

# ALPHA ANALYTICAL, INC.

## Quality Assurance Manual

### Volume I

Rev. 15

January, 2007



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# **Section 1**

## **Quality Assurance Manual Identification Form**

Alpha Analytical, Inc.  
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Page: 1 of 1

### QUALITY ASSURANCE MANUAL IDENTIFICATION FORM

Document Title: Quality Assurance Manual for Alpha Analytical Inc.

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Plan Coverage: This is a document describing Alpha Analytical's Quality Assurance Plan (QAP). The plan covers all environmental chemistry data generated from samples submitted to Alpha for analysis. The coverage in this plan will be as resources and priorities allow.

Laboratory Approval:

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# **Section 2**

## **T**able of Contents

**TABLE OF CONTENTS**  
**QA PLAN ELEMENTS**  
**VOLUME I**

<b>SECTION</b>	<b>TITLE</b>	<b>PAGE #</b>	<b># OF PAGES</b>
1.0	QUALITY ASSURANCE MANUAL IDENTIFICATION FORM	1 - 1	1
2.0	TABLE OF CONTENTS	2 - 1	4
3.0	STATEMENT OF POLICY	3 - 1	13
4.0	ORGANIZATION AND RESPONSIBILITY	4 - 1	8
5.0	QUALITY ASSURANCE ROUTINES TO ASSESS PRECISION, ACCURACY AND THE CALCULATION OF METHOD DETECTION LIMITS	5 - 1	9
6.0	SAMPLING PROCEDURES	6 - 1	13
7.0	SAMPLE CUSTODY	7 - 1	7
8.0	ANALYTICAL PROCEDURES	8 - 1	26
9.0	CALIBRATION PROCEDURES AND FREQUENCY	9 - 1	58
10.0	EQUIPMENT AND INSTRUMENT MAINTENANCE	10 - 1	14
11.0	QUALITY CONTROL PROCEDURES TO ASSESS LABORATORY DATA	11 - 1	60
12.0	DATA REDUCTION, REVIEW, AND STORAGE	12 - 1	10
13.0	CORRECTIVE ACTIONS AND CONTROL OF NONCONFORMING ENVIRONMENTAL TEST WORK	13 - 1	12
14.0	SYSTEM AND TECHNICAL AUDITS	14 - 1	21
15.0	QUALITY ASSURANCE REPORTS	15 - 1	9
16.0	LABORATORY REPORTS AND REPORTING PROCEDURES	16 - 1	8
17.0	LIST OF ACRONYMS AND ABBREVIATIONS	17 - 1	7
18.0	GLOSSARY OF TERMS	18 - 1	11

**TABLE OF CONTENTS**  
**QA PLAN ELEMENTS**  
**VOLUME II**

<b>SECTION</b>	<b>TITLE</b>	<b>PAGE #</b>	<b># OF PAGES</b>
<b>APPENDIX A</b>			
A.1	TECHNICAL PERSONNEL RESUMES	A - 1	49
<b>APPENDIX B</b>			
B.1	FIELD SAMPLING PLAN	B.1 - 1	1
B.2	FIELD SAMPLING DOCUMENTATION	B.2 - 1	1
B.3	QUALITY CONTROL FIELD SAMPLES	B.3 - 1	3
B.4	VOLATILE SAMPLING TECHNIQUES 524.2	B.4 - 1	3
B.5	VOLATILE SAMPLING TECHNIQUE - WATER (624/8260)	B.5 - 1	2
B.6	SOIL AND SEDIMENT SAMPLING TECHNIQUES	B.6 - 1	1
B.7	SVOC SAMPLING TECHNIQUE - WATER	B.7 - 1	2
B.8	GROUND WATER SAMPLING - MONITORING WELLS	B.8 - 1	2
B.9	FIELD DECONTAMINATION AND WASTE DISPOSAL	B.9 - 1	2
B.10	SAMPLE PACKING AND TRANSPORTATION	B.10 - 1	2
<b>APPENDIX C</b>			
C.1	SAMPLE TRACKING PLAN	C.1 - 1	1
C.2	SAMPLE IDENTIFICATION	C.2 - 1	2
C.3	LABELING FIELD SAMPLES	C.3 - 1	2
C.4	SAMPLE RECEIVING AND PROJECT COMMUNICATION	C.4 - 1	8
C.5	SAMPLE CONTAINERS, PRESERVATION, HOLDING TIME AND GENERAL SAMPLE RECEIPT PROTOCOLS	C.5 - 1	7
C.6	SAMPLE ACCEPTANCE POLICY	C.6 - 1	3
C.7	MANUAL CHAIN-OF-CUSTODY PROCEDURES	C.7 - 1	3
C.8	LIMS GENERATED CHAIN-OF-CUSTODY PROCEDURES	C.8 - 1	4
C.9	INTERNAL CHAIN-OF-CUSTODY PROCEDURES	C.9 - 1	5
C.10	SAMPLE LOG-IN LEDGER	C.10 - 1	3
C.11	SAMPLE RECEIPT STORAGE	C.11 - 1	2

C.12	SAMPLE TRACKING	C.12 - 1	4
C.13	MAINTENANCE OF CUSTODY	C.13 - 1	1
C.14	E-MAILING FINAL ANALYTICAL DATA	C.14 - 1	3
C.15	SAMPLE SCHEDULING AND DISCREPANCY REPORTING	C.15 - 1	1
C.16	WASTE DISPOSAL OPERATIONS	C.16 - 1	19
C.17	REVIEW OF REQUESTS, TENDERS AND CONTRACTS	C.17 - 1	3

#### APPENDIX D

D.1	DOCUMENT CONTROL PLAN (DCP)	D.1 - 1	3
D.2	DOCUMENT CONTROL FILING SYSTEM	D.2 - 1	5
D.3	LABORATORY SAMPLE DOCUMENT FLOW	D.3 - 1	2
D.4	PROCEDURE FOR THE PREPARATION, REVIEW, APPROVAL, REVISION, AND DISTRIBUTION OF SOPS AND OTHER TECHNICAL DOCUMENTS.	D.4 - 1	6
D.5	LABORATORY TRAINING PROGRAM	D.5 - 1	17
D.6	EMPLOYEE SIGNATURE/INITIAL LOG	D.6 - 1	2
D.7	INSTRUMENT SEQUENCE LOGBOOK RECORD KEEPING PROCEDURE	D.7 - 1	1
D.8	INSTRUMENT SEQUENCE AND MAINTENANCE LOGBOOK	D.8 - 1	1
D.9	ANALYTICAL BALANCE LOGBOOK	D.9 - 1	3
D.10	SAMPLE EXTRACTION LOG	D.10 - 1	3
D.11	ANNUAL THERMOMETER CALIBRATION PROCEDURE	D.11 - 1	5
D.12	IR TEMPERATURE PROCEDURE	D.12 - 1	7
D.13	TEMPERATURE LOG	D.13 - 1	4
D.14	PROCEDURE FOR COLD STORAGE TEMPERATURE EXCURSIONS	D.14 - 1	1
D.15	REFRIGERATOR DOCUMENT CONTROL	D.15 - 1	4
D.16	MECHANICAL VOLUMETRIC DISPENSING DEVICES (MVDD) PROCEDURE	D.16 - 1	3

#### APPENDIX E

E.1	ANALYTICAL AND EXTRACTION SUPPORT PROCEDURES	E.1 - 1	1
E.2	DISHWASHER AND STEAM SCRUBBER OPERATIONS	E.2 - 1	1
E.3	MANUAL GLASSWARE CLEANING PROCEDURE	E.3 - 1	1

E.4	SAMPLE CONTAINER CLEANING PROCEDURE	E.4 - 1	3
E.5	PREVENTION OF SAMPLE CONTAMINATION	E.5 - 1	2
E.6	STANDARD PREPARATION PROCEDURE	E.6 - 1	18
E.7	STORAGE BLANK PROCEDURE	E.7 - 1	4
E.8	A PRACTICAL APPLICATION GUIDE FOR PERFORMING A DETERMINATION OF CAPABILITIES (DOC) AND METHOD DETECTION (MDL) STUDY	E.8 - 1	15
E.9	POLICY FOR MANUAL & AUTOMATED INTEGRATION PROCEDURES	E.9 - 1	15
E.10	PREPARATION OF REAGENT GRADE WATER	E.10 - 1	7
E.11	SUBSAMPLING PROCEDURES AND SAMPLE COMPOSITING	E.11 - 1	8
E.12	A PRACTICAL APPLICATION GUIDE FOR PERFORMING INSTRUMENT CALIBRATION, CALIBRATION MODEL DETERMINATION AND CALIBRATION VERIFICATION	E.12 - 1	15
APPENDIX F			
F.1	SOFTWARE QUALITY ASSURANCE PLAN	F.1 - 1	1
F.2	COMPUTER SOFTWARE OPERATIONS	F.2 - 1	5
F.3	DATA COLLECTION & STORAGE	F.3 - 1	2
F.4	DATA FILE UPLOADING	F.4 - 1	8
F.5	ELECTRONIC DISKETTE DELIVERABLES (EDDs)	F.5 - 1	2
F.6	MSACCESS2003 DATABASES	F.6 - 1	3
F.7	DATA ARCHIVING	F.7 - 1	4
F.8	PC/SERVER INTEGRITY & SOFTWARE VALIDATION	F.8 - 1	2
F.9	SAMPLE LOG IN (COMPUTER ENTRY)	F.9 - 1	3
F.10	SAMPLE PREPARATION OMEGA SOP	F.10 - 1	3
APPENDIX G			
G.1	LABORATORY ETHICS/FRAUD PREVENTION AND DATA INTEGRITY PROGRAM	G.1 - 1	8
APPENDIX H			
H.1	STATE CERTIFICATION AND PARAMETERS OF ANALYSIS	H.1 - 1	3



# **Section 3**

## **Statement of Policy**

**ALPHA ANALYTICAL, INC.**  
**QUALITY ASSURANCE MANUAL**

**3.0 INTRODUCTION**

- 3.0.1 Alpha Analytical, Inc. is an analytical laboratory specializing in the analysis of environmental samples. The staffing, methods, and quality control procedures are designed for the determination of analytes in diverse environmental matrices.
- 3.0.2 This quality assurance manual describes our quality systems incorporating laboratory activities so as to provide the client with data of known and documented quality.
- 3.0.3 This system includes the description of analytical methods, how methods are continuously monitored and evaluated and how their performance is documented.
- 3.0.4 Our quality assurance program is designed to meet the requirements established in the use of Performance Based Measurements Systems (PBMS) in environmental testing and to provide the foundation for PBMS implementation of these standards.
- 3.0.5 References

The following two references provides the foundation of this Quality Assurance (QA) manual to include the standardization and organization while provided guidance on the implementation of our quality systems.

3.0.5.1 National Environmental Laboratory Accreditation Conference's (NELAC)  
Chapter 5 Quality System, Version 3, June 2003.

3.0.5.1 Quality Systems Manual (QSM) for Environmental Laboratories, Department  
fo Defense, Final Version 3, January 2006.

Note: NELAP and DOD incorporated the requirements of ISO 9001 and ISO 9002 that are relevant to the scope of environmental testing services into their quality systems documents.

Therefore, by complying with NELAC and DOD/QSM, Alpha's quality system have been established in accordance with ISO 9001 and ISO 9002.

**3.1 STATEMENT POLICY FROM MANAGEMENT**

- 3.1.1 It is Alpha's policy and commitment from top management to perform all activities under good professional practices and to produce quality analytical data while

servicing our clients under the guidelines of NELAC as described in this Quality Assurance Manual.

- 3.1.2 It is Alpha's policy and commitment from top management to define our quality assurance policies and document our quality control objectives in this Quality Assurance Manual.
- 3.1.3 It is Alpha's policy and commitment from top management to provide the necessary guidance and to require all personnel concerned with environmental testing activities to thoroughly familiarize themselves with the quality documentation and to implement the policies and procedures in their work.
- 3.1.4 This commitment includes Alpha's CEO, President, Laboratory Director, Laboratory Manager and QA Officer.

## **3.2 PURPOSE**

The purpose of the QA program is to:

- a) Provide a consistent framework for the generation of analytical data in support of the programs enforced by various regulatory agencies;
- b) Establish standard practices which permit inter-laboratory comparison of data; and,
- c) Establish procedures for demonstrating that analytical systems are in control.

## **3.3 OBJECTIVES**

More specifically, the objectives of the QA program are to:

- a) Provide a uniform basis for sample handling, instrument conditions, methods control, performance evaluations, and analytical data generation and reporting that will provide legally defensible results in a court of law;
- b) Estimate the quality of each analytical system, which includes Precision, Accuracy, Representativeness, Comparability, and Completeness, "PARCC", that is sufficient for the needs of each project;
- c) Assist in the early recognition of deficiencies which affect the quality of data;
- d) Enable Alpha Analytical to identify and implement actions that are necessary to ensure the validity of laboratory data; and,
- e) Require sufficient documentation to verify the quality of data submitted.

### **3.4 SCOPE**

3.4.1 This document outlines the purpose, policies, organization, and operations of the QA Program established to support chemical analyses conducted for various programs and projects. All routine laboratory tasks have written Standard Operating Procedures (SOP's) to increase production uniformity, and are consistent with sound scientific principles. Advances in quality control procedures will be reflected in updated versions of this plan.

3.4.2 Implementation of this QA plan will help ensure the validity of data and provide a reliable foundation on which to base decisions. It is a basic policy of Alpha Analytical to provide accurate, precise, complete, and representative determinations of chemical constituents in submitted samples, and to sufficiently document the analytical QC procedures.

3.4.3 In implementing the QA Program, it is important to understand the difference between QA and QC.

QA refers to the system through which the organization provides assurance that monitoring of quality-related activities has occurred. Frequently, QA is interpreted as a record-keeping system to ensure documentation of all activities, including traceability, completeness, and security of documents.

QC refers to specific actions taken to guarantee that system performance is consistent with established limits regarding accuracy, precision, and comparability of results. QC activities are conducted within a system of QA for proof that QC exists.

3.4.4 Implementation of the QA Program is designed to assure data are collected under in-control conditions, rather than assuring documentation of poorly conducted analyses. The QA Program is intended to establish a QA system and proper QC guidelines and procedures.

### **3.5 APPLICATION**

3.5.1 The emphasis of this QA Program is on activities which generate analytical data, and includes those aspects of field sampling that may affect the integrity of samples. Alpha is not a sampling company; and therefore, have no ultimate control over the QA/QC procedures conducted in the field. Therefore our role is one of advising field samplers as to the acceptable practices.

3.5.2 Specific requirements are provided for sampling and chemical analysis of groundwater, surface water, soil, and sediment samples. In general, principles described herein are applicable to most field/laboratory activities.

- 3.5.3 This program has been written and designed specifically for Alpha Analytical and represents the system used by our staff during normal operating conditions. The QA plan may be superseded or appended with different or additional QA/QC activities related to a specific project or Statement of Work (SOW).
- 3.5.4 If a SOW or Quality Assurance Project Plan (QAPP) is used to supplement or append this QA/QC plan, then the Data Quality Objectives (DQO's) should be reflected in that particular QA/QC data package when requested.

### **3.6 GENERAL POLICIES**

#### **3.6.1 Ethics and Data Integrity Policy**

The QA Manual and EPA methods are written as a set of policies and procedures that define what laboratory personnel are required to do; however, our ethics policies and our Laboratory Ethics Program, Appendix G, is written to ensure that employees are educated as to what they are not allowed to do.

Our data integrity training program includes discussions regarding the critical need for honesty, full disclosure in all analytical reporting and other related data integrity issues.

3.6.1.1 It is Alpha's policy to provide ethics and data integrity training to all new employees and provide this training to current employees on an annual basis.

3.6.1.2 Alpha Analytical has a zero tolerance policy regarding unethical situations, scientific misconduct and intentional lack of compliance with required procedures.

3.6.1.3 It is Alpha's policy to encourage laboratory personnel to come forward and report inappropriate activities.

3.6.1.4 It is managements philosophy and Alpha's policy to be proactive in the training, and education in the prevention of improper, unethical or illegal actions.

3.6.1.5 It is managements philosophy and Alpha's policy to clearly document how all analytical values were obtained and to supply the data user all data necessary to re-create and/or document final data results.

#### **3.6.2 Manual Integration Policy**

Manual integration is an important subsection of data integrity, but is delineated to bring attention and to highlight the importance of this issue. Our manual integration training program includes discussions regarding the critical need for honesty, full

disclosure in all analytical reporting and other related data integrity issues.

3.6.2.1 It is Alpha's policy to produce analytical data using automated and manual integration practices, in a manner that is non-arbitrary (meaning standards, control samples, and client samples are all integrated using consistent integration parameters).

3.6.2.2 It is Alpha's policy to produce analytical data using automated and manual integration practices, in a manner that is rational and can be backed up with the reason for a particular integration practice.

3.6.2.3 It is Alpha's policy to encourage laboratory personnel to come forward and report inappropriate activities.

### 3.6.3 Subcontract Laboratories

3.6.3.1 It is Alpha's policy to subcontract out analytical services not performed by Alpha, to laboratories that have been certified for those methods, to the best of our ability.

3.6.3.2 It is Alpha's policy, when subcontracting work because of unforeseen reasons (e.g., workload, need for further expertise or temporary incapacity, etc.), this work is placed with:

- a) a laboratory accredited under NELAP for the tests to be performed, or
- b) with a laboratory that meets applicable statutory and regulatory requirements to perform the required analytical testing.

3.6.3.3 It is Alpha's policy to first subcontract analytical services, not performed by Alpha, to those laboratories that have been requested by the client and when possible, gain the approval of the client in writing.

3.6.3.4 If for any reason the subcontract laboratory is unable to perform the duties, then Alpha will procure an alternate laboratory of our choosing.

3.6.3.5 It is Alpha's policy to maintain a register of subcontract laboratories and a record of the evidence of appropriate certification.

3.6.3.6 It is Alpha's policy, to report all data issued by the subcontract laboratory on their official letter head and not retype and or reissue hard copy data on Alpha's letter head. If this can not be accomplished, the subcontract

laboratory data must be clearly labeled as to the laboratory conducting the analysis.

3.6.3.7 It is the subcontract laboratories responsibility and business liability to ensure they have and maintain the appropriate State Certifications, method and compound certifications, program certifications and any other certifications or approvals necessary to perform the related tasks. It is the responsibility of all subcontract laboratories to ensure Alpha has been notified of those samples that cannot be analyzed due to:

- Inadequate sample / extraction holding time;
- Inadequate sample volume;
- Inappropriate sampling procedures;
- Inappropriate sample containers/preservatives;
- Loss of instruments or power failure;
- Loss of certifications or approvals to perform the requested task; or,
- Any other circumstances that would prevent the appropriate or adequate analysis of those affected samples.

3.6.3.8 It is Alpha's policy not to assume any liability of any subcontract laboratory data or the preponderance of the defenseability, documentation or data quality produced by any subcontract laboratory. This is the responsibility of the subcontract laboratory and their respective certifying agents or authorities.

#### 3.6.4 Training Policy

3.6.4.1 It is Alpha's policy to hire personnel which have appropriate education and/or On-the-Job-Training (OJT) adequate to perform their job duties.

3.6.4.2 It is Alpha's policy to conduct a training program that includes initial and continuing training of laboratory personnel.

3.6.4.3 It is Alpha's policy to ensure the competence of technical staff personnel who operate analytical equipment, evaluate results, and sign test reports.

However, it is the responsibility of the trainee to ensure they have received adequate initial and continuing training and the documentation of that training to achieve and maintain skills commensurate with their responsibilities.

#### 3.6.5 Policy for the Procurement of Supplies and Materials

3.6.5.1 It is Alpha's policy to *evaluate* supply vendors, chemicals, reagents and any

other supplies which are critical to method performance to include additional testing to verify their quality before use.

Supply vendors are typically required to provide information to substantiate their ability to provide suitable quality e.g., Certificates of Analysis ( C of As), and their mechanism to assure consistent delivery.

3.6.5.2 It is Alpha's policy to *purchase* equipment, services and material supplies that affect the quality of environmental testing, at a level which will meet and/or exceed method/project criteria. These services and supplies are typically thought of as:

- Instruments,
- Solvents,
- Reagents,
- Gasses,
- Reference material,
- Sample containers, and
- Other laboratory supplies.

3.6.5.3 If a method specifies a particular brand name, it is Alpha's policy to purchase the item that meets or exceeds method specification, but not necessarily those brand name items specified in the method.

3.6.5.4 If no industry standard is available which specifies the appropriate quality grade, then it is Alpha's policy to determine what testing may be required to evaluate usability of supplies and materials.

#### 3.6.5.5 Material Supplies

- a) It is Alpha's policy to purchase solvents, reagents, etc, which does not contain any target analytes observed at greater than one-half of the targeted reporting limit and no interfering non-target analytes at the reporting limit.
- b) Once chemical purity, grade or quality has been established, it is Alpha's policy to repurchase these same items and document their continuing quality with the associated certificate of analysis for historical records.
- c) Materials and supplies which have associated certificate of analysis will be inspected upon receipt. If the quality of supplies or materials are suspect, certain actions will be required on the part of the analyst or extraction chemist.



- i. The analyst/extraction chemist will first verify the lot number and C of A of the material in question and assure that it has met vendor/supplier specification.
- ii. Secondly, the analyst/extraction chemist will document the quality issue and inform the Laboratory Director, QA Officer and Purchasing Agent of the problem and any additional correction taken on his/her part.
- iii. Thirdly, the Laboratory Director or QA Officer, will take the necessary actions to rectify and document the problem and the solution to the problem.

3.6.5.6 It is Alpha's policy to maintain purchasing documents for items affecting data quality. These items are reviewed and approved for technical content by the purchasing agent prior to release. Such items are as follows:

- a) Materials such as standards, reagents, solvents, chemicals, etc. are documented with:
  - i. date of receipt,
  - ii. expiration date (if applicable),
  - iii. identification of vendor or manufacturer,
  - iv. lot number, and
  - V. certificate of analysis (if applicable.)

See the Preparation of Standards SOP for additional details.

- b) Services such as balance calibration are documented by the vendors certificates as described in the Analytical Balance Logbook SOP.
- c) The documentation of accuracy and precision for S class weights is described in the Analytical Balance Log Book SOP.
- d) The documentation of accuracy and precision for Class A glassware and manual volumetric dispensing devices is described in the Manual Volumetric Dispensing Device SOP.
- e) The documentation of accuracy and precision for thermometers is described in the Annual Thermometer Calibration SOP.

### 3.6.6 Computer Hardware/ Software Operation Policy

It is the policy of the laboratory, that the computer department has the responsibility for all software loading, upgrades, coding changes, debugging and hardware/software retirements.

It is Alpha's policy to ensure that:

- a) policies and procedures are in place to protect the clients' electronic storage and transmission of results;
- b) Only authorized, trained personnel are allowed to perform these functions;
- c) Only authorized software is to be loaded on any company computer;
- d) Only authorized, trained personnel are allowed to use company connections to the network or internet; and
- e) No hardware, software, or raw data be removed from the laboratory without without written authorization.

### 3.6.7 Policy for Testing of Proficiency Evaluation (PE) Samples

3.6.7.1 It is Alpha's policy to ensure adequate quality control procedures are in place for monitoring the validity of environmental testing through the participation of a proficiency-testing program.

3.6.7.2 It is Alpha's policy to participate in two single-blind, single-concentrate Proficiency Testing (PT) studies, per year for each field of proficiency testing to maintain accreditation.

3.6.7.3 It is Alpha's policy to obtain PE samples from a Proficiency Testing Oversight Body (PTOB) / Proficiency Test Provider Accreditor (PTPA) approved PT provider.

3.6.7.4 It is Alpha's policy to analyze and submit PE sample results to the PT provider within their allotted time schedule as defined by the PT provider.

3.6.7.5 It is Alpha's policy to analyze and treat PE samples in a manner consistent with real environmental samples using the same staff, methods, procedure, equipment, facilities, etc. as used for routine analysis of that sample.

3.6.7.6 It is Alpha's policy that all staff members comply with the following restrictions on the transfer of PE samples and communication of PE sample

results prior to the time results of the study are released:

- a) No staff member shall send any PE sample to another laboratory for an analysis for which accreditation is being requested;
- b) No staff member shall knowingly receive any PE sample or portion of a PE sample from another laboratory for any analysis for which the sending laboratory is seeking accreditation;
- c) No staff member shall communicate with any individual at another laboratory concerning the PE sample; and,
- d) No staff member shall attempt to obtain assigned values of any PE sample from the PE provider.

#### 3.6.8 Policy of Laboratory Organization and Staffing

It is Alpha's policy to have an organization that:

- a) has sufficient managerial and technical staff personnel with the authority and resources needed to carry out their duties and to identify the occurrence of departures from the quality system or from the procedures for performing environmental tests, and to initiate actions to prevent or minimize such departures;
- b) has processes to ensure that management and staff personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work;
- c) has policies and procedures to ensure the protection of client's confidential information and proprietary rights;
- d) has policies and procedures to avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgement or operational integrity;
- e) defines the organization and management structure of the laboratory, and the relationship between quality management, technical operations, and support services;
- f) specifies the responsibility, authority and interrelationships of personnel who manage, perform or verify work affecting quality of the environmental tests;

- g) provides adequate supervision of environmental testing staff, including trainees, by persons familiar with methods and procedures, purpose of each environmental test, and with the assessment of the environmental test results;
- h) in powers the technical management with the overall responsibility for the technical operations and for providing the resources needed to ensure the required quality of laboratory operations;
- i) has a member of the technical staff appointed as the quality assurance officer who, irrespective of other duties and responsibilities, has the defined responsibility and authority for ensuring that the quality system is implemented and followed at all times;
- j) appoints deputies for key managerial personnel, including the technical director and quality assurance officer; and
- k) participates in a proficiency testing program to qualify for and maintain accreditation.

It is Alpha's policy to ensure the quality assurance officer has direct access to the highest levels of management at which decisions are made on laboratory policy or resources.

### 3.6.9 Waste Disposal Policy

Handling and disposal of wastes received or generated by Alpha requires a thorough understanding of the many waste streams within Alpha. Proper handling is important for obvious health concerns and the proper disposal is important to insure Alpha meets the requirements and documentation set forth by the Resource Conservation and Recovery Act (RCRA).

3.6.9.1 A complete description of our Hazardous Waste SOP's are found in Appendix C. There are two basic types of wastes within Alpha: 1) Hazardous, and 2) Non-hazardous. The important distinction between the two is that **all** hazardous waste generated at the 255 Glendale Avenue laboratory has to stay within that facility. **NO** hazardous waste can be generated, then moved to the Freeport facility behind the laboratory.

### 3.6.10 Change in Ownership / Business Termination

For a change in ownership the following policies are to be met:

3.6.10.1 Alpha agrees to be accountable for any analyses, data and reports

generated up to the time of legal transfer of ownership; and,

- 3.6.10.2 The buyer must agree in writing to be accountable for any analyses, data and reports generated after the legal transfer of ownership occurs.

It is Alpha's policy to ensure that analytical records are maintained or transferred according to the clients instructions, if either a change in ownership or business termination were to occur.

### 3.6.11 Service to Clients

The environmental testing business is a service oriented business, requiring a large amount of interaction with our clients. It is in our best interest, to emphasis the importance of conducting client communication in an environment that is professional, informational and confidential.

- 3.6.11.1 It is Alpha's policy to cooperate with our clients or their representatives to clarify the client's request and to monitor the analytical performance in relation to the work performed on their project, and to provide this service in a climate that ensures confidentiality to other clients.

- 3.6.11.2 Service to clients is a proactive engagement with our clients require staff to notify clients of problem situations such as:

- a) incorrect or improper method requests;
- b) the need to optimize methods to ensure data quality objectives are met for difficult matrix or poor performing analytes;
- c) lack of project guidance; and
- d) problems with sampling or analysis that may impact results (e.g., improper preservation of sample).

### 3.6.12 Customer Complaints

- 3.6.12.1 It is Alpha's policy to respond to complaints and/or problems in a reasonable time frame and in a courtesy manner that is both polite and professional to the customer.

Alpha Analytical, Inc.  
Section No.: 3.0  
Revision No.: 15.0  
Date: January, 2007  
Page: 13 of 13

The documentation of customer complaints, the response to these complaints, and their resolution is useful information to improving the quality of our client service. This information, as part of our quality system, helps identify patterns of problems and is important in formulating a corrective response to those problems.

# **Section 4**

## **Organization and Responsibility**

## **4.0 ORGANIZATION AND RESPONSIBILITY**

### **4.1 INTRODUCTION**

- 4.1.1 Alpha Analytical, Inc. is a business entity (environmental laboratory) incorporated in the state of Nevada that is legally responsible for all activities performed at this laboratory.
- 4.1.2 The guidelines described in this manual have been developed to ensure data quality is documented and controlled and data responsibilities are delegated and executed.
  - 4.1.2.1 The Laboratory Director is ultimately responsible for the quality of data collected and reported in support of the various programs.
  - 4.1.2.2 The Laboratory Manager is responsible for the implementation of the policies and procedures and for overseeing key operations within the laboratory.
  - 4.1.2.3 The Quality Assurance Officer (QAO) is responsible for ensuring the quality system is implemented and followed at all times.
  - 4.1.2.4 Staff members are responsible for assuming the accountability and reliability of the generated data.
- 4.1.3 All Alpha Analytical employees are trained and have a clear understanding of the laboratory's responsibility for producing data that are accurate, complete, documented, and meet the requirements of precision, representativeness, and comparability.

All employees have access to Alpha's Quality Assurance Manual and are trained on the specific portions that apply to their responsibilities.

All employees participate in on-going discussions of the lab's quality assurance procedures and are trained in the importance of the quality control data acquired during normal sample analysis.

### **4.2 RESPONSIBILITIES AND AUTHORITY**

It is Alpha's policy to ensure the laboratory has sufficient managerial and technical staff personnel to support the analytical process and to identify the occurrence of departures from the quality system, and to initiate actions to prevent or minimize such departures.

Personnel are assigned responsibilities delineating their specific jobs, i.e., clerical, analysis, and sample preparation.

The relationships between the organization and the responsibilities involved in Alpha Analytical's QA/QC are outlined below. The following descriptions assign certain tasks to



various levels of responsibility. The organizational chart is show in Table 4-1. The resumes of Alpha Analytical's technical employees are included in Appendix A.

#### 4.2.1 Laboratory Director

The Laboratory Director is responsible for the overall collection and analysis of data produced and reported by Alpha Analytical. The following describes some of the more important duties that are performed by the Laboratory Director. These duties include:

- a) Ensures that all analytical activities are performed according to methods and protocols specified in the QA Manual;
- b) Ensures that daily operations function smoothly within the operating conditions and guidelines established by Alpha Analytical;
- c) Coordinates analytical work to ensure the completion of tasks are within established time frames;
- d) Ensures the analytical data review process is functioning properly;
- e) Oversees preventative maintenance activities;
- f) Evaluates and implements changes in methods and quality control measures;
- g) Identifies quality control problems and takes measures to correct or eliminate the problem source;
- h) Assumes the responsibility for determining the level of qualification, experience and skills necessary for staffing all positions in the laboratory to perform the technical duties with the required quality control;
- i) Ensures that the training of each member of the technical staff is kept current;
- j) Ensures that all technical laboratory staff members have demonstrated capability in the activities for which they are responsible, such as IDC and MDL studies;
- k) Oversees personnel and evaluates their performance; and
- l) Ensures the laboratory is participating in a proficiency testing program and that corrective actions are implemented after testing and evaluating the effectiveness of the corrective actions.

#### 4.2.2 Laboratory Manager

The Laboratory Manager is responsible for the implementation of the policies and procedures as described by the QAM with the direction of the Laboratory Director. The Laboratory Manager is responsible for overseeing key operations within the laboratory to ensure the work flow is efficient, balanced and within the guidelines established in the QAM. The following describes some of the more important duties that are performed by the Laboratory Manager. These duties include:

- a) Ensures the Sample Custody Officer has the appropriate staffing, training and experience necessary to carry out the responsibility for receiving and logging in samples as they arrive at the laboratory;
- b) Ensures the Document Control Officer has the appropriate staffing, training and experience necessary to carry out the responsibility for implementing the Document Control Program;
- c) Ensures that the Director of Client Services, Technical Representatives and Project Coordinators have the appropriate staffing, training, experience and/or education necessary to carry out their responsibilities and duties;
- d) Ensures that the Laboratory Information Management (LIMS) Administrator has the appropriate staffing, training and experience necessary to carry out the responsibility of implementing the Software Quality Assurance Plan (SQAP);
- e) Coordinates with the Laboratory Director and the QA Officer to assure all activities, both sampling and analysis are performed according to the specific QA Project Plans (QAPP), methods and protocols specified in this QA Plan;
- f) Ensures that daily operations function smoothly within the operating conditions and guidelines established by Alpha Analytical;
- g) Ensures that completion of all tasks are within the established time frames;
- h) Ensures the laboratory is participating in a proficiency testing program and that corrective actions are implemented after testing and evaluating the effectiveness of the corrective actions; and,
- i) Responsible for the assessing, selecting and use of subcontract laboratories to meet project specific criteria.

#### 4.2.3 Quality Assurance Officer

4.2.3.1 The Quality Assurance Officer (QAO) has the responsibility and authority for ensuring that the quality system is implemented and followed at all times. The QA Officer:

- 1) is the focal point for QA/QC and is responsible for the oversight and/or review of quality control data;
- 2) is independent from laboratory operations;
- 3) objectively evaluates laboratory data and performs assessments without outside managerial influence;
- 4) has documented training and experience in QA/QC procedures and is knowledgeable in the quality system as defined under NELAC;
- 5) has a general knowledge of the analytical test methods for which data review is performed;
- 6) arranges for and/or conducts internal audits annually; and,
- 7) notifies laboratory management of deficiencies in the quality system and monitors corrective actions.

4.2.3.2 In addition, the QA Officer is responsible for reviewing and advising on all aspects of QA/QC, including the following:

- a) assisting the data requester in specifying the QA/QC procedure to be used during the testing program;
- b) makes recommendations to the data requester and Laboratory Director, if problems are detected, to ensure that appropriate corrective actions are taken;
- c) oversees the review of quality control data to determine if test data is acceptable;
- d) updates and supervises the updates of quality control markers, such as accuracy, precision, and method detection limits;
- e) Coordinates and oversees the preparation of quality assurance plans;
- f) reviews new or proposed protocols to determine appropriate use;
- g) reviews method validation data; and
- h) ensures continuous improvement at the laboratory through the use of control charts and other method performance indicators.

#### 4.2.4 Analysts

4.2.4.1 Analysts are primarily responsible for ensuring they are completely familiar with the quality systems documentation and the implementation of the policies and procedures affecting their work. The following describes some of the more important responsibilities and duties that are performed by the analysts. These responsibilities include:

- 1) Analysts are responsible for ensuring that they perform the required analyses according to test methods specified by rule, permit, QAPPS and/or SOPs;
- 2) Analysts are responsible for ensuring that the instrument and related equipment is working to acceptable standards; and,
- 3) Analysts are responsible for ensuring the required supplies are available for their particular instrument.

4.2.4.2 Some of the various duties that analysts perform include the following:

- a) Ensures that all analytical equipment has been properly calibrated before beginning analysis;
- b) Ensures that all identifying information, including sample identification numbers, have been accurately transcribed into records or computer databases;
- c) Ensures that all calculations are correct;
- d) Ensures that appropriate confirmatory tests or procedures have been completed;
- e) Identifies, documents, and begins corrective actions on any quality control problem that relates to the analytical test; and,
- f) Maintains equipment in working condition and documents all preventative maintenance and repairs.

#### 4.2.5 Extraction Technicians

Extraction Technicians are responsible for a large number of duties and activities which support personnel performing sample analysis. These activities are necessary to ensure that quality extracts are prepared for instrumental analysis.

Extraction Technicians are responsible for ensuring they are completely familiar with the quality systems documentation and the implementation of the policies and

procedures affecting their work. The following describes some of the more important responsibilities and duties that are performed by the extraction technician. These duties and responsibilities include:

- a) Performs the required extraction, clean-up procedures, and final concentration steps according to the test methods used by Alpha Analytical;
- b) Ensures that all extraction equipment is properly maintained before and after use;
- c) Ensures that all analytical equipment, such as pH meters and balances, have been properly calibrated before use;
- d) Ensures that all identifying information, including sample identification numbers, have been accurately transcribed into the extraction logs and other pertinent areas;
- e) Documents with meticulous accuracy all procedures performed on the sample and notes any irregularities observed during the sample extraction which may affect the analysis; and,
- f) Communicates to analysts and all affected personnel irregularities or observations of the sample prior to analysis to ensure that proper decisions are made regarding that particular sample.

#### 4.2.6 Sample Custody Officer

The Sample Custody Officer (SCO) has one basic area of responsibility, which is ensuring that the proper handling and documentation of all samples are performed by the person who has legal custody of that sample during all phases of laboratory work. The SCO performs the following duties:

- a) Assumes the responsibility for receiving and logging in samples as they arrive at the laboratory;
- b) Obtains the documentation required to complete the chain of custody form for each specific sample;
- c) Notes any irregularities of the sample and/or inquires of the client regarding these abnormalities;
- d) Notes special project requirements with detailed information on the chain of custody;
- e) Informs all personnel affected by special projects of the requirements and

that the project is in-house; and,

- f) Assumes responsibility for placing environmental samples in the proper storage area to prevent possible sample cross-contamination.

#### 4.2.7 Document Control Officer

The Document Control Officer (DCO) is responsible for implementing, updating, and maintaining Alpha Analytical's Document Control Program. The Document Control Officer is primarily responsible for overseeing data assembly and documentation of client files. The DCO is responsible for the following areas:

- a) Ensures that all documents concerning a client/sample file are accounted for when a project is completed;
- b) Responsible for the organization and assembly of all documents related to a client sample file according to established SOP's;
- c) Maintains control of confidential information;
- d) Directs and coordinates the Document Control Program; and,
- e) Reports directly to the Laboratory Manager.

#### 4.2.8 Laboratory Information Management System (LIMS) Administrator

The LIMS Administrator is responsible for implementing, updating and maintaining Alpha Analytical's Software Quality Assurance Plan (SQAP). When computers are used for the capture, processing, manipulation, recording, reporting, storage and retrieval of analytical data, the LIMS Administrator ensures the following:

- a) All requirements of the SQAP are being met;
- b) Computer software is documented and adequate for use;
- c) Ensures procedures are established and implemented for protecting the integrity of data, such as data entry or capture, data storage, data transmission and data processing;
- d) Ensures computer equipment is adequately maintained to function properly;
- e) Establishes and implements appropriate procedures for the maintenance and security of data including the prevention of unauthorized access to, and the unauthorized amendment of computer records.

#### 4.2.9 Training Coordinator

This person is responsible for the oversight of the training program and the maintenance of training and qualification records. This person coordinates the training program by updating Trainers and/or the Training Committee Members of when and what type of training is needed, and the overall recommendations to the Training Program.

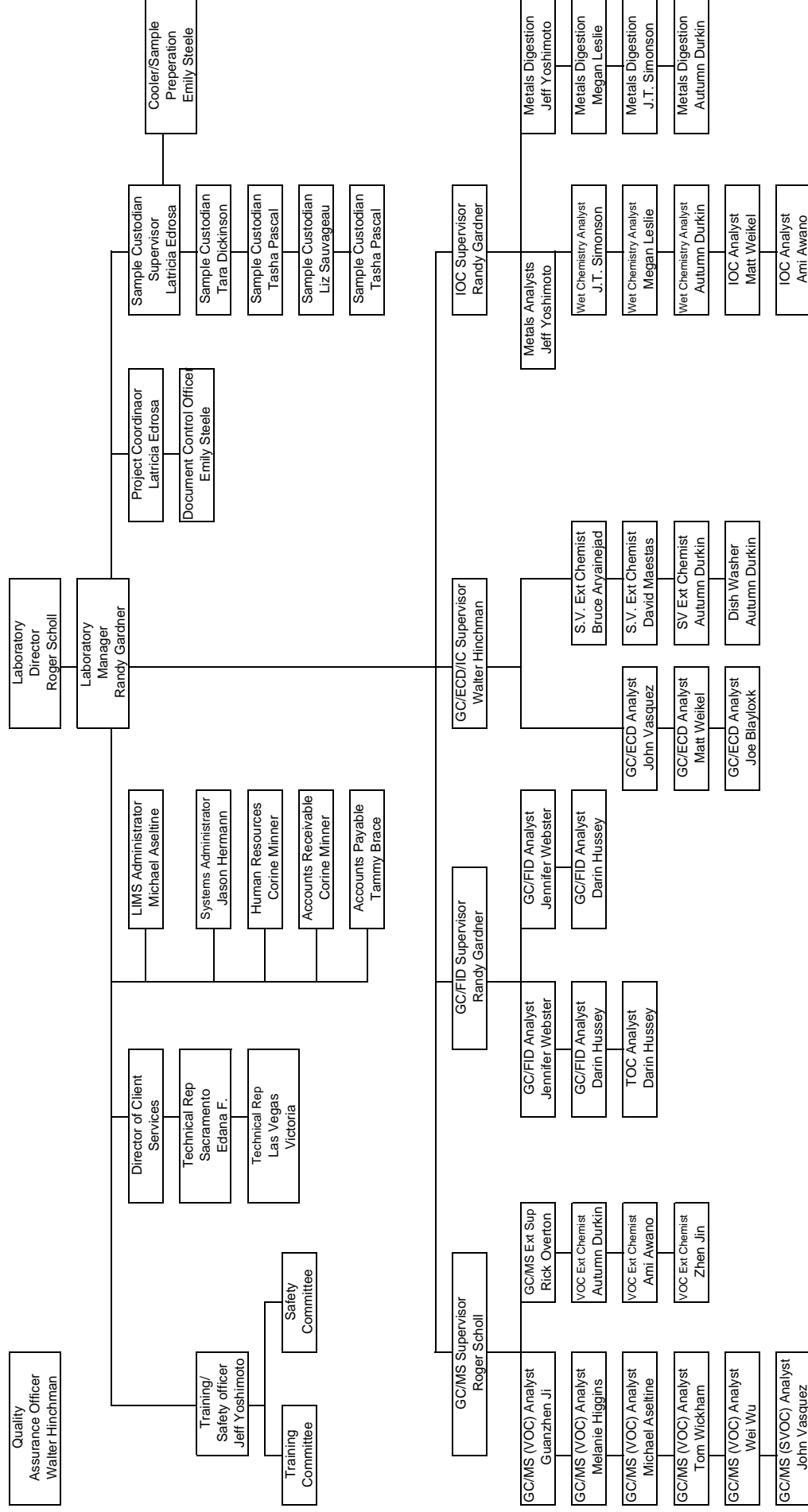
### 4.3 WORK STOPPAGE

The following personnel have the authority to stop work in response to quality problems. They have the authority to approve or disapprove of analytical and/or extraction batches and the authority to approve or disapprove of final analyses.

- 1) Laboratory Director
- 2) Quality Assurance Officer
- 3) Laboratory Manager

# Alpha Analytical, Inc.

## Organizational Chart





# **Section 5**

**Quality Assurance Routines to  
Assess Precision, Accuracy and  
the Calculation of Method  
Detection Limits**

## **5.0 QUALITY ASSURANCE ROUTINES TO ASSESS PRECISION, ACCURACY AND THE CALCULATION OF METHOD DETECTION LIMITS**

### **5.1 GENERAL DATA QUALITY OBJECTIVES**

Data Quality Objectives (DQOs) for each method and analyte are determined to ensure analytical and method-specific goals are met. DQOs for analytical measurements are commonly interpreted as Precision, Accuracy, Representativeness, Completeness and Comparability, "PARCC." Precision and accuracy are the two most common criteria used to define DQOs.

Accuracy and precision are generally determined for each EPA method target compound by data presented as either single laboratory or spooled data from multiple laboratories. This type of data, represents Method Derived Data Quality Objectives. As part of our QA program, Alpha statistically determines in-house Laboratory Derived Data Quality Objectives and ensures these DQO's are comparable to method derived DQO's.

The last three, representativeness, completeness and comparability, are DQOs that are laboratory and project specific. Therefore, these three elements do not have method specified DQOs. The determination of method detection limits is also a useful tool to evaluate project or analytical goals.

### **5.2 ROUTINE METHODS USED TO ASSESS PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS AND COMPARABILITY**

One of the primary objectives of this QA plan is to establish a framework that can estimate the quality of each analytical system, including Precision, Accuracy, Representativeness, Completeness, and Comparability. Specific data quality objectives for accuracy, precision, and completeness are based on prior knowledge of the measurement system employed and method validation studies using duplicates, spikes, standards, recovery studies, etc.

At the present time, the EPA has established only a couple of select PARCC guidelines that must be met by data generated in support of a few methods of analysis. However, for analysis which PARCC criteria do exist, Alpha uses those method defined QA goals.

#### **5.2.1 Precision**

Repetitive measurements of the same parameters in a sample creates a distribution curve in which the spread or dispersion about the mean can be calculated or expressed as precision. The calculations for precision where duplicate or replicate analysis have been performed are accomplished by the analysis of laboratory control samples and matrix spikes.

Field duplicate samples and matrix spike duplicates are analyzed to assess field sampling precision. Laboratory control samples and laboratory control sample duplicates are analyzed to assess analytical precision. Precision measurements are determined using Relative Percent Difference (RPD) between the duplicate sample

results and, for replicate analyses, the Relative Standard Deviation (RSD) is calculated and used to determine precision.

#### 5.2.2 Accuracy

The accuracy of an analytical measurement is defined as the amount of agreement between an experimental measurement of the concentration of a parameter and the known true concentration of that parameter. Accuracy is commonly expressed as a percent recovery of spiked analytes. Analytical accuracy is assessed by comparing the percent recovery of analytes spiked into an LFB or LCS to a defined control limit. For organic methods of analysis, surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed.

Percent recovery, where applicable, is calculated using concentration limits, such as µg/Kg or µg/L, and converted to a percentage. Accuracy criteria is both compound and method specific.

#### 5.2.3 Representativeness

The representativeness of samples describes the degree to which the sample represents an undisturbed matrix. Obtaining representative samples is a difficult task that requires site and project specific planing prior to any field sampling. Matrix, target compounds, methods of analyses, sampling depths, sampling equipment, time of sampling, etc. are all contributing factors in obtaining a representative sample. In order to minimize random errors introduced by non-uniform sampling procedures, the SOP's cited in the FSP are suggested procedures to be followed for sample collection. The use of standard operating procedures will help to provide uniformity for the sample collection work.

#### 5.2.4 Completeness

Data completeness is defined as the percentage of total tests conducted that are deemed satisfactory for a specific analysis or matrix. General criteria for data completeness is determined and compared to project specific DQOs. Completeness is expressed as a percent of the overall data generated and is calculated as follows:

$$C = V/T \times 100$$

where,

C = Percent completeness;

V = Number of measurements judged satisfactory; and,

T = Total number of planned measurements.

Alpha's goal is 95% completeness for aqueous samples with a relatively clean

background matrix (i.e., SDWA samples), a 90% completeness for methods which may contain a some degree of background interferences (i.e., CWA) and 85% completeness for methods which more than likely will contain significant degrees of background contamination as well as matrix bias (i.e., RCRA type samples).

#### 5.2.5 Comparability

Comparability of data is expressed as a measure of confidence where multiple sets of data may be used to evaluate a common analyte by a standard method of analysis.

Data comparability can be scrutinized by collecting independent samples in the same manner during different sampling episodes and by processing samples using the same procedures in the laboratory. Field sampling and laboratory analytical procedures for each parameter should remain as constant as possible at all times.

Alpha uses standard EPA methods to insure inter-laboratory comparability of data. In addition, data generated by Alpha Analytical is expressed in units consistent with the data generated by other laboratories reporting similar analyses to allow comparability of data between organizations. SOPs are an important element in both inter- and intra-laboratory comparison of data. These procedures allow laboratory activities to become routine and standardized, which minimizes random errors.

### 5.3 CONTROL CHARTS

#### 5.3.1 Shewhart Control Chart

The Shewhart control chart is used by Alpha to monitor variations in the precision and accuracy of routine analysis and to detect possible trends in those variations.

This control chart is constructed from data representing the performance of a complete analytical method in order to monitor all activities associated with the analytical procedure. Although tabulations of precision and accuracy are used to evaluate if a datum point falls within the prescribed limits, trends are difficult to discern from tables.

Therefore, control charts consist of a graphical portrayal of that information. The Shewhart control chart consists of a central line or the average percentage recovery, and an upper and lower limiting line that establishes the window of acceptability.

Recovery data is used and graphed on a Shewhart control chart to monitor and establish the validity of individual sample analysis. Data are plotted chronologically, in terms of instrument analysis, to monitor instrument performance.

Several methods require laboratories to achieve a particular level of precision and accuracy, that are described and built into the methods. However, many of these methods establish precision and accuracy requirements from the results of a single laboratory. These criteria are often times not indicative of the method or how

laboratories are being evaluated on a national scale. Therefore, NELAP requires laboratories to determine and use on a regular basis, the accuracy and precision limits that reflect actual in-house control windows or criteria generated from laboratory data.

#### 5.3.1.1 Upper and Lower Warning Limits

The calculation of limiting lines or the windows of acceptability is based on the distribution of recoveries about the mean. There are two sets of limiting lines that may be used in the construction of the Shewhart control chart. The inner window or warning limit is defined by the upper or lower warning limits (UWL) and (LWL), and is calculated as:

$$\begin{aligned} \text{UWL} &= X + 2s \\ \text{LWL} &= X - 2s, \end{aligned}$$

where X is the mean and s is the standard deviation.

Statistically, one in twenty will fall outside the inner control lines, provided the data are statistically uniform (i.e., 95% confidence interval).

#### 5.3.1.2 Upper and Lower Control Limits

The second window is defined by the Upper and Lower Control Limit (UCL) and (LCL) and is calculated as:

$$\begin{aligned} \text{UCL} &= X + 3s \\ \text{LCL} &= X - 3s \end{aligned}$$

and is statistically defined at the 99% confidence level. When possible control charts are prepared for each surrogate compound using data from actual sample analysis and plotted as a percent recovery. Percent recoveries are used to allow for minor variations in standard solution concentrations.

Limits of acceptability are calculated using a minimum of thirty samples of the same sample matrix and are periodically reviewed. Only samples evaluated as in-control are used for establishing and updating control limits. Out-of-control or outlier points are plotted; however, these points are not used in control limit calculations.

## 5.4 METHOD EVALUATION

5.4.1 For all environmental organic and inorganic analytical methods of analysis, the method is evaluated for those parameters that adversely affect data quality. These parameters are things such as method detection limit, reporting limit, accuracy and precision.

This includes both standard and non-standard methods of analysis, test methods used outside of their published scope, and amplifications and modifications of standard methods to confirm that the method is fit for its intended use.

Method validation is as extensive as is necessary to meet the needs of the application. Method validation results and the procedure used for the validation are recorded. The following list briefly describes the minimum method parameters that are critically evaluated and documented prior to performing a new method.

5.4.1.1 An Initial Demonstration of Capability (IDC) is performed prior to the analysis of samples or when a significant change in instrument, personnel, matrix or test method has taken place. Continuing Demonstrations of Capability (DOC) are performed annually. This data is used to initially determine method accuracy and precision.

5.4.1.2 Method Detection Limit studies (MDL's) - MDL studies are conducted to determine the minimum amount of a substance that an analytical process can detect. This is also referred to as a Limit of Detection (LOD) study. Some methods state the MDLs and/or estimated MDLS that were achieved in multi-laboratory studies as a means to help the laboratory evaluate its method data.

