Appendix A

Personnel

1.0 Personnel

1.1 General Requirements for Laboratory Staff

1.1.1 Technical staff positions are filled with personnel that fulfill the necessary requirements of education, training, technical knowledge, and experience for their assigned functions. This includes a general knowledge of laboratory operations, test methods, quality assurance/quality control procedures and records management.

1.1.2 In accordance with our training policies and procedures, Alpha maintains documentation that certifies technical personnel have the appropriate educational and/or technical background to perform all accredited test procedures.

1.2 Staffing Policies

1.2.1 It is Alpha's policy to have a laboratory organized with sufficient managerial staff with the authority and resources needed to discharge their duties.

1.2.2 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.

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

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

1.3 Personnel Responsibilities

1.3.1 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.

1.3.2 It is the responsibility of all staff personnel to comply with all quality assurance/quality control requirements that pertain to their technical function in the laboratory.

2.0 Personnel Qualifications

2.1 Technical Director

2.1.1 Definition

The technical director means a full-time member of staff who exercises day-to-day supervision of the laboratory operations which may include reporting of laboratory results for the associated fields of accreditation.

2.1.2 Working Status

Technical directors who are absent for a period of time exceeding 15 consecutive calendar days must designate another full-time staff member listed below meeting the qualifications of the technical director to temporarily perform this function. If this absence exceeds 65 consecutive calendar days, the primary accrediting authority is notified in writing.

2.1.3 Titles

The title of such person(s) may include but is not limited to:

a) Laboratory Director;
b) Technical Director;
c) Laboratory Supervisor; and
d) Laboratory Manager

Where staffing is limited the Quality Assurance Officer may also be the technical director.

2.1.4 Duties

Technical director(s) minimum duties may include:

a) monitoring standards of performance in QA/QC;
b) monitoring the validity of the analyses performed; and
c) monitoring the data generated to assure reliable data.

2.1.5 Qualifications of Technical Directors

2.1.5.1 Laboratory Director

The Laboratory Director must have a bachelors degree in chemical, environmental, biological sciences, physical sciences or engineering, with at least 24 college semester credit hours in chemistry and at least 2 years of experience in environmental analysis.

A masters or doctoral degree may be substituted for 1 year of experience or a BS degree in a science or science related field plus 10 years of analytical experience may be substituted for the chemistry credit requirement.

2.1.5.2 Laboratory Manager

The Laboratory Manager must have a bachelors degree in chemical, environmental, biological sciences, physical sciences or engineering and at least one year of experience in environmental analysis. The Laboratory Manager must have a working knowledge of quality assurance principals.

2.1.5.3 Qualifications of Quality Assurance Officer

The Quality Assurance Officer must have a bachelors degree in chemical, environmental, biological sciences, physical sciences or engineering with at least 24 college semester credit hours in chemistry and at least 2 years experience in environmental analysis.

A masters or doctoral degree may be substituted for 1 year of experience or a BS degree in a science or science related field plus 10 years of analytical experience may be substituted for the chemistry credit requirement.

2.3 Qualifications of Technical Staff

2.3.1 It is the responsibility of Alpha's management to formulate the goals with respect to education, training and skills of the technical and non-technical staff members.

2.3.2 Alpha's training program specifies the training policies and procedures for identifying the training needs and providing training of personnel. The qualification of all staff members is a combination of education, experience and training and are critical element in maintaining the qualifications of our staff.

2.3.3 Laboratory Analyst

The Laboratory Analyst must have a bachelors degree in science or a science related field and at least 1 year of experience in environmental analysis. If the analyst is responsible for the operation of analytical instrumentation, they must complete specialized training offered by the manufacturer or another qualified training facility or served a minimum of a six month apprenticeship under an experienced analyst.

2.3.4 Laboratory Technicians

The Laboratory Technician must have at least a high school diploma or

equivalent, completed the in-house training program under an experienced analyst/technician and must have served a minimum of a six month apprenticeship under an experienced analyst/technician.

2.3.5 Laboratory Apprentice

The Laboratory Apprentice is a laboratory analyst or technician who has not fulfilled either the educational or experience requirements and is performing the job duties of one of those positions. The apprenticeship requirements are judged on a case-by-case basis. However, all employees performing job duties as an Apprentice are required to be under the direct tutelage of a senior Analyst or Technician who reviews all data or work produced until such time that the educational and experience requirements can be met.

Appendix B

Field Sampling Plan

Appendix B

Standard Operating Procedure

SOP B.1
Field Sampling Plan

1.0 FIELD SAMPLING PLAN (FSP)

1.1 Alpha is not generally responsible for sample collection. For most sites, specific work plans or Quality Assurance Project Plans (QAPPs) and SOP's are developed and designed for each unique site. However, when Alpha does collect samples for analysis, it follows the procedures outlined in the procedures described in the Field Sampling Plan.

1.2 The FSP has been written to assure uniformity and consistency of the sampling procedures. The FSP should be implemented at the sampling site when no other field sampling plan has been developed by the engineers or field samplers. These procedures should be performed during all phases of the sampling plan.

1.3 The objective of the FSP is to discuss subjects and sampling protocols that need to be controlled in the field to ensure the validity of the test results. Generalized field procedures used in the FSP are described by SOP's to be followed while performing field work.

1.4 The scope of the FSP includes descriptions of sampling documentation, sampling procedures, decontamination procedures and field QC procedures. Discussions of data quality objectives, laboratory quality control, sample labeling, shipment and custody records, and quality assurance oversight are provided in the Quality Assurance Manual.

1.5 Management Review of Potential New Work

1.5.1 Before sampling is performed at any site, the Project Manager should meet with a representative of Alpha Analytical to establish the sampling methodology to be employed, and the tests which will be performed on the samples. A sample collection plan should be determined, areas of responsibility delineated and a logistical plan developed.

1.5.2 After the planning meeting, the appropriate laboratory personnel are alerted of the need for specific laboratory analyses. Laboratory personnel are then provided with the information required to satisfy all specifications outlined in the Statement of Work (SOW) or QAPP. In addition, the logistical criteria such as sample containers, sample volume, preservatives, transportation and the appropriate numbers and types of QC samples needed is finalized, and finally the laboratory is assessed to ensure we have the appropriate facilities and resources to complete the project.

Appendix B

Standard Operating Procedure

SOP B.2
Field Sampling Documentation

1.0 FIELD SAMPLING DOCUMENTATION

1.1 Sample integrity begins from the time the sample is actually taken. Typically, a bound logbook is used to record all pertinent information regarding the sampling trip to ensure proper and complete documentation.

2.0 STANDARD OPERATING PROCEDURE

2.1 The logbook must contain information to distinguish samples and sample locations from any other. Entries into the logbook should include the following:

- Client/Project name for which the sampling is being conducted,
- Matrix sampled (i.e., groundwater, soil, etc.),
- Specific sampling location in sufficient detail to allow the re-sampling at the same location (diagrams, maps etc.),
- Sampling date and time,
- Environmental conditions (if relevant),
- Sample depth,
- Sample procedure,
- Volume of water removed during well purge,
- Sample type taken (e.g., duplicate, split or field sample etc.),
- Analytes or methods of interest,
- Preservation techniques,
- Field sample ID,
- Significant observations made during the sampling process,
- Field measurements taken,
- Statistics the sampling procedure was based on (if appropriate),
- Decontamination procedures, and
- Printed name and signature of the person performing the sampling.

2.2 Sampling situations vary widely and no general rules can specify the extent of information that must be entered in a logbook or standardized form. However, records should contain sufficient information so that someone can reconstruct the sampling activity without relying on the collector's memory.

Prior to the collection of any samples, the sampling location should be verified using site maps if available. If discrepancies are noted, sampling locations should be verified with the Project Coordinator before sampling.

2.3 If the client requires deviations, additions or exclusions from the documented sampling plan or from a particular sampling procedure, this activity is to be recorded in detail along with the appropriate sampling data. This information is to be included in all documents containing environmental test results and the deviations are to be communicated to the appropriate personnel.

Appendix B

Standard Operating Procedure

SOP B.3
Quality Control Field Samples

1.0 QUALITY CONTROL FIELD SAMPLES

1.1 The following quality control samples should be collected to ensure the reliability and integrity of field and analytical data for water and soil samples.

2.0 STANDARD OPERATING PROCEDURE

Section 11 of the QA plan addresses both laboratory and field QC samples to include purpose, frequency, preparation procedures, acceptance criteria, and corrective actions.

2.1 Water QC Samples

Table B.3-1 lists the number and types of aqueous field QC samples that should be collected for each sampling event.

2.1.1 The same type of QC samples should be collected for groundwater and surface water samples. Field duplicates and three types of quality control blanks are typically used (trip blanks, field blanks and equipment blanks).

2.1.2 Trip Blanks are typically only prepared when VOC field samples are taken and consist of a sample bottle filled with organic free water. Trip blanks are prepared in the laboratory and sent out in the field. Trip blanks are transported to the site in the same ice chest used in the field and transported back to Alpha along with samples obtained in the field.

2.1.3 Equipment and/or Rinsate Blanks are used to ensure non-dedicated sampling devices, e.g., bailers, filtering equipment, pumps, etc. have been effectively decontaminated. Field equipment is rinsed with reagent free water, collected and transferred to a sample bottle for future analysis.

2.1.4 Field blanks consist of reagent free water placed in a sample container at the laboratory and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation and all analytical activities. The purpose of the field blank is to determine if method analytes or other interferences are present in the field environment.

2.1.5 Duplicates should be collected and analyzed on a ten percent basis. The relative percent difference between duplicate measurements should provide an estimate of sampling precision.

2.1.6 Matrix spike/matrix spike duplicates are the only samples which requires additional sample volume. When determining sampling logistics make sure this is taken into consideration.

TABLE B.3 - 1
SUMMARY OF QUALITY CONTROL SAMPLES

	Field QC	
Sample Type	**Target Frequency %**	**Comment**
Trip Blank		1 per cooler for VOC's / Analyze as needed
Equipment/Rinsate Blanks	10%	1 per analysis per day / analyze as needed
Field Blanks		1 per source per event analyzed for VOC's as needed
Field Duplicates	10%	ensure adequate volume
	Laboratory QC	
Sample Type	**Target Frequency %**	**Comment**
Duplicate	5%	1 per analytical method
Blank		1 per analytical or extraction batch
Matrix Spike/Matrix Spike Duplicate	5%	1 per analytical method
Surrogate Spike	Method Specific	Method Specific
Laboratory Control Spike	5%	1 per analytical or extraction batch

2.2 Soil/Solid QC Samples

Table B.3-2 lists the number and types of soil field QC samples that should be collected for each sampling event.

2.2.1 Trip Blanks should be sent with each cooler when volatile organic analysis is required. The trip blanks should be analyzed for volatile organic comounds only.

2.2.2 Equipment and/or Rinsate Blanks should be used to ensure non-dedicated sampling equipment has been effectively decontaminated. Samples should be collected each day soil sampling is conducted. Only a representative number should be analyzed. Additional blanks may be analyzed if a problem is perceived.

2.2.3 Field Duplicates should be collected at a frequency of ten percent of the total number of soil samples collected. Field duplicates are collected and analyzed to determine the precision of field sampling.

TABLE B.3 - 2
SUMMARY OF QUALITY CONTROL SAMPLES

	Field QC	
Sample Type	**Target Frequency %**	**Comment**
Trip Blank		1 per cooler for VOC's / Analyze as needed
Equipment/Rinsate Blanks	10%	1 per analysis per day / analyze as needed
Field Duplicates	10%	ensure adequate volume
	Laboratory QC	
Sample Type	**Target Frequency %**	**Comment**
Duplicate	5%	1 per analytical method
Blank		1 per analytical or extraction batch
Matrix Spike/Matrix Spike Duplicate	5%	1 per analytical method
Surrogate Spike	Method Specific	Method Specific
Laboratory Control Spike	5%	1 per analytical or extraction batch

Appendix B

Standard Operating Procedure

SOP B.4
Volatile Sampling Technique - 524.2

1.0 VOLATILE SAMPLING TECHNIQUE - 524.2

1.1 This sampling procedure is written specifically for those samples requiring analysis by method 524.2 under the Safe Drinking Water Act.

1.2 Reference

Methods for the Determination of Organic Compounds in Drinking Water, USEPA, EMSL, Cincinnati OH, Supplement III; EPA-600/R-95/131, Method 524.2, Revision 4.1, 1995.

2.0 STANDARD OPERATING PROCEDURE

2.1 Collect samples in triplicate using 40mL clear VOA vials. If samples are suspected to contain residual chlorine add approximately 25mg of ascorbic acid per 40ml of sample to the sample bottle before filling, (pre-preserved in the laboratory).

2.2 Adjust the pH of all samples to < 2 at the time of collection (field preserve), but after dechlorination, by carefully adding two drops of 1:1 HCL for each 40mL VOA vial. Seal the sample vial and mix for 1 minute. Do not mix the ascorbic acid with HCL in the sample bottle prior to sampling.

2.3 When sampling for Trihalomethanes (THM's) analysis only, acidification may be omitted if sodium thiosulfate (3mg per 40ml sample) is used to dechlorinate the sample. This exception to acidification does not apply if ascorbic acid is used for dechlorination.

2.4 If a sample foams vigorously when HCL is added, discard the sample. Collect a set of samples but do not acidify them. These samples must be flagged as "not acidified" and must be stored at 4° C. These samples must be analyzed within 24 hours of collection time if they are to be analyzed for any compounds other than THM's.

2.5 Place the septum, Teflon side down, on the sample, and screw on the cap without dislodging the septa.

2.6 Invert the sample and lightly tap the lid to ensure the absence of entrapped air bubbles. If air bubbles are trapped in the vial, add additional sample until sample container is free of air bubbles.

2.7 As each VOA vial is filled, enter the sample information on the label and pack the vial in the shipping container at 4° C. Samples must be refrigerated at the time of collection and maintained at that temperature, analyze within 14 days of collection. Samples not received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C on arrival at the laboratory.

Table B.4 - 1
Sample Preservation and Holding time Table
Method 524.2

Description	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, 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

2.8 Never allow the sample to freeze during transportation. If samples are refrigerated with ice, pack the vials to ensure contact between the ice and sample vials are minimized to avoid potential freezing.

2.9 Never filter VOC samples.

2.10 Never sample for volatile organic compounds near a running motor or any type of exhaust system because discharged fumes and vapors may contaminate the sample.

2.11 VOC samples may also be contaminated by diffusion of volatile organics through the septa during shipment and storage. To monitor possible contamination, a trip blank prepared from reagent grade water, is carried throughout the sampling storage, shipping and analytical process. Additional QC samples may be collected (i.e., field blanks, rinse blanks, field duplicates, etc.), but should be considered on a case-by-case basis.

2.12 When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized usually about 5 to 10 minutes. Adjust the flow to about 500 ml/min and collect the samples from the flowing stream.

2.13 When sampling from an open body of water, fill a wide mouth bottle or a beaker with sample from a representative area, and carefully fill sample bottles from the container.

Appendix B

Standard Operating Procedure

SOP B.5
Volatile Sampling Technique - 624/8260

1.0 VOLATILE SAMPLING TECHNIQUE-WATER (624/8260B)

1.1 There are several requirements common to most VOC sampling events. Overall care must be taken in regard to equipment and, container handling, storage, decontamination, and record keeping.

1.2 Reference

1.2.1 Method 624: Purgeables, Federal Register 43250, Volume 49, NO.209, October 26, 1984 as updated in 40CFR Part 136, Appendix A.

1.2.2 Method 8260B: Volatile Organic Compounds by GC/MS, Revision 2, December 1996, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, 1996.

2.0 STANDARD OPERATING PROCEDURE

2.1 Sample Collection

Samples must be collected in glass containers with zero head-space. At a minimum, aqueous samples should be collected in 40 mL (nominally 43 mL) VOA vials in triplicate. While sampling, completely fill sample vials full such that they form a meniscus at the top of the vial taking care not to flush out the preserving agents.

2.1.1 Sample vials should not contain any bubbles exceeding 5 - 6mm (pea sized) as they may cause significant degassing and loss of volatiles.

2.1.2 Place the septa, teflon side down, on the sample, and screw on the cap with out dislodging the septa.

2.1.3 Invert the sample and lightly tap the lid to ensure the absence of entrapped air bubbles. If air bubbles are trapped in the vial, add additional sample until sample plus the duplicate vials are free of air bubbles.

2.1.4 Never filter VOC samples.

2.1.5 Never sample for volatiles near a running motor or any type of exhaust system because discharged fumes and vapors may contaminate the sample.

2.1.6 VOC samples may be contaminated by diffusion of volatiles through the septa during shipment and storage. To monitor possible contamination, a trip blank is carried throughout the sampling, storage, shipping and analytical process. Additional QC samples may be collected (i.e., field blanks, rinse blanks, field duplicates, etc.), and should be considered on a case-by-case basis.

2.1.7 When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized usually about 5 to 10 minutes). Adjust the flow to about 500 ml/min and collect the samples from the flowing stream.

2.1.8 When sampling from an open body of water, fill a wide mouth bottle or a beaker with sample from a representative area, and carefully fill sample bottles from the container.

2.1.9 If protective gloves are used, they should be clean, new, disposable, and nitrile. Gloves should be changed between sampling events to prevent the possibility of cross-contamination. Care should be taken to prevent the sample from touching the gloves while filling the containers or touching the inside of the container with the gloves.

2.2 Sample Dechlorination

If samples contain residual chlorine they must be dechlorinated with sodium thiosulfate prior to sample acidification.

2.2.1 Method 624 suggests 10 mg per 40 ml sample volume is sufficient to dechlorinate a water sample which contains up to 5 mg/L residual chlorine. If a sample contains residual chlorine greater than 5 mg/L, sodium thiousulfate is added proportional to the concentration of residual chlorine.

2.2.2 Method 8260B suggests 0.008% (3.2mg per 40ml) sodium thiosulfate.

2.3 Sample Preservation

Adjust all samples to a pH of < 2, by carefully adding, a suggested volume, of four drops of 1:1 HCl for each 40 ml vial. Seal the vial and mix by inverting the vial several times. Sample vials may be preserved prior to field sampling for ease of use; however, it is incumbent upon the sampler to ensure the proper pH has been achieved.

2.3.1 Method 624 - acidification is only required in sample being analyzed for the aromatic compounds.

1) Clarification: All samples should be preserved with HCl acid regardless of method. Samples not preserved with HCl should be analyzed within 24 hours or footnoted accordingly.

2.4 Sample Storage

2.4.1 Both water and soil samples should be refrigerated at ≤6°C from the time of collection. Samples that will not be received at the laboratory on the day of

collection must be packaged for shipment with sufficient ice to ensure a temperature of ≤6°C will be maintained on arrival at the laboratory.

2.4.2 Never allow the samples to freeze during transportation. If samples are refrigerated with ice, pack the vials to ensure contact between the ice and sample vials are minimized to avoid potential freezing.

2.5 Sample Holding Time

Water samples must be analyzed within fourteen days of sample collection. Soil samples must be extracted and analyzed within fourteen days of sample collection.

Note: See VOC appendix for soil sample variations by method 5035A.

2.5.1 Method 624 - samples which are not being analyzed for aromatic compounds and are non-acidified must be analyzed within 7 days of sample collection.

Sample Collection, Preservation and Holding Time for Volatile Organics
Table B.5-1

Method	Matrix	Sample Collection Container	Volume Sample	Preservation	Holding Time
624	Water	40mL VOA vial, glass with Teflon septa.	3x40mL	pH<2; HCl, Cool ≤ 6°C 10mg per 40mL $Na_2S_2O_3$	14 days to analyze
624 (Non-Aromatics only	Water	40mL VOA vial, glass with Teflon septa.	3x40mL	Cool ≤ 6°C 10mg per 40mL $Na_2S_2O_3$ NO ACID	7 days to analyze
8260B	Water	40mL VOA vial, glass with Teflon septa.	3x40mL	pH<2; HCl, Cool 4°C 0.008% $Na_2S_2O_3$	14 days to analyze
8260B	Solid	Brass tube or 8oz wide mouth glass container	Brass tube or 8oz glass jar	Cool 4°C	14 days to extract and analyze See note above

Appendix B

Standard Operating Procedure

SOP B.6
Soil Sampling Technique

1.0 SOIL SAMPLING TECHNIQUE

1.1 Soil or sediment samples are typically collected in 8oz/250 ml or 4oz/125mL wide-mouth glass bottles according to the prescribed protocol below.

2.0 STANDARD OPERATING PROCEDURES

2.1 Fill each container, as a grab sample or by using a sampling device, with the soil sample. Avoid aeration of the sample to minimize the loss of volatile organic compounds if VOC methods are to be taken from the same sampling containers.

2.2 As each container is filled, enter the applicable information on the label and pack the bottle in the shipping container at ≤6°C. Samples must be refrigerated at the time of collection and maintained at that temperature until analysis. Samples not received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure a temperature of ≤6°C on arrival at the laboratory.

2.3 Never sample near a running motor or any type of exhaust system because discharged fumes and vapors may contaminate the sample.

2.4 Samples may also be contaminated by the use of non-dedicated sampling equipment such as a scoop or split sampling device. To monitor possible contamination a rinse blank and/of equipment blank should be collected each day soil sampling is conducted and analyzed for the compounds of interest.

2.5 If protective gloves are used they should be clean, new, disposable, and nitrile. Gloves should be changed between sampling events to prevent the possibility of cross-contamination. Care must be taken to prevent the sample from touching the gloves while filling the containers or touching the inside of the container with the gloves.

Appendix B

Standard Operating Procedure

SOP B.7
Semi-volatile Water Sampling Technique

1.0 SEMI-VOLATILE SAMPLING TECHNIQUE - WATER

1.1 When sampling water for semi-volatile organic compounds, samples are typically collected in 1 liter amber bottles according to the prescribed protocol below. In addition to the following procedures, overall care must be taken in regard to equipment and container handling, storage, decontamination, and record keeping.

2.0 STANDARD OPERATING PROCEDURE

2.1 Collect all samples in duplicate by slowly filling each container, minimizing the amount of sediment collected, so that the head space is no greater than the threaded portion of the neck.

2.2 Add the appropriate preservatives to the samples as described in the QA manual. It is important the right amounts of preservative be added by checking the pH, residual chlorine etc. for this verification. Cap the bottles with a Teflon lined cap and invert the sample to ensure the preservatives are well dispersed in the sample.

2.3 As each bottle is filled, enter the applicable information on the label and pack the bottle in the shipping container at $\leq 6°C$. Samples should be refrigerated at the time of collection and maintain refrigeration until extraction. Samples not received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure a temperature of $\leq 6°C$ is maintained on arrival at the laboratory.

2.4 Never allow the sample to freeze during transportation. If samples are refrigerated with ice, pack samples such that contact between the ice and sample bottles are minimized to avoid potential freezing.

2.5 Precautions must be taken to limit the contamination of samples from outside sources. Hands should be washed and gloves worn prior to sampling. The order of sampling should be from the least contaminated well to the most contaminated well.

2.6 When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 5 to 10 minutes). Adjust the flow to about 500 ml/min and collect the samples from the flowing stream.

2.7 Samples from springs, surface water or other open bodies of water should be collected as grab samples. These samples should be carefully collected to minimize the turbulence and amount of sediment collected with the water samples.

2.8 If protective gloves are used, they should be clean, new, disposable, and nitrile. Gloves should be changed between sampling events to prevent the possibility of cross-contamination. Care must be taken to prevent the sample from touching the gloves while filling the containers or touching the inside of the container with the gloves.

Appendix B

Standard Operating Procedure

SOP B.8
Ground Water Sampling - Monitoring Wells

1.0 GROUNDWATER SAMPLING - MONITORING WELLS

1.1 Monitoring wells are particularly important to understanding the hydrology and remediation efforts at a project site. Well information is used to define the geochemical baseline and it is therefore extremely important to sample each well exactly the same. The following procedures incorporate the necessary aspects of sampling QA and should be used each time a monitoring well is sampled.

2.0 STANDARD OPERATING PROCEDURE

2.1 Whenever feasible, wells not expected to be contaminated should be sampled first, followed by wells with increasing levels of contamination.

2.2 In most wells, a submersible pump, if available, should be used to facilitate purging. The pump is generally powered by a portable generator. The generator must be operated down wind and as far away as possible from the actual sampling location. Actual samples to be submitted for analysis should be taken by a bailer. In low-yield wells, purging and sampling should be conducted by bailing.

2.3 Immediately prior to well purging and sample collection activities, the static water level below the top of the well casing should be measured with an electronic sounder and recorded in the logbook. Water levels should be recorded to the nearest 0.01 foot.

2.4 Measure and record the depth from the top of the casing to the bottom of the sediment/water interface. Subtract the depth to top of the water from the depth to the bottom of the sediment/water interface and determine the height of standing water in the casing.

2.5 Measure the well diameter and determine the water volume using the following equation:

$$\text{well volume} = (pi)\ (r^2)\ (h)$$

Where;

r = radius measured in feet
h = height of water measured in feet
pi = 3.141

Well volume calculated in cubic feet. The conversion of cubic feet to gallons is:

$$1\ ft^3 = 7.48 \text{ gallons}$$

2.6 Prior to sample collection purge three well volumes from the well. Purging and sampling should be performed in a manner that minimize the agitation of sediments in the water column and to reduce the potential of organic chemical volatilization.

2.7 If the well goes dry during purging, it is assured of removing all water which has prolonged well casing or air contact. If the recovery rate is quick, allow the well to recover to its original level before subsequent purging of the well. If water recovery is very slow, samples should be taken when sufficient water is available.

2.8 Samples for volatile organics should be collected first and immediately sealed in 40ml VOA vials with no head space. All samples should be checked for the presence of bubbles that may bias the analytical data. Samples for volatile organics should not be homogenized, composited or filtered.

2.9 All samples should be taken in pre-cleaned containers. Semi-volatile samples should be placed in 1 liter amber glass bottles in duplicate, volatile organic samples should be sampled in three 40 ml VOA vials and metals or inorganic parameters should be sampled in 125 to 1000 ml polyethylene containers. Add the appropriate preservatives and cap the sample containers for storage and transportation to the laboratory.

2.10 Refrigerate all samples in an ice chest at $\leq 6°C$ immediately after sampling and deliver to the laboratory as soon as possible.

2. 11 In addition to the record keeping requirements of the QA plan, the following information should be recorded each time a well is purged and sampled:

- Depth to water before and after purging,
- Well casing volume calculations,
- Condition of each well,
- Apparent thickness of any floatable hydrocarbon layer; and
- Any required field parameter (i.e., pH, EC, temperature).

2.12 All non-dedicated purging and sampling equipment should be decontaminated between wells. In addition, non-dedicated bailers should be rinsed once with well water prior to collecting a sample.

Appendix B

Standard Operating Procedure

SOP B.9
Field Decontamination and Waste Disposal

1.0 FIELD DECONTAMINATION AND WASTE DISPOSAL

1.1 Sampling equipment must be cleaned prior to and after each use to minimize cross contamination of samples. Good house-keeping practices are reflected in the analysis of clean equipment or rinsate blanks.

2.0 STANDARD OPERATING PROCEDURE

2.1 Equipment Decontamination

2.1.1 Sampling equipment should be cleaned prior to sample collection, in a controlled environment, preferably at the laboratory and transported to the field pre-cleaned and ready to use.

2.1.2 Sampling equipment should be cleaned between sample locations, and at the end of sampling activities. Large HDPE drums should be used to hold wash and rinse solutions.

2.1.3 The following general decontamination procedure should be used when collecting field samples:

- Clean all sampling equipment with tap water and a non phosphate detergent such as Alconox or Detergent-8. Brush if necessary to remove particulate matter or surface film;
- Rinse thoroughly with tap water;
- Rinse thoroughly with deionized or distilled water. Enough water should be used to insure all surfaces are flushed with water;
- Rinse twice with hexane or methanol. One rinse may be used as long as all surfaces are thoroughly wetted with free flowing solvent;
- Rinse thoroughly with deionized or distilled water;
- Rinse with organic free water and allow to air dry; and
- Clean sampling equipment should be wrapped in aluminum foil or protected by other means to prevent contamination during storage or transportation to the field.