5.4.1.3 Determination of the Limit of Quantitation (LOQ) - LOQ is defined as the minimum level, concentration or quantity of a target analyte that can be reported with specified degree of confidence. The LOQ was formerly known as the Practical Quantitation Limit (PQL), Minimum Reporting Limit (MRL) or simply the reporting limit.

5.4.1.4 Calibrations- Calibration protocols are method specific. A summary of the method calibration criteria is found in individual method SOP's and in section 9 of the QAM.

5.4.1.5 Proficiency Evaluation (PE) test samples- The results of PE sample analysis, if available, are used to evaluate our ability to produce accurate data.

See QAM, Vol II, Appendix E.8, A Practical Application Guide for Performing a Determination of Capabilities (DOC) and Method Detection Limit (MDL) Study for additional details.

#### 5.4.2 Demonstration of Capability (DOC)

5.4.2.1 An initial Demonstration of Capability (IDC) is performed prior to using test methods or when a significant change in instrument, test methods or personnel have been made. DOC does not test the performance in real world samples, but in available clean matrix ( a sample matrix in which no target analytes or interferences are present at concentrations that would impact the

results of a specific test method).

5.4.2.2 Once the IDC has been performed a continuing demonstration of capability is performed annually.

5.4.2.3 In all cases, the appropriate forms and the Certification Statement is completed and retained by Alpha. All associated supporting data, such as quantitation reports etc., necessary to reproduce the analytical results summarized in the Certification Statement is also retained by Alpha.

5.4.2.4 Procedure for Demonstration of Capability.

The following guidelines are used when completing a initial demonstration of capability or continuing demonstration of capability study.

5.4.2.4.1 A Quality Control (QC) sample is obtained from an outside source or the QC sample is prepared using stock standards that are independently prepared from those used in instrument calibration.

5.4.2.4.2 The analytes are spiked into a volume of clean matrix sufficient to prepare four aliquot at the method specified concentration or at a concentration 1-10 times the limit of quantitation.

5.4.2.4.3 At least four aliquots are prepared and analyzed according to the test method either concurrently or over a period of days.

5.4.2.4.4 Using all the results calculate the mean recovery in the appropriate reporting units and the standard deviations of the population sample (n-1) for each parameter of interest is determined.

5.4.2.4.5 The information determined above is compared to the corresponding acceptance criteria for precision and accuracy in the associated method. If all parameters meet the acceptance criteria, the analysis of samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter and the test needs to be repeated.

5.4.2.4.6 When one or more of the target compounds fail the acceptance criteria then the analyst should proceed according to the following:

- a) Locate and correct the source of the problem and repeat the test, and

- b) Beginning with (3) above, repeat the test for all parameters that failed to meet the method criteria. Repeated failure, however, generally confirms a problem with the measurement system. If this occurs, locate the source of the problem and repeat the test for all compounds of interest beginning with (3) above.

#### 5.4.3 Method Detection Limits/ Limits of Detection

5.4.3.1 The Method Detection Limit (MDL) is the minimum concentration a substance can be measured and reported with 99% confidence that the analyte concentration is greater than zero. A properly documented MDL study requires that all sample processing steps of the analytical method be included in the determination of the method detection limit. Alpha establishes MDLs for each method and analyte used at our laboratory. Alpha re-validates these MDLs on a regular basis.

Alpha follows the procedures as described in 40 CFR, Part 136, Appendix B, Definition and Procedure for the Determination of the Method Detection Limit. This procedure is summarized in the following manner:

5.4.3.1.1 Obtain the concentration value that corresponds to:

- a) An instrument signal/noise ratio within the range of 2.5 to 5.0, or
- b) The region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the curve.

5.4.3.1.2 Analyze a minimum of seven replicates containing the analytes of interest at a concentration of three to five times the estimated MDL;

5.4.3.1.3 Calculate the Standard Deviation (SD) of each of the analytes; and,

5.4.3.1.4 Using the Student t-Test, determine the MDL for each analyte as follows:

$$\text{MDL} = (t_{n-1}) (\text{SD}),$$

where  $(t_{n-1})$  is the student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.



#### 5.4.4 Limit of Quantitation

5.4.4.1 The LOQ or reporting limit is critical to the evaluation and reporting of analytical data. The LOQ is technically defined as the minimum level, concentration or quantity of a target analyte that can be reported with a specified degree of confidence.

5.4.4.2 The lowest initial calibration point must be at or below the established LOQ, but they may also be at the same concentration.

5.4.4.3 The LOQ is generally established at a concentration no less than 3 times the LOD.

5.4.4.3.1 If a client requires a reporting limit below the established LOQ, typically method modification is required or the client will be required to accept the established LOQ as the lowest technically valid value that can be provided.

5.4.4.3.2 If analytes are reported below the established LOQ, they should be flagged.

**TABLE 5-1**  
**STATISTICAL CALCULATIONS**

STATISTIC	SYMBOL	FORMULA	DEFINITION	USES
Mean	$\bar{x}$	$\frac{\sum_{i=1}^n X_i}{n}$	Measure of central tendency	Determine average value of measurements
Standard Deviation	SD	$\left( \frac{\sum (x_i - \bar{X})^2}{(n - 1)} \right)^{\frac{1}{2}}$	Measure of relative scatter of the data	Calculating variation of measurements
Relative Standard Deviation	RSD	$\left( \frac{s}{\bar{x}} \right) \times 100$	Relative standard deviation, adjusts for magnitude of observations	Assess precision for replicate results
Percent Difference	% D	$\frac{x_i - x_2}{x_1} \times 100$	Measure of the difference of 2 observations	Assess accuracy
Relative Percent Difference	RPD	$\left( \frac{(x_1 - x_2)}{\left( \frac{x_1 + x_2}{2} \right)} \right) \times 100$	Measure of the variability that adjusts for the magnitude of observations	Assess total and analytical precision of duplicate measurements
Percent Recovery	% R	$\left( \frac{x_{measured}}{x_{true}} \right) \times 100$	Recovery of spiked compound in pure matrix	Assess accuracy
Percent Recovery	% R	$\frac{\text{Value of Spiked Sample} - \text{Value of Unspiked Sample}}{\text{Value of Added Spike}} \times 100$	Recovery of spiked compound in sample matrix	Assess matrix effects and total precision

x = Observation (concentration) n = Number of observations

# **Section 6**

## **Sampling Procedures**

## **6.0 SAMPLING PROCEDURES**

### **6.1 INTRODUCTION**

Alpha is not generally responsible for sample collection. When Alpha does collect samples for analysis, it follows the procedures outlined in this section or other specific field sampling standard operating procedures. These procedures are designed to obtain samples that are proper representations of the sampled matrix. Once a sample has been collected, it should be properly stored and preserved to maintain the chemical and physical properties that it possessed at the time of collection. Trace levels of contaminants from sources external to the sample must be eliminated through the use of good sampling techniques. Sample management and stringent documentation are the key factors as outlined in Alpha's Field Sampling Plan (FSP).

The specific methods, techniques and equipment used for collecting environmental samples are provided in Appendix B, FSP. Subjects discussed include field activities, sampling plans and decontamination procedures. Field sampling practices should follow established protocols.

Once samples have been taken, they are expeditiously sent to the laboratory. As a general rule, storage at low temperature is the best way to preserve most samples; however, the length of time a sample can be held even at low temperature varies with the analyte and matrix. Bottles are packaged for shipping in insulated containers, and constructed to ensure that sample bottles will arrive intact.

When samples are received by Alpha, the time lapse between sampling activity and analysis should not exceed the times shown in the sample holding times tables.

### **6.2 SAMPLE HANDLING**

Samples are collected and handled in a manner that attempts to maintain sample integrity and preserves the potential contaminants being analyzed. Samples are collected in containers specific to the matrix and requested analysis.

Gloves should be used during the sampling and extraction procedures for protection against possible exposure to carcinogens and to minimize accidental contamination of samples by the collector. When wearing gloves, the person must be careful not to let the gloves come into contact with the sample, the interior of the container, or allow solvents to touch both the sample and any extract.

### **6.3 SAMPLE SHIPMENT**

Samples requiring refrigeration are carefully placed in coolers with bagged ice to maintain a temperature of 4°C. Glass sample containers are securely packaged in ice chests using bubble wrap or other packaging material, to avoid breakage in transit. Chain-of-Custody forms should be completed at the sampling site and sent with the sample to maintain sample

integrity at all times. Samples are then transported to the laboratory by a courier or field personnel as soon as possible.

## **6.4 CONTAINERS**

Sample containers are determined by the requested analysis. However, the following containers are generally used for environmental analysis:

- Standard 40 ml clear glass screw-cap Volatile Organic Analysis (VOA) vials with Teflon-faced silicone septum are used for volatile analysis;
- Narrow mouth, 1 L amber Boston round glass bottles with Teflon-lined lids are used for semi-volatile, analysis;
- Large mouth, 8oz, 4oz glass bottles or brass tubes are typically used for soil and sediment samples; and,
- Narrow mouth, 125, 250, and 500 mL polyethylene bottles are typically used for the analysis of metals and other general inorganic parameters.

All sample containers are cleaned according to EPA established protocols. Factory cleaned sample containers require no further cleaning prior to sample collection, and are the containers of choice. Sample containers are not reused. Alpha maintains a sequestered supply of sample containers to eliminate the possibility of contamination of the sample from the container. Containers are sequestered by lot to track QA/QC procedures associated with that group of sample containers.

## **6.5 SAMPLE PRESERVATION**

The purpose of sample preservation is to prevent or retard chemical degradation or modification during transit and storage. Most solid samples require cooling as the only preservation technique. Water sample are subject to a variety of specific preservation techniques, depending on the target analytes. Alpha Analytical preserves samples according to analytical methods and programs by which the sample will be analyzed. Preservatives are generally grouped into one of two types:

- a) Acid preservatives, i.e., HCl, HNO<sub>3</sub>, or H<sub>2</sub>SO<sub>4</sub>, used to stop biological activity; and,
- b) Sodium thiosulfate, ascorbic acid, or ammonium chloride used to stop the chlorination process.

Efforts to preserve the integrity of the samples are initiated at the time of sampling or immediately upon sample receipt at the laboratory. Alpha accomplishes sample preservation in one of two ways:

- a) Send or take bottles to preserve at the sample site; or

- b) Add preservatives to sample containers prior to going to the field.

Preservations and storage requirements are provided in Tables 6-1 through 6-8.

## **6.6 SAMPLE HOLDING**

The holding time before analysis is of critical, practical and regulatory importance. The time that a preserved sample may be held between sampling and analysis is based on the specific analytical method and analytes of interest. Holding time limitations described in all standardized methods are intended to minimize chemical change in a sample before it is analyzed.

Holding times, outlined in tables 6-1 through 6-8, are the maximum times allowable between sample collection, extraction and analysis. Allowable holding times apply to both solid and aqueous samples. The holding time clock starts the moment of sampling and ends with the beginning of the extraction or analytical procedure.

Samples analyzed after holding times have been exceeded are considered out-of-control and analytical results are unacceptable to report unless requested by the Client.

To expedite analysis and minimize the possibility of exceeding holding times, overnight courier service, such as Federal Express, UPS, etc., or other reliable methods of transportation are used.

Sample cold storage is terminated only after all analysis has been finished and the minimum holding time requirements have been met.

## **6.7 FIELD SAMPLING**

### **6.7.1 Surface Water Sample Collection**

Surface water samples from springs or other surface waters may be taken under many different site specific conditions.

At the time of sampling, the Project Coordinator should designate the appropriate sampling techniques for the site-specific setting.

Before sampling, all equipment is rinsed downstream or away from the sampling point, taking care not to disturb sediments at the sampling point. After sampling each location, the equipment is rinsed with distilled water and decontaminated before further use.

All samples are placed in containers that have been pre-cleaned or have been cleaned according to established protocols. Organic samples are collected in amber glass bottles, with Teflon-lined lids and samples for inorganic chemical analyses are

collected in separate glass or polyethylene bottles.

Sample filtration is determined by the Client prior to sampling. Samples are then collected according to the QAPP or the sampling techniques described in the FSP and documented accordingly.

#### 6.7.2 Ground Water Sample Collection

Groundwater sampling should occur only after wells have been completely developed. Well development disturbs natural groundwater systems and should remain undisturbed for several days to allow the groundwater system to return to chemical equilibrium.

All equipment used to measure and sample the groundwater system (e.g., bailer, pumps, tapes, ropes, etc.) is cleaned before use to prevent cross-well contamination. When sediments adhere to sampling equipment, scrubbing is required in addition to the normal rinsing.

Samples are placed in containers that have been pre-cleaned or have been cleaned according to established protocols. Organic samples are collected in glass bottles, with Teflon-lined lids and samples for inorganic chemical analyses are collected in separate glass or polyethylene bottles.

#### 6.7.3 Soil Sampling

The sampler is ultimately responsible for collecting representative samples from the site to accurately reflect project site conditions. The Project Coordinator/Client must specify the method of analysis, and the procedure to collect the sample that will represent the matrix of interest. The sampler should remove all items that are not integral components of the matrix of interest.

The Project Coordinator/Client should develop a sampling plan with sample site locations, chosen to be representative of the area being investigated. These plans should be followed during the sampling excursions.

Compositing multiple samples into a single sample can be used as part of the initial sampling strategy to identify plumes of contamination and as a screening technique. Individual samples are subsequently collected and analyzed to describe the sampling points within that area of investigation.

Sampling points are typically marked with a stake and labeled with the appropriate site identification information. Prior to sampling, surface vegetation, rocks, pebbles, leaves, twigs, and other debris should be cleared from the sample point to allow for the collection of a representative soil sample.

Background samples should be taken at distances outside the investigation area, but

within a location that is geologically similar to the actual sampling site. These samples give the Principle Investigators (PI) additional information concerning concentration levels above the background (i.e. baseline concentration). The number and location of background surface samples should be specified in the QAPP.

Soil samples should be collected in containers cleaned according to established protocols of the appropriate size. Samples are then labeled and placed in a temperature controlled ice chest immediately after sampling and delivered to Alpha as soon as possible.

After sampling each location, all equipment should be thoroughly cleaned to prevent cross contamination of samples. Equipment should be scrubbed and rinsed with distilled water.



**SDWA TABLE OF SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS  
ORGANIC ANALYSIS**

**Table 6-1**

METHOD	PARAMETERS	PRESERVATION	CONTAINER	HOLDING TIME
*504.1	EDB/DBCP	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 3mg/40ml	40ml, G, Cool, 4 °C	Extract and analyze within 14 days
505	Organohalide Pesticides and PCB's	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 3mg/40ml	40ml, G, Cool, 4 °C	Extract within 7 days / Analyze immediately after Extraction
507	Nitrogen / Phosphorus Pesticides	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 80mg/L	1L, G, Cool, 4 °C	Extract within 14 days / Analyze within 14 days of Extraction
508	Chlorinated Pesticides	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 80mg/L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 14 days of Extraction
515.1	Acid Herbicides	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 80mg/L	1L, G, Cool, 4 °C	Extract within 14 days / Analyze within 28 days of Extraction
531.1	Carbamates	Monochloroacetic Acid Buffer pH-3 1.2ml/40ml	40ml, G, Cool, 4 °C	Analyze within 28 days
547	Glyphosate	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 4mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days / 18 months if frozen
548.1	Endothall	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 80mg/L	500ml, G, Cool, 4 °C	Extract within 7 days / Analyze within 14 days of Extraction
549.2	Diquat	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 50mg/0.5L	500ml, P, Amber, Cool, 4 °C	Extract within 7 days / Analyze within 21 days of Extraction
525.2	SVOCs	Na <sub>2</sub> SO <sub>3</sub> 50mg/1L**	1L, G, Cool, 4 °C	Extract within 14 days / Analyze within 30 days of Collection
*524.2	VOCs	pH<2, 1:1 HCl + Ascorbic Acid 25mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days
*551.1	Disinfectant Byproducts	Na <sub>2</sub> HPO <sub>4</sub> 2g + KH <sub>2</sub> PO <sub>4</sub> 198g + NH <sub>4</sub> Cl 1.2g Buffer Salts	40ml, G, Cool, 4 °C	Extract within 14 days / Analyze within 14 days of Extraction / Store Extracts at -10 °C
*551.1	Chloral Hydrate only	Na <sub>2</sub> HPO <sub>4</sub> 2g + KH <sub>2</sub> PO <sub>4</sub> 198g + Na <sub>2</sub> SO <sub>3</sub> 1.2g Buffer Salts	40ml, G, Cool, 4 °C	Extract within 14 days / Analyze within 14 days of Extraction / Store Extracts at -10 °C
552.2	Haloacetic Acids	NH <sub>4</sub> Cl 100mg/L	40ml, G, Cool, 4 °C	Extract within 14 days / Analyze within 7 days of Extraction when stored at 4 °C / Analyze within 14 days of Extraction when stored at -10 °C

\*Note-Zero head space (no air bubbles) is required for these methods.

Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> - Sodium Thiosulfate is used for chlorinated source water only.

Na<sub>2</sub>SO<sub>3</sub> - Sodium Thiosulfite is used for chlorinated source water only.

G - Glass P - Plastic

\*\* Sample pH is field adjusted <2 HCL if acid compounds like PCP are to be determined.

**SDWA SAMPLE PRESERVATION AND HOLDING TIME TABLE  
METHOD 524.2**

**Table 6-2**

DECSRIPTION	SAMPLE VOLUME	DECHLORINATION	SAMPLE PRESERVATION	ANALYSIS HOLDING TIME
Full List Compounds	3 x 40mL	25mg ascorbic acid per 40mL sample	pH<2, 2 drops 1:1 HCL Field preserved, cool 4°C	14 days
Full List Compounds sample foams when HCL is added carbonaceous waters	3 x 40mL	25mg ascorbic acid per 40mL sample	No acid	Analyze within 24 hours
THM's only	3 x 40mL	25mg ascorbic acid per 40mL VOA vial	pH<2, 2 drops 1:1 HCL Field preserved, cool 4°C	14 days
THM's only	3 x 40mL	Sodium Thiosulfate 3mg/40mL sample	No acid	14 days
THM's only sample foams when HCL is added carbonaceous waters	3 x 40mL	Sodium Thiosulfate 3mg/40mL sample	No acid	14 days

**SDWA TABLE OF SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS**  
**INORGANIC ANALYSIS**

**Table 6-3**

METHOD	PARAMETERS	PRESERVATION	CONTAINER	HOLDING TIME
120.1/SM2510B	Conductivity	Cool, 4 °C	250ml, G/P	Analyze within 28 days
150.1/SM4500H	pH	Cool, 4 °C (no headspace if possible)	250ml, G/P	Analyze immediately
160.1/SM2540C	TDS	Cool, 4 °C	250ml, G/P	Not specified for method 160.1. Analyze within 7 days for method SM2540C. Will use 7 days.
180.1/SM2130B	Turbidity	Cool, 4 °C	250ml, G/P	Not specified (as soon as possible) will use a 14 day in-house criteria
300.0	Anions	None, if analyzed within 48 hours, for nitrate, nitrite and ortho-phosphate or else pH < 2 H <sub>2</sub> SO <sub>4</sub> .	250ml, G/P	48 hours non preserved, 28 days preserved.
310.1/SM2320B	Alkalinity	Cool, 4 °C, minimize headspace and cap at all times	250ml, G/P	Not specified. Will use a 14 day in-house criteria.
314.0	Perchlorate	None	250ml, G/P	Analyze within 28 days
330.5/SM4500Cl G	Residual Free Chlorine	Cool 4 °C, no headspace	0.1-L, amber glass, protect from light	Not specified, other than stating analysis must be started immediately, (In house criteria 24 hours).
350.3/SM4500NH <sub>3</sub> D	Ammonia	Cool, 4 °C, <u>May</u> be preserved with H <sub>2</sub> SO <sub>4</sub> , 2ml/ L.	250ml,G/ P	Not specified. Will use a 14 day in-house criteria.
SM3500Cr D	Chromium VI	Cool, 4 °C	250ml, G/P	24 hours.
200.8	Metals (total)	1:1 HNO <sub>3</sub> , 3ml per 1L, pH<2 Sample may be preserved in the laboratory up to 2 weeks following sample collection.	500mL, G/P	Digest and analyze within 6 months of collection.
SM2340B	Hardness (Calculated)	Same as for 200.8 metals		
200.8	Metals (dissolved)	Field filtered through a 0.45 $\mu$ m filter. 1:1 HNO <sub>3</sub> , 3ml per 1L, pH<2 Sample may be preserved in the laboratory up to 2 weeks following sample collection.	500mL, G/P	Digest and analyze within 6 months of collection.
SM3500-Fe D	Iron (Total)	Cool, 4 °C, pH<2 HNO <sub>3</sub>	250ml,G/ P	Digest and analyze within 6 months of collection.
SM3500-Fe D	Iron (Dissolved)	Cool, 4 °C, Field filtered through a 0.45 $\mu$ m filter. pH<2 HNO <sub>3</sub>	250ml,G/ P	Digest and analyze within 6 months of collection.
SM3500-Fe D	Iron (Ferrous)	Cool, 4 °C, Field filtered through a 0.45 $\mu$ m filter. pH<2 HCl	250ml,G/ P	Not specified, (as soon as possible). In house criteria will require samples be color developed within 72 hours of sample collection and samples analyzed within 72 hours of color development.
G - Glass P - Plastic				

**CWA TABLE OF SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS  
ORGANIC ANALYSIS**

**Table 6-4**

METHOD	PARAMETERS	PRESERVATION	CONTAINER	HOLDING TIME
*601	Purgeable Hydrocarbons	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 10mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days
*602	Purgeable Aromatics	pH<2, 1:1 HCl, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 10mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days
*603	Acrolein / Acrylonitrile	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 10mg/40ml, pH 4-5 (HCL/NaOH)	40ml, G, Cool, 4 °C	Analyze within 14 days
604	Phenols	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 80mg/L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
605	Benzidine	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 80mg/L, pH 2-7 H <sub>2</sub> SO <sub>4</sub>	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 7 days of Extraction
606	Phthalate Esters	No Preservation	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
607	Nitrosamines	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 80mg/L, pH 7-10 (H <sub>2</sub> SO <sub>4</sub> /NaOH)	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
608	Organochlorine Pesticides/PCB's	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 80mg/L, pH 5-9 (H <sub>2</sub> SO <sub>4</sub> /NaOH)	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
609	Isophrone	No Preservation	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
610	Polynuclear Aromatic Hydrocarbons	Na <sub>2</sub> SO <sub>3</sub> 80mg/1L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
611	Haloethers	Na <sub>2</sub> SO <sub>3</sub> 80mg/1L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
612	Chlorinated Hydrocarbons	No Preservatives	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
613	Dioxin	Na <sub>2</sub> SO <sub>3</sub> 80mg/1L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
614	Organophosphorus Pesticides	pH 6-8 (H <sub>2</sub> SO <sub>4</sub> /NaOH)	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
615	Chlorinated Herbicides	No Preservatives	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
619	Triazine Pesticides	Not Specified	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
622	Nitrogen / Phosphorus Pesticides	pH 6-8 (H <sub>2</sub> SO <sub>4</sub> /NaOH)	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
*624	Purgeables	pH<2, 1:1 HCl, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 10mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days
625	Base Neutral Acids	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 80mg/L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
632	Carbamate Pesticides	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 80mg/L	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 28 days of Extraction
*Note-Zero head space (no air bubbles) is required for these methods      Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> - Sodium Thiosulfate    G - Glass    P - Plastic				

**CWA/RCRA TABLE OF SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS  
INORGANIC ANALYSIS**

**Table 6-5**

<b>METHOD</b>	<b>PARAMETER</b>	<b>PRESERVATION</b>	<b>CONTAINER</b>	<b>HOLDING TIME</b>
EPA 120.1/SM2510B/9050A	Conductivity	Cool, 4 °C	250 ml, G/P	Analyze within 28 days
EPA 150.1/SM4500H/9040C	pH	Cool, 4 °C	250 ml, G/P	Analyze immediately,
EPA 160.1/SM2540C	TDS	Cool, 4 °C	250 ml, G/P	Not specified for method 160.1. Analyze with 7 days method SM2540C
EPA 160.2/SM2540D	TSS	Cool, 4 °C	250 ml, G/P	Not specified for method 160.2. Analyze with 7 days method SM2540D
EPA 160.3/SM2540B	TS	Cool, 4 °C	250 ml, G/P	Not specified for method 160.2. Analyze with 7 days method SM2540B
EPA 180.1/SM2130B	Turbidity	Cool, 4 °C	250 ml, G/P	Not specified (as soon as possible) will use a 14 day criteria.
EPA 300/9056	Anions	None ,if analyzed with 48 hrs, for nitrate, nitrite and ortho-phosphate or else pH <2 H <sub>2</sub> SO <sub>4</sub>	250 ml, G/P	48 Hours non preserved, 28 days preserved
EPA 305.1/SM2310B	Acidity	Cool, 4 °C	250 ml, G/P	Not specified. Will use a 14 day in-house criteria
EPA 310.1/SM2320B	Alkalinity	Cool, 4 °C	250 ml, G/P	Not specified. Will use a 14 day in-house criteria
EPA 314.0	Perchlorate	None	250 ml, G/P	Analyze within 28 days
EPA 330.5/SM4500Cl G	Chlorine	Cool, 4 °C no headspace, protect from light	0.1-L, Amber glass	Not specified, other than stating analyze immediately. Will use 24 hr criteria
EPA 350.3/SM4500 NH <sub>3</sub> D	Ammonia	Cool, 4 °C <u>May</u> be preserved with H <sub>2</sub> SO <sub>4</sub> pH<2	250 ml, G/P	Not specified. Will use a 28 day in-house criteria
EPA 351.4/SM4500N C	Total Kjeldahl - N	Cool, 4 °C <u>May</u> be preserved with H <sub>2</sub> SO <sub>4</sub> pH<2	250 ml, G/P	Not specified. Will use a 28 day in-house criteria
EPA 365.2/SM4500P E	Total Phosphorus	pH<2 H <sub>2</sub> SO <sub>4</sub> , Cool, 4 °C	250 ml, G/P	Not specified. Will use a 28 day in-house criteria
EPA 376.2/SM4500S D	Sulfide	0.2 mL 2N zinc acetate per 0.1-L( glass is preferred), 0.2 ml 6 N NaOH, pH >9, no head-space Cool, 4 °C	250 ml, Glass	Not specified. Will use a 14 day in-house criteria if preserved
EPA 377.1	Sulfite	Cool, 4 °C	500 ml, G/P	24 hours
EPA 410.4/SM5520D	COD	pH<2 H <sub>2</sub> SO <sub>4</sub> , Cool, 4 °C	250 ml, G/P	Not specified. Will use a 28 day in-house criteria
SM 5210B	BOD	Cool, 4 °C	2L, Plastic	48 hours
EPA 425.1	MBAS	Cool, 4 °C	2 L, Clear Glass	48 hours

EPA 415.1/SM5310C	TOC	pH<2 H <sub>2</sub> SO <sub>4</sub> , Cool, 4 °C, protect from sunlight	125 ml G/P	28 days
EPA 245.1/7470	Mercury	pH<2 HNO <sub>3</sub> Cool, 4 °C	250 ml, Plastic	28 days
200.8/6020	Metals, ICP-MS	pH<2 HNO <sub>3</sub> Cool, 4 °C	250 ml, G/P	6 months
SM3500Cr D/7196A	Cr <sup>+6</sup>	Cool, 4 °C	250 ml, G/P	24 hours
SM3500Fe D	Total Iron	pH<2 HNO <sub>3</sub> Cool, 4 °C	250 ml, G/P	6 months
SM3500Fe D	Ferrous Iron	Field filter, then acidify pH <2 HCL, Cool, 4 °C	250 mL, G/P	Not specified, will use 24 hr criteria
1664A	Oil and Grease	pH<2 HCL or H2SO4, Cool, 4 °C	1-L Glass only	28 days

## RCRA TABLE OF WATER / AQUEOUS SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS ORGANIC ANALYSIS

**Table 6-6**

METHOD	PARAMETERS	PRESERVATION	CONTAINER	HOLDING TIME
*8010	Halogenated Volatiles	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .008%	40ml, G, Cool, 4 °C	Analyze within 14 days
*8011	EDB/DBCP	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 3mg/40ml	40ml, G, Cool, 4 °C	Analyze within 14 days
*8021B	Aromatic Volatiles	pH<2, 1:1 HCL, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .008%	40ml, G, Cool, 4 °C	Analyze within 14 days
8041	Phenols	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8081A	Organochlorine Pesticides	pH 5-9 (H <sub>2</sub> SO <sub>4</sub> /NaOH) Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8082	Polychlorinated Biphenyls (PCBs)	pH 5-9 (H <sub>2</sub> SO <sub>4</sub> /NaOH) Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8141A	Organophosphorus Pesticides	pH 5-9 (H <sub>2</sub> SO <sub>4</sub> /NaOH)	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8151A	Chlorinated Herbicides	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
*8260	Volatile Organics	pH<2, 1:1 HCL, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .008%	40ml, G, Cool, 4 °C	Analyze within 14 days
8270	BNAs	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8310	Polynuclear Aromatics	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .008%	1L, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8318	N-methyl carbamates	Monochloroacetic Acid Buffer pH 4-5; 1.2mL per 40mL	40ml, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8015B/Purgeable*	TPH/GRO	pH<2, 1:1 HCL	40ml, G, Cool, 4 °C	Analyze within 14 days
8015B/Extractable	TPH/DRO	No Preservation	40ml, G, Cool, 4 °C	Extract within 7 days / Analyze within 40 days of Extraction
8015B	Nonhalogenated VOC's	pH<2, 1:1 HCL, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .008%	40mL, G, Cool, 4 °C	Analyze within 14 days
*Note-Zero head space (no air bubbles) is required for these methods Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> - Sodium Thiosulfate G - Glass P - Plastic				

**RCRA TABLE OF SOIL / WASTE SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS**  
**ORGANIC ANALYSIS**  
**TABLE 6-7**

METHOD	PARAMETERS	PRESERVATION	CONTAINER	HOLDING TIME
8081A	Organochlorine Pesticides and PCBs	Soil - Cool to 4 °C Waste - None	4 to 8 oz., G, Cool, 4 °C	Extract within 14 days / Analyze within 40 days of Extraction
8082	PCB's (Aroclor)	Soil - Cool to 4 °C Waste - None	4 to 8 oz., G, Cool, 4 °C	Extract within 14 days / Analyze within 40 days of Extraction
8141A	Organophosphorus Pesticides	Soil - Cool to 4 °C Waste - None	4 to 8 oz., G, Cool, 4 °C	Extract within 14 days / Analyze within 40 days of Extraction
8151A	Chlorinated Herbicides	Soil - Cool to 4 °C Waste - None	4 to 8 oz., G, Cool, 4 °C	Extract within 14 days / Analyze within 40 days of Extraction
*8260	Volatile Organics	Soil - Cool to 4 °C Waste - None	4 to 8 oz., G, Cool, 4 °C	Analyze within 14 days
8270	BNAs	Soil - Cool to 4 °C Waste - None	4 to 8 oz., G, Cool, 4 °C	Extract within 14 days / Analyze within 40 days of Extraction
6020	Metals	Soil - Cool to 4 °C Waste - None	4 to 8 oz., G, Cool, 4 °C	Digest and analyze within 6 months.
*Note - Zero Headspace is required for these methods G - Glass P - Plastic				

**TABLE OF TCLP/SPLP PRESERVATION AND HOLDING TIME REQUIREMENTS  
ORGANIC AND INORGANIC ANALYSIS**

**Table 6-8**

<b>Method</b>	<b>Parameters</b>	<b>Preservation</b>	<b>Container</b>	<b>Holding Time</b>
1311/1312	VOCs	No Preservation	2L/300g, G, Cool, 4° C	See Table Below
1311/1312	SVOCs	No Preservation	2L/300g, G, Cool, 4° C	See Table Below
1311/1312	Metals	No Preservation	2L/300g, G/P, Cool, 4° C	See Table Below
G - glass P - Plastic				

<b>Method</b>	<b>Parameters</b>	<b>From Field Collection to TCLP/SPLP Extraction</b>	<b>From TCLP/SPLP Extraction to Preparative Extraction</b>	<b>From Preparative Extraction to Determinative Analysis</b>	<b>Total Elapsed Time</b>
1311/1312	VOCs	14 days	NA	14 days	28 days
1311/1312	SVOCs	14 days	7 days	40 days	61 days
1311/1312	Metals (except Hg)	180 days	NA	180 days	360 days
1311/1312	Mercury	28 days	NA	28 days	56 days



# **Section 7**

## **Sample Custody**

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## **7.0 SAMPLE CUSTODY**

### **7.1 SAMPLE CUSTODY PROCEDURES**

Traditionally, record keeping is the primary emphasis of a QA program and this is the case in sample custody. Without a rigid and formal record keeping system, the QC procedures would not be documented appropriately.

All samples, from receipt through analysis, are handled under our Sample Tracking Plan. This portion of the QA program helps ensure the maintenance of sample integrity. It also helps to ensure all test procedures are performed in a timely and efficient manner.

The Sample Tracking Plan (STP, Appendix C) is a set of SOPs written with the maintenance of custody as a primary objective interwoven through all of the SOPs. Alpha is responsible for sample tracking and the maintenance of custody once it arrives and continues through sample analysis.

The Sample Custody Officer (SCO ) is responsible for sample custody and for the overall implementation of the STP. This responsibility includes assuring proper handling and that the documentation of all samples are performed according to the described SOPs.

### **7.2 SAMPLE DOCUMENTATION PROCEDURES**

Sample documentation, identification and chain-of-custody procedures are designed to assure accountability and control of all samples. Analytical records are kept and maintained in sufficient detail to track and recreate all analytical activities to ensure sample integrity. Project and client communication is also an extremely important aspect of the sample documentation procedures.

SOP topics associated with the sample custody and documentation procedures are as follows:

- Sample Receiving and Project Communication,
- Manual Chain-of-Custody Procedure,
- LIMS Generated Chain-of-Custody Procedure,
- Sample Identification Procedure,
- Labeling Field Samples,
- Internal Chain-of-Custody Procedure,
- Sample Log-In Ledger,
- Sample Storage Procedure,
- Maintenance of Custody,
- Sample Tracking Procedure,
- Client Communication, and
- Sample Scheduling Procedure.

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#### 7.2.1 Chain-of-Custody (COC)

Samples are documented on a Chain-of-Custody form and signed by both the client and laboratory. This document formalizes the sample transaction and is critical to the maintenance of sample custody. Information required on this document is described in Section C.:

- Manual Chain-of-Custody Procedures,
- LIMS Generated Chain-of-Custody Procedures, and
- Internal Chain-of-Custody Procedure.

The Chain-of-Custody is generally regarded as a legal document and should be completely filled out as legibly and as error free as possible.

#### 7.2.2 Sample Log-in Ledger

Upon arrival at the laboratory, the Sample Custodian logs the sample with the pertinent information into the LIM System.

A long term ledger is maintained as a permanent record of samples logged into our LIM system. Approximately every 2 weeks the LIM system is queried to print all samples logged-in during that period. A macro is used which parses out the relevant information from each chain-of-custody produced by the laboratory. This information is printed and stored in a 3-ring binder labeled "Log-In". Sample information contained in the "Log-In" book includes:

- Initials,
- Date of sample receipt,
- Laboratory's sample identification,
- Client's sample identification,
- Matrix type,
- Analysis requested,
- Turn-around-time (TAT),
- Date sampled, and
- Work order comments.

This document is used primarily by the sample custodian as a quick source to check historical information on the chain-of-custody records.

#### 7.2.3 Sample Scheduling

The Laboratory Supervisors coordinate sample scheduling to maintain an even production flow while ensuring samples are extracted and analyzed within their prescribed holding times.

Scheduling is coordinated with the appropriate personnel to maximize production according to the numbers and types of analyses to be performed during the analytical or extraction batch. Scheduling is an art and requires a working knowledge of instruments, personnel and lab activities. This balance is often shifted by the presence of “rush” analysis. A rush analysis takes precedence in the scheduling of samples. All other normal analyses are pushed back by these requests, and are scheduled according to their sampling date.

Large sample projects are coordinated with the Laboratory Manager and Director before sample arrival. This preplanning eases potential workload difficulties while ensuring the appropriate level of QA/QC is maintained. All discrepancy reporting is handled by the Laboratory Director to ensure the problem is expressed to the client and is properly documented.

### **7.3 LABORATORY LOGBOOK POLICY**

Bound logbooks are the preferred method of record keeping. However, certain laboratory functions are formalized enough to use standard forms (e.g., sample preparation log). These activities are documented and recorded using spiral bound or loose leaf three-ring binders. In this case, pages are dated in chronological order which helps reference data.

Each analyst, instrument or specific laboratory function has its own logbook to track and document lab activities, dates and times more effectively. When more than one analyst shares a common logbook, they delineate their data insertions by initialing and dating data entries.

All logbook entries are made in ink. Corrections are made by drawing one line through the incorrect entry, and then entering the correct information, initialing and dating this change. Complete information is entered so that during an examination it can be decided what was done, by whom, when and what the results were. All logbook entries are signed by the analyst or technician recording those entries.

#### **7.3.1 Instrument Sequence Logbook**

Associated with each instrument is a sequence logbook in which all tuning, calibration and analytical activities conducted on that instrument are recorded. Analytical schedules are the preferred method of tracking analytical instrument activities. This logbook is instrument-specific, not person-specific.

At the end of each day or upon completion of an analytical batch, each analyst must sign and date all pages that contain data entries for that day.

With automated data acquisitions systems, the data files for each standard and sample are recorded.

Appropriate information contained in the standard preparation logbook may be annotated in the instrument logbook. This has the advantage of correlating standards, QC checks, lot numbers, etcetera, to the appropriate analytical batch without additional searching of records.

#### 7.3.2 Analytical Data Record Keeping System

The need for a single, yet efficient, procedure for analytical data record keeping is paramount in reconstructing historical analytical records. Therefore, the analytical data record keeping system is designed for this procedure.

For each analytical instrument there is an associated record keeping system. Every analytical run made by an instrument is partially or completely documented by the system. A complete description of this procedure is detailed in Section D. of the QAM.

### 7.4 PHYSICAL SECURITY AND DOCUMENT CONFIDENTIALITY

Data may be compromised in many ways other than QA/QC measures normally associated with the validation of generated data. An important aspect of data integrity is answering the question and eliminating the possibility that data may have or could have been compromised by the lack of or inappropriate security measures. Physical security, security measures, document confidentiality and employee policies regarding ethics, waste, fraud and abuse are addressed and continuously monitored in order to generate data of the highest quality that will stand a legal challenge. Physical security, security measures and document confidentiality are generally of the following type:

- Building security,
- Perimeter - door security,
- Visitor security,
- Document confidentiality, and,
- Sample security.

#### 7.4.1 Building Security

Alpha Analytical, Inc. has installed a Z1100 security system through ADT Security Systems. The Z1100 is a digital communicator system monitored 24 hours a day by a central station. When the monitoring station receives an alarm from Alpha, it immediately contacts the correct response agency (ambulance, police or fire).

The Z1100 is an arming system programmed with a pre-alarm and automatic bell cutoff. Alpha has door contacts at all entrances. Passive and infrared motion detectors are strategically located throughout the laboratory to detect intruders in sensitive areas. Alpha has smoke and heat detectors in all areas where flammable

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chemicals or heat-generating equipment is located. Alpha also has glass breakage detectors on all high travel perimeter glass windows, where intruder entrance through a window could take place.

Alpha continuously updates the security codes to prevent unwanted entry by former employees or others who may have had access to security codes.

#### 7.4.2 Perimeter Door Security

The normal layout of Alpha's laboratory includes a series of perimeter access doors which would not intentionally be designed in the physical plant of a laboratory such as ours. However, since our laboratory is situated in an office building complex, there are a large number of perimeter access doors which necessitates additional perimeter security. The types of perimeter access doors have been identified and are as follows:

- Perimeter doors which will remain open at all times during our normal laboratory hours (e.g., the two main entrance doors);
- Perimeter doors not in direct line with any associated travel patterns of our employees, and which otherwise have no need to be opened for normal business practice; and,
- Perimeter doors in direct line with the travel path of most employees, and need to be secured in a way which allows unfettered access to employees while also being a deterrent to outside intrusions.

The majority of perimeter doors are one-way locking doors such that an employee may exit a perimeter door without a key. However, to gain access to the building would require an access code.

#### 7.4.3 Visitor Security

Unwanted physical intrusions may occur at any time unless a number of security measures are implemented. Once the premise has been secured, attention is now addressed to the non-employees whom may obtain access to our facility.

7.4.3.1 Visitors (e.g. non-employees) are given a visitor tag upon entrance to our facility.

Note: Clients logging in samples or visitors constrained to the main reception area are not required to sign the visitor logbook.

7.4.3.2 Visitors are required to sign-in upon arrival at the front desk, and answer the following questions:

- Date/Time of arrival,
- Company or institute they represent,
- Personal signature,
- Badge or ID number,
- Person whom they are visiting, and
- Date/Time of departure.

7.4.3.3 Visitors are required to place this badge on their garment in such a way to be highly visible at all times.

7.4.3.4 Visitors are required to be escorted to their area of interest and accompanied while on premises at all times.

7.4.3.5 Visitors without a badge will be escorted immediately to the front area and asked to remain there until their party arrives.

7.4.3.6 Visitors without badges also will be questioned to obtain the seriousness and extent of this breach of security and the possibility of data invalidation.

7.4.3.7 Visitors are required to return the badge and sign out at the front desk upon leaving our facility.

#### Visitor Log-In

Page: \_\_\_\_\_

Badge ID	Printed Name	Signed Name	Company you represent	Person Visiting	Date/Time Arrival	Date/Time Departure

#### 7.4.4 Document Confidentiality

7.4.4.1 All samples and project documents are considered to be confidential. Standard Business Records Confidentiality practices apply to all documents, materials and relevant information.

7.4.4.2 Specific procedures that are followed to maintain legal confidentiality include the following:

- 
- 7.4.4.2.1 All documents and files are secured in locked file cabinets or equally secured areas, i.e., secured building, during other than normal working hours, unless the files are personally attended by someone authorized to have access to such files;
- 7.4.4.2.2 Employees other than management, DCO or SCO are not allowed immediate or direct access to confidential files or documents without approval of the Laboratory Director; and,
- 7.4.4.2.3 All sample documents and any verbal information will only be released to the client or Principle Investigator (PI) who requested sample analysis.

Note: Persons or organizations, other than the client, requesting such information may only receive the information upon approval to release the data.

If there are any doubts concerning the identity of the organization or authority, then they must show proof of identification before Alpha will release information.

#### 7.4.5 Sample Security

Sample security is the responsibility of the person who has custody of the sample at any particular time. The overall responsibility resides with the SCO to ensure sample custody practices and procedures are being followed.

Sample storage refrigerators are not locked for most routine samples; however, occasionally a higher level of sample security and custody is required by a sensitive project. Alpha Analytical is prepared and can use locked and secured storage facilities for this purpose.

Samples that require separately locked storage facilities will be the responsibility of the SCO. Staff members associated with that particular project will be assigned a storage key and sample custody will remain with the designated project-specific personnel during laboratory activities.



# **Section 8**

## ***A*nalYTical Procedures**

## **8.0 ANALYTICAL PROCEDURES**

### **8.1 INTRODUCTION**

- 8.1.1 Before using an analytical method to analyze environmental samples, Alpha demonstrates the ability to perform that method of analysis with the prescribed degree of precision and accuracy.
- 8.1.2 Standardized analytical methods used by Alpha are generally published methods from recognized federal agencies (i.e., ASTM, SW-846, EPA's Methods for the Determination of Organic Compounds in Drinking Water etc.).
- 8.1.3 Most standardized methods require an initial demonstration of capability study which generates precision and accuracy data establishing a baseline typical of routine analysis. The initial demonstration of capability studies are used primarily to preclude a laboratory from analyzing unknown samples via a new, unfamiliar method prior to obtaining some experience with it.

### **8.2 ANALYTICAL METHODS**

Standardized analytical methods are described by a set of written procedures completely defining the techniques to be used to process a sample and obtain analytical results. Descriptions of analytes, sample matrix, sample preparation, types and quantities of reagents, instrumental calibration and measurement parameters, and computations are all integral parts of a complete method.

#### **8.2.1 Selection of Methods**

Analytical methods are selected for environmental testing, to meet the needs of our clients and which are cited in regulation as the appropriate methods for the specific regulatory programs.

#### **8.2.2 Sources of Standardized Methods**

- 8.2.2.1 Alpha uses standardized methods for commonly encountered analytes in order to provide a common point of reference, and to establish standard practices that allow inter-laboratory comparison of data. Alpha uses methods that are program specific and are typically referenced in the regulatory literature.

In addition to specifying sample preparation and analytical procedures, each method also typically specifies calibration procedures, calibration acceptance criteria, methods of preparing standard solutions, and preparation of QC samples. Laboratory methods used on a routine basis are provided in the following references.

- Test Methods for Evaluating Solid Waste, Physical/Chemical methods, SW-846, 3rd Edition, Final Update III, 1996 and various updates,
- Guidelines Establishing Test Procedures for the Analysis of Pollutants, 40 CFR, Part 136, Appendix A as updated,
- Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88/039, December 1988, Revised July 1991, EMSL Cincinnati, OH.
- Methods for the Determination of Organic Compounds in Drinking Water, Supplement I, EPA-600/4-90/020, July 1990, EMSL Cincinnati, OH,
- Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA-600/R-92/129, August 1992, EMSL Cincinnati, OH,
- Methods for the Determination of Organic Compounds in Drinking Water, Supplement III, EPA-600/R-95/131, 1995, EMSL Cincinnati, OH,
- Methods for the Determination of Inorganic Substances in Environmental Samples, EPA600/R-93/100, August 1993, EMSL Cincinnati, OH,
- Methods for the Determination of Metals in Environmental Samples, Supplement I, EPA-600/R-94/111, May 1994, EMSL Cincinnati, OH,
- Technical Notes on Drinking Water, EPA/600/R-94/173, October 1994, EMSL Cincinnati, OH,
- Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup> edition, 1992, and updates,
- Methods for Chemical Analysis of Water and Waste, EPA-600/4-79-020, Revised March 1983, EMSL, Cincinnati, OH.

8.2.2.2 The latest valid edition of these referenced methods are used unless it is not appropriate or impossible to do.

8.2.2.3 Requested methods of analysis are used unless it is not appropriate, or if we are not certified for the requested method and an equivalent certified method

exists, then that method will be used.

8.2.2.4 When the client does not specify a method to be used, Alpha only uses methods that have been fully documented and validated.

### 8.2.3 Procedure Manual

Each analytical method has an associated in-house analytical SOP and in total comprise our Procedure Manual. These SOPs are written, reviewed, approved, and distributed according to the procedures outlined in Appendix D. When the referenced methods are ambiguous or does not provide sufficient detail, our in-house analytical SOPs clarifies these issues as Clarification Boxes.

### 8.2.4 Laboratory Developed Methods

If an analytical test method is developed by Alpha, it will be planned and executed in a well organized, scientifically supported manner.

### 8.2.5 Non-Standard Methods

In the event that analyses must be conducted for compounds for which no reliable method exists, development of a method will be conducted under the supervision of the Laboratory Director. As part of the method development, and to ensure continuous quality of data, QC criteria is initially proposed and established that is consistent with similar methods or technology. At a minimum the following QC requirements are addressed:

- Calibration,
- Contamination,
- Precision and accuracy,
- Interference, and
- Analyte identification.

When testing of the analytical procedure has been successfully completed, the method is evaluated for scientific and technical soundness and is documented in the standardized format.

## 8.3 SUMMARY OF ANALYTICAL PROCEDURES

The analytical and extraction procedures presented in the following sections as outlined in Tables 8-1 through 8-5 are methods currently used at Alpha Analytical in support of the various environmental regulatory programs. A brief description of the methods are in the subsections following the tables.

## 8.4 ANALYTICAL PROCEDURES IN SUPPORT OF THE SAFE DRINKING WATER ACT (SDWA)

A series of inorganic methods of analysis are found in Methods for Chemical Analysis of Water and Wastes. These are a series of wet chemistry and various metals methods used in support of the SDWA. Standard Methods for the Examination of Water and Wastewater is also used in support of the SDWA; however, this reference contains inorganic wet chemistry, metals and organic methods of analysis. An additional series of methods written by the EPA covering all methods required under the SDWA is found in Methods for the Determination of Organic and Inorganic Compounds in Drinking Water and associated supplements. Methods of analysis Alpha Analytical uses in support of the SDWA is as follows:

### SDWA Methods of Analysis

Table 8-1

EPA METHODS	OTHER METHODS	PARAMETERS
Inorganic		
120.1	SM2510B	Conductivity
150.1	SM4500H B	pH
160.1	SM2540C	TDS
180.1	SM2130B	Turbidity
200.8		Metals
300.0		Anions
310.1	SM2320B	Alkalinity
314.0		Perchlorate
330.5	SM4500Cl G	Free Residual Chlorine
	SM3500Cr D	Chromium VI
	SM2340B	Hardness (calculated)
Organic		
524.2		Volatile Organics

### 8.4.1 Conductivity - EPA Method 120.1/Standard Method 2510B

Conductivity is the ability of a solution to pass a current. The amount of current a solution may conduct is proportional to the number of ions present in the sample. Therefore, conductivity is a measure of the total ionic concentration in a sample. Specific conductance of a sample is determined by the use of a self-contained conductivity meter at 25°C, thus standardizing the measurement by compensating for cell geometry and temperature.

#### 8.4.2 pH - EPA Method 150.1/Standard Method 4500H B

The pH of a sample is determined electrometrically using a combination electrode. The pH meter is calibrated using a series of standard pH buffers at a known pH.

#### 8.4.3 Total Dissolved Solids (TDS) - EPA Method 160.1/Standard Method 2540C

A well mixed sample is filtered through a standard glass-fiber filter, and the filtrate is evaporated to dryness in a weighed dish and dried to a constant weight of 180°C. The increase in dish weight represents the total dissolved solids.

#### 8.4.4 Turbidity - EPA Method 180.1/Standard Method 2130B

Turbidity measurement is based upon a comparison of the intensity of light scattered by the sample with the intensity of light scattered by a standard reference suspension.

#### 8.4.5 Metals - EPA Method 200.8

This procedure is a multi-elemental procedure for the determination of analytes by ICP-MS in environmental samples. Elements in solution are introduced by pneumatic nebulization and the resulting aerosol is transported by argon gas into a radio frequency plasma where the energy transfer process causes desolvation of the elements followed by atomization and ionization. The ions produced by high temperatures are entrained in the plasma gas and introduced, by means of a vacuum interface, into a mass spectrometer. The ions produced are sorted according to their mass-to-charge ratios by a quadrupole mass spectrometer and detected with the assistance of an electron multiplier. Isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents and sample matrix are corrected by the data acquisition software.

8.4.5.1 For the determination of total recoverable metals, analytes are solubilized by gentle refluxing with nitric and hydrochloric acids i.e., block digestion. After cooling, the sample is brought back to its original volume, mixed and centrifuged or allowed to settle overnight prior to filtration and sample analysis.

8.4.5.2 For the determination of dissolved metals in a filtered sample, or the direct analysis of analytes in drinking water samples where the sample turbidity is <1 NTU, the sample is made ready for analysis by the addition of nitric acid prior to sample analysis.

#### 8.4.6 Anions - EPA Method 300.0

A small volume of sample is introduced into an ion chromatograph to flush and fill a fixed volume sample loop. The sample is then injected into a mobile phase eluent

of carbonate-bicarbonate. The anions are separated and measured using an Ion Chromatograph (IC) comprised of a guard column, an analytical column, a suppressor device and the conductivity detector. The suppressor device reduces the amount of background conductivity of the carbonate-bicarbonate eluent to a negligible level. Anions are identified based on their retention times compared to known standards. An extraction procedure is performed on soil and/or solid samples prior to sample analysis.

#### 8.4.7 Alkalinity - EPA Method 310.1/Standard Method 2320B

An unaltered sample is titrated to an electrometrically determined end point of pH 4.5 for total alkalinity and to a second endpoint of 8.3 if the speciation of alkalinity is required. The sample is not filtered, diluted, concentrated, or altered in any way.

For samples of low alkalinity (less than 20 mg CaCO<sub>3</sub>/L) an extrapolation technique is used to determine the equivalence point. The amount of standard acid required to reduce the pH exactly 0.30 pH units beyond the normal end point of 4.5 corresponds to an exact doubling of the hydrogen ion concentration.

#### 8.4.8 Perchlorate - EPA Method 314.0

A volume of sample is introduced into an ion chromatograph to flush and fill a fixed 1.0 mL volume sample loop. The sample is then injected into a mobile phase eluent of 50 mM KOH. The perchlorate anion is separated and measured using an IC comprised of a guard column, an analytical column, a suppresser device and the conductivity detector. The suppresser device reduces the amount of background conductivity of the KOH eluent to yield a baseline with no more than 2-3 nanosiemen (nS) noise/drift per minute. Perchlorate is identified based on its retention time compared to known standards. Quantitation is accomplished by measuring peak area and comparing it to a calibration curve generated from known standards.

#### 8.4.9 Free Residual Chlorine - EPA Method 330.5/Standard Method 4500Cl G

Free residual chlorine also known as free available chlorine exists in most waters as hypochlorous acid or hypochlorite ion. These analytes react immediately with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color. The intensity of the pink color is proportional to the chlorine concentration. Chlorine is measured at a wavelength of 530nm.

#### 8.4.10 Chromium VI - Standard Method 3500Cr D

Hexavalent chromium is determined colorimetrically by a reaction with diphenylcarbazide in an acid solution. A purple color will appear if hexavalent chromium is present in the absence of interfering analytes. The concentration of

hexavalent chromium is determined by its absorbance measured photometrically at a wavelength of 540 nm.

#### 8.4.11 Hardness (Calculated) - Standard Method 2340B

Total hardness is defined as the sum of calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter. Although hardness can be determined a number of ways, the preferred procedure is to compute hardness from the results of separate determinations of calcium and magnesium as a ratio to calcium carbonate. Therefore, the calculation of "Hardness (calc), mg equivalent  $\text{CaCO}_3$  =  $2.497 [\text{Ca, mg/L}] + 4.118 [\text{Mg, mg/L}]$ .

#### 8.4.12 Volatile Organics - EPA Method 524.2

VOCs are purged from the sample by bubbling helium through a 25 mL aqueous sample. The purgeable organics are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are trapped. When purging is complete, the sorbent tube is heated and backflushed to desorb the trapped compounds into a gas chromatographic column. The GC is temperature programmed to separate the purgeable target analytes. Compounds eluting from the GC column are identified by comparing their measured mass spectra and retention times to reference spectra and retention times. Analytes are quantitated using an internal standard procedural calibration process.

### 8.5 ANALYTICAL PROCEDURES SUPPORTING THE CLEAN WATER ACT (CWA)

The Clean Water Act includes a promulgated series of methods enacted to satisfy the analytical requirements of a facility holding an NPDES discharge permit. The 600 series organic methods of analysis are found in 40 CFR, Appendix A, Part 136, and Methods for Organic Analysis of Municipal and Industrial Waste Water. These are a series of GC, GC/MS and HPLC methods for the determination of compounds that may be found in municipal and industrial discharges.

The inorganic methods of analysis are found in Methods for Chemical Analysis of Water and Wastes. These are a series of wet chemistry and various metals methods used in support of the CWA. These are the same procedures as used in support of the SDWA. Standard Methods for the Examination of Water and Wastewater is also used in support of the CWA. Methods of analysis Alpha Analytical uses in support of the CWA is as follows:



**Table 8-2**

EPA METHODS	OTHER METHODS	PARAMETERS
Inorganic		
120.1	SM2510B	Conductivity
150.1	SM4500H B	pH
160.1	SM2540C	TDS
160.2	SM2540D	TSS
160.3	SM2540B	TS
180.1	SM2130B	Turbidity
200.8		Metals
300.0		Anions
305.1	SM2310B	Acidity
310.1	SM2320B	Alkalinity
314.0		Perchlorate
330.5	SM4500Cl G	Total and Free Residual Chlorine
350.3 (350.2-distillation)	SM4500NH3 D (NH3 B-dist)	Ammonia
351.4	SM4500Norg C	Total Kjeldahl-N
365.2	SM4500P B&E	Total Phosphorus
376.2	SM4500S D	Sulfide
410.4	SM5220 D	COD
415.1	SM5310C	TOC
	SM2340B	Hardness (calculated)
	SM3500Cr D	Chromium VI
	SM3500Fe D	Ferrous or Total Iron
1664A		n-Hexane Extractable Material (Oil and Grease)
Organic		
608	SM6630C	Organochlorine Pesticides and PCBs
624		Purgeables
625		Semivolatile Base/Neutal and Acids

#### 8.5.1 Total Suspended Solids (TSS) - EPA Method 160.2/Standard Method 2540D

A well mixed sample is filtered through a weighed standard glass-fiber filter and the residue retained on the filter is dried to a constant weight of 103-105°C. The increase in weight of the filter represents the total suspended solids.

#### 8.5.2 Total Solids (TS) - EPA Method 160.3/Standard Method 2540B

A well mixed sample is evaporated in a weighed dish and dried to a constant weight in an oven at 103 to 105°C. The increase in weight over that of the empty dish represents the total solids.

#### 8.5.3 Acidity - EPA Method 305.1/Standard Method 2310B

The sample pH is determined and a measured amount of standard acid is added to lower the pH to 4 or less. If the initial sample pH is less than 4.0, the addition of acid is not required. This is an arbitrary inflection point, because accurate identification of inflection points may be difficult or impossible in buffered or complex mixtures.

The sample is oxidized with hydrogen peroxide because samples of industrial wastes, acid mine drainage, or other solutions that contain appreciable amounts of hydrolyzable metal ions such as iron, aluminum or manganese may exist in other reduced forms of the polyvalent cations. The sample is subsequently boiled to hasten hydrolysis.

The sample is cooled and titrated electrometrically with standard alkali to a pH of 8.3. The titration to an end point of 8.3 corresponds to the stoichiometric neutralization of carbonic acid to bicarbonate and is reported as total acidity (pH 8.3). This end point is generally accepted as the standard of total acidity, including CO<sub>2</sub> and most weak acids. However, for more complex mixtures or buffered solutions such as waste waters or grossly polluted waters, use two end points, 3.7 and 8.3 for standard acidity determinations where simple carbonate equilibria cannot be assumed. In this case acidity is reported as “methyl orange acidity” (pH 3.7) and total acidity (pH 8.3).

#### 8.5.4 Total Residual Chlorine - EPA Method 330.5/SM4500-Cl G

##### 8.5.4.1 Free Residual Chlorine (A fraction)

Free residual chlorine also known as free available chlorine exists in most waters as hypochlorous acid or hypochlorite ion. These analytes react immediately with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color. The intensity of the pink color is proportional to the chlorine concentration. Chlorine is measured at a wavelength of 530nm (A reading).

##### 8.5.4.2 Combined Chlorine (B-A fraction) + ( C-A fraction)

Chloroanimes analyzed and reported as combined chlorine may be estimated with an additional sample preparation procedure.

#### Monochloramine (B-A fraction)

The monochloramine fraction (B-A) is estimated by the addition of 0.1mg of KI per 10 mL sample volume using the same sample as used in the free residual analysis followed by immediate sample analysis (B reading).

#### Dichloramine ( C-A fraction)

The dichloramine fraction may further be estimated by the addition of 0.2g KI per 10 mL sample volume using this same sample followed by immediate analysis.

A simplified approach for determining monochloramine and dichloramine together as combined chlorine would be to add 0.2g KI per 10 mL sample volume and not fractionate the combined chlorine subfractions (total residual chlorine). To determine the concentration of the combined chlorine, run a free residual chlorine test. Subtract the results of the free chlorine test from the total chlorine test to obtain the combined chlorine concentration.

#### 8.5.4.3 Total Chlorine (A fraction) + (B-A fraction) + ( C-A fraction)

Total chlorine may be determined as the addition of free residual and combined chlorine. This is accomplished in a single spectrophotometric reading by adding the full amount of KI, 0.2g per 10 mL sample volume, at the start of the analysis along with the DPD indicator.

Total chlorine analysis is actually a determination of the iodine present in a sample. Iodine is produced in a stoichiometric relationship with combined chlorine from the addition of KI. The combined chlorine oxidizes iodide in the reagent to iodine. The iodine and free chlorine react with DPD to form a red color which is proportional to the total chlorine concentration.

#### 8.5.5 Ammonia - EPA Method 350.3/SM4500NH<sub>3</sub> D

##### 8.5.5.1 Distillation Step - EPA Method 350.2/SM4500NH<sub>3</sub> B

Samples are buffered to a pH of 9.5 with a borate buffer solution to decrease hydrolysis of organic nitrogen compounds. Samples are then distilled into a weak sulfuric acid solution for final ammonia determination.

##### 8.5.5.2 Analysis

The ammonia concentration of a sample is determined potentiometrically using an ion selective gas-sensing combination ammonia electrode. Dissolved ammonia ( $\text{NH}_{3(\text{aq})}$  and  $\text{NH}_4^+$ ) are converted to  $\text{NH}_{3(\text{aq})}$  by raising the pH to above 11 with a strong base. The gas sensing electrode responds to dissolved ammonia gas in solution. The dissolved ammonia gas diffuses across the membrane into a small volume of buffer, specific to the ammonia electrode. Reaction of the gas with the buffer causes a pH change sensed by an internal pH electrode. The fixed level of chloride in the internal fill solution is sensed by a chloride ion-selective electrode that serves as the reference electrode. Because the reference electrode is built-in, a separate reference electrode is not necessary.

This same procedure using a gas-sensing electrode can also be used to measure ammonium ions after conversion to ammonia, or organic nitrogen after Kjeldahl digestion of the sample.

#### 8.5.6 Total Kjeldahl Nitrogen (TKN)-EPA Method 351.4/Standard Method SM4500NH<sub>3</sub>org C

Prior to the distillation or analysis of ammonia as described above, the sample is heated in the presence of concentrated sulfuric acid, potassium sulfate and copper sulfate until the solution becomes colorless or pale blue-green. The ammonia is subsequently distilled from the sample and determined potentiometrically.

#### 8.5.7 Total Phosphorus - EPA Method 365.2/Standard Method SM4500P B&E

The determination of phosphorus can generally be summed up in two steps: a) conversion of the various forms of phosphorous to ortho-phosphate and b) colorimetric determination of ortho-phosphate.

Total phosphorus procedure converts organic and inorganic phosphorous to the ortho-phosphate form by a persulfate digestion.

Acid-hydrolyzable phosphorous or polyphosphates forms of phosphorus are converted to the orthophosphate form by sulfuric acid hydrolysis. This form of phosphorus will contain free orthophosphate plus a small amount of organically bound phosphorous. Therefore acid-hydrolyzable phosphorous is reported as the difference between the results obtained using the hydrolysis procedure and the results of ortho-phosphate analyzed directly without acid hydrolysis.

These preparatory procedures are then followed by the analysis of orthophosphate. Ortho-phosphate also known as reactive phosphorus is determined directly without any sulfuric acid hydrolysis or persulfate digestion prior to sample analysis. Reactive phosphorus is essentially a measure of ortho-phosphate, plus a small fraction of condensed or polyphosphate that may have been hydrolyzed during the reaction. Orthophosphate reacts with molybdate in an acid medium to produce a

phosphomolybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.

#### 8.5.8 Sulfide - EPA Method 376.2/Standard Method SM4500S D

The methylene blue method is based on the reaction of sulfide, ferric chloride, and N,N-dimethyl-p-phenylenediamine in a strongly acidic solution to form the dye methylene blue. The excess color due to ferric chloride is removed by the addition of diammonium hydrogen phosphate.

##### 8.5.8.1 Total Sulfide

Hydrogen sulfide and acid-soluble metal sulfides are collected and preserved with zinc acetate which forms the insoluble ZnS, and further basified with sodium hydroxide. This preservation and extraction treatment limits the loss (volatilization) of potential sulfide prior to sample analysis. Interferences are removed from the sample (and the sample concentrated) by carefully withdrawing the supernatant liquid from the ZnS precipitate, and either replacing the removed water with deionized water or leaving at the lesser volume for sample concentration. Sulfide is then color developed by the reaction with N,N-dimethyl-p-phenylenediamine sulfate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. Sulfide is measured at a wavelength of 665 nm.

##### 8.5.8.2 Dissolved Sulfide

Dissolved sulfide may be determined after the suspended solids have been removed by flocculation and settling prior to color development and sample analysis.

#### 8.5.9 Chemical Oxygen Demand (COD) - EPA Method 410.4/Standard Method SM5520D

The mg/L COD results are defined as the mg of  $O_2$  consumed per liter of sample under conditions outlined in this procedure. Samples are prepared by a closed-reflux digestion procedure with sulfuric acid along with an excess of potassium dichromate ( $K_2Cr_2O_7$ ) followed by sample analysis. The COD reagents also contain silver and mercury ions. Silver sulfate ( $Ag_2SO_4$ ) serves as a catalyst to assist oxidation of straight-chain hydrocarbons such as diesel fuel and motor oil and mercury is used to control the chloride interferences. The sample is digested at a temperature of  $150^\circ C$  for two hours to drive the reaction to completion. During digestion the samples carbon bearing compounds are oxidized by the acid and potassium dichromate, reducing the dichromate ion ( $Cr_2O_7^{2-}$ ) to green chromic ion ( $Cr^{3+}$ ) while the organic matter and inorganic carbon compounds are oxidized to  $CO_2$  and  $H_2O$ . Colorimetric analysis is suitable for COD because the two chromium ions absorb at different wavelengths in the visible range  $Cr^{3+}$  at 620nm and  $Cr^{6+}$  at 420 nm.

Note: Method 410.4 is designed to measure only the chromic ion ( $\text{Cr}_3^+$ ) and Method SM5220 D has the option to calibrate and analyze either the dicromate ion or the chromic ion. We use the low level option by measuring the  $\text{Cr}_6^+$  ion for calibration and quantitation.

#### 8.5.10 Total Organic Carbon (TOC) - EPA Method 415.1/Standard Method 5310C

Water samples typically contain both organic and inorganic carbon. Inorganic carbon, (typically in the forms of carbonate and bicarbonate) is removed by acidification and purging prior to sample analysis. Organic carbon in the sample is then converted to carbon dioxide ( $\text{CO}_2$ ) by persulfate oxidation and UV irradiation. The  $\text{CO}_2$  formed is stripped from the sample with a stream of gas and measured directly by a non-dispersive infrared (NDIR) detector. The amount of  $\text{CO}_2$  is directly proportional to the concentration of organic carbon material in the sample.

#### 8.5.11 Ferrous and Total Iron - Standard Method SM3500Fe D

##### 8.5.11.1 Total Iron

All forms of iron are brought into solution by an acid digestion prior to color development. FerroVer Iron Reagent converts, by reducing, all soluble iron and most insoluble forms of iron in the sample to the soluble ferrous iron. Three molecules of 1,10-phenanthroline chelate each atom of ferrous iron to form an orange-red complex. The color in solution obeys Beer's law and is proportional to the iron concentration. The concentration of total iron is determined by its absorbance measured photometrically at a wavelength of 510 nm.

##### 8.5.11.2 Ferrous Iron ( $\text{Fe } 2^+$ )

The 1,10-phenanthroline indicator in the Ferrous Iron Reagent pillow reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react.

##### 8.5.11.3 Ferric Iron ( $\text{Fe } 3^+$ )

The ferric iron concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test.

#### 8.5.12 Pesticides and PCBs - EPA Method 608/Standard Method 6630C

This procedure is a GC method used to determine Organochlorine pesticides and PCBs. A measured volume of water sample is extracted with methylene chloride,

dried, exchanged into hexane and concentrated. The extract is separated by a GC equipped with an Electron Capture Detector for the identification of the target compounds.

#### 8.5.13 Purgeables - EPA Method 624

Method 624 is a GC/MS method used in the determination of a number of volatile organics in industrial and municipal wastewater. Helium is bubbled through a 25 ml water sample contained in a specially 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 trap where the purgeables are trapped. After purging is completed, the trap is heated and back-flushed to desorb the purgeables onto the GC. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

#### 8.5.14 Base/Neutrals and Acids - EPA Method 625

Method 625 is a GC/MS procedure used in the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. A measured sample volume is extracted with methylene chloride at a pH greater than eleven and again at a pH less than two using a separatory funnel or by mechanical tumbling. The methylene chloride extract is dried, concentrated and analyzed by GC/MS.

### 8.6 ANALYTICAL PROCEDURES IN SUPPORT OF THE RESOURCE CONSERVATION AND RECLAMATION ACT (RCRA)

Several of the hazardous waste regulations under Subtitle C of RCRA require that specific test methods described in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Final Update III (SW-846) be employed for certain applications. Specific requirements are found in 40 CFR, part 140 through 290.

SW-846 provides the test procedures and guidelines for field and laboratory quality control, sampling, determining hazardous constituents in waste, determining the hazardous characteristics of waste (toxicity, ignitability, reactivity, and corrosivity) and for determining the physical properties of waste. Methods of analysis Alpha Analytical uses in support of RCRA is as follows:

**RCRA Methods of Analysis**  
**Table 8-3**

EPA METHODS	OTHER METHODS	PARAMETERS
Inorganic		

SW6020/6020A		Metals
SW7196A		Chromium (VI)
SW9040B/9045C		Corrosivity
SW9050A		Conductivity
SW9056		Anions
SW9058		Perchlorate
SW9060		TOC
Organic		
SW8015B-DRO	NWTPH-dx & 8015AZ	Total Petroleum Hydrocarbons (Diesel Range)
SW8015B-GRO	NWTPH-gx & 8015AZ	Total Petroleum Hydrocarbons (Gasoline Range)
SW8081A		Organochlorine Pesticides
SW8082		Polychlorinated Biphenyls (PCBs)
SW8260B		Volatile Organics
SW8270C		Semi-volatile Organics

#### 8.6.1 Metals - EPA Method SW6020/6020A

An aliquot of a well mixed, aqueous or solid sample is weighed or measured for sample processing. For total metals analysis, analytes are solubilized by acid digestion. After cooling, the sample is made up to volume prior to analysis.

This procedure is a multi-elemental procedure for the determination of analytes by ICP-MS in environmental samples. This method measures ions produced by a radio frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol is transported by argon gas into the plasma torch. The ions produced by high temperatures are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with an electron multiplier.

#### 8.6.2 Hexavalent Chrome - EPA Method 7196A

Hexavalent chromium is determined colorimetrically by a reaction with diphenylcarbazide in an acid solution. A purple color will appear if hexavalent chromium is present in the absence of interfering analytes. The concentration of hexavalent chromium is determined by its absorbance measured photometrically at a wavelength of 540 nm.

An alkaline digestion procedure using a 0.28M Na<sub>2</sub>CO<sub>3</sub>/0.5M NaOH solution and



heating the digestate to 90-95°C for 60 minutes is required to extract hexavalent chromium in soils prior to color development and spectrophotometric analysis. The pH of the sample digest must be carefully adjusted during the digestion procedure and temperature monitored to ensure the complete dissolution of Cr(VI) and stabilize it against reduction to Cr(III).

#### 8.6.3 Conductivity - EPA Method 9050A

Conductivity is the ability of a solution to pass a current. The amount of current a solution may conduct is proportional to the number of ions present in the sample. Therefore, conductivity is a measure of the total ionic concentration in a sample. Specific conductance of a sample is determined by the use of a self-contained conductivity meter at 25°C, thus standardizing the measurement by compensating for cell geometry and temperature.

#### 8.6.4 Anions - EPA Method SW9056

A small volume of sample is introduced into an ion chromatograph to flush and fill a fixed volume sample loop. The sample is then injected into a mobile phase eluent of carbonate-bicarbonate. The anions are separated and measured using an Ion Chromatograph (IC) comprised of a guard column, an analytical column, a suppressor device and the conductivity detector. The suppressor device reduces the amount of background conductivity of the carbonate-bicarbonate eluent to a negligible level. Anions are identified based on their retention times compared to known standards. An extraction procedure is performed on soil and/or solid samples prior to sample analysis.

#### 8.6.5 Perchlorate - EPA Method 9058

A volume of sample is introduced into an ion chromatograph to flush and fill a fixed 1.0 mL volume sample loop. The sample is then injected into a mobile phase eluent of 50 mM KOH. The perchlorate anion is separated and measured using an IC comprised of a guard column, an analytical column, a suppresser device and the conductivity detector. The suppresser device reduces the amount of background conductivity of the KOH eluent to yield a baseline with no more than 2-3 nanosiemen (nS) noise/drift per minute. Perchlorate is identified based on its retention time compared to known standards. Quantitation is accomplished by measuring peak area and comparing it to a calibration curve generated from known standards.

#### 8.6.6 Total Organic Carbon (TOC) - EPA Method SW9060

Inorganic Carbon (IC), carbonate and bicarbonate is removed by acidification and purging. Sample purging also removes Purgeable Organic Carbon (POC) so that the organic carbon measurement made after eliminating IC interferences is actually a

Non Purgeable Organic Carbon (NPOC) determination. Therefore, in practice, the NPOC determination is substituted for TOC. Organic carbon in the sample is then converted to carbon dioxide (CO<sub>2</sub>) by persulfate oxidation. The CO<sub>2</sub> formed is measured directly by a non-dispersive infrared detector. The amount of CO<sub>2</sub> is directly proportional to the concentration of carbonaceous material in the sample.

#### 8.6.7 Total Petroleum Hydrocarbons (TPH) - Extractable

##### 8.6.7.1 8015B-DRO

This method is applicable to the analysis of semi-volatile petroleum hydrocarbons, commonly referred to as Diesel Range Organics (DRO). DROs typically correspond to the range of petroleum compounds from C<sub>13</sub> to C<sub>22</sub>; however, this range may be changed as required. Diesel fuel is used as the default standard for quantitation of petroleum hydrocarbons identified in this C range. Samples are solvent extracted and analyzed by GC/FID.

##### 8.6.7.2 NWTPH-dx

This method covers the analysis of semi-volatile petroleum products (i.e., kerosene through heavy fuel oils), by pattern matching ("fingerprinting"), and quantitation of petroleum products based on that particular petroleum fuel. When the petroleum product is unknown, diesel is used as the default standard for quantitation. In general, those petroleum products which do not contain a substantial volatile "gasoline" fraction, should be analyzed by this method. A clean-up procedure, which may be used to aid in the removal of non-petroleum based organic interferences, i.e. biogenic interferences, has also been included.

##### 8.6.7.3 8015AZ

This method was written for the analysis of petroleum hydrocarbons in the range of C<sub>10</sub> - C<sub>32</sub> in soil. The C<sub>10</sub> - C<sub>22</sub> range is known as DRO (Diesel Range Organics) and the C<sub>22</sub> - C<sub>32</sub> range is known as the ORO (Oil Range Organics). Samples are quantitated against a diesel or 30 weight standard based on the petroleum product determined in the samples. Samples are solvent extracted and analyzed by GC/FID.

#### 8.6.8 Total Petroleum Hydrocarbons (TPH) - Purgeable

##### 8.6.8.1 8015B-GRO

This method is applicable to the analysis of volatile petroleum hydrocarbons, commonly referred to as Gasoline Range Organics (GRO). The SW846

Method 8015B-GRO specifies a C range of C<sub>6</sub> to C<sub>10</sub> using 2-methylpentane and 1,2,4-trimethylbenzene as the C range markers. Our policy is to define GROs as those hydrocarbons which correspond to the range of alkanes from C<sub>4</sub> to C<sub>13</sub>; however, this may be changed as required. Gasoline is used as the default standard for quantitation and includes compounds from C<sub>4</sub> to C<sub>13</sub>. Samples are analyzed by the GC/MS purge-and-trap procedure.

#### 8.6.8.2 NWTPH-gx

This method covers the analysis of volatile petroleum hydrocarbons (e.g. gasoline, naptha, mineral spirits, etc.), by pattern matching ("fingerprinting"), and quantitation of petroleum products based on that particular petroleum fuel if possible. When the petroleum product is unknown, regular unleaded gasoline is used as the default standard for quantitation. In general, those petroleum products which contain a substantial volatile fraction (i.e., the majority of the components eluting within the gasoline range), should be analyzed by this method.

#### 8.6.8.3 8015AZ

This method was written for the analysis of petroleum hydrocarbons in the range of C<sub>10</sub> - C<sub>32</sub> in soil. This method may also be extended to quantitate C<sub>6</sub> - C<sub>10</sub> hydrocarbon range using a purge-and-trap procedure. C<sub>6</sub> - C<sub>10</sub> is known as GRO (Gasoline Range Organics) and are quantitated against a gasoline standard. Soil samples are extracted in methanol.

#### 8.6.9 Organochlorine Pesticides - EPA Method SW8081A

Method 8081A is used to determine the concentration of various Organochlorine Pesticides in extracts from solid and liquid matrices. A measured volume or weight of sample is extracted using the appropriate sample extraction technique. After the sample has been extracted and dried it is exchanged into hexane for final concentration. The extract is injected into a GC equipped with an Electron Capture Detector for separation and quantitation. All compounds identified tentatively in the primary analysis are confirmed on a dissimilar GC column.

#### 8.6.10 Polychlorinated Biphenyls (PCBs) - EPA Method SW8082

Method 8082 is used to determine the concentration of the several common PCBs as Aroclors or as individual PCB congeners in extracts for solid and liquid matrices. A measured volume or weight of sample is extracted using the appropriate sample extraction technique. After the sample has been extracted and dried, it is exchanged into hexane for final concentration. The extract is injected into a GC equipped with

an Electron Capture Detector for separation and quantitation. All compounds identified tentatively in the primary analysis are confirmed on a dissimilar GC column.

#### 8.6.11 Volatile Organics - EPA Method SW8260B

Volatile (or purgeable) organics in water and soil samples are analyzed using method SW8260B. This method uses a gas chromatography mass spectrometry technique. Volatile compounds are introduced into the GC by purge and trap (SW5030B). Helium gas is bubbled through the sample to transfer the purgeable organic compounds from the liquid to vapor phase. Soil samples are extracted with methanol before purging or are directly sparged with a special purge and trap device. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is back- flushed and heated to desorb the purgeable organics onto a capillary GC column where they are separated and then detected with a mass spectrometer. The Internal Standard (IS) procedure is used for the quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample.

#### 8.6.12 Semi-volatiles - EPA Method SW8270C

Semi-volatile organics (also known as base/neutral and acid extractables) in water and soil samples are analyzed using method SW8270C. This technique quantitatively determines the concentration of a number of SVOCs. Samples are extracted and both base/neutral and acid extracts are then combined and concentrated through evaporation. Compounds of interest are separated and quantified using a capillary column GC/MS. The IS procedure is used for quantitation of target analytes. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample.

### 8.7 SAMPLE EXTRACTION

#### 8.7.1 Water Sample Preparation

The need to filter water samples depends on whether total or dissolved contaminants are of interest. The Project Coordinator or client will determine this prior to sample collection for each specific site/project.

Samples which are analyzed only for dissolved analytes such as metals must be filtered prior to chemical preservation. Analysis for volatile organic compounds and oil/grease are the only two universal exceptions to this guideline; they are never filtered. Samples may be filtered in the laboratory prior to extraction if requested by the client. The filter material used by Alpha is chosen on the basis of compatibility factors between the filter paper and the analytes of interest. Compatibility is defined in the following way:

- The sample being filtered is not changed by the filter material; and,
- The filter paper does not absorb or leach out the chemical analytes of interest.

Generally, particulate matter is not considered to be a natural component of groundwater, and normally will be filtered through a 0.45 micron filter prior to analysis, especially if the particulate matter is suspected of interfering with sample workup except for VOCs and Oil and Grease analyses. Filtration of drinking water or tap water will occur if specified by the method of analysis (e.g. all HPLC methods require filtration prior to analysis).

Organic samples for RCRA analysis are prepared using the EPA 3500 series methods when appropriate. In particular, water samples are prepared according to methods 3510C, separatory funnel, method 3520C continuous liquid-liquid extractions, and method SW3535 Solid Phase Extraction (SPE). These preparation methods are used specifically with the 8000 series methods of analysis.

Inorganic RCRA samples are generally prepared by one of two procedures, EPA method 3010, block digestion or method 3015, microwave digestion. These two acid digestion procedures are used with analytical method 6020 as well as various other analytical metals procedures.

Methods of analysis in support of the SDWA and CWA have their extraction or digestion procedures written into the analytical procedure. Even though they do not have an extraction method assigned to them; they are essentially the same procedures as outlined in the equivalent RCRA procedures. Method specific extraction procedures can be found in the various analytical SOPs.

#### 8.7.2 Soil/Sediment Sample Preparation

Soil and sediment samples are complex mixtures, even within a single sample site. Therefore, surrogate and analyte recovery depends on many factors, including organic content, mineral content, particle size and moisture content of the soil. Soil and sediment samples are analyzed in the condition they are received. Soil samples are generally prepared for extraction or digestion as follows:

The sample is mixed as thoroughly as possible in the original wide-mouth glass bottle by shaking or stirring. Glass rods are used for stirring. If samples have analyses for both volatile organic compounds and other analyses, the VOC sample preparation activity takes precedence before any other sample work-up and subsequent sample homogenization.

Generally, samples are quantitated on a “wet-weight” basis. When necessary, samples are quantitated on a “dry-weight” basis and the percent dry weight content

of the sample is determined.

For each soil sample, an aliquot of the sample is dried according to the procedure established in Standard Operating Procedure for Percent Dry Weight and Percent Moisture. Soils are weighed and dried at 105°C for no less than four hours. The calculated % dry weight for each sample is determined and used in final analytical determinations.

The determination of % dry weight is calculated as follows:

$$\% \text{ dry weight} = \frac{\text{Sample Weight (Dry)}}{\text{Sample Weight (Wet)}} \times 100$$

Composite samples are proportioned according to the number of samples and % dry weight content is determined.

Organic sample extractions for soil are prepared according to EPA Methods 3540C, soxhlet extraction; Method 3545, Pressurized Fluid Extraction (PFE) or Method 3550A, sonication. Exceptions to these techniques are method specific as outlined in the SOPs found in Appendix E.

Inorganic soil sample digestion procedures for the analysis of metals are generally prepared according to EPA method 3051, micro-wave digestion.

#### 8.7.3 Sample Batch

Samples are routinely analyzed by a batch system. Alpha uses two types of batch systems: 1) an extraction batch and 2) an analytical batch. An extraction batch consists of a maximum of 20 samples, that can be extracted together. An analytical batch is any number of samples that can be analyzed during an 8 or 12 hr GC/MS period which is associated with a MS tune.

GC methods usually require calibration verification standards analyzed at a specific sample frequency; however, this does not preclude the analyses of additional standards interspersed with samples. The rate of sample collection or shipment does not determine maximum batch size, although it may limit the number of samples available for analysis at a given time. A batch may consist of samples from more than one client. However, all samples in one batch must be completely processed through any given step in the same time period.

### 8.8 SAMPLE PREPARATION METHODS

Most methods of analysis have an associated extraction or digestion procedure to prepare

the sample prior to final analysis. RCRA procedures separates the preparatory procedure from the analytical procedure in well defined discrete written protocols. The SDWA and CWA have these preparatory procedures written within the analytical method. These procedures are essentially the same regardless of program; however, the following preparatory methods are those specified and delineated in the RCRA procedures.

#### 8.8.1 Semi-volatile Organic Preparation Methods

Water, soil, sediment, sludge and waste samples requiring organic analysis such as; base/neutral and acid extractables, or organochlorine pesticides, must undergo solvent extraction prior to sample analysis. There are six primary methods used to extract the semi-volatile target analytes from the sample matrix when the 8000 series of analytical methods are requested. However, Alpha typically uses only two of these procedures on a regular basis; Method 3510C and Method 3545. Extraction procedures for liquid and solid matrices presented in this section are outlined in Table 8-4.

### **SAMPLE PREPARATION METHODS FOR SEMI-VOLATILE ORGANIC ANALYSIS**

**Table 8-4.**

<b>SAMPLE PREPARATION METHOD NUMBER</b>	<b>DESCRIPTION</b>	<b>MATRIX</b>	<b>ASSOCIATED ANALYTICAL METHODS</b>
SW3510C	Liquid-Liquid Extraction	Water	SW8081A, SW8082, SW8270C
SW3545	Pressurized Fluid Extraction	Soil	SW8081A, SW8082, SW8270C

##### 8.8.1.1 Separatory Funnel Liquid-Liquid Extraction - EPA Method SW3510C

Method SW3510C is designed to quantitatively extract non-volatile and semi-volatile organics from liquid samples using standard separatory funnel techniques. The sample and the extracting solvent must be immiscible in order to yield recovery of target compounds. Dichloromethane is the solvent most commonly used for this application. Subsequent cleanup and detection methods are described in the organic analytical method used to analyze the extract.

##### 8.8.1.2 Pressurized Fluid Extraction (PFE) - EPA Method SW3545

Method SW3545 is a procedure for extracting water soluble or slightly water soluble semi-volatile compounds for solids. The method uses elevated temperature (100°C) and pressure (1500-2000 psi) to extract compounds using less solvent and time than equivalent procedures.

#### 8.8.2 Volatile Organic Preparation Methods

Soil, sediment, sludge and waste samples requiring volatile organic analysis must undergo one of two primary preparation procedures. EPA method SW5030B is a direct purge and trap procedure where samples are analyzed directly with no sample preparation and are therefore, analyzed as a direct injection. The second procedure is method SW5035 which sparges the VOC compounds directly from the soil sample diluted into water using a specially designed sparging chamber and is also a soil preparation procedure. VOC preparatory procedures for solid matrices presented in this section are outlined in Table 8-5.

**SAMPLE PREPARATION METHODS  
FOR VOLATILE ORGANIC ANALYSIS**

**Table 8-5.**

<b>SAMPLE PREPARATION METHOD NUMBER</b>	<b>DESCRIPTION</b>	<b>MATRIX</b>	<b>ASSOCIATED ANALYTICAL METHODS</b>
SW5030B	Purge-and-Trap	Water / Soil	SW8260B, SW8015B (GRO)
SW5035	Purge-and-Trap	Soil	SW8260B, SW8015B (GRO)

**8.8.2.1 Purge-and-Trap - EPA Method SW5030B**

Method SW5030B is a procedure for the extraction and introduction of VOCs for analysis. This method is applicable to nearly all types of sample matrix including such things as aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, water, tar, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils and sediments.

Helium gas is bubbled through the sample solution at ambient temperature to transfer the volatile components to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. When the purge is completed, the solvent cartridge is heated and back flushed with inert gas to desorb the volatile components onto the GC column.

Water samples are analyzed directly for volatile organic compounds by this purge-and-trap technique. Liquids that are immiscible in water, solids and wastes are extracted with methanol prior to the purge-and-trap procedure.

**8.8.2.2 Closed System Purge-and-Trap - EPA Method SW5035**

This method is a closed-system purge-and-trap procedure for the analysis of VOCs in solid materials. This method has two very distinct preparation procedures; one for low levels of VOCs and a high level VOC procedure.

The low level procedure uses a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis.



Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transportation, handling, and analysis are negligible.

The high level procedure uses a methanolic extraction procedure in conjunction with method SW5030B for final sample analysis.

### 8.8.3 Inorganic Preparation Methods

There are two primary digestion procedures used to prepare samples for final metals analysis. The following description of the preparatory procedures may be used for ICP-MS, ICP-OES, or many other furnace methods of analysis.

#### 8.8.3.1 Micro-wave Assisted Digestion of Aqueous Matrices - EPA Method 3015

An aqueous sample is digested in concentrated nitric acid or, optionally, a mix of concentrated nitric acid and concentrated hydrochloric acid, for a specific time using microwave heating. The temperature of the acid-sample mixture is brought to a predetermined maximum temperature and maintained at this temperature for a specific time to accelerate the extraction process. The sample and acids are placed in an acid resistant micro-wave safe TFM vessel. The vessel is sealed and heated in the microwave unit. After cooling, the vessel contents are filtered, centrifuged, or allowed to settle and then diluted to volume and analyzed.

#### 8.8.3.2 Micro-wave Assisted Digestion of Solid Matrices - EPA Method 3051

A soil sample is digested in a mix of concentrated nitric and hydrochloric acid for a specific time using microwave heating. The temperature of the acid-sample mixture is brought to predetermined maximum temperature and maintained at this temperature for a specific time to accelerate the digestion process. The sample and acids are placed in an acid resistant micro-wave safe TFM vessel. The vessel is sealed and heated in the microwave unit. After cooling, the vessel contents are filtered, centrifuged, or allowed to settle and then diluted to volume and analyzed.

### 8.8.4 Extraction Test Procedures for Hazardous Waste

There are two primary extraction tests using buffered reagents to simulate particular environmental conditions in order to determine if a solid waste exhibits the characteristic of toxicity. These procedures are referred to as: 1) Toxicity Characteristic Leaching Procedure (TCLP) or 2) Synthetic Precipitation Leaching Procedure (SPLP). These procedures are used if the total concentration in the waste equals or exceeds the Maximum Concentration of Contaminants for the Toxicity

Characteristic (TC) Limits.

#### 8.8.4.1 Toxicity Characteristic Leaching Procedure (TCLP) - EPA Method SW1311

Method SW1311 is an extraction procedure, using a buffer system similar to acid rain used for the determination of the concentration of organic (volatile and semi-volatile) and inorganic analytes that are leachable from waste or other materials.

#### 8.8.4.2 Synthetic Precipitation Leaching Procedure (SPLP) - EPA Method SW1312

Method SW1312 is designed to determine the mobility of both organic and inorganic analytes present in liquids, solids, and wastes. This procedure is exactly like the TCLP procedure with the exception of a different buffering medium.

### 8.9 GENERAL LABORATORY OPERATIONS

There are numerous activities required by our laboratory to be executed with minimal mistakes on a routine basis. Many of these activities are critical in the overall production of analytical methods and in some way influence the QA and QC of the laboratory. Many of these procedures have SOPs not only because they are routine activities, but also because they are regulated by law or by a regulatory agency. Activities other than analytical methods and extraction procedures which have written SOPs are as follows:

- Dish Washing and Steam Scrubber Operations, Appendix E,
- Manual Glassware Cleaning, Appendix E,
- Sample Container Cleaning Procedure, Appendix E,
- Prevention of Sample Contamination, Appendix E,
- Standards Preparation, Appendix E,
- Storage Blank Procedures, Appendix E,
- A Practical Guide for Performing a Demonstration of Capabilities (DOC) and Method Detection Limit (MDL) Study, Appendix E,
- Manual and Automated Integration Procedures, Appendix E,
- Waste Disposal, Appendix C,
- Preparation of Reagent Grade Water, Appendix E,

- Sample Compositing and Sub-sampling Procedure, Appendix E, and
- A Practical Guide to Performing Initial Calibration, Calibration Model Determination and Calibration Verification, Appendix E,

# **Section 9**

## **C**alibration Procedures and Frequency

## **9.0 CALIBRATION PROCEDURES AND FREQUENCY**

### **9.1 Instrument Calibration**

The following standard specifies the essential elements that define the procedures and documentation for initial calibration and continuing instrument calibration verification to ensure that the data is of a known quality and is appropriate for a given regulation or decision.

This standard does not specify the detailed procedural steps (“how to”) for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical method prescribed procedures and statistical approaches currently applicable for calibration.

#### **9.1.1 Initial Instrument Calibration**

The following items are defined as essential elements of initial instrument calibration:

- a) The details of the initial instrument calibration procedures including calculations, integrations, acceptance criteria and associated statistics is included and/or referenced in the test method SOP (Procedural Manual).
- b) Raw data records are retained to permit the reconstruction of the initial calibration. Raw data records include such things as: calibration date, test method, instrument, analysis date, each analyte name, analyst’s initials or signature, concentration and response, calibration curve or response factor, or the unique equation or coefficient used to reduce the instrument response to concentration.
- c) Sample results are quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification, unless otherwise required by method or program.
- d) Second Source Standard (Initial Calibration Verification Standards)

All initial instrument calibrations are verified with a standard obtained from a second manufacturer or lot if the lot can be demonstrated from the manufacturer as prepared independently from other lots. Commercially purchased standards are traceable to a national standard as documented with the Certificate of Analysis (C of A).

Note 1: The use of standards from a second lot is acceptable when only one manufacturer, not vendor, of the calibration exists.

Note 2: The requirement for a second source standard for the initial

calibration is waived if a second source standard is used for the calibration verification.

Note 3: The date of preparation of each second source standard is considered when evaluating its suitability for use. This consideration includes an assessment of the stability of the standard solution, as well as its natural degradation rate.

Note 4: The second source standard is prepared at a concentration at or near the middle of the calibration range. Since most methods do not require the analysis of this standard and no method criteria exists, the criteria of acceptance is generally established using the calibration verification acceptance criteria.

e) The initial calibration criteria established and documented in the method SOPs are derived directly from the referenced method, if available. If the referenced method does not provide calibration criteria, then the in-house criteria detailed in the method SOP is established to be appropriate to the calibration technique employed.

f) The limit of quantitation, reporting limit, can not be established lower than the lowest initial calibration standard.

Note 1: Data reported below the limit of quantitation, or the lowest initial calibration point is considered to have an increased uncertainty and is only reported using data flags or footnotes.

Note 2: It is Alpha's policy to not report data below our lowest established calibration point and to only report data below the established limit of quantitation, if required by the client.

g) The highest calibration standard is the highest concentration for which quantitative data is reported.

Note 1: Data reported above the highest calibration point is considered to have an increased quantitative uncertainty and is only reported using data flags or footnotes.

Note 2: It is Alpha's policy to not report data above our highest calibration point and to use dilutions as necessary to report all data below the highest concentration point established in the calibration curve.

h) Analyte concentrations reported outside the established working calibration range, are reported as having less certainty and are reported using data flags

or footnotes. The lowest calibration standard is above the established limit of detection.

Noted NELAP Exceptions:

If a method or instrument technology (such as ICP/MS) employs a single point calibration strategy, then the following are required:

- 1) Prior to the analysis of samples a zero point (i.e., calibration blank) and a single point calibration must be analyzed and the linear calibration range of the instrument must be established by analyzing a series of standards, one which is at the lowest quantitation limit. Sample results reported within the established linear calibration range do not require data flags.
- 2) Zero points, (calibration blanks) and single point calibration standards must be analyzed with each analytical batch.
- 3) A standard corresponding to the limit of quantitation must be analyzed with each analytical batch and must meet established acceptance criteria.
- 4) The linearity is verified at a frequency established by the method and/or the manufacturer.

Note: See the ICP/MS method SOP for details.

- i) If the initial instrument calibration results are outside of the established acceptance criteria, corrective actions are performed and all associated samples reanalyzed. If reanalysis of the samples is not possible, data associated with an unacceptable initial calibration is reported with the appropriate data flags or footnotes.
- j) If the referenced analytical method does not specify the number of initial calibration points, then the minimum number is two (one of which must be at the limit of quantitation), not including blanks, or zero standards, with the exception of methods which only require a single point calibration. The minimum number of calibration points are established and referenced in each of the analytical SOPs.

Note: It is Alpha's policy to establish the minimum number of calibration points as 3 for inorganic analysis and 5 for organic analysis with the noted exceptions for some multi-component analytes, such as PCBs, toxaphene and technical chlordane.

### 9.1.2 Initial Calibration Verification

See section 9.1.1 d above for details

### 9.1.3 Calibration Verification

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration is verified prior to sample analyses by an acceptable continuing calibration verification standard with each analytical batch. As long as the continuing calibration verification is acceptable, a new initial instrument calibration is not necessary. The following items are essential elements of continuing instrument calibration verification:

- a) The details of the continuing instrument calibration procedure, calculations and associated statistics are included or referenced in the analytical method SOP.
- b) Calibration is verified for each discrete (single response) analyte or element except for mult-component analytes such as PCBs, toxaphe, technical chlordane or total petroleum hydrocarbons where a single response standard mix is used.
- c) Instrument calibration verification is performed as follows:
  - 1) at the beginning and end of each analytical batch (except, if an internal standard is used, only one verification standards needs to be performed at the beginning of the analytical batch);  
  
Noted exception is ICP/MS which uses an internal standard calibration procedure, the initial calibration is verified by the analysis of the CV at a frequency of once every 10 samples and the samples must be bracketed.
  - 2) whenever it is expected that the analytical system may be out of calibration or might not meet the verification acceptance criteria;
  - 3) if the time period for calibration or the most previous calibration verification has expired; or
  - 4) if the method specifies a different calibration frequency.

When the method specifies that the CV be analyzed at a specific sample interval (for example, every 10 or 20 samples), the count of these samples may include field samples only. However, QC samples must be analyzed with their associated batches.



Note: The grouping of QC samples from a variety of batches is unacceptable.

- d) Raw data records are retained to permit the reconstruction of the continuing instrument calibration, e.g., analyst's name, test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor to convert instrument response into concentrations. Continuing calibration records and data are maintained in a manner to explicitly connect the continuing calibration to the initial instrument calibration.
- e) The criteria for the acceptance of a continuing instrument calibration verification is established and detailed in the individual method SOPs.

#### Calibration Acceptance Criteria

If the continuing calibration verification results obtained are outside of the established acceptance criteria, corrective action is generally required. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either, after corrective action, the next two consecutive calibrations verification standards has to be acceptable or a new initial instrument calibration should be performed.

If samples are analyzed on an instrument that has failed the continuing instrument calibration, then the results are flagged for the failing analytes. Data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

- 1) when the acceptance criteria for the continuing instrument calibration verification is exceeded high, (i.e., high bias), and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification should be reanalyzed after corrective action and an acceptable calibration verification or a new calibration curve has been established, evaluated and accepted.
- 2) when the acceptance criteria for the continuing instrument calibration verification is exceeded low, (i.e. low bias), those samples may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable calibration verification should be reanalyzed after corrective action and an acceptable calibration verification or a new calibration curve has been established, evaluated and accepted.

## 9.2 Reference Standards and Reagents

### 9.2.1 Policy

It is Alpha's policy to purchase calibration and/or verification and validation standards, to include S class weights, and thermometers, that are traceable to national standards and measurements when possible. These are typically documented with a Certificate of Analysis.

It is Alpha's policy to participate in a inter laboratory comparison program such as a proficiency testing program in an effort to provide additional evidence of correlation of sample results.

### 9.2.2 Reference Material

During standard calibration and sample analysis, solutions containing known target compounds at known concentrations are prepared. These standards are used to calibrate instruments and quantitate analytical data.

Alpha Analytical uses these three types of reference material:

- commercial standards;
- commercially prepared custom standards; and
- custom-made standards prepared from reagent grade or neat chemical.

#### 9.2.2.1 Commercial Standards

Commercial standards are the primary source of reference material Alpha Analytical uses in the determination of analytical data. Alpha compares commercial standards with a secondary source (e.g., ICV standards) of commercial standards to verify standard concentrations and analytes.

Individual and standard mix solutions procured from commercial vendors are purchased for specific methods of analysis and are factory prepared for ease of secondary dilutions. Alpha Analytical, except for unusual standards, purchases reference and neat standards from the sources listed in table 9-1.

#### 9.2.2.2 Neat Standards

Custom-made standards is an additional source of reference standard material used in the determination of analytical data. Chemicals used in the preparation of custom made standards are typically ACS reagent grade of 99+% purity.

**SOURCES OF COMMERCIAL STANDARDS**  
**Table 9-1**

Absolute Standards, Inc. P.O. Box 5585 Hamden, CT 06518 800-368-1131	Inorganic Ventures, Inc. 195 Lehigh Ave, Suite 4 Lakewood, NJ 08701 800-569-6799	ChemService, Inc. 660 Tower Lane, P.O. Box 599 West Chester, PA 19381-0599 800-452-9994
Supelco, Inc. Supelco Park Bellefonte, PA 16823-0048 800-247-6628	Aldrich Chemical Co. Inc. 940 West Saint Paul Ave. Milwaukee, WI 53233 800-558-9160	Ultra Scientific 250 Smith Street North Kingston, RI 02852 800-338-1745
NSI Solutions Inc. 7517 Precision Drive, Suite 101 Raleigh, NC 27617 800-234-7837	Accu Standards, Inc 125 Market Street New Haven, CT 06513 800-442-5290	Environmental Research Associates 5540 Marshall Street Arvada, CO 80002 800-372-0122

## 9.2.3 Transport and Storage

### 9.2.3.1 Reference Material

The Standard Preparation SOP, found in Appendix E, describes in detail our procedure for safe handling, transport, storage and use of reference material (i.e., instrument calibration standards) in order to prevent contamination or deterioration and in order to maintain and protect its integrity.

Semi-volatile reference materials are generally stored in a refrigerator at 4°C and volatile reference materials are generally stored in a freezer at -20°C. Most inorganic reference material is either stored at 4°C or at room temperature.

### 9.2.3.2 Reagents

Laboratory reagents and chemicals must be stored according to method guidelines and the manufacturer's instructions. All solvents used for VOC analyses (i.e. methanol) must be isolated and stored separately from solvents which may be target analytes.

Reagents are stored and segregated according to compatibility groups (e.g. flammable solvents and non flammable solvents). Storage of all chemicals and solvents follow all OSHA requirements.

## 9.2.4 Documentation and Labeling of Standards and Reference Material

### a) Reference Material Logbook

To insure the proper quantification of sample analytes, all standards used by Alpha Analytical, Inc. are the finest quality available.

To insure the integrity of sample quantitation, records are retained for all standards, reagents, and reference material and documented as follows:

- i. the manufacturer/vendor;

- ii. the manufacturer's Certificate of Analysis or purity;
- iii. lot number;
- iv. the date of receipt;
- v. expiration date;

Note: standards may not be used past their expiration date unless its reliability is verified.

If standards are prepared, the following additional information is required:

- vi. preparation date,
- vii. amounts and concentration of all source reagents and compounds used; and
- viii. signature and initials of preparer.

Upon receipt of the standards and certificates, the technician or chemist responsible for the preparation and traceability of that standard, dates and initials all certificates. The certificates are then placed in a 3-ring binder for historical reference.

- b) Original containers (such as those provided by the manufacturer or vendor) must be labeled with an expiration date.
- c) Standard Preparation Logbook

Standard receipt, preparation and dilution information is stored with the standard preparation logbook. Measurements and calculations made during the preparation of the standards and dilutions are also recorded and stored in the Standard Preparation logbooks.

Alpha maintains a general policy to label and properly store all bottles, flasks, beakers or vials that contain samples, sample extracts or standard solutions.

- d) Standard and standard dilutions are prepared according to the procedure found in Appendix E. Once commercial standards are logged in they are repackaged into screw capped vials and labeled. Information such as lot number, date prepared, preparer's name, solvent, standard ID, etc. is described on the label; as well as, on subsequent standard dilutions when prepared. The information provided on these labels can be used as a reference for future traceability.

#### 9.2.5 Documentation and Labeling of Reagents

- a) Reference Material Logbook

A record of all reagents and chemicals are maintained Reference Material Logbook. At a minimum these records shall document

To insure the proper preparation of samples, all reagents used by Alpha Analytical, Inc. are the finest quality available.