2.2 Disposal of Field Waste Material

2.2.1 All discarded material or other objects should be handled in a way to minimize waste and to preclude the potential for spreading contamination or causing

litter to be left on-site. The major waste material generated during field activity is purged well water and fluids collected during decontamination.

2..2.2 Well Purge Water

All water collected from monitoring wells should be collected in approved containers. The containerized liquids should be handled according to the analytical results of the well water. Those waters purged from existing wells showing the presence of contaminants, should also be containerized and handled accordingly.

2.2.3 Decontamination Water

Similar to purged well water, water generated from decontamination activities should also be containerized, sampled and handled according to approved disposal procedures.

Appendix B

Standard Operating Procedure

SOP B.10
Sample Packing and Transportation

1.0 SAMPLE PACKING AND TRANSPORTATION

1.1 Samples taken in the field must be packaged in a manner to prevent breakage, or cross-contamination during transportation to the laboratory. Samples must be refrigerated with sufficient ice to ensure a temperature of < 6°C on arrival at the laboratory.

2.0 STANDARD OPERATING PROCEDURE

2.1 Samples should be segregated by site, sampling location, or by sample analysis type during the packing of coolers. Sample segregation may follow this scheme or any other scheme that is sensible and well thought out. These schemes are dependent on the levels of contamination present, the number of bottles to be transported, the size of the bottles etc.

2.2 VOC samples from different sources may be placed into the same cooler to reduce the number of required trip blanks.

2.3 All samples requiring thermal preservation should be packed in thoroughly insulated coolers with wet ice.

2.4 Samples in breakable containers should be packed with material such as bubble wrap or foam sleeves to prevent breakage.

2.5 Sample coolers should be sealed with strapping tape or other means to prevent tampering. Custody seals may also be placed on the container lids.

2.6 Packed samples should be delivered to the laboratory by the sampling team or common carrier. If sent by a common carrier, all documentation (transmittal forms, bills-of-lading, COC's etc.) should be sealed and placed inside the shipping containers prior to sealing it. It is recommended to place all forms inside a plastic bag and tape it to the underside of the cooler lid.

2.7 Secondary Containment

2.7.1 To prevent melted ice from leaking out of the sample cooler during transportation, it is recommended to encapsulate all samples and cooler ice in a garbage bag. This is accomplished by double bagging the ice-chest and unfolding the garbage bags lining the bag in the cooler. Place all samples, packing material etc. in the first of the two garbage bags. This first or inner garbage bag is then pigtailed and tied.

2.7.2 If a temperature blank is to be used, place the blank in the second garbage bag. To prepare a temperature blank place a temperature blank which contains a thermometer in a 40 ml VOA vial into a larger container, preferably a 500

ml HDPE (plastic) container. The plastic container filled with water acts as secondary containment as well as providing a much larger thermal mass to help stabilize the temperature readings. Finally, pre-cool the temperature blanks prior to placement into the coolers. Reducing the thermal mass of a volume of water from ambient temperature to < 6°C takes approximately 4 to 6 hours. Therefore pre-cooling the temperature blank will help expedite the thermal transfer.

2.7.3 Place crushed ice not block ice in the second garbage bag, filling the remaining space of the cooler to produce contact with the ice and the samples that are in the first garbage bag.

2.7.4 Tie the inner garbage bag which contains the samples to ensue any breakage or spillage will be encapsulated in this container.

2.7.5 Finally, tie the outer garbage bag which now contains: a) the inner garbage bag with field samples, b) the temperature blank, and c) the loose ice and prepare to ship the cooler to the laboratory.

Appendix C

Sample Tracking Plan

Appendix C

Standard Operating Procedure

SOP C.1
Sample Tracking Plan

1.0 SAMPLE TRACKING PLAN (STP)

1.1 The Sample Tracking Plan is a set of SOPs designed and written specifically for maintaining the integrity of a sample throughout its life at Alpha. These SOPs are strictly followed during all phases of the sample's stay at Alpha.

1.2 The STP includes procedures for the transportation, receipt, handling, protection storage, and disposal of samples, including the procedures necessary to protect the integrity of the sample, and to protect the interests of the laboratory and client.

1.3 Sample tracking and the maintenance of custody starts with the sample collection activities in the field and continues throughout the sample's progress in the lab. Sample collection is the responsibility of the field sampler; however, once the samples arrive at the laboratory, sample custody is turned over to our laboratory.

1.4 We cannot control the custody or the maintenance of custody until it arrives at our laboratory. Therefore, standard chain of custody procedures are typically used for all samples unless otherwise required.

1.5 Sample integrity is an equally important factor in all samples, regardless of sample type.

1.6 The Sample Custody Officers (SCO) is responsible for the implementation of the STP. This responsibility includes assuring that the proper handling and documentation of all samples are performed according to the SOPs described in this plan. Occasionally, a QAPP or SOW will require additional or different procedures to be followed for their samples.

Appendix C

Standard Operating Procedure

SOP C.2
Sample Identification

1.0 SAMPLE IDENTIFICATION

1.1 Alpha Analytical, Inc. has developed a system for uniquely identifying samples to ensure traceability of samples while in the possession of Alpha Analytical, Inc. and to maintain sample identity while in-house. Each sample received is assigned a unique lab ID number.

2.0 STANDARD OPERATING PROCEDURE:

2.1 The sample identification assigned to a sample is retained throughout the life of the sample at the laboratory.

2.2 The sample identification system is specifically designed and operated to ensure that samples cannot be confused physically or when referred to in records or other documents.

Once samples have been assigned a sample identification number, this identification is retained for the sample, sub-samples, subsequent extracts, and/or digestates related to that original sample.

2.3 The sample identification system is specifically designed to accommodate the grouping of sample (e.g., SVOC grouping from TPH-P) and the transfer of samples within and from the laboratory.

2.4 The Alpha Analytical Identification (AAI) is generated to assign a unique identification code to each sample container received by the laboratory.

1) Clarification: NELAC notes the use of container shape, size, or other physical characteristics, such as amber glass, or purple top is an unacceptable practice. Since we use a unique ID system this is not an issue but is stated only to make the point.

2.5 The AAI number assigned to each sample container is also the same ID used to unequivocally link the sample containers, extracts, etc. to the ID assigned in the field by the sampling team.

This link is unequivocally established using the Chain of Custody where the relationship between the field ID and laboratory ID are defined.

2.6 The laboratory ID is placed on the sample container using a durable sample label.

2.7 Sample containers and extract vials are labeled with a lab ID number in the following format:

XXXYYMMDDSC- ZZ

Where:

XXX represents a 3-letter prefix unique to each individual client,

YY refers to the last two digits of the year,

MM refers to the month,

DD refers to the day,

SC refers to the sample custodian who logged in the sample, and

ZZ refers to the sample number

For example: JDI11011030-05 would be a sample belonging to John Doe Incorporated. The sample was received on January 10, 2011 and logged in by the assistant SCO assigned to ID number 30. The -05 means this is the fifth sample in the sample set.

2.8 This lab ID number is used by Alpha Analytical for continuous identification of the sample from receipt to completion of analysis. Samples which are received as a fraction or subsequently extracted in the laboratory are identified using the lab ID number and a suffix identifying the sample type, whether its a matrix spike, matrix spike duplicate, etc.

Appendix C

Standard Operating Procedure

SOP C.3
Labeling Field Samples

1.0 LABELING FIELD SAMPLES

1.1 To ensure sample integrity, Alpha uses company labeled tags on samples. This tag is designed to make all entries visible on a white background, and is used to discourage the use of non-waterproof labeling material.

2.0 STANDARD OPERATING PROCEDURE:

2.1 Alpha provides customized waterproof labels to be used in the field. This is the preferred procedure; however, not all clients use our containers or sampling materials.

2.2 These labels should be affixed to the sample containers and completed using waterproof ink. If necessary, clear tape can be placed over the label. Alpha encourages the use of these labels to help our staff visually check and verify labels through continuity and standardization.

2.3 Each label is documented with the following information:

a) Analysis requested,
b) Preservation type,
c) Sample location,
d) Clients identification,
e) Date and time sampled, and
f) Alpha's sample identification.

2.4 Not all information is essential on the label. However, the following information is strongly recommended:

- Preservation Type,
- Client's Identification, and,
- Date and Time sampled.

Appendix C

Standard Operating Procedure

SOP C.4
Sample Receiving and Project/Client Communication

1.0 SAMPLE RECEIVING AND PROJECT/CLIENT COMMUNICATION

1.1 Sample receiving and project/client communication go hand-in-hand and is difficult to separate the two activities since one is associated with the other. The following synopsis discusses, in general terms, the primary activities associated with these procedures. The procedural order may vary somewhat, but this is generally the proper sequential order used to receive in-coming samples.

2.0 STANDARD OPERATING PROCEDURE

2.1 Background

2.1.1 Client Information

Specific project information is communicated throughout the laboratory using the "Client Information" menu in the Omega database. Whenever a change/policy is made by the client, it is recorded in this menu. The specific policy changes are entered and the name of the person making the request is recorded. The person typing the requests initials and dates the entry to indicate who typed the change and when the change was made.

2.1.2 Work Order Information

All client contacts for a particular work order are typed in this menu of the Omega database, initialed and dated by the person typing the request. The work order information is then printed and placed in the main file folder. This menu is similar to the Client Information screen; however, this screen is used for a particular work order only and the Client Information screen is used for policies that apply to entire projects.

2.1.3 Sample Receipt Checklist

A sample receipt checklist is automatically created for every work order/chain-of-custody that comes into the laboratory. After logging the samples into the Omega database, the sample receipt checklist is printed and faxed or e-mailed to the client immediately. The checklist identifies any sample integrity issues associated with a particular group of samples or work order and gives the client instructions on communicating with the laboratory how to resolve those abnormalities/nonconformities.

2.2 Sample Receiving Procedure

2.2.1 Alpha's manual chain-of-custody is completed and signed by both the relinquishing and receiving parties.

2.2.2 Samples are given a unique sample identification number.

2.2.3 Information described on the manual chain-of-custody is entered into the Omega LIM System.

2.2.4 Samples are labeled.

2.2.5 Documentation of Sample Integrity and Compliance

Upon receipt of the sample, the condition, including any abnormalities or departures from normal or specified conditions in the environmental test method, is recorded on the sample receipt checklist. The client is consulted and the conversations documented by the use of the sample receipt checklist for the following general items:

- When there is doubt as to the suitability of a sample for a particular environmental test,
- When a sample does not conform to the description provided, or
- When the test required is not specified in sufficient detail.

If doubt exist, the sample receipt personnel, consults the client for additional instructions and records those discussions before completing the sample receipt protocols.

2.2.6 A master file and method specific colored file folders are created which include a LIMS generated sample report indicating the target analyte list.

2.2.7 If the client has specific requirements associated with a work order, the client's comments are typed into the LIMS "Work Order" or "Comments" information section and this is printed out and placed into all associated files.

2.2.8 Samples and file folders are disseminated throughout the laboratory.

2.2.9 A copy of the chain-of-custody and the sample receipt checklist is faxed or e-mailed as a pdf file to the client.

2.2.10 The faxed or e-mailed confirmation report is placed into the master file.

2.2.11 If the chain-of-custody and/or sample receipt checklist sent by fax or e-mail to the client does not initiate changes to the chain-of-custody within one day, that is confirmation the chain-of-custody and associated information is correct without error.

If the fax or e-mail of the chain-of-custody and/or sample receipt checklist initiates a response, then those phone conversations, amendments to the chain-of-custody or other communications are annotated to the master and associated method files.

2.3 Subcontract Laboratories

It is Alpha's policy to subcontract out analytical services not performed by Alpha, to laboratories that have been certified by the appropriate state agencies, methods, and programs to the best of our ability.

2.3.1 If samples can not be analyzed in-house, then the sample custodian must determine if the client has specified a subcontract laboratory.

2.3.2 If a subcontract laboratory has not been identified by the client, then the sample custodian must review the subcontract laboratory register and determine which laboratory is most suitable to subcontract the work to.

This determination should take into account such things as; appropriate laboratory certification, methods of analysis, capabilities, is the subcontract laboratory capable of receiving the samples and reporting the data with the specified data quality objectives etc.

1) Clarification: The DoD requires subcontract laboratories meet the requirements of the DoD QSM. In addition, these subcontract laboratories must also be accredited by DoD and have received project-specific approval from the DoD client prior to sample analysis.

2.3.3 During the sample receipt process, samples are identified and segregated to be subcontracted. All sample receipt and COC protocols are followed for subcontracted sample analysis.

2.3.4 After the subcontract laboratory has been chosen, a subcontract chain-of-custody and a subcontract sample receipt checklist are sent along with the samples.

2.3.5 Once the samples have been received in the subcontract laboratory's facility, a return copy of the sample receipt checklist is requested.

Alpha request this checklist be returned to document and identify any potential abnormalities/nonconformities with the samples and gives the sub-contract laboratory instructions on communicating with our SCO if the samples are acceptable or not.

Thus we have confirmation regarding the subcontracted samples' receipt,

preservation, holding time and any other potential problems prior to the commencement of sample analysis by the subcontractor.

2.4 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.

2.4.1 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.

2.4.2 Service to clients is a proactive engagement with our clients which requires staff to notify clients of problem situations such as:

a) incorrect, obsolete 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 documents, such as a QAPP, or the need for clarification of requirements in the document; and

d) problems with sampling or analysis that may impact sample results (e.g., improper preservation of sample).

2.5 Customer Complaints

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

2.5.1 Customer complaints are directed to the sample custodian supervisor. These complaints are documented in the customer complaint logbook. Customer complaint documentation includes information such as:

a) client name,
b) date,
c) complaint,
d) information on who received the complaint,
e) a remedy of those complaints, and
f) initials of the Sample Coordinator or person receiving that complaint.

2.5.2 If complaints can be resolved immediately than the remedy for that particular complaint is documented. Conversely, if the complaint cannot be resolved immediately, and the remedy is more complicated, than the sample custodian supervisor relinquishes the duty of finding a remedy to the complaint to the laboratory manager.

2.5.3 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.

2.6 Document Confidentiality

2.6.1 All sample documents to include: telephone conversations, electronic data deliverables, faxes, or work order information is strictly confidential and will only be released to the client or Principal Investigator (PI) who originally requested the sample analysis.

Persons or organizations which request 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 releasing information. This is documented by the SCO, see Fig C.4-1.

REPORT RELEASE REQUEST

To: _____ **Fax:** _____

From: _____ **Date:** _____

RE: _____

Please respond

Work Order Number:

_____ is requesting the above listed report/s. Due to client confidentiality we are unable to release these report/s without your written permission. Please sign below and fax back to us at (775) 355-0406 if we are allowed to fax the above named report/s requested.

Thank you,
Alpha Analytical

_____
your signature

Fig C.4-1

Appendix C

Standard Operating Procedure

SOP C.5
Sample Containers, Preservation, Holding Times and General Sample Receipt Protocols

1.0 SAMPLE CONTAINERS, PRESERVATION, HOLDING TIMES AND GENERAL SAMPLE RECEIPT PROTOCOLS

1.1 Once samples are received it is important to verify and document the sample has maintained its integrity and is in compliance with the requested test method. This SOP is used in conjunction with the Sample Receipt Checklist to document sample integrity.

2.0 STANDARD OPERATING PROCEDURE

2.1 Introduction

Sample integrity issues are most often written into the methods of analysis. This would include such issues as sample collection, preservation and holding time. These items may be critical to the final data results and are important factors that must be addressed by the field sampler and verified in the laboratory.

For instance, if samples are collected and thermally cooled in the field, but had an inadequate amount of wet ice added and are received at the laboratory without ice and are warm; then sample integrity has been lost and the sample is noncompliant with the particular test method as data results may now be compromised.

2.2 Documentation of Sample Integrity and Method Compliance

Upon receipt of the sample, the condition, including any abnormalities or departures from normal or specified conditions in the environmental test method, is throughly checked and documented on the sample receipt checklist.

Sample containers, preservation, holding times, field and laboratory generated COC's, etc. are all reviewed and checked to verify sampling integrity and method compliance.

2.3 Corrective Actions for Non Compliant Samples

2.3.1 If the sample does not meet the sample receipt acceptance criteria, the SCO must retain any correspondence records of conversations concerning the final disposition of the rejected sample; or document the decision to proceed with the analysis of samples not meeting the acceptance criteria. These corrective action decisions are documented as follows:

i. The conditions of these abnormalities must be noted on the COC and/or on the sample receipt checklist.

ii. The sample analysis is appropriately footnoted on the final report.

2.3.2 If sample information errors are discovered, then these errors or discrepancies are recorded on the sample receipt checklist.

2.3.3 The client is faxed a copy or e-mailed the completed COC and the sample receipt checklist. Therefore, an unequivocal accurate record, which documents all laboratory activities is produced and maintained.

2.4 Sample-Receipt-Review-Items to Verify Sample Integrity and Method Compliance

2.4.1 Chain of Custody Information

The following items are reviewed by the SCO to verify the specific COC items are correct and documented.

2.4.1.1 SCO should review and verify the carrier name and ensure accompanying transportation documents are retained.

2.4.1.2 SCO should review and verify the field completed COC is present.

2.4.1.3 SCO should review and verify if custody seals are used and intact on the shipping container.

2.4.1.4 SCO should review and verify if custody seals are used and intact on individual sample bottles.

2.4.1.5 SCO should review and verify if COC was signed by the relinquishing party and signed by Alpha, the receiver.

Note: Sample custody begins with the field sampler. Therefore, technically the field sampler, should also be the first person to relinquish custody as documented on the COC.

2.4.1.6 SCO should review and verify if the COC agrees with the sample labels.

2.4.1.7 SCO should review and verify if the date and time of collection are noted by the client on the COC.

Note: Time stamps should be consistent within a COC; that is use either military or standard time for all data entries and not a mix of the two. If standard time is used, the use of a.m. or p.m. should also be used to avoid time confusions.

2.4.1.8 SCO should review and verify if an internal or evidentiary COC is requested or required.

2.4.1.9 SCO should review and verify if a subcontract laboratory is required and if so was a subcontract laboratory specified.

2.4.2 Sample Containers

The following items are reviewed by the SCO to verify the specific items relating to samples containers are correct and documented.

2.4.2.1 SCO should review and verify the cooler and the individual samples are intact and in good condition.

2.4.2.2 SCO should review and verify the COC completed in the field matches the sample containers for number of containers, types, sample identification etc.

2.4.2.3 SCO should review and verify sample container types and sizes are appropriate for the requested method of analysis.

2.4.2.4 SCO should review and verify the sample volume is appropriate for the requested method of analysis.

2.4.3 Thermal Preservation

The following items are reviewed by the SCO to verify the specific items relating to sample/cooler temperature are correct and documented.

2.4.3.1 SCO should review and verify that the samples are received at the correct temperature.

2.4.3.1.1 Temperature measurements are typically taken through the use of temperature blanks or by using an IR gun.

2.4.3.1.2 For samples with a specified temperature of 4°C, require samples with a temperature range from just above freezing temperature of water to 6°C is acceptable.

Note: The use of blue ice or reusable cold packs is not an acceptable alternative to wet ice.

2.4.3.1.3 Samples that are hand delivered to the laboratory on the same day they are collected may not meet these criteria. In these cases, the samples are considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

2.4.4 Chemical Preservation

The following items are reviewed by the SCO to verify the specific items relating to sample preservation are correct and documented.

2.4.4.1 SCO should review and verify that the samples are received with the correct preservation unless it is not technically acceptable to check preservation upon receipt. If any of the following conditions exist, chemical preservation should be checked at a later time, or rechecked in the laboratory:

a) continued preservation of the sample is in question (e.g., the sample may not be compatible with the preservation);

b) it is not technically acceptable to check preservation upon receipt (e.g., in the case of VOC samples); or

c) deterioration of the preservation is suspected.

2.4.4.1.1 Techniques for Verifying Sample Preservation

Samples that are preserved in the field are also checked at the laboratory. Essentially all samples are checked and verified prior to sample analysis or extraction/digestion. There are separate techniques to verify this without contaminating the samples. For instance:

2.4.4.1.1.1 There are multiple vials collected for VOC analysis. The first vial is used to verify and record the sample pH with pH paper. Since this vial is not used for final sample analysis, the pH strip can be dipped directly into the sample vial.

2.4.4.1.1.2 Aqueous samples which require extraction are checked for pH prior to any sample preparation typically using pH paper. The

sample pH and any necessary pH adjustments are then recorded on the sample preparation log.

2.4.4.1.1.3 TOC samples are the only universal exception to checking and verify sample preservation at the lab bench. Sample preservation for TOC is verified using pH paper at the time of sample receipt.

2.4.4.1.2 Prevention of Sample Contamination when Verifying Preservatives

Concern over possible contamination of the sample resulting from dipping a probe or a test strip into the sample suggests that a slight modification in how samples are obtained may be in order.

2.4.4.1.2.1 The most common suggestion to field samplers is to take an identical sample, and to use that sample to determine the preservation requirements. For example, if it is found that 2.3 ml of nitric acid is required to lower the sample to a pH <2, then the addition of the same amount of acid to the other sample containers will achieve the correct preservation, without contaminating the sample to be sent to the laboratory.

2.4.4.1.2.2 When only a limited number of sample bottles are taken, sample preservation is checked and documented by placing a sample drop using the disposable pipette or stir rod directly onto the pH paper. The pH paper is not dipped into the sample container in an effort to limit the possibility of contamination by the pH paper.

2.4.5 VOC Methods Requiring No Head Space

The following items are reviewed by the SCO to verify the specific items relating to VOC head space is correct and documented.

2.4.5.1 SCO should review and verify the VOC sample vials were received with zero head space.

2.4.5.1.1 Sample vials should not contain any bubbles exceeding 5 - 6mm (pea sized) as they may cause significant degassing and loss of volatile organic compounds. Conversely, sample vials containing bubbles smaller then 5-6 mm in size (total head space) are acceptable.

2.4.5.2 SCO should review and verify aqueous VOC samples were collected in 40 ml (nominally 43 ml) VOA vials in triplicate.

2.4.6 Sample Holding Time

The following items are reviewed by the SCO to verify the specific items relating to sample holding time is correct and documented.

2.4.6.1 SCO should review and verify that the samples were received within the method specified holding time.

2.4.6.2 SCO should review and verify that the samples have a sufficient amount of holding time left to conduct the sample analysis.

Appendix C

Standard Operating Procedure

SOP C.6
Sample Acceptance Policy

1.0 SAMPLE ACCEPTANCE POLICY

1.1 Once samples have been received by the laboratory, they are inspected to ensure they meet our sample acceptance policy. The sample acceptance policy is a series of sample inspection criteria outlined in the following procedure which defines the circumstances under which samples are accepted or rejected.

This inspection is documented thereby maintaining the documentation audit trail and is important to ensure the samples have maintained their integrity and are in compliance with the requested test method. This SOP is used in conjunction with the Sample Receipt Checklist to document sample integrity.

2.0 STANDARD OPERATING PROCEDURE

2.1 Data from any samples which do not meet the following criteria is flagged to clearly define the nature and substance of the variation. This policy is an important centerpiece for correctly recording the sample receipt checklist and COC documents.

2.2 Chain of Custody

2.2.1 The COC must be properly, fully and completely documented, which includes:

i. sample identification,
ii. sample or project location,
iii. date and time of sample collection,
iv. collector's name,
v. preservation type,
vi. sample type, and
vii. any special remarks concerning the sample.

2.2.2 See the following documents for additional details:

- QAM, Vol II, Appendix C, SOP C.7, Manual COC Procedures,
- QAM, Vol II, Appendix C, SOP C.8, LIMS COC Procedures,
- QAM, Vol II, Appendix C, SOP C.9, Internal COC Procedures.

2.3 Sample Labeling

2.3.1 Sample labels are completed using Alpha's sample identification scheme to uniquely identify and label all sample containers.

2.3.2 Sample labels used in the field should be water resistant and field samplers are encouraged to use indelible ink.

2.3.3 See the following documents for additional details:

- QAM, Vol II, Appendix C, SOP, C.2, Sample Identification, and
- QAM, Vol II, Appendix C, SOP, C.3, Labeling Field Samples.

2.4 Sample Containers - See the following documents for details:

- QAM, Vol I, Section 6.4 and Table 6-1 through Table 6-8.
- QAM, Vol, II, Appendix C, SOP C.5, Sample Receipt Protocols.

2.5 Sample Holding Times - See the following documents for details:

- QAM, Vol I, Section 6.6 and Table 6-1 through Table 6-8.
- QAM, Vol, II, Appendix C, SOP C.5, Sample Receipt Protocols.

2.6 Sample Preservation - See the following documents for details

- QAM, Vol I, Sections 6.3 and 6.5, and Table 6-1 through 6-8.
- QAM, Vol, II, Appendix C, SOP C.5, Sample Receipt Protocols.

2.7 Sample Volume - See the following documents for details:

- QAM, Vol I, Section 6.4 and 6.7, and Table 6-1 through Table 6-8.

1) Clarification: Since Alpha does not take field samples, all sampling supplies are furnished by Alpha to meet the sample acceptance policy criteria.

2.8 New Work

All new work is approved for sample acceptance by either the Laboratory Director or Laboratory Manager. This is a case-by-case approach and is not entirely a QA/QC issue. Items that affect the acceptance of new work include the following:

a) is the work in a state or under an agency with current laboratory approval;
b) are the requested methods of analysis, methods we are approved for;
c) are the target analytes, compounds we are approved for and analyze on a normal basis;
d) what are the reporting limits or LOQs requirements;
e) what are the QC requirements;
f) what are the turn around requirements;
g) what is the sample matrix;
h) what are the number of samples to be analyzed, and over what time period will they arrive;
i) do we have the appropriate facilities and resources before commencing the work; and

j) can the samples be analyzed without overly stressing the current staff.

This is not a comprehensive list of questions to be determined, but is a general starting point for all new projects. In addition, these do not answer the business questions regarding payment, pricing etc. which are always a factor when evaluating new work.

Appendix C

Standard Operating Procedure

SOP C.7
Manual Chain-of-Custody Procedures

1.0 MANUAL CHAIN-OF-CUSTODY PROCEDURES

1.1 The chain-of-custody can be regarded as a legal document in some situations and should be completely filled out and as error free as possible. All samples received by Alpha are entered into our Sample Tracking System to enhance the legal defensability of all data produced at Alpha. 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.

2.0 STANDARD OPERATING PROCEDURE

2.1 The Sample Custodian handles and/or processes samples dropped off at the laboratory or sample shipments, including pickup of samples at Reno-Tahoe International Airport, bus station, Federal Express, UPS, or other carrier service within Alpha Analytical's geographic area. The Sample Custodian is available to receive sample shipments at any time the delivery service is operating, including weekends.

2.1.1 Alpha's manual COC record is completed with the following information:

a) Client name, address, phone/FAX number,
b) Who the report should be addressed to,
c) Sampler,
d) Date Sampled,
e) Time of collection,
f) Matrix,
g) Lab identification number,
h) Client identification/sample description,
i) Number of containers,
j) Analysis requested, and
k) All necessary signatures/dates/times.
l) Billing Information
m) PO #, PWS #, Job # or any additional information

Note: The log-in person should be consistent when annotating times, (i.e., use either standard time or military time) and if standard time is chosen, use am and/or pm when applicable.

2.1.2 Examine the condition of the sample and note in the comments section:

a) The integrity of the sample container,
b) The amount of sample for the analysis,
c) The presence of air bubbles in VOA vials,
d) Whether the sample was preserved according to the method prescribed preservation, and
e) Note any other irregularities with sample condition.

2.1.3 Compare all documents (client vs. laboratory) to verify information. In the event errors are discovered, record the discrepancies in the remarks section.

2.1.4 Issue the yellow copy to the client, and retain the white copy as the laboratory's copy.

2.1.5 All entries made on this document must be filled out in ink.

2.1.6 Samples received in the absence of the Sample Custodian or other designated recipients are placed in their appropriate refrigerator until the correct personnel are available to process the samples according to established protocol.