To insure the integrity of sample quantitation, records are retained for all reagents, and reference material and documented as follows:

- i. Storage conditions and location for reagents (these parameters are outlined in laboratory SOPs and the Chemical Hygiene Plan ),
- ii. the manufacturer/vendor;
- iii. the manufacturer's Certificate of Analysis or purity;
- iv. lot number;
- v. the date of receipt;
- vi. expiration date;

Note: reagents may not be used past their expiration date unless its reliability is verified.

Upon receipt of the reagents and certificates, the technician or chemist responsible for the receipt and storage of that material, dates and initials all certificates. The certificates are then placed in a 3-ring binder for historical reference.

- b) Original containers (such as those provided by the manufacturer or vendor) must be labeled with an expiration date.
- c) If reference material are to be diluted, or prepared in any way, then they are labeled and documented as detailed above and in the Standard Preparation Logbook.

## **9.3 CONTROL OF PROCURED ITEMS**

### **9.3.1 Supplies and Materials**

Alpha Analytical has contracted with major environmental suppliers for the procurement of critical and non-critical items.

Items purchased on a regular basis that have an impact on the final data results are sequestered by lot to maintain quality uniformity and controlled conformity. Items such as containers, solvents, standards, etc. have been recognized by the industry to be critical to environmental laboratories; therefore, suppliers have specialized in producing these items to meet and exceed method specification.

These items are controlled by our procurement process by repeatedly buying the same product. If an item does not meet the required specification, our procurement process will have the documentation to pull these items from our laboratory and return them to the supplier.

All critical items are constantly checked by their daily use, i.e., methanol is checked by analyzing daily method blank and standards are checked against the response factors of independent standards. These activities occur daily and are documented in various areas of the laboratory such as the analysts' logbooks and the instrument document control books.

#### **9.4 Summary of Method Calibration Procedures**

- 9.4.1 The following tables are designed to list calibration procedures for each analytical method. These tables also include minimum frequencies, acceptance criteria, and possible corrective actions for method specified calibration. These tables are an outline of the method specified procedures and, when in doubt, or a discrepancy exists, the method defined procedure takes precedence.

**SDWA  
ORGANIC METHODS**

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 524.2  
TABLE 9-2**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
524.2 VOCs	Initial Demonstration of Capabilities (IDC) Study	Once per analyst, annually	Analyze 4 replicates of LFB at a concentration range of 2.0 -5.0 ug/L from a source independent of the IC. Recoveries must be $\pm 20\%$ of the expected value and RSD must be $< 20\%$ .	If any analyte fails, correct the source of the problem and repeat the test for that compound.
	Tuning	25 ng BFB at the beginning of each 12 hour shift.	Method Table-3	Take remedial action and repeat the test.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, lowest 2-10 xMDL. A minimum of 3 CALs for a calibration range of a factor of 20, 4 CALs for a factor of 50 and 5 CALs for a factor of 100.	If RDS is $\leq 20\%$ linearity is assumed and average RF used. Alternatively, use a calibration curve.	Correct the problem then repeat the initial calibration.
	Quality Control Sample (QCS)	QCS should be analyzed quarterly from a source independent from the IC	Recoveries should be $\pm 30\%$ of the expected value.	Rerun. If repeat failure occurs locate and correct the source of the problem.
	Continuing Calibration Check (CCC)	Mid-level calibration standard analyzed at the beginning of each 12 hour shift.	RF must be within $\pm 30\%$ of the IC. Absolute areas of quant ions for IS and surrogate must not decrease more than 30% from previous CCC or by more than 50% for IC.	Adjust sensitivity, repeat test using fresh calibration, or generate a new IC.
	Surrogates	Every sample, spike and standard.	Recoveries should be $\pm 30\%$ of the expected value.	Locate possible errors, and reanalyze. If reanalysis fails, report data as suspect.
	Internal Standard (IS)	Every sample, spike and standard.	Absolute areas of quant ions for IS must not decrease more than 30% from previous CCC or by more than 50% for IC.	Locate possible errors, and reanalyze. If reanalysis fails, report data as suspect.
	Standard Expiration	Liquid standards stable for at least 4 weeks. Gas standards stable up to 1 week.	CCC acceptance criteria	Remake fresh standards.



**SDWA and CWA  
INORGANIC METHODS**

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 120.1/SM2510B  
TABLE 9-3**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
120.1 SM2510B  Specific Conductance	Initial Calibration (IC)	Initial calibration prior to analysis. Manufacturer suggests, 2 EC points, 1 in air.	Prepare calibration curve to bracket sample concentrations.	Repeat the test.
	Initial Calibration Verification (ICV)	After each new initial calibration. Per our SOP we will add: and daily before sample analysis.	Not specified. However, we will use method 314.0 criteria. Daily ICV at 1410 $\mu$ S/cm with an acceptance criteria of 1380 to 1440 $\mu$ S/cm.	Repeat the test. If failure occurs recalibrate with a new standard. Samples measured after criteria was exceeded must be reanalyzed.
	Calibration Verification (CV)	Not specified. Will verify daily and bracket sample analysis.	Not specified. However, we will use method 314.0 criteria. Daily CV at 1410 $\mu$ S/cm with an acceptance criteria of 1380 to 1440 $\mu$ S/cm.	Repeat the test. If failure occurs recalibrate with a new standard. Samples measured after criteria was exceeded must be reanalyzed.
	Standard Expiration	Not specified. Minimum of one year or vendor expiration date	Not specified.	If degradation or contamination is suspected, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 150.1/SM4500H B  
TABLE 9-4**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
150.1 SM4500H B  pH	Initial Calibration (IC)	Initial calibration prior to analysis, minimum of 2 pH buffers.	Prepare calibration curve to bracket sample concentration. Manufacturer suggests a curve slope of 92 - 102%	Repeat the test. If failure occurs recalibrate.
	IC for Corrosivity Characterization	Include pH buffer at 1.68 for acidic wastes and pH buffer at 12.45 for caustic wastes	Not specified. The same as above.	Repeat the test. If failure occurs recalibrate.
	Initial Calibration Verification (ICV)	Not specified. Lab suggests after each new initial calibration.	Not specified. However, PE criteria is $\pm 10\%$ . Therefore response should be $\pm 10\%$ of the expected value.	Repeat the test. If failure occurs recalibrate.
	Calibration Verification (CV)	Not specified. Manufacturer suggests to verify the calibration every 2 hours.	Not specified. However, PE criteria is $\pm 10\%$ . Therefore response should be $\pm 10\%$ of the expected value.	Repeat the test. If failure occurs recalibrate. Samples measured after criteria was exceeded must be reanalyzed.
	pH Buffer Expiration	Not specified. Minimum of six months or vendor expiration date.	Not specified.	If degradation or contamination is suspected, replace the standards.
	Working Standard Expiration	Not specified. Daily	Not specified	If degradation or contamination is suspected, replace the standards.

**SUMMARY OF CALIBRATION PROCEDURES  
 FOR METHOD 160.1/160.2/160.3 and  
 SM2540C/SM2540D/SM2540B**

**TABLE 9-5**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
TDS-160.1/2540C	Initial Calibration (IC)	NA		
TSS-160.2/2540D	Calibration Verification (CV)	NA		
TS-160.3/2540B	Working standard Expiration	NA		

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 180.1/SM2130B  
TABLE 9-6**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
180.1 SM2130B  Turbidity	Initial Calibration (IC)	Initial calibration prior to analysis.	Prepare calibration curve to bracket sample concentrations. ( 0 and 40 NTU standards)	Repeat the test. If failure occurs recalibrate.
	Initial Calibration Verification (ICV)	Not required. Lab suggests after each new initial calibration.	Not required. In-house established criteria is $\pm 10\%$ .	Repeat the test. If failure occurs recalibrate.
	Linearity Check	Not required. Lab suggests analyzing 3 standards.	Not required. In-house established criteria is $\pm 10\%$ .	Repeat the test. If failure occurs recalibrate.
	Calibration Verification (CV)	Not required. Lab suggests after each 10 samples and at the end of the sequence.	Not required. In-house established criteria is $\pm 10\%$ .	Repeat the test. If failure occurs recalibrate.
	Stock Standard Expiration	Formazin Stock (1 month) AMCO-AEPA-1 (1 year)	Not specified.	If degradation or contamination is suspected, replace standards.
	Working standard Expiration	Formazin (1 week) AMCO-AEPA-1 (1 year)	Not specified.	If degradation or contamination is suspected, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 200.8  
TABLE 9-7**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
200.8 Metals	Demonstration of Capabilities (DOC)	LCR, QCS, MDL.	See SOP and Method, Section 9.0 for details.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Instrument Detection Limits (IDL)	Instrument detection limit studies are performed initially during instrument start-up.	Calculate the average of the standard deviation from the analysis of a reagent blank analyzed 10 times.	
	Tuning	Daily, pre-calibration, prior to sample analysis.	Analyze tuning solution (1 to 10 ug/L Be, Mg, Co, In, Pb) 5 times. RSD of absolute signals $\leq 5\%$ . Mass calibration $< 0.1$ amu from true value, resolution $< 0.75$ amu at 5% peak height.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Daily. Initial calibration prior to sample analysis. Minimum of calibration blank and 1 level using the average of 3 integrations.	Per instrument manufacturer's specifications. Alpha performs a multipoint calibration defining the low end of the calibration. See SOP for details	Correct the problem then repeat the initial calibration.
	Quality Control Sample (QCS)  Same as:  Initial Calibration Verification (ICV)	Verify each element calibration with an ICV prepared from a source independent from the calibration standards at a concentration equivalent to or near the midpoint of the calibration curves and at a concentration different than that used for instrument calibration.	Percent Recovery (%R) of three replicates = 90 - 110% of expected value. If using as an on-going basis, three replicates are not necessary.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test. If the problem occurs twice then recalibrate, or the ICV must pass on the third and fourth consecutive tests.
	Internal Standards (IS)	Add IS to all standards. Recommended IS: Li, Sc, Y, In, Tb, Ho and Bi.	IS intensity levels must agree to within 60-125% of the level in the CB.	Stop the analysis, correct the problem, re-calibrate, and re-analyze the affected samples.
	Calibration Verification (CV)	Analyze CV after IC, every 10 samples and at the end of the analytical run.	Percent Recovery (%R) = 90 - 110 %.	Same as the ICV
	Calibration Blank (CB)	Analyze CB prior to the CV, every 10 samples and at the end of the analytical run.	No specified criteria.	
	Interference Check Solution (ICS)	Not required.	Not required.	
	Standard Expiration		No criteria specified for stock standards. 2 weeks for calibration standards.	

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 300.0  
TABLE 9-8**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
300.0 Anions	Linear Calibration Range (LCR)	Every 6 months	Blank and 3 standards over the expected linear range. LCR standards must be $\pm 10\%$ of the expected value.	If any of the 3 LCR standards exceed $\pm 10\%$ of the expected value, linearity must be reestablished.
	MDL	Should be conducted every 6 months, new operator, or change in instrument response.	MDL must be less then the minimum reporting limit	If MDL is above RL, reevalutate RL or conduct MDL study a second time.
	Initial Calibration (IC)	Initial calibration prior to analysis, minimum of 3 concentration levels covering the working range of the instrument	Prepare calibration curve to bracket sample concentration. No other method criteria.	If sample analytes exceed cal range, dilute sample or recalibrate at a higher concentration point.
	QCS/ICV	Each time a new IC is prepared and quarterly	Second source standard, $\pm 10\%$ of the expected value.	If the QCS exceeds $\pm 10\%$ of the expected value, reanalyze second time or recalibrate.
	Instrument Performance Check Calibration Verification IPC/CV	Initially, after every 10 samples and at the end of the sample run.	Response should be $\pm 10\%$ of the expected value.	Repeat the test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Retention Time Window	Each initial calibration and calibration verification over the course of the day.	$\pm 3$ times the standard deviation centered around the mean.	If CV's or other QC checks are outside the window recheck and/or recalculate window
	Stock Standard Expiration	Minimum of one month, SOP 1 year or vendor expiration date	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	Weekly except for nitrite,nitrate and o-phos. 48 hours.	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
 FOR METHOD 305.1/SM2310B  
 TABLE 9-9**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
305.1 SM2310B  Acidity	Initial Calibration (IC)	See pH SOP		
	Calibration Verification (CV)	See pH SOP		
	Standardization of titrants	Prior to sample analysis.	NA	
	Standardized Titrant Expiration	1 week for Na <sub>2</sub> CO <sub>3</sub> only. No other expiration date for any other titrant.	NA	



**SUMMARY OF CALIBRATION PROCEDURES  
 FOR METHOD 310.1/SM2320B  
 TABLE 9-10**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
310.1 SM2320B  Alkalinity	Initial Calibration (IC)	See pH SOP		
	Calibration Verification (CV)	See pH SOP		
	Standardization of titrants	Prior to sample analysis.	NA	
	Standardized Titrant Expiration	1 week for Na <sub>2</sub> CO <sub>3</sub> only. No other expiration date for any other titrant.	NA	

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 314.0  
TABLE 9-11**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
314.0 Perchlorate	Initial Demonstration of Accuracy (IDA)	Once per analyst.	Analyze 7 replicate LFBs at 25 ug/L. Average recovery must be $\pm 10\%$ of true value.	If test fails, correct the source of the problem and repeat the test.
	Initial Demonstration of Precision (IDP)	Once per analyst.	Calculate %RSD of IDA replicates, The %RSD must be $< 10\%$ .	If test fails, correct the source of the problem and repeat the test.
	Method Detection Limit (MDL)	Initially and verified periodically.	MDL must be less then the minimum reporting limit	If MDL is above RL, reevaluate RL or conduct MDL study a second time.
	Initial Calibration (IC)	Initial calibration prior to analysis, minimum of 3 points for one order of magnitude and 5 points for 2 orders of magnitude covering the working range of the instrument	If $\%RSD \leq 15\%$ linear through zero. Alternatively, prepare a calibration curve.	Correct the problem, then repeat the initial calibration.
	Quality Control Sample (QCS) (Same as ICV)	Each time a new IC is prepared.	Second source standard, $\pm 10\%$ of the expected value.	If the QCS exceeds $\pm 10\%$ of the expected value, reanalyze second time or recalibrate.
	Instrument Performance Check (IPC)	Prior to sample analysis to verify MCT and instrument performance.	See Method/SOP criteria.	Repeat the test. If failure occurs determine the source of the problem and repeat the test.
	Initial Continuing Calibration Standard (ICCS)	Prior to any sample analysis at the MRL.	Response should be $\pm 25\%$ of the expected value.	Repeat the test. If failure occurs recalibrate.
	Continuing Calibration Check Standard (CCCS)	Analyze mid and high level CCCS/ECCS after every 10 samples and at end of analytical batch.	Response should be $\pm 15\%$ of the expected value.	Repeat the test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Retention Time Window	Each initial calibration and calibration verification over the course of the day.	$\pm 3$ times the standard deviation centered around the mean.	If CCCS or other QC checks are outside the window recheck and/or recalculate window
	Stock Standard Expiration	1 year or vendor expiration date	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	1 year or vendor expiration date	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 330.5/SM4500Cl G  
TABLE 9-12**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
330.5 SM4500Cl G  Free and Total Residual Chlorine	Initial Demonstration of Proficiency	Not required by the method, required by NELAP. Once per analyst.	Prepare and analyze 4 replicates. Neither method 4500-Cl G or 330.5 specifies accuracy or precision. Should pass the in-house criteria to be acceptable.	If the analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Not specified by the method. Prepare prior to sample analysis.	NELAP suggests when this situation occurs, a minimum of a 3 point calibration curve be generated, and the IC should have a correlation coefficient of >0.995 to be acceptable.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	Should pass the in-house criteria, of $\pm 15\%$ to be acceptable.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Not required by the method. However, NELAP requires samples to be bracketed with CVs and suggests after each 10 samples.	Not specified. Response should pass the in-house criteria of $\pm 15\%$ of the expected value to be acceptable.	Repeat the test. If failure occurs re-calibrate. Samples analyzed after criteria was exceeded must be re-analyzed.
	Stock Standard Expiration	No criteria specified. However, a 1 year or manufacturer's expiration date will be used as the default.	Not specified.	If comparison to second source indicates degradation, replace the standard.
	Working Standard Expiration	No criteria specified. However, a 1 day or manufacturer's expiration date will be used as the default.	Not specified.	If comparison to second source indicates degradation, replace the standard.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 350.3/SM4500NH<sub>3</sub>D**

**TABLE 9-13**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
350.3 SM4500NH <sub>3</sub> D  Ammonia   351.4 SM4500N <sub>org</sub> C  TKN	Initial Calibration (IC)	Initial calibration prior to analysis.	Prepare calibration curve to bracket sample concentrations. In-house suggested curve slope of -60 ±10%.	Repeat the test. If failure occurs recalibrate.
	Initial Calibration Verification (ICV)	Not required. NELAP requires after each new initial calibration.	Not required. In-house established criteria is ±20 %.	Repeat the test. If failure occurs recalibrate.
	Calibration Verification (CV)	Not required. Manufacturer suggests to verify the calibration every 2 hours.	Not required. In-house established criteria is ±10 %.	Repeat the test. If failure occurs recalibrate. Samples measured after criteria was exceeded must be reanalyzed.
	Stock Standard Expiration	Not specified. Minimum of 6 months or vendor expiration date	Not specified.	If degradation or contamination is suspected, replace standards.
	Working Standard Expiration	Not specified. Daily	Not specified.	If degradation or contamination is suspected, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 365.2/SM4500P E**

**TABLE 9-14**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
365.2 SM4500P E  Total and Reactive Phosphorus	Initial Demonstration of Proficiency (IDP)	Not required by the method, required by NELAP. Once per analyst.	Prepare and analyze 4 replicates. Both EPA method 365.2 and SM 4500-P E report some accuracy and precision data. IDP data is evaluated against a accuracy window of (80-118%) based on the tightest windows of these two combined methods	If the analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	A minimum of a 6 point linear calibration curve through zero. Prepare prior to sample analysis.	Not specified. NELAP suggests in such situations a correlation coefficient "r" of > 0.995 is required.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	Should pass the in-house CV criteria, of $\pm 15\%$ to be acceptable.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Required by both methods but somewhat ambiguous to frequency. NELAP requires samples to be bracketed with CVs and suggests after each 10 samples.	Should pass the in-house CV criteria, of $\pm 15\%$ to be acceptable.	Repeat the test. If failure occurs re- calibrate. Samples analyzed after criteria was exceeded must be re-analyzed.
	Stock Standard Expiration	Not specified. In-house will use a one year expiration date or manufacturers suggested expiration date.	Not specified.	If comparison to second source indicates degradation, replace the standard.
	Working Standard Expiration	Not specified. In-house will use a 48 hour expiration date, same as method 300.0 for ortho- phosphate.	Not specified.	If comparison to second source indicates degradation, replace the standard.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 376.2/SM4500S D  
TABLE 9-15**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
376.2 SM4500S D  Sulfide	Initial Demonstration of Proficiency (IDP)	Not required by the method, required by NELAP. Once per analyst.	Prepare and analyze 4 replicates. Neither method SM4500S or 376.2 specifies accuracy or precision. Should pass the in-house criteria to be acceptable.	If the analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Method only suggests using a 5 point calibration procedure for color comparison. No other criteria specified by the method. Prepare prior to sample analysis.	NELAP suggests when this situation occurs, a minimum of a 3 point calibration curve be generated, and the IC should have a correlation coefficient of >0.995 to be acceptable.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	Should pass the in-house criteria, of $\pm 15\%$ to be acceptable.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Not required by the method. However, NELAP requires samples to be bracketed with CVs and suggests after each 10 samples.	Not specified. Response should pass the in-house criteria of $\pm 15\%$ of the expected value to be acceptable.	Repeat the test. If failure occurs re- calibrate. Samples analyzed after criteria was exceeded must be re-analyzed.
	Stock Standard Expiration	No criteria specified. However, a 1 month or manufacturer's expiration date will be used as the default.	Not specified.	If comparison to second source indicates degradation, replace the standard.
	Working Standard Expiration	No criteria specified. Working standards should be prepared daily.	Not specified.	If comparison to second source indicates degradation, replace the standard.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 410.4/SM5220D  
TABLE 9-16**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
410.4 SM5220D  COD	Initial Demonstration of Proficiency (IDP)	Not required by the method, required by NELAP. Once per analyst.	Prepare and analyze 4 replicates. EPA method 410.4 does not specify any accuracy or precision criteria. However SM5220D uses a window of 72-128% to be acceptable	If the analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	A minimum of a 5 point calibration curve. Prepare prior to sample analysis.	Not specified. NELAP suggests in such situations a correlation coefficient "r" of > 0.995.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	Should pass the method LCS criteria, of 72-128% to be acceptable.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Not specified. However, NELAP requires samples to be bracketed with CVs and suggests after each 10 samples.	Should pass the method CV criteria, of $\pm 5\%$ to be acceptable.	Repeat the test. If failure occurs re- calibrate. Samples analyzed after criteria was exceeded must be re-analyzed.
	Stock Standard Expiration	3 months	Not specified.	If comparison to second source indicates degradation, replace the standard.
	Working Standard Expiration	3 months	Not specified.	If comparison to second source indicates degradation, replace the standard.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 415.1/SM5310C**

**TABLE 9-17**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
415.1 SM5310C  TOC	Initial Demonstration of Proficiency (IDP)	Not required. Once per analyst.	Not specified. Analyze 4 replicates. Evaluate accuracy and precision against method defined criteria.	If analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Not specified. NELAP requires a minimum of 3 calibration points with the lowest at or below the reporting level and the other two covering the working range of the calibration.	Not Specified.  Instrument manufacturer only allows the use of a linear calibration. NELAP requires a correlation coefficient of > 0.995 when not specified.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established	In-house criteria, should pass the CV criteria to be acceptable.	Repeat the test. If repeat failure occurs correct the problem and next two ICVs must pass or recalibrate.
	Calibration Verification (CV)	Method 9060A specifies a CV evry 15 samples. However, in-house batch size is 20 samples; therefore, minimum of 2, before sample analysis and at the end of the sequence or once every 20 samples and preferably once every 10 samples.	Not Specified. Response should be with in $\pm 20\%$ of the expected value.	Repeat the test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Stock Standard Expiration	Not specified. One year, store in the dark at 4°C.	Not specified	If comparison to secound source indicates degradation, replace the standards.
	Working Standard Expiration	Not specified. One year, store in the dark at 4°C.	Not specified	If comparison to secound source indicates degradation, replace the standards.



**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD SM3500Cr D/SW7196A**

**TABLE 9-18**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SM3500Cr D SW7196A  Cr (VI)	Initial Demonstration of Proficiency (IDP)	Not required by the method, required by NELAP. Once per analyst.	Digest and analyze 4 replicates. Neither method SM3500Cr or 7196A specifies accuracy or precision. In-house $\pm 15\%$ criteria same as specified for MS. Method 3060A soil does specify a soil LCS criteria of $\pm 20\%$ and will be used to assess the soil IDC>	If the analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Not specified by the method. Prepare with each new batch of samples prior to sample analysis.	Not specified. NELAP suggests a minimum of a 3 point calibration. NELAP suggests when this situation occurs, the IC should have a correlation coefficient of >0.995 to be acceptable.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established	In-house criteria, should pass the CV criteria of $\pm 10\%$ to be acceptable.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Method 7196A requires a CV ever 15 <sup>th</sup> sample. In-house criteria is as follows: Verify the IC at the beginning, after every 10 samples and at the end of the analytical sequence.	Not specified. Response should be $\pm 10\%$ of the expected value.	Repeat the test. If failure occurs re- calibrate. Samples analyzed after criteria was exceeded must be re-analyzed.
	Stock Standard Expiration	No criteria specified. However, a 1 year or manufacturer's expiration date will be used as the default.	Not specified.	If comparison to second source indicates degradation, replace the standard.
	Working Standard Expiration	No criteria specified. In-house criteria: prepare a new set of calibration standards with each set of samples to be analyzed.	Not specified.	If comparison to second source indicates degradation, replace the standard.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD SM3500Fe D**

**TABLE 9-19**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SM 3500Fe D  Ferrous Iron  Total and Dissolved Iron	Initial Demonstration of Proficiency (IDP)	Not required by the method, required by NELAP. Once per analyst.	Prepare and analyze 4 replicates. Method SM3500Fe D suggests only general accuracy or precision. In-house $\pm 15\%$ criteria same as used for the LCS.	If the analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Not specified by the method. Prepare with each new batch of samples prior to sample analysis.	Not specified. NELAP suggests a minimum of a 3 point calibration. NELAP suggests when this situation occurs, the IC should have a correlation coefficient of >0.995 to be acceptable.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established	In-house criteria, should pass the CV criteria of $\pm 10\%$ to be acceptable.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Not required by the method. In-house criteria is as follows: Verify the IC at the beginning, after every 10 samples and at the end of the analytical sequence.	Not specified. Response should be $\pm 10\%$ of the expected value.	Repeat the test. If failure occurs re- calibrate. Samples analyzed after criteria was exceeded must be re-analyzed.
	Stock Standard Expiration	One year or manufacturer expiration date prior to color development.	Not specified.	If comparison to second source indicates degradation, replace the standard.
	Working Standard Expiration	Non-colored working standards are prepared daily. Method specifies a 6 month criteria for color developed standards provided they are sealed and protected from light. (In-house 1 month for ferrous iron standards).	Not specified.	If comparison to second source indicates degradation, replace the standard.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 1664A/SW9070A  
TABLE 9-20**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
1664A SW9070A  HEM and SGT-HEM (Oil and Grease)	Initial Proficiency and Recovery (IPR)	An initial one-time demonstration per analyst or any significant change to method.	Extract and analyze 4 replicates of the PAR/LCS spike mix at a concentration of 40 mg/L. (HEM Accuracy must meet accuracy window of 83-101% and a precision of <11%.) (SGT-HEM Accuracy must meet accuracy window of 83-116% and a precision of <28%.)	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Balance Calibration Verification (CV)	Before and after the extraction batch.	2mg S class weight must be within $\pm 10\%$ ( $\pm 0.2\text{mg}$ )  1000 mg S class weight must be within $\pm 0.1\%$ ( $\pm 1\text{mg}$ ).  5000 mg S class weight must be within $\pm 0.1\%$ ( $\pm 5\text{mg}$ ).	Re-calibrate the balance and repeat the test.

**CWA**  
**ORGANIC METHODS**

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 608/SM6630C**

**TABLE 9-21**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
608 SM6630C  Pesticides PCBs	Initial Demonstration of Proficiency (IDP)	An initial one-time demonstration per analyst.	Extract and analyze 4 replicates of the QC check standard. Compare accuracy and precision results to method Table-3.	If data are not comparable, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 3 levels, lowest near but above MDL	If RSD is $\leq 10\%$ linearity is assumed and average CF is used. Alternatively, prepare a calibration curve.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires response to be within $\pm 20\%$ of the expected value.	Repeat test. If repeat failure occurs correct the problem and next two ICVs must pass or recalibrate.
	Calibration Verification (CV)	One or more calibration standards analyzed daily.	Response should be $\pm 15\%$ of the expected value.	Repeat test. If failure occurs recalibrate or flag data.
	Retention Time Window	Each initial calibration and/or calibration verification.	Suggest, $\pm 3$ times the standard deviation of each analyte's retention time.	Correct the problem then reanalyze all samples since the last retention time check.
	Endrin and DDT Breakdown Check	Not required		
	Stock Standard Expiration	One year, store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	6 months, store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 624  
TABLE 9-22**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
624 VOCs	Initial Demonstration of Capabilities (IDC)	An initial one-time demonstration per analyst.	Analyze 4 replicates of the QC check standard. Compare accuracy and precision results to method Table-5 or 6.	If data are not comparable, correct the source of the problem and repeat the test.
	BFB Tune	50 ng BFB, analyzed at the beginning of each day.	BFB must meet method Table-2 criteria.	Retune the mass spectrometer and repeat the test until all criteria are achieved.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 3 levels, lowest near but above MDL	If RSD is $\leq 35\%$ linearity is assumed and average RF is used. Alternatively, prepare a calibration curve.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires response to be within $\pm 25\%$ of the expected value.	Repeat test. If repeat failure occurs correct the problem and next two ICVs must pass or recalibrate.
	Calibration Verification (CV)	Daily.	Compare % recovery to "Q" found in method Table-5 criteria.	Repeat test. If failure occurs recalibrate or flag data.
	Retention Time Window	Each initial calibration and/or calibration verification.	Qualitative compound identification is confirmed if RT is within $\pm 30$ s of the RT of the mid-point standard of the IC.	Correct the problem then reanalyze all samples since the last retention time check.
	Stock Standard Expiration	Liquid standards stable for at least 1 month. Gas standards stable up to one week. Store with minimal head space in the dark at $-10^{\circ}$ to $-20^{\circ}\text{C}$ .	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	Aqueous standards can be stored up to 24 h, if held in sealed vials with zero headspace.	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 625  
TABLE 9-23**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
625 SVOCs	Initial Demonstration of Capabilities (IDC)	An initial one-time demonstration per analyst.	Extract and analyze 4 replicates of the QC check standard. Compare accuracy and precision results to method Table-6 or 7	If data are not comparable, correct the source of the problem and repeat the test.
	DFTPP Tune	50 ng DFTPP, analyzed at the beginning of each day.	DFTPP must meet method Table-9 criteria.	Re-tune the mass spectrometer and repeat the test until all criteria are achieved.
	Column Performance Check (CPC)	At the beginning of each day.	Confirm benzidine peak tailing factor is less than 3.0 for B/N fraction. Confirm pentachlorophenol peak tailing factor is less than 5.0 for the acid fraction	Correct the problem, then repeat the column performance check.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 3 levels, lowest near but above MDL	If RSD is $\leq 35\%$ linearity is assumed and average RF is used. Alternatively, prepare a calibration curve.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires response to be within $\pm 25\%$ of the expected value.	Repeat test. If repeat failure occurs correct the problem and next two ICVs must pass or recalibrate.
	Calibration Verification (CV)	One or more calibration standards analyzed daily.	Response should be $\pm 20\%$ of the expected value.	Repeat test. If failure occurs recalibrate or flag data.
	Retention Time Window	Each initial calibration and/or calibration verification.	Qualitative compound identification is confirmed if RT is within $\pm 30$ s of the RT of the mid-point standard of the IC.	Correct the problem then reanalyze all samples since the last retention time check.
	Stock Standard Expiration	6 months if tests indicate a problem, longer if not. Store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	6 months if tests indicate a problem, longer if not. Store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 625 (PNAs by SIMs)**

**TABLE 9-24**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
625 (PNA by SIMs)	Initial Demonstration of Capabilities (IDC)	An initial one-time demonstration per analyst.	Extract and analyze 4 replicates of the QC check standard. Compare accuracy (X) and precision (s) results to method Table-6 or 7	If data are not comparable, correct the source of the problem and repeat the test.
	DFTPP Tune	50 ng DFTPP, analyzed at the beginning of each day.	DFTPP mut meet method Table-9 criteria.	Re-tune the mass spectrometer and repeat the test until all criteria are achieved.
	Column Performance Check (CPC)	At the beginning of each day.	Confirm 3,3'- dichlorobenzidine peak tailing factor is less than 3.0.	Correct the problem, then repeat the column performance check.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 3 levels, lowest near but above MDL	If RSD is $\leq$ 35% linearity is assumed and average RF is used. Alternatively, prepare a calibration curve.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires response to be within $\pm 25\%$ of the expected value.	Repeat test. If repeat failure occurs correct the problem and next two ICVs must pass or re- calibrate.
	Calibration Verification (CV)	One or more calibration standards analyzed daily.	Response should be $\pm 20\%$ of the expected value.	Repeat test. If failure occurs re-calibrate or flag data.
	Retention Time Window	Each initial calibration and/or calibration verification.	Qualitative compound identification is confirmed if RT is within $\pm 30$ s of the RT of the mid-point standard of the IC.	Correct the problem then re-analyze all samples since the last retention time check.
	Stock Standard Expiration	One year, store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	6 months, store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.



Alpha Analytical, Inc  
Section No.: 9.0  
Revision No.: 15.0  
Date: January, 2007  
Page 37 of 58

**RCRA**  
**ORGANIC METHODS**

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 8015B-DRO**

**TABLE 9-25**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
8015B-DRO  TPH-E	Initial Demonstration of Proficiency (IDP)	An initial one-time demonstration per analyst	Extract and analyze 4 replicates. Method 8015B does not specify accuracy and precision. Use laboratory defined criteria.	If the analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.	If RSD is $\leq 20\%$ linearity through the origin is assumed and average CF is used. If the RSD is $>20\%$ and correlation coefficient is $> 0.99$ then linear calibration not forced through zero is acceptable. Alternatively, prepare a calibration curve with a coefficient of determination $>0.99$ . Prepare calibration with Diesel standards or standards appropriate to the sampling site	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires response to be within $\pm 20\%$ of the expected value.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or recalibrate.
	Calibration Verification (CV)	Minimum of 2, before sample analysis and at the end of the sequence or every 12 hours. Calibration standard must also be run after every 20 samples (every 10 is recommended).	Response should be within $\pm 15\%$ of the expected value.	Repeat test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Retention Time Window	Each initial calibration and updated with the CV	Method 8015B suggest C10 to C28.	Correct the problem then reanalyze all samples since the last retention time check.
	Stock Standard Expiration	One year, store in the dark at $4^{\circ}\text{C}$ .	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	One year, store in the dark at $4^{\circ}\text{C}$ .	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD NWTPH-dx  
TABLE 9-26**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
NWTPH-dx  TPH-E	Initial Demonstration of Proficiency (IDP)	Not required.	NA Note: will use 8015B-DRO criteria.	NA
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.	Calibration curve must have a correlation coefficient >0.990 and none of the standards may vary from their true known value by more than $\pm 15\%$ .  (Prepare calibration with Diesel standard or standard appropriate to the sampling site.)	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires response to be within $\pm 20\%$ of the expected value.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or recalibrate.
	Calibration Verification (CV)	Minimum of 2, before sample analysis and at the end of the sequence. An increase in the minimum frequency is recommended.	Response should be within $\pm 15\%$ of the expected value.	Repeat test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Retention Time Window	Each initial calibration and updated with the CV	Use the same retention time window for quantitation of the sample as used for the IC.	Correct the problem then reanalyze all samples since the last retention time check.
	Stock Standard Expiration	One year, store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	One year, store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 8015AZ  
TABLE 9-27**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
8015AZ  TPH-E	Initial Demonstration of Proficiency (IDP)	Reporting Limit Verification (RLV) study performed initially, then annually.	7 replicates spiked at the reporting limit. Each replicate must have a recovery of $\pm 30\%$ of the true value. No statistical analysis is required, this study is to prove R.L. are achievable.	If the replicates fail, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.	If RSD is $\leq 20\%$ linearity through the origin is assumed and average CF is used. Alternatively, prepare a calibration curve with a coefficient of determination $> 0.995$ .  (Prepare calibration curve for diesel and 10w30 oil) Note: gasoline may also be used to quantitate C6-C10 range hydrocarbons.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Immediately after the IC has been established.	Response should be within $\pm 20\%$ of the expected value with a second source standard.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or recalibrate.
	Calibration Verification (CV)	Verify the IC at the beginning, after every 10 samples and at the end of the analytical sequence.	Response should be within $\pm 30\%$ of the expected value.	Repeat test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Retention Time Window	Each initial calibration and updated with the CV or as frequently as needed.	Not specified. Method 8015AZ suggests C10, C22 and C32 RT markers.	Correct the problem then reanalyze all samples since the last retention time check.
	Stock Standard Expiration	In-house criteria: one year, store in the dark at $4^{\circ}\text{C}$ .	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	In-house criteria: one year, store in the dark at $4^{\circ}\text{C}$ .	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 8015B-GRO**

**TABLE 9-28**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
8015B-GRO  TPH-P	Initial Demonstration of Proficiency (IDP)	An initial one time demonstration per analyst	Extract and analyze 4 replicates. Method 8015B does not specify accuracy and precision. Use laboratory defined criteria.	If the analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.	If RSD is $\leq 20\%$ linearity through the origin is assumed and average RF is used. If the RSD is $> 20\%$ and correlation coefficient is $> 0.99$ then linear calibration not forced through zero is acceptable. Alternatively, prepare a calibration curve with a coefficient of determination $> 0.99$ . Prepare calibration with gasoline standard or standard appropriate to the sampling site.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	In-house criteria, should generally pass the CV criteria to be acceptable.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Every 12 hours.	Response should be within $\pm 15\%$ of the expected value.	Repeat test. If failure occurs re-calibrate. Samples injected after criteria was exceeded must be re-analyzed.
	Retention Time Window	Each initial calibration and updated periodically.	Method 8015B-GRO suggests C6 and C10. Note will use C4 - C13.	Correct the problem then reanalyze all samples since the last retention time check.
	Stock Standard Expiration	Six months, store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	Not specified. Will used 8260 criteria. 24 hours for aqueous working standards.	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD NWTPH-gx  
TABLE 9-29**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
NWTPH-gx  TPH-P	Initial Demonstration of Proficiency (IDP)	Not required.	NA Note: will use 8015B-GRO criteria	NA
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.	Calibration curve must have a correlation coefficient is > 0.990 and none of the standards may vary from their true known value by more than $\pm 15\%$ .	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	In-house criteria, should pass the CV criteria to be acceptable.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Minimum of 2, before sample analysis and at the end of the sequence. An increase in the minimum frequency is recommended.	Response should be within $\pm 20\%$ of the expected value. Verification is accomplished by the measurement of a mid-range fuel standard.	Repeat test. If failure occurs re-calibrate. Samples injected after criteria was exceeded must be re-analyzed.
	Retention Time Window	Each initial calibration and updated as needed	Use the same retention time window for quantitation of the sample as used for the IC. At a minimum the gasoline integration must include toluene through naphthalene.	Correct the problem then reanalyze all samples since the last retention time check.
	Stock Standard Expiration	One year, store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	One year, store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 8015AZ**

**TABLE 9-30**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
8015AZ  TPH-P	Initial Demonstration of Proficiency (IDP)	Reporting Limit Verification (RLV) study performed initially, then annually.	7 replicates spiked at the reporting limit. Each replicate must have a recovery of $\pm 30\%$ of the true value. No statistical analysis is required, this study is to prove R.L. are achievable.	If the replicate fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.	If RSD is $\leq 20\%$ linearity through the origin is assumed and average CF is used. If the RSD is $> 20\%$ and correlation coefficient is $> 0.995$ then linear calibration not forced through zero is acceptable. Alternatively, prepare a calibration curve with a coefficient of determination $> 0.995$ .	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Immediately after the IC has been established.	Response should be within $\pm 20\%$ of the expected value with a second source standard.	Repeat the test. If repeat failure occurs correct the problem and the next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Verify the IC at the beginning, after every 10 samples and at the end of the analytical sequence.	Response should be within $\pm 30\%$ of the expected value. Verification is accomplished by the measurement of the gasoline standard.	Repeat test. If failure occurs re-calibrate. Samples injected after criteria was exceeded must be re-analyzed.
	Retention Time Window	Each initial calibration and updated with the CV or as frequently as needed.	Not specified. Method 8015AZ suggest C6 and C10 RT markers	Correct the problem then reanalyze all samples since the last retention time check.
	Stock Standard Expiration	In-house criteria: 6 months, store in the dark at $4^{\circ}\text{C}$ .	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	In-house criteria: aqueous standards 24 hours.	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD SW8081A**

**TABLE 9-31**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8081A  Pesticides	Initial Demonstration of Proficiency (IDP)	Once per analyst.	Extract and analyze 4 replicates. Method 8081A does not specify accuracy and precision. Use Laboratory defined criteria.	If any analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.	If RSD is $\leq 20\%$ linearity through the origin is assumed and average CF is used. If RSD is $> 20\%$ and correlation coefficient is $> 0.99$ then linear calibration not forced through zero is acceptable. Alternatively, prepare a minimum of a 6 point calibration curve with a coefficient of determination $> 0.99$ .	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires response to be within $\pm 20\%$ of the expected value.	Repeat test. If repeat failure occurs correct the problem and next two ICVs must pass or recalibrate.
	Calibration Verification (CV)	At the beginning of the analysis, once every 10 samples and at the end of the sequence.	Response should be within $\pm 15\%$ of the expected value.	Repeat test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Retention Time Window	Each initial calibration and/or calibration verification.	$\pm 3$ times the standard deviation of each analyte's retention time.	Correct the problem then reanalyze all samples since the last retention time check.
	Endrin and DDT Breakdown Check	Each 12 hour shift.	Degradation $\leq 15\%$ or relative response must be consistent during an analytical batch.	Repeat breakdown check.
	Stock Standard Expiration	One year, store in the dark at $4^{\circ}\text{C}$ .	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	6 months, store in the dark at $4^{\circ}\text{C}$ .	Not specified.	If comparison to second source indicates degradation, replace standards.



**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD SW8082**

**TABLE 9-32**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8082  PCBs	Initial Demonstration of Proficiency (IDP)	Once per analyst.	Extract and analyze 4 replicates. Compare to laboratory defined criteria.	If any analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.	If RSD is $\leq 20\%$ linearity through the origin is assumed and average CF is used. If RSD is $> 20\%$ and correlation coefficient is $> 0.99$ then linear calibration not forced through zero is acceptable. Alternatively, prepare a calibration curve with a coefficient of determination $> 0.99$ .	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires the response to be within $\pm 20\%$ of the expected value.	Repeat test. If repeat failure occurs correct the problem and next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	At the beginning of the analysis, once every 10 samples and at the end of the sequence.	Response should be within $\pm 15\%$ of the expected value.	Repeat test. If failure occurs re-calibrate. Samples injected after criteria was exceeded must be re-analyzed.
	Retention Time Window	Each initial calibration and calibration verification.	$\pm 3$ times the standard deviation from the average of each congener's retention time.	Correct the problem then reanalyze all samples since the last retention time check.
	Stock Standard Expiration	One year, store in the dark at $4^{\circ}\text{C}$ .	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	6 months, store in the dark at $4^{\circ}\text{C}$ .	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD SW8260B  
TABLE 9-33**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8260B VOCs	Initial Demonstration of Proficiency (IDP)	Once per analyst.	Analyze 4 replicates. Method 8260B specifies accuracy and precision as guidance only until laboratory has defined its own accuracy and precision. Use Laboratory defined criteria and/or method Table-7 as a guide.	If any analyte fails, correct the source of the problem and repeat the test.
	BFBTune	50 ng BFB initially and every 12 hour shift.	Confirm all key m/z criteria in method Table-4, CLP or Method 524.2.	Re-tune the mass spectrometer and repeat the test until all criteria are achieved.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. Lowest level must be at or below the minimum reporting limit.	If RSD is $\leq 15\%$ linearity through the origin is assumed and average RF is used. If RSD is $> 15\%$ and correlation coefficient is $> 0.99$ then linear calibration not forced through zero is acceptable. Alternatively, prepare a minimum of a 6 point calibration curve with a coefficient of determination $> 0.99$ . RSD for CCCs must be $\leq 30\%$ , RF for SPCCs must be $\geq 0.300$ for chlorobenzene, and 1,1,2,2-tetrachloroethane and $\geq 0.100$ for chloromethane, 1,1-dichloromethane, and bromoform..	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires response to be within $\pm 25\%$ of the expected value.	Repeat test. If repeat failure occurs correct the problem and next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Mid level calibration standard run at the beginning of every 12 hour shift.	RF for CCCs, must be $< 20\%$ difference from the IC. RF for SPCCs same as for the IC. Internal standard areas must be $-50\%$ to $+200\%$ of the initial calibration. Note: See criteria for Arizona and DOD	Repeat test. If failure occurs re-calibrate. Samples injected after criteria was exceeded must be re-analyzed.
	Retention Time Window	Each initial calibration and/or calibration verification.	RRT must be $\pm 0.06$ RRT units from mid point standard.	Correct the problem then re-analyze all samples since the last retention time check.
	Stock Standard Expiration	Liquid standards stable for at least 6 months. Gas standards stable up to one week. Store with minimal head space in the dark at $-10^{\circ}$ to $-20^{\circ}\text{C}$ .	Fresh standards for liquids or gasses should be prepared if a check against the initial calibration exceeds a 20% drift.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	Aqueous standards can be stored up to 24 hr, zero headspace.	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD SW8270C**

**TABLE 9-34**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8270C  SVOCs	Initial Demonstration of Proficiency (IDP)	Once per analyst.	Extract and analyze 4 replicates. Method 8270C specifies accuracy and precision as guidance only until laboratory has defined its own accuracy and precision. Use Laboratory defined criteria and/or method Table-6 or 7 as a guide. (Same table as 625)	If any analyte fails, correct the source of the problem and repeat the test.
	DFTPP Tune	50 ng DFTPP initially and every 12 hour shift.	Confirm all key m/z criteria in method Table-3 are achieved.	Retune the mass spectrometer and repeat the test until all criteria are achieved.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. Lowest level must be at or below the minimum reporting limit.	If RSD is $\leq 15\%$ linearity through the origin is assumed and average RF is used. If RSD is $>15\%$ and correlation coefficient is $> 0.99$ then linear calibration not forced through zero is acceptable. Alternatively, prepare a minimum of a 6 point calibration curve with a coefficient of determination $>0.99$ . RSD for CCCs must be $\leq 30\%$ Table-4 and RF for SPCCs must be $\geq 0.050$ .	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires response to be within $\pm 25\%$ of the expected value.	Repeat test. If repeat failure occurs correct the problem and next two ICVs must pass or recalibrate.
	Calibration Verification (CV)	Mid level calibration standard run at the beginning of every 12 hour shift.	CCCs must be $<20\%$ difference from the expected value. RF for SPCCs $> 0.050$ .	Repeat test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Retention Time Window	Each initial calibration and/or calibration verification.	Relative Retention Time (RRT) must be within $\pm 0.06$ RRT units of the daily standard.	Correct the problem then reanalyze all samples since the last retention time check.
	Stock Standard Expiration	One year, store in the dark at $4^{\circ}\text{C}$ .	Replace if tests indicate a problem.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	Store according to manufacturers recommended holding time and temperature.	Replace if tests indicate a problem.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD SW8270C (PNAs by SIMs)**

**TABLE 9-35**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8270C  (PNA by SIMs)	Initial Demonstration of Proficiency (IDP)	Once per analyst.	Extract and analyze 4 replicates. Method 8270C specifies accuracy and precision as guidance only until laboratory has defined its own accuracy and precision. Use Laboratory defined criteria and/or method Table-6 or 7 as a guide.	If any analyte fails, correct the source of the problem and repeat the test.
	DFTPP Tune	50 ng DFTPP initially and every 12 hour shift.	Confirm all key m/z criteria in method Table-3 are achieved.	Re-tune the mass spectrometer and repeat the test until all criteria are achieved.
	Column Performance Check (CPC)	NOT REQUIRED (Use 625 criteria) At the beginning of each day.	Confirm 3,3'-dichlorobenzidine peak tailing factor is less than 3.0.	Correct the problem, then repeat the column performance check.
	Initial Calibration (IC)	Initial calibration prior to sample analysis, minimum of 5 levels covering the working range of the instrument. Lowest level must be at or below the minimum reporting limit.	If RSD is $\leq 15\%$ linearity through the origin is assumed and average RF is used. If RSD is $>15\%$ and correlation coefficient is $> 0.99$ then linear calibration not forced through zero is acceptable. Alternatively, prepare a minimum of a 6 point calibration curve with a coefficient of determination $>0.99$ . RSD for CCCs must be $\leq 30\%$ Table-4 and RF must be $\geq 0.050$ .	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established.	No method/NELAP criteria. DoD requires response to be within $\pm 25\%$ of the expected value.	Repeat test. If repeat failure occurs correct the problem and next two ICVs must pass or re-calibrate.
	Calibration Verification (CV)	Mid level calibration standard run at the beginning of every 12 hour shift.	Target analytes must be $<20\%$ difference from the IC. RF must be $> 0.050$ .	Repeat test. If failure occurs re-calibrate. Samples injected after criteria was exceeded must be re-analyzed.
	Retention Time Window	Each initial calibration and/or calibration verification.	Relative Retention Time (RRT) must be within $\pm 0.06$ RRT units of the daily standard.	Correct the problem then re-analyze all samples since the last retention time check.
	Stock Standard Expiration	One year, store in the dark at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	Store according to manufacturers recommended holding time and temperature.	Not specified.	If comparison to second source indicates degradation, replace standards.

Alpha Analytical, Inc  
Section No.: 9.0  
Revision No.: 15.0  
Date: January, 2007  
Page 49 of 58

**RCRA**  
**INORGANIC METHODS**

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 6020  
TABLE 9-36**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
6020 Metals	Demonstration of Capabilities (DOC)	Not specified by the method  NELAP requires once per analyst, and annually thereafter.	Extract and analyze 4 replicates. Method 6020 specifies the lab to determine its own accuracy and precision.	
	Instrument Detection Limits (IDL)	Instrument detection limit studies are performed quarterly.	Calculate the average of the standard deviation of 3 runs on 3 non-consecutive day from the analysis of a reagent blank and 7 consecutive measurements per day. (Will used 10 measurements per day, method 200.8)	
	Tuning	Daily, pre-calibration, prior to sample analysis.	Analyze tuning solution (1 to 10 ug/L Li, Co, In, Tl) 4 times. (Will use 5 replicates) RSD of absolute signals $\leq 5\%$ . Mass calibration $< 0.1$ amu from true value, resolution $< 0.9$ amu at 10% peak height. (Will use resolution of $< 0.75$ amu at 5% peak height, method 200.8).	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Initial Calibration	Daily. Initial calibration prior to sample analysis. Minimum of calibration blank and 1 level using the average of 3 integrations.	Per instrument manufacturer's specifications. Alpha performs a multipoint calibration defining the low end of the calibration. See SOP for details	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Verify each element calibration with an ICV prepared from a source independent from the calibration standards at a concentration equivalent to or near the midpoint of the calibration curves and at a concentration different than that used for instrument calibration.	Percent Recovery (%R) = 90 - 110	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test. If the problem occurs twice than recalibrate, or the ICV must pass on the third and fourth consecutive tests.
	Internal Standards (IS)	Add IS to all standards. Recommended IS Li, Sc, Y, In, Tb, Ho and Bi.	IS intensity levels of the CB, ICV, and CV must agree to within $\pm 20\%$ of the level in the IC.	Stop the analysis, correct the problem, re-calibrate, and re-analyze the affected samples.
	Calibration Verification (CV)	Analyze CV after IC, every 10 samples and at the end of the analytical run.	Percent Recovery (%R) = 90 - 110	Same as the ICV
	Calibration Blank (CB)	Analyze CB prior to the CV, every 10 samples and at the end of the analytical run.	Results must be less than 3 times IDL per element.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test or re-evaluate the IDL level.
	Interference Check Solution (ICS)	Analyze ICS containing interfering elements and TAL to verify correction factors at the beginning of the run and every 12 hours.	No criteria specified. Arizona ADEQ requires the criteria to be statistically derived windows.	Evaluate on a case-by-case basis.