Appendix C

Standard Operating Procedure

SOP C.8
LIMs Generated Chain-of-Custody Procedures

1.0 LIMS GENERATED CHAIN-OF-CUSTODY PROCEDURES

1.1 Once samples have been manually logged-in to the laboratory they are also entered into the LIM system. Essentially, the same information used in the original manual chain-of-custody is used to complete the log-in procedures for the LIM system.

2.0 STANDARD OPERATING PROCEDURE:

2.1 The Omega system is entered from the main computer menu by double clicking the Omega icon. When the log-in prompt appears, type "System" and click the "O.K."button. This procedure will take you to the main Omega IV menu. From this menu, double click "Work Orders."

2.2 To add a sample, double click the "Add" key in the lower left-hand corner of the screen and amend the order numbers to correspond with the correct sequence:

Lead SCO: 1-19
1st Assistant SCO: 20-39
2nd Assistant SCO: 40-59
3rd Assistant SCO: 60-79
4th Assistant SCO: 80-89
5th Assistant SCO: 90-99

2.3 Sample Log-in Procedure

2.3.1 Alpha's LIMS generated Chain-of-Custody record is completed with the following information:

a) Client name, address, phone/FAX number;
b) Who the report should be addressed to;
c) Sampler;
d) Date Sampled;
e) Time of collection;
f) Matrix;
g) Lab identification number;
h) Client identification/sample description;
i) Number of containers;
j) Analysis requested; and
k) All necessary signatures/dates/times.
l) Billing Information
m) PO #, PWS #, DWR #, Job # or any additional information

2.3.2 Click once in the "Client Sample ID' box and enter the clients' three letter code. The company name, point-of-contact, address, phone and fax numbers will automatically appear.

2.3.3 The sample Turn-Around-Time (TAT) is automatically indexed to 10 days for all samples. If the TAT is shorter than 10 days is required, amend the TAT box as needed.

2.3.4 If a project name is required, click the "Order Name" box and type the project name.

2.3.5 Double click the "Received" box and a calender will appear with the current date. Double click the date and the system will automatically enter the date. Enter the sample state from the pull-down menu.

2.3.6 Double click the "Date Due" box and the system will count ahead the number of days selected in the TAT box (excluding weekends) and automatically enter the information.

2.3.7 Click on the "Login" key and enter the sample ID for the first sample.

2.3.8 Open the "Collection Date" box and enter the appropriate information. This information should be written on the sample label - otherwise, ask the client.

2.3.9 If a sample time is provided, double space after the "Date Sampled" information box, and enter the correct information converted to military time.

2.3.10 Open the sample "Matrix" box and enter the appropriate information. This information may be typed by hand or entered by clicking the down arrow key adjacent to the matrix box, and selecting the proper matrix.

2.3.11 Enter the number of sample containers currently being logged-in. There is a tab labeled "Bottle Information". Enter the type and number of bottles provided for each sample.

2.3.12 Click once in the "Test Groups" box. Enter the appropriate test and click the down arrow key next to the box to view the list of possible choices. Tests are segregated by state and test method.

2.3.13 Trip Blanks

2.3.13.1 Many sample coolers will have associated Trip Blanks. Trip Blanks are treated as separate samples.

2.3.13.2 For "Client Sample ID" type in Trip Blank.

2.3.13.3 Enter a TAT that corresponds to the sample being logged-in.

2.3.13.4 Enter the collection date which corresponds to the earliest sample date on the chain-of-custody.

Trip Blank collection dates are entered in this fashion to prevent the LIM System from creating an artificial holding time problem. The true Trip Blank preparation date should be listed on the vial label.

2.3.13.5 TB original preparation date information and the place of origin are then entered into the comments section.

2.3.13.6 Trip Blanks are normally not analyzed unless requested by the client. Therefore, under "Test Groups" type "Hold". This will tell the analyst that the sample should not be analyzed.

2.3.14 For each new sample, click the "Add Sample" box and repeat the sample log-in procedure starting with a new client ID.

2.3.15 Add any additional comments in the "Comments" box on the "Main" Screen by clicking on the box and typing the relevant information. Information such as rush TAT, California samples, special QA/QC, etc. are entered into this section.

2.3.16 If a client has a Purchase Order (PO) number, click the "Invoice Info" box at the top of the screen and enter the PO number.

2.3.17 Print the chain-of-custody by clicking the "WO COC" button at the top of the screen.

2.3.18 The analyte list then needs to be generated. Click on "Print Test" and put in the sample number to be printed. Staple the analyte list on the inside cover of the appropriate test folder.

2.3.19 Laboratory ID labels are then generated for the sample containers. Click on the "Labels" button, enter the number of bottles present for each sample and print the labels.

2.3.20 When all relevant information has been entered, click the "out-the-door" box at the lower right hand corner of the screen and exit.

Appendix C

Standard Operating Procedure

SOP C.9
Internal Chain-of-Custody Procedures

1.0 INTERNAL CHAIN-OF-CUSTODY PROCEDURES

1.1 Occasionally the use of internal COC protocols may be required by some clients and/or programs. This SOP is designed to supplement the record keeping system as discussed in various sections of the QAM.

2.0 Standard Operating Procedure

2.1 Internal COC Client Management

It must be stated and understood in the strongest terms that the use of this SOP will be implemented only when specifically requested by the client. This request must be documented on the Sample Receipt Checklist and acknowledged by the client. If the client does not request or acknowledge that this requirement is needed, then the normal record keeping system has precedence and is the system of choice.

1) Clarification: A complete evidentiary COC begins at sample collection, unless otherwise specified by the client, and ends after laboratory analysis of the sample is completed and the sample is ready for disposal. Samples are disposed of in accordance with the normal sample disposal procedures.

2.2 Internal Chain-of-Custody

The internal COC record is designed to produce an intact, continuous record of the physical possession, storage and disposal of samples, sample aliquots and extracts. For ease of use, samples, sample aliquots and extracts are referred to as samples.

2.2.1 A sample is in someone's custody if:

a) it is in one's actual physical possession;

b) it is in one's view, after being in one's physical possession;

c) it is one's physical possession and then locked up so that no one can tamper with it; or

d) it is kept in a secured area, restricted to authorized personnel only.

2.2.2 The internal COC record is filled out and should account for all time periods associated with the sample.

2.2.3 The internal COC is also initialed and dated each time an individual physically handles the sample.

2.2.4 Clients usually provide multiple samples for each analysis; therefore, samples will have more than one bottle. Therefore, when any of the vial/bottles are consumed, the analyst or extraction technician must document the consumption of that vial/bottle (e.g. vial 1/3 consumed) on the internal COC. The vial consumption documentation must also include the date of consumption and the initials of the person that used that vial.

2.2.5 The internal COC contains a "master work order sheet" which is kept in a master logbook. This sheet provides information on when the samples were moved into the primary refrigerator, secondary storage, and the date that the entire sample set was disposed. The same information (except when samples were moved into the primary refrigerator) is recorded for the sample extracts.

This master work order sheet along with the internal COC and the client COC details the custody trail of the samples from the sampling event to final disposal.

2.2.6 The internal COC includes a sign-off sheet for each different test. These sheets are stapled to the inside rear cover of each file folder. The analyst/extraction technician that handles the sample must maintain internal custody by initialing and dating the COC when logging samples in and out of the refrigerator.

2.3 Evidentiary Chain-of-Custody Procedures

2.3.1 The COC procedures as described in C.7 and C.8 are followed completely and error free as possible.

2.3.2 It is important not to break the continual record of physical possession of the samples. Therefore, it is critical to ensure the sampler has signed off or relinquished the samples to either a second party or to the laboratory.

2.3.3 If samples are mailed, then they should be registered with return receipt requested. If samples are sent by common courier, receipts should be retained as part of the permanent COC documentation.

2.3.4 Once samples are received by Alpha, then the responsibilities for the care and custody of the samples are in our hands.

2.4 Sample Disposal

2.4.1 Disposal of the sample will occur 60 days after sample receipt as outlined in the waste disposal SOP.

2.4.2 All conditions of disposal and correspondence between parties concerning the

final disposition of the sample is recorded and retained as part of the permanent record.

2.4.3 Internal COC records indicate the date of disposal, the nature of disposal (i.e., sample depleted, sample disposed of in hazardous waste facility or sample returned to client) and the name of the person who performed the task.

Appendix C

Standard Operating Procedure

SOP C.10
Sample Log-in Ledger

1.0 SAMPLE LOG-IN LEDGER

1.1 Upon arrival at the laboratory, the Sample Custodian completes a LIMS generated COC. From the LIMS generated chain-of-custody a macro routine is used to parce information to be placed in a 3-ring binder called the Sample Log-In Ledger. This is a notebook used to keep a running total of all samples received by Alpha. This ledger is used in conjunction with other Log-in documents to rectify sample receipt problems.

2.0 STANDARD OPERATING PROCEDURE:

2.1 NELAC Requirements

2.1.1 NELAC requires the Sample Log-In Ledger to record the following minimum information:

i. client/project name,
ii. date and time of sample receipt,
iii. laboratory sample identification, and
iv. signature or initials of the person making the entries.

2.1.2 NELAC also requires the following additional information to be unequivocally linked between the COC and Sample Log-In Ledger.

i. The client/field identification on each container is linked to the laboratory identification.

ii. The date and time of sample collection is linked to the sample container and to the date and time of sample receipt.

iii. The requested analyses is linked to the laboratory identification.

iv. Any comments resulting from inspection for sample rejection is linked to the laboratory identification.

2.2 Since the Sample Log-In Ledger is parced from the LIMS generated COC essentially any information contained on the COC can be unequivocally linked to COC log in records.

2.2.1 All COC and sample log-in records/information are entered into our LIM system and stored as permanent archived records. These records are easily retrieved upon request and readily available to all staff members who will process the samples.

2.2.2 The Sample Log-In Ledger records the following information:

a) initials,

b) date of sample receipt,

c) laboratory's sample identification,

d) client's sample identification,

e) matrix type,

f) analysis requested,

g) Turn-Around-Time (TAT),

h) date sampled,

i) temperature upon sample receipt,

j) work order comments, and

k) any additional comments.

2.3 Approximately every 2 weeks the SCO or assistant SCO activates the Sample Log-In Ledger macro and initiates a computer parcing routine which identifies the requested information as stated above.

All samples and associated sample information entered into the LIMS, regardless of the person whom actually logged-in the sample, will be printed out in chronological order.

Once this information is printed, the SCO or Assistant SCO checks the information for accuracy, paginates and initials the ledger.

Appendix C

Standard Operating Procedure

SOP C.11
Sample Storage Procedure

1.0 SAMPLE STORAGE PROCEDURE

1.1 Alpha maintains sample integrity through disciplined sample processing and storage procedures. All samples received by the lab are placed in the appropriate storage area immediately after log in. If the sample is removed from the storage area for extraction or analysis, this activity is documented in the technician's extraction or the analyst's instrument logbooks. Therefore, samples can be tracked in the laboratory by reconstructing historical data.

2.0 STANDARD OPERATING PROCEDURE

2.1 Samples requiring thermal preservation are stored under refrigeration which is ± 2°C of the specified preservation temperature unless method specific criteria exists. For samples with a specified storage temperature of 4°C, storage at a temperature of above freezing point of water to 6°C is acceptable. Freezer temperatures are kept within an ideal temperature of -10 °C to -20 °C and are maintained according to their particular use.

2.2 The samples removed from the shipping container are stored in their original containers unless damaged. Damaged samples are disposed of in an appropriate manner and documented.

2.3 Alpha maintains a large inventory of refrigerators and freezers in order to facilitate proper segregation of samples, extracts, standards, etc. which require thermal preservation according to the specifications in the test methods.

2.3.1 General Storage Considerations - the following items are isolated from one another and stored separately to prevent environmental and/or chemical cross contamination:

1) samples,
2) standards,
3) extracts and digests, and
4) solvents and reagents.

2.3.2 Food Storage - food is isolated and stored separately to prevent any possibility of contaminating food from environmental samples, or chemicals used in the analysis of samples.

2.3.3 Samples Storage - samples are stored away from all standards, reagents, food and other potentially contaminating sources. In addition, the following considerations are used when isolating samples:

1) class of analytes to be measured;

2) volatile organic samples are completely isolated from all other types of sample streams;

3) samples suspected of containing high levels of volatile organics are further isolated from other volatile organic samples; and

4) method of analysis.

2.3.4 Extracts and Reagents Storage - these chemical streams are stored and isolated from one another as well as storing and isolating them from samples to help prevent any possible cross contamination. In addition these chemical streams are isolated and stored away from food to prevent contaminating the food and causing a health issue.

2.4 All samples are placed in cold storage until they have been analyzed, and then held for a minimum of sixty days.

2.5 Disposal records are maintained to demonstrate that samples have been properly disposed of, in accordance with Federal, State and local regulations.

2.6 Refer to SOP D.14, Refrigerator/Freezer Document Control, for a listing of the sample and extract storage areas.

Appendix C

Standard Operating Procedure

SOP C.12
Sample Tracking Procedure

1.0 SAMPLE TRACKING PROCEDURE

1.1 Sample tracking is a fundamental responsibility of the SCO. This person must, at any given time, know where a particular sample or client file is located and have the ability to track the sample's analytical progression through the laboratory. The large number of samples in-house requires a procedure for tracking and documenting this activity.

2.0 STANDARD OPERATING PROCEDURE

2.1 The SCO must properly fill out a COC, identify, label and store all samples upon receipt, according to established SOPs.

2.2 Once this activity has taken place, multiple copies of the COC are made and placed in various locations.

2.3 A master client-file is then generated for the purpose of physically tracking analytical data produced by the various analyses. Each sample or group of samples from an individual client is given a client file with the client and sample identification written on the folder's tab. Each method of analysis has its own folder and is discriminated between methods by the file folder color. Table C.12-1 is a listing of file folder colors associated with a particular method of analysis.

2.4 Bin World

2.4.1 Bin World is a part of the physical tracking system used in conjunction with the master files to carry out the sample tracking procedure. Bin World is a set of various discrete bins associated with a given day used to segregate primary sample streams. The various discrete bins include:

- Other lab data only,
- Alpha + other lab,
- Alpha only,
- Alpha 5-day,
- Amendments, and
- Rush.

2.4.2 Master files are placed in the Bin World according to the discrete primary sample stream. Secondly, master files are placed in Bin World according to their "due date". This enables client data to be tracked effectively according to due date.

2.4.3 If the analytical data is not produced in a manner reflective of the client requested due date, or if QA/QC data is to be sent at a later date, then the master file is relocated to another set of discrete bins. There are three primary areas which may delay final data production and therefore have an associated

bin. They are as follows:

- Delayed Alpha reports,
- Delayed Inorganic reports, and
- Needs QC Alpha / Inorganic.

2.5 Master Files

2.5.1 A master file is generated for each group of samples represented by a single COC. A manilla file folder is used for this master file. If the client is requesting a single method of analysis, then the master file is generated using the same file folder color as the color used for the requested method of analysis. The master file is labeled with the sample identification number attached to the folder's tab. The master file initially contains the following items:

- manually produced, hand written COC;
- LIMS generated COC;
- air bills or bills-of-landing;
- memos or transmittal forms;
- nonconformance letters or a sample receipt checklist;
- documentation confirming that the sample submitting party has received, and was notified of, the sample receipt Checklist;
- client communications in regards to special requests or changed orders; and
- any other relevant information.

2.6 Analytical Files

2.6.1 Once the analytical file folders are generated, they are placed in the extraction or analytical lab depending upon the matrix and analysis requested. This gives the extraction chemist or the analyst two places to check and verify sample information:

1) the client file; and
2) the LIMS System.

2.6.2 The extraction chemist or analyst will then perform the required work and place the relevant data in the respective client file folder. If the data can be uploaded, it is then uploaded into the LIM system which will indicate the work has been completed.

2.6.3 Once the analysis has been completed, the client file is reviewed by several staff members. This is a rigorous four-tiered review to ensure the data has been calculated, evaluated and reported correctly.

2.6.4 Once the analytical report has been signed, the method files and all of their associated analytical data is then collected into the master file.

2.6.5 The DCO and SCO both monitor the master file to determine and physically track the status of analytical data. This procedure continues until all data has been produced and physically collected in the master file.

2.6.6 Final analytical data is then faxed, or e-mailed as a scanned pdf file and a final hard copy is sent to the client.

File Folder Tracking Table
Table C.12-1

Analytical Method	File Folder Color
524.2	Yellow
8260 (Water) / (Soil)	Brown/Lavender
624	Blue
8010 / 601 / (Water) / (Soil)	Blue
8081/8082 /608 /(Water)/ (Soil)	Orange
8270 / 625 / (Water) / (Soil)	Red
TPH-Purgeable (Water) / (Soil)	Lavendar
TPH-Extractable (Water) / (Soil)	Green/Red
TCLP Volatiles	Blue
TCLP Pest / Herb/ Sv	Gray
S / O Separators (11 Regulated)	Blue
TOC	Pink
Methanol	White
Metals	Gray
Wet Chemistry	Teal/Pink
Gravimetric	Maroon
COD/Phosphourus	Navy Blue
Anions	Light Blue
Perchlorate	White
Methane	White
Organic Acids	Yellow

Appendix C

Standard Operating Procedure

SOP C.13
Sample Custody Procedure

1.0 SAMPLE CUSTODY PROCEDURE

1.1 The maintenance of custody defines who is responsible for sample integrity at any given time. The responsibility of sample custody changes during the sample's life in the laboratory. Sample custody is documented on bench logs, analysts logs and other QA documents while in the laboratory, from receipt until sample disposal.

2.0 STANDARD OPERATING PROCEDURE

2.1 The following narrative describes the change of sample custody and responsibility during normal laboratory activities.

2.1.1 The Sample Custody Officer is responsible for sample receipt and placing samples in the correct cold storage facility.

2.1.2 Sample custody changes when an extraction technician or analyst removes that sample from storage. Samples will remain in that person's custody until returned to storage.

2.1.3 Once the sample arrives at the lab bench, the analyst or technician records all procedures completed on the sample in the proper logbooks.

2.1.4 The remaining sample or extract is returned to the appropriate storage facility and custody returns to the Sample Custodian.

2.1.5 Those samples requiring security are stored in a locked storage area, with the Sample Custodian or a designated analyst having access. Sample custody remains that person's responsibility at all times.

Appendix C

Standard Operating Procedure

SOP C.14
Electronic Transmission of Results

1.0 ELECTRON TRANSMISSION OF RESULTS

1.1 An e-mail of the original data scanned as a pdf file is the preferred method of relaying client information. The following procedures outline the steps taken when e-mailing client information.

1.2 The electron transmission of results must be conducted to ensure that all reasonable steps have been taken to preserve sample and client confidentiality.

2.0 STANDARD OPERATING PROCEDURE

2.1 Analytical Data Verification

2.1.1 DCO should review and verify the analytical folder disseminated to the various analysts; match the final report generated by the report writer.

2.1.2 DCO should review and verify the analytical folder was correctly placed in the appropriate master file located in Bin World under the day the reports are due.

2.1.3 DCO should review and verify the final analytical reports match the requested methods of analysis on the COC.

2.1.4 DCO should review and verify that all analytical data and final reports are included in the master file.

2.1.4.1 If the master file is not complete, place the analytical folders with their reports standing up in the master file and place them in the Bin World under the day the reports are due.

2.1.4.2 If at the end of the day, folders have not been completed, then place the master file in the appropriate secondary bin (e.g. delayed data, needs inorganic results, etc.).

2.1.5 DCO should review and verify clients' specific instructions. These "Client SOPs" need to be checked for special reporting instructions before removing the master file from the due date bin to the secondary bins. These special reporting instructions include such things as: "e-mail results as soon as possible," etc.

2.1.6 If a master file is complete, it is ready for e-mailing. The master file is then removed from the Bin World and placed in the "Reports to be E-mailed" bin next to the scanner until it is e-mailed.

2.2 E-mailing

2.2.1 Prior to e-mailing, the entire report is scanned as a pdf file and saved in a sub-directory by month using the work order and job name to identify the file.

2.2.2 A permanent record is completed daily which lists all reports e-mailed during that day. This record includes the following:

- Alpha's W.O. number,
- The test being reported, and
- Noting if the final data reported was a completed or partial data report.

2.2.3 Items to be e-mailed include the final reports, and client's original COC. If the client's original COC is amended, then the amended COC is included with the e-mail.

2.2.4 Send the e-mail to the person listed under the "Report Attention" column located on the COC.

2.2.5 Refer to the client specific SOP for any special instructions, such as e-mailing to a group of people.

2.2.6 Once the e-mail process has been initiated, place the reports and COC back into the master file and place this file into the Awaiting Confirmation bin next to the scanner.

2.2.7 After the e-mail confirmation has been issued, stating the e-mail transmission was successful, the confirmation is placed in the master file as part of its permanent record.

2.2.8 Once the reports have been e-mailed, the master file is then forwarded to one of the following areas:

- Client specific QC,
- Electronic Data Deliverables(EDDs),
- Invoicing.

If the folder is not complete, place the folder back in Bin World in one of the secondary bins associated with its deficiency.

2.2.9 Once the daily e-mailing requirements has been completed, the list of e-mailed client reports are entered into the LIM System under

- Work Order, and
- Fax Update

Additionally, the daily permanent record listing all reports e-mailed during that day is archived in a 3-ring binder labeled "E-mailed Report Binder," segregated by month.

Appendix C

Standard Operating Procedure

SOP C.15
Sample Scheduling and Discrepancy Reporting

1.0 SAMPLE SCHEDULING AND DISCREPANCY REPORTING

1.1 The Sample Custody Officer and Supervisors discuss and communicate all sample integrity issues such as sample containers, preservation and holding times with the appropriate personnel to ensure samples are received and documented appropriately. The SCO will also help schedule sample receipt and analysis with the client and notify that person as to any analytical irregularities.

2.0 STANDARD OPERATING PROCEDURE

2.1 The laboratory supervisors will coordinate sample scheduling with the appropriate personnel to maximize economy of effort with the number and types of analysis to be performed.

2.2 The laboratory supervisors will update the client as necessary throughout the analytical process as required by that client.

2.3 Irregularities with sample documentation, or problems encountered during sample analysis will be reported immediately to the laboratory director.

2.4 The director or analysts will document all discrepancies associated with the sample, before the problem is communicated to the client.

2.5 It is the responsibility of the laboratory director to communicate and resolve any out-of-control problems with a client.

2.6 In reporting data discrepancies, the client's sample identification is used both verbally and in correspondence.

Appendix C

Standard Operating Procedure

SOP C.16
Waste Disposal Procedure

1.0 WASTE DISPOSAL PROCEDURE

1.1 Alpha Analytical, Inc. is committed to insuring to our customers and our employees that all wastes generated and/or accepted at the laboratory are disposed of in a proper and responsible manner.

2.0 CLASSIFICATION AND ORIGIN OF WASTE STREAMS

To work within the proper waste disposal program at Alpha, it is important to understand the different waste streams and their proper handling and disposal operations.

2.1 Types of Waste

2.1.1 **Non-Hazardous Waste:** Most materials, solids and liquids, that have not come in contact with solvents can be disposed of as non-hazardous wastes. These include unused soi/water samples, rinse waters, glass containers, etc.

2.1.2 **Hazardous Waste:** Those materials which are listed or characteristic wastes as defined in 40 CFR. Specifically, these include spent solvents, spent soil and water extracts, specific inorganic wastes, fuel samples and any material which comes into contact with large amounts (>10%) of any solvent.

2.2 Origin of Waste Stream

It is important to understand that a hazardous material is not a hazardous waste until its use is no longer viable within the laboratory. Only when a hazardous material is ready for disposal does it become a hazardous waste. Once it becomes a hazardous waste, it must be disposed of within the facility and can not be moved.

Tracking a soil sample for VOC analysis can better explain this concept. An aliquot of soil is removed from a brass sleeve and extracted into Methanol in a 40ml vial. This sample extract can be moved from the lab to the storage area on Freeport Way. The sample remains there for the 60-day retention period required by the client. Once the retention time is complete, the sample extract is ready for disposal and then becomes a hazardous waste. Other wastes, such as waste solvent from the SVOC extraction process has no benefits after the extraction process and therefore becomes a hazardous waste immediately. Because of this, the spent solvents from the SVOC lab must stay within the lab facility and can not be transported to the waste facility on Freeport.

It is important to understand that Alpha holds two EPA manifest sites: the lab facility on Glendale Avenue and the waste facility behind the lab on Freeport Way. All wastes generated within either facility must be manifested and disposed of from that facilities waste stream. If you have any questions regarding the status of a waste or the proper placement of the waste, please contact Randy Gardner for information.

3.0 Waste Streams - Per Facility

3.1 Laboratory Facility

All hazardous waste that is generated in the laboratory facility, located on Glendale Avenue, must be temporarily stored in the waste area of the extraction lab or in the inorganic lab. The wastes that are located in the extraction lab are as follows:

3.1.1 Waste Solvent: All waste solvents, including Methylene chloride, Methanol, Hexane, Ether and any other rinse or spent solvents, are combined and manifested as F listed wastes.

3.1.1.1 Extracted Liquids: All extracted liquids for such analyses as SVOC and Pesticides.

3.1.1.2 Extracted Solids: All extracted solids, or any other solids that come in contact with solvents (i.e. sodium sulfate). Also, all unused client samples are disposed of as extracted F002 waste.

3.1.2 Wastes located in the inorganic laboratory include:

3.1.2.1 Metals Liquid Waste: Waste reagents from spectrophotometric procedures, e.g., COD, Cr^{+6}, Sulfide, etc., and other inorganic procedures that produce wastes containing high levels of metals. Such wastes include silver, mercury, hexavalent chrome and other such metals.

3.2 Waste/Storage Laboratory

All samples and sample extracts that are stored in the waste laboratory behind the lab facility (located on Freeport Way) are not classified as hazardous waste until they are ready to be disposed of. Again, all sample and sample extracts stored in the waste area have beneficial use and are stored there in case there is a need for a further analysis or a reanalysis for confirmation purposes. Alpha's general policy is to store these samples and their extracts for 60 days. After the 60-day storage period, the samples are ready to be disposed and therefore have no beneficial use, thus classifying themselves as hazardous waste.

3.2.1 The hazardous wastes located in the waste/storage lab are as follows:

3.2.1.1 Waste Solvent: All solvent waste that has been decanted from the extracted samples. These are typically TPH and VOC soil samples that have been extracted and stored in a 40-ml closed-top vial. This waste solvent is primarily made up of Hexane and Methanol.

3.2.1.2 Extracted Solids: All extracted solids, or any other solid samples that come in contact with organic solvents, i.e., sodium sulfate. Once the solvent is decanted from the TPH or VOC extraction vials, soil samples are placed in the extracted soil container. All unused client samples are also disposed of as extracted soils.

3.2.2 The non-hazardous wastes located in the waste/storage lab are as follows:

3.2.2.1 Non-extracted Solids: All non-extracted soils, such as duplicate soil containers and unused soil samples are disposed of as ~~non-~~hazardous waste. These soils are stored in a 55-gallon drum and disposed of as Petroleum Contaminated Soils.

3.2.2.2 Non-extracted Liquids: All non-extracted liquids, such as duplicate containers and unused samples are treated as non-hazardous waste. Because liquids for TPH-Diesel (EPA Method 8015) use 1 ml of hexane for the extraction, these liquids are separated from the hexane and treated as non-hazardous. These liquids are collected and evaporated as described in the following section.

4.0 Waste Streams - Per Usage

4.1 Following is a designation of all hazardous and non-hazardous wastes that Alpha deals within conjunction with its laboratory functions. Also, listed are the correct handling and disposal processes.

4.1.1 Waste Solvent: All spent solvent, both chlorinated and non-chlorinated must be disposed of as waste solvent. Several temporary waste containers are available throughout the laboratory. Once full, these containers must be emptied into the 55-gallon drum in the extraction or waste laboratory. The drum is self-contained. It is important that this drum remain under the fume hood at all times and that the drum is never tipped or rolled. Each time solvent is added to this drum, the level must be marked and dated. A weekly visual examination must be made of the drum to ensure that acidic or chlorinated solvents have not corroded the metal barrel. The lids must be kept on the barrels when not in use.