	Standard Expiration	ICS is prepared weekly Stock standards are prepared yearly.	No criteria specified. However, a 1 year or manufacturer's expiration date will be used as the default.	
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**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 9040C/9045D  
TABLE 9-37**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
9040C 9045D  pH	Initial Calibration (IC)	Initial calibration prior to analysis, minimum of 2 pH buffers.	Prepare calibration curve to bracket sample concentration. Manufacturer suggests a curve slope of 92 - 102%	Repeat the test. If failure occurs recalibrate.
	IC for Corrosivity Characterization	Include pH buffer at 1.68 for acidic wastes and pH buffer at 12.45 for caustic wastes	Not specified. The same as above.	Repeat the test. If failure occurs recalibrate.
	Initial Calibration Verification (ICV)	Not specified. Lab suggests after each new initial calibration.	Not specified. However, PE criteria is $\pm 10\%$ . Therefore response should be $\pm 10\%$ of the expected value.	Repeat the test. If failure occurs recalibrate.
	Calibration Verification (CV)	Not specified. Manufacturer suggests to verify the calibration every 2 hours.	Not specified. However, PE criteria is $\pm 10\%$ . Therefore response should be $\pm 10\%$ of the expected value.	Repeat the test. If failure occurs recalibrate. Samples measured after criteria was exceeded must be reanalyzed.
	pH Buffer Expiration	Not specified. Minimum of six months or vendor expiration date.	Not specified.	If degradation or contamination is suspected, replace the standards.
	Working Standard Expiration	Not specified. Daily	Not specified	If degradation or contamination is suspected, replace the standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 9050A**

**TABLE 9-38**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
9050A  Specific Conductance	Initial Calibration (IC)	Initial calibration prior to analysis. Manufacturer suggests, 2 EC points, 1 in air.	Prepare calibration curve to bracket sample concentrations.	Repeat the test.
	Initial Calibration Verification (ICV)	After each new initial calibration. Per our SOP we will add: and daily before sample analysis.	Not specified. However, we will use method 314.0 criteria. Daily ICV at 1410 $\mu$ S/cm with an acceptance criteria of 1380 to 1440 $\mu$ S/cm.	Repeat the test. If failure occurs recalibrate with a new standard. Samples measured after criteria was exceeded must be reanalyzed.
	Calibration Verification (CV)	Not specified. Will verify daily and bracket sample analysis.	Not specified. However, we will use method 314.0 criteria. Daily CV at 1410 $\mu$ S/cm with an acceptance criteria of 1380 to 1440 $\mu$ S/cm.	Repeat the test. If failure occurs recalibrate with a new standard. Samples measured after criteria was exceeded must be reanalyzed.
	Standard Expiration	Not specified. Minimum of one year or vendor expiration date	Not specified.	If degradation or contamination is suspected, replace standards.



**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 9056**

**TABLE 9-39**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
9056  Anions	Initial Demonstration of Proficiency	Not specified		
	Initial Calibration	Initial calibration prior to analysis, minimum of 3 concentration levels. One level near, but above, the reporting limit.	Prepare a calibration curve using a linear least squared calibration model to bracket sample concentrations. No other method criteria.	If sample analytes exceed cal range, dilute sample or recalibrate at a higher concentration point.
	Initial Calibration Verification ICV	Not specified.		
	Calibration Verification CV	Daily, or whenever eluent is changed, after every 10 injections and for every batch of samples.	Midrange standard. Response should be $\pm 5\%$ of the expected value. Note: This is unreasonable and method 300.0 criteria of $\pm 10\%$ will be used.	Repeat the test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Retention Time Window	Each initial calibration and calibration verification over the course of the day.	$\pm 3$ times the standard deviation centered around the mean.	If CV's or other QC checks are outside the window recheck and/or recalculate window
	Stock Standard Expiration	Minimum of one month, when stored at 4°C.	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working standard Expiration	Weekly except for nitrite, nitrate and o-phos. Should be prepared daily.	Not specified.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR METHOD 9060  
TABLE 9-40**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
9060 TOC	Initial Demonstration of Proficiency (IDP)	Not required. Once per analyst.	Not specified. Analyze 4 replicates. Evaluate accuracy and precision against method defined criteria.	If analyte fails, correct the source of the problem and repeat the test.
	Initial Calibration (IC)	Not specified. NELAP requires a minimum of 3 calibration points with the lowest at or below the reporting level and the other two covering the working range of the calibration.	Not Specified.  Instrument manufacturer only allows the use of a linear calibration. NELAP requires a correlation coefficient of > 0.995 when not specified.	Correct the problem then repeat the initial calibration.
	Initial Calibration Verification (ICV)	Not required by the method, required by NELAP. Immediately after the IC has been established	In-house criteria, should pass the CV criteria to be acceptable.	Repeat the test. If repeat failure occurs correct the problem and next two ICVs must pass or recalibrate.
	Calibration Verification (CV)	Method 9060A specifies a CV evry 15 samples. However, in-house batch size is 20 samples; therefore, minimum of 2, before sample analysis and at the end of the sequence or once every 20 samples and preferably once every 10 samples.	Not Specified. Response should be with in $\pm 20\%$ of the expected value.	Repeat the test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Stock Standard Expiration	Not specified. One year, store in the dark at 4°C.	Not specified	If comparison to secound source indicates degradation, replace the standards.
	Working Standard Expiration	Not specified. One year, store in the dark at 4°C.	Not specified	If comparison to secound source indicates degradation, replace the standards.

**SUMMARY OF CALIBRATION PROCEDURES  
 FOR METHOD ASTM D2216  
 TABLE 9-41**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ASTM D2216  Percent Moisture	Initial Calibration (IC)	Not applicable		
	Initial Calibration Verification (ICV)	Not applicable		
	Calibration Verification (CV)	Not applicable		
	Standard Expiration	Not applicable		
	Working standard Expiration	Not applicable		

Alpha Analytical, Inc  
Section No.: 9.0  
Revision No.: 15.0  
Date: January, 2007  
Page 56 of 58

**IN-HOUSE  
DEVELOPED  
METHODS**

**SUMMARY OF CALIBRATION PROCEDURES  
FOR DISSOLVED GASES  
TABLE 9-42**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SOP RSK-175  Dissolved Gases	Initial Demonstration of Performance (IDP)  Not specified by RSK-175	IDP should be conducted annually.	Prepare and analyze 4 replicate LCSs. Average recoveries must be within the laboratory established criteria.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem. IDP should be conducted prior to sample analysis.
	Initial Calibration (IC)  Not specified by RSK-175	Initial calibration prior to analysis, minimum of 5 concentration levels covering the working range of the instrument	Prepare calibration curve to bracket sample concentrations.	If sample analytes exceed cal range, dilute sample or recalibrate at a higher concentration point.
	Initial Calibration Verification (ICV)  Not specified by RSK-175	Each time a new IC is prepared.	Second source standard, $\pm$ 25% of the expected value.	If the ICV exceeds $\pm$ 25% of the expected value, reanalyze second time or recalibrate.
	Calibration Verification (CV)  Not specified by RSK-175	Daily, after every 10 samples and at the end of the sample run.	Response should be $\pm$ 25% of the expected value.	Repeat the test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Stock Standard Expiration  Not specified by RSK-175	Manufacturers expiration date	Compare to check standards.	If comparison to second source indicates degradation, replace standards.
	Working standard Expiration  Not specified by RSK-175	As needed	Compare to check standards.	If comparison to second source indicates degradation, replace standards.

**SUMMARY OF CALIBRATION PROCEDURES  
FOR ORGANIC ACIDS**

**TABLE 9-43**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Organic Acids	Initial Demonstration of Performance (IDP)	Should be conducted annually.	Prepare and analyze 4 replicate LCSs. Average recoveries must be within the laboratory established criteria.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem. IDA should be conducted prior to sample analysis.
	MDL	Should be conducted annually, new operator, or change in instrument response.	MDL must be less the minimum reporting limit	If MDL is above RL, reevaluate RL or conduct MDL study a second time.
	Initial Calibration (IC)	Initial calibration prior to analysis, minimum of 5 concentration levels covering the working range of the instrument	Prepare calibration curve to bracket sample concentrations.	If sample analytes exceed cal range, dilute sample or recalibrate at a higher concentration point.
	Initial Calibration Verification (ICV)	Each time a new IC is prepared.	Second source standard, $\pm$ 30% of the expected value.	If the ICV exceeds $\pm$ 30% of the expected value, reanalyze second time or recalibrate.
	Calibration Verification (CV)	Daily, or whenever eluent is changed, after every 10 samples and at the end of the sample run.	Response should be $\pm$ 20% of the expected value.	Repeat the test. If failure occurs recalibrate. Samples injected after criteria was exceeded must be reanalyzed.
	Retention Time Window	Each initial calibration and calibration verification over the course of the day.	$\pm$ 3 times the standard deviation centered around the mean.	If CV's or other QC checks are outside the window recheck and/or recalculate window
	Stock Standard Expiration	Minimum of one 6months.	Not specified.	If comparison to second source indicates degradation, replace standards.
	Working Standard Expiration	Weekly.	Not specified.	If comparison to second source indicates degradation, replace standards.

# **Section 10**

## **Equipment and Instrument Maintenance**

## **10.0 EQUIPMENT**

10.0.1 Alpha Analytical, Inc. provides a full range of environmental analyses for contaminants in soil, water, industrial waste and other matrices. Alpha has analytical capabilities including sophisticated instrumentation for the detection of metals, and other inorganic analytes, pesticides, herbicides, industrial solvents, PCBs, and other analytes encompassing the range of Hazardous Substances, Priority Pollutants, and the identification of thousands of organic compounds by Gas Chromatography/Mass Spectrometry (GC/MS).

10.0.2 It is Alpha's policy to purchase equipment capable of achieving the accuracy, precision, sensitivity, and selectivity required for the intended use of the analytical test methods.

## **10.1 EQUIPMENT OPERATION POLICY**

10.1.1 Analytical test instruments are only operated by authorized personnel. Instrument manuals on the use and maintenance of this equipment is available for use by the analysts.

## **10.2 EQUIPMENT IDENTIFICATION**

10.2.1 Analytical test instruments and their associated software are uniquely identified. This identification is used on all documents to reference that particular instrument.

10.2.2 Instrument identification documents are maintained to record the following information:

- a) the identity of the item of equipment and its software;
- b) the manufacturer's name, type of equipment, and serial an/or model number;
- c) date received and date placed in service (if available); and
- d) if available, condition when received (e.g., new, used, reconditioned).

## **10.3 MAJOR EQUIPMENT**

Alpha Analytical is equipped with modern instrumentation to provide the quality of data required by the regulatory agencies and to provide redundancy for all major systems.

### **10.3.1 Gas Chromatograph/Mass Spectrometers for Volatile Organic Analysis**

Alpha is equipped with six Hewlett-Packard (HP) 5890 and 5890 series II GCs attached to HP 5970 and 5972 MSDs and six Agilent Technologies (AT) 6890GC's attached to AT 5973 MSD's. The MSDs are quadrupole mass analyzers and are equipped with turbo molecular pumps. Mass spectral data is acquired using windows based PCs and the HP/AT ChemStation and Enviroquant software.

Water samples are introduced into the GC's using Tekmar Liquid Sample



Concentrators (LSC) and Tekmar Automatic Liquid Samplers (ALS). These (GC/MS) systems are dedicated to the analysis of Volatile Organic Compounds (VOCs) by methods 624, 8260B, 524.2 and total petroleum hydrocarbons..

#### 10.3.2 Gas Chromatograph/Mass Spectrometer for Semi-volatile Analysis

Alpha's semi-volatile instruments use the same hardware as described for the volatiles GC/MS systems excluding the purging devices. The instruments are configured for automatic sample injection using the HP7673 or AT 7683 auto-towers. Data acquisition and reduction is acquired through the use of a windows based PC, HP/AT ChemStation and Enviroquant Software. This instrument is dedicated to the analyses of semi-volatile organic compounds by methods 625 and 8270C.

#### 10.3.3 Gas Chromatographs with Flame Ionization Detectors for Petroleum Hydrocarbons, Dissolved Gases and General Screening Analysis

Alpha is equipped with three HP 5890 GC's and three AT 6890 each with Flame Ionization Detectors (FID). Four of these systems are configured with the ProSep® large volume injection inlets and capillary columns for Method 8015B, DRO Total Petroleum Hydrocarbon (TPH) analysis. One system is configured with a headspace analyzer for the analysis of dissolved gases, and the other for screening SVOCs.

#### 10.3.4 Gas Chromatographs with Electron Capture Detectors for Pesticides, and PCB Analysis

Alpha has two HP 5890 and one AT 6890 Gas Chromatographs; each equipped with dual Electron Capture Detectors (ECD). These instruments are configured with dual detectors for simultaneous confirmation of analytes. These instruments are configured for methods: 608, SM6630C, 8081A, 8082, and PCBs in oil. These systems use personal computers for data acquisition, reduction and storage. The GC/ECDs are configured for automatic sample injection using the HP 7673 and AT 7683 auto towers. These are dual tower automatic sample injection systems which, in conjunction with the dual detectors, can perform both analysis and confirmation simultaneously.

#### 10.3.5 High Pressure Liquid Chromatograph (HPLC)

Alpha has a HP1050 (TI) HPLC quaternary pump suitable for both gradient and isocratic instrument conditions. This system is configured with a Ultra Violent (UV) and fluorescence detector in series. Data is acquired on a PC through an A/D converter box using AI-450 software.

The second HPLC system is configured with a Diode-Array Detector (DAD), and fluorescence detectors which simultaneously collects continuous UV spectra and a

fluorescence detectors in series. This UV spectra is used to confirm analysis with suspected real compounds by comparing its UV spectra for positive identification. Data is acquired on a PC using the HP Phoenix Photo Diode-Array software.

#### 10.3.6 Ion Chromatograph (IC) with Conductivity Detector for the Analysis of Perchlorate and Other Common Ions

Ions are determined by the use of a Dionex DX 500 and DX 600 Ion Chromatography system. The DX500 is a modular system consisting of the GP40 pump, CD-20 conductivity detector, LSC 25 column heater department and AS40 auto-sampler.

The DX600, IC is configured essentially the same as above; however this instrument has a GP 50, a CD25 detector. Both systems use the Chromellian software for data acquisition.

Perchlorates are determined by the use of a Dionex ICS2000 Ion Chromatography system. This is a non-modular system where the pump, detector and column heater are built into a single instrument. The AS-40 auto-sampler is modular and is the same auto-sampler as used for the other two systems. This instrument also uses the Chromellian software for data acquisition.

#### 10.3.7 Inductively Coupled Plasma / Mass Spectrometer (ICP-MS) for Metals Analysis

Most metals analysis is performed on an AT7500i ICP-MS. This system is configured with the Cetax ASX510AS autosampler and a Neslab NSLCF-100 chiller. Data acquisition is accomplished with the Agilent ChemStation software. This instrument is dedicated to metals analysis by methods 200.8 and 6020.

#### 10.3.8 Accelerated Solvent Extractor (ASE 200)

The ASE 200 is used by method SW3545 for accelerated solvent extraction of base/neutral and acids (BNAs), polychlorinated biphenyls (PCBs) and chlorinated pesticides. The ASE 200 accelerates the extraction of solid matrices by using solvents at elevated temperature and pressure. Increased temperature accelerates the extraction kinetics, while elevated pressure keeps the solvent below the boiling point, thus enabling safe and rapid extraction.

#### 10.3.9 Microwave Digestor for the Digestion of Metals

The digestion of RCRA samples for metals analysis is performed using a Milestone Ethos Plus microwave digestion instrument. This microwave digestion instrument is designed with a complete temperature and pressure control system, thus preventing over-pressurization. The instrument is controlled with a PC using the EasyWAVE software for automatic parameter control.

**ANALYTICAL INSTRUMENT LIST**  
**TABLE 10-1**

<b>INSTRUMENT ID</b>	<b>MODEL</b>	<b>DETECTOR</b>	<b>AUTO-SAMPLER</b>	<b>DATA ACQUISITION SYSTEM</b>	<b>METHODS OF ANALYSIS</b>
GC/MSD # 1	HP5890	HP5970B	LSC-3000 Aqua Tek-70	HP ChemStation	VOC Screens
GC/MSD # 2	HP5890	HP5970B	HP7673A ProSep 800 plus	HP ChemStation	MeOH, EtOH
GC/MSD # 3	HP5890 (Series II)	HP5970	LSC-2000 ALS-2050	HP ChemStation	524.2
GC/MSD # 4	HP5890 (Series II)	HP5972A	LSC-2000 ALS-2050	HP ChemStation	VOC Screens
GC/MSD # 5	HP5890A	HP5970B	LSC-2000 Precept II	HP ChemStation	VOC Screens
GC/MSD # 6	HP5890 (Series II)	HP5972	LSC-3000 Aqua Tek-70	HP ChemStation	624, 8260, TPH-G
GC/MSD # 7	AT6890	AT5973	Velocity XPT Solatex-72	AT Chem Station	624, 8260, TPH-G
GC/MSD # 8	AT6890	AT5973	LSC-3100 Aqua Tek-70	AT ChemStation	624, 8260, TPH-G
GC/MSD # 9	AT6890N	AT5973N	LSC-3000 Aqua Tek-70	AT ChemStation	624, 8260, TPH-G
GC/MSD # 10	AT6890	AT5973	LSC-3000 Aqua Tek-70	AT ChemStation	624, 8260, TPH-G
GC/MSD # 11	HP5890	HP5970	HP7673A ProSep 800 plus	HP ChemStation	MeOH, EtOH
GC/MSD # 12	AT6890N	AT5973N	LSC-3100 Aqua Tek-70	AT ChemStation	624, 8260, TPH-G
GC/MSD # 14	AT6890N	AT5973Inert	AT7683	AT ChemStation	625, 8270C, PNA SIMs
GC/MSD #15	AT6890N	AT5973Inert	Velocity XPT Aqua Tek-70	AT Chem Station	624, 8260, TPH-G

# **ANALYTICAL INSTRUMENT LIST**

**Continued**  
**TABLE 10-1**

<b>INSTRUMENT ID</b>	<b>MODEL</b>	<b>DETECTOR</b>	<b>AUTO-SAMPLER</b>	<b>DATA ACQUISITION SYSTEM</b>	<b>METHODS OF ANALYSIS</b>
GC/FID #1	AT 6890	FID	AT 7683B ProSep 800 Plus	Dionex AI-450	TPH-D
GC/FID #2	AT 6890	FID	AT 7683B ProSep 800 Plus	Dionex AI-450	TPH-D
GC/FID #3	HP 5890 Series II	FID	HP 7673A ProSep 800 Plus	Dionex AI-450	TPH-D
GC/FID #4	HP 5890 Series II	FID	HP 7673A ProSep 800 Plus	Dionex AI-450	TPH-D
GC/FID #5	HP 5890 Series II	FID	HP 7673A	HP Chem Station	SV Screen
GC/FID #6	AT 6890N	FID	AT G1888 Headspace Sampler	AT Chem Station	Dissolved Gases
GC/ECD # 2	HP5890 (Series II)	Dual ECD	HP7673B	DIONEX AI-450	Screen
GC/ECD # 4	HP5890 (Series II)	Dual ECD	HP7673B	DIONEX AI-450	608, 8081A, 8082, PCB/OIL
GC/ECD # 5	AT6890 (plus)	Dual Micro-ECD	AT7683	DIONEX AI-450	608, 8081A, 8082, PCB/OIL
HPLC # 1	HP1050 (TI)	HP1046A HPDAD	HP7673/1050	DIONEX AI-450	610, 8310
HPLC # 3	HP1050 (TI)	HP1046A HP1050UV	HP7673/1050	DIONEX AI-450	Organic acids
IC #1	DX500	CD-20	AS-40	Chromellian	300.0
IC #2	DX600	CD-25	AS-40	Chromellian	300.0
IC #3	ICS2000	D-56	AS-40	Chromellian	314.0
TOC	Dohrmann Phoenix 8000	UV	Dohrman STS 8000	TOC Talk	415.1 / 9060
ICP-MS	Agilent 7500i	MS	Cetax ASX510AS	ChemStation	200.8, 6020/6020A

**EQUIPMENT IDENTIFICATION FORM**  
**Table 10-2**

1) Date Reviewed:			
2) Equipment Identification Configuration			
3)	GC	Equipment Manufacturer	
		Model Number	
		Serial Number	
		Purchase Date	
		Date Placed in Service	
4)	Detector	Equipment Manufacturer	
		Model Number	
		Serial Number	
		Purchase Date	
		Date Placed in Service	
5)	Liquid Sample Concentrator	Equipment Manufacturer	
		Model Number	
		Serial Number	
		Purchase Date	
		Date Placed in Service	
6)	Auto sampler	Equipment Manufacturer	
		Model Number	
		Serial Number	
		Purchase Date	
		Date Placed in Service	
7)	Computer	Equipment Manufacturer	
		Model Number	
		Serial Number	
		Purchase Date	
		Date Placed in Service	
8)	Software	Software Name	
		Software Revision	

## **10.4 SUPPORT EQUIPMENT**

Support equipment is defined as those devices that may not be the actual test instrument, but are necessary to support general laboratory operations. This equipment include such things as: balances, ovens, refrigerators, freezers, water baths, temperature measuring devices, and volumetric dispensing devices (such as Eppendorf pipetors and automatic dispensing devices). Procedures for individual instruments are described in detail in Appendix D. In general the following are included in those SOPs:

- a) All support equipment is maintained in proper working order and a record of all repair and maintenance activities including service calls are maintained in their respective maintenance logbooks.
- b) All support equipment is calibrated or verified at least annually using NIST traceable references when available, over the entire range of use. The results of these calibrations or verifications are maintained to ensure the equipment is within the specification required of the application for which the equipment is used. If the equipment does not meet these specifications then:
  - 1) the equipment is removed from service until repaired; or
  - 2) records are maintained to establish correction factors to correct all measurements.
- c) Raw data records are maintained to document equipment performance.
- d) Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths are checked in the expected range with NIST traceable references where commercially available. The acceptability of use or continued use to established to the needs of the analysis or application for which the equipment is being used.
- e) Mechanical volumetric dispensing devices (MVDD) (excluding Class A glassware) is checked for accuracy on at least a quarterly basis.

Note: Glass microliter syringes are considered the same as Class A glassware, but a certificate attesting to accuracy is documented and maintained.

## **10.5 INSTRUMENT MAINTENANCE**

### **10.5.1 Policy**

It is Alpha's policy to maintain, inspect and clean all equipment, and to document maintenance procedures in the maintenance log book.

### **10.5.2 Purpose**

Alpha's Preventative Maintenance Program (PMP) establishes the basic procedures for maintaining test and measurement equipment used to conduct sample analyses. These procedures are established to ensure laboratory equipment perform their intended functions in a timely and effective manner.

### 10.5.3 Responsibility

Preventative maintenance is a critical element of the quality assurance program at Alpha Analytical. Responsibility for preventative maintenance lies with the analyst and their direct supervisor in charge of monitoring equipment. Our analytical staff is dedicated to the implementation of the preventative maintenance program and are always watchful for signs that there is a need for maintenance activities.

#### 10.5.3.1 In-house Maintenance

Analysts perform routine preventive maintenance such as the replacement of parts, cleaning of components, and changing of pump oil. Analytical instruments such as GCs, GC-MS and ICP-MS systems are serviced and maintained by in-house personnel.

#### 10.5.3.2 Outsourced Maintenance

Occasionally there are instances when service personnel are contracted to replace or service instruments that cannot be serviced by our personnel. Other instruments such as analytical balances are serviced on a routine basis by a contracted company. All instrument maintenance is recorded in an instrument maintenance logbook specifically associated with that particular instrument.

### 10.5.4 Monitoring

Instruments are constantly monitored by the use of daily standards, sensitivity, and response checks to determine if maintenance is required. In the event that an instrument does fail, every effort is made to meet obligations to the client's holding times and analysis due dates.

Laboratory support equipment such as refrigerators and ovens are also monitored and serviced regularly. The laboratory quality assurance program is designed to reduce data loss by monitoring and recording the functioning of these systems, allowing rapid correction of any malfunction before data loss can occur.

Alpha Analytical's Preventative Maintenance Program concentrates on four primary areas of concern and they are as follows:

- 1) A suggested PM schedule. See Table 10-3;
- 2) Documentation of all maintenance and repairs;

- 3) Vendor/manufacturing operation and maintenance manuals available for all instruments; and,
- 4) Alpha Analytical's Analytical Contingency Plan.

#### 10.5.5 Maintenance Schedule

##### 10.5.5.1 Equipment Purchase

When Alpha Analytical was established in 1987, one of the primary goals in the start-up operation was to buy the finest equipment available to reduce down time. Hewlett-Packard/Agilent Technologies instruments were purchased for most major instruments used for organic analyses.

##### 10.5.5.2 Suggested Maintenance Schedules

Maintenance that is performed on a regular schedule consists of changing pump oil, changing septum and injection inserts, cleaning syringe barrels on automatic sample injectors, etc. Most other types of maintenance are those that cannot be prevented by regular servicing, such as electronic board failure, filament burnout, detection degradation, etc.

Table 10-3 identifies suggested preventative maintenance activities by instrument type and recommended frequencies. It should be noted that it may be necessary to perform activities more frequently depending on workload, sample types analyzed, and/or instrument performance. Frequency of instrument maintenance activities incorporates both laboratory experience and instrument manufacturer's recommended PM frequency.

## 10.6 MAINTENANCE LOGBOOK

- 10.6.1 An individual instrument maintenance logbook is assigned to each instrument. This logbook is used to record preventive maintenance checks and services in addition to emergency maintenance procedures. The maintenance logbook contains descriptions of instrument problems, solutions, replacement parts, and maintenance personnel.

Each maintenance period or problem is signed and dated to record the maintenance history of each piece of equipment.

## 10.7 MAINTENANCE MANUALS

- 10.7.1 As stated earlier in this section, Alpha Analytical's instrumentation consists primarily of Hewlett-Packard/Agilent Technologies GCs, GC-MSs, ICP-MSs and



HPLCs. Hewlett-Packard generally publishes three to four separate service books associated with each analytical instrument and they are as follows:

- 1) Operators Handbook - A description of the use and operation of a particular instrument;
- 2) Installation and Maintenance Guide - A handbook that describes how to install, troubleshoot, and maintain each instrument;
- 3) Getting It All Together - A handbook which describes how to connect different modules to make a complete system; and,
- 4) Parts Manual - This handbook displays blow-ups of various sub-components of a system while giving part numbers and electronic schematics.

Alpha Analytical maintains a library of maintenance manuals for all Hewlett-Packard equipment and PM manuals for all other equipment Alpha Analytical uses on a regular basis.

## **10.8 CONTINGENCY PLANS**

### **10.8.1 Instrument Capacity/Back up Instruments**

For most methods, we have the instrument capacity to perform analysis on multiple instruments. This capability changes over time based on sample work load and the availability of it's backup.

### **10.8.2 Spare Parts Inventory**

In the event of complete instrument failure, a number of decisions need to be made quickly in order to prevent invalidating the current sample work load. All priorities and effort will be addressed to resolve the instrument problem. Alpha Analytical maintains a sizeable inventory of spare parts for this scenario. However, it is impractical and impossible to predict and maintain an inventory of all possible spare parts. Parts can be delivered overnight for repair the next day.

### **10.8.3 Service Calls**

The second decision which is made at the onset of instrument failure is if our in-house personnel are able to repair the problem at hand. If not, then a service call is made to determine the logistical requirements of getting a service person to the laboratory at the earliest possible date. Service calls also are useful in talking or resolving an instrument problem over the phone. The conversation usually entails a series of diagnostic activities, which will guide the analyst or person in charge of that instrument to a reason for the instrument failure. This activity is the first plan of attack when an instrument fails for non-obvious reasons. If the problem can still not

be resolved, then the service call goes one step further and a repair person is dispatched to the laboratory.

Alpha Analytical's service priorities are as follows:

- 1) Service our clients by performing the work within their contract or specified turn-around time;
- 2) Perform this work under the guidance of a certification program; and,
- 3) Provide quality analytical data.

#### 10.8.4 Sub-contracting Laboratories

In the event of instrument failure, it is Alpha's goal to accomplish and perform all testing and analysis in-house. When it becomes apparent that Alpha Analytical cannot meet these obligations, Alpha Analytical will then sub-contract this work to one of several laboratories that are available.

**TABLE 10-3**  
**SUGGESTED PREVENTATIVE MAINTENANCE ACTIVITIES**

INSTRUMENT	ACTIVITY		FREQUENCY
Gas Chromatographs	General	Check septa, cylinder gas pressure, and oxygen, moisture traps	D
		Bake out injection body	2
		Check electronics (voltages, waveforms, etc.)	3, 4
	Columns	Change glass inserts, shorten ends of columns, change glass wool plugs, replace ferrules, and check for leaks	3, 5
	Electron Capture Detector	Wipe tests	A
		Return detector to factory for cleaning and refoiling	2,3, 4
	Flame Ionization Detector	Clean	Q
		Replace flare tip	A
		Replace flare ignition	1
	Nitrogen Phosphorous Detector	Clean	Q
		Replace bead	1, 3, 4
Mass Spectrometers	General	Replace vacuum pump oil	A (1)
		Check ion source and analyzer (dismantle and clean, replace parts as needed)	A (1, 2, 3, 4, and 5)
		Check mechanical (vacuum pump, gas pressures, and flows)	Q
	Purge and Trap	Bake vessels	2
		Change trap	A
		Bake trap	2
		Check purge flow	M
		Check for leaks	M

Key to Frequencies: (1) Replace as necessary; (2) High background; (3) Loss of sensitivity or failing resolution; (4) Erratic response; (5) QC failure; (A) Annually; (D) Daily; (M) Monthly; (Q) Quarterly; (BA) Bi-annually; (SA) Semi-annually; and (W) Weekly.

**TABLE 10-4**  
**SUGGESTED PREVENTATIVE MAINTENANCE ACTIVITIES**

INSTRUMENT	ACTIVITY		FREQUENCY
High Pressure Liquid Chromatographs	General	Gas Lines Checked for leaks	D
		Clean mobile phase flow system with 10% nitric acid	BA (3,4)
		Clean detector flow cells	BA(3,4)
		Clean injection valve (replace rotors and seals)	A(1)
		Check solvent filters	W
		Check pump seals and check valve assemblies (clean and replace as pressures and flows of mobile phase indicate)	D(1)
		Lubricate post column reagent pumps	M
		Check valves and replace post column restrictors and frits or clean restrictors with 10% nitric acid	M(1,3,4)
Ion Chromatographs	General	Replace or quick start suppressor	1,2,3,4,5
		Clean detector flow cell	2,3,4
		Clean injection valve (replace rotors and seals)	A(1)
		Check pump seals (clean and replace as pressures and flows of mobile phase indicate)	D(1)
ICP-MS	General	Check cylinder gas pressure	D(1)
	ICP	Clean sample and skimmer cones	2,3,4,5
		Clean torch and spray chamber assemblies	2,3,4,5
		Replace liquid sample lines	M(2,4,5)
	MS	Check turbo pump	Q
		Replace vacuum pump oil	A(1)
		Check ion source and analyzer	A(1,2,3,4,5)
Auto Samplers	General	Check needles/syringes	D(1)
		Replace motors, belts, carriage assemblies, and sensors	1,4
		Clean	Q
Data Systems	General	Clean computers, check battery backup, and check ventilation fans	A

Key to Frequencies:

(1) Replace as necessary; (2) High background; (3) Loss of sensitivity or failing resolution; (4) Erratic response; (5) QC failure; (A) Annually; (D) Daily; (M) Monthly; (Q) Quarterly; (BA) Bi-annually; (SA) Semi-annually; and (W) Weekly.

**TABLE 10-5  
SUGGESTED PREVENTATIVE MAINTENANCE ACTIVITIES**

INSTRUMENT	ACTIVITY		FREQUENCY
Refrigerators/Ovens	General	Clean interiors	SA
		Check thermometer temperatures against NBS certified thermometer	A
Analytical Balances	General	Clean pan and compartment	D
		Check with S class weights	D
pH and Ion Selective Electrodes	Probes	Check probe for cracks and proper levels of filling solution; check reference junction; clean electrode	D(1)
	Meter	Check electronics or batteries for loose connections and cracked leads	D(1)
Spectrophotometers	General	Clean sample compartment interior	SA
		Check wave-length calibration with holmium-oxide filter	A
TOC Instrument	General	Check carrier gas, and reagents	D
		Clean UV reactor and IC sparger	W(2,3,4)
		Inspect chlorine scrubber	M(1,2,3,4,5)
		Inspect permanganate dryer	M(1,2,3,4,5)
Thermometers	General	Check for cracks in the glass and gaps in the fluid	D(1)

Key to Frequencies:

(1) Replace as necessary; (2) High background; (3) Loss of sensitivity or failing resolution; (4) Erratic response; (5) QC failure; (A) Annually; (D) Daily; (M) Monthly; (Q) Quarterly; (BA) Bi-annually; (SA) Semi-annually; and (W) Weekly.

# **Section 11**

## **Quality Control Procedures to Assess Laboratory Data**

## **11.0 QUALITY CONTROL PROCEDURES TO ASSESS LABORATORY DATA**

### **11.1 ELEMENTS OF QUALITY CONTROL CHECKS**

This section presents QC requirements relevant to analysis of environmental samples that are followed during all analytical activities. The purpose of this QC Program is to provide quantitative evidence that the entire analytical method is performed within the specified criteria. Data generated from control samples are used to monitor day-to-day variations in routine analysis, which provides a mechanism for ongoing control and evaluation of data quality measurements through the use of QC material.

### **11.2 INTRA LABORATORY QUALITY CONTROL**

Intra laboratory quality control is performed as described in the Standard Methods for the Examination of Water and Wastewater, 1996, 20<sup>th</sup> Edition, Environmental Protection Agency Methods for Organics in Finished Drinking Water, and Test Methods for Evaluating Solid Waste, 3rd Edition. Quality Control data is used to review and determine method precision and accuracy.

### **11.3 FIELD QC SAMPLES**

The following QC check samples are the basic requirements needed to ensure the reliability and integrity of field data. For details, see the Quality Control Field Samples SOP located in Appendix B.

#### **11.3.1 Equipment/Rinsate Blanks**

These are field QC check samples used to ensure non-dedicated sampling devices (bailers, filtering equipment, pumps, etc.) have been effectively decontaminated. After field washing and decontamination, sampling equipment should be rinsed with reagent free water. This rinse water is then transferred to a sample bottle, and returned to the laboratory for analysis. A representative number of the equipment / rinsate blanks should be analyzed, depending on the SOW.

#### **11.3.2 Field Reagent Blanks (FRB)/ Field Transfer Blanks (FTB)**

These types of QC samples are reagent grade water placed in a sample container at the laboratory and treated exactly as a sample in all respects, including exposure to sampling site conditions, storage, preservation and all analytical procedures. The purpose of the FRB/FTB is to determine if the method analytes or other interferences are present as air-borne constituents in the field environment. These types of QC samples are analyzed for project specific compounds.

#### **11.3.3 Trip Blanks (TB)**

Trip Blanks (TB) are analogous to FRBs in all respects except they are not opened or exposed to the field environment at any time. The purpose of the TB is to determine if method analytes or other air-borne interferences are present in the environment to which actual samples were exposed, and perhaps contaminated or cross-contaminated samples by air-borne infusion through the sample septum. TBs are most typically sent in coolers with samples requesting VOC analysis.

#### 11.3.4 Field Duplicates

Field duplicates are two separate samples collected at the same time, under identical circumstances, and treated exactly the same throughout the field and laboratory procedures. Analysis of field duplicates give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures. Field duplicates are typically collected for an analysis at a frequency of 10% of the total samples taken for each parameter group.

### 11.4 LABORATORY QUALITY CONTROL SAMPLES

Laboratory control samples are those samples introduced into the train of environmental samples to function as monitors on the performance of the analytical method. All QC samples are prepared and processed through the complete analytical method. Stock solutions used to spike QC samples are prepared independently of stock standards used for calibration standards.

Method Blanks (negative controls) and laboratory control samples(positive controls) are used to monitor day-to-day performance of routine analytical methods. Internal standards are used to monitor the performance of the instrument. Surrogates, matrix spikes and sample duplicates are used to assess the effects of extraction efficiency, sample matrix and sampling, respectively, on the analytical data. The following descriptions indicate the types of QC samples that are included in an analytical or an extraction batch.

#### 11.4.1 Method Blank (MB)

##### 11.4.1.1 Matrix Composition and MB Definition

Method blanks are prepared from a matrix similar to the batch of associated samples (e.g., reagent grade water for water matrices, or ottowa sand, sodium sulfate or teflon chips for soil matrices) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

##### 11.4.1.2 Purpose



The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps. The method blank is processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure.

#### 11.4.1.3 Frequency

The method blank is prepared and analyzed at a minimum of 1 per preparation batch not to exceed 20 samples. In those instances for which no separate preparation method is used (example: volatiles in water) the batch is defined as the group of environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

#### 11.4.1.4 Evaluation Criteria

The goal for the method blank is to have no detectable contamination, however each method blank is critically evaluated as to the nature of interferences and the effect on the analysis of each sample within the batch. The source of contamination is investigated and measures are taken to minimize or eliminate the problem.

#### 11.4.1.5 Corrective Actions

If a blank exceeds the detectable limit for reporting of target analytes, then the analytical and extraction systems are evaluated and the appropriate corrective actions are taken. These may include such things as: cessation of further sample analysis to determine the source of contamination; reanalyses of all samples processed with that blank, or reporting the results with the appropriate footnotes. See section 13 for additional details.

### 11.4.2 Laboratory Control Sample (LCS)

#### 11.4.2.1 Matrix Composition and LCS Definition

Laboratory control samples are prepared from a sample matrix, similar to the batch of associated samples (e.g., reagent grade water for water matrices, or ottowa sand, sodium sulfate, or teflon chips for soil matrices), free from the analytes of interest and spiked with verified known amounts of analytes or material containing known or verified amounts of analytes.

Note: The matrix spike may be used in place of the LCS as long as the acceptance criteria are as stringent as for the LCS. Alternatively the LCS may be prepared from a matrix which contains known and verified concentrations of analytes or as a Certified Reference Material (CRM).

All analyte concentrations must be within the calibration range of the method.

#### 11.4.2.2 Spike Composition

The components spiked into the LCS are those specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components LCS samples are spiked as follows:

- a) For those components that interfere with an accurate assessment (i.e., spiking multi-component analytes together such as spiking simultaneously with technical chlordane, toxaphene and PCBs) the spike is chosen that represents the chemistries and elution patterns of the components to be reported.
- b) For those test methods that have extremely long lists of analytes, a representative number may be chosen. The selected analytes are chosen in a manner representing all reported analytes. The following criteria is used for determining the minimum number of analytes to be spiked. However, all target analytes are spiked and evaluated over a 2 year period.
  - 1) For methods that include 1-10 targets, all compounds are spiked and evaluated;
  - 2) For methods that included 11-20 targets, at least 10 or 80% of those compounds whichever is greater are spiked and evaluated;
  - 3) For methods that include more than 20 targets, a minimum of 16 compounds are spiked and evaluated.

#### 11.4.2.3 Purpose

The LCS is used to evaluate the performance of the total analytical

system, including all preparation and analysis steps.

#### 11.4.2.4 Frequency

An LCS is analyzed at a minimum of 1 per preparation batch. Exceptions for this frequency are for those methods/analytes for which no spiking solutions are available such as pH. In those instances for which no separate preparation method is used (Example: volatiles in water) the batch is defined as the group of environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

#### 11.4.2.5 Evaluation Criteria

The results of the individual batch LCS analytes are calculated in percent recovery and compared to established acceptance criteria. These calculations are documented on method worksheets and/or on LIMs generated summary LCS reports. LCS results are compared to acceptance criteria as follows:

- a) If LCS criteria is method specified, than the LCS is compared to the criteria as published in the mandated test method.
- b) If LCS criteria is not method specified, than the LCS is compared to laboratory derived criteria, and the method used to establish the limits are documented.
- c) Client specified criteria.

A LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be “out-of-control” are considered suspect and the samples are reprocessed and re-analyzed or the data reported with the appropriate footnote.

Marginal Exceedance Limits (Optional, if used with in-house limits)

If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control, therefore, corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits may be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit

(3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.

The number of allowable marginal exceedances is based on the number of target analytes evaluated in the LCS. If more analytes exceed the LCS control limits than are allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes such as Methods 8260B and 8270C. This evaluation does not apply to target analyte lists with fewer than 11 analytes.

The number of allowable marginal exceedances is as follows:

- 1) >90 analytes in LCS, 5 analytes allowed in ME;
- 2) 71-90 analytes in LCS, 4 analytes allowed in ME;
- 3) 51-70 analytes in LCS, 3 analytes allowed in ME;
- 4) 31-50 analytes in LCS, 2 analytes allowed in ME;
- 5) 11-30 analytes in LCS, 1 analytes allowed in ME;
- 6) <11 analytes in LCS, no analytes allowed in ME.

Marginal exceedances must be random. Analytes which repeatedly exceed the ME are not random and is an indication of a systemic problem. If this occurs, the source of the problem is located and corrective action is taken.

#### 11.4.2.6 Corrective Actions

Any affected samples associated with an out-of-control LCS is reprocessed for re-analysis or the results are reported with the appropriate footnotes.

### 11.4.3 Matrix Spike (MS)

#### 11.4.3.1 Matrix Composition and MS/MSD Definition

Matrix spike samples are prepared by adding a known mass of target analytes to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the matrix on a methods recovery efficiency.

Matrix spike duplicate (MSD) is a replicate aliquot of the same sample taken through the entire analytical procedure. The results from this analysis indicates the precision of the results for the specific

sample using the selected method.

Client samples used as the batch MS/MSD are randomly picked to ensure spiked samples are rotated through the client sample stream.

#### 11.4.3.2 Spike Composition

The components spiked into the MS are those specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components MS samples are spiked as follows:

- a) For those components that interfere with an accurate assessment (i.e., spiking multi-component analytes together such as spiking simultaneously with technical chlordane, toxaphene and PCBs) the spike is chosen that represents the chemistries and elution patterns of the components to be reported.
- b) For those test methods that have extremely long lists of analytes, a representative number may be chosen. The selected analytes are chosen in a manner representing all reported analytes. The following criteria is used for determining the minimum number of analytes to be spiked. However, all target analytes are spiked and evaluated over a 2 year period.
  - 1) For methods that include 1-10 targets, all compounds are spiked and evaluated;
  - 2) For methods that included 11-20 targets, at least 10 or 80% of those compounds whichever is greater are spiked and evaluated;
  - 3) For methods that include more than 20 targets, a minimum of 16 compounds are spiked and evaluated.

#### 11.4.3.3 Purpose

Matrix specific samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these controls is sample and matrix specific and is not normally used to determine the validity of the entire batch.

In other words, the MS is extracted and analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results.

#### 11.4.3.4 Frequency

Each preparatory batch, not to exceed 20 samples, must contain an associated MS and MSD using the collected project samples.

If adequate sample material is not available, then the lack of MS/MSDs are noted. Additionally, MS/MSD frequency may be project specified or test method specified.

#### 11.4.3.5 Evaluation Criteria

The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R), relative percent difference (RPD), or other appropriate statistical technique that allows comparison to establish acceptance criteria. These calculations are documented on method worksheets and on LIMs generated summary MS reports. MS/MSD results are compared to acceptance criteria as follows:

- a) If MS criteria is method specified, than the MS is compared to the criteria as published in the mandated test method.
- b) If MS criteria is not method specified, than the MS is compared to laboratory derived criteria, and the method used to establish the limits are documented.
- c) Client specified criteria.

The background concentration of the analytes in the sample matrix are first determined in a separate non-spiked aliquot, and the measured values in the spiked MS/MSDs are corrected for background concentrations.

#### 11.4.3.6 Corrective Actions

If matrix spike, spike duplicate results are outside the established criteria, for either accuracy or precision, corrective action is documented or the results are reported with the appropriate footnotes.

Often-times, MS recoveries exhibit matrix interference and are

outside the range of acceptability. In these cases the LCS is used to qualify analytical data; therefore, the recovery problem encountered with the spiked sample is judged to be matrix related, not system related, provided both LCS and MS were extracted in the same extraction batch.

#### 11.4.4 Matrix Duplicate (Sample Duplicate)

##### 11.4.4.1 Matrix Composition and Duplicate Definition

Laboratory duplicates are two sample aliquots taken from the same sample and analyzed separately with identical procedures. The composition is usually not known.

Client samples used as sample duplicates are randomly picked to ensure they are rotated through the client stream.

##### 11.4.4.2 Purpose

Sample duplicate is a replicate aliquot of the same sample taken through the entire analytical procedure. The results from this analysis indicates the precision of the results for the specific sample using the selected method. The sample duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.

##### 11.4.4.3 Frequency

If target analytes are known in a sample duplicate then it may be analyzed in place of an MSD. Duplicate analysis whether MSD or sample duplicates must be performed at a minimum frequency of once per preparatory batch not to exceed 20 samples.

Note: Since it is not typically known if a sample contains target analytes, preparing and analyzing a sample duplicate in place of a MSD is impractical and is discouraged. However, if a client or a project requires a sample duplicate, then both a sample duplicate and a MSD are prepared and analyzed.

##### 11.4.4.4 Evaluation Criteria

The results from a sample duplicate containing target analytes is primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). These calculations are documented on method worksheets and on

LIMs generated summary sample duplicate reports. Duplicate results are compared to acceptance criteria as follows:

- a) If sample duplicate criteria is method specified, than the duplicate data is compared to the criteria as published in the mandated test method.
- b) If sample duplicate criteria is not method specified, than the duplicate data is compared to laboratory derived criteria, and the method used to establish the limits are documented.
- c) Client specified criteria.

#### 11.4.4.5 Corrective Actions

If sample duplicate data results are outside of the established criteria, corrective actions are documented or the data is reported with the appropriate footnotes.

### 11.4.5 Surrogate Spikes

#### 11.4.5.1 Purpose

Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Surrogates are added prior to sample preparation/extraction and provide a measure of recovery for every sample matrix.

#### 11.4.5.2 Frequency

Surrogates are added to all samples, standards, and blanks for all appropriate test methods, except where the matrix precludes its use.

#### 11.4.5.3 Spike Composition

Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method. They are most often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of selected compounds.

#### 11.4.5.4 Evaluation Criteria

The results from surrogate spikes is used to monitor the effect of the matrix on the accuracy of the analysis. Surrogates are also used to



assess the recovery of the method and to detect any systematic extraction problems. Aberrations are reported in terms of percent recovery. These calculations are documented on method worksheets and on LIMs generated summary reports. Surrogate results are compared to acceptance criteria as follows:

- a) If surrogate criteria is method specified, than the surrogate recovery data is compared to the criteria as published in the mandated test method.
- b) If surrogate criteria is not method specified, than the surrogate recovery data is compared to laboratory derived criteria, and the method used to establish the limits are documented.
- c) Client specified criteria.

#### 11.4.5.5 Corrective Action

Surrogates outside the acceptance criteria are evaluated to determine if the aberration is indicating an effect on the individual sample results. The appropriate corrective action are generally guided by the data quality objectives or other site specific requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria are reported appropriately.

#### 11.4.6 System Monitoring Compounds

System Monitoring Compounds are added to every blank, sample, matrix spike, matrix spike duplicate and standard for volatile organic analysis, and are used to evaluate the performance of the entire analytical system. These compounds serve essentially the same purpose as the surrogates used in extractable analysis.

#### 11.4.7 Internal Standards

##### 11.4.7.1 Definition

A known amount of standard is added to an aliquot of sample to be analyzed as a reference for evaluating and controlling the precision and bias of the applied analytical method.

##### 11.4.7.2 Purpose

Internal standards are used in internal standard calibration methods to correct sample results affected by changing instrument sensitivity,

injection and purging losses, by measuring and comparing the relative responses of method analytes that are components of the same solutions.

11.4.7.3 Frequency

Internal standards are added to all samples, standards, and blanks for all appropriate test methods, except where the matrix precludes its use.

11.4.7.4 Spike Composition

Internal standards are chosen to represent the various chemistries of the target analytes in the method. They are most often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of selected compounds.

11.4.7.5 Evaluation Criteria

The results from internal standards are used to monitor the effect of the matrix on the accuracy of the analysis. Internal standard results are compared to acceptance criteria as published in the mandated test method.

11.4.7.6 Corrective Action

Internal standard results outside the acceptance criteria are evaluated to determine if the aberration is an effect of the individual sample matrix or an instrument problem. Typically samples which have exceeded the method criteria are re-analyzed. If the re-analyses indicates a matrix effect, then results reported from analyses with internal standard recoveries outside the acceptance criteria are footnoted appropriately.

## 11.5 SUMMARY OF METHOD QC PROCEDURES

The following tables are designed to list QC procedures for each analytical method used by Alpha. These tables include minimum frequencies, acceptance criteria, and possible corrective actions for method specified QC samples. These tables are an outline of the method specified procedure.

**SDWA  
ORGANIC METHODS**

**SUMMARY OF QC PROCEDURES  
FOR METHOD 524.2  
TABLE 11-1**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
524.2 VOCs	Laboratory Reagent Blank (LRB)	Once per analytical shift or one per 20 samples, before any samples are analyzed.	No analytes above MRL.	Determine the source of the contamination and eliminate the interference before proceeding.
	Laboratory Fortified Blank (LFB)*	Analyze LFB frequency of one LFB per 20 samples at a concentration of 2.0 - 5.0ug/L. This can be used interchangeably as a CCC.	Recovery should be $\pm$ 30% of the expected value.	Rerun the second LFB. If repeat failure occurs, locate and correct the source of the problem.
	Laboratory Fortified Matrix (LFM)	Not required unless matrix effects are suspected to be causing low surrogate/ internal standards recoveries.	Not specified	Flag sample results and report LFM data.
	System Monitoring Compounds (Surrogates)	Every sample, spiked sample, and method blank.	Recoveries should be $\pm$ 30% of the expected value.	Locate possible errors, and reanalyze. If reanalysis fails, report data as suspect.
	Internal Standards	Every sample, spiked sample, and method blank.	Absolute areas of quant ions should not vary more than 50% from the initial calibration or by 30% from the continuing calibration standard.	Locate possible errors, and reanalyze. If reanalysis fails, report data as suspect.
	Preservation and Storage Conditions	Every sample.	25 mg/40 mL ascorbic acid or 3mg/40mL sodium thiousulfate. Field preserve 2 drops 1:1 HCL pH<2.0, cool 4°C	If samples are not correctly preserved/stored footnote data accordingly.
	Holding Time	Every sample.	Analyze within 14 days if preserved. 24 hours if unpreserved.	If samples are analyzed outside the holding time footnote data accordingly.

Note: \*Since the LFB and the daily calibration check are made in the same way and since procedural standards are used, the LFB and daily calibration may be used interchangeably

Alpha Analytical, Inc.  
Section No.: 11.0  
Revision No.: 15.0  
Date: January, 2007  
Page: 15 of 60

**SDWA and CWA  
INORGANIC METHODS**

**SUMMARY OF QC PROCEDURES  
FOR METHOD 120.1/SM2510B**

**TABLE 11-2**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
120.1 SM2510B  Specific Conductance	Sample measurement	All samples	Measure samples in a temperature range of 23 - 27°C using ATC.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Sample Duplicate	Analyze a sample in duplicate at a frequency of 10% of the total samples.	No specified criteria for RPD. Will use statistically determined criteria.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Preservation and Storage Conditions	All samples	cool 4°C	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples	Method 120.1 -If samples are not analyzed within 24 hours samples <b>should</b> be filtered through a 0.45 $\mu$ m filter.  Method 9050A - Analyze samples within 28 days.  SOP - Since this is a should statement samples will be analyzed within 28 days.	If samples are analyzed outside the 28 day holding time footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
 FOR METHOD 150.1/SM4500H<sup>+</sup> B**

**TABLE 11-3**

<b>METHOD</b>	<b>PARAMETER</b>	<b>MINIMUM FREQUENCY</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CORRECTIVE ACTION</b>
150.1 SM4500H <sup>+</sup> B  pH	Sample Measurement	All samples	Measure sample until pH determinations are less than 0.1 pH units apart. Report the first determination.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Preservation and Storage Conditions	Not specified		
	Holding Time	All samples	As soon as possible. No other specified criteria.	

**SUMMARY OF QC PROCEDURES  
FOR METHOD 160.1/160.2/160.3 and  
SM2540B/SM2540C/SM2540D  
TABLE 11-4**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
TDS- 160.1/2540C  TSS- 160.2/2540D  TS- 160.3/2540B	Evaporative Dish Blank (In-house procedure)	Per gravimetric batch of sample up to 20 samples.	Dry samples to a constant weight if possible (weight loss is less than 0.5 mg). This entails multiple drying- cooling-weighing cycles for each determination.	Determine the source of the contamination and eliminate the interference before proceeding.
	Method Blank (In-house procedure)	Per gravimetric batch of sample up to 20 samples.	Dry samples to a constant weight if possible (weight loss is less than 0.5 mg). This entails multiple drying- cooling-weighing cycles for each determination.	Determine the source of the contamination and eliminate the interference before proceeding.
	Laboratory Control Sample (LCS) (In-house procedure)	Per gravimetric batch of sample up to 20 samples.	Arizona ADEQ requires the criteria to be statistically determined when no method criteria exists.	Repeat the test. If repeat fails report data as suspect.
	Sample Measurement	All samples	Dry samples to a constant weight if possible (weight loss is less than 0.5 mg). This entails multiple drying- cooling-weighing cycles for each determination.	Repeat the test. If repeat fails report data as suspect.
	Sample Duplicate	Analyze 10% of all samples in duplicate	Duplicate determinations should agree within 5% of their average weight as RPD	Repeat the test. If repeat fails report data as suspect.
	Preservation and Storage Conditions	All samples	Cool, 4°C	If samples are not correctly preserved/stored footnote data accordingly.
	Holding Time	All samples	7 days	If samples are analyzed outside the 7 day holding time footnote the data accordingly.



**SUMMARY OF QC PROCEDURES  
FOR METHOD 180.1**

**TABLE 11-5**

<b>METHOD</b>	<b>PARAMETER</b>	<b>MINIMUM FREQUENCY</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CORRECTIVE ACTION</b>
180.1 SM2130B  Turbidity	Method Blank (MB)	Not required. In-house criteria, one MB per batch of 20 samples.	Not required. In-house criteria, turbidity < the reporting limit of 0.10 NTU	Determine the source of the problem and eliminate before proceeding.
	Laboratory Control Sample (LCS)	Not required. Same as the ICV.	Not required. In-house criteria, same as the ICV $\pm$ 10% of the true value.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Sample Measurement	All samples	Measure sample until turbidity meter has stabilized. Report turbidity as ____ NTU.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Preservation and Storage Conditions	cool 4°C.		
	Holding Time	All samples	Not specified. (As soon as possible) Will use an in-house criteria of 14 days.	

**SUMMARY OF QC PROCEDURES  
FOR METHOD 200.8 USING  
DIGESTION PROCEDURES 200.2  
TABLE 11-6**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
200.8 Metals	Method Blank (MB)	One method blank with each batch of samples digested.	Less than the reporting limit.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Fortified Blank (LFB)	Analyze one LFB with each batch of 20 samples or less.	% Recovery 85-115%	Repeat the test. If repeat failure occurs, locate and correct the source of the problem. Re-analyze any samples during the out-of-control condition.
	Laboratory Fortified Matrix (LFM)	One LFM per 10 samples.	% Recovery 70-130%	If % recovery fails and the LFB is acceptable, problem is judged to be matrix related. Report data as suspect.
	Internal Standards (IS)	Add IS to all samples, spikes, standards and blanks. Recommended IS: Li, Sc, Y, In, Tb, Ho and Bi.	IS intensity must be between 60 -125% of level in the CB for method 200.8. A default of 60-120% criteria will be used in order to combine both methods 6020 and 200.8.	Dilute fresh aliquot of sample 2 times, add IS, and re-analyze. Repeat procedure until sample IS intensities fall within the prescribed window.
	Preservation and Storage Conditions	All samples and digests	Aqueous: pH $\leq$ 2 HNO <sub>3</sub> Solid and Hg Store at 4°C	If samples are not correctly preserved/stored footnote data accordingly.
	Holding Time	All samples and digests	6 months 28 days for mercury	If samples are analyzed outside the 6 month/28 day H.T. footnote the data.
	Sample Amount Required	All samples	Aqueous: 1 liter P/G Solid: 200grams P/G	

**SUMMARY OF QC PROCEDURES  
FOR METHOD 300.0**

**TABLE 11-7**

<b>METHOD</b>	<b>PARAMETER</b>	<b>MINIMUM FREQUENCY</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CORRECTIVE ACTION</b>
300.0 Anions	Laboratory Reagent Blank (LRB)	One method blank per extraction/analytical batch up to 20 samples or when new reagents are used.	No analytes above MDL. SOP, no analytes above the reporting limit.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Fortified Blank (LFB)	With each batch of samples or at a frequency of 5%.	Recovery should be $\pm 10\%$ of the expected value.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem. Re-analyze any samples during the out- of- control condition.
	Laboratory Fortified Matrix (LFM)	With each batch of samples or at a frequency of 10%.	Recovery should be $\pm 20\%$ of the expected value.	If % recovery fails and the LFB is acceptable, problem is judged to be matrix related. Report data as suspect.
	Preservaion and Storage Conditions	All samples and soil extracts.	Fl, Cl, Br no preservation all others cool to 4° C. pH<2, H2SO4 for Total-N	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and soil extracts.	Fl, Cl, Br, SO4 28 days. NO2, NO3 and PO4 48 hrs. Total -N 28 days.	If samples are received and/or analyzed outside of hold time footnote data accordingly.

**SUMMARY OF QC PROCEDURES  
 FOR METHOD 305.1/SM2310B**

**TABLE 11-8**

<b>METHOD</b>	<b>PARAMETER</b>	<b>MINIMUM FREQUENCY</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CORRECTIVE ACTION</b>
305.1 SM2310B  Acidity	Sample Measurement	All samples	Measure sample until pH determinations are less than 0.1 pH units apart. Report the first determination.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Preservaion and Storage Conditions	Not specified		
	Holding Time	All samples	As soon as possible. No other specified criteria.	

**SUMMARY OF QC PROCEDURES  
 FOR METHOD 310.1/SM2320B**

**TABLE 11-9**

<b>METHOD</b>	<b>PARAMETER</b>	<b>MINIMUM FREQUENCY</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CORRECTIVE ACTION</b>
310.1 SM2320B  Alkalinity	Sample Measurement	All samples	Measure sample until pH determinations are less than 0.1 pH units apart. Report the first determination.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Preservaion and Storage Conditions	Not specified		
	Holding Time	All samples	As soon as possible. No other specified criteria.	

**SUMMARY OF QC PROCEDURES  
FOR METHOD 314.0**

**TABLE 11-10**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
314.0 Perchlorate	Laboratory Reagent Blank (LRB)	One method blank per extraction/analytical batch up to 20 samples or when new reagents are used.	No analytes above ½ MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Fortified Blank (LFB)	With each batch of samples or at a frequency of 5%.	Recovery should be 85-115 % of the expected value.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem. Re-analyze any samples during the out- of- control condition.
	Laboratory Fortified Matrix (LFM)	With each batch of samples or at a frequency of 5%.	Recovery should be 80-120 % of the expected value.	If % recovery fails and the LFB is acceptable, problem is judged to be matrix related. Report data as suspect.
	Laboratory Fortified Matrix Duplicate (LFMD)	With each batch of samples or at a frequency of 5%.	Recovery should be 80-120 % of the expected value and RPD must be ≤15%.	If % recovery or % RPD fails and the LFB is acceptable, problem is judged to be matrix related. Report data as suspect.
	Preservaion and Storage Conditions	No preservation required. Room temperature.		
	Holding Time	All samples	28 days	If samples are received and/or analyzed outside of hold time footnote data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 330.5/SM4500Cl G**

**TABLE 11-11**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
330.5 SM4500Cl G  Free and Total Residual Chlorine	Method Blank (MB)	Not required by the method. One method blank with each batch of samples extracted.	No criteria specified. Less than the reporting limit.	Determine the source of the contamination and eliminate interference before proceeding.
	Laboratory Control Sample (LCS)	Not required by the method. Analyze one LCS with each batch of 20 samples or 5% frequency. LCS and ICV may be used interchangeably.	No criteria specified. Should pass the in-house criteria to be acceptable.	Repeat the test. If repeat fails report data as suspect.
	Matrix Spike (MS)	Not required by the method. Analyze one MS with each batch of 20 samples or 5% frequency.	No criteria specified. MS spiked at the same concentration as the LCS. Should pass the in-house criteria to be acceptable.	Repeat the test. If repeat fails report data as suspect.
	Preservation and Storage Conditions	All samples	Cool 4°C, 0.1 L amber, glass container, minimum head-space.	If samples are not correctly stored footnote data accordingly.
	Holding Time	All samples	Not specified, other than stating analysis must be started immediately.	Report date and time sample collected and analyzed.

**SUMMARY OF QC PROCEDURES  
 FOR METHOD 350.3/SM4500NH<sub>3</sub> D**

**TABLE 11-12**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
350.3 SM4500NH <sub>3</sub> D  Ammonia  351.4 SM4500Norg C  TKN	Method Blank (MB)	Not required. In-house criteria, one MB per batch of 20 samples.	Not required. In-house criteria, no ammonia or TKN above the reporting limit.	Determine the source of the problem and eliminate before proceeding.
	Laboratory Control Sample (LCS)	Not required. In-house criteria, one LCS per batch of 20 samples.	Not required. In-house criteria is used to evaluate sample data.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Matrix Spike (MS)	Not required. In-house criteria, MS with each batch of samples or at a frequency of 10%.	Not required. In-house criteria is used to evaluate the sample data.	If % recovery fails and the LFB is acceptable, problem is judged to be matrix related. Report sample data as suspect.
	Sample Measurement	All samples	Measure sample until pH/ISE meter has stabilized. Report ammonia as mg NH <sub>3</sub> -N/L	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Preservation and Storage Conditions	All samples	2 ml concentrated H <sub>2</sub> SO <sub>4</sub> per liter, cool 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples	28 days if preserved, 24 hours un-preserved.	If samples are received and/or analyzed outside of the holding time, footnote the data accordingly.



**SUMMARY OF QC PROCEDURES  
FOR METHOD 365.2/SM4500P E  
TABLE 11-13**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
365.2 SM4500P E  Total and Reactive Phosphorus	Method Blank (MB)	One method blank with each batch of samples extracted.	Less than the reporting limit.	Determine the source of the contamination and eliminate interference before proceeding.
	Laboratory Control Sample (LCS)	Not required by the method. Analyze one LCS with each batch of 20 samples or 5% frequency. LCS and ICV may be used interchangeably.	Both EPA method 365.2 and SM4500P E report some accuracy and precision data. LCS data is evaluated against a method accuracy window of 80-118% based on the tightest window of the two methods.	Repeat the test. If repeat fails report data as suspect.
	Matrix Spike (MS)	Not required by the method. Analyze one MS with each batch of 20 samples or 5% frequency.	MS spiked at the same concentration as the LCS. MS data is evaluated against a method accuracy window of 80 - 118% based on the tightest window of the two methods. No precision criteria is stated in the methods. A statistically derived in-house precision criteria is used.	Repeat the test. If repeat fails report data as suspect.
	Preservation and Storage Conditions	All samples	Total-P - Cool 4°C, pH <2 H <sub>2</sub> SO <sub>4</sub> Ortho-P - Cool 4°C	If samples are not correctly preserved/ stored footnote data accordingly.
	Holding Time	All samples	Not method specified criteria. Will use a in-house holding time of 28 days for preserved samples and 48 hours for non-preserved samples and ortho-P.	If samples are analyzed outside the holding time footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 376.2/SM4500S D**

**TABLE 11-14**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
376.2 SM4500S D  Sulfide	Method Blank (MB)	Not required by the method. One method blank with each batch of samples extracted.	No criteria specified. Less than the reporting limit.	Determine the source of the contamination and eliminate interference before proceeding.
	Laboratory Control Sample (LCS)	Not required by the method. Analyze one LCS with each batch of 20 samples or 5% frequency. LCS and ICV may be used interchangeably.	No criteria specified. In-house criteria of acceptance is $\pm 15\%$ of expected value.	Repeat the test. If repeat fails report data as suspect.
	Matrix Spike (MS)	Not required by the method. Analyze one MS with each batch of 20 samples or 5% frequency.	No criteria specified. The MS recoveries are compared to in-house generated acceptance criteria for data evaluation.	Repeat the test. If repeat fails report data as suspect.
	Preservation and Storage Conditions Total Sulfide	All samples	0.2ml of 2N zinc acetate, and 0.2ml of 6N NaOH per 100ml sample.  Cool 4°C, no head-space.	If samples are not correctly stored footnote data accordingly.
	Holding Time Total Sulfide	All samples	Not specified. If preserved will use a in-house holding time of 14 days.	If samples are analyzed outside the holding time footnote the data accordingly.
	Preservation and Storage Conditions Dissolved Sulfide	All samples	0.2ml of 6N NaOH per 100ml sample.  Cool 4°C, no head-space.	If samples are not correctly stored footnote data accordingly.
	Holding Time Dissolved Sulfide	All samples	Not specified. If non-preserved will use a in-house holding time of 48 hrs.	If samples are analyzed outside the holding time footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 410.4/SM5220D  
TABLE 11-15**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
410.4 SM5220D  COD	Method Blank (MB)	One method blank with each batch of samples extracted.	Less than the reporting limit.	Determine the source of the contamination and eliminate interference before proceeding.
	Laboratory Control Sample (LCS)	Not required by the method. Analyze one LCS with each batch of 20 samples or 5% frequency. LCS and ICV may be used interchangeably.	EPA method 410.4 does not specify any accuracy or precision criteria. However SM5220D uses a 72-128% of the true value to be acceptable	Repeat the test. If repeat fails report data as suspect.
	Matrix Spike (MS)	Not required by the method. Analyze one MS with each batch of 20 samples or 5% frequency.	MS spiked at the same concentration as the LCS. Use the same windows as specified in SM5220D, 72-128% of the true value to be acceptable.	Repeat the test. If repeat fails report data as suspect.
	Preservation and Storage Conditions	All samples	pH <2 H <sub>2</sub> SO <sub>4</sub> Cool 4°C	If samples are not correctly stored footnote data accordingly.
	Holding Time	All samples	Not method specified. Hach suggests a holding time of 28 days.	If samples are analyzed outside the 28 day holding time footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 415.1/SM5310C**

**TABLE 11-16**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
415.1 SM5310C  TOC	Method Blank (MB)	Not specified. One method blank per batch up to 20 samples or when new reagents are used.	Not specified. No analytes above the MRL.	Determine the source of the problem and eliminate before proceeding.
	Laboratory Control Sample (LCS)	Not specified. Analyze LCS at a frequency of 5% of sample load.	Compare results to Method 415.1 established limits of 74-125%.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Matrix Spike (MS)	Not specified. One MS at a frequency of 10% or one MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Compare results to laboratory established limits.	If % recovery fails and the LCS is acceptable, problem is judged to be matrix related. Report data as suspect.
	Preservation and Storage Conditions	All samples	pH < 2 H <sub>2</sub> SO <sub>4</sub> Cool to 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples	Not specified. 28 day laboratory defined.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD SM3500Cr D/SW7196A**

**TABLE 11-17**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SM3500Cr D SW7196A  Chromium (VI)	Method Blank (MB)	One method blank with each batch of samples extracted.	Less than the reporting limit.	Determine the source of the contamination and eliminate interference before proceeding.
	Laboratory Control Sample (LCS)	Analyze one LCS with each batch of 20 samples or 5% frequency.	No criteria specified. In-house criteria of acceptance is $\pm 15\%$ of expected value.	Repeat the test. If repeat fails report data as suspect.
	Matrix Spike (MS)	Analyze one MS with each batch of 20 samples or 5% frequency.	MS spiked at the same concentration as the LCS. No criteria specified by SM3500Cr D, but SW7196A specifies a criteria of $\pm 15\%$ of expected value and is used.	If MS/MSD yield recoveries less than 85%, sample should be re-tested to determine if low recoveries are due to the presence of residual reducing agent. See SOP for specifics.
	Preservation and Storage Conditions	All samples	Cool 4°C, acidify during sample digestion.	If samples are not correctly stored footnote data accordingly.
	Holding Time	All samples	24 hours	If samples are analyzed outside the 24 hour holding time footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD SM3500Fe D  
TABLE 11-18**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SM 3500Fe D  Ferrous Iron  Total and Dissolved Iron	Method Blank (MB)	Not required by the method. In-house criteria prepare one method blank with each batch of samples	Less than the reporting limit.	Determine the source of the contamination and eliminate interference before proceeding.
	Laboratory Control Sample (LCS)	Not required by the method. In-house criteria prepare and analyze one LCS with each batch of 20 samples or 5% frequency.	No criteria specified. In-house criteria of acceptance is $\pm 15\%$ of expected value.	Repeat the test. If repeat fails report data as suspect.
	Matrix Spike (MS)	Not required by the method. In-house criteria prepare and analyze one MS with each batch of 10 samples or MS/MSD at a 5% frequency.	No criteria specified. MS spiked at the same concentration as the LCS. In-house criteria of acceptance is $\pm 30\%$ of expected value and a precision criteria of $< 20\%$ RPD.	Repeat the test. If repeat fails report data as suspect.
	Preservation and Storage Conditions	All samples	Ferrous iron - field filter with a $0.45\mu\text{m}$ filter and acidify with HCL, pH $< 2.0$ , cool $4^{\circ}\text{C}$  Total iron - acidify nitric acid, pH $< 2.0$ , cool $4^{\circ}\text{C}$  Dissolved iron - filter with a $0.45\mu\text{m}$ filter and acidify with nitric acid, pH $< 2.0$ , cool $4^{\circ}\text{C}$	If samples are not correctly stored footnote data accordingly.
	Holding Time	All samples	Ferrous iron - 48-72 hours to color develop and 72 hours to analyze after color development (In-house criteria).  Total iron - 6 months	If samples are analyzed outside the holding time footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 1664A/SW9070A  
TABLE 11-19**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
1664A SW9070A  HEM and SGT-HEM (Oil and Grease)	Analytical Batch	A set of samples started through the extraction process in a 12 hour shift, to a maximum of 20 samples.		
	Evaporative Dish Blank (In-house procedure)	Per gravimetric batch up to 20 samples.	Dry samples to a constant weight (weight loss is less than 0.5 mg). Evaporative dish blank should be less than the reporting limit to be acceptable. This entails multiple drying-cooling-weighing cycles for each determination.	Determine the source of the contamination and eliminate before proceeding.
	Method Blank (MB)	One method blank with each batch of samples extracted.	Less than the reporting limit of 5 mg/L.	Determine the source of the contamination and eliminate interference before proceeding.
	Laboratory Control Sample (LCS)	Analyze one LCS with each batch of 20 samples or 5% frequency.	LCS should be spiked at a concentration of 40 mg/L. HEM window of 78-114% SGT-HEM window of 64-132%.	Repeat the test. If repeat fails report data as suspect.
	Quality Control Sample (QCS)	Not method required, but the method highly recommends to analyze regularly to verify the LCS spike solution.	Not specified. Use LCS criteria.	Repeat the test. If repeat fails verify LCS spike solution and re-analyze.
	Matrix Spike (MS)	Analyze one MS with each batch of 20 samples or 5% frequency. A MSD is recommended but not required.	MS spiked at the same concentration as the LCS. HEM window of 78-114% HEM precision < 18% SGT-HEM window of 64-132%. SGT-HEM precision < 34%	In order to be valid, all samples must be associated with a valid MS. If LCS is acceptable and MS failed, the batch has failed until MS is acceptable.
	Preservation and Storage Conditions	All samples	pH <1 1:1 HCl or 1:3 H <sub>2</sub> SO <sub>4</sub> Cool 4°C	If samples are not correctly preserved/stored footnote data accordingly.
	Holding Time	All samples	28 days	If samples are analyzed outside the 28 day holding time footnote the data accordingly.

Alpha Analytical, Inc.  
Section No.: 11.0  
Revision No.: 15.0  
Date: January, 2007  
Page: 34 of 60

**CWA**  
**ORGANIC METHODS**



**SUMMARY OF QC PROCEDURES  
FOR METHOD 608/SM6630C  
TABLE 11-20**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
608 SM6630C  Pesticides PCBs	Method Blank (MB)	One method blank with each batch of samples extracted or when new reagents are used.	No analytes above MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze one QC check standard with every 10 samples. Frequency may be reduced if MS recovery meets QC criteria.	Compare % recovery to method 608 Table-3 or optional QC acceptance criteria calculated for the specific concentration Table-4.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	One MS per 10 samples or 1 MS per month, which ever is more frequent.	Compare % recovery to method 608 Table-3 or optional QC acceptance criteria calculated for the specific concentration Table-4.	If % recovery fails then analyze a QC check standard. If QC check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Not required	See Method derived DQO.	
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C. If aldrin is to be analyzed and sample has residual chlorine, add 80 mg/L sodium thiousulfate., pH 5.0-9.0.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	7 day extract hold time for waters. 40 day hold time from extraction to analysis.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 624  
TABLE 11-21**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
624 VOCs	Method Blank (MB)	One method blank with each batch of samples analyzed.	No analytes above MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze one QC check standard at a frequency of 5%. Frequency may be reduced if MS recovery meets QC criteria.	Compare % recovery (P) to method 624 Table-5 or optional QC acceptance criteria calculated for the specific concentration Table-6.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	Analyze one MS at a frequency of 5% of sample load, or 1 MS per month, which ever is more frequent.	Compare % recovery (P) to method 624 Table-5 or optional QC acceptance criteria calculated for the specific concentration Table-6.	If % recovery fails then analyze a QC check standard. If QC check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogate to all samples, spiked samples, standards and blanks.	Not specified. Use laboratory derived criteria.	Check for errors and/or report data accordingly.
	Internal Standard (IS)	IS added to all samples, spiked samples, standards, etc.	Criteria not specified. Use 8260B criteria.	Check for errors. If reanalysis still exceeds limits then report data as suspect.
	Preservation and Storage Conditions	All samples	For chlorinated samples add 10 mg sodium thiousulfate per 40ml sample volume.. 2 drops 1:1 HCL pH < 2.0, required for aromatic compounds only. Cool to 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples	Analyze within 14 days of sample collection.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 625  
TABLE 11-22**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
625 SVOCs	Method Blank (MB)	One method blank with each batch of samples extracted or when new reagents are used.	No analytes above MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze one QC check standard at a frequency of 5%. Frequency may be reduced if MS recovery meets QC criteria.	Compare % recovery (P) to method 625 Table-6 or optional QC acceptance criteria calculated for the specific concentration Table-7.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	Analyze one MS at a frequency of 5% of sample load, or 1 MS per month, which ever is more frequent.	Compare % recovery (P) to method 625 Table-6 or optional QC acceptance criteria calculated for the specific concentration Table-7.	If % recovery fails then analyze a QC check standard. If QC check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogate to all samples, spiked samples, standards and blanks.	Not specified. Use laboratory derived criteria.	Check for errors and/or report data accordingly.
	Internal Standard (IS)	IS added to all samples, spiked samples, standards, etc.	Criteria not specified. Use 8270C criteria.	Check for errors. If reanalysis still exceeds limits then report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C. If chlorinated water source add 80 mg/L sodium thiousulfate.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	7 day extract hold time for waters. 40 day hold time from extraction to analysis.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 625 (PNAs by SIMs)  
TABLE 11-23**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
625 (PNAs by SIMs)	Method Blank (MB)	One method blank with each batch of samples extracted or when new reagents are used.	No analytes above MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze one QC check standard at a frequency of 5%. Frequency may be reduced if MS recovery meets QC criteria.	Compare % recovery (P) to method 625 Table-6 or optional QC acceptance criteria calculated for the specific concentration Table-7.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	Analyze one MS at a frequency of 5% of sample load, or 1 MS per month, which ever is more frequent.	Compare % recovery (P) to method 625 Table-6 or optional QC acceptance criteria calculated for the specific concentration Table-7.	If % recovery fails then analyze a QC check standard. If QC check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogate to all samples, spiked samples, standards and blanks.	Not specified. Use laboratory derived criteria.	Check for errors. If both surrogates fail rerun extract or flag data as suspect.
	Internal Standard (IS)	A minimum of 3 IS added to all samples, spiked samples, standards, and blanks.	Criteria not specified. Use 8270C criteria.	Check for errors. If more than 2 IS fail rerun extract. If extract still exceeds limits then reextract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C. If chlorinated water source add 80 mg/L sodium thiousulfate.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	7 day extract hold time for waters. 40 day hold time from extraction to analysis.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**RCRA**  
**ORGANIC METHODS**

**SUMMARY OF QC PROCEDURES  
FOR METHOD 8015B-DRO  
TABLE 11-24**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8015B  TPH-E	Method Blank (MB)	One method blank per extraction batch up to 20 samples or when new reagents are used.	No analytes above MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze LCS at a frequency of 5% of the sample load.	Compare results to laboratory established limits.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	One MS at a frequency of 10% or one MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Compare results to laboratory established limits.	If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogate to all samples, spiked samples, standards and blanks.	Compare results to laboratory established limits.	Check for errors. Rerun extract. If extract still exceeds limits then re-extract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	14 day extract hold time if water. 14 day extract hold time for soils. 40 day hold time from extraction to analysis.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD NWTPH-dx  
TABLE 11-25**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
NWTPH-dx  TPH-E	Method Blank (MB)	One method blank per extraction batch up to 20 samples or when new reagents are used.	Not specified. Note: will use 8015-DRO criteria.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Not required.	NA Note: will use 8015B-DRO criteria.	NA
	Matrix Spike (MS)	One MS/MSD or duplicate sample at a frequency of 10% or each batch of samples, which ever is more frequent.	Not specified. Note: will use 8015B-DRO criteria.	If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogates to all samples, spiked samples, standards and blanks.	Acceptance range of recovery is $\pm 50\%$ of the expected value.	Check for errors. Rerun extract. If extract still exceeds limits then re-extract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	7 day extract holding time for waters non-preserved. 14 day extract holding time if water is preserved to a pH<2. 14 day extract hold time for soils. 40 day hold time from extraction to analysis.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 8015AZ  
TABLE 11-26**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
8015AZ TPH-E	Method Blank (MB)	One method blank per extraction batch up to 20 samples or when new reagents are used.	Not specified. Note: will use 8015-DRO criteria.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze LCS at a frequency of 5% of the sample load.	Acceptance range of recovery is $\pm 30\%$ of the expected value.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	One MS at a frequency of 10% or one MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Acceptance range of recovery is $\pm 30\%$ of the expected value.	If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogates to all samples, spiked samples, standards and blanks.	Acceptance range of recovery is $\pm 30\%$ of the expected value.	Check for errors. Rerun extract. If extract still exceeds limits then re-extract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	Extract and analyze within 14 days of sampling.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.



**SUMMARY OF QC PROCEDURES  
FOR METHOD 8015B-GRO  
TABLE 11-27**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8015B  TPH-P	Method Blank (MB)	One method blank per extraction batch up to 20 samples or when new reagents are used.	No analytes above MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze LCS at a frequency of 5% of the sample load.	Compare results to laboratory established limits.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	One MS at a frequency of 10% or one MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Compare results to laboratory established limits.	If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogate to all samples, spiked samples, standards and blanks.	Compare results to laboratory established limits.	Check for errors. Rerun extract. If extract still exceeds limits then re-extract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	14 day hold time if water. 14 day to extract and analyze for soils.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD NWTPH-gx  
TABLE 11-28**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
NWTPH-dx  TPH-p	Method Blank (MB)	One method blank per extraction batch up to 20 samples or when new reagents are used.	Not specified. Note: will use 8015-GRO criteria.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Not required.	NA Note: will use 8015B-GRO criteria.	NA
	Matrix Spike (MS)	One MS/MSD or duplicate sample at a frequency of 10% or each batch of samples, which ever is more frequent.	Not specified. Note: will use 8015B-GRO criteria.	If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogates to all samples, spiked samples, standards and blanks.	Acceptance range of recovery is $\pm 50\%$ of the expected value.	Check for errors. Rerun extract. If extract still exceeds limits then re-extract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	7 day extract holding time for waters non-preserved. 14 day extract holding time if water is preserved to a pH<2. 14 day extract and analytical hold time for soils.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 8015AZ  
TABLE 11-29**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
8015AZ TPH-P	Method Blank (MB)	One method blank per extraction batch up to 20 samples or when new reagents are used.	Not specified. Note: will use 8015-GRO criteria.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze LCS at a frequency of 5% of the sample load.	Acceptance range of recovery is $\pm 30\%$ of the expected value.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	One MS at a frequency of 10% or one MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Acceptance range of recovery is $\pm 30\%$ of the expected value.	If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogates to all samples, spiked samples, standards and blanks.	Acceptance range of recovery is $\pm 30\%$ of the expected value.	Check for errors. Rerun extract. If extract still exceeds limits then re-extract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	Extract within 3 days and analyze within 14 days of sampling.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD SW8081A  
TABLE 11-30**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8081A Pesticides	Method Blank (MB)	One method blank per extraction batch up to 20 samples or when new reagents are used.	No analytes above MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze LCS at a frequency of 5% of the sample load at 20 x EQL.	Compare results to laboratory established limits.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	One MS at a frequency of 10% and/or an MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Compare results to laboratory established limits.	If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add TCMX/DCBP to all samples, spiked samples, standards and blanks.	Results of one of two surrogates must fall within laboratory established control limits.	Check for errors. Rerun extract. If extract still exceeds limits then re-extract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C. If chlorinated water source add 80 mg/L sodium thiousulfate, pH 5.0-9.0.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	7 day extract hold time for waters. 14 day extract hold time for soils. 40 day hold time from extraction to analysis.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD SW8082  
TABLE 11-31**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8082  PCBs	Method Blank (MB)	One method blank per extraction batch up to 20 samples or when new reagents are used.	No analytes above MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze LCS at a frequency of 5% of the sample load.	Compare results to laboratory established limits.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	One MS at a frequency of 10% and/or an MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Compare results to laboratory established limits.	If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add TCMX/DCBP to all samples, spiked samples, standards and blanks.	Results must fall within laboratory established control limits.	Check for errors. Rerun extract. If extract still exceeds limits then re-extract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C. Adjust water samples to a pH 5.0-9.0.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	7 day extract hold time for waters. 14 day extract hold time for soils/oils. 40 day hold time from extraction to analysis.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD SW8260B  
TABLE 11-32**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8260B  VOCs	Method Blank (MB)	One method blank per analytical batch up to 20 samples .	No analytes above MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze LCS at a frequency of 5% of the sample load.	Compare results to laboratory established limits.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	One MS at a frequency of 10% and/or an MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Compare results to laboratory established limits.	Repeat the test. If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogate to all samples, spiked samples, standards and blanks.	Compare results to laboratory established limits.	Check for errors and/or report data as suspect.
	Internal Standard (IS)	Add IS to all samples, spiked samples, standards, and method blanks.	RT must be within $\pm 30s$ from the RT of the mid-point IC standard and area must be within -50 to +200%.	Check for errors and re-analyze. If sample still exceeds limits then report data as suspect.
	Preservation and Storage Conditions	All samples	0.008% sodium thiousulfate for chlorinated samples 2 drops 1:1 HCL pH < 2.0, refrigeration at 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples	Analyze within 14 days of sample collection	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD SW8270C  
TABLE 11-33**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8270C SVOCs	Method Blank (MB)	One method blank per extraction batch up to 20 samples or when new reagents are used.	No analytes above RL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze LCS at a frequency of 5% of the sample load.	Compare results to laboratory established limits.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	One MS at a frequency of 10% and/or an MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Compare results to laboratory established limits.	If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogate to all samples, spiked samples, standards and blanks.	Compare results to laboratory established limits.	Check for errors. If more than 2 surrogates fail rerun extract. If extract still exceeds limits then re-extract or report data as suspect.
	Internal Standard (IS)	Add IS to ass samples, spiked samples, standards, and method blanks.	RT must be within $\pm 30s$ from the RT of the mid-point IC standard and area must be within 50-100%.	Check for errors. If more than 2 IS fail rerun the extract. If extract still exceeds limits then re-extract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C and protect from light.. If chlorinated water source add 80 mg/L sodium thiousulfate.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	7 day extract hold time for waters. 14 day extract hold time for soils. 40 day hold time from extraction to analysis.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD SW8270C (PNAs by SIMs)  
TABLE 11-34**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW8270C  (PNA by SIMs)	Method Blank (MB)	One method blank per extraction batch up to 20 samples or when new reagents are used.	No analytes above MRL.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze LCS at a frequency of 5% of the sample load.	Compare results to laboratory established limits.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.
	Matrix Spike (MS)	One MS at a frequency of 10% and/or an MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Compare results to laboratory established limits.	If % recovery fails then analyze a LCS. If LCS check standard is acceptable then problem is judged to be matrix related. Report data as suspect.
	Surrogate	Add surrogate to all samples, spiked samples, standards and blanks.	Compare results to laboratory established limits.	Check for errors. If both surrogates fail rerun extract or flag report data as suspect.
	Internal Standard (IS)	Add IS to all samples, spiked samples, standards, and method blanks.	RT must be within $\pm$ 30s from the RT of the mid-point IC standard and area must be within 50-100%.	Check for errors. If more than 2 IS fail rerun the extract. If extract still exceeds limits then re- extract or report data as suspect.
	Preservation and Storage Conditions	All samples and extracts.	Preserve by refrigeration at 4°C and protect from light.. If chlorinated water source add 80 mg/L sodium thiousulfate.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and extracts.	7 day extract hold time for waters. 14 day extract hold time for soils. 40 day hold time from extraction to analysis.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.



Alpha Analytical, Inc.  
Section No.: 11.0  
Revision No.: 15.0  
Date: January, 2007  
Page: 51 of 60

**RCRA**  
**INORGANIC METHODS**

**SUMMARY OF QC PROCEDURES  
FOR METHOD 6020 USING  
DIGESTION PROCEDURES 3015/3051  
TABLE 11-35**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
6020 Metals	Method Blank (MB)	One method blank with each batch of samples extracted .	No criteria specified.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	Analyze one LCS with each batch of 20 samples or less. An LCS/LCSD is preferred.	Evaluate against laboratory derived windows.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem. Re-analyze any samples during the out-of-control condition.
	Matrix Spike (MS)	MS/MSD per batch of samples.	% Recovery, % RPD not specified. Evaluate against laboratory derived windows	If % recovery fails and the LCS is acceptable, problem is judged to be matrix related. Report data as suspect.
	Post - Digestion Spike	Frequency not specified. Spike level based on the indigenous element concentration.	%R = 75-125%, otherwise dilute sample and reanalyze.	Dilute and reanalyze samples that are above the linear range or measure an alternate less abundant isotope.
	Sample Duplicate	Duplicate sample analysis after each 20 samples or analytical batch, whichever is more frequent.	RPD < 20% for analyte values greater than 100 times the IDL.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem . Re-analyze any samples during the out-of-control condition
	Dilution Test (DT)	Perform 1+4 dilution on sample containing analytes > 100 times the reagent blank. One DT per 20 samples.	Results of dilution should agree within $\pm 10\%$ of the original measurement.	Repeat the test. If repeat failure occurs, re-analyze the ICS and determine if corrections factors are appropriate. If not, correct the problem and reanalyze all affected samples.
	Internal Standards (IS)	Add IS to all samples, spikes, standards and blanks. Recommended IS Li-6, Sc, Tb, Re and Bi.	IS intensity must be between 30 -120% of level in the IC for method 6020. A default of 60-120% criteria will be used in order to combine both methods 6020 and 200.8.	Dilute fresh aliquot of sample 5 times, add IS, and re-analyze. Repeat procedure until sample IS intensities fall within the prescribed window.
	Preservation and Storage Conditions	All samples and digests	Aqueous: pH $\leq 2$ HNO <sub>3</sub> Solid and Hg Store at 4°C	If samples are not correctly preserved/stored footnote data accordingly.
	Holding Time	All samples and digests	6 months 28 days for mercury	If samples are analyzed outside the 6 month /28 day H.T. footnote the data.

**SUMMARY OF QC PROCEDURES  
 FOR METHOD 9040C/9045D  
 TABLE 11-36**

<b>METHOD</b>	<b>PARAMETER</b>	<b>MINIMUM FREQUENCY</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CORRECTIVE ACTION</b>
9040C 9045D  pH	Sample Measurement	All samples	Measure sample until pH determinations are less than 0.1 pH units apart. Report the first determination.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Preservation and Storage Conditions	Not specified		
	Holding Time	All samples	As soon as possible. No other specified criteria.	

**SUMMARY OF QC PROCEDURES  
FOR METHOD SW9050A  
TABLE 11-37**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW9050A  Specific Conductance	Sample measurement	All samples	Measure samples in a temperature range of 23 - 27°C using ATC.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Sample Duplicate	Analyze a sample in duplicate at a frequency of 10% of the total samples.	No specified criteria for RPD. Will use statistically determined criteria.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Preservation and Storage Conditions	All samples	cool 4°C	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples	Method 120.1 -If samples are not analyzed within 24 hours samples <b>should</b> be filtered through a 0.45 $\mu$ m filter.  Method 9050A - Analyze samples within 28 days.  SOP - Since this is a should statement samples will be analyzed within 28 days.	If samples are analyzed outside the 28 day holding time footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD 9056  
TABLE 11-38**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
9056 Anions	Method Blank (MB)	One method blank per extraction/analytical batch up to 20 samples or when new reagents are used.	No analytes above the reporting limit.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	With each batch of samples or at a frequency of 5%.	Not specified. Will use laboratory derived windows of acceptability.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem. Re-analyze any samples during the out- of- control condition.
	Matrix Spike (MS)	With each batch of samples or at a frequency of 5%.	Not specified Will use laboratory derived windows of acceptability.	If % recovery fails and the LCS is acceptable, problem is judged to be matrix related. Report data as suspect.
	Sample Duplicate	Analyze duplicate one in every 10 samples 10%.	Not specified.	
	Preservation and Storage Conditions	All samples and soil extracts	Preserve by refrigeration at 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples and soil extracts	SW-846 Chpt 2 Nitrate 48 hrs, Sulfate 28 days. Will use method 300.0 criteria	If samples are received and/or analyzed outside of holding time footnote data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD SW9060  
TABLE 11-39**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW9060  TOC	Method Blank (MB)	Not specified. One method blank per batch up to 20 samples or when new reagents are used.	Not specified. No analytes above the MRL.	Determine the source of the problem and eliminate before proceeding.
	Laboratory Control Sample (LCS)	Not specified. Analyze LCS at a frequency of 5% of sample load.	Compare results to Method 415.1 established limits of 74-125%.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem.
	Matrix Spike (MS)	Not specified. One MS at a frequency of 10% or one MS/MSD at a frequency of 5% or each batch of samples, which ever is more frequent.	Compare results to laboratory established limits.	If % recovery fails and the LCS is acceptable, problem is judged to be matrix related. Report data as suspect.
	Preservation and Storage Conditions	All samples	pH < 2 H <sub>2</sub> SO <sub>4</sub> Cool to 4°C.	If samples are not correctly preserved/stored footnote the data accordingly.
	Holding Time	All samples	Not specified. 28 day laboratory defined.	If samples are received and/or analyzed outside of holding time, footnote the data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR METHOD ASTM D2216  
TABLE 11-40**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ASTM D2216  Percent Moisture	Method Blank (MB)	Not required.		
	Laboratory Control Sample (LCS)	Not required.		
	Matrix Spike (MS)	Not required.		
	Sample Duplicate	Not required. Suggested to analyze at a 10% frequency	Not required. Suggest criteria of 15% RPD.	Repeat the test. If repeat failure occurs, report both values.
	Sample measurement	All samples	Measure sample until dry	Repeat the test. If repeat failure occurs, report both values.
	Preservation and Storage Conditions	cool, 3 to 30°C.		
	Holding Time	All samples	Not specified. (As soon as possible) Will use an in-house criteria of 28 days.	

Alpha Analytical, Inc.  
Section No.: 11.0  
Revision No.: 15.0  
Date: January, 2007  
Page: 58 of 60

**IN-HOUSE  
DEVELOPED  
METHODS**



**SUMMARY OF QC PROCEDURES  
FOR DISSOLVED GASES  
TABLE 11-41**

METHOD	PARAMETER	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
RSK-175  Dissolved Gases	Method Blank (MB)  Not specified by RSK-175	One method blank per preparatory/analytical batch up to 20 samples.	No analytes above the reporting limit.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)  Not specified by RSK-175	With each batch of samples or at a frequency of 5%.	Response should be $\pm 30\%$ of the expected value or compare results to laboratory established limits.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem. Re-analyze any samples during the out- of- control condition.
	Matrix Spike (MS)  Not specified by RSK-175	With each batch of samples or at a frequency of 10%.	Response should be $\pm 30\%$ of the expected value or compare results to laboratory established limits.	If % recovery fails and the LFB is acceptable, problem is judged to be matrix related. Report data as suspect.
	Preservation and Storage Conditions	All samples	pH < 2, cool to 4° C.	If samples are not correctly stored footnote the data accordingly.
	Holding Time	All samples	14 days preserved 7 days non-preserved	If samples are received and/or analyzed outside of hold time footnote data accordingly.

**SUMMARY OF QC PROCEDURES  
FOR ORGANIC ACIDS  
TABLE 11-42**

<b>METHOD</b>	<b>PARAMETER</b>	<b>MINIMUM FREQUENCY</b>	<b>ACCEPTANCE CRITERIA</b>	<b>CORRECTIVE ACTION</b>
Organic Acids	Method Blank (MB)	One method blank per extraction/analytical batch up to 20 samples or when new reagents are used.	No analytes above the reporting limit.	Determine the source of the contamination and eliminate interferences before proceeding.
	Laboratory Control Sample (LCS)	With each batch of samples or at a frequency of 5%.	Compare results to laboratory established limits.	Repeat the test. If repeat failure occurs, locate and correct the source of the problem. Re-analyze any samples during the out- of- control condition.
	Matrix Spike (MS)	With each batch of samples or at a frequency of 10%.	Compare results to laboratory established limits.	If % recovery fails and the LFB is acceptable, problem is judged to be matrix related. Report data as suspect.
	Preservation and Storage Conditions	All samples and soil extracts.	Cool to 4° C.	If samples are not correctly stored footnote the data accordingly.
	Holding Time	All samples and soil extracts.	28 days.	If samples are received and/or analyzed outside of hold time footnote data accordingly.

# **Section 12**

## **Data Reduction, Review and Storage**

## **12.0 DATA REDUCTION, REVIEW AND STORAGE**

### **12.1 CONTROL OF DATA**

The data reduction, review, reporting, and verification procedures described in this section are established to help ensure data are correctly reported. The primary focus of this multi-tiered peer review is as follows:

- Reported data is free from transcription and calculation errors;
- Quality control measures are reviewed, and evaluated before data is reported;
- Calibrations, manual calculations and manual integrations are correct; and
- Complete documentation is maintained.

Laboratory data reduction and review procedures are required to ensure that the overall objectives of analysis and reporting meet method or project specifications.

All laboratory activities and procedures are documented where possible to ensure maximum sample integrity. Data reduction, review, and reporting activities are included in all client data files and/or the Analytical Data Record Keeping System.

The overall Data Quality Objectives (DQOs) for the analytical activities can only be met if the data generated can be proven to be valid.

### **12.2 DATA REDUCTION**

Alpha Analytical maintains written Standard Operating Procedures (SOPs) governing all aspects of the data acquisition and reporting process. This record keeping procedure makes it possible to reanalyze data at a future date and is used in support of the experimental conclusions.

Data reduction is the process of converting measurements collected by analytical data systems into an expression of parameters and information from which conclusions about the sample or site can be made. This process must be performed with acceptable precision and accuracy. All calculations and data entries are reviewed to maintain the accuracy of this process.

Laboratory QC samples (such as matrix spikes, matrix spike duplicates, method blanks, surrogate spikes, and laboratory control samples) are analyzed and data generated to evaluate and assess the accuracy and precision of the data. Accuracy and precision data are used to determine if errors are produced through the analytical procedure.

In addition, the QC field samples (such as trip, equipment/rinse blanks, and field duplicate samples) are analyzed to determine any systematic or random errors introduced by field procedures.

#### 12.2.1 Compound Identification

When appropriate, identification and quantitation is based on internal standards, such as those specified in EPA Methods 524.2, 624, 625 and 200.8. When internal standards methods are not satisfactory, external standard methods are used to quantitate analytical data.

Chromatographic compound identification is routinely accomplished by comparison of its retention time to the retention times of standard reference chromatograms. If the retention time of an unknown compound in a sample corresponds, within the retention time (RT) limits which are established by standard calibrations, then identification is considered positive for the analytical column only.

#### 12.2.2 Analytical Column

For GC analysis of single response analytes, which use RT as the only source of compound identification, an alternative technique is employed to confirm peak identification. When possible, sample confirmation is determined by GC/MS. This may be limited by trying to confirm compounds at very low levels of detection where other GC detectors are more sensitive.

#### 12.2.3 Confirmation Column

The second technique that Alpha uses for positive peak identification is through the use of a confirmation column. Each confirmation column is selected with a different polarity than the analytical column; therefore, the retention time and the retention time order are different for each column. If the unknown compound is within the prescribed retention time windows on the confirmation column and compound quantitation is similar to the analytical column, then compound identification is considered positive.

Multi-response analytes (i.e., TPH and PCBs) do not require additional confirmation because they each have a unique chromatographic signature that positively identifies each of these types of compounds.

#### 12.2.4 Retention Time Windows

Retention time windows are calculated and used in chromatographic methods of analysis for qualitative identification of analytes. They are generally calculated from the analyses of standards over the course of an analytical sequence. The standard deviation of the retention time of multiple injections for each single component or

analyte in question is calculated. Typically plus or minus three times the standard deviation from the mean of the retention time of each standard is generally used to define the retention time window.

In those cases where the standard deviation is zero, the standard deviation of a similar close eluting compound is used to develop a valid retention time window. The procedure and calculation methods are given in SW 846, Method 8000B. This is not a hard and fast criteria, and often times compound and instrument behavior is more heavily used in the interpretation of chromatography and the establishment of retention time windows.

#### 12.2.5 Compound Quantitation

Analyte concentrations in the sample are calculated from the response of those analytes used in the calibration procedures.

If an internal standard calibration procedure is used, the concentration (C) in the sample is calculated using the Response Factor (RF) ratio to the appropriate internal standard. RF is calculated for each analyte and surrogate using the following equations:

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

where:  $A_s$  = Response of the analyte;  
 $A_{is}$  = Response of the internal standard;  
 $C_{is}$  = Concentration of the internal standard; and,  
 $C_s$  = Concentration of the analyte to be measured.

and:

$$C = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:  $I_s$  = Amount of internal standard added to each extract; and,  
 $V_o$  = Volume or weight extracted or purged.

If the external standard calibration procedure is used, calculate the peak area response by using the calibration curve or calibration factor determined from the initial calibration. The concentration (C) in the sample can then be calculated from the following equations:

$$C = \frac{(A)(V_t)}{(V_i)(V_s)},$$

where :      A = Calculated peak area response;  
                   $V_i$  = Volume of extract injected;  
                   $V_t$  = Volume of total extract; and,  
                   $V_s$  = volume or weight of sample extracted.

### 12.3 DATA VERIFICATION (Review)

Data validations are not performed by the laboratory. However, Alpha does provide data review to assure the analytical documentation is provided and the analysis is carried out in accordance with the data user's project specifications so that future third-party data validations can be performed. Alpha maintains records sufficient to recreate each extraction and analytical event during a sample's progress throughout the laboratory. Records are assembled and kept in the client file or by the QA officer.

#### 12.3.1 Data Integrity

The following is a list of the important records that are checked and maintained by Alpha:

- Chain-of-custody forms,
- Extraction and analytical logbooks,
- Instrument and Document Control Logbooks,
- Initial and continuing calibration records,
- Standard preparations logbooks,
- Internal standard results,
- Surrogate recovery charts,
- Method blank analyses,
- Spike and spike duplicate records and results,
- Initial method demonstrations, and
- Raw data including instrument printouts, chromatogram and quant reports.

Alpha maintains and uses written procedures for analytical QA/QC functions when appropriate.

#### 12.3.2 Data Review

Before releasing any analytical data, it is our policy to review and verify that the data has met all of the method criteria and is scientifically correct. Data reviews include the evaluation of information, as presented by the analyst or staff member, for accurate representation of the samples submitted. Analytical data are generally subjected to a four-person tiered review before it is released with each successive check performed by a different person.

#### 12.3.2.1 Analyst Review (Tier-1)

First the analyst will run, quantitate, and review analytes found in a particular sample or sample set. This includes reviewing and performing the following activities:

- Calibrations, tunes, blanks, and any other instrument quality control criteria were met and in-control during the analysis reported;
- Calculations of individual analytes and detection limits were met;
- Verify holding times or extraction times were met; and,
- Make notes or footnotes on the quantitation report if abnormalities occurred during the analysis or any other QA/QC problems associated with the sample.

#### 12.3.2.2 QC/Peer Review (Tier-2)

Samples pass through a two way QC review prior to final sample signature. The first half of this review includes a QC review of the calibration data and the other half of the review is a general QC review of all other QC batch data. Often times this review occurs simultaneously.

##### Calibration Review

Initial calibrations, initial calibration verifications, and daily calibration verifications are reviewed for correctness against the method criteria or other in-house established criteria prior to releasing the analytical data associated with that particular calibration.

##### Batch QC Review

Once the data has been worked-up by the analyst and the data has passed the first phase of the calibration review the data proceeds to the QC review department. The QC review person, then verifies that all dates, sample identification, reporting limits, reported analyte values, concentration units, header information, and footnotes or comments were transcribed accurately. All information on the final report that can be verified against the chain-of-custody is checked for errors and completeness.



#### 12.3.2.3 General Data Review (Tier-3)

When this step is completed, the client file, which includes chain-of-custody, chromatograms, quantitation reports, draft reports, and final reports, are reviewed by a third person. This person is typically another analyst, QC reviewer (QC upload person), or an assistant to the person signing the final report.

This person may review such things as:

- Chain-of-Custody records were analytically followed;
- Calculations and quantitation were performed correctly;
- Analytical holding times were met;
- Correct methods were used;
- Quality control criteria were met;
- Reporting limits were calculated properly;
- Correct concentration units were reported; and,
- Follow up and verify that any abnormalities which may have occurred during the analysis did not affect the final report. If abnormalities did occur, this person verifies the QA documentation and footnotes to determine if they are appropriate.

#### 12.3.2.4 Final Review/Data Signature (Tier-4)

The Laboratory Director, Laboratory Manager, or QA Officer will, at any time a problem is encountered, question the appropriate staff members and make determinations concerning the quality of the data.

Finally, the client file is checked and verified by the Laboratory Director or other designee who is signing final reports. This person spot checks activities associated with the log-in, tracking, extraction, sample analysis, and final reporting for technical and scientific soundness.

Many of the same activities reviewed by the Laboratory Director are also spot checked by the QA Officer, or designee as part of the internal quality assurance program. The QAO reviews approximately 10% of all data for technical completeness and accuracy. Once this has been accomplished, the final reports or summary reports are signed indicating they have been approved for release to the client.