4.1.2 VOC Soil Extracts: Soils being analyzed for volatile organic (VOC) are extracted with methanol. The soils are extracted into methanol in 40 ml vials. After analysis, the extracts are moved to the waste facility for the required retention period. After the retention period, the vials are crushed with the glass crusher and the solid material (both glass and extracted soil) are disposed of as extracted soil. The solvent is screened off and disposed of as waste solvent.

4.1.3 VOC Waters: Those aqueous samples requiring VOC analysis are purged directly onto the instrument and are not solvent extracted with methanol. The purged water, along with the duplicate vials, are crushed with the glass crusher and the solid material (glass vials) are disposed of as non-extracted soil/solid. The remaining water is screened off and disposed of as non-extracted water.

4.1.4 TPH-E and SVOC Soil Extracts: Soil samples being analyzed for TPH-E or Semi-Volatile Organics are extracted with Hexane/Acetone or Methylene Chloride. The soils are extracted in 40 ml vials. After analyses, the extracts are moved to the waste facility for the required retention period. After the retention period, the vials are crushed with the glass crusher and the solid material (both glass and extracted soil) are disposed of as extracted soil. The solvent is screened off and disposed of as waste solvent.

4.1.5 TPH-E Water Extracts: Those waters that require TPH-E analysis are extracted with Hexane in 40 ml vials. After analyses, the extracts are moved to the waste facility for the required retention period. After the retention period, the vials are crushed with the glass crusher. The water is separated from the solvent and disposed of as non-extracted water and the solvent is disposed of as solvent waste.

4.1.6 Extracted Soil for Semi-volatile Analyses (Requiring ASE Extraction) (SVOC, and Pesticides etc): These solvent extracts are captured into 40 ml vials. The extracted soil samples are removed from the ASE extraction containers and are disposed of in the laboratory as extracted soils. Because the extracted soils from the ASE are not retained for further use, they become hazardous waste immediately and cannot be moved to the waste facility. ALL ASE extracted soils are manifested from the laboratory facility. After analysis, the extracts and/or autovials are moved to the waste facility for retention. After retention, the vials are crushed with the glass crusher and the solid material is disposed of as extracted soil. The extract is disposed of as waste solvent.

4.1.7 Extracted Liquids for Semi-volatile Analysis (SVOC and Pesticides, etc): Liquids requiring any semi-volatile analysis are solvent extracted. The solvent extract is concentrated down and placed in the appropriate vials. The extracted water is disposed of in the laboratory as extracted water. Because the extracted waters are not retained for further use, they become hazardous waste immediately and can not be moved to the waste facility. ALL such extracted waters are manifested from the laboratory facility. After analysis, the extracts and/or autovials are moved to the waste facility for retention. After retention, vials are crushed with the glass crusher and the solid material is disposed of as extracted soil and the extract is disposed of as waste solvent.

4.1.8 Auto-vialed Extracts: All samples that require a semivolatile extraction are transferred into 2ml autovials. These autovials are then placed on the appropriate instrument and analyzed. After analysis, these vials are moved to the waste facility for retention. After the retention period, they are disposed of by crushing the vials with the small autovial crusher located under the waste fume hood. The solid material is disposed of as an extracted soil/solid. The solvent extract is disposed of as waste solvent.

4.1.9 Inorganic Laboratory Liquid Waste: All extracted and non-extracted waste water from the inorganic shop must be neutralized before being disposed of as a non-hazardous waste.

4.1.10 Inorganic Metals Waste: All reagents that contain hazardous levels of metals. Such wastes include many of the spectrophotometric method wastes from the analysis of COD, Sulfide and Cr^{+6}. These wastes have high levels of Silver, Mercury and Cr^{+6}.

5.0 Storage of Hazardous Waste

5.1 One of the fundamental concepts of hazardous waste management is the 90-day limitation for accumulating and storing hazardous waste on-site before they must be transported off-site for treatment or disposal.

Whereas the 90-day limitation for on-site storage of hazardous waste is a requirement of hazardous waste management, it is important to know and understand the exceptions to this rule, namely the *Satellite Rule*. This exception is important in that it would be costly and inefficient for Alpha to accumulate its many different waste streams and to dispose of them every 90 days. However, compliance should be followed meticulously. Therefore it is imperative that the rules are understood.

The following is a quick reference summary of the accumulation and storage rules:

5.1.1 On-site storage of Hazardous Waste without a storage facility permit is limited to 90 days unless the generator qualifies for satellite rule accumulation.

5.1.2 The accumulation start date which begins the 90-day period and must be identified on the label of each container is different for large and small quantity generators. If more than 100 Kg (220 pounds), 27 g of hazardous waste or 1 kg of extremely or acutely hazardous waste are generated at the entire facility, the 90-day period begins when the first drop hits the container. If not, the small quantity generator may accumulate up to these amounts before beginning the 90-day clock.

5.1.3 The *satellite rule* provides relief for generators who qualify by allowing accumulation of up to 55 gallons of hazardous waste, or 1 qt of extremely or

acutely hazardous waste, or one year, whichever comes first, at or near the point of generation. This rule allows a reasonable accumulation period for smaller volume waste streams as they are usually generated in the laboratory.

5.1.4 Any facility which stores hazardous wastes over the 90-day period (unless the satellite rule applies) must obtain a permit or variance from the NDEP.

5.2 Accumulation Exemptions: (40 CFR 262.34)

5.2.1 A generator who generates greater than 100 Kg but less than 1000 Kg of Hazardous Waste in a calendar month may accumulate hazardous waste on-site for 180 days or less without a permit or without having interim status provide that:

1) The quantity of waste never exceeds 6000 Kg

2) That requirements of subpart I of 265, except 265.176 are in compliance. These requirements are:

a) The condition of the containers are intact;

b) Stored wastes are compatible;

c) The management of the containers is prudent (i.e., container must be kept closed, and handled with care);

d) The containers must be inspected at least weekly;

e) The containers are clearly labeled and the accumulation dates are legible;

f) At least one employee must be available at all times to respond to an emergency at the facility;

g) The following information must be posted near the telephone:

1) Name and phone number of emergency coordinator;

2) Location of fire extinguishers, spill control material and fire alarms; and,

3) Telephone number of local fire department unless the facility has a direct line through the fire alarm.

3) Alpha must ensure that all employees are thoroughly familiar with

proper waste handling and emergency procedures, relevant to their responsibilities during normal facility operations and emergencies.

4) Immediate emergency response levels are:

FIRE: Call the fire department and/or attempt to extinguish it.

SPILL: In the event of a spill, contain the flow of HW to the extent possible, and as soon as is practicable, clean up the HW and any contaminated materials or soil.

OTHER: In the event of a fire, explosion, or other release which could threaten human health outside the facility or when the generator has knowledge that a spill has reached surface water, the SO must immediately notify the National Response Center (800/424-8802). The report must include the following information:

1) Name, address, and USEPA ID generator's number;

2) Date, time and type of incident (spill or fire, etc);

3) Quantity and type of HW involved;

4) Extent of injured; and

5) Estimated quantity and disposition of any recovered materials.

If the generator meets the above criteria, and must transport his waste, or offer his waste for transport, over a distance of 200 miles or more for off-site treatment, storage or disposal may accumulate HW on-site for 270 days or less without a permit or without having interim status.

6.0 Disposal of Empty Containers

6.1 Containers which are empty and no longer needed must be disposed of properly. Container disposal shall be as directed by 40 CFR 261.7 "Residues of hazardous waste in empty containers." Containers which have held acute hazardous materials as defined in 40 CFR 261.31, 261.32, or 261.33 require special handling. Please contact Randy Gardner for further information and assistance.

The following guidelines are used to assist in determining if an empty container is regulated. A container shall be considered "empty" if all the following conditions exist (for this section, a container shall be considered to be a primary container or an inner liner):

6.1.1 The container contained none of the chemicals that are listed in 40 CFR 261.33(e),

6.1.2 All chemicals have been removed that can be removed using practices commonly employed to remove materials from that type of container (i.e., pouring, pumping, aspirating, etc.),

6.1.3 There is less than one inch of residue left in the bottom of the container,

6.1.4 There is less than 3% by weight of residue left in the container (0.3% for >110 gal. containers),

The container shall be considered empty **only** if the container has been triple rinsed or cleaned by another method that has been shown in the scientific literature to achieve equivalent removal. If the container has not been cleaned as stated above, the container shall become hazardous waste. Once a container has been declared "empty" by the above criteria, it can be placed in the normal refuse.

7.0 Waste Area Mechanical Operation

All waste area technicians must have training in the operation of the jaw crusher, pump operation, evaporator operation and storage procedures before being allowed access to the waste area. Following is listing of the equipment in the waste laboratory and their standard operating procedures.

7.1 Waste water evaporator and holding/settling tank operation

7.1.1 The waste water evaporator is designed for the evaporation of non-hazardous wastewater. Utmost care must be given to ensure that hazardous waste, solvents or pure fuel products are not introduced to either the holding tank or the evaporator. Wastewater may be introduced into the evaporator by either discharging directly into the evaporator or by transferring wastewater from the holding tank to the evaporator. The holding tank is designed to prevent sediment transfer directly into the evaporator. In essence it is a settling tank. Once evaporator wastewater has been pumped into the settling tank, a period of no less than twenty-four hours of undisturbed settling time is required before the second transfer is initiated from the settling tank to the evaporator. Wastewater directly dumped into the evaporator must be free of sediment or very carefully decanted to eliminate a sediment buildup of the evaporation unit. Waste streams that can be evaporated include the following:

- Mass spectrometer rinse water,
- Client water samples,
- Neutralized inorganic wastewater
- TPH-E water extracts (decanting the hexane solvent layer),

- HPLC waste, except acetontrile waste waters; and,
- Non-extracted lab waters.

7.1.2 The evaporator requires routine maintenance in an effort to keep it as clean as possible and to preclude the build up of heavy metals and salts. Over time this type of scaling can be harmful to the heating coils. On a three-month frequency, the evaporator water should be cooled, then pumped out and disposed of as non-hazardous wastewater. The evaporator should be thoroughly cleaned including wire brushing scale deposits off the heating coils and sides of the evaporator and thorough checks should be made for cracking or unusual wear. Once a year, the combustion chambers' air to fuel mixture should be checked and adjusted according to the owner's manual specifications. The full maintenance procedure is found in section 4.3 of the owners manual.

7.1.3 Extreme care should be given when transferring wastewater from the crusher to the settling tank and on to the evaporator. This operation may require two people to prevent possible over-spillage.

7.2 Jaw Crusher Operation

7.2.1 The jaw crusher is used for the crushing of 40mL VOA vials and 1 liter bottles. DO NOT crush auto vials or screw cap vials smaller than 20 ml VOA's in the jaw crusher. All crushed glass collected in the lower strainer bin must be triple rinsed with water before being dumped into the trash container.

7.2.2 The jaw crusher is a very powerful piece of equipment. The following safety elements should be followed when operating the jaw crusher:

7.2.2.1 Never operate the crusher without the fan belt and fly wheel shrouds bolted in place. Use common sense when in operation;

7.2.2.2 Do not put your hands near any moving parts;

7.2.2.3 Always ensure no one is near the crusher when the machine is being tuned on;

7.2.2.4 Always use an extender such as a stick or a bar to free glass stuck in the loading chute;

7.2.2.5 Always wear safety glasses;

7.2.2.6 Always wear cotton or leather gloves;

7.2.2.7 Never over-fill the hopper, VOA vials will clog the hopper jaws and not feed properly;

7.2.2.8 The hopper lid must be installed to prevent glass from flying out of the hopper when crushing liter and smaller bottles;

7.2.2.9 Remove all liter and large bottle lids prior to crushing;

7.2.2.10 Bottles must be fed into the hopper one bottle at a time with the neck of the bottle towards the jaws;

7.2.2.11 All glass collected in the lower strainer bin must be triple rinsed before dumping into the trash container;

7.2.2.12 Always know what is being placed in the crusher;

7.2.2.13 Never leave uncrushed material in the hopper;

7.2.2.14 Keep the crusher and surrounding area clean and free of litter;

7.2.2.15 Clean all spills immediately;

7.2.2.16 Keep the white crusher "roll-around-bins" as clean and sediment free as possible;

7.2.2.17 Avoid vacuuming sediment in the pick-up hose when transferring water from the white crusher-bins to the settling tank; and,

7.2.2.18 Grease the rotating crushing shaft after each crushing event to prevent the crushing jaws from freezing-up.

7.3 Auto vial crusher operations

7.3.1 The autovial crusher is used to crush auto vials, and 2 and 8 ml screw cap vials. This operation is preformed under the waste hood to capture solvent vapors. Avoid over filling the hopper and always ensure the discharge hose is securely placed in the solvent waste container under the crusher. The crushed glass is disposed of as extracted soil.

8.0 TPH-E Disposal Procedures

8.1 There are primary waste streams used in the disposal of TPH samples. The following standard operating procedures describe the disposal practices for each TPH-E waste stream.

8.2 Wastewater

The following TPH-E waste streams are bulked together and disposed of as Non-hazardous wastewater. These streams include the following:

8.2.1 Unused Client Water Samples: Unused client water samples are packed in boxes labeled TPH WASTEWATER with the sample receipt date. These boxes are transferred for storage to the waste laboratory. After the required retention, the samples are disposed of as non-hazardous wastewater.

Cold Storage: Refrigerator SAR-4B (duplicate samples)
Cold Storage Sample Removal: 2 weeks from date of sample receipt
Cold Storage Removal frequency: Weekly
Total Storage Period: 60 days from date of sample receipt
Waste Stream: Non-hazardous wastewater

Note: Water samples are bulked into the non-hazardous wastewater stream. The containers are crushed. Once crushed, the glass shards are triple rinsed with water and discarded in the trash bin.

8.2.2 Extracted Water: All extracted water is packed in boxes and labeled TPH EXTRACTED WATER with the disposal date. These boxes are transferred for storage to the waste laboratory and placed on shelves marked TPH EXTRACTIONS.

Cold Storage: Refrigerator EXT-37A
Cold Storage Sample Removal: Monthly
Total Storage Period: 60 days from date of sample receipt
Waste Stream: Non-hazardous wastewater

Note: The extracted water is decanted from the vials and the water fraction is bulked with the non-hazardous wastewater and the hexane solvent is bulked with the waste solvent.

8.2.3 TOC Waste: All TOC waste water is treated as non-hazardous wastewater. TOC wastewater is transferred to the waste room for storage, then disposed of as non-hazardous wastewater.

Cold Storage: Refrigerator SAR-4B (duplicate samples)
Cold Storage Sample Removal: 2 weeks from date of sample receipt
Cold Storage Removal frequency: Weekly
Total Storage Period: 60 days from date of sample receipt
Waste stream: Non-hazardous wastewater

8.3 Hazardous Solids

The following TPH waste streams are bulked and disposed of as hazardous solids. Hazardous solid waste stream includes:

8.3.1 Extracted Soils: Soil extracts are packed in boxes labeled TPH SOIL EXTRACTS with the disposal date. Soil extracts are transferred for storage to the waste laboratory. These boxes are stored on the shelves marked TPH EXTRACTIONS.

Cold Storage: Refrigerator EXT-37A
Cold Storage Sample Removal: Monthly
Total Storage Period: 60 days from date of sample receipt
Waste Stream: Hazardous solid and Solvent waste

Note: The hexane is decanted and bulked with the solvent waste. The remaining soil extract and glass containers are crushed together, collected and bulk packed with the hazardous solid waste.

8.3.2 Unused Client Soil Samples: Unused client soil samples older than 14 days are transferred for storage to the waste laboratory. The samples are stored in order of date received and are disposed of in accordance to their disposal dates.

Cold Storage: Refrigerator SAR-11A
Cold Storage Sample Removal: 14-days
Total Storage Period: 60 days from date of sample receipt
Waste Stream: Non-hazardous solid

8.5 Pure Fuel Product Samples

8.5.1 Pure product samples are removed for storage to the waste laboratory in sealed paint cans. They are labeled with their disposal date and placed on the Pure Product shelves in the storage area, according to the date of disposal.

Cold Storage: Refrigerator SAR-2B
Cold Storage Sample Removal: Monthly
Total Storage Period: 60 Days from date of sample receipt
Waste Stream: Waste Solvent

9.0 VOC Waste Disposal Procedures

9.1 There are four primary waste streams used in the disposal of VOC samples. The following SOP describes the disposal practices for VOC samples and their associated wastes.

9.2 Wastewater

The following VOC subwaste streams are bulked together and disposed of as Non-hazardous Wastewater. These waste streams include the following:

9.2.1 Unused Client Water Samples: Unused Client water samples older than 14 days are removed from the refrigerator and packed in boxes labeled VOC NON-EXTRACTED WATER with the date of sample receipt. The VOA rack labels indicating client name and date are removed, and attached to the inside lid of the storage box. These boxes are transferred to the waste area and placed in the storage racks designated for VOC samples. After 60 days from the date of receipt, samples are discarded as non-hazardous wastewater.

Cold Storage: Refrigerator SAR-1B
Cold Storage Sample Removal: 2 weeks from date of sample receipt
Cold Storage Removal Frequency: Daily
Total Storage Period: 60 days from date of sample receipt
Waste Stream: non-hazardous wastewater

Note: Water samples are bulked into the non-hazardous wastewater stream. The VOA vials are crushed. Once crushed, these glass shards are triple rinsed with water and discarded in the trash bin.

9.2.2 Mass Spectrometer Rinse Water: The mass spectrometer rinse water is collected from each instrument daily. Rinse water is transferred to the waste room for disposal as non-hazardous wastewater.

Removal Frequency: Daily
Waste Stream: non-hazardous wastewater

9.2.3 Client Drinking Water Samples: VOC drinking waters are removed from the refrigerator every 30 days. Samples are packed into boxes labeled VOC DRINKING WATER with the date of disposal. These boxes are transferred to the sample storage area and retained for 60 days from date of sample receipt. Samples are disposed of as non-hazardous wastewater.

Cold Storage: Refrigerator SAR-10A
Cold Storage Sample Removal: 30 Days from date of sample receipt
Cold Storage Removal Frequency: 30 Days
Total Sample Storage Period: 60 Days from date of sample receipt
Waste Stream: non-hazardous wastewater

Note: Water samples are bulked into the non-hazardous wastewater waste stream. The VOA vials are crushed. Once crushed, these glass shards are triple rinsed with water and discarded in the trash.

9.2.4 Unused Client Soil Samples: Unused Client soil samples older than 14 days are removed from the refrigerator and placed into green plastic tote boxes. Tote boxes are labeled with the date of sample receipt. These tote boxes are transferred to the waste area and placed on shelves. Sample are retained for a period of 60 days from the date of sample receipt. Samples are discarded as non-hazardous solids.

Cold Storage :Refrigerator SAR-11A
Cold Storage Sample Removal: Date of analysis or 2 weeks from date of sample receipt
Cold Storage Removal Frequency: Daily
Total Storage Period: 60 Days from date of sample receipt
Waste Stream: non-hazardous solids

Note: Soil samples and their glass jars are bulked into the non-hazardous solids waste stream.

9.4 Hazardous Solids

The following VOC waste streams are bulked together and disposed of as hazardous solids. These waste streams include the following:

9.4.1 Extracted Soil/Solid Samples: VOC extracts older than 30 days are removed from the refrigerator monthly. The extracts are packed into boxes labeled MEOH SOIL EXTRACTS with the date of disposal. The boxes of extracts are transferred to the sample storage area and placed on designated racks in the waste room. After a storage period of 60 days from date of sample receipt, the extracts are discarded as hazardous solids.

Cold Storage: Refrigerator EXT-36B
Cold Storage Sample Removal: 30 Days from date of sample receipt
Cold Storage Removal Frequency: 30 Days
Total Sample Storage Period: 60 Days from date of sample receipt
Waste Stream: Hazardous Solids and Waste Solvent

Note: The methanol is decanted and bulked with the solvent waste. The remaining soil extract and glass containers are crushed together, collected and bulk packed with the hazardous solids.

9.4.2 Methanolic Air Extracts: Methanol air extracts older than 60 days are discarded every 3 months. The methanol and carbon material in the vials are crushed in the autovial crusher and the resulting glass shards are disposed of as non-hazardous solids.

Cold Storage: Refrigerator SAF-6B
Cold Storage Sample Removal: 60 Days from date of sample receipt
Cold Storage Removal Frequency: 90 Days
Total Sample Storage Period: 60 Day from date of sample receipt
Waste Stream: non-hazardous solids

9.5 VOC Solvent Waste

The following VOC subwaste streams are bulked together and disposed of as Solvent Waste and non-hazardous solids. These subwaste streams include the following:

9.5.1 Solvents and Fuel Products: Client samples containing pure products older than 14 days are removed from the refrigerator monthly. Pure product samples are placed in labeled paint cans. Samples are transferred to the sample storage area and placed on VOC storage racks. After a period of 60 days from the date of sample receipt, samples are discarded as waste solvent.

Cold Storage: Refrigerator SAR-2A
Cold Storage Sample Removal: 14 Days from date of sample receipt
Cold Storage removal Frequency: Monthly
Total Storage Period: 60 Days from date of sample receipt
Waste Stream: Waste Solvent

Note: Pure product samples are decanted and bulked with the solvent waste stream. The glass containers are crushed. Once crushed, these glass shards are triple rinsed with water and discarded in the trash bin.

10.0 Semi-volatile/Wet Lab Waste Disposal Procedure

10.1 There are six primary waste streams used in the disposal of Semi-Volatile wastes extracts and spent samples. They are non-hazardous wastewater, hazardous wastewater, waste solvent, non-hazardous solids, and hazardous solids. The following standard operation procedures describe the disposal practices for Semi-Volatile samples and their associated wastes.

A special note concerning all extracts from the semi-volatile extraction laboratory. All extracts can be moved and stored in all parts of both the laboratory and the storage area. This is due to the fact that they have a beneficial use within the laboratory (for analysis, both present and future) and are not hazardous wastes until the time that they are ready for disposal.

All solvent-extracted soils and waters, and all waste solvent generated within the extraction laboratory must stay within the extraction laboratory where they were generated. This is due to the fact that these materials do not have any further beneficial use and are ready for disposal.

10.2 Non-hazardous Wastewater

The following semi-volatile waste streams are bulked together and disposed of as non-hazardous wastewater. These waste streams include the following:

10.2.1 Unused Client Water Samples: Unused client water samples older than 14 days are removed from the refrigerator and moved to the waste laboratory. The samples are stored on racks according to their disposal date and disposed of as non-hazardous wastewater.

Cold Storage: Refrigerator SAR-3A
Cold Storage Sample Removal: 14Days from date of sample receipt
Cold Storage Removal Frequency: Daily
Total Storage period: 60 Days from date of sample receipt
Waste Stream: non-hazardous wastewater

10.2.2 Unused TCLP Extracts: Unused TCLP extracts older than 14 days are removed from the refrigerator and moved to the waste laboratory. The samples are stored on racks according to their disposal date and disposed of as non-hazardous wastewater.

Cold Storage: Refrigerator SAR-3A
Cold Storage Sample Removal: 14Days from date of sample receipt
Cold Storage Removal Frequency: Daily
Total Storage period: 60 Days from date of sample receipt
Waste Stream: non-hazardous wastewater

10.3 Hazardous Wastewater

The following semi-volatile waste stream is bulked together and disposed of as hazardous wastewater. This waste stream includes the following:

10.3.1 Solvent Extracted Water Samples: Spent Water samples extracted with >10% solvent are manifested and disposed of in the extraction laboratory under the waste hood.

Solvent Extracted Water Removal: As needed
Waste Stream: Hazardous Wastewater

10.4 Hazardous Solids

The following semi-volatile waste stream is bulked together and disposed of as hazardous solids. This waste stream includes the following:

10.4.1 Extracted Soil Samples: All solvent extracted soils from the ASE extractor must be manifested and disposed of in the extraction laboratory under the waste hood.

ASE Extracted Soil Removal: As needed
Waste Stream: Hazardous Soil

10.4.2 Saturated Extraction Supplies (Sodium Sulfate/Filters, etc):

All solvent extracted soils, filter paper, extraction discs and sodium sulfate from semi-volatile extractions where the materials have come into contact with solvents must be manifested and disposed of in the extraction laboratory under the waste hood.

Removal: As needed
Waste Stream: hazardous Soil

10.5 Non-hazardous Solids

The following semi-volatile extractable waste stream is bulked together and disposed of as non-hazardous solids. This waste stream includes the following:

10.5.1 Unused Client Samples: Unused Client soil samples older than 14 days are removed from the refrigerator and moved to the waste laboratory. These soil containers are placed on racks according to the sample disposal date.

Cold Storage: Refrigerator SAR-3A
Cold Storage Sample Removal: 14 days from date of sample receipt
Cold Storage Removal Frequency: Daily
Total Storage Period: 60 days from date of sample receipt
Waste Stream: Non-hazardous Solids

Note: Soil samples are bulked into the non-hazardous solids waste stream. The wide mouth glass jars are crushed. Once crushed, the glass shards are disposed of along with the soil as non-hazardous soils.

10.6 Waste Solvent (From the Extraction Laboratory)

The following waste streams are bulked together and disposed of as waste solvent. It is again important to emphasis that all waste solvent generated within the extraction lab must be manifested and disposed of from the extraction lab. Those solvents decanted from the stored samples and autovials in the waste laboratory on Freeport must be manifested and disposed of from the Freeport facility. These waste streams (from the extraction lab) include the following:

10.6.1 Glassware Rinse Solvent: Glassware solvent rinse, ASE waste solvent, waste solvent from standard preparation, and excess waste solvent from extractions are bulked in the extraction laboratory.

10.6.2 Solvent Extracts: ASE generated semi-volatile solvent extracts older than one month are removed from refrigerator EXT 33C. Extracts are placed into boxes labeled SEMI-VOLATILE EXTRACTS with the sample disposal date. These boxes are placed on racks in the waste laboratory and after a period of 60 days from date of sample receipt, the extracts are discarded as waste solvent.

Cold Storage: Refrigerator EXT-33C
Cold Storage Sample Removal: Monthly
Cold Storage Removal Frequency: Monthly
Total Storage Period: 60 days from date of sample receipt
Waste Stream: Waste solvent.

10.6.3 Semi-Volatile Auto Vials: Once semi-volatile analyses are completed, all auto vials are removed from their respective refrigerators. Autovials are packed into paint cans labeled SEMI-VOLATILE AUTO VIALS with a disposal date. The cans are transferred to the waste laboratory and placed on their appropriate rack and stored for 60 days before disposal.

Cold Storage Autovials: Refrigerator EXT-33C
Total Storage Period: 90 Days
Waste Stream: Waste Solvent, Hazardous Solids

Note: Autovials are crushed while collecting the waste solvent for disposal. The crushed glass is then bulked and disposed with the hazardous solids.

11.0 Inorganic Lab Waste Disposal Procedure

11.1 Inorganic Reagents (From the Inorganic Laboratory): The following waste streams are bulked together and disposed of as D-listed metals waste. This waste stream includes such metals as silver, mercury and chrome. The common wastes that are disposed of in this stream are those waste reagents/extracts associated with COD, Cr^{+6}, Sulfide and other such inorganic analyses.

Appendix C

Standard Operating Procedure

SOP C.17
Review of Requests, Tenders, and Contracts

1.0 REVIEW OF REQUESTS, TENDERS AND CONTRACTS

1.1 Often times new work is associated with a request, tender and/or a contract. This may be a long drawn out and complicated process. Occasionally, new work requires no contract, and samples simply arrive unexpectedly at the sample receiving area. This SOP describes the general procedures for the review of requests, tenders and contracts and the maintenance of those contracts.