### 12.3.3 Quality Control Data Reporting

The results for each analyte in the spiked QC samples are determined using the same calibration curve used for environmental samples in that batch. Values less than the reporting limit are reported as "Not Detected" (ND).

QC data is typically reported in terms of accuracy and precision, which translates into percent recovery of spiked compounds and relative percent difference of spiked analytes between duplicate analyses. This is the most commonly accepted practice for all analytical data. Alpha reports QC data in several different formats depending on the type of analysis that the client or regulatory agency requests. We frequently modify or change QC summary reports to satisfy the requirements of a particular SOW.

#### 12.3.4 Transcription Errors

It is a general policy of Alpha Analytical to minimize any errors associated with data reduction, validation and the reporting procedures including transcription errors.

If transcription errors are discovered during any part of the data review process, those errors challenged by the reviewer are taken to the person where the error was first propagated and discussed. During this discussion, the alleged transcription error will either proven to be a transcription error or not.

If the alleged transcription error is found to be a true error than the following are required:

##### 12.3.4.1 LIMS Transcription Errors

- LIMS generated COC are amended with the correct information. The mistake is annotated in the comments section, with the name of the person making the correction, and a new blue colored COC is issued to all affected personnel.
- LIMS generated extraction batch reports are amended with the correct information. The mistake is annotated and a mistake free batch report is generated. The new batch report is attached to the old batch report as a way to track the initial error.
- LIMS generated final reports are reissued with the correct information. The mistake is annotated as a footnote which clearly indicates the mistake, i.e., "This report replaces the report issued 1/10/07 due to a change of client ID"

##### 12.3.4.2 Non-LIMS Transcription Errors

- Instrument quantitation reports and any other non-LIMS generated data are amended (hand written) with the correct information, dated and initialed by the analyst.

## **12.4 DATA STORAGE**

### **12.4.1 Client File Data Assembly**

Following final data review, client files are organized in a manner to enhance future referencing of the data. Data files are organized by the DCO to facilitate easy data review and reconstruction of laboratory activities. Data files are generally organized in the following order:

- Computer generated chain of custody,
- Client chain of custody,
- Sub-contract lab chain of custody (if present),
- Work order information (if present),
- Sample receipt checklist,
- Sample receipt checklistfax confirmation,
- Final Alpha analytical reports,
- Alpha QA/QC data reports (if present),
- Final sub-contract lab reports (if present),
- Sub-contract lab QA/QC data reports (if present),
- Alpha invoice-always make sure than an invoice is present,
- Sub-contract lab invoices (if present),
- Raw data:
- Final report raw data. The initials of the analyst indicating final data should be indicated somewhere on all of the sheets and should be paper clipped together; and,
- Screen reports, re-runs, etc. this is indicated on the top sheet and should be stapled together,
- Air bill,
- Correspondence; and,
- Report fax or e-mailed confirmation.

If there were amendments made to the work order, put the amended COC on top of

the previous COCs. Put amended final reports on top of other lab reports.

#### 12.4.2 Archival Storage

##### Analytical Data

All analytical instrument data is permanently archived on the LIMS Server Disk as well as on Compact Disks (CD's) as a backup to hard copy data. The archival storage of data allows samples to be reevaluated at any time, providing proof of previous identifications, and the ability to search for other non-target compounds if GC-MS or ICP-MS data is being reevaluated.

##### Client File Data

All client data, which includes final reports, calculation sheets, chains-of-custody, records, chromatograms, quantitation reports, correspondence, and other associated data, are maintained by Alpha Analytical, Inc. for no less than ten years. This hard-copy data is stored on location at the laboratory for approximately one year. After this period of time, the data is scanned and stored electronically as a PDF file at an off-site location.

### 12.5 COLLECTIONS AND VALIDATIONS OF COMPUTERIZED DATA

In a computerized environment there are unique problems which must be considered in order to assure data integrity. Particularly unique data attributes, data archival, data security and soft-ware program integrity.

The computerized data collection and handling systems used by Alpha are designed such that data entries and data files are uniquely identified so that data can be reliably stored and retrieved without loss. Additionally each datum is supported by at least one hard copy output or laboratory notebook entry.

It is the responsibility of the LIMS Administrator to ensure the computerized data handling systems is used by trained personnel such that data corruption is prevented. Additionally, the LIMS Administrator is responsible for ensuring the integrity and accuracy of data handling and reduction programs are maintained to include the following:

- a) computer software developed by Alpha is documented in sufficient detail and is suitably validated as being adequate for use;
- b) established procedures are implemented for protecting the data; such as confidentiality of data entry, data storage, data transmission and data processing;
- c) computers are maintained to ensue proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of the test

data; and

- d) to establish and implement appropriate procedures for the maintenance of data security, including the prevention of unauthorized access to, and the unauthorized amendment of computer records.

Note: Commercial off-the-shelf software (e.g., word processing, database and statistical programs) used within their designated application range are considered sufficiently validated. However, analytical data acquisition software only needs to be validated initially, and again if modifications have been made to the software.

A complete description of Alpha's Software Quality Assurance Plan (SQAP) is found in Appendix F of the QAM. Specific items found in the SQAP are as follows:

- Computer Software/Hardware Operations,
- Data Collections and Storage,
- Data File Uploading,
- Electronic Diskette Deliverables (EDD's),
- MSAccess 97 DataBases,
- Data Archiving,
- PC Server Integrity and Software Validation,
- Sample Log-In, and
- Sample Prep Omega SOP.

# **Section 13**

## **Corrective Actions and Control of Nonconforming Environmental Testing Work**

## **13.0 CORRECTIVE ACTIONS AND CONTROL OF NONCONFORMING ENVIRONMENTAL TESTING WORK**

### **13.0.1 Summary**

Analytical methods require strict adherence to initial calibration, calibration verification, defined accuracy and precision limits, as well as a host of other critical QC elements with prescribed criteria. These QC elements are continuously monitored by the analyst, supervisors and QA Officer to ensure they are conforming to the prescribed method criteria.

In an imperfect world, occasionally, one or more of these QC elements do not comply with the method and/or project defined criteria and are identified as a nonconforming sample.

There are four primary areas where a sample may be noncompliant or nonconforming. They are generally described as:

- i. the sample is nonconforming due to a sampling or sample receiving issue;
- ii. the sample is nonconforming due to a sample matrix effect issue;
- iii. the sample is nonconforming due to a sample preparation issue; or
- iv. the sample is nonconforming due to a instrument issue.

In addition, samples may be nonconforming due to a combination of the potential issues or causes as described above.

### **13.0.2 Types of Corrective Actions**

The Laboratory Director, Laboratory Manager, analyst, and QA Officer may all be involved in corrective actions. Corrective actions are of two kinds:

1. Immediate, to correct or repair non-conforming equipment and systems. The need for this type of action is most frequently identified by the analyst as a result of calibration checks and QC sample analyses.
2. Long-term, to eliminate causes of non conformance. The need for such actions is normally identified by audits. Examples of this type of action include:
  - a) staff training in technical skills or in implementing the QA Program;
  - b) rescheduling of laboratory operations to ensure analysis within allowed holding time;
  - c) identifying vendors who supply reagents of sufficient purity; and
  - d) revision of the QA Program or replacement of personnel.

### 13.0.3 Procedural Outline

The following procedure is implemented when samples are determined to be noncompliant either by method or laboratory defined criteria, or by client requirements. The following outline describes this procedure:

- 1) the responsibilities and authorities for the management of nonconforming work is designated and actions (including halting of work and withholding test reports, as necessary) are defined and taken when nonconforming work is identified;
- 2) an evaluation of the significance of the nonconforming work is determined;
- 3) correction are taken, together with any decision about the acceptability of the nonconforming work;
- 4) where the data quality is impacted, the client is notified; and
- 5) the responsibility for authorizing the resumption of work is defined.

### 13.0.4 Conclusion

A comprehensive discussion which would include a compendium of situations, various scenarios to resolve these issues, ways to improve or minimize these issues, the corrective actions associated with these issues, and their respective follow up is beyond the scope of this discussion.

However, the following discussion, identifies the key elements of our policy and defines the procedures to implement these findings in a manner to correct and minimize nonconforming samples.

The following discussion is an extremely important aspect of environmental testing and is critical to improving our quality systems.

If the evaluation of nonconforming work indicates the issue could reoccur or a systemic problem is discovered, the corrective actions procedures described below is promptly followed.

## 13.1 CORRECTIVE ACTIONS

When, as a result of audits or QC sample analysis, analytical systems are shown to be unsatisfactory, a corrective action is implemented. Generally, quality control information is reviewed by several individuals. The responsibility for the initial assessment of a quality control measure lies with the analyst who identifies a problem with the sample or procedure and has access to the test results.



#### 13.1.1 Initial QC Assessment and Assignment of Responsibility and Authority

The individual responsible for operating the analytical instrument is responsible for performing the first initial data review (Tier-1 review).

Table 13-1 identifies typical quality control checks that may be required by the various test methods. The acceptance range or the source of the acceptance range is also identified in this table. This table does not include sample receiving criteria but focuses on sample matrix, sample preparation and instrument criteria.

If one or more of these key QC elements fail, then the sample is noncompliant. The root cause of the failure is investigated, and corrective actions are taken to resolve these issues.

The following QC samples are typically reviewed by the analyst:

- Calibration, which may includes initial, calibration verification, continuing calibration and tuning standards when specified;
- Blank, which may include, method or reagent blanks, equipment and trip blanks;
- Spikes, which may include matrix spikes, control spikes, duplicate spikes; and
- Surrogate and internal Standards when required.

#### 13.1.2 Secondary QC Assessment and Assignment of Responsibility and Authority

In addition to the analyst, the following controls are also reviewed by a second person (Tier-2 review) such as a QC assistant. This generally includes the following:

- Standards,
- Blanks,
- Spiked samples (matrix and blank), and
- Duplicates.

#### 13.1.3 Corrective actions are ultimately the responsibility of the individual in oversight authority (i.e., supervisor, Laboratory Director, Laboratory Manager, or QA Officer).

GENERAL ACCEPTANCE CRITERIA FOR QUALITY CONTROL CHECKS TABLE 13-1	
QC CHECKS	ACCEPTANCE CRITERIA
Calibrations	
Initial Calibration	Method Acceptance Criteria
Initial Calibration Verification	
Daily Calibration Verification	
Tuning Criteria	
Blanks	
Method Blanks	< Reporting Limit
Reagent Blanks	
Equipment Blanks	
Trip Blanks	
Spikes	
Matrix Spikes	Within Method or Laboratory Specified Accuracy Limits
Blank Spikes (Laboratory Control Samples)	
Duplicates	
Laboratory Duplicates	Within Method or Laboratory Specified Precision Acceptance Limits
Matrix Spike Duplicates	
Field Duplicates	
Others	
Surrogates	Within Method or Laboratory Specified Accuracy Limits
Internal Standards	Method Acceptance Criteria
Split Samples	Meets Precision Criteria

#### 13.1.4 Cause Analysis

Finding the source of a QC problem involves identifying probable sources of error and checking each source to determine if the protocols were properly followed.

Common sources of error and expected follow-up protocols are outlined in Tables 13-2, 13-3, 13-4, and 13-5.

#### 13.1.5 Selection and Implementation of Corrective Actions

When the source of a QC error has been identified, a corrective action is selected and implemented. The selection of a particular corrective action and its implementation is premised on the action(s) most likely to eliminate the problem and to prevent future recurrence.

Corrective actions are selected and implemented to match the degree that is appropriate to the magnitude and the risk of the problem.

#### 13.1.6 Documentation of Corrective Actions

13.1.6.1 If a quality control measure fails to meet acceptance criteria (i.e., immediate or short-term corrective actions), the QC measure and the procedures used to correct the problem is typically documented using analytical corrective action worksheets.

If systems changes are required resulting from corrective action investigations (i.e., long-term corrective actions), these changes and the implementation of these changes are also documented.

13.1.6.2 The selection and implementation of corrective actions are documented by the appropriate individuals and procedures. For example, documentation does not imply a formal memo but may be documented in the following fashion:

1. Corrective actions that are initiated during an on-going analytical run typically documented on the chromatogram as well as in the instrument analytical log book; and
2. Corrective actions that require input or intervention of more than one individual is typically documented in the related log books and records.

Both of these types of immediate or short-term corrective actions are documented using the instrument corrective action worksheets.

13.1.6.3 If an identified quality control problem affects more than one set of data or multiple projects, then the documentation associated with identifying and resolving the problem is cross-referenced to all associated projects.

### 13.1.7 Monitoring of Corrective Actions

For either immediate or long-term corrective actions, steps comprising a closed-loop corrective action system is employed as follows:

- Define the problem;
- Assign responsibility for investigating the problem;
- Investigate and determine the cause of the problem;
- Assign and accept responsibility for implementing the corrective action;
- Establish effectiveness of the corrective action and implement the corrections; and
- Monitor and verify that the corrective action has eliminated the problem.

Occurrence of problems, corrective actions employed, and verification that the problem has been eliminated are examined by the QA Officer and/or Laboratory Director.

Historical corrective action items are periodically reviewed during the Internal Data Quality Audits to monitor conformity and to identify long-term trends or recurring problems.

## 13.2 Additional Audits

### 13.2.1 Internal Audits

When the identification of nonconformance or departures cast doubts on method and/or program compliance, or with policies and procedures established by Alpha, additional audits of the effected areas of activity are initiated as soon as possible. A detailed list and description of our audit policies and procedures are found in section 14.

### 13.2.2 External Audits

The need to initiate corrective action may be the result of activities or audits from external sources. Sources include system audits, performance audits, split samples, blind QC samples, and findings from project or data validation review. A description of external audits policies and procedures are also found in section 14.

## 13.3 Technical Corrective Action

A detailed explanation of probable sources and expected corrective action for each QC measure is included herein and also found in section 9, which lists calibration criteria and section 11 which lists the batch QC criteria.

Section 9 and section 11 are method summary tables included in each analytical SOP and reprinted in those sections as a quick reference source. Corrective actions procedures used for assessing, evaluating, implementing, monitoring and documenting technical corrective actions, are identical to those protocols defined above.

Since many QC problems have unique solutions, the corrective action protocols are not limited to those listed below. Further assessment, based on an individual's experience and knowledge, may be warranted.

13.3.1 The first QC measure focuses on calibrations. Table 13-2 outlines some probable sources of QC problems and expected review procedures.

<b>SOURCES AND EXPECTED REVIEW PROCEDURES FOR CALIBRATIONS TABLE 13-2</b>	
<b>SOURCES</b>	<b>EXPECTED REVIEW PROCEDURES</b>
Improperly prepared or outdated standards	Review preparation logs for calculations or dilution errors and use of expired standards
Improperly prepared or outdated check standards	Verify check standard
Poor instrument response	Determine if preventative maintenance is required
Incorrect calculations	Review and verify all calculations
Contamination problems	See Table 13-3

The following is a description of expected corrective actions for calibrations:

- Recalculate calibration curve;
- Prepare fresh standards;
- Re-calibrate instrument;
- Perform preventative maintenance;
- Perform mass calibration and retune;
- Re-analyze all samples bracketing those from the previous acceptable QC check through the next acceptable QC check; and
- Take measures to eliminate sources of contamination.

13.3.2 The second QC measure focuses on blanks. While the goal is to have no detectable contaminants, each method blank is critically evaluated as to the nature of compound interferences and the effect on the analysis of each sample within the batch. The source of the contamination is investigated and measures are taken to minimize or eliminate the problem and affected samples are reprocessed or data is appropriately qualified. Table 13-3 outlines some probable sources of contamination and expected review procedures.

<b>SOURCES AND EXPECTED REVIEW PROCEDURES FOR BLANKS TABLE 13-3</b>	
SOURCES	EXPECTED REVIEW PROCEDURES
Contaminated reagents	Verify reagent sources
Environmental cross contamination (sample collection, and sample and analysis conditions)	Review sample handling and storage protocols
Improper or incomplete laboratory or field decontamination procedures	Review cleaning protocols
Contaminated sample containers	Verify source and storage conditions
Contaminated source water	Verify water source

The following is a description of expected corrective actions for blanks:

- Take measures to determine the source of the problem and eliminate future problems such as discarding reagents, revising protocols, performing preventative maintenance, or changing the use of interfering chemicals.
- Review data with respect to reported contamination levels. If the concentration of a target analyte is found in the blank at or above the reporting limit, AND the concentration in the blank is greater than 1/10th of the amount measured in the associated sample batch, then the associated samples should be re-extracted, the client notified, and resampled or the data footnoted.
- If sample concentrations are significantly higher than blanks, or contaminants are not detected in the sample, then report the sample data and flag the analytes by reporting the concentrations in the blank and if required, footnote the analytical data.

13.3.3 The third QC measure focuses on spikes, surrogate spikes, and internal standards. Table 13-4 outlines some probable sources of QC Problems and expected review procedures.

<b>SOURCES AND EXPECTED REVIEW PROCEDURES            FOR SPIKES, SURROGATE SPIKES, AND INTERNAL STANDARDS</b> <b>TABLE 13-4</b>	
SOURCES	EXPECTED REVIEW PROCEDURES
Error in calculation	Review and recheck all calculations
Error in preparing or using spike solution	Review all preparation logs and analytical logs for proper dilutions, solvents, etc.
Outdated standards	Review expiration dates and standard preparation logs
Contamination problems	See blanks above
Poor instrument response	Determine if preventative maintenance is required

The following is a description of expected corrective actions for spikes, surrogate spikes, and internal standards:

- Take measures to eliminate contamination problems, reprocess or reextract samples, and reanalyze as necessary;
- Perform required maintenance and revise PM schedules;
- Review preparations, calculations, and record keeping to determine if additional training or more stringent protocols are required; and,
- If the sample matrix produces consistently unacceptable recoveries, and none of the sources discussed above are responsible for the problem, then the sample should be re-extracted and re-analyzed. If re-analysis produces the same results, then the associated samples should be reported with a footnote to qualify the results.

13.3.4 The fourth QC measure focuses on duplicates. The Table 13-5 outlines some probable sources of QC problems and expected review procedures.

**SOURCES AND EXPECTED REVIEW PROCEDURES  
FOR DUPLICATES  
TABLE 13-5**

SOURCES	EXPECTED REVIEW PROCEDURES
Non-representative sample	Review sample collection procedures
Error in calculations	Recheck calculations
Contamination problems	See blanks above
Error in preparing or using spike solutions	Review all preparation logs and analytical logs for proper dilutions, solvents, etc.
Outdated standards	Review expiration dates and standard preparation logs
Poor instrument response	Determine if preventative maintenance is required

The following is a description of expected corrective actions for duplicates:

- Report data with a footnote and explanation;
- Revise sample collection or sample processing procedures to assure a representative sample;
- Take measures to eliminate contamination problems; and
- Reprocess and re-analyze the sample set.

#### **13.4 CLIENT NOTIFICATION OF NONCONFORMITY**

##### **13.4.1 Nonconformance Associated with Sample Receiving**

There are many potential levels of sample nonconformance which may be the result of the submitted sample or a laboratory error. In general, if a sample is received and noted that it contains a nonconforming item, then the client is notified of the samples nonconformance, see Appendix C. It is the responsibility of Alpha to identify and notify the client of the sample nonconformance. Subsequently once the client has been notified it is the responsibility of the client to determine the final status of that sample.

##### **13.4.2 Nonconformance Associated with Analytical Data**



13.4.2.1 It is Alpha's policy to report, to the extent possible, samples only if all quality control measures are acceptable.

13.4.2.2 Nonconformance associated with analytical data production is not always black and white, but is an issue of the significance of the nonconformity.

13.4.2.3 Critical QC Elements

Analytical data associated with the following items are not released as final data until the nonconforming item is technically reviewed and all associated data is re-analyzed with the analytical QC in control or the data appropriately footnoted. These critical items are:

- BFB or DFTPP;
- Initial calibration;
- Calibration verification;
- Internal standards;
- Method blank; and
- LCS recovery.

13.4.2.4 Reporting Nonconforming Data Results Associated with Critical QC Elements

If data are analyzed during a nonconforming situation, and it is impossible to re-analyze those affected samples; then it is the decision of the client, if possible, to decide the fate of that data.

If the data is to be released, then at a minimum all affected data are footnoted with a description of the failed QC parameters. See section 16, Data Reports, for a description of possible footnotes.

However, it is our first priority to identify the problem, correct the problem, and re-analyze all associated samples.

If the problem can not be corrected in a timely manner they are re-analyzed on a second instrument. It is one of the primary objectives of our laboratory to have redundant back-up instruments available for just this type of case. This situation is outlined in Section 10 "Contingency Plan."

13.4.2.5 Less Critical QC Elements

There are additional analytical QC items which are not as critical.

These items are generally attributed to the sample matrix which may cause analytical QC parameters to fail. These less critical QC elements are described as follows:

- The ending calibration fails for one or more compounds;
- One or more surrogates are not recovered within the QC limits of acceptability; and
- Matrix Spike recovery values are not within the QC limits of acceptability.

#### 13.4.2.6 Reporting Nonconforming Data Results Associated with Less Critical QC Elements

If these less critical QC criteria fails, then a decision is made regarding the scientific defensability, technical soundness, and end users data quality objectives, whether these samples will be re-analyzed or not.

Most of the time these problems are matrix related and re-analysis will confirm the nonconformity is matrix related by the repeated QC failure.

In either case this data is footnoted with a description of the failed QC parameters and the data is released. See section 16, Data Reports, for a description of possible footnotes.

# **Section 14**

## **System and Technical Audits**

## **14.0 SYSTEMS AND TECHNICAL AUDITS**

### **14.1 Definitions**

Audit - a systematic evaluation to determine the operational quality of a particular system or function.

System audits - verify compliance with our laboratories quality system (e.g., QA Manual, Vol I and II) based on the NELAC Quality System. Examples of these types of audits would include audits such as sample acceptance policies, and sample tracking procedures.

Technical audits - verify compliance with method-specific requirements, as well as operations related to the test method (e.g., sample preparation).

Note: NELAP makes a distinction between the two types of internal audits, however, in practice most internal audits are verifying the operational quality of the entire laboratory (i.e., both system and technical audit elements are intertwined).

### **14.2 INTERNAL AUDITS**

14.2.1 Internal audits are an independent check used to verify that laboratory policies and procedures continue to comply with the requirements of the quality system as defined by the NELAP standards and detailed in this QA Manual.

14.2.2 Internal audits are conducted to encourage staff members to adopt good quality assurance practices at all levels of the organization. Staff members are also encouraged to use these audits as an educational opportunity.

14.2.3 The internal audit program addresses all elements of the quality systems, including the environmental testing activities and is conducted on an annual basis.

14.2.4 It is the responsibility of the QA Officer to plan, organize and schedule internal audits. Audits not conducted by the QA Officer or carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited.

#### **14.2.5 Audit Findings and Corrective Actions**

When audit findings cast doubt on the effectiveness of the operations or validity of the test results, corrective actions are taken and clients are immediately notified if investigations indicate results were affected. All internal audit review finding and any corrective actions that may arise from them are documented accordingly.

If corrective actions were taken, follow-up audits are conducted to verify and record the implementation and effectiveness of the corrective action taken.

## **14.3 INTERNAL SYSTEMS AUDITS**

### **14.3.1 Quality Assurance Manual Audit**

The EPA requires that each laboratory must have a written and approved Quality Assurance Manual as well as individual analytical Standard Operating Procedures. The EPA and NELAP specifies the QA Manual must present in specific terms, the policies, organizations, objectives, functional activities, and specific QA/QC activities designed to achieve the data quality goals of specific methods or projects.

14.3.1.1 One of the primary duties and responsibilities of the QA Officer is to prepare, review, approve, revise and distribute the Quality Assurance Manual, Standard Operating Procedures and other technical documents. The QA manual, including Volume I and Volume II, is reviewed annually for accuracy and adequacy, and updated as appropriate.

14.3.1.2 This review takes account of reports from laboratory personnel, the outcome of recent internal audits, assessments of external auditing agencies, the results of inter-laboratory comparisons or proficiency tests, changes in the volume and type of work undertaken, feedback from clients, corrective actions, and other relevant factors.

14.3.1.3 All staff members are required to attend annual training sessions covering the material outlined in the chapters, or sections, contained in Volume I of the QA Manual. This required training is described in the training SOP, D.5. Internal system audits are typically conducted simultaneously with this training to encourage feedback and to address any shortcomings with the QA Manual and related procedures, see internal audit schedule.

14.3.1.4 The documentation that internal systems audits have been conducted are outlined in Table 14-1 and 14-2 and are available for external audit and reviews.

### **14.3.2 Staff Audits**

Training and auditing of the systems and procedures described in Volume II of the QA Manual are generally conducted with the staff members and documented using Table 14-3.

14.3.2.1 These audits are conducted with individual staff members covering the various aspects of their work or work related activities that have a bearing on the overall quality of the data produced. Internal staff audits consist of observations and verification to the adherence of approved practices and procedures. Deviations are noted and

discussed with the affected staff members.

- 14.3.2.2 Table 14-3 checklist is the same audit checklist as described in the RCRA Laboratory Audit Inspection Guidelines Document, RCRA Enforcement Division, USEPA OSWER 9950.4. This is a general audit checklist and is used as a foundation for this internal system audit which can be added to or subtracted from as needed.

## **14.4 INTERNAL TECHNICAL AUDITS**

- 14.4.1 Internal systems audits tend to focus on laboratory procedures rather than data quality. Therefore, these types of systems audits do not completely address and identify potential data quality issues and problems.

Data quality (technical) audits are conducted for the purpose of determining whether data of acceptable quality is being generated. There are three general types of technical audits:

- a) SOP document audits,
- b) Internal data quality audits or methods audits, and
- c) Records audits.

- 14.4.2 SOP document audits and methods audits are conducted annually for all analytical and extraction procedures. The documentation that internal technical audits have been conducted and audit schedules are outlined in Table 14-4 and 14-5. Documentation of specific audit topics are outlined in Tables 14-6 and 14-7.

### **14.4.3 Records and Data Audits**

An internal records audit is conducted to verify the QA record systems is functioning properly and is being adequately filed and maintained for protection and accessability. This records audit is also conducted to ensure data packages as generated by Alpha are adequate and fulfill the QA deliverables package as requested by the client or project. The QA Officer typically reviews records to verify the following general items:

- QA contents,
- QA format, and
- Completeness of data package in relation to the appropriate deliverable requirement.

## **14.5 GENERAL REVIEWS**

14.5.1 Internal audits and reviews are conducted not only on PE samples, and QA documents and procedures; but in addition, other quality control procedures are continuously reviewed to ensure quality data is being provided to clients. The following quality control procedures are regularly reviewed:

- Use of certified reference material or second source for QC sample analysis,
- Participation in proficiency-testing program,
- Replicate testing such as quarterly QC samples, and/or annual DOC studies,
- Re-testing of retained samples, and
- Calculation of results for different parameters of a sample (i.e. comparing gravimetrically determined TDS results with a cross check calculation using conductivity data).

#### **14.6 PERFORMANCE EVALUATION REVIEWS**

14.6.1 Alpha Analytical participates in a proficiency-testing program to help ensure our laboratory has adequate quality control procedures in place for monitoring the validity of environmental test methods and procedures.

14.6.2 It is a policy of Alpha and a NELAC requirement that laboratories participate in two single-blind, single-concentrate Proficiency Testing (PT) studies, per year for each field of testing to maintain accreditation.

14.6.3 Performance Evaluation (PE) samples are purchased and prepared as follows:

- 14.6.3.1 Performance evaluation samples are obtained from a laboratory accredited as a provider of Proficiency Testing (PT) samples, under the auspices of the National Institute of Standards and Technology (NIST), the USEPA and the National Voluntary Accreditation Program (NVLAP);
- 14.6.3.2 Aqueous samples are typically prepared in analyte-free water or prepared as whole volume samples by the PT provider, and soil samples are typically sent in a pre-spiked soil matrix.
- 14.6.3.3 These are blind PE samples; therefore, the analysts is not aware of the analyte concentration values in the PE audit sample. PE samples are inserted into the routine stream of laboratory sample analysis.

14.6.4 Performance Evaluation Findings and Corrective Actions

If Alpha's PE study results, determined by score of pass/fail criteria is deemed fully

acceptable, corrective actions are not required. However, if Alpha's performance is determined to be unacceptable on any individual fractions, then corrective actions are taken to locate the problem, identify the problem, implement corrective actions and to document these corrective actions. Once the problem has been identified and the corrective action implemented, a remedial or supplemental PE sample is analyzed for that fraction.

## **14.7 ANNUAL MANAGEMENT REVIEW**

14.7.1 The analytical quality systems and all other ancillary quality systems are reviewed annually by management to ensure its continuing suitability and effectiveness. If systems are found to be ineffective; then this review will discuss and introduce necessary changes or improvements to the quality systems and/or laboratory operations.

14.7.2 This review takes into account:

- a) the suitability of policies and procedures;
- b) reports from management and supervisory personnel;
- c) the outcome of recent internal audits;
- d) corrective and preventive actions;
- e) assessments by external bodies;
- f) the results of inter-laboratory comparisons or proficiency tests;
- g) changes in the volume and type of work undertaken;
- h) client feedback;
- i) complaints; and
- j) other relevant factors such as quality control activities, resources and staffing.

14.7.3 The documentation that management reviews have been conducted are outlined in Table 14-8.

## **14.8 EXTERNAL AUDITS**

14.8.1 External Systems Audit



External audits are conducted by individual clients or the various regulatory agencies (i.e., state certifying agencies, EPA regional agencies, etc.). This is an on-site inspection and review of our quality control system. Their visit to the laboratory is to review and discuss any shortcomings and discrepancies in an actual sample walk through. Audits performed by an external Quality Assurance Officer normally will address all applicable elements of this QA Plan or contract requirements as it pertains to their QAPP or SOW. It is Alpha's policy to comply fully with audits conducted by certifying agencies, regulatory agencies and clients.

#### 14.8.2 External Performance Audits

External performance audits are Performance Evaluation (PE) samples submitted and analyzed as unknown sample concentrates as double-blind samples. These PE samples are typically obtained from proficiency test (PT) providers and submitted as samples within the clients normal sample stream.

### 14.9 PREVENTIVE ACTION

14.9.1 Preventive action is a pro-active process used to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

Section 13 is a description of corrective actions and is the reactionary process after a problem has been identified. This section, Section 14 describes the audit and review process which, by its very nature, is a preventive action procedure.

14.9.2 Once potential sources of nonconformance, either technical or concerning the quality system, is identified, preventive action is taken.

If preventive action is required, a plan of action is developed, implemented and monitored to reduce the likelihood of the occurrence of such nonconformances and to take advantage of the opportunities for improvement.

The procedure as described in section 13, Corrective Action, is the same basic procedure as used for preventive actions.

**Internal System Audit  
Document Review  
QA Manual Volume I  
Table 14-1**

Section	Audit Schedule (Tentative)	2007		2007 Additional Review (if required)	
		Review		Review	
		Date	Revision No.	Date	Revision No.
Section 1	January (week 1)				
Section 2	January (week 1)				
Section 3	January (week 1)				
Section 4	January (week 3)				
Section 5	January (week 3)				
Section 6	February (week 1)				
Section 7	February (week 1)				
Section 8	February (week 3)				
Section 9	February (week 3)				
Section 10	March (week 1)				
Section 11	March (week 1)				
Section 12	March (week 3)				
Section 13	March (week 3)				
Section 14	April (week 1)				
Section 15	April (week 1)				
Section 16	April (week 1)				
Section 17	April (week 1)				
Section 18	April (week1)				

Comments:

**Internal System Audit  
Document Review  
QA Manual Volume II  
Table 14-2**

[illegible]

Comments:

**Internal System Audit Checklist**  
**(NELAP check list may also be used)**  
**Table 14-3**

Date: \_\_\_\_\_

Employee: \_\_\_\_\_

Auditor: \_\_\_\_\_

REVIEW SAMPLE HANDLING PROCEDURES	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
1) Is an individual appointed to log in incoming samples?				
2) Does this individual know the sampling requirements (i.e. type of container, preservation, storage container, etc.) for each analysis or have that material available?				
3) If no individual is appointed, are the individuals logging in samples aware of the sampling requirements for each analysis?				
4) Does this individual know the storage process for storage of incoming samples?				
5) Is a sample label affixed to each container?				
6) Do the sample labels contain complete information in order to identify the sample and ensure that it has been sampled in the correct manner?				
7) Are samples collected in the type of container specified for each analysis?				
8) Are samples preserved as required and/or cooled to 4°C?				
9) Do samples which are shipped to the laboratory arrive at the correct temperature to ensure that the sample has remained in a preserved state?				
10) Are volatile samples received with no air bubbles?				
11) Are trip blank, field blanks, and field duplicates used as required?				
12) If so are they identified as such?				
13) If used, are spiked samples identified?				
14) Is a chain-of-custody filled out and kept on file?				
15) Does the information on the sample label and chain-of-custody match?				
16) Are laboratory numbers assigned to all incoming samples?				
17) Does the laboratory maintain a master schedule sheet or logbook of all samples being analyzed, indexed by laboratory number, client, date of arrival, and analysis to be performed?				

Notes:

**Internal System Audit Checklist**  
**(NELAP check list may also be used)**  
**Table 14-3**

Date: \_\_\_\_\_

Employee: \_\_\_\_\_

Auditor: \_\_\_\_\_

REVIEW SAMPLE HANDLING PROCEDURES	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
18) Is the laboratory number written on the sample label, the master schedule sheet, and any document related to that sample?				
19) Are completed sample analysis request sheets available for each sample?				
20) Does each sample have a separate file for each analysis or group of analyses to be performed?				
21) After analyses have been completed, are all data collated in a master file with all appropriate summary sheets for each analysis?				
22) Are samples analyzed within the correct amount of time?				
23) Are sample maintained at the correct temperature until time of analysis?				
24) Are adequate facilities provided for storage of incoming samples, including cold storage?				
25) Are volatile samples stored separately from non or semi-volatile samples?				
26) Is the temperature of cold storage recorded daily?				
27) Are temperature excursions noted and are appropriate actions taken when required?				
28) If used, are sample containers cleaned properly?				
29) Are the possession and handling of samples traceable from the time and date of collection to time and date of analysis and reporting?				
30) Are written SOPs available for receipt and storage of samples?				
31) Are SOPs and procedures being followed?				
32) Are sampling letters being sent out to all SDWA clients?				

Notes:

**Internal System Audit Checklist**  
**(NELAP check list may also be used)**  
**Table 14-3**

Date: \_\_\_\_\_

Employee: \_\_\_\_\_

Auditor: \_\_\_\_\_

REVIEW CALIBRATION PROCEDURES	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
1) Are initial and daily instrument calibration procedures specified in the QA/QC program?				
2) Are these procedures used in daily laboratory analysis as specified in the QA/QC program?				
3) Are standard curves and check samples used in covering the analytical range of interest for calibrating analytical instruments to ensure that calibration accurately encompass the range of environmental samples?				
4) Does the analyst stop analysis when an analytical system is deemed "out of control" and implement corrective procedures?				
5) Are corrective action procedures clearly defined and documented?				
6) Is the analyst initiating corrective action procedures when necessary?				
7) Is there a prompt notification of errors in reporting data or "loss" of a sample and a prompt request for resampling from the same point?				
8) Are MVDDs calibrated to ensure the amount marked coincides with the amount delivered?				
9) Is glassware cleaned correctly after each use to ensure that there will be no contamination with the next use?				
10) Is the analytical balance located away from drafts and areas subject to rapid temperature change?				
11) Has the balance been checked within one year by a certified technician?				

Notes:

**Internal System Audit Checklist**  
**(NELAP check list may also be used)**  
**Table 14-3**

Date: \_\_\_\_\_

Employee: \_\_\_\_\_

Auditor: \_\_\_\_\_

REVIEW OF QUALITY CONTROL PROGRAM	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
1) Is one matrix spike used for every analytical batch or every twenty samples whichever is most frequent?				
2) Are accuracy results of matrix spikes and surrogates measured for each method to indicate the closeness of an individual measurement?				
3) Are precision results of sample replicates measured for each method to indicate reproducibility among individual measurements?				
4) Are matrix spikes and surrogates analyzed to establish that the analytical measurement system is functioning properly and with the desired sensitivity?				
5) Are these precision and accuracy results organized in the form of quality control charts?				
6) Are LCS used one per analytical batch or every 20 samples, whichever is more frequent?				
7) Is one blank used per analytical batch or every 20 samples, whichever is more frequent?				
8) Is the analytical system calibrated each day according to the requirements of the method?				
9) Is a surrogate spikes added to appropriate method blank, standards, samples, and QA samples?				
10) Are trip blanks, field blanks, and laboratory blanks used as needed, to ensure the water contains no contaminants which may interfere with the analysis?				
11) Are laboratory method blanks extracted and analyzed with the same procedures used to extract and analyze samples?				

Notes:

**Internal System Audit Checklist**  
**(NELAP check list may also be used)**  
**Table 14-3**

Date: \_\_\_\_\_

Employee: \_\_\_\_\_

Auditor: \_\_\_\_\_

<b>REVIEW PROCEDURES FOR DATA HANDLING REPORTING AND RECORD KEEPING</b>	<b>Y/N/NA</b>	<b>Corrective Action Due Date</b>	<b>Corrective Action Completion Date</b>	<b>Comments</b>
1) Is the criteria for the data review documented and are limits on operational parameters, calibration data, special checks, statistical tests, and manual checks included?				
2) Are there procedures for data handling and reporting, including the recording of data on standard forms and in laboratory notebooks?				
3) Are sample calculations available for inspection?				
4) Are bound notebooks used for laboratory activities?				
5) Is raw data being archived and documented properly?				
6) Are records readily available for review?				
7) Are records maintained for a minimum of ten years?				
8) Is the instrument maintenance logbook available for review?				
9) Are analytical methods available to the analysts?				
10) Are analytical SOPs available and maintained as to revisions?				
11) Are IDCs and MDL studies available?				
12) Are control charts available?				
13) Are logbooks signed on each page?				
14) Are corrective actions documented by the analysts?				
15) Are the analysts keeping their training logs updated?				

Notes:



**Internal Technical Audit  
Document Review  
QA Manual Volume III  
Table 14-4**

SOP	Method	Comments	Audit Schedule (Tentative)	2007		2007 Additional Review (if required)	
				Review		Review	
				Date	Revision No.	Date	Revision No.
E.20	524.2		May (week 1)				
E.30	Pesticides		May (week 2)				
E.31	PCB		May (week 3)				
E.33	VOCs		May (week 4)				
E.34	SVOCs		June (week 1)				
E.35	PNA (SIMs)		June (week 2)				
E.36	Alcohols (SIMs)		June (week 3)				
E.37	TPH-DRO		June (week 4)				
E.38	TPH-GRO		July (week 1)				
E.50	TCLP		July (week 2)				
E.51	SPLP		July (week 3)				
E.52	STLC		July (week 4)				
E.55	ASE (3545)		July (week 4)				
E.56	Liq-Liq (3510)		July (week 4)				

Comments:

**Internal Technical Audit  
Document Review  
QA Manual Volume III  
Table 14-5**

SOP	Method	Comments	Audit Schedule (Tentative)	2007		2007 Additional Review (if required)	
				Review		Review	
				Date	Revision No.	Date	Revision No.
E.60	Metals		August (week 1)				
E.64	Organic Acids		August (week 3)				
E.65	Anions		August (week 4)				
E.66	Perchlorate		August (week 4)				
E.67	TOC		August (week 3)				
E.70	3015		August (week 2)				
E.71	3051		August (week 2)				
E.72	200.2		August (week 2)				
E.75	Conductivity		September (week 1)				
E.76	pH		September (week 1)				
E.77	Ammonia/TKN		September (week 2)				
E.78	Turbidity		September (week 2)				
E.80	TDS/TSS/TS		September (week 3)				
E.81	% Moisture		September (week 3)				
E.82	1664A		September (week 4)				
E.85	Alkalinity/Acidity		September (week 4)				
E.90	Chromium VI		October (week 1)				
E.92	Sulfide		October (week 1)				
E.93	Chlorine		October (week 2)				
E.94	COD		October (week 2)				
E.95	Total-P		October (week 3)				
E.96	Fe		October (week 3)				

Comments:

**Internal Data Quality Audit  
Checklist  
Table 14-6**

<b>Item</b>	<b>Date Completed</b>	<b>Comments</b>	<b>Initial</b>
1) Method review			
2) CAR external auditors review			
3) SOP review			
4) Section 9 Calibration review			
5) Section 11 QC review			
6) IDC review			
7) MDL review			
8) Standards review			
9) Data Package review			
10) PE review			
11) Data Quality Audit review			
12) Analyst training			
13) Report review			
14) DQO review			
15) QC Summary Report review			
16) Bench records review			
17)			
18)			

Comments:

**Internal Data Quality Audit**  
**IDC/MDL Review**  
**Table E.14-7A**

Date: \_\_\_\_\_  
 Analyst: \_\_\_\_\_  
 Peer: \_\_\_\_\_

Method: \_\_\_\_\_  
 Instrument: \_\_\_\_\_

Matrix: \_\_\_\_\_

DESCRIPTION	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
<b>1) Initial Demonstration of Capability</b>				
a) Was an IDC performed for this method by this analyst?				
b) Was each target analyte included at the required concentrations?				
c) Was each target analyte evaluated for spike value, found value, average percent recovery, standard deviation and percent relative standard deviation?				
d) Does the average percent recovery and standard deviation of each analyte meet the method specified acceptance range?				
e) Is the IDC documented on the standard form with the correct information and required signatures?				
<b>2) Method Detection Limits</b>				
a) Was an MDL study performed for this method by this analyst?				
b) Was each target analyte and data point included?				
c) Was the MDL study generated using the preparatory and cleanup procedures routinely used on samples?				
d) Were any data points deleted from the MDL study?				
e) Was each target analyte evaluated for spike value, found value, average percent recovery, standard deviation and calculated MDL?				
f) Is calculated MDL higher than the spike concentration, and if so was the study repeated at a higher concentration?				
g) Is the calculated MDL greater than 1/10 of the spike concentration, and if not was the study repeated at a lower concentration.				
h) Was a MDL verification check standard analyzed at approximately 2 times the MDL? And was the check standard detected or have a signal to noise ratio greater than 3?				
i) Is the calculated MDL higher than the LOQ, and if so was the study repeated at a lower level or the LOQ elevated above 3 times the MDL?				
j) Was a LOQ verification check standard analyzed at 1-2 times the LOQ and if so were the recoveries within the LCS windows of acceptability?				

**Internal Data Quality Audit  
Calibration Review  
Table E.14-7B**

Date: \_\_\_\_\_  
Analyst: \_\_\_\_\_  
Peer: \_\_\_\_\_

Method: \_\_\_\_\_  
Instrument: \_\_\_\_\_  
Matrix: \_\_\_\_\_

DESCRIPTION	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
<b>3) Initial Calibration Data</b>				
a) Was an initial calibration performed for each target analyte?				
b) Were all target analytes included in the calibration standard?				
c) Were the concentration values used for each analyte in the calibration table or curve appropriate for the method and do the concentration values in the method table match the actual standard concentrations?				
d) Are analytes reported by GC/MS assigned the right characteristic ions or isotopes by ICP/MS?				
e) Are GC or IC retention time windows correctly calculated?				
f) Is a second GC confirmation column or alternate detector used for analyte confirmation?				
g) Are multi response analytes being evaluated for pattern match?				
h) Was peak integration performed properly with no indication of improper data manipulation?				
i) Were all manual integrations properly documented with a before and after chromatogram and appropriate explanation?				
j) Does the low level standard have a signal to noise response greater than 3?				
k) Are integration routines used on calibration points acceptable?				
l) Do calibration points either high or low need to be deleted from the IC?				
m) Does IC meet method criteria for the chosen mathematical model?				
n) Are the same instrument conditions used for IC the same used for production analysis?				

Notes:

**Internal Data Quality Audit  
Calibration Review  
Table E.14-7B  
(Continued)**

Date: \_\_\_\_\_  
Analyst: \_\_\_\_\_  
Peer: \_\_\_\_\_

Method: \_\_\_\_\_  
Instrument: \_\_\_\_\_  
Matrix: \_\_\_\_\_

DESCRIPTION	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
<b>4) Instrument Performance Check (IPC)</b>				
a) Was the appropriate IPC analyzed? 1) BFB or DFTPP tune standard for GC/MS 2) Metals tune standard for ICP/MS 3) GC or IC method specific IPC	1) 2) 3)			
b) Was the GC/MS or ICP/MS tune standard analyzed and checked each 12 hours of sample analysis or method required frequency?				
c) For GC/MS are ion abundance and relative ion abundance within the method acceptance criteria?				
d) For ICP are mass calibration, resolution and RSD within method criteria?				
e) For IC or GC methods, does the ICP meet method criteria?				
<b>5) Initial Calibration Verification (ICV)</b>				
a) Was an ICV performed using a standard independent from the IC?				
b) Was a midpoint concentration value used for the ICV?				
c) Are the % recovery values acceptable? (Typically evaluated against the CV criteria?)				
<b>6) Calibration Verification (CV)</b>				
a) Are the concentration values appropriate for the CV?				
b) Are high and low concentration values being used for GC methods?				
c) Do assigned concentration values match the actual concentration values provided with the calibration standard?				
e) Are % difference or % recovery values for each target analyte or CCC within method criteria?				

Notes:

**Internal Data Quality Audit  
Data Package Review  
Table E.14-7C**

Date: \_\_\_\_\_  
Analyst: \_\_\_\_\_  
Peer: \_\_\_\_\_

Method: \_\_\_\_\_  
Instrument: \_\_\_\_\_  
Matrix: \_\_\_\_\_

DESCRIPTION	Y/N/NA	Corrective Action Due Date	Corrective Action Completion Date	Comments
<b>7) Method Blank (MB) [Negative Control]</b>				
a) Are analytes present above the LOQ in the blank? If so did this affect any of the associated samples?				
<b>8) Laboratory Control Spike (LCS) [Positive Control]</b>				
a) Are the appropriate analytes and spike levels included in the LCS?				
b) Was the LCS prepared independently from the IC or from a second source?				
c) Do the assigned analyte concentrations match the values from the source?				
d) Do the % recovery range of each analyte compare with method stated or laboratory derived acceptance ranges?				
<b>9) Matrix Spike (MS) [Sample Specific Control]</b>				
a) Is the same spike mix and spike levels used in the LCS also used in the MS?				
b) Do MS/MSD analytes meet acceptable RPD criteria?				
<b>10) Sample Data</b>				
a) Are surrogates and internal standards recoveries within the method criteria?				
b) If not was sample re-analyzed or was sample data appropriately footnoted?				
c) Were second column confirmation, GC retention time windows, elution order, and pattern recognition criteria correctly used for reported analytes by GC?				
d) Were concentrations for found analytes calculated and reported correctly?				
<b>11) Standards Preparation</b>				
a) Are all standards traceable to the Certificate of Analysis?				
b) Were all standards completely documented with lot numbers, expiration dates, correct concentration units, solvents, initials etc.?				
c) Was the DOC/MDL/ICV/LCS/MS prepared and clearly documented from a standard source independent from the IC?				

**Management Review**  
**Table 14-8**

Date: \_\_\_\_\_

Personnel Present: \_\_\_\_\_ / \_\_\_\_\_ / \_\_\_\_\_ / \_\_\_\_\_

Review Items	Specific Items of Interest	Comments
1) Suitability of policies and procedures		
2) Reports from managerial and supervisory personnel		
3) Outcome of internal audits		
4) Corrective and preventive actions		
5) Outcome of external audits		
6) Results of PE data results		
7) Changes in volume and types of work		
8) Complaints		
9) Other		
Recommended Changes		



# **Section 15**

## **Quality Assurance Reports**

## **15.0 QUALITY ASSURANCE REPORTS**

Quality assurance reports are designed to keep staff members informed of the performance of QA/QC activities. Quality assurance reporting documents the quality control and quality assurance activities in the laboratory and provides communications and an accountability link among analysts, management and clients. Analytical report formats, which include selected quality control data and evaluations, are referred to as Quality Control Reports or QA deliverables. These provide a direct link between the analyst and the data user concerning the quality of the data.

In some instances, projects need to pay attention to particular quality assurance controls and assessment. In such situations Alpha will provide technical guidance and, if requested, periodic quality assurance reports to keep the project guided toward achieving those quality goals. These reports, both verbal and written, may include subjects that address the validity and documentation of data generation activities.

QA reports typically list significant problems and discuss the solutions and corrective actions implemented concerning QA/QC activities. QA reports may include such things as internal and external audits, QA/QC summary data sheets associated with a batch of analytical samples, PE sample results, etc.

### **15.1 QUALITY CONTROL REPORTING**

Data reported for quality control, including MDL studies will have at least three significant figures unless specified otherwise in the method (i.e., any analytical result being used for quality control calculations should carry three significant figures).

Quality control data reported needs to be expressed in the proper units. Matrix spike and other QC analytical results prior to performing quality control calculations are expressed in the same units as the samples. Likewise, method blank data is expressed in the same units as the samples.

Alpha's standard analytical report does not include quality control documentation. However, a certification that all QC requirements were met is implicit by the signature of the final report.

### **15.2 INTERNAL QA REPORTS**

Internal QA reports are distributed as necessary to keep staff members informed of the performance of QA/QC activities. This information is provided in the form of verbal communication, formal memorandums, or reports to ensure sound QA/QC practices.

### 15.3 Data Deliverables

In some specific situations, samples from a project may need to be technically evaluated by a third party review. Methods of analysis, SOW, QAM and other documents are used to assist the evaluator in the technical review of analytical data generated by our QA Program. Data deliverables is the process of gathering and collating analytical data in a manner which helps organize the data in a logical sequence. Table 15-2 through 15-5, gives guidance on collating and organizing a data deliverables package. Data deliverables may be project specific; therefore, it is important to organize the data deliverables specific to the SOW.

**QA Summary Reports**  
**DQO Reference Table 15-1**

<b>Method</b>	<b>Surrogate Window of Acceptability</b>	<b>LCS Window of Acceptability</b>	<b>MS Window of Acceptability</b>	<b>Matrix</b>
200.8	Not Required	Method Specified	Method Specified	Water
524.2	Method Specific	Method Specified	Not Required	Water
608	Laboratory Derived	Method Specified	Method Specified	Water
610	Laboratory Derived	Method Specified	Method Specified	Water
624	Laboratory Derived	Method Specified	Method Specified	Water
625	Laboratory Derived	Method Specified	Method Specified	Water
6020	Not Required	Laboratory Derived	Laboratory Derived	Water/Soil
8015B-DRO	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8015B-GRO	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8081A	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8082	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8260B	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8270C	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil
8310	Laboratory Derived	Laboratory Derived	Laboratory Derived	Water/Soil

**Example**  
**GC/MS Level IV Deliverables**  
**Table 15-2**

<b>Item #</b>	<b>Deliverables</b>	<b>Page</b>
1	Case Narrative	Page _____ through _____
2	Table of Contents	Page _____ through _____
3	Chain of Custody	Page _____ through _____
4	Sample results with analysis and extraction/preparation dates	Page _____ through _____
5	Raw data which includes chromatograms and quantitation reports	Page _____ through _____
6	Summary of MS/MSD/Duplicate recoveries and control limits (linking native samples)	Page _____ through _____
7	Raw data associated with the MS/MSD/Duplicate which includes chromatograms and quantitation reports (linking native samples)	Page _____ through _____
8	Summary of LCS recoveries and control limits	Page _____ through _____
9	Raw data associated with the LCS which includes chromatograms and quantitation reports	Page _____ through _____
10	Summary of method blank results	Page _____ through _____
11	Raw method blank data which includes chromatograms and quantitation reports	Page _____ through _____
12	Summary of internal standard areas/RT's, and summary of surrogate recoveries	Page _____ through _____
13	Summary of initial calibration data (RF, and %RSD)	Page _____ through _____
14	Raw data associated with the initial calibration which includes chromatograms, quantitation reports, and the calibration plots, indicating correlation coefficients if required	Page _____ through _____
15	Summary of continuing calibration data (% Difference reports from calculated concentrations, and from RRF)	Page _____ through _____
16	Raw data associated with the continuing calibration which includes chromatograms and quantitation reports	Page _____ through _____
17	Summary of instrument tuning (listing associated samples and injection times) for all applicable analytical shifts, including those in which initial calibration levels, QC samples, and client samples were analyzed	Page _____ through _____
18	Instrument sequence/injection logs	Page _____ through _____
19	Extraction/preparation logs and sample dilution logs	Page _____ through _____

**Example**  
**ICP/MS Level IV Deliverables**  
**Table 15-3**

<b>Item #</b>	<b>Deliverables</b>	<b>Page</b>
1	Case Narrative	Page _____ through _____
2	Table of Contents	Page _____ through _____
3	Chain of Custody	Page _____ through _____
4	Sample results with analysis and digestion/preparation dates	Page _____ through _____
5	Raw data which includes quantitation reports	Page _____ through _____
6	Summary of MS/MSD/Duplicate recoveries and control limits (linking native samples)	Page _____ through _____
7	Raw data associated with the MS/MSD/Duplicate which includes quantitation reports (linking native samples)	Page _____ through _____
8	Summary of LCS/LCSD recoveries and control limits	Page _____ through _____
9	Raw data associated with the LCS/LCSD which includes quantitation reports	Page _____ through _____
10	Summary of method blank results	Page _____ through _____
11	Raw method blank data which includes quantitation reports	Page _____ through _____
12	Summary of initial calibration data (CPS, and Linear Regression Equations)	Page _____ through _____
13	Raw data associated with the initial calibration which includes quantitation reports and the calibration plots, indicating correlation coefficients if required	Page _____ through _____
14	Raw data associated with the ICV which includes quantitation reports.	Page _____ through _____
15	Raw data associated with the ICSA/ICSB which includes quantitation reports.	Page _____ through _____
16	Raw data associated with the ICB/CCB which includes quantitation reports.	Page _____ through _____
17	Summary of continuing calibration data	Page _____ through _____
18	Raw data associated with the CCV dwhich includes quantitation reports	Page _____ through _____
19	Summary of instrument tuning	Page _____ through _____
20	Instrument sequence/injection logs	Page _____ through _____
21	Digestion/preparation logs	Page _____ through _____

Alpha Analytical, Inc.  
Section No.: 15.0  
Revision No.: 15.0  
Date: January, 2007  
Page: 5 of 9

**EXAMPLE**  
**QC SUMMARY SHEETS**



# Alpha Analytical, Inc.

255 Glendale Ave. • Suite 21 • Sparks, Nevada 89431-5778  
(775) 355-1044 • (775) 355-0406 FAX • 1-800-283-1183

Date:  
06-Oct-06

## OC Summary Report

Work Order:  
06091223

### Method Blank

Type **MBLK** Test Code: **EPA Method 624/SW8260B**

File ID: **D:\MSDCHEM\MS12\DATA\060913\06091333.D**

Batch ID: **MS12W0913C**

Analysis Date: **09/13/2006 21:58**

Sample ID: **MBLK MS12W0913C**

Units: **µg/L**

Run ID: **MSD\_12\_060913B**

Prep Date: **09/13/2006**

Analyte	Result	PQL	SpkVal	SpkRefVal	%REC	LowLimit	HighLimit	RPDRefVal	%RPD(Limit)	Qual
Chloromethane	ND	2								
Vinyl chloride	ND	1								
Chloroethane	ND	1								
Bromomethane	ND	2								
Trichlorofluoromethane	ND	1								
1,1-Dichloroethene	ND	1								
Dichloromethane	ND	2								
trans-1,2-Dichloroethene	ND	1								
Methyl tert-butyl ether (MTBE)	ND	1								
1,1-Dichloroethane	ND	1								
cis-1,2-Dichloroethene	ND	1								
Chloroform	ND	1								
1,2-Dichloroethane	ND	1								
1,1,1-Trichloroethane	ND	1								
Carbon tetrachloride	ND	1								
Benzene	ND	1								
1,2-Dichloropropane	ND	1								
Trichloroethene	ND	1								
Bromodichloromethane	ND	1								
cis-1,3-Dichloropropene	ND	1								
trans-1,3-Dichloropropene	ND	1								
1,1,2-Trichloroethane	ND	1								
Toluene	ND	1								
Dibromochloromethane	ND	1								
Tetrachloroethene	ND	1								
Chlorobenzene	ND	1								
Ethylbenzene	ND	1								
Bromoform	ND	1								
1,1,2,2-Tetrachloroethane	ND	1								
1,3-Dichlorobenzene	ND	1								
1,4-Dichlorobenzene	ND	1								
1,2-Dichlorobenzene	ND	1								
Xylenes, Total	ND	1								
Surr: 1,2-Dichloroethane-d4	10.3		10		103	76	127			
Surr: Toluene-d8	9.75		10		98	84	113			
Surr: 4-Bromofluorobenzene	9.38		10		94	79	119			

### Laboratory Control Spike

Type **LCS** Test Code: **EPA Method 624/SW8260B**

File ID: **D:\MSDCHEM\MS12\DATA\060913\06091331.D**

Batch ID: **MS12W0913C**

Analysis Date: **09/13/2006 21:15**

Sample ID: **LCS MS12W0913C**

Units: **µg/L**

Run ID: **MSD\_12\_060913B**

Prep Date: **09/13/2006**

Analyte	Result	PQL	SpkVal	SpkRefVal	%REC	LowLimit	HighLimit	RPDRefVal	%RPD(Limit)	Qual
1,1-Dichloroethene	10.3	1	10		103	80	120			
Benzene	10.7	0.5	10		107	81	122			
Trichloroethene	10.7	1	10		107	74	125			
Toluene	10.3	0.5	10		103	80	120			
Chlorobenzene	10.5	1	10		105	79	124			
Ethylbenzene	10.7	0.5	10		107	80	120			
Xylenes, Total	21.4	0.5	20		107	81	128			
Surr: 1,2-Dichloroethane-d4	10.6		10		106	76	127			
Surr: Toluene-d8	9.95		10		100	84	113			
Surr: 4-Bromofluorobenzene	9.69		10		97	79	119			



# Alpha Analytical, Inc.

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Date:

06-Oct-06

## QC Summary Report

Work Order:

06091223

### Sample Matrix Spike

Type MS

Test Code: EPA Method 624/SW8260B

File ID: D:\MSDCHEM\MS12\DATA\060913\06091348.D

Batch ID: MS12W0913C

Analysis Date: 09/14/2006 03:15

Sample ID: 06091223-02AMS

Units: µg/L

Run ID: MSD\_12\_060913B

Prep Date: 09/14/2006

Analyte	Result	PQL	SpkVal	SpkRefVal	%REC	LowLimit	HighLimit	RPDRefVal	%RPD(Limit)	Qual
1,1-Dichloroethene	47.5	2.5	50	0	95	65	127			
Benzene	49.4	1.3	50	0	99	74	125			
Trichloroethene	45.8	2.5	50	0	92	66	126			
Toluene	45.3	1.3	50	0	91	76	120			
Chlorobenzene	47	2.5	50	0	94	76	124			
Ethylbenzene	47.3	1.3	50	0	95	77	124			
Xylenes, Total	94.7	1.3	100	0	95	75	130			
Surr: 1,2-Dichloroethane-d4	55.8		50		112	76	127			
Surr: Toluene-d8	48.1		50		96	84	113			
Surr: 4-Bromofluorobenzene	47.7		50		95	79	119			

### Sample Matrix Spike Duplicate

Type MSD

Test Code: EPA Method 624/SW8260B

File ID: D:\MSDCHEM\MS12\DATA\060913\06091349.D

Batch ID: MS12W0913C

Analysis Date: 09/14/2006 03:37

Sample ID: 06091223-02AMSD

Units: µg/L

Run ID: MSD\_12\_060913B

Prep Date: 09/14/2006

Analyte	Result	PQL	SpkVal	SpkRefVal	%REC	LowLimit	HighLimit	RPDRefVal	%RPD(Limit)	Qual
1,1-Dichloroethene	51.4	2.5	50	0	103	65	127	47.53	7.8(17)	
Benzene	52.9	1.3	50	0	106	74	125	49.4	6.9(13)	
Trichloroethene	50.2	2.5	50	0	100	66	126	45.75	9.2(13)	
Toluene	49.4	1.3	50	0	99	76	120	45.26	8.7(13)	
Chlorobenzene	50.4	2.5	50	0	101	76	124	46.96	7.1(12)	
Ethylbenzene	51.2	1.3	50	0	102	77	124	47.32	7.9(13)	
Xylenes, Total	103	1.3	100	0	103	75	130	94.66	8.2(13)	
Surr: 1,2-Dichloroethane-d4	54.9		50		110	76	127			
Surr: Toluene-d8	48.7		50		97	84	113			
Surr: 4-Bromofluorobenzene	48		50		96	79	119			

### Comments:

Calculations are based off of raw (non-rounded) data. However, for reporting purposes, all QC data is rounded to three significant figures. Therefore, hand calculated values may differ slightly.





# Alpha Analytical, Inc.

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Date:

06-Oct-06

## OC Summary Report

Work Order:

99999999

### Method Blank

Type **MBLK** Test Code: **EPA Method 200.8**

File ID: **100406.B\031AICB.D\**

Batch ID: **15759**

Analysis Date: **10/04/2006 19:46**

Sample ID: **MB-15759**

Units : **mg/L**

Run ID: **ICP/MS\_061004A**

Prep Date: **10/04/2006**

Analyte	Result	PQL	SpkVal	SpkRefVal	%REC	LowLimit	HighLimit	RPDRefVal	%RPD(Limit)	Qual
Beryllium (Be)	ND	0.004								
Vanadium (V)	ND	0.003								
Chromium (Cr)	ND	0.005								
Cobalt (Co)	ND	0.005								
Nickel (Ni)	ND	0.01								
Copper (Cu)	ND	0.01								
Zinc (Zn)	ND	0.1								
Arsenic (As)	ND	0.005								
Selenium (Se)	ND	0.005								
Molybdenum (Mo)	ND	0.005								
Silver (Ag)	ND	0.005								
Cadmium (Cd)	ND	0.005								
Antimony (Sb)	ND	0.005								
Barium (Ba)	ND	0.005								
Mercury (Hg)	ND	0.001								
Thallium (Tl)	ND	0.002								
Lead (Pb)	ND	0.005								

### Laboratory Control Spike

Type **LCS** Test Code: **EPA Method 200.8**

File ID: **100406.B\032ALCS.D\**

Batch ID: **15759**

Analysis Date: **10/04/2006 19:51**

Sample ID: **LCS-15759**

Units : **mg/L**

Run ID: **ICP/MS\_061004A**

Prep Date: **10/04/2006**

Analyte	Result	PQL	SpkVal	SpkRefVal	%REC	LowLimit	HighLimit	RPDRefVal	%RPD(Limit)	Qual
Beryllium (Be)	0.264	0.004	0.25		106	84	118			
Vanadium (V)	0.255	0.003	0.25		102	84	118			
Chromium (Cr)	0.242	0.005	0.25		97	84	118			
Cobalt (Co)	0.258	0.005	0.25		103	84	118			
Nickel (Ni)	0.257	0.01	0.25		103	84	118			
Copper (Cu)	0.256	0.01	0.25		102	84	118			
Zinc (Zn)	0.249	0.1	0.25		99.6	84	118			
Arsenic (As)	0.252	0.005	0.25		101	84	118			
Selenium (Se)	0.253	0.005	0.25		101	84	118			
Molybdenum (Mo)	0.252	0.005	0.25		101	84	118			
Silver (Ag)	0.246	0.005	0.25		98	84	118			
Cadmium (Cd)	0.262	0.005	0.25		105	84	118			
Antimony (Sb)	0.259	0.005	0.25		103	84	118			
Barium (Ba)	2.78	0.005	2.5		111	84	118			
Mercury (Hg)	0.00554	0.001	0.005		111	72	119			
Thallium (Tl)	0.225	0.002	0.25		90	84	118			
Lead (Pb)	0.26	0.005	0.25		104	84	118			

### Laboratory Control Spike Duplicate

Type **LCSD** Test Code: **EPA Method 200.8**

File ID: **100406.B\033ALCS.D\**

Batch ID: **15759**

Analysis Date: **10/04/2006 19:56**

Sample ID: **LCSD-15759**

Units : **mg/L**

Run ID: **ICP/MS\_061004A**

Prep Date: **10/04/2006**

Analyte	Result	PQL	SpkVal	SpkRefVal	%REC	LowLimit	HighLimit	RPDRefVal	%RPD(Limit)	Qual
Beryllium (Be)	0.258	0.004	0.25		103	84	118	0.2639	2.1(9)	
Vanadium (V)	0.252	0.003	0.25		101	84	118	0.2549	1.3(9)	
Chromium (Cr)	0.239	0.005	0.25		95	84	118	0.2422	1.5(9)	
Cobalt (Co)	0.254	0.005	0.25		102	84	118	0.2579	1.4(9)	
Nickel (Ni)	0.257	0.01	0.25		103	84	118	0.2571	0.2(9)	
Copper (Cu)	0.258	0.01	0.25		103	84	118	0.2562	0.5(9)	
Zinc (Zn)	0.252	0.1	0.25		101	84	118	0.2489	1.4(9)	
Arsenic (As)	0.251	0.005	0.25		100	84	118	0.252	0.6(9)	
Selenium (Se)	0.25	0.005	0.25		99.9	84	118	0.2525	1.1(9)	
Molybdenum (Mo)	0.251	0.005	0.25		100	84	118	0.2517	0.4(9)	
Silver (Ag)	0.24	0.005	0.25		96	84	118	0.2462	2.7(9)	
Cadmium (Cd)	0.259	0.005	0.25		103	84	118	0.2621	1.4(9)	
Antimony (Sb)	0.255	0.005	0.25		102	84	118	0.2587	1.4(9)	
Barium (Ba)	2.74	0.005	2.5		110	84	118	2.784	1.5(9)	
Mercury (Hg)	0.0052	0.001	0.005		104	72	119	0.00554	6.3(11)	
Thallium (Tl)	0.232	0.002	0.25		93	84	118	0.2252	3.1(9)	
Lead (Pb)	0.26	0.005	0.25		104	84	118	0.2603	0.3(9)	



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Date:  
06-Oct-06

## OC Summary Report

Work Order:  
99999999

### Sample Matrix Spike

Type MS Test Code: EPA Method 200.8

File ID: 100406.B\035MSL.D\

Batch ID: 15759

Analysis Date: 10/04/2006 20:06

Sample ID: 06092934-03AMS

Units : mg/L

Run ID: ICP/MS\_061004A

Prep Date: 10/04/2006

Analyte	Result	PQL	SpkVal	SpkRefVal	%REC	LowLimit	HighLimit	RPDRefVal	%RPD(Limit)	Qual
Beryllium (Be)	0.249	0.004	0.25	0	99.6	75	126			
Vanadium (V)	0.24	0.003	0.25	0	96	75	126			
Chromium (Cr)	0.227	0.005	0.25	0	91	75	126			
Cobalt (Co)	0.227	0.005	0.25	0	91	75	126			
Nickel (Ni)	0.242	0.01	0.25	0	97	75	126			
Copper (Cu)	0.243	0.01	0.25	0	97	75	126			
Zinc (Zn)	0.234	0.1	0.25	0	94	75	126			
Arsenic (As)	0.241	0.005	0.25	0	96	75	126			
Selenium (Se)	0.241	0.005	0.25	0	96	75	126			
Molybdenum (Mo)	0.245	0.005	0.25	0.005198	96	75	126			
Silver (Ag)	0.236	0.005	0.25	0.01618	88	75	126			
Cadmium (Cd)	0.252	0.005	0.25	0	101	75	126			
Antimony (Sb)	0.249	0.005	0.25	0	99	75	126			
Barium (Ba)	2.68	0.005	2.5	0	107	75	126			
Mercury (Hg)	0.00521	0.001	0.005	0	104	75	126			
Thallium (Tl)	0.22	0.002	0.25	0.008011	85	75	126			
Lead (Pb)	0.251	0.005	0.25	0	100	75	126			

### Sample Matrix Spike Duplicate

Type MSD Test Code: EPA Method 200.8

File ID: 100406.B\036MSD.D\

Batch ID: 15759

Analysis Date: 10/04/2006 20:11

Sample ID: 06092934-03AMSD

Units : mg/L

Run ID: ICP/MS\_061004A

Prep Date: 10/04/2006

Analyte	Result	PQL	SpkVal	SpkRefVal	%REC	LowLimit	HighLimit	RPDRefVal	%RPD(Limit)	Qual
Beryllium (Be)	0.249	0.004	0.25	0	99.6	75	126	0.249	0.0(10)	
Vanadium (V)	0.25	0.003	0.25	0	100	75	126	0.2401	4.1(10)	
Chromium (Cr)	0.238	0.005	0.25	0	95	75	126	0.2268	4.7(10)	
Cobalt (Co)	0.246	0.005	0.25	0	99	75	126	0.2266	8.3(10)	
Nickel (Ni)	0.253	0.01	0.25	0	101	75	126	0.2416	4.5(10)	
Copper (Cu)	0.255	0.01	0.25	0	102	75	126	0.243	4.8(10)	
Zinc (Zn)	0.245	0.1	0.25	0	98	75	126	0.2342	4.7(10)	
Arsenic (As)	0.25	0.005	0.25	0	99.9	75	126	0.2411	3.5(10)	
Selenium (Se)	0.249	0.005	0.25	0	99	75	126	0.2407	3.2(10)	
Molybdenum (Mo)	0.251	0.005	0.25	0.005198	98	75	126	0.2447	2.6(10)	
Silver (Ag)	0.235	0.005	0.25	0.01618	87	75	126	0.2364	0.7(10)	
Cadmium (Cd)	0.256	0.005	0.25	0	102	75	126	0.252	1.4(10)	
Antimony (Sb)	0.252	0.005	0.25	0	101	75	126	0.2486	1.4(10)	
Barium (Ba)	2.72	0.005	2.5	0	109	75	126	2.679	1.5(10)	
Mercury (Hg)	0.00528	0.001	0.005	0	106	75	126	0.005208	1.4(19)	
Thallium (Tl)	0.232	0.002	0.25	0.008011	90	75	126	0.2202	5.2(10)	
Lead (Pb)	0.256	0.005	0.25	0	102	75	126	0.2506	2.0(10)	

### Comments:

Calculations are based off of raw (non-rounded) data. However, for reporting purposes, all QC data is rounded to three significant figures. Therefore, hand calculated values may differ slightly.

# **Section 16**

## **Laboratory Reports and Reporting Procedures**

## **16.0 LABORATORY REPORTS AND REPORTING PROCEDURES**

### **16.1 DATA REPORTING**

#### **16.1.1 Policy**

It is Alpha's policy to report environmental test results accurately, clearly, unambiguously and objectively, and in accordance with any specific instruction in the test method. Analytical test data is reported on a final analytical test report which summarizes all the information requested by the client and that which is necessary for the interpretation of the test results and all information required by the method.

#### **16.1.2 Test Reports**

The generation of analytical data requires the use of computerized data systems for acquisition, storage, retrieval, and reporting of data. After the data has been acquired, reduced, and reviewed, it is then transcribed onto a final analytical report. For most analytical final reports, the report will include the following information:

- a) Title: "Analytical Report,"
- b) Alpha's Name and Address,
- c) A unique serial number (project identification) at the bottom of the report, and page number,
- d) Title of project or job number,
  - i. Client address,
  - ii. Client phone number,
  - iii. Point of contact,
- e) Analytical method,
- f) Sample identification,
  - i. Alpha Analytical identification number,
  - ii. Client identification number,
- g) Dates
  - i. Date sample collected,
  - ii. Date sample received,
  - iii. Date sample analyzed,

Note: If the time of sample extraction or analysis is less than 72 hours from sample collection, than they are on the final report.

- h) Sample matrix,
- i) Test results,
  - i. Reporting limits,
  - ii. Concentration units,
  - iii. List of compounds analyzed,
  - iv. Observations or comments such as failed quality control,
- j) Signature or approval block and date of issue.

### 16.1.3 Test Report Description

#### 16.1.3.1 Footnotes or Data Qualifiers

Data is reported when the sample analysis occurred during periods that the calibration and systems were in-control. If a quality control measure is found to be out-of-control, and the data are to be reported, then the failed QC measure is reported with the appropriate data qualifiers.

#### 16.1.3.2 Concentration Units

All numerical results are reported in terms of concentrations (i.e., µg/Kg for soils or µg/L for waters) in the environmental sample.

#### 16.1.3.3 Reporting Limits

Reporting limits are required for all methods to evaluate method performance. All values less than the reporting limit are reported as Not Detectable, "ND". Reporting limits are dependent on the matrix of the sample that is being tested. Interferences frequently require sample dilution, which may change the analytical reporting limit.

### 16.1.4 Test Report Format

16.1.4.1 Alpha Analytical has designed final reports and reporting formats which facilitate the analytical data review process. These reports are formatted for specific regulatory programs and designed to display information accordingly.

16.1.4.2 Final reports may contain multiple analyses on a given report from a set of samples with the same type of analysis.

16.1.4.3 Final reports may contain analytical information from several methods of analysis from the same sample.

- 16.1.4.4 Reports can be customized to virtually any client request or QAPP specific request. Not only can reports be customized, data can also be reported on a number of different spreadsheets or databases (i.e., Excel) and transmitted electronically.

#### 16.1.5 Analytical Reports Signature Block

The following personnel have the authority to sign final analytical data reports and to approve or disapprove other final analytical data reports that do not require a signature block such as Electronic Data Deliverables (EDD) or other data packages.

- 1) Laboratory Director
- 2) Quality Assurance Officer
- 3) Laboratory Manager

#### 16.1.6 Verbal Analytical Data Release

The following personnel have the authority to verbally release final analytical data to clients that has previously been signed:

- 1) Laboratory Director,
- 2) Quality Assurance Officer,
- 3) Laboratory Manager,
- 4) Director of Client Services,
- 5) Project Coordinators,
- 6) Supervisors, and
- 7) LIMS Administrator

## 16.2 SIGNIFICANT FIGURES

### 16.2.1 Definition

Significant figures is the count of the number of digits in a number which properly represents a measured quantity when the number is expressed in scientific notations.

### 16.2.2 Purpose

When properly rounded, the result of a measurement is expressed so that the last digit remaining shows where the uncertainty in the measurement begins. The nature of the measurement process, accounting for all cumulative errors from consecutive operations, determines the number of significant figures.

### 16.2.3 Methods

There are two methods used to signify uncertainty. The most definitive technique is to follow the measured value with the  $\pm$  and the amount of uncertainty (e.g.,  $10 \pm 2$ ). The less informative and more widely used method is to indicate the degree of uncertainty by the number of significant figures in the reported value. The last figure shown is the one in which there is uncertainty.

#### 16.2.4. Procedure

The proper use of the concept of significant figures requires adherence to some conventions.

##### 16.2.4.1 Rules of Significant Figures

Zeros may be significant digits, for example 50, 2.0, or 505. However, there are two different functions for which zeros are not considered significant digits.

- a) Zeros may flag a decimal, for example 0.52.
- b) Zeros may also define magnitude, for example 500, 50, or 0.05.

When reporting data which follows the conventions for significant figures, zeros will not be added to make each number have the same number of digits past the decimal point (i.e., we report 520 not 520.0)

##### 16.2.4.2 Rounding Rules

In reporting results, rounding to the correct number of significant figures occurs only after all calculations and data manipulations are completed. Premature rounding can significantly affect the final result. When the calculation or instrument gives more figures than needed, it is necessary to round off. The following rules shall be used:

- Rule 1: If the next digit beyond the rounding point is less than 5, leave the previous digit unchanged (e.g., 21.4 becomes 21).
- Rule 2: If the next digit beyond the rounding point is greater than 5, increase the previous digit by one (e.g., 21.6 becomes 22).
- Rule 3: If the next digit beyond the rounding point is equal to 5, with no digits other than zero following the 5,

round the previous digit to the nearest even number (e.g., 21.5 and 22.5 both become 22).

Rule 4: If the next digit is a 5 followed by other digits, then treat the case as in rule 2 - for greater than 5 (e.g., 21.51 becomes 22).

Rule 5: If there are not enough numbers to get to the required number of significant figures, for example 2.3 when working with three significant figures, do not add extra zeros.

Rule 6: When performing calculations, carry at least one extra significant figure through the process and round only the final result. Rounding data before a calculation introduces a cumulative error. Carrying at least one extra digit minimizes this error.

### **16.3 CONTRACT LABORATORY PROGRAM (CLP) DATA QUALIFIERS**

16.3.1 To the extent possible, samples are reported only if quality control measurements are acceptable. If a quality control measure is found to be out of control, and the data are to be reported, the failed QC measure is reported with the appropriate footnote or data qualifier.

16.3.2 Many times CLP data qualifiers do not accurately describe or represent the associated sample problem. In these cases custom footnotes or footnotes requested by a state agency which more accurately describe the sample situation is appended to the bottom of the associated sample reports.

16.3.3 The CLP describes a set of data qualifiers that are often requested by the end user. The following definitions provide brief explanations of the CLP qualifiers assigned to results in the data review process. Additional data qualifiers may be requested by data validators when evaluating data usability.

- U - Undetected at the limit of detection. The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
- J - Estimated: The analyte was positively identified; the quantitation is an estimation.
- N - Nontarget analyte: The analyte is a tentatively identified compound (using mass spectroscopy).



- NJ - The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
- UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte.
- R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
- B - Blank contamination: The analyte was detected above the reporting limit in an associated blank.
- Q - One or more quality control criteria failed. Data usability should be carefully assessed by the project team.

Note: This is a DoD footnote only.

Alpha Analytical, Inc.  
Section No.: 16.0  
Revision No.: 15.0  
Date: January, 2007  
Page: 7 of 8

**EXAMPLE  
ANALYTICAL REPORT**



# Alpha Analytical, Inc.

255 Glendale Ave. • Suite 21 • Sparks, Nevada 89431-5778

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## ANALYTICAL REPORT

Alpha Analytical, Inc.--Sparks Office  
255 Glendale Avenue  
Sparks, NV 89431  
Job#: Example Project Workorder

Attn: Randy Gardner  
Phone: (775) 355-1044  
Fax: (775) 355-0406

Alpha Analytical Number: AAI99999999-01A  
Client I.D. Number: WATER

Sampled: 11/20/06  
Received: 11/20/06  
Analyzed: 11/20/06

### Volatile Organics by GC/MS EPA Method SW8260B

Compound	Concentration	Reporting Limit	Compound	Concentration	Reporting Limit
1 Dichlorodifluoromethane	ND	1.0 µg/L	36 Bromoform	ND	1.0 µg/L
2 Chloromethane	ND	2.0 µg/L	37 Styrene	ND	1.0 µg/L
3 Vinyl chloride	ND	1.0 µg/L	38 o-Xylene	ND	0.50 µg/L
4 Chloroethane	ND	1.0 µg/L	39 1,1,2,2-Tetrachloroethane	ND	1.0 µg/L
5 Bromomethane	ND	2.0 µg/L	40 1,2,3-Trichloropropane	ND	2.0 µg/L
6 Trichlorofluoromethane	ND	1.0 µg/L	41 Isopropylbenzene	ND	1.0 µg/L
7 1,1-Dichloroethene	ND	1.0 µg/L	42 Bromobenzene	ND	1.0 µg/L
8 Dichloromethane	ND	2.0 µg/L	43 n-Propylbenzene	ND	1.0 µg/L
9 trans-1,2-Dichloroethene	ND	1.0 µg/L	44 4-Chlorotoluene	ND	1.0 µg/L
10 1,1-Dichloroethane	ND	1.0 µg/L	45 2-Chlorotoluene	ND	1.0 µg/L
11 cis-1,2-Dichloroethene	ND	1.0 µg/L	46 1,3,5-Trimethylbenzene	ND	1.0 µg/L
12 Bromochloromethane	ND	1.0 µg/L	47 tert-Butylbenzene	ND	1.0 µg/L
13 Chloroform	ND	1.0 µg/L	48 1,2,4-Trimethylbenzene	ND	1.0 µg/L
14 2,2-Dichloropropane	ND	1.0 µg/L	49 sec-Butylbenzene	ND	1.0 µg/L
15 1,2-Dichloroethane	ND	1.0 µg/L	50 1,3-Dichlorobenzene	ND	1.0 µg/L
16 1,1,1-Trichloroethane	ND	1.0 µg/L	51 1,4-Dichlorobenzene	ND	1.0 µg/L
17 1,1-Dichloropropene	ND	1.0 µg/L	52 4-Isopropyltoluene	ND	1.0 µg/L
18 Carbon tetrachloride	ND	1.0 µg/L	53 1,2-Dichlorobenzene	ND	1.0 µg/L
19 Benzene	ND	0.50 µg/L	54 n-Butylbenzene	ND	1.0 µg/L
20 Dibromomethane	ND	1.0 µg/L	55 1,2-Dibromo-3-chloropropane (DBCP)	ND	3.0 µg/L
21 1,2-Dichloropropane	ND	1.0 µg/L	56 1,2,4-Trichlorobenzene	ND	2.0 µg/L
22 Trichloroethene	ND	1.0 µg/L	57 Naphthalene	ND	2.0 µg/L
23 Bromodichloromethane	ND	1.0 µg/L	58 Hexachlorobutadiene	ND	2.0 µg/L
24 cis-1,3-Dichloropropene	ND	1.0 µg/L	59 1,2,3-Trichlorobenzene	ND	2.0 µg/L
25 trans-1,3-Dichloropropene	ND	1.0 µg/L			
26 1,1,2-Trichloroethane	ND	1.0 µg/L			
27 Toluene	ND	0.50 µg/L			
28 1,3-Dichloropropane	ND	1.0 µg/L			
29 Dibromochloromethane	ND	1.0 µg/L			
30 1,2-Dibromoethane (EDB)	ND	2.0 µg/L			
31 Tetrachloroethene	ND	1.0 µg/L			
32 1,1,1,2-Tetrachloroethane	ND	1.0 µg/L			
33 Chlorobenzene	ND	1.0 µg/L			
34 Ethylbenzene	ND	0.50 µg/L			
35 m,p-Xylene	ND	0.50 µg/L			

ND = Not Detected

*Roger Scholl*

*Randy Gardner*

*Walter Hinchman*

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Alpha Analytical, Inc. currently holds appropriate and available NDEP certifications for the data reported - certification #NV16.

Report Date

Page 1 of 1

# **Section 17**

## **List of Acronyms and Abbreviations**

## 17.0 LIST OF ACRONYMS AND ABBREVIATIONS

### A

<b>AAI</b>	Alpha Analytical, Inc.
<b>ACS</b>	American Chemical Society
<b>ALS</b>	automatic liquid sampler
<b>A2LA</b>	American Association for Laboratory Accreditation
<b>APHA</b>	American Public Health Association
<b>ASCII</b>	American Standard Code Information Interchange
<b>ASE</b>	accelerated solvent extraction
<b>ASTM</b>	American Society for Testing and Materials
<b>AWWA</b>	American Water Works Association

### B

<b>BFB</b>	4-bromofluorobenzene
<b>BLK</b>	blank
<b>BN</b>	base/neutral
<b>BNA</b>	base/neutral acid
<b>BOD</b>	biological oxygen demand
<b>BTEX</b>	benzene, toluene, ethyl benzene, xylene

### C

<b>C</b>	concentration
<b>CAS</b>	Chemical Abstract Service
<b>CCC</b>	calibration check compounds
<b>CV</b>	calibration verification
<b>CERCLA</b>	Comprehensive Environmental Response, Compensation, and Liability Act
<b>CF</b>	calibration factor
<b>CFR</b>	Code of Federal Regulation
<b>CLP</b>	Contract Laboratory Program
<b>COC</b>	Chain-of-Custody
<b>COD</b>	chemical oxygen demand

### D

<b>DAD</b>	diode-array detector
<b>DCBP</b>	decachlorobiphenyl
<b>DCO</b>	document control officer
<b>DDD</b>	dichlorodiphenyldichloroethane
<b>DDE</b>	dichlorodiphenyldichloroethene
<b>DDT</b>	dichlorodiphenyltrichloroethane

**DFTPP** decafluorotriphenylphosphine  
**DHS** Department of Health Services  
**DI** de-ionized  
**DQO** data quality objective  
**DRO** diesel range organics

## **E**

**EC** electrolytic conductivity  
**ECD** electron capture detector  
**EDB** ethylene dibromide  
**EDD** electronic diskette deliverables  
**EDL** estimated detection limit  
**EICP** extracted ion current profile  
**EMSL** Environmental Monitoring Support Laboratory  
**EPA** Environmental Protection Agency  
**EQL** estimated quantitation limits  
**ERM** Environmental Resource Management

## **F**

**FID** flame ionization detector  
**FRB** field reagent blank  
**FSP** field sampling plan  
**FTB** field transfer blank

## **G**

**G** glass  
**GC** gas chromatography  
**GC/MS** gas chromatography/mass spectroscopy  
**GLP** good laboratory practice  
**GRO** gasoline range organics  
**GS** gas spike

## **H**

**HCl** hydrochloric acid  
**HDPE** high density polyethylene  
**HNO<sub>3</sub>** nitric acid  
**HP** Hewlett Packard  
**HPLC** high-performance liquid chromatography  
**H<sub>2</sub>SO<sub>4</sub>** sulfuric acid

## I

<b>IB</b>	instrument blank
<b>IC</b>	inorganic carbon
<b>ICL</b>	inner control limit
<b>ICP</b>	inductively coupled plasma
<b>ICS</b>	interference check standard
<b>ID</b>	identification
<b>IDC</b>	initial demonstration of capabilities
<b>IOC</b>	inorganic compounds
<b>IPC</b>	instrument performance check
<b>IS</b>	internal standard

## K

<b>K-D</b>	Kuderna-Danish
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## L

<b>LC</b>	liquid chromatography
<b>LCL</b>	lower control limit
<b>LCS</b>	laboratory control sample
<b>LFB</b>	laboratory fortified blank
<b>LFM</b>	laboratory fortified matrix
<b>LIMS</b>	laboratory information management systems
<b>LPC</b>	laboratory performance check sample
<b>LRB</b>	laboratory reagent blank
<b>LSC</b>	liquid sample concentrator
<b>LUFT</b>	leaking underground fuel tanks

## M

<b>MB</b>	method blank
<b>MBAS</b>	methylene blue active substances
<b>MDL</b>	method detection limit
<b>MeOH</b>	methanol
<b>MeCl<sub>2</sub></b>	methylene chloride
<b>MRL</b>	method reporting limit
<b>MS</b>	matrix spike
<b>MSD</b>	matrix spike duplicate
<b>MSD</b>	mass selective detector
<b>MTBE</b>	methyl-tert-butyl ether

## N

<b>N</b>	normal
<b>N<sub>2</sub></b>	nitrogen
<b>NA</b>	not applicable
<b>NaCl</b>	sodium chloride
<b>NaOH</b>	sodium hydroxide
<b>Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub></b>	sodium thiosulfate
<b>Na<sub>2</sub>SO<sub>3</sub></b>	sodium thiosulfite
<b>Na<sub>2</sub>SO<sub>4</sub></b>	sodium sulfate
<b>NBS</b>	National Bureau of Standards
<b>ND</b>	not detected
<b>NEDTS</b>	Navy Environmental Data Transfer Standard
<b>NEIC</b>	National Enforcement Investigations Center
<b>NELAC</b>	National Environmental Laboratory Accreditation Conference
<b>NELAP</b>	National Environmental Laboratory Accreditation Program
<b>NDEP</b>	Nevada Department of Environmental Protection
<b>NH<sub>4</sub>Cl</b>	ammonium chloride
<b>NIH</b>	National Institute of Health
<b>NIOSHA</b>	National Institute of Occupational Safety and Health Administration
<b>NIST</b>	National Institute of Standards and Technology
<b>NPd</b>	nitrogen phosphorus detector
<b>NPDES</b>	National Pollution Discharge Elimination System
<b>NPOC</b>	non-purgeable organic carbon
<b>NVLAP</b>	Nevada Laboratory Accreditation Program

## O

<b>OJT</b>	on-the-job training
<b>ORO</b>	oil range organics
<b>OSHA</b>	Occupational Safety and Health Administration
<b>OSWER</b>	Office of Solid Waste Environmental Regulations

## P

<b>P</b>	polyethylene
<b>PAH</b>	polynuclear aromatic hydrocarbon
<b>PARCC</b>	precision, accuracy, representativeness, comparability, and completeness
<b>PCB</b>	polychlorinated biphenyl
<b>PE</b>	performance evaluation
<b>PFE</b>	pressurized fluid extraction
<b>PHP</b>	potassium hydrogen phthalate
<b>PI</b>	principle investigator



<b>PID</b>	photo ionization detector
<b>POC</b>	point of contact; purgeable organic carbon
<b>ppb</b>	parts per billion
<b>ppm</b>	parts per million
<b>PQL</b>	practical quantitation limit
<b>PSI</b>	pounds per square inch
<b>PT</b>	proficiency testing

## Q

<b>QA</b>	quality assurance
<b>QAMS</b>	quality assurance management system
<b>QAP</b>	quality assurance plan
<b>QAPP</b>	quality assurance project plan
<b>QAO</b>	quality assurance officer
<b>QC</b>	quality control
<b>QCS</b>	quality control sample

## R

<b>R</b>	recovery
<b>RAAS</b>	robotic arm automatic sampler
<b>RCRA</b>	Resource Conservation and Recovery Act
<b>RF</b>	response factor
<b>RIC</b>	reconstructed ion chromatograph
<b>RPD</b>	relative percent difference
<b>RRF</b>	relative response factor
<b>RRT</b>	relative retention time
<b>RSD</b>	relative standard deviation
<b>RT</b>	retention time

## S

<b>S</b>	soil
<b>SARA</b>	Superfund Amendments and Reauthorization Act
<b>SB</b>	storage blank
<b>SCO</b>	sample custody officer
<b>SD</b>	standard deviation
<b>SDWA</b>	Safe Drinking Water Act
<b>SLC</b>	software life cycle
<b>SOP</b>	standard operating procedure
<b>SOW</b>	statement of work
<b>SPCC</b>	system performance check compound
<b>SPE</b>	solid phase extraction
<b>SPLP</b>	synthetic precipitation leaching procedure

**SQAP** software quality assurance plan  
**SRS** self regenerating suppressor  
**STD** Standard Deviation  
**STP** sample tracking plan  
**SVOC** semivolatile organic compound

**T**

**TAT** turn-around time  
**TB** trip blank  
**TCL** target compound list  
**TCLP** toxicity characteristic leaching procedure  
**TCMX** tetrachloro-meta-xylene  
**TDS** total dissolved solids  
**THMs** trihalomethanes  
**TIC** tentatively identified compound  
**TOC** total organic compounds  
**TPH** total petroleum hydrocarbon

**U**

**UCL** upper control limit  
**USEPA** United States Environmental Protection Agency  
**UV** ultraviolet

**V**

**V** volume  
**VOA** volatile organic analysis  
**VOC** volatile organic compound  
**VOST** volatile organic sampling train

**W**

**W** water  
**WP** Water Pollution Study  
**WPCF** Water Pollution Control Federation  
**WS** Water Supply Study

**Z**

**ZHE** zero headspace extraction

## 17.1 SYMBOLS

<b>°C</b>	degrees Celsius
<b>g</b>	gram
<b>Kg</b>	kilogram
<b>L</b>	liter
<b>µg</b>	microgram
<b>µl</b>	microliter
<b>mg</b>	milligram
<b>ml</b>	milliliter
<b>mm</b>	millimeter
<b>mg/Kg</b>	milligrams per kilogram
<b>mg/L</b>	milligrams per liter
<b>ng</b>	nanogram
<b>nm</b>	nanometer
<b>oz</b>	ounce
<b>µg/Kg</b>	micrograms per kilogram
<b>µg/L</b>	micrograms per liter

# **Section 18**

## **Glossary of Terms**

## 18.0 GLOSSARY OF TERMS

The following definitions are used in the text of the NELAP Quality System and Alpha's Quality Assurance Manual.

**Acceptance Criteria** - Specified limits placed on characteristics of an item, process, or service defined in required documents.

**Accreditation** - The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one.

**Accuracy** - the difference between individual analytical measurements and the true value, corresponding to the sum of systematic and random errors.

**Aliquot** - a measured portion of a sample taken for analysis.

**Analyte** - a chemical component for which analysis is conducted.

**Analytical Method** - a set of written instructions completely defining the procedure to be adopted by the analyst in order to obtain an analytical result.

**Audit** - a systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types; 1) performance/internal audits in which quantitative data are independently obtained for comparison with routinely obtained data in a measurement system; or 2) system/external audits of a qualitative nature that consist of an on-site review of a laboratory's quality assurance system and physical facilities for sampling, calibration and measurement.

**Autozero** - zeroing the instrument (typically a spectrophotometer for inorganic analysis) at the proper wavelength. It is equivalent to running a standard blank with the absorbance set at zero.

**Bar Graph Spectrum** - a plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

**Batch** - Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lots of reagents. A **preparation batch** is composed of one to 20 environmental samples of the sample matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

**Blank** - A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

**Blind Sample** - A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

**4-Bromofluorobenzene (BFB)** - the compound chosen to establish mass spectral instrument performance for volatile analyses.

**Calibration Curve** - The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument responses.

**Calibration Standard** - A substance or reference material used to calibrate an instrument.

**Certification** - approval by a certifying agency to use an analytical method for analysis of specific analytes following submission of a performance data package.

**Chain of Custody** - formalized system of creating an accurate written record which can be used to trace the possession and handling of a sample from the moment of collection.

**Comparability** - confidence with which one data set can be compared to another.

**Confirmation** - Verification of the identity of a component through the use of an approach with a different scientific principle from the original method.

**Conformance** - An affirmative indication or judgment that a product or service has met the requirements or the relevant specifications, contracts, or regulation; also the state of meeting the requirements.

**Control Samples** - samples introduced into the train of environmental samples as monitors of the performance of the analytical method.

**Corrective Action** - The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

**Data Audit** - A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

**Data Quality** - totality of features and characteristics of a data set that bears on its ability to satisfy a given purpose.

**Data Reduction** - The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc. and collation into a more useable form.

**Data Validation** - a systematic process for reviewing a body of data against a set of criteria to provide assurance that the data are adequate for their intended use. Data validation consists of data editing, screening, checking, auditing, verification, certification, and review.

**Decafluorotriphenylphosphine (DFTPP)** - a compound chosen to establish mass spectral tuning performance for semi-volatile analysis.

**Demonstration of Capability** - A procedure to establish the ability of the analyst to generate acceptable accuracy.

**Detection Limit** - The lowest concentration or amount of the target analyte that can be Identified, measured, and reported with confidence that the analyte concentration is not a false positive value.

**Document Control** - The act of ensuring that documents are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

**Dry Weight** - the weight of a sample based on percent solids. The weight after drying in an oven.

**Duplicate** - a second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

**Equipment Blank** - usually an organic or aqueous solution that is free of analytes and is transported to the sampling site, opened in the field, and poured over or through the sample collection device, collected in a sample container, and returned to the laboratory. This serves as a check on sampling device cleanliness.

**Extractable** - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include BNA, pesticide and PCB compounds.

**Field Blank** - a sample to which no analytes of interest have been added. It is transported to the sampling site and back to ensure that no contamination is introduced during shipment. This sample may be opened near the sampling location to determine if air-borne contaminants are contributing to the sample contaminations.

**Field Duplicate** - two samples, collected at the sample site, that are treated exactly the same throughout field and laboratory procedures. Analysis of field duplicates provides a measure of the precision associated with sample collection, preservation and storage, as well as with

laboratory procedures.

**Heavy Metals** - metallic elements with high atomic weights, such as mercury, chromium, cadmium, arsenic, and lead. They can damage living things at low concentrations and tend to accumulate in the food chain.

**Holding Time** - the maximum time allowable between sample collection and analysis or extraction.

**Hydrocarbons** - any of a series of chemical compounds that consist entirely of carbon and hydrogen.

**Initial Calibration** - the analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the instrument to the target compounds.

**Inspection** - An activity such as measuring, examining, testing or gauging one or more characteristics of an entity comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic.

**Instrument Blank** - A clean sample processed through the instrumental steps of the measurement process; used to determine instrument contamination.

**Instrument Performance Check** - a solution of method analytes, used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

**Interferents** - substances which affect the analysis for the analyte of interest.

**Internal Standards** - compounds added to every standard, blank, matrix spike, matrix spike duplicate and sample (for VOAs, and semi-volatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

**Laboratory Control Sample (LCS)** - A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyte-specific precision and bias or to assess the performance of all or a portion of the measurement system.

**Laboratory Duplicate** - Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

**Laboratory Fortified Blank (LFB)** - an aliquot of laboratory reagent water to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the method is in control and whether the laboratory is capable of making accurate and precise measurements.



**Limit of Detection (LOD)** - An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent.

**Limit of Quantitation (LOQ)** - The minimum levels, concentrations, or quantities of a target analyte that can be reported with a specified degree of confidence.

**Linear Dynamic Range** - (Inorganic Analysis) The concentration range over which the ICP or IC analytical curve remains linear.

**Matrix** - the predominant material of which the sample to be analyzed is composed. For most purposes, a sample matrix is either water or soil/sediment.

**Matrix Spike (spiked sample or fortified sample)** - A sample prepared by adding a known mass of target analyte to a specified amount of the matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

**Matrix Spike Duplicate (spiked sample or fortified sample duplicate)** - A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery for each analyte.

**Method Blank** - A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

**Method Detection Limit (MDL)** - the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is statistically determined from analysis of samples in a given matrix containing the analyte at a predefined low concentration level.

**National Environmental Laboratory Accreditation Conference (NELAC)** - A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP.

**National Environmental Laboratory Accreditation Program (NELAP)** - The overall National Environmental Laboratory Accreditation Program of which NELAC is a part.

**Negative Control** - Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

**Outlier** - an extreme observation that is shown to have a low probability of belonging to a

data population.

**Percent Difference (%D)** - An arithmetic calculation to compare two values. The percent difference indicates both the direction and the magnitude of the comparisons, i.e., the percent difference may be either negative, positive, or zero.

**Percent Moisture** - an approximation of the amount of water in a soil/sediment sample obtained by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at 105°C, in addition to water.

**Percent Solids** - the proportion of the solid in a soil sample determined by drying an aliquot of the sample.

**Performance Audit** - The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

**Performance Evaluation (PE) Sample** - a sample of known composition provided by a third party (unknown composition by the laboratory) used to evaluate laboratory performance.

**Positive Control** - Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

**Precision** - the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

**Preservation** - Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

**Proficiency Testing** - A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

**Proficiency Testing Oversight Body/Proficiency Testing Provider Accreditor (PTOB/PTPA)** - An organization with technical expertise, administrative capability and financial resources sufficient to implement and operate a national program of PT provider evaluation and oversight that meets the responsibilities and requirements established by NELAC standards.

**Proficiency Testing Program** - The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results,

statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.

**Proficiency Testing Study Provider** - Any person, private party, or government entity that meets stringent criteria to produce and distribute NELAC PT samples, evaluate study results against published performance criteria and report the results to the laboratories, primary accrediting authorities, PTOB/PTPA and NELAP.

**Proficiency Test Sample (PT)** - A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

**Protocol** - A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

**Purge and Trap (device)** - an analytical technique (device) used to isolate volatile organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

**Quality Assurance (QA)** - An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

**Quality Assurance Project Plan (QAPP)** - A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

**Quality Control (QC)** - The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

**Quality Control Sample** - A sample used to assess the performance of all or a portion of the measurement system. QC samples may be Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking.

**Quality Manual** - A document stating the management policies, objectives principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

**Quality System** - A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products, and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

**Random Error** - the deviation in any step in an analytical procedure that can be explained by standard statistical techniques.

**Raw Data** - Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study.

**Reagent Blank (method reagent blank)** - A sample consisting of reagents, without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all the subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

**Reagent Water** - water in which an interferant is not observed at or above the minimum reporting limit for the parameters of interest.

**Reconstructed Ion Chromatogram (RIC)** - a mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

**Recovery** - a determination of the accuracy of the analytical procedure made by comparing measured values for a fortified sample against the known spike values.

**Reference Material** - A material or substance one or more properties of which are sufficiently well established to be used for calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

**Reference Standard** - A standard, generally of the highest quality available at a given location, from which measurements made at that location are derived.

**Relative Percent Difference (RPD)** - An analytical technique used to compare two values. The relative percent difference is based on the mean of the two values, and is reported as an absolute value.

**Relative Response Factor** - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

**Replicate Analyses** - The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval.

**Resolution** - the separation between peaks on a chromatogram, calculated by dividing the height of the valley between the peaks by the average peak height of the two peaks being resolved, multiplied by 100.

**Retention Time Window** - usually defined as three times the standard deviation of the absolute or relative RT of an analyte, positioned around a defined absolute retention time.

**Sample** - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

**Sample Number** - (AAI Sample Number) - a unique identification number designated by Alpha for each sample. The sample number appears on the final report which documents information on that sample.

**Selectivity** - The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.

**Semi-volatile Compounds** - compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

**Sensitivity** - instrument response (counts, peak area, etc.) observed for the absolute quantity of analyte introduced into the instrument at the reporting limit.

**Serial Dilution** - the dilution of a sample when corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits.

**Significant Figures** - the number of digits used to express a result in scientific notation. All digits are expected to be known definitely, except the last digit, which may be in doubt.

**Spike** - A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

**Standard** - The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

**Standard Deviation** - the positive square root of the expected value of the square of the difference between a random variable and its mean.

**Standard Method** - A test method issued by an organization generally recognized as competent to do so.

**Standard Operating Procedure (SOP)** - a written document 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.

**Standard Reference Material (SRM)** - A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.

**Supervisor** - The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision

of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.

**Surrogate** - A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.

**System Monitoring Compounds** - Compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard for volatile analysis and used to evaluate the performance of the entire purge and trap, gas chromatograph/ mass spectrometer system. These compounds are brominated or deuterated compounds not expected to be detected in environmental media.

**Target Analyte** - specific analytes reported for every sample analyzed by a given method.

**Target Concentration** - known spiked concentration.

**Technical Director** - Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.

**Tentatively Identified Compounds (TIC)** - compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. These peaks are subjected to mass spectral library searches for tentative identification.

**Test Method** - An adoption of a scientific technique for performing a specific measurement as documented in a laboratory SOP or as published by a recognized authority.

**Traceability** - The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

**Tune** - An injected standard required by the method as a check on instrument performance for mass spectrometry.

**Twelve-hour Time Period** - the twelve (12) hour time period for GC/MS system tuning and standard calibrations which begins at the moment of injection of the DFTPP or BFB. The time period ends after 12 hours has elapsed according to the system clock.

**Validation** - The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

**Verification** - Confirmation by examination and provision of evidence that specified requirements have been met.

**Volatile Compounds** - compounds amenable to analysis by the purge and trap technique.

Used synonymously with purgeable compounds.

**Work Cell** - A well-defined group of analysts that together perform the method of analysis. The members of the group and their specific functions within the work cell must be fully documented.