2.0 STANDARD OPERATING PROCEDURE

2.1 Review Items

The purpose of this review is to establish that we possess the resources necessary for the performance of the contract or project. Items that affect the review of requests, tenders and contracts for acceptance of new work include the following:

a) are the requested methods of analysis adequately defined, documented and understood;

b) do we have the appropriate facilities and resources to meet the contract requirements to include:

i. do we have the required physical space;

ii. do we have the personnel with the skills and expertise for the performance of the tests in question;

iii. do we have the appropriate information and technical resources;

iv. what are the number of samples to be analyzed, and over what time period will they arrive;

v. can these samples be analyzed without overly stressing the current staff;

vi. are their performance evaluation (PE) samples available for the requested method of analysis; and if not are their QC samples available with know values in order to determine uncertainties of measurement, detection limits, confidence limits, or other essential quality control requirements;

c) can we provide the specified methods of analysis to include:

vii. is the work in a state or under an agency which we have approval;

viii. are the requested methods of analysis, methods we are approved for;

ix. are the target analytes, compounds we are approved for and analyze on a normal basis;

d) can we provide the specified data quality objectives to include:

x. the requested reporting limits or LOQs;

xi. the requested accuracy and precision;

xii. the requested turn around times; and

xiii. are we capable of analyzing the constituents in the requested matrix.

This is not a comprehensive list of questions to be determined, but is a general starting point for all new projects. In addition, these do not answer the business questions regarding payment, pricing etc which are always a factor when evaluating new work.

2.2 Review Result

After the initial request of review is completed, the client is informed of the review results, to include:

a) any potential conflict with ongoing projects;

b) any deficiencies in the request for analysis;

c) any lack of appropriate accreditation status; or

d) the inability to complete the client's work.

2.3 Resolution of Differences

Any differences between the requested contract and our capabilities must be resolved before any work commences. The contract, if one exists, must be acceptable to both the laboratory and client.

Note: NELAC defines a contract as any written or oral agreement when providing a client with environmental testing services.

2.4 Documentation of Review

2.4.1 Records of reviews, including any significant contract changes are retained by

Alpha. Most often these are oral contracts and those changes are documented in the "Client Information" section of the Omega LIM system.

2.4.2 These records may also include ongoing discussions with a client relating to the client's requirements or the results of the work during the period of execution of the project or contract.

2.4.3 For the review of routine and other simple tasks, the date and the initials of the person, in the laboratory, responsible for carrying out the contracted work is considered adequate.

For repetitive routine work, the review is made only once at the beginning of the contract or on-going work under a general agreement with the client, provided that the client's requirements remain unchanged.

For new, complex or advanced environmental testing tasks, a more comprehensive record is maintained.

2.4.4 This contract review also includes any work that is subcontracted.

2.5 Deviations from the Contract

2.5.1 It is Alpha's policy to inform the client of deviations from the contract.

2.6 Contract Amendments

2.6.1 If a contract needs to be amended after work has commenced, the same contract review process is repeated and any amendments are communicated to all affected personnel.

2.6.2 Suspension of accreditation, revocation of accreditation, or voluntary withdrawal of accreditation is reported to the client.

Appendix D

Document Control Plan

Appendix D

Standard Operating Procedure

SOP D.1
Document Control Plan

1.0 DOCUMENT CONTROL PLAN (DCP)

2.0 STANDARD OPERATING PROCEDURE

2.1 The opening statement to our QA manual explains the purpose and objectives of our quality assurance system. These statements are fundamental principals upon which we have built our QA system and upon which policy decisions are made. The QA manual devotes much attention to fulfilling these objectives such as:

- Providing a uniform basis for analytical generation and reporting;
- Assisting in the early recognition of deficiencies which affect the quality of data; and,
- Requiring sufficient documentation to verify the quality of data submitted.

2.1.1 The quality assurance system is only as good as the record keeping system. If historical data cannot be maintained and retrieved, and the construction of those historical records are in doubt, than a house-of-cards has been created, and the entire system is brought into question and the data is dubious at best.

2.1.2 In keeping with good laboratory practice, laboratory activities including observations, data production and calculations should be recorded at the time they are made and be identifiable to the specific task.

2.2 Control of Records

It is a basic policy of Alpha to provide accurate, precise, complete, and representative determinations, and to document sufficient QA/QC of the analytical procedures. The procedure designed to document and maintain these as historical records is the Document Control Plan. This plan has established a set of SOPs written specifically for maintaining uniformity and consistency in support of these goals.

2.3 Record Keeping System and Design

The record keeping system is designed for the identification, collection, indexing, accessing, filing, storing, maintenance and disposal of quality and technical records. These records include reports from internal audits, and management reviews as well as records of corrective and preventive actions.

2.3.1 A major component in the design of the record keeping system is a procedure which allows the historical reconstruction of the laboratory activities that produced the analytical data. The sample history is easily reconstructed through this documentation system. Such records include:

2.3.1.1 records identifying the person involved in sampling, sample receipt, sample preparation, calibration and analysis;

2.3.1.2 information relating to laboratory equipment, analytical test methods, and related laboratory activities such as sample receipt, sample preparation or data verification; and

2.3.1.3 sufficient information for each environmental test to help identify factors that may affect the uncertainty of the test result; as well as enabling the environmental test to be repeated under conditions as close as possible to the original sample analysis.

2.3.2 The record keeping system is designed to facilitate the retrieval of working files and archiving records for inspection and verification purposes. This is accomplished by the following description.

2.3.2.1 All document entries are initialed by the responsible staff member. The reason for the signature or initials is indicated on all quantitation and QC reports by using a stamp indicating the checking of analytical data. This stamp describes the basic activities, and the person responsible for those activities, initials it. The stamp is as follows:

- Result,
- Peer/QC,
- Report, and
- Final

2.3.2.2 All generated data, except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent ink.

2.3.2.3 Entries in records are not to be obliterated by methods such as erasures, overwritten files or other markings. All record keeping corrections are made by a single line marked through the error. The individual making the correction will initial and date the correction. This criterion applies to electronic records as well, to avoid the loss or change of original data. When corrections are due to reasons other than transcription errors, the reason for the correction should also be documented.

2.4 Records Management and Storage

2.4.1 All records, certificates and reports are safely stored, held secure and in confidence to the client.

2.4.2 All records are stored in an environment to prevent damage or deterioration and prevent record loss. Records are stored as follows:

- all records are scanned as pdf files and are electronically stored and filed.; and
- when needed, hard copy data is stored off-site with a company specializing in record management.

2.4.3 All records are retained for a minimum of five years.

2.4.4 Records stored or generated by computers are duplicated with either hard copies or write-protected backups.

All electronic data is stored and supported by the hardware and software necessary for the retrieval, see Appendix F for details.

2.4.5 The information necessary for the historical reconstruction of data is maintained with these files. This includes, original observations, (e.g., quantitation reports), calculations and derived data, calibration records and a copy of the test report.

2.4.6 The Document Control Filing System SOP, found in this appendix, describes the identification scheme used to archive and retrieve hard copy data records.

2.4.7 Electronic data is stored and archived in a manner to facilitate retrieval and security. Appendix F, Software Quality Assurance Plan (SQAOP), has two SOPs which describe this process, SOP F.3, Data Collection and Storage, and SOP F.7, Data Archiving.

2.4.8 Alpha has established a record management system for control of laboratory notebooks, instrument logbooks, standard logbooks, and other records for data reduction, validation storage and reporting.

2.4.9 Access to archival information maintained off-site is documented with an access log. Off-site archival records are protected with a company specializing in record management, against fire, theft, loss and environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources.

Appendix D

Standard Operating Procedure

SOP D.2
Document Control Filing System

1.0 DOCUMENT CONTROL FILING SYSTEM

1.1 The document control filing system is designed to retain laboratory records of original observations and other derived data in sufficient detail to establish an audit trail of calibration records, staff records, and issued test reports for a five-year minimum period. These records include reports from internal audits, and management reviews as well as records of corrective and preventive actions.

1.2 This system of document control and record keeping allows for the historical reconstruction of all laboratory activities that produced the analytical data. The record keeping system is designed for the identification, collection, indexing, accessing, filing, storing, maintenance and disposal of quality and technical records.

2.0 STANDARD OPERATING PROCEDURE

2.1 Our goal is to keep one year of the current past files in our main filing room. All older files are scanned and archived on the LIMs server.

2.2 Access Log

2.2.1 Access to off-site archival information is documented with an access log. Off-site archival records are protected with a company specializing in record management, against fire, theft, loss and environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources.

2.2.2 The access log also known as the "File Check-out and Return" log book maintained in the main file room.

Any person who retrieves a file is required to sign the access log. When the file is returned, the person returning the file is also required to sign the log to indicate the file was returned.

There are only two people allowed to re-file: Document Control Officer, and Assistant Document Control Officer. We have specific, designated areas in our main file room for:

- Files that need to be collated,
- Returned files from cabinets,
- New folders ready to be filed in the cabinets.

2.3 Data Retention of Laboratory Support Activities

A description of our data retention policies and procedures is found in SOP D.1, Document Control Plan. The following list describes the activities that are documented and retained.

a) all original raw data, used for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms and other instrument response readout records);

b) the analytical method SOP used for a specific test method to include a description of the specific computational steps used to translate parametric observations into a reportable analytical value;

c) copies of final reports;

d) archived SOPs;

e) correspondence relating to laboratory activities for a specific project;

f) all corrective action reports, audits and audit responses;

g) proficiency test results and raw data; and

h) results of data review, verification, and cross checking procedures.

2.4 Data Retention of Analytical Records

The essential information associated with data analysis, such as chromatograms, tabular printouts, computer files, analytical notebooks, and run logs are also retained. The specific information retained is as follows:

a) laboratory sample ID;

b) date of analysis and time of analysis, if the holding time is 72 hours or less, or when time critical steps are included in the analysis;

c) instrument identification and instrument operating conditions/parameters;

d) analysis type;

e) all manual calculations, e.g., manual integrations;

f) analyst's or operators's initials/signature;

g) sample preparation including cleanup, separation protocols, sample IDs, volumes, weights, instrument printouts, meter readings, calculations, reagents;

h) sample analysis;

i) standard and reagent origin, receipt, preparation, and use;

j) calibration criteria, frequency and acceptance criteria;

k) data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

l) quality control protocols and assessment;

m) electronic data, software documentation and verification, software and hardware audits, backups; and

n) method performance criteria including expected quality control requirements.

2.5 Data Retention of Administrative Records

The following administrative records are also retained:

a) personnel qualifications, experience and training records;

b) records of demonstration of capability for each analyst; and

c) a log of names which include the initial and signatures for all individuals who are responsible for signing or initialing any laboratory record.

2.6 Collating Client Folders

The data records mentioned above are retained in a number of locations. A large portion of the data associated directly with the final analytical data results is stored in the client file.

2.6.1 The following information is stored, maintained and retained in the client files as follows:

- Computer generated chain-of-custody,
- Amended chain-of-custody (if present),
- Client chain-of-custody,
- Sub-contract laboratory chain-of-custody (if present),
- Internal/evidentiary chain-of-custody (if present),
- Work order information (if present),
- Sample Receipt checklist,
- Sample Receipt checklist fax confirmation,
- Final Alpha reports,
- Alpha QA/QC (if present),
- Copy of EDD report (if present),

- Final sub-contract lab reports (if present),
- Sub-contract lab QA/QC (if present),
- Alpha invoice-always make sure than an invoice is present,
- Sub-contract lab invoices (if present),
- Raw Data,
- Final report raw data (initials of the analyst indicating final data are indicated on all of the sheets),
- Screen Reports, re-runs, etc. (indicated on the top sheet),
- Air bills,
- Correspondence, and
- Report of e-mail (pdf file) confirmation.

2.7 Files are scanned as pdf files and hard copy data are retained for approximately one year. All data can be accessed from the LIMs server as a scanned electronic copy.

REPORT CHECK-OUT LIST
Table D.2-1

REPORT NUMBER	NAME	DATE CHECKED-OUT	DATE RETURNED

Appendix D

Standard Operating Procedure

SOP D.3
Document Flow

1.0 LABORATORY SAMPLE DOCUMENT FLOW

1.1 This flow diagram is to assist all personnel in understanding document production flow from sample receipt through analysis to the final report.

2.0 STANDARD OPERATING PROCEDURE

2.1 Sample Receipt - Sample Custodian

Documents: Chain-of-Custody, sample receipt checklist, air bills/shipping manifests, and sample receipt log.

2.2 Sample Storage - Sample Custodian

Documents: Refrigerator Temperature Log and evidentiary COC records.

2.3 Sample Preparations - Extraction Chemist

Documents: Semi-volatile extraction logbook, volatile sample extraction logbook, metals digestion logbook, standards reagent logbook, and balance logbooks.

2.4 Sample Analysis - Analyst

Documents: Instrument sequence logs, maintenance logs, quantitation reports, chromatograms, calculations, draft reports, instrument document control logbook, calibration reports, tunes, corrective action reports, and QC data reports.

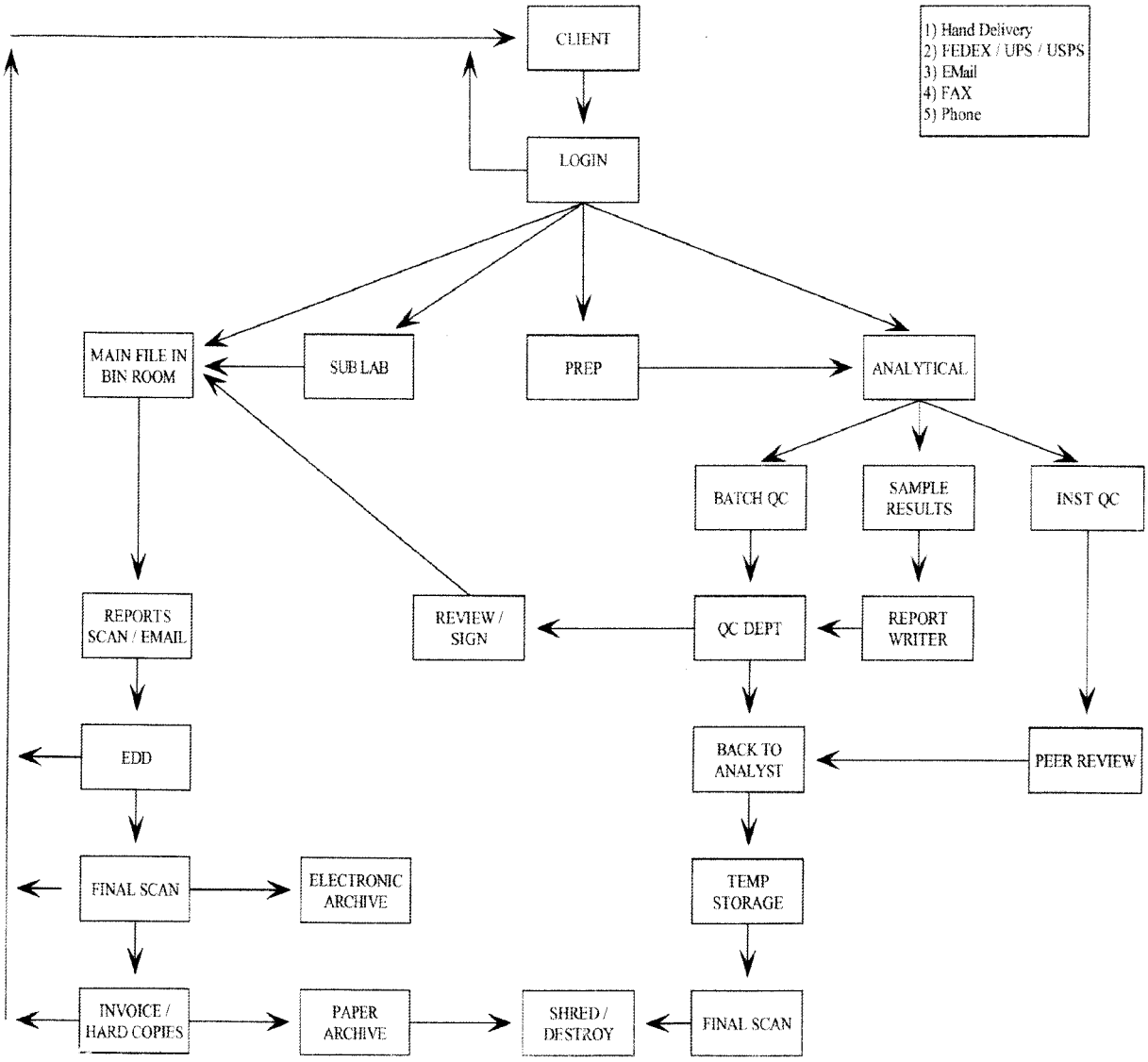
2.5 Data Review - Lab Director/QA officer.

Documents: All previous records, plus: final reports, archived SOPs, correspondence relating to laboratory activities, corrective action reports, proficiency test results and associated raw data and results of data review, verification and cross checking procedures.

2.6 Assembly - Document Control Officer

Documents: All

FIGURE D.3 -1


CLIENT
1) Hand Delivery
2) FEDEX / UPS / USPS
3) EMail
4) FAX
5) Phone
LOGIN
MAIN FILE IN BIN ROOM
SUB LAB
PREP
ANALYTICAL
BATCH QC
SAMPLE RESULTS
INST QC
REPORTS SCAN / EMAIL
REVIEW / SIGN
QC DEPT
REPORT WRITER
EDD
BACK TO ANALYST
PEER REVIEW
FINAL SCAN
ELECTRONIC ARCHIVE
TEMP STORAGE
INVOICE / HARD COPIES
PAPER ARCHIVE
SHRED / DESTROY
FINAL SCAN

Appendix D

Standard Operating Procedure

SOP D.4
Procedure for the Preparation, Review, Approval, Revision and Distribution of the QAM, SOP's and other Technical Documents

1.0 PROCEDURE FOR THE PREPARATION, REVIEW, APPROVAL, REVISION AND DISTRIBUTION OF THE QAM, SOP'S AND OTHER TECHNICAL DOCUMENTS

1.1 It is of fundamental importance and a primary objective that our QA manual, SOP's and other technical documents follow strict adherence to established guidelines for their preparation, review, approval, revision and distribution in maintaining uniformity and consistency of laboratory activities and the documentation that these procedures were being followed historically.

2.0 STANDARD OPERATING PROCEDURE

2.1 This procedure is intended to ensure that all records required under the National Environmental Laboratory Conference (NELAC), Chapter 5 Systems, are retained, as well as establishing the procedures for the control and maintenance through a document control system.

2.2 This procedure ensures that all standard operating procedures, manuals, or other technical documents clearly indicates the time period for which the procedure or document was in force.

2.3 Standard operating procedure formats are modeled after suggestions from the National Environmental Laboratory Conference (NELAC). In addition, the USEPA, Quality Assurance Management Staff (QAMMS), has written guidelines and specifications for elements of a QA plan/manual and recommended formats to be followed and, specifies how plans should be reviewed and approved.

2.4 Document Control

All quality system documents such as the QA Manual, SOPs, Quality Assurance Project Plans (QAPP's) and other technical documents are prepared using a document control format placed in the upper right-hand corner of each document page. This document control number uniquely identifies all quality system documents. The document control number includes the following information:

- Laboratory Name,
- Section Number,
- Revision Number,
- Date (of revision), and
- Page number and total pages of the document

Document control is a key element for referencing and archiving active and historical technical procedures.

2.5 Elements of a QA Manual and QAPP

Each of the sixteen items listed below must be considered for inclusion in each QAPP and are the minimum requirements for a QA Manual:

1) Title page with provision for approval signature,
2) Table of contents,
3) Project/plan description,
4) Project/plan organizations and responsibility,
5) QA objectives for measurements data in terms of precision, accuracy, completeness, representatives and comparability;
6) Sampling procedures,
7) Sampling custody,
8) Calibration procedures and frequency,
9) Analytical procedures,
10) Data reduction, validation and reporting,
11) Internal quality control checks and frequency,
12) Performance and system audits and frequency,
13) Preventive maintenance procedures and schedules,
14) Specific routine procedures to be used to assess data precision, accuracy and completeness of specific measurement parameters involved,
15) Corrective actions, and
16) Quality assurance reports to management

2.6 Standard Operating Procedure

2.6.1 Analytical SOP's are written for each accredited test method. These are maintained in the Procedure Manual. The Procedure Manual consists of copies of the referenced test method as well as the in-house written analytical SOP. In cases where modifications to the published test method have been made or where the referenced test method is ambiguous or provides insufficient detail, these cases are described in the SOPs as Clarification Boxes. The following elements are addressed and documented in each analytical SOP where applicable as follows:

1) identification of test method;
2) applicable matrix or matrices;
3) detection limit;
4) scope and application, including components to be analyzed;
5) summary of the test method;
6) definitions;
7) interferences;
8) safety;
9) equipment and supplies;
10) reagents and standards;
11) sample collection, preservation, shipment and storage;
12) quality control;

13) calibration and standardization;
14) procedure;
15) data analysis and calculations;
16) method performance;
17) pollution prevention;
18) data assessment and acceptance criteria for QC measures;
19) corrective actions for out-of-control data;
20) contingencies for handling out-of-control or unacceptable data;
21) waste management;
22) references; and
23) any tables, diagrams, flowcharts and validation data.

In addition, to items 1) through 23) above, the SOP addresses equipment, instrument maintenance, computer hardware and software and troubleshooting when appropriate.

2.6.2 Non-analytical SOPs are written primarily for those laboratory activities conducive to standardization and procedural formalization. They do not necessarily need to address all 23 elements required for an analytical SOP as stated above.

2.6.3 These documents are internally prepared procedures written with adequate detail to allow someone similarly qualified, other than the analyst, to reproduce the procedures used to generate the test result.

2.7 Preparation and Responsibilities

2.7.1 Standard Operating Procedures

2.7.1.1 It is the responsibility of the Laboratory Director, QA Officer, Supervisors, and all affected personnel to discuss the need for SOP's and other technical documents.

2.7.1.2 It is the responsibility of the Laboratory Director, Laboratory Manager and QA Officer to ensure that all personnel, either technical or non-technical staff, are implementing the tasks described in the SOPs, and/or are made individually aware that changes to a SOP has occurred.

2.7.1.3 SOP's and other technical documents will continue to be written revised and distributed as laboratory activities change with time. All such reviews are documented and made available for assessment. A copy, either paper or electronic, of the updated SOP is made available in close proximity to the work area of the affected personnel.

2.7.2 QA Manual

2.7.2.1 The QA Officer is responsible for the preparation of a written QA Manual that involves all phases of environmental test measurements. The QA Officer must ensure that the QA Manual contains procedures to document and report sufficient information for the assurance of precise, accurate and complete data generation.

2.7.2.2 It is the responsibility of the Laboratory Director, Laboratory Manager and QA Officer to ensure that all personnel, either technical or non-technical staff, are implementing the tasks described in the QA Manual, and/or are made individually aware that changes to the manual has occurred.

2.7.2.3 The QA Officer is responsible for the annual review of the QA Manual. However, the QA Manual may be revised more often and these revisions to the QA Manual are documented through the distribution of an Addendum to the QA Manual or the distribution of a new complete revision of the QA Manual.

2.7.2.4 The QA Officer is responsible for ensuring a copy, either paper or electronic, of the QA Manual is made available in close proximity to the work area of the affected personnel.

2.7.3 Quality Assurance Project Plans

2.7.3.1 Project Managers (PMs) working in close coordination with the QA Officer have a responsibility to see that a written QA Project Plan is prepared for projects requesting site/project specific plans. The elements of a QAPP are typically identified from the QA Manual and written as a document independent from the QA Manual.

2.8 Plan Review, Approval and Distribution

2.8.1 Review and Approval

2.8.1.1 The review and approval of quality systems documents are conducted by the Laboratory Director, Laboratory Manager, supervisors and/or the QA Officer.

2.8.1.2 Changes to documents are reviewed and approved by the same function as that performed by the original review.

2.8.1.3 The reviewing person has the authority to access any of the quality system documents and may use any additional pertinent background information upon which to base the review and approval process.

Note: If practical, the altered document or new text may be identified in the document or appropriate attachments.

2.8.1.4 Completion of reviews and approvals are shown by signatures on the title page of the QA Manual and/or a signature block at the end of the technical SOP.

2.8.1.5 The driving force to revise the QA Manual or other technical documents is typically due to:

1) changing regulations regarding certification;
2) updates or revisions to existing methods of analysis;
3) the promulgations of new methods and/or lab procedures;
4) expansions or contractions of analytical capabilities and/or services;
5) changes in general laboratory policies or procedures; and
6) recommendations and/or findings, resulting from on-site laboratory inspections from external and internal audits.

2.8.2 Distribution

2.8.2.1 The QA Officer is responsible for the distribution of the QA Manual and associated SOP's. The QA Manual will be numbered or inventoried to monitor the distribution of this publication and for the purpose of updating those distributed documents to the affected organizations.

2.8.2.2 The QA Officer is responsible for ensuring only authorized editions of the technical documents, to include the QA Manual and analytical SOPS, are available at all locations where operations essential to the effective functioning of the laboratory are performed.

2.8.2.3 The QA Officer is responsible for promptly removing invalid or obsolete documents to ensure against their unintended use.

2.8.2.4 The QA Officer is responsible for retaining for either legal or knowledge preservation purposes, e.g., for their historical value, obsolete documents.

2.8.2.5 The QA Officer is responsible for maintaining a master list identifying the current status and distribution of documents in the quality system to preclude the use of invalid and/or obsolete documents, see Fig D.4-1.

QA Manual Master List
Document Control
Inventory Log

Alpha Analytical, Inc.
Document: QA Manual
Rev:
Date:

Name	Distribution Number	Date Signed Out	Initial		Date Signed In	Initial
	1					
	2					
	3					
	4					
	5					
	6					
	7					
	8					
	9					
	10					
	11					
	12					
	13					
	14					
	15					

Fig D.4-1

Appendix D

Standard Operating Procedure

SOP D.5
Laboratory Training Program

1.0 LABORATORY TRAINING PROGRAM

1.1 This SOP outlines requirements for initial and continuing demonstration of staff proficiency in the methods or procedures which they perform. The ultimate goal of Alpha's training program is to provide comprehensive, effective, consistent and documented training for employees to ensure reliable and efficient job performance.

2.0 STANDARD OPERATING PROCEDURE

2.1 General

2.1.1 Application

Many factors determine the correctness and reliability of environmental test methods performed by Alpha. These factors include contributions from such things as:

a) human factors;
b) accommodation and environmental conditions;
c) environmental test methods and method validation;
d) equipment;
e) measurement traceability;
f) sampling; and
g) the handling of samples.

The total contribution of these factors differ considerably between methods of analysis. These factors are taken into account when developing analytical methods of analysis, the selection of equipment, training and the qualification of the personnel associated with individual methods of analysis. Therefore, our training program is customized to the individuals needs and the degree of difficulty of a particular procedure.

2.1.2 Definitions

2.1.2.1 Required reading- Reading of documents, procedures or publications identified by the Laboratory Director, QA Officer, Supervisor or Trainer as necessary to fulfill the duties of the position.

2.1.2.2 Method/SOP Training- Used to train analysts to perform analyses without the supervision of the Laboratory Director or another qualified analyst. Also used to train any employee to a task implemented through an approved SOP.

2.1.2.3 Seminars- Classroom type training either led by a company representative or analytical/supply vendor.

2.1.2.4 Work Cells - A work cell is considered to be all individuals who see a sample through the complete process of preparation, extraction and analysis.

2.1.3 Alpha only uses personnel who are employed by or under contract to, the laboratory. When contract labor is used, whether to fulfill a technical or non-technical position, Alphas ensures that such personnel are supervised and competent and that they work in accordance with the laboratory's quality system.

2.2 Summary

2.2.1 Several types of training tools are used for staff training, including:

a) Required reading,
b) Method/SOP training,
c) Seminars,
d) Vendor supplied training,
e) Individual mentoring by a senior staff member,
f) MDL/IDC studies,
g) Performance Evaluation (PE) samples and
h) Multi-Media Interactive Computer Presentation.

2.2.2 Training files are maintained by each employee for the current year and historical records are maintained by the Training Coordinator. These files contain all associated training documents.

2.2.3 Safety, when not the topic of the training session, will be stressed during the applicable training sessions.

2.2.4 All training conducted by Alpha is documented.

2.3 Responsibilities

2.3.1 Management Responsibilities

Laboratory management is responsible for formulating training goals with respect to education, training and skills of laboratory personnel. This includes the procedures for identifying training needs and to provide the training of personnel. A basic goal of Alpha's training policy is to ensure all staff members are trained accordingly. This is to include:

- initial and on-going or continuing training;
- ensures all staff members are supervised, competent and implementing the procedures described in the QA Manual and Procedural Manul;
- ensures staff members are made aware of changes to an SOP; and
- ensures a copy, either paper or electronic, of the updated SOP is available in close proximity to the work area;

In addition, Alpha:

- maintains current job descriptions for all personnel who manage, perform, or verify work affecting the quality of environmental testing;
- authorizes and assigns specific personnel to perform environmental testing, who may issue test reports, give opinions and interpretations and who may operate particular types of equipment;
- maintains records of the relevant authorization, competence, educational and professional qualifications, training, skills and experience of all technical personnel including contracted personnel; and,
- makes available the information regarding when the authorization and/or competence is confirmed.

2.3.2 Training Coordinator Responsibilities

The Training Coordinator is responsible for the implementation of the Training Program. The Training Coordinator will remind Trainers to conduct training for each staff member. It is the responsibility of the Training Coordinator to ensure that all historical staff training records are retained. It is the responsibility of the Training Coordinator to ensure that each staff member new and old are assigned the appropriate Trainers. It is the responsibility of the Training Coordinator to ensure that new staff members have been given a laboratory orientation to include required documentation found in the Training SOP.

2.3.3 Trainer Responsibilities

Each staff member is placed under the direct supervision of a senior staff member upon entering employment at Alpha. The senior staff member will conduct an orientation meeting at which time a training guide listing those training requirements will be discussed. The trainer will provide the new staff

member with a training log along with this training program document which will list their required annual training. It is the responsibility of the senior staff member to ensure that assigned staff members under their guidance are given adequate training to perform their duties. Trainers are generally assigned as follows:

- QA Officer will oversee general QA procedures for all staff members;
- LIMS Administrator will oversee all LIMS and PC training;
- GC/MS Supervisor will oversee all GC/MS analysts;
- IC/HPLC Supervisors will oversee all IC/HPLC analysts;
- GC/FID Supervisors will oversee all GC/FID analysts/extraction chemists;
- VOC Prep Supervisor will oversee all VOC extraction chemists;
- SV Prep Supervisors will oversee all SV extraction chemists;
- Metals Supervisor will oversee all metals and wet chemistry chemists;
- Sample Custodian will oversee all personnel associated with these duties; and
- Safety Officer will oversee safety training for all employees.

2.3.4 Trainee Responsibility

It is the responsibility of all Trainees to ensure that they have been adequately trained in their duties as prescribed in the various SOPs. It is the responsibility of the Trainees to ensure all training, to include initial and continuing training, has been properly documented by the Trainer and Training Coordinator.

2.4 Annual Training Requirements

2.4.1 Each staff member who performs sample preparation, analytical procedures or is a technical staff member of a work cell must demonstrate his/her ability to successfully execute each method. In order to qualify to perform a given analytical method, that staff member must, at a minimum, successfully process an IDC study. After initial qualification on a given method, staff proficiency is demonstrated on a continuing basis. At least annually each staff member who performs a method must successfully produce a continuing demonstration of capability study.

2.4.2 Laboratory Ethics/Fraud Prevention and Data Integrity Program

Training courses in ethical and legal responsibilities including the potential punishment and penalties for improper, unethical or illegal actions are conducted and documented on an annual basis.

2.4.3 Laboratory Ethics/Fraud Prevention and Manual Integration Program

Training is conducted annually for all analysts regarding manual integration. This training outlines the use of manual integrations, how manual integrations are to be produced, documentation of manual integrations, analysts responsibilities and the potential penalty for improper, and/or unethical use of manual integrations.

2.5 Procedure

2.5.1 Seminars

Seminars are used to train employees on general topics such as health and safety. Seminars are generally conducted by the Laboratory Director, QA Officer or a Laboratory Supervisor.

Seminar topics and course objectives will be approved by the Laboratory Director, QA Officer or Supervisors.

Seminar attendance will be assigned by the Laboratory Director, QA Officer or Supervisors, and notices will be sent to all affected employees. Upon completion of the seminar, the individual conducting the seminar will ensure the seminar is documented on the individual training records of all attendees.

2.5.2 Required Reading

Required reading is used to convey important information to the staff. Required reading is of two types: 1) reading which does not require attendance at a seminar or "hands on" experience; and 2) reading which is part of mandatory attendance at a seminar. Typical required reading would be such things as: 1) EPA regulatory information; 2) company polices; 3) technical articles; and 4) SOPs which the employee performs. The trainer will document these requirements.

2.5.3 Method/SOP Training

This form of training is used for new employees, training of analysts for new methods and SOPs, and on-going training. The training is documented on their individual training records.

2.5.4 Vendor Training

Employees may be trained by a Vendor representative either at our facility or off-site. If this training is conducted at our facility, the training is documented on their individual training record.

2.6 Training

2.6.1 General

Each new employee is given a personal laboratory notebook. This notebook is a standard laboratory notebook for employees to use as they deem necessary. Alpha encourages employees to write notes in this logbook which may help them understand the required material.

In addition, each employee is given a Personal Training Logbook. This is a standardized logbook used by all employees to document laboratory wide required training. General topics to be included in a Personal Training Logbook include:

- dates and descriptions of training,
- detailed information regarding the training topics discussed,
- changes to a method/SOP or general laboratory practice or policy,
- audit deficiencies, and
- documentation of annual training.

All training is assigned by the Laboratory Director, QA Officer or Supervisor. All new employees will be under the direct supervision of a senior staff member for their initial and continuing training. SOP training for non-analysts are under the direct supervision of a senior staff member.

2.6.2 Initial Demonstration of Proficiency

A new analyst/extraction chemist is one who has not met the training requirements for a method or any analyst/extraction chemist who has not performed the method for greater than 1 year. Before a new analyst/extraction chemist can perform work independently on a method, the following steps should be completed:

- the analyst/extraction chemist will read all pertinent QA and method SOPs, EPA methods, manual or other documents as assigned by the trainer;

- the analyst will perform a minimum of 2-4 analytical sequences under the direct supervision of an experienced analyst;

- the extraction chemist will perform a minimum of 2-4 extraction batches under the direct supervision of an experienced extraction chemist; and

- the analyst will complete the outlined training tasks, including an MDL/IDC study.

2.6.3 Continued Demonstration of Proficiency

Each analyst/extraction chemist who has met the initial training criteria for a method must demonstrate and document continued proficiency by at least one of the following once per year:

- acceptable performance of a blind sample;
- another demonstration of capability, i.e. in-house PE;
- successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS Volatiles by purge and trap for methods by either 524.2, 624 or 5035/8260); or
- at least 4 consecutive laboratory control samples with acceptable levels of precision and accuracy.

Training/Continuing Education Program

Statement of Training Responsibilities:
I have received, read, understood and will implement all relevant items described in the QA Manual and all relevant addendums.

Signature:_____
Employee:_____
Years in Service:_____
Department:_____

QA Manual Revision: 17.0
Date:_____
Issued:_____
Supervisor: _____

#	Training Code	Description	Training Time	Date Completed	Employee Initial	Trainer Initial
1	RR/SEM	**QAM Vol. I, Section 3.0** General Statement of Policy				
2	RR/SEM	**QAM Vol. I, Section 4.0** Organization and Responsibility				
3	RR/SEM	**QAM Vol. I, Section 5.0** Quality Assurance Objectives				
4	RR/SEM	**QAM Vol. I, Section 6.0** Sampling Procedures				
5	RR/SEM	**QAM Vol. I, Section 7.0** Sample Custody				
6	RR/SEM	**QAM Vol. I, Section 8.0** Analytical Procedures				
7	RR/SEM	**QAM Vol. I, Section 9.0** Calibration Procedures and Frequency				
8	RR/SEM	**QAM Vol. I, Section 10.0** Instrument Maintenance				
9	RR/SEM	**QAM Vol. I, Section 11.0** Quality Control Checks				
10	RR/SEM	**QAM Vol. I, Section 12.0** Data Reduction, Validation and Reporting				
11	RR/SEM	**QAM Vol. I, Section 13.0** Corrective Actions				
12	RR/SEM	**QAM Vol. I, Section 14.0** Performance and System Audits				
13						
14						

Continuing Education Program

Employee: _____ **Issued:** _____

#	Training Code	Description	Training Time	Date Completed	Employee Initial	Trainer Initial
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						

Continuing Education Program

Employee: _____ **Issued:** _____

#	Training Code	Description	Training Time	Date Completed	Employee Initial	Trainer Initial
26						
27						
28						
29						
30						
31						
32						
33						
34						
35						
36						
37						
38						
39						
40						
41						
42						
43						
44						
45						
46						
47						
48						
49						
50						

Continuing Education Program

Employee: _____ **Issued:**_____

#	Training Code	Description	Training Time	Date Completed	Employee Initial	Trainer Initial
51						
52						
53						
54						
55						
56						
57						
58						
59						
60						
61						
62						
63						
64						
65						
66						
67						
68						
69						
70						
71						
72						
73						
74						
75						

Continuing Education Program

Employee: _____ **Issued:**_____

#	Training Code	Description	Training Time	Date Completed	Employee Initial	Trainer Initial
76						
77						
78						
79						
80						
81						
82						
83						
84						
85						
86						
87						
88						
89						
90						
91						
92						
93						
94						
95						
96						
97						
98						
99						
100						

Safety Education Program

Employee: _____ **Issued:** _____

#	Training Code	Description	Training Time	Date Completed	Employee Initial	Trainer Initial
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						

Personnel Information

Name:
Title:
Laboratory: Alpha Analytical, Inc.
Street: 255 Glendale Ave., Ste. 21
City: Sparks State: NV Zip Code: 89431

Education				
	Name and Address	Semester Credit Hours in Chemistry and Biology	Year Graduated	Degree and Major Area of Study
High School				
Technical School or College				
Graduate School				

Experience			
Laboratory & Address	Date Employed		Duties Including specific analyses performed
	From	To	
Alpha Analytical, Inc Sparks, NV			

Employee Name _____ Signature _____ Date _____

Certification
of
Education/Technical Background

Alpha Analytical, Inc.
255 Glendale Ave., Ste 21
Sparks, NV 89431

I the undersigned, **CERTIFY** that:

1) The educational and/or training requirements as specified in Alpha's QAM have been met.

2) The representation of my college degree and/or additional training is correct.

Degree	School of Degree	Date

Name	Signature	Date

Laboratory Ethics / Fraud Prevention
and
Data Integrity Program

Alpha Analytical, Inc.
255 Glendale Ave., Ste 21
Sparks, NV 89431

I the undersigned, **CERTIFY** that:

I have read, acknowledged and understand the personal, ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions.

This includes, data integrity and/or data authentication issues such that the analytical process can be completely reviewed by recreating the paper trail.

Employee Name	Signature	Date

Senior Management Name	Signature	Date

Laboratory Ethics / Fraud Prevention

Program for Manual Integration

Alpha Analytical, Inc.
255 Glendale Ave., Ste 21
Sparks, NV 89431

The following scenarios constitute an ethics and/or fraud violation when integrating analytical data:

a) No manual integration will be performed exclusively on QC samples in order to meet QC acceptance criteria. Manual integration is acceptable as long as consistency rules are applied to all standards and samples, and there is a reason why the manual integration is necessary.

b) Under no circumstance is peak shaving allowed for the sole purpose for obtaining QC acceptance.

c) Under no circumstance may an analyst add area under the baseline of a peak, in an effor to create additional peak area.

These are specific examples of fraudulent and unethical manual integration practices, and are not intended to be a comprehensive list; rather this list is intended to give the analyst an idea of the types of unethical behavior that will not be tolerated.

I the undersigned, **CERTIFY**, that:

I have read, acknowledged and understand the personal ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions regarding manual integrations.

Employee Name	Signature	Date

Certification
of
Initial and/or Continued Proficiency Requirement

Alpha Analytical, Inc.
255 Glendale Ave., Ste 21
Sparks, NV 89431

I the undersigned, **CERTIFY** that:

I have read, understood, and agree to perform the most current version of the test method, standard operating procedure and to keep current the initial and continued proficiency requirements for the following:

Method	**SOP**			**Initial**
	Doc No	**Revision**	**Date Issued**	
EPA Method 524.2	E.20			
EPA Method 608/8081A	E.30			
EPA Method 8082	E.31			
EPA Method 624/8260B	E.33			
EPA Method 625/8270C	E.34			
EPA Method 8270C SIMs	E.35			
EPA Method 8260B SIMs	E.36			
EPA Method 8015B/D-DRO/NW-TPHdx	E.37			
EPA Method 8015B/D-GRO/NW-TPHgx	E.38			
RSK-175 Dissolved Gases	E.40			
EPA Method 1311 TCLP	E.50			
EPA Method 1312 SPLP	E.51			
DHS Method (WET) Waste Extraction Test	E.52			
Organic Acids	E.64			
EPA Method 300.0/9056	E.65			
EPA Method 314.0	E.66			
EPA Method 9060A/SM5310C	E.67			

Name _____ Signature _____ Date _____

Certification
of
Initial/Continued Proficiency Requirement

Alpha Analytical, Inc.
255 Glendale Ave., Ste 21
Sparks, NV 89431

I the undersigned, **CERTIFY** that:
I have read, understood, and agree to perform the most current version of the test method, standard operating procedure and to keep current the initial and continued proficiency requirements for the following:

Method	**SOP**			**Initial**
	Doc No	**Revision**	**Date Issued**	
EPA Method 200.8/6020 (Metals)	E.60			
EPA Method 3015 Aq Microwave Digestion	E.70			
EPA Method 3051 Solid Microwave Digestion	E.71			
EPA Method 200.2/3010 Block Digestion	E.72			
EPA Method 120.1/9050A/SM2510B (Conductivity)	E.75			
EPA Method 150.1/9040C/9045D (pH)	E.76			
Standard Method SM4500NH3 D (Ammonia/TKN)	E.77			
EPA Method 180.1 (Turbidity)	E.78			
Standard Method SM4500 O G (DO)	E.79			
Standard Method SM2540 (TDS/TSS/TS)	E.80			
ASTM 2216 (Percent Dry Weight and Percent Moisture)	E.81			
EPA Method 1664A (Oil and Grease)	E.82			
Standard Method SM2310B (Acidity)	E.85			
Standard Method SM2320B (Alkalinity)	E.86			
Standard Method SM5210B (BOD)	E.87			
EPA Method 7196A/SM 3500-Cr D Cr(VI)	E.90			
Standard Method SM4500-S D (Sulfide)	E.92			
Standard Method SM4500-Cl G (Residual Chlorine)	E.93			
Standard Method SM 5520-D (COD)	E.94			
EPA Method 365.3/SM4500P E (Total Phosphorus)	E.95			
Standard Method SM3500-Fe D (Ferrous Iron)	E.96			

Name _____ Signature _____ Date _____

PERFORMANCE EVALUATION
(PE)
SUMMARY DATA

Study Number/Date	Analyte/Method	Acceptance (Yes/No)

INITIAL DEMONSTRATION OF CAPABILITIES
(IDC)
SUMMARY DATA

Method	SOP	IDC Date

METHOD DETECTION LIMIT
(MDL)
SUMMARY DATA

Method	SOP	MDL Date

QUARTERLY LOD/LOQ
SUMMARY DATA

Method	SOP	LOD/LOQ Study Date
		1st Qtr
		2nd Qtr
		3rd Qtr
		4th Qtr
		1st Qtr
		2nd Qtr
		3rd Qtr
		4th Qtr
		1st Qtr
		2nd Qtr
		3rd Qtr
		4th Qtr
		1st Qtr
		2nd Qtr
		3rd Qtr
		4th Qtr
		1st Qtr
		2nd Qtr
		3rd Qtr
		4th Qtr
		1st Qtr
		2nd Qtr
		3rd Qtr
		4th Qtr

Appendix D

Standard Operating Procedure

SOP D.6
Signature Log

1.0 EMPLOYEE SIGNATURE/INITIAL LOG

1.1 An employee signature and initial log is recorded and maintained by Alpha in order to identify personnel from their initials or signatures on laboratory documents. The list contains the analyst's typed names, initials, written signatures, and written initials.

TABLE D.6 - 1

LABORATORY SIGNATURE LIST

Name	Initials	Signature	Initials
Ami Awano	AA		
Behrooz Aryainejad	BA		
Cheryl Gamble	CG		
Corinne Miner	CM		
Darin Hussey	DH		
David Maestas	DM		
Elizabeth Adcox	EA		
Emily Steele	ES		
Guanzhen Ji	GJ		
Jason Herrmann	JH		
Jeff Yoshimoto	JY		
Jennifer Webster	JW		

Name	Initials	Signature	Initials
Joseph Mauro	JM		
Kathryn Murray	KM		
Melanie Wickham	MW		
Michael Aseltine	MA		
Randy Gardner	RG		
Reyna Vallejo	RV		
Rickey Overton	RO		
Roger Scholl	RS		
Tammy Brace	TB		
Tara Dickinson	TD		
Tom Wickham	TW		
Walter Hinchman	WH		
Wei Wu	WW		
Zhen Jin	ZJ		

Appendix D

Standard Operating Procedure

SOP D.7
Instrument Sequence Log

1.0 INSTRUMENT SEQUENCE LOGBOOK RECORD KEEPING PROCEDURE

1.1 Quality control can only be proven by the documentation that it exists. QC data, therefore, is fundamental in the operation, implementation and maintenance of a QA plan. The need for a simple yet efficient procedure for this activity is paramount in reconstructing historical analytical records.

2.0 STANDARD OPERATING PROCEDURE

2.1 For each instrument there is an associated analytical data control record keeping file system. This record keeping file system is uniquely associated with a particular instrument for the sole purpose of reconstructing historical analytical records.

2.2 Each analytical run made on an instrument is partially or completely documented in the Instrument Sequence Logbook. The following is a description of how to maintain and document these analytical runs.

2.3 At the end of the analytical sequence a photocopy of the instrument sequence logbook is placed at the beginning of the analytical quantitation reports. This serves as an index to the data which follows.

2.4 Analytical runs are placed in chronological order as they were analyzed on the analytical sequence log.

2.5 Final data, which is placed in the client file, need not be photocopied. However, data such as CV's, tunes, blanks and other QC data should be included in the logbook with the analytical sequence data.

2.6 The only exception to this policy is the final data which is placed into the client file.

2.7 The reconstruction of an analytical sequence(s) is easily accomplished by the use of this record keeping system and the associated client files.

2.8 This record keeping system also serves as a secondary backup to the original instrument logbook.

Appendix D

Standard Operating Procedure

SOP D.8
Maintenance Log

1.0 INSTRUMENT SEQUENCE AND MAINTENANCE LOGBOOK

1.1 All analytical instruments have two separate log books: 1) the Instrument Sequence Logbook; 2) and the Maintenance Logbook. The log books are annotated by the analyst to track and monitor all activities associated with that particular instrument. Complete documentation is a mandatory criteria that all personnel abide by

2.0 STANDARD OPERATING PROCEDURE

2.1 Instrument Sequence Logbook

This is not a complete list of all the types of activities which can be documented in the log book. However, the following is a list of the minimum information which should be annotated on a daily basis.

- Date;
- Instrument conditions, if they have been changed from the previous days conditions;
- Minor instrument adjustments such as changing the multiplier etc.;
- Full descriptions of each analytical run including: sample/client identification, amount injected or purged, analytical run# or file ID;
- Initials on each page; and
- If the instrument is operating other than normal, state conditions.

2.2 Instrument Maintenance Logbook

Any type and description of the maintenance performed on an instrument should be annotated in this book. There are no required information; however, common sense should indicate the types of entries that should be placed into this logbook.

2.3 All log book entries are documented with the date and the initials of the person making the entry.

Appendix D

Standard Operating Procedure

SOP D.9
Analytical Balance Logbook

1.0 ANALYTICAL BALANCE PROCEDURE

1.1 Balances are the primary analytical test equipment used for several gravimetric methods of analysis and are crucial instruments in support of various other analytical methods. In either case, final quantitative results are dependent on their accuracy and the need to track, monitor, and document their status is important to maintaining data integrity.

2.0 STANDARD OPERATING PROCEDURE

2.1 The following procedure details the practices, operations, performance checks and acceptance criteria used to monitor our laboratory analytical balances.

2.2 Definitions

2.2.1 Analytical balance - an analytical balance is used to measure mass to a very high degree of precision and accuracy. The measuring pan of a high precision, usually 0.1 mg or better, analytical balance is inside a transparent enclosure with the doors so that dust does not collect and so any air currents in the room do not affect the balance's operation.

2.2.2 Top-loading balance - a top-loading balance is a type of scale, usually digital, which measures the mass of an object after that object is placed on a metal platform which rests on top of the scale. Usually a top loading balance measured in grams, but many are also able to convert to other measurements units such as milligrams, ounces or pounds. Generally, this type of scale can measure the weight of items anywhere from one up to three decimal places, which means some top-loading balances can round to the nearest thousandth of a gram or one milligram.

2.3 Daily Performance Checks and Accuracy Criteria

2.3.1 Purpose - To ensure the quantitative results produced by the analytical balances are accurate by verifying the balance calibration.

2.3.2 Frequency - Verify daily or before use with S - class weights.

2.3.3 Procedure - Perform the daily balance check, using two S class weights chosen to bracket the anticipated target weight and record the weights of the balance checks in the balance logbook.

2.3.4 Acceptance Criteria - The results of the daily performance check must be within the acceptance criteria to be acceptable. They are as follows:

a) Top-loading balance - ± 2% or ± 0.02 g, whichever is greater.

b) Analytical balance - ±0.1% or ± 0.5 mg, whichever is greater.

2.3.5 Corrective Action - If the results are not within the specified guidelines then:

1) the balance is removed from service until repaired and/or recalibrated; or

2) the balance can be used if a correction factor can be reliably established and maintained.

2.4 Semi-annual Scheduled Maintenance and Traceability Check

2.4.1 Purpose - To ensure quantitative results produced by the analytical balances are accurate using second source balance weights.

2.4.2 Frequency - This maintenance and traceability check is performed twice a year (semi-annually).

Note: NELAC requires only an annual servicing by a certified technician.

2.4.3 Procedure

2.4.3.1 A analytical balance service company is contracted to provide semi-annual preventative and scheduled maintenance. This service call includes cleaning, lubricating, and adjusting all balances to the original manufactures specifications and testing for errors. In addition, built in weights are cleaned and tested for errors.

2.4.3.2 After balances have been serviced, they are calibrated and checked against NIST traceable weights to verify accuracy. The calibration also includes a linearity and corner-load check.

2.4.3.3 Balances are then certified for use with certificates of accuracy.

2.4.3.4 All records of repair and maintenance activities, including the documentation of certificates of accuracy, are kept by a lab technician responsible for these duties.

2.4.4 Acceptance Criteria - Manufacturers specification.

2.4.5 Corrective Action - If the results are not within the specified guidelines:

1) the balance is removed from service until repaired; or

2) the balance can be used if a correction factor can be reliably established and maintained.

2.5 S Class Weights

2.5.1 Purpose - To ensure the mass of the S class weights are within the prescribed accuracy criteria used for verifying laboratory balances on a daily basis.

2.5.2 Frequency - S class weights used for daily balance accuracy check are certified at a minimum every 5 years.

2.5.3 Procedure - These weights are sent to a company certified under ISO/IEC 17025-1999 ANSI/NCSL Z540-1-1994 program to perform this procedure.

They use reference standards traceable to NIST using NIST IR 6969 SOP 4 Double Substitution Weighing Design Protocol.

2.5.4 Acceptance Criteria - The S class weights are verified against NIST mass acceptance criteria.

2.5.5 Corrective Action - If the results are not within the specified guidelines:

1) the S class weight is removed from service until repaired; or

2) the weight can be used if a correction factor can be reliably established and maintained.

TABLE D.9-1
GENERAL GUIDELINES FOR BALANCE WEIGHT ACCEPTABILITY

Balance Model	Balance Type	Location	Alpha ID	Sensitivity	True Weight	Range of Acceptability	Criteria
Mettler AB-204S	Analytical	organic ext lab	1A	0.0001 g ± 0.1 mg	5.0 mg	5.5 - 4.5 mg	0.5 mg
					5.00 g	5.005 - 4.995 g	0.1 %
Mettler PM-300	Top-loading	organic ext lab	2A	0.01 g ± 10 mg	500 mg	490 - 510 mg	10 mg or 2% *
					200.0 g	199.8 - 200.2 g	0.1%
Mettler PB602S	Top-loading	volatile prep lab	3A	0.01 g ± 10 mg	5.00 g	5.01 - 4.99 g	0.2 % *
					50.0 g	50.05 - 49.95 g	0.1 %
Mettler AJ-100	Analytical	volatile prep lab	4A	0.0001 g ± 0. 1mg	5.0 mg	5.5 - 4.5 mg	0.5 mg
					5.00 g	5.005 - 4.995 g	0.1 %
Mettler AB-204S	Analytical	inorganic wet chem	5A	0.0001 g (± 0.1 mg	5.00 g	5.005 - 4.995 g	0.1 %
					50.0 g	50.05 - 49.95 g	0.1 %
Mettler AB-204S	Analytical	inorganic Metals	6A	0.0001 g ± 0.1 mg	0.10005 g	0.10005-0.0995 g	0.5mg
					5.00 g	5.005 - 4.995 g	0.1 %
					50.0 g	50.05 - 49.95 g	0.1 %
Mettler AB-204S	Analytical	inorganic gravimetric	7A	0.0001 g ± 0.1 mg	5.00 g	5.005 - 4.995 g	0.1 %
					50.0 g	50.05 - 49.95 g	0.1 %
Sartorius BP-310S	Analytical	TPH-D ext lab	8A	0.00 1g ± 1 mg	500 mg	501 - 499 mg	1 mg or 0.2 %*
					50.0 g	50.05 - 49.95 g	0.1 %
Mettler AB-204S	Analytical	TPH-D ext lab	9A	0.0001 g ± 0.1 mg	500 mg	500.5 - 499.5 mg	0.1 %
					50.0 g	50.05 - 49.95 g	0.1 %

Note: Criteria is established as 0.1% or 0.5mg, whichever is larger.
Note: * Criteria check is at its limit of sensitivity.

Appendix D

Standard Operating Procedure

SOP D.10
Extraction/Digestion Logbook

1.0 SAMPLE EXTRACTION/DIGESTION LOG

1.1 All sample preparations are documented in the Sample Extraction/Digestion Logbook. Any changes in the protocol during preparation must be authorized by the Laboratory Director.

2.0 STANDARD OPERATING PROCEDURE

2.1 The following information is recorded by the LIM System:

- Batch No.;
- Matrix;
- Analysis;
- Date;
- Page;
- Extraction method;
- Cleanup procedure;
- Solvent;
- Initials of extraction chemist;
- Crucible number if applicable;
- AAI Lab ID No.;
- Client ID;
- Sample weight information;
- Remarks on odor or color, etc.;
- Information on surrogates or spike solution added; and
- Information on any specific difficulties (i.e. emulsions) with sample.
-

2.2 The extraction or digestion procedure used is referenced with adequate information to repeat the procedure at a later date.

- 3510 - Separatory funnel extraction;
- 3545 - Accelerated solvent extraction;
- 3550 - Sonication extraction;
- 3511 - Aqueous micro-extraction;
- 3570 - Soil micro-extraction;
- 5035 - Closed system purge-and-trap of soils;
- 200.2 - SDWA metals block digestion;
- 3015 - Microwave assisted digestion of waters; and
- 3051 - Microwave assisted digestion of soils.

2.3 The sample cleanup procedure used is referenced such as:

- (AW) Sulfuric acid cleanup (SW 3665A);
- (SG) Silica gel cleanup (SW 3630C);
- (SGM) Modified silica gel cleanup (NWTPH-dx);

- (Al) Alumina cleanup (SW3610B);
- (Al-TPH) Alumin cleanup for TPH) (SW3611B); and,
- (Ba/Ag/H) Perchlorate pre-treatment cleanup (314.0 x-up).

2.4 At the end of an extraction, the extracted material is labeled with the AAI sample number, solvent, date, initial and analytical method.

2.5 If an error is made, a single line is drawn through the information, dated and initialed.

2.6 LIMS Procedure for starting a Preparation Batch

2.6.1 Single click left button of mouse on "sample prep";

2.6.2 Click left button of mouse on "Add" in the upper right corner of the screen;

2.6.3 Type in the method number followed by an underline and P or B (no spaces) in the space provided, designated "Prep Code" i.e., 515_P or use select key;

2.6.4 Choose the name of the technician, who will be extracting these samples, by clicking on the downward pointing arrow adjacent "technician";

2.6.5 If all samples available are to be extracted click on "User Select". If samples are not to be extracted deleted them from the user select screen.

2.6.6 Type in the necessary number of matrix spike samples in this column by typing the client ID followed by the client number and MS, i.e., 09101540-01 AMS;

2.6.7 Click on "Reagents/Spikes" positioned left of center at the top of the screen;

2.7 After all spikes and surrogates are recorded click "main" at the top left of the screen to return to the main page of this prep Batch.

2.8 Choose an "end date" for when this batch will be ready for analysis and enter "end date".

2.9 Exit the file by using the icon in the upper left hand corner. Upon exiting this file the computer will ask to "complete the status of this Prep Batch report," yes or no. Once this has been completed, the Prep Batch and all associated samples are annotated in the LIMS system.

Appendix D

Standard Operating Procedure

SOP D.11
Annual Thermometer Calibration Procedure

1.0 ANNUAL THERMOMETER CALIBRATION PROCEDURE

1.1 Method SM250B: Determination of Temperature Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

2.0 Standard Operating Procedure

2.1 Temperature readings are used to monitor the thermal preservation of all samples received at the laboratory and for environmental samples stored in the laboratory. In addition, thermometers are used in a variety of other analytical techniques in the laboratory such as in the calculation of alkalinity, in studies of saturation and stability with respect to calcium carbonate, etc.

2.2 For laboratory operations which report temperature, and for reference thermometers, temperature measurements should be made preferably with a spirit-filled or digital thermometer. The spirit-filled thermometer should have a scale marked for every 0.1 to 0.2° C, with markings etched for glass capillary thermometers.

2.3 Thermometers are assigned a unique identifier when calibrated and placed into service. This identifier is an internal sequential number assigned by QA and is used to identify the thermometers for calibration and temperature monitoring logs.

2.4 Definition

Note: Most laboratory errors in temperature measurement results from using the wrong thermometer and the incorrect usage of that thermometer.

2.4.1 Partial Immersion Thermometers

A partial immersion thermometer is designed to indicate the actual temperature when a specified portion of the thermometer stem is exposed to the temperature being measured. Correct temperature readings can only be obtained when the partial immersion thermometer is immersed in the fluid to the level (mm) marked on the stem or to the inscribed ring.

Note: The immersion line is a quick and easy visual indication to the user. The thermometer should be immersed to this line for correct temperature indication.

2.4.2 Total Immersion Thermometers

A total immersion thermometer is designed to indicate the actual temperature

when the bulb and the entire liquid column are exposed to the temperature being measured. In practice, a short length of the liquid column (usually one-half inch) is permitted to extend above the surface of the liquid being measured to allow reading of the thermometer.

For example, if you are checking a temperature of 35°C, the 35°C temperature mark of the thermometer stem and mercury or spirit level will be just below or above the top of the fluid.

Note: Most total immersion thermometers can also be used in a condition of complete immersion, where the entire thermometer is exposed to the temperature being measured, as with the inside of a refrigerator, freezer, incubator or other chamber.

Note: Total immersion thermometers are sometimes a little tricker to identify. Some manufacturers inscribe TOTAL or TOTAL IMMERSION on the reverse of the thermometer, but this is not an industry-wide practice. The photo above is a typical total immersion thermometer; there is no immersion line, and these is no TOTAL IMMERSION marking on the reverse. If there is no inscription on the reverse indicating immersion, it should be assumed the thermometer is designed for total immersion.

2.4.3 Complete Immersion Thermometer

A complete immersion thermometer is designed to indicate the actual temperature when the entire thermometer is exposed to the temperature being measured.

2.5 Thermometer Calibration

2.5.1 Purpose - To ensure the quantitative results displayed by the laboratory thermometers are accurate by a process of temperature verification or calibration.

2.5.2 Frequency - Laboratory thermometers are calibrated against a National Institute of Standards and Technology (NIST) reference thermometer or NIST traceable thermometer.

2.5.2.1 Liquid in glass thermometers - calibrated before first use and annually.

2.3.2.2 Electronic thermometers - calibrated before first use and quarterly.

2.5.3 Procedure

2.5.3.1 Reference thermometers used for calibration should be the same type of thermometer as being calibrated, i.e., use a partial immersion reference thermometer to calibrate a partial immersion laboratory thermometer.

2.5.3.1.1 Hot Water Bath Thermometers

Calibrated with partial emersion reference thermometer

When calibrating thermometers for general use above room temperature, a standard water bath is heated to approximately 100°C or at the normal operational temperature of the batch. Place the (partial emersion) reference thermometer and the thermometers to be calibrated in the water batch and adjust them to their appropriate level in the fluid. Compare the values of the working thermometers against that of the reference thermometer at the temperature of interest and record three separate temperature readings.

2.5.3.1.2 Oven Thermometers

Calibrated with total emersion reference thermometer

When calibrating thermometers for use in ovens, heat an oven to approximately 104°C or 180°C. Place the (total emersion) reference thermometer and the thermometers to be calibrated in the oven thus the reference thermometer is totally immersed. Compare the values of the working thermometers against that of the reference thermometer at the temperature of interest and record three separate temperature readings.

2.5.3.1.3 Refrigerator Thermometers

Calibrated with partial emersion reference thermometer

When calibrating thermometers for general use in a refrigerator, a standard water bath is chilled with ice to approximately 4°C. Place the (partial emersion) reference thermometer and the thermometers to be calibrated in the water bath and adjust them to their

appropriate level in the fluid. A second method of calibrating this type of thermometer, would be to place them in a refrigerator at approximately 4°C and calibrate the thermometers as a group. Compare the values of the working thermometers against that of the reference thermometer at the temperature of interest and record three separate temperature readings.

2.5.3.1.4 Freezer Thermometers

Calibrated with total emersion reference thermometer

Thermometers are calibrated at approximately -15°C when calibrating thermometers for use in sample freezer. Place the (total emersion) reference thermometer and the thermometers to be calibrated in the freezer thus the reference thermometer is totally emersed. Compare the values of the working thermometers against that of the reference thermometer at the temperature of interest and record three separate temperature readings.

2.5.3.2 Complete the information on the calibration form Figure D.11-1 as follows:

a) identify each thermometer with the assigned unique identification number;

b) indicate the date of calibration;

c) temperature of normal use;

d) calculated calibration factor;

e) serial numbers of the reference thermometer and working thermometer; and,

f) analyst performing the calibration.

Thermometer Calibration Form

	Ref Thermometer	Ref Thermometer SN	Ref Thermometer Type	
	XYZ Instrument Co	123456	Partial Immersion	
Therm Style	Thermometer ID	Serial Number (SN)	Thermometer Type	Date in Service
Electronic	SAR-1B	ABCDEF	Stem	1/1/2011

Note: Electronic - calibrate quarterly at two temperatures that bracket the temperature against NIST- traceable thermometer. If only a single temp is used, calibrate at the temperature of use. Liquid in glass-annually.

	1st Quarter Cal		2nd Quarter Cal		3rd Quarter Cal		4th Quarter Cal	
Calibration Date	Date:		Date:		Date:		Date:	
	Ref Therm	Cal Therm	Ref Therm	Cal Therm	Ref Therm	Cal Therm	Ref Therm	Cal Therm
Temp #1 °C								
Temp #2 °C								
Temp #3 °C								
Average Temp								
CF=ref-therm								

Initials							
Acc of Ref Therm at temp of interest							

Comments _____

Figure D.11-1

2.5.3.3 Calibration

The calibration factor determined during thermometer calibration is defined as the difference between the working thermometer and the reference thermometer at the point of interest. It is the number of degrees that have to be added or subtracted from the measured temperature to get the true temperature.

2.5.3.3.1 Determine the Calibration Factor (CF) as follows:

CF = RT - WT

Where;

RT = Temperature of the reference thermometer

WT = Temperature of the working thermometer

2.5.3.3.2 If the calibration factor is positive, that amount is added to the measured temperature to get the true temperature.

2.5.3.3.3 If the calibration factor is negative, that amount is subtracted from the measured temperature to get the true temperature.

2.5.4 Acceptance Criteria

2.5.4.1 In order for the NIST reference thermometer to be used for the internal calibration of thermometers it should meet the following criteria:

a) the reference thermometer should have divisions of 0.1 to 0.2°C; and,

b) have listed NIST calibration points at 0°C, 100°C and 180°C (temperature of use).

2.5.4.2 If the calibration factor is greater than ±2°C that particular thermometer should be discarded and taken out of service and replaced with a newly calibrated thermometer.

Appendix D

Standard Operating Procedure

SOP D.12
IR Thermometer Procedure

1.0 IR Thermometer Standard Operating Procedure

1.1 Reference - Users manual for the Linear C-1000 IR thermometer

2.0 Applicable Matrix or Matrices

This method is applicable to the determination of temperature, using an IR gun, on essentially any type of material container.

3.0 Method Detection Limit

The IR gun has a temperature sensitivity of ± 1°C or F.

4.0 Scope and Application

Thermal energy is radiated by all objects and a portion of this thermal energy is radiated as infrared energy. An IR gun is a temperature measuring device which consists of a hand-held probe with a lens to focus the infrared energy onto a detector. Once the infrared energy is detected it is converted by a microprocessor to temperature and is displayed. The unique benefits of measuring temperature with this type of IR probe are as follows:

a) measurements are made without physical contact,
b) measurements are made rapidly,
c) heat is not removed from the measured object so the accuracy of the readings are ossured,
d) Objects that are contaminated can be easily measured.

5.0 Summary of Method

An Infrared (IR) thermometer is used to determine and record sample temperature.

6.0 Definitions

6.1 Emissivity - The ratio of energy emission of an object being measured to that of a black object at the same temperature.

6.2 IR Gun Functions

6.2.1 ARROWS - used in Sample or Emissivity mode to increase or decrease the emissivity. Depressing either key will adjust emissivity in steps of 0.01 for six steps then switch to an accelerated mode for rapid adjustment.

6.2.2 AVERAGE - used to display the average temperature of readings taken in the Sample mode.

6.2.3 °C and °F - used to change the display units

6.2.4 CONTINUAL - used to enter Continual mode for constant monitoring of the target.

6.2.5 EMMISSIVITY - used to enter Emissivity mode.

6.2.6 MAXIMUM - used to display the maximum temperature reading obtained in either the Sample or Continual mode.

6.2.7 MINIMUM - used to display the minimum temperature obtained in either the Sample or Continual mode.

6.2.8 SAMPLE - used to enter sample mode and to take an individual reading of the target.

7.0 Interferences

7.1 Temperature measurements made at a distance greater than 3 times the sample size may affect the temperature reading. Measurements can be made at virtually any distance from the bottle. However, as the distance from the bottle is increased, the diameter of the measured area increases proportionally. When the 3:1 distance to size ratio is breached and the distance is greater than 3 times the sample size, than background temperature will start interfering with the determination of the sample temperature.

7.2 Measurements must be made perpendicular to the object being measured, as non-perpendicular measurements will slightly increase the measured area and the 3:1 distance to size ratio may not be maintained and could affect the displayed temperature.

7.3 Avoid contact of the IR gun cone with warm objects for more than a few seconds, as this could cause the signal to drift several degrees.

7.4 Prolonged readings of large, hot sources are also to be avoided.

8.0 Safety

The IR gun is used to measure sample containers which may be contaminated. Sample and sample containers need to be treated with caution. See appropriate SOPs for details. Also, see Alpha's Laboratory Safety/Hazardous Communications Manual and Chemical Hygiene Plan for additional information and details.

9.0 Equipment and Supplies

9.1 Linear C-1000 Infrared Thermometer

9.2 9 volt alkaline battery

10.0 Reagents and Standards

Not applicable.

11.0 Sample Collection, Preservation, and Storage

Not applicable. See the individual methods of analysis.

12.0 Quality Control

See D.11, Standard Operating Procedure for Annual Thermometer Calibration

13.0 Calibration and Standardization

See D.11, Standard Operating Procedure for Annual Thermometer Calibration and see section 14.5 below.

14.0 Procedure

14.1 Maintenance

A 9 volt alkaline battery is used to power the IR gun. When the battery needs replacing, a "Lo" appears on the LCD display. The battery is located in the probe. To gain access to the battery, first slide the probe off of the display unit. Lightly press down on the arrow on the battery cover while sliding the battery cover in the direction of the arrow. Replace the battery. To replace the cover, simply slide it back into the probe.

14.2 Modes of operation:

a) Sample, and

b) Continual.

For our purpose the IR gun should be placed in the Sample Mode. In this mode, a sample measurement is taken each time the SAMPLE key is pressed. The measurement remains on the LED display until another sample measurement is made or another mode is selected.

A running average is calculated of all the samples taken. The average temperature can be displayed by pressing the AVERAGE key. Pressing either the MAXIMUM or MINIMUM key will display the maximum or minimum temperature measured in any mode since the unit was turned on.

DO NOT RECORD THIS AS THE SAMPLE TEMPERATURE!

14.3 Measuring Temperature

14.3.1 To measure temperature, simply hold the probe perpendicular to the object being measured at a distance not greater than three times the size of the object and press "SAMPLE." It is important that this 3:1 ratio is maintained for accurate temperature measurements.

14.4 Calculating Distance

14.4.1 To measure an 8 inch sample bottle, hold the probe perpendicular to the bottle within 24 inches of the bottle.

14.5 Emissivity Adjustment

14.5.1 An IR gun temperature measuring instrument reports temperature by adjusting the measured temperature against the emissivity of the object being measured.

Emissivity is the ratio of energy emission of an object being measured to that of a black object at the same temperature which produces an emissivity constant. This ratio is unique to the surface material being measured. Therefore, IR guns measurements are adjusted by multiplying the measured IR temperature by this emissivity ratio to correct for differences in energy emissions.

Once the emissivity function on the IR gun is adjusted, the temperature reading on the IR gun display has then been corrected for errors associated with surface emission.

14.5.2 Adjusting emissivity

14.5.2.1 If the emissivity is known, enter the EMISSIVITY mode button and adjust the emissivity ratio using the ARROW key.

14.5.2.2 If the emissivity is unknown, it can be determined as follows:

14.5.2.2.1 Place a black sticker on the object to be measured.

14.5.2.2.2 Enter the Emissivity mode and adjust the emissivity to 1.0.

14.5.2.2.3 Save the new emissivity by exiting the Emissivity mode by pressing the EMISSIVITY key.

14.5.2.2.4 Measure the temperature on the sticker by pressing SAMPLE key and note the temperature.

14.5.2.2.5 Next measure an area next to the sticker by pressing the SAMPLE key.

14.5.2.2.6 If the reading is the same as the previous one, the emissivity is calibrated for the surface.

14.5.2.2.7 If it two temperatures are not the same, keep the probe pointed at the area next to the sticker. Press the down ARROW key. (An emissivity value will briefly appear on the display and then the temperature compensated with the new emissivity will remain. Note the reading.

14.5.2.2.8 If it is not the same temperature as obtained on the sticker, press the down ARROW key until the measurements matches that of the black sticker. At this point the emissivity is calibrated for the surface.

Note: If a black sticker cannot be used, flat black paint can be used. Paint a small area on the object to be measured and follow the procedure just described.

Emissivity Table
Table D.12-1

Material	Emissivity (%)
Glass	92
Glass, frosted	96
Plastic	99
Ice	97
Water	98
Wood	85
Rubber	95
Quartz	93

14.6 Procedure

14.6.1 Turn the IR gun "ON" using the switch located on the left side of the IR gun.

14.6.2 Place the IR gun to the SAMPLE mode.

14.6.3 Enter the emissivity.

14.6.4 Point the IR gun perpendicular to the sample at a distance no greater than 3 times the size of the object being measured.

14.6.5 Press SAMPLE and note the measured temperature for recording. Take the average of three consecutive measurements when recording temperature. DO NOT USE THE AVERAGE function key for this procedure.

Note: The final temperature recorded in not corrected if the IR gun has a correction factor (this is to maintain standardization across all temperature recordings in the laboratory.).

14.6.6 Turn the IR gun "OFF" using the switch located on the left side of the IR gun.

15.0 Calculations

Not applicable.

Note: Temperature is reported in °C for most applications

16.0 Method Performance

Not applicable.

17.0 Pollution Prevention

No solvents, acids or bases are used with this procedure. The sample containers which are being measured by the IR gun may be contaminated and need to be treated with caution. See appropriate SOPs for details. Also, see Alpha's Laboratory Safety/Hazardous Communications Manual and Chemical Hygiene Plan for additional information and details.

18.0 Data Assessment and acceptance criteria for quality control measures

Not applicable.

19.0 Corrective Actions for Out-of-Control Data

Not applicable.

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

20.1 Failed Instrument

See instrument maintenance and/or use another calibrated thermometer.

21.0 Waste Management

Reference Alpha Analytical's Sample Waste SOP.

Appendix D

Standard Operating Procedure

SOP D.13
Temperature Log

1.0 TEMPERATURE LOG

1.1 All thermometers or thermistors used to monitor temperatures are calibrated against a National Institute and Technology (NIST) calibrated or traceable thermometer on an annual basis for liquid filled glass thermometers and quarterly for electronic thermometers.

2.0 STANDARD OPERATING PROCEDURE

2.1 Definition - The process of tracking, monitoring, and documenting temperature for various laboratory operations.

2.2 Purpose - Temperature readings are used to monitor the thermal preservation of samples received at the laboratory and for environmental samples stored in the laboratory. In addition, thermometers are used in a variety of other analytical techniques in the laboratory such as in the calculation of alkalinity, in studies of saturation and stability to calcium carbonate etc. Therefore, the need to track, monitor, and document temperature is important to maintaining data integrity.

2.3 Frequency

2.3.1 The sample custodian or other delegated person is responsible for monitoring and recording temperatures.

2.3.2 Temperature readings for non-analytical techniques such as refrigerators, ovens etc. should be taken at approximately the same time every day to avoid any potential cyclical temperature variations. In addition, it is recommended, to take these temperature readings, early in the morning, to avoid temperature variations, due to the opening and closing of doors.

2.3.3 This information is recorded on the Temperature Log daily, and/or as needed.

1) Clarification: DoD requires refrigerator/freezer temperature to be recorded 7 days per week.

2.4 Procedure

2.4.1 Each refrigerator/freezer and oven is assigned a calibrated thermometer. The thermometer and the device being monitored for temperature use the same identification scheme and therefore, should have the same identification.

2.4.2 If possible, this identification number is written directly on the thermometer to avoid the switching of thermometers. If the thermometer identification has been obscured, or damaged, replace the identification and verify the identification by its serial number.

2.4.3 Daily temperature readings are recorded directly from the thermometer without making adjustments to account for the thermometer correction factor.

However, if a corrected temperature were required, then the following procedure is used:

2.4.3.1.1 If the calibration factor is positive, then that amount is added to the thermometer temperature to calculate the corrected true temperature.

2.4.3.1.2 If the calibration factor is negative, then that amount is subtracted to the thermometer temperature to calculate the corrected true temperature.

2.4.4 Ovens (Spirit Filled Thermometers)

2.4.4.1 Oven thermometers are typically total immersion, spirit filled thermometers. These types of thermometers are particularly useful in this situation, because they can withstand the oven temperatures. Occasionally, the liquid spirit will separate and need to be shaken down and/or replaced.

2.4.4.2 Oven thermometers are placed in a sand-filled beaker to buffer oven temperature variations.

2.4.4.3 Placing these thermometers in a sand bath helps minimize temperature variations by increasing the thermal mass, thus mimicking more closely actual sample storage temperatures.

Secondly, it facilitates the reading of the thermometer and eliminates the temperature probe from coming into direct contact with a non-temperature equilibrated surface.

2.4.5 Refrigerators (Steel Stemmed Thermometers)

2.4.5.1 Refrigerator thermometers are total immersion, steel stemmed digital thermometers and are particularly useful for easy reading.

2.4.5.2 The refrigerator thermometer stems are placed through the septum of a 40 ml VOA vial or a 1 liter container filled with water to buffer refrigerator temperature variations.

2.4.5.3 Placing these thermometers in a 40ml VOA vial or 1-L containers helps minimize temperature variations by increasing the thermal mass, thus mimicking more closely actual sample storage temperatures.

Secondly this practice facilitates the reading of the thermometer and eliminates the temperature probe from coming into contact with a non-temperature equilibrated surface.

2.4.6 Refrigerators (Wireless Thermometers)

2.4.6.1 Electronic thermometers or thermistors configured with a wireless data logger are particularly useful for the continuous monitoring of refrigerators and freezers.

2.4.6.2 The refrigerator thermistor are placed through the septum of a 40ml VOA vial or a 1 liter amber container filled with water in order to buffer refrigerator temperature variations.

2.5 Acceptance Criteria

2.5.1 Alpha has established temperature range criteria for all ovens, refrigerators and freezers containing samples, sample extracts and standards. They are as follows:

2.5.1.1 Refrigerators - 0° to 6°C

2.5.1.2 Freezers - ≤-10°C

2.5.1.3 Drying Ovens ±5% at the temperataure of use

2.5.1.4 TDS Oven 180° ± 2°C

2.6 Corrective Action (Refrigerators, Freezers and Ovens)

2.6.1 The person taking temperature reading can make minor refrigerator/oven adjustments to the temperature control in order to compensate seasonal fluctuations, sample mass to be cooled or heated, or any other contributing factor.

2.6.2 All adjustments are recorded in the comments section of the Temperature Log.

2.6.3 The Laboratory Director or Laboratory Manager must be informed of refrigerators or ovens with temperatures out side of their designated range, wildly fluctuating temperatures or abnormalities which might affect the sample integrity. All corrective actions and maintenance will be recorded on its log record.

2.6.4 Procedure for Cold Sample Storage and Oven Temperature Excursions

If the refrigerator/freezer or oven temperature continuously drifts outside the prescribed range of acceptability the following steps should be taken:

2.6.4.1 Notify the Laboratory Director and/or the QA Officer immediately.

2.6.4.2 Monitor the refrigerator or oven for a minimum of three consecutive days with a thermograph or wireless thermistor to establish a real-time temperature baseline.

Note: Temperature excursions may be an isolated occurrence, therefore constant monitoring is required to document a more precise determination of a problem.

2.6.4.3 If the temperature excursions persist for three consecutive days then the samples or extracts located in that refrigerator or oven should be moved to a back-up and the out-of-control unit repaired or replaced.

2.6.4.4 The three-day period may be circumvented if it is deemed necessary.

Appendix D

Standard Operating Procedure

SOP D.14
Refrigerator Document Control

1.0 REFRIGERATOR DOCUMENT CONTROL

1.1 Refrigerator logs and the maintenance of these historical records is an important part in the overall continuity of sample integrity. Records are kept and maintained to ensure there is no breach in sample integrity or the process of documenting this.

2.0 STANDARD OPERATING PROCEDURE

2.1 Each refrigerator is assigned a one-time unique identification number to distinguish it from any other refrigerator in use by the laboratory. This identification number has the added feature in identifying refrigerators temporally.

2.2 Refrigerators/freezers are labeled with an identification number in the following format:

XXXNNZ

where;

XXX represents a 3-letter prefix unique to each type of refrigerator/freezer

such as;

SAR refers to sample refrigerator
SAF refers to sample freezer
STR refers to a standards refrigerator
STF refers to a standards freezer
EXT refers to an extraction refrigerator/freezer

and;

NN represents a two-digit number identifying the refrigerator/freezer from another refrigerator/freezer with the same prefix. The following sets of numbers (NN) are used:

1-19	refers to sample refrigerators/freezers
20-40	refers to standard refrigerators/freezers
31-50	refers to extract refrigerators/freezers

and:

Z represents whether the unit is the original unit or a replacement unit.

For example:

If the VOC refrigerator, ID SAR-1A, happened to be replaced for whatever reason the ID for the new refrigerator would be SAR-1B. By using this scheme, refrigerators

which are replaced can be tracked by time. This is particularly useful when tracking samples throughout the lab. Samples are placed in a particular refrigerator and therefore the confusion of which refrigerator and how it was operating during a particular time period can easily be traced.

2.3 Refrigerator adjustments and replacements are all annotated on the specific temperature log and are kept as historical record. Tables D.14-1-through 3 lists the refrigerators/freezers, their particular identification and the types of samples or standards which are stored in them.

Sample Refrigerator/Freezer Identification Table
Table D.14-1

Refrigerator Name	Use
SAR-1B	VOC water samples post analysis
SAR-2C	Contaminated VOC/TPH samples (in paint cans)
SAR-3A	8270/8081/8082 and general SV samples prior to extraction
SAR-4B	TPH/TOC water samples post analysis and non-extracted waters
SAR-5D	Laboratory prepared VOC trip and field blanks
SAF-6B	Air samples and methanolic air extracats
SAR-7A	TPH-E/TOC samples prior to analysis
SAR-8A	VOC water samples prior to screening and prepared sequences awaiting analysis
SAR-9B	Inorganic samples post analysis
SAR-10A	524.2 sample prior to analysis and storage after analysis
SAR-11A	TPH/VOC soil samples prior to analysis and storage after analysis
SAR-12A	VOC water samples after screening and prior to analysis and Alcohol waters
SAR-13A	Inorganic samples
SAR-14A	Department of Defense (DOD) samples
SAR-15B	Incoming sample (Sample Receipt)
SAR-16A	Dissolved Gases
SAR-17A	Alcohols not completed
SAR-18A	Incoming Sample Overflow
SAR-19B	Organic Acids

Standard Refrigerator/Freezer Identification Table
Table D.14-2

Refrigerator Name	Use
STR-20A	TPH exractable standards
STF-21B	VOC methanolic standards
STR-22A	625/8270 standards
STR-23B	Metals/Inorganic standards
STR-24C	VOC standards
STR-25B	Anions standards
STR-27C	Alcohols standards
STR-28A	ECD standards
STF-29C	524.2 standards
STR-30A	TPH/TOC standards
STR-32A	Wet Chem standards

Extract Refrigerator/Freezer Identification Table
Table D14-3

Refrigerator Name	Use
EXT-33C	ECD extracts
EXT-36B	MeOH VOC soil extracts
EXT-37A	TPH extracts
EXT-38A	SV extracts requiring internal C-O-C procedures (DoD extracts)

Appendix D

Standard Operating Procedure

SOP D.15
Standard Operating Procedure for the Calibration Verification of Mechanical Volumetric Dispensing Devices (MVDD) and Volumetric Labware

Standard Operating Procedure for the Calibration Verification of Mechanical Volumetric Dispensing Devices (MVDD) and Volumetric Labware

1.0 Identification of the Test Method

1.1 Confidential Business Information (CBI)

The information contained in this document is confidential, and any specification or infringement of proprietary rights is strictly forbidden. This document is not to be distributed, copied, disseminated, or used without written authorization of Alpha Analytical, Inc.

1.2 References

1.2.1 Method E542-01: Standard Practice for Calibration of Laboratory Volumetric Apparatus (Re-approved 2007); American Standard Test Methods (ASTM); ASTM Committee E.41, approved November 1, 2007.

1.2.2 Method E969-02: Standard Specification for Glass Volumetric (Transfer Pipets (Re-approved 2007); American Standard Test Methods (ASTM); ASTM Committee E.41, approved November 1, 2007.

Note: Both Methods E542 and E969 have been approved for use by agencies of the Department of Defense (DoD).

1.2.3 ASTM E694-99, Standard Specification for Laboratory Glass Volumetric Apparatus, American Society for Testing and Materials, 2010.

2.0 Applicable Matrix

2.1 NA

3.0 Detection Limit

3.1 This procedure is intended to encompass labware with a capacity between 0.1mL and 2000 mL in volume. Typical products falling within the purview of this practice are burets, graduated cylinders, volumetric flasks, measuring and dilution pipets, and transfer and capacity pipets.

3.2 This procedure is not recommended for calibration of apparatus with capacities below 0.1 mL, such as microsyringes and small micro-pipetor tips.

4.0 Scope and Application

4.1 Volumetric glassware/labware is used to measure liquids with accuracy ranging from

moderate to a very high level of accuracy. Many standard beakers and flasks have graduations accurate to ± 5%. This level of accuracy is sufficient for many routine tasks in the laboratory. However, often there is a need to measure liquids much more precisely. Volumetric labware allows the measurement of liquids to less than 0.1% depending on its type, design and quality.

4.2 Analytical results are dependent on their accuracy, as in standard preparation, and dispensing of liquids or dilution into a specified volume making these types of support-equipment necessary in the determination of quantitative analysis.

4.3 The primary purpose of this procedure is to provide uniform practices that may be used to accurately calibrate and verify calibration of a wide variety of volumetric ware. The techniques are simple in concept and can provide reliable results, provided the procedures are throughly followed. Accordingly, this practice should provide a means of checking the original calibration of glassware and similar apparatus and for periodic rechecks as the need should arise.

4.4 The following procedure defines volumetric and non-volumetric labware and establishes accuracy and precision criteria that must be met in support of analytical testing and measurement procedures used throughout the laboratory.

5.0 Summary of the Test Method

5.1 The practice of labware calibration and calibration verification is based upon a determination of the volume of liquid either contained in or delivered by the dispensing device.

5.2 By using water as the determinant liquid, liquid volume may be calculated based on the gravimetric determination of the quantity of water either contained or delivered, and the conversion of this value to true volume at the standard temperature of 20°C by means of suitable equations and standardized tables.

5.3 Mechanical volumetric dispensing devices such as solvent/acid dispensers and burettes are checked for accuracy on a quarterly basis. Automatic pipetors are checked daily, and glass microliter syringes are considered in the same manner as Class A glassware and are checked for accuracy upon evidence of deterioration.

6.0 Definitions

6.1 Capacity - The basic International Standard (SI) unit for volume is the cubic meter.

Note: Due to its large size, it is rarely used in volumetric calibration. Rater, the cubic centimeter, cm^3, is used and will be employed in this practice. The unit, milliliter (ml) is considered as equivalent to the cubic centimeter.

6.2 Class A Volumetric Glassware - Glassware which provides the highest accuracy.

Note: Class A glassware complies with the Class A tolerances defined in ASTM E694, (±1% volume error up to 50 ml and ±0.5% error for glassware greater than 50 ml). Class A glassware must be permanently labeled as Class A, and is supplied with a serialized certificate of precision.

Note: Many manufacturers supply "generic" Class A vessels which meet Class A tolerances but are not technically Class A because they are not serialized or certified. All Class A volumetric glassware is actually glass; volumetric plasticware is not eligible for Class A status.

6.3 Class B Volumetric Glassware - Glassware which has tolerances twice those of Class A (except graduated cylinders, which have rules of their own).

Note: Class B volumetric glassware must comply within the Class B tolerances defined in ASTM E694 and must be permanently labeled as Class B.

6.4 Mechanical Volumetric Dispensing Devices (MVDD) are devices that dispense liquids with a high degree of accuracy using a pre-selected dispensing volume.

Note: These devices include both mechanical hand pumps and electronic computerized pumps.

6.5 Meniscus - The curve in the upper surface of a standing body of liquid, produce in response to the surface of the container or another object. A liquid meniscus can either be convex or concave. A convex meniscus occurs when the molecules have a stronger attraction to each other. Conversely, a concave meniscus occurs when the molecules of the liquid attract those of the container wall by capillary action.

6.6 Non-volumetric Labware - Glass or plastic labware which were not designed, manufactured or labeled as Class A or B glassware and carry no accuracy tolerance limits. This type of labware, is typically thought of as labware used by the laboratory by the process of calibrating a sample container or other piece of labware. Non-volumetric labware is typically used for measuring the initial sample volume or the final extract/digestate volume.

6.7 Standard Temperature and Pressure (STP) - In chemistry, standard conditions for temperature and pressure are standard sets of conditions for experimental measurements, to allow comparisons to be made between different sets of data. This is most typically defined as a temperature of 0°C (32°F) and a pressure of 1 atmosphere (760 mm Hg). However, many scientific organizations, use other stated conditions for STP. Volumetric ware is almost universally calibrated at 20°C. The procedures described below provide for such a calibration.

6.8 To Contain (TC) - A vessel marked TC contains the amount specified when it is filled to the graduation line.

6.9 To Deliver (TD) - A vessel marked TD delivers the amount when it is filled to the graduation line and emptied using the proper procedure.

Note: The difference between TC and TD arises because of drainage hold-back error. For example, volumetric flasks are rated TC; if you fill a 500 ml volumetric flask to the graduation line, it contains exactly 500 ml of solution (within its tolerance). If you empty that flask into another container, a bit less than 500 ml will transfer. That's because some of the solution remains in the flask, wetting the inner surface.

7.0 Interferences

7.1 Class A Glassware

Class A borosilicate volumetric glassware will hold its calibration indefinitely provided that it is not exposed to hydrofluoric acid, hot phosphoric acid, or strong, hot alkalis and that it is not heated above 150°C when dry. A frosting of the glass surface (viewed when dry) indicates that chemical attack has occurred, and re-calibration may be in order.

8.0 Safety

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible extent. A reference file of material safety data sheets are available. See Alpha's Laboratory Safety/Hazardous Communications Manual and Chemical Hygiene Plan for additional information and details.

9.0 Equipment and Supplies

9.1 Balance - Typically a four place balance (e.g., 0.1g which is approximately 0.1mL). The sensitivity of the balance is the limiting factor in the accuracy of the measurements.

9.2 Thermometer - For measuring the temperature of the water. The accuracy of the calibration will depend upon the accuracy requirement of the volumetric calibration. An accuracy of ± 0.5°C is required to achieve the specified tolerences.

9.3 Barometer - Capable of providing atmospheric pressure measurements of ± 20 mm Hg. Alternatively, the barometric pressure may be obtained from the local weather service.

10.0 Reagents and Standards

10.1 Reagent Water - Deionized water 10.0 *uS*/cm or better. Water should contain particles no larger than 0.20 µm.

11.0 Sample Collection, Preservation, Shipment and Storage

11.1 NA

12.0 Quality Control

12.1 Reading and Setting of a Liquid Meniscus

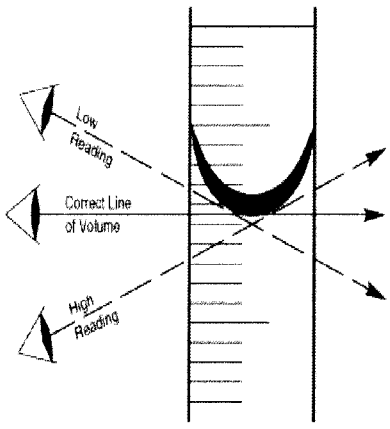
12.1.1 Reading of the Meniscus

A characteristic of liquids in glass containers is that they curve at the edges. This meniscus makes the determination of volume somewhat difficult. With water in glass, the meniscus will curve up (concave) at the edges and down in the center, so we would typically say to read the bottom of the meniscus.

For all volumetric labware calibrated by this procedure, the reading is made on the lowest point of the meniscus. In order that the lowest point may be observed, it may be necessary to place a shade of some dark material immediately below and/or behind the meniscus, which renders the profile of the meniscus dark and clearly visible against a light background.

12.1.2 Setting the Meniscus

The position of the lowest point of the meniscus with reference to the graduation line is horizontally tangent to the plane of the upper edge of the graduation line. The position of the meniscus is obtained by having the eye in the same plane of the upper edge of the graduation line.



13.0 Calibration and Standardization

13.1 Calibration Procedure for Class A Glassware (To Deliver)

13.1.1 Definition - Determination of Class A volumetric glassware volume by using a gravimetric procedure corrected for temperature and barometric pressure.

13.1.2 Purpose - To determine if a device which measures or dispenses a liquid is performing that operation to within a predetermined set of tolerance limits.

13.1.3 Frequency

13.1.3.1 Class A borosilicate volumetric glassware will hold its calibration indefinitely provided that it is not exposed to hydrofluoric acid, hot phosphoric acid, or strong, hot alkalis and that it is not heated above 150°C when dry. A frosting of the glass surface (viewed when dry) indicates that chemical attack has occurred, and recalibration may be in order.

13.1.3.2 If there is any evidence of deterioration, verify calibration.

13.1.4 Procedure

13.1.4.1 Weight Determination

13.1.4.1.1 Do not dry the vessel that is being calibrated TO DELIVER (TD) prior to the test.

13.1.4.1.2 Weigh an empty receiving flask and record the weight or simply tare the receiving flask.

13.1.4.1.3 Fill the vessel being tested to the capacity test line with water.

13.1.4.1.4 Rapidly empty the test vessel by gradually inclining the vessel as to avoid splashing on the walls of the empty receiving flask as much as possible.

13.1.4.1.5 When the main drainage stream has ceased, the flask will be nearly vertical. Hold this position for 30 s and touch off the drop of water adhering to the top of the test vessel.

13.1.4.1.6 Determine the final weight of the receiving flask

immediately to ensure no evaporation has taken place. Calculate the difference between the original empty weight and final fill weight and record.

13.1.4.1.7 Determine the water temperature by placing a thermometer in the filling beaker and record.

13.1.4.1.8 Take 10 replicate measurements at the determined volume of use.

13.1.5 Acceptance Criteria - From those 10 replicate measurements, calculate the average, standard deviation and Relative Standard Deviation (RSD).

13.1.5.1 The average must be within ± 2% of the volume measured.

13.1.5.2 The RSD must be ≤1% of the 10 replicates.

13.1.6 Corrective Action - Class A glassware which does not meet the minimum RSD criteria, can not be used and must be taken out of service.

13.2 Calibration Procedure for Class A Glassware (To Contain)

13.2.1 Definition - Same as section 13.1

13.2.2 Purpose - Same as section 13.1

13.2.3 Frequency - Same as section 13.1

13.2.4 Procedure

13.2.4.1 Weight Determination

13.2.4.1.1 After cleaning **and drying**, weigh the empty vessel. Fill the vessel with water just below the capacity line to be measured. When filling take care and avoid wetting the vessel above the capacity line.

13.2.4.1.2 Complete the filling by watching the meniscus approach the capacity line. A pipet or dropper with a finely drawn tip may be required to adjust the meniscus.

13.2.4.1.3 Determine the water temperature by placing a thermometer in the filling beaker.

13.2.4.1.4 Determine the final weight. Calculate the difference between the original empty weight and final fill weight and record.

13.2.4.1.5 Take 10 replicate measurements at the determined volume of use.

13.2.5 Acceptance Criteria - Same as section 13.1

13.2.6 Corrective Action - Same as section 13.1

13.3 Calibration Procedure for Class B Glassware (To Contain and/or To Deliver)

13.3.1 Definition - Same as section 13.1

13.3.2 Purpose - Same as section 13.1

13.3.3 Frequency - Same as section 13.1

13.3.4 Procedure - Same as section 13.1

13.3.5 Acceptance Criteria - Technically, Class B glassware has tolerances twice those of Class A. However, Class A tolerance limits will be observed. From those 10 replicate measurements, calculate the average, standard deviation and Relative Standard Deviation (RSD).

13.3.5.1 The average must be within ± 2% of the volume measured.

13.3.5.2 The RSD must be ≤1% of the 10 replicates.

13.3.6 Corrective Action - Same as section 13.1

13.4 Calibration Procedure for Non-Volumetric Glassware (To Contain)

13.4.1 Definition - Determination of non-volumetric labware volume using a volumetric procedure by dispensing from a Class A volumetric glassware.

13.4.2 Purpose - Non-volumetric labware may be used for measuring initial sample volume or final extract/digestate volume by comparison.

13.4.3 Frequency - These are monitored by lot, before first use or upon evidence of deterioration.

13.4.4 Procedure - By using Class A volumetric glassware, a volumetric procedure is used for establishing a predetermined TO CONTAIN volume.

13.4.4.1 Volumetric Determination

13.4.4.1.1 Using Class A glassware, determine the volume of liquid needed and fill the Class A glassware to the test line.

13.4.4.1.2 Decant this liquid into the non-volumetric labware and place a volume capacity line on the container.

Note: This container may have multiple volume capacity lines if using this to determine initial sample volume and or extract/digestate volumes.

13.4.4.1.3 Take 10 replicate measurements at the determined volume of use.

13.4.5 Acceptance Criteria - From those 10 replicate measurements, calculate the average, standard deviation and Relative Standard Deviation (RSD).

13.4.5.1 The average must be within ± 3% of the volume measured.

13.4.5.2 The RSD must be ≤3% of the 10 replicates.

13.4.6 Corrective Action - Non-volumetric glassware which does not meet the average and/or the minimum RSD criteria, can not be used and must be taken out of service.

13.5 Calibration Procedure for Burets (Same as Class A Glassware - To Deliver)

13.5.1 Definition - Same as section 13.1

13.5.2 Purpose - Same as section 13.1

13.5.3 Frequency - Quarterly

13.5.4 Procedure - By using a Class A cylinder, either the volume or weight can be used to verify calibration of the buret.

13.5.4.1 Set Up

13.5.4.1.1 Clamp the buret vertically on a support stand. Place a beaker of fill water next to the buret stand large enough to hold a thermometer.

13.5.4.1.2 Fill the buret from a storage bottle, in which the water has reached equilibrium with room temperature and check to verify that there is neither leakage from the tip nor from the stockcock plug.

13.5.4.1.3 Fill the burett approximately 10 mm above the zero mark with water and fill the beaker that holds the thermometer; record the temperature.

13.5.4.1.4 Set the meniscus on the zero mark using the buret stopcock to lower the liquid level and remove any excess water from the burett tip with a kimwipe.

13.5.4.1.5 Use a Class A volumetric cylinder as the receiving flask and place the volumetric cylinder in such a way as to make contact with the tip of the buret (the volumetric cylinder will be at a slight angle) so that the water will slide down the side of the cylinder and prevent any loss due to spattering.

13.5.4.1.6 Fully open the stopcock until the water is only a few millimeters above the line being tested and then the stream is slowed as to make an accurate setting. When the setting or volume has been reached, move the volumetric cylinder horizontally, breaking the contact with the buret and recheck the volume setting on the buret.

13.5.4.2 Volumetric Determination

13.5.4.2.1 Dispense the desired volume of liquid from the buret into a Class A glassware vessel and record the volume. Take 10 replicate measurements at the test volume.

13.5.4.3 Weight Determination

13.5.4.3.1 Determine the empty weight of the Class A receiving vessel and record.

13.5.4.3.2 Record the temperature in the test tube next to the buret if weight is to be used.

13.5.4.3.3 Dispense the desired volume of liquid from the buret into a Class A glassware vessel.

13.5.4.3.4 Weigh the Class A volumetric cylinder a second time, and record the weight.

13.5.4.3.5 Take 10 replicate measurements at the test volume.

13.5.5 Acceptance Criteria - From those 10 replicate measurements, calculate the average, standard deviation and Relative Standard Deviation (RSD).

13.4.5.1 The average must be within ± 2% of the volume measured.

13.4.5.2 The RSD must be ≤1% of the 10 replicates.

13.5.6 Corrective Action - Burets which do not meet the average and/or the minimum RSD criteria, can not be used and must be taken out of service.

13.6 Calibration Procedure for Mechanical Volumetric Dispensing Devices (Solvent/acid Dispensers Same as Class A Glassware - To Deliver)

13.6.1 Definition - Same as section 13.1

13.6.2 Purpose - Same as section 13.1

13.6.3 Frequency - Quarterly

13.6.4 Procedure - By using a Class A cylinder, either the volume or weight can be used to verify calibration of the MVDD

13.6.4.1 Volumetric Determination

13..6.4.1.1 Dispense the desired test volume of liquid from the MVDD into a Class A glassware vessel and record the volume.

13.6.4.1.2 Take 10 replicate measurements at the test volume.

13.6.4.2 Weight Determination

13.6.4.2.1 Use a Class A volumetric cylinder as the weighing flask and determine the empty weight and record.

Note: It may be helpful to place the volumetric cylinder to make contact with the tip of the MVDD (the volumetric cylinder will be at a slight angle) so that the liquid will slide down

the side of the cylinder and prevent any loss due to spattering.

13.6.4.2.2 If the liquid being dispensed from the MVDD is anything other than water, than the gravimetric procedure to determine volume is not used.

If the liquid being dispensed from the MVDD is water, than the volume determination using gravimetry is corrected for temperature or barometric pressure.

13.6.4.2.3 Dispense the desired volume of liquid from the MVDD into a Class A glassware vessel

13.6.4.2.4 Weigh the Class A volumetric cylinder a second time, and record the weight.

13.6.4.2.5 Take 10 replicate measurements at the test volume.

13.6.5 Acceptance Criteria - From those 10 replicate measurements, calculate the average, standard deviation and Relative Standard Deviation (RSD).

13.6.5.1 The average must be within ± 2% of the volume measured.

13.6.5.2 The RSD must be ≤ 1% of the 10 replicates.

13.6.6 Corrective Action - Devices which do not meet the average and/or the minimum RSD criteria, can not be used and must be taken out of service.

13.7 Calibration Procedure for Automatic Pipetors

13.7.1 Definition - Same as section 13.1

13.7.2 Purpose - Same as section 13.1

13.7.3 Frequency - Quarterly

13.7.4 Procedure

13.7.4.1 Weight Determination

13.7.4.1.1 Use a beaker or other collection flask as the weighing flask and determine the empty weight and record or simply tare the collection flask.

13.7.4.1.2 Place a beaker of water next to the collection flask and let it come to room temperature. Record the temperature in the beaker next to the collection flask.

13.7.4.1.3 Dispense the desired volume of liquid from the pipetor into the collection flask.

13.7.4.1.4 Determine the dispensed weight and record the weight.

13.7.4.1.5 Take 10 replicate measurements at the test volume.

13.7.5 Acceptance Criteria - From those 10 replicate measurements, calculate the average, standard deviation and Relative Standard Deviation (RSD).

13.7.5.1 The average must be within ± 2% of the volume measured.

13.7.5.2 The RSD must be ≤ 1% of the 10 replicates.

13.7.6 Corrective Action - Automatic pipetors which do not meet the acceptance criteria can not be used and must be taken out of service.

14.0 Procedure

14.1 Weighing Procedures

14.1.1 Two weighings are required, namely V_L referring to the loaded vessel and V_E referring to the empty vessel.

14.1.2 Complete both of the required weighings in as short a time interval as possible to assure that they have been weighed under similar conditions.

14.1.3 Weighings should be made with care and made expeditiously to minimize evaporation losses which would constitute a source of error.

14.1.4 The balance should be checked with S class weights prior to using and in prime working condition.

14.1.5 The vessels that are weighed should be clean externally, and handled carefully to avoid contamination.

14.1.6 Vessels may be wiped with a clean cloth as required.

14.2 Temperature and Barometric Pressure Determinations

14.2.1 If the liquid being dispensed from the MVDD is anything other than water, than the gravimetric procedure to determine volume is not used.

If the liquid being dispensed from the MVDD is water, than the volume determination using gravimetry is corrected for temperature or barometric pressure.

14.2.2 Temperature Corrections

14.2.2.1 Determine temperature preferably as °C (celsius).

14.2.3 Barometric Pressure Corrections

14.2.3.1 Determine barometric pressure preferably as mm Hg.

15.0 Data Analysis and Calculations

15.1 Volume Determination using Gravimetry

15.1.1 Calculate the volume from the weight of the water, contained or delivered (at standard temperature of 20°C) as follows:

$$V_{20} = (V_L - V_E)(Q)(1/P_W - P_A)(1 - P_A/P_W)[1 - \alpha(t-20)]$$

Where:

$V_L - V_E$ = the difference, in grams obtained by subtracting the balance indication in grams associated with the empty weighing flask from that associated with the loaded flask,

Q = the apparent mass conversion factor that differs from unity for different types of balances, depending upon the actual density of the weights and the apparent mass scale to which they have been adjusted by the manufacturer. The factor has a maximum value of 1.000013, hence may be considered as unity for volumetric calibrations,

$1/P_W - P_A$ and $1 - P_A/P_W$ = two density terms which require knowledge of air density, P_A, water density, P_W, and the density of the balance weights, P_B (density of balance weights taken as 7.78 g/cm^3), and

$1-\alpha(t-20)$ = the thermal expansion factor used to convert the volume from the temperature of measurement in degrees Celsius, to the standard temperature of 20°C. The symbol α represents the coefficient of cubical expansion of the vessel.

15.1.2 The equation $(Q)(1/P_W-P_A)(1-P_A/P_W)[1-\alpha(t-20)]$ can be substituted by an appropriate Z factor. Therefore, by multiplying the observed mass of water (V_L-V_E) by the Z factor, the volume of the glassware at the standard temperature of 20°C may be obtained.

Where:

$$V_{20} = (V_L-V_E)(Q)(1/P_W-P_A)(1-P_A/P_W)[1-\alpha(t-20)]$$

And:

$$Z = (Q)(1/P_W-P_A)(1-P_A/P_W)[1-\alpha(t-20)]$$

So:

$$V_{20} = (V_L-V_E)Z$$

Z Values as a Function of Temperature and Pressure for Use in Calibration of Type I, Class A, Borosilicate Glass
Table D.16-1

Barometric Pressure	Temperature, °C								
mm Hg	16	17	18	19	20	21	22	23	24
600	1.00196	1.00211	1.00228	1.00245	1.00264	1.00283	1.00304	1.00326	1.00349
620	1.00199	1.00214	1.00231	1.00248	1.00267	1.00287	1.00308	1.00330	1.00353
640	1.00201	1.00216	1.00233	1.00251	1.00270	1.00290	1.00311	1.00333	1.00356
660	1.00204	1.00219	1.00236	1.00254	1.00272	1.00292	1.00313	1.00335	1.00358
680	1.00207	1.00222	1.00239	1.00256	1.00275	1.00295	1.00316	1.00338	1.00361
700	1.00209	1.00225	1.00242	1.00259	1.00278	1.00298	1.00319	1.00341	1.00364

Z Values as a Function of Temperature and Pressure for Use in Calibration of Type I, Class B, Borosilicate Glass
Table D.16-2

Barometric Pressure	Temperature, °C								
mm Hg	16	17	18	19	20	21	22	23	24
600	1.00198	1.00212	1.00229	1.00246	1.00265	1.00284	1.00304	1.00326	1.00348
620	1.00201	1.00215	1.00232	1.00249	1.00267	1.00287	1.00307	1.00328	1.00351
640	1.00203	1.00218	1.00234	1.00251	1.00270	1.00289	1.00310	1.00331	1.00354
660	1.00206	1.00221	1.00237	1.00254	1.00272	1.00292	1.00312	1.00334	1.00357
680	1.00209	1.00224	1.00240	1.00257	1.00275	1.00295	1.00316	1.00337	1.00359
700	1.00211	1.00226	1.00243	1.00259	1.00278	1.00298	1.00318	1.00340	1.00362

Z Values as a Function of Temperature and Pressure for Use in Calibration of Autop-pipetors (Eppendorf type pipetors) with plastic pipet tips
Table D.16-3

Barometric Pressure	Temperature, °C								
mm Hg	16	17	18	19	20	21	22	23	24
600	1.0019	1.0021	1.0022	1.0024	1.0026	1.0028	1.0031	1.0033	1.0035
638	1.0020	1.0021	1.0023	1.0025	1.0027	1.0029	1.0031	1.0033	1.0036
675	1.0020	1.0022	1.0023	1.0025	1.0027	1.0029	1.0032	1.0034	1.0036
713	1.0021	1.0022	1.0024	1.0026	1.0028	1.0030	1.0032	1.0034	1.0037
750	1.0021	1.0023	1.0025	1.0026	1.0028	1.0031	1.0033	1.0035	1.0037

15.1.3 Table 1, 2 and Table 3 are provided to facilitate the calculation of the volume from the observed weighings, when using water, for the weighing conditions and practices commonly used.

15.1.3.1 Temperature

The temperature of at the time of measurement is also assumed to be the temperature of the laboratory air. Fahrenheit to Celsius conversion is as follows:

°F = (9/5 * °C) + 32 or

°C = (°F -32)* 5/9

The range of air temperature most commonly found in the laboratory is from 68°F - 76°F which corresponds to 20°C - 24.4°C with an average of 72°F or 22.2°C.

13.1.3.2 Barometric Pressure

Reno sits at an elevation of approximately 4500 feet above sea level. The range of barometric pressures most commonly reported at the Reno Nevada airport ranges from 25.1 to 25.9 inches of Hg which corresponds to a range of 638 to 658 mm Hg.

15.1.4 A single nominal correction value of 1.00300 may be used because the variation of typical temperature and pressure will not introduce a significant error.

16.0 Method Performance

16.1 A competent operator should be able to repeat volumetric calibrations within the limits indicated in Table D.16-4

Precision Data
Table D.16-4

Vessel	Nominal Size, mL	Reproducibility, mL
Pipet	1	0.002
	2	0.002
	5	0.002
	10	0.003
	25	0.005
	50	0.007
	100	0.0010
Flask	10	0.005
	50	0.007
	100	0.011
	250	0.017
	500	0.021
	1000	0.042
Buret	10	0.003
	50	0.007
	100	0.012

16.2 The largest source of experimental error associated with this measurement is in the adjustment of the meniscus, which will depend on operator care and is related to the cross section of the vessel where the meniscus is located.

17.0 Pollution Prevention

17.1 No solvents, acids or other chemicals are used in this method. Nothing in this procedure pose a threat to the environment.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 Class A Glassware

18.1.1 From those 10 replicate measurements the average must be within ± 2% of the volume measured and the RSD must be ≤ 1%.

Note: This acceptance criteria is the same for burets, Class B glassware, automatic/Eppendorf pipetors and mechanical volumetric dispensing devices

18.2 Non-Volumetric Glassware

18.2.1 From those 10 replicate measurements the average must be within ± 3% of the volume measured and the RSD must be ≤ 3%.

19.0 Corrective Actions for Out-of-Control Data

19.1 Failed Instrument Parameters - Repeat the test. If repeat failure occurs, locate and correct the source of the problem and repeat the test.

19.2 Failed QC Parameters - Repeat the test. If repeat failure occurs, locate and correct the source of the problem.

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

20.1 Failed Instrument Parameters - If upon re-analysis the corrective actions fail to solve the problem, then the following shall be performed:

20.1.1 Devices and/or glassware which do not meet the acceptance criteria can not be used and must be taken out of service.

21.0 Waste Management

21.1 Reference Alpha Analytical's Sample Waste SOP.

Appendix E

Analytical and Extraction Support Procedures

Appendix E

Standard Operating Procedure

SOP E.1
Analytical and Extraction Support Procedures

1.0 ANALYTICAL AND EXTRACTION SUPPORT PROCEDURES

1.1 The QA manual and associated SOPs have been written to provide the basis for QA and QC in both field sampling and laboratory analysis. Data generated by Alpha could potentially be used to support litigation; therefore, documentation of laboratory procedures is essential to maintain a defensible audit trail for the generation of sample results. One part of this trail of documentation is the need and a compilation of Standard Operating Procedures to assure the various activities were performed and documented with uniformity and consistency.

Not all activities have written SOPs, however, procedures which are performed on a routine basis that may have a significant impact regarding the Data Quality Objectives have SOPs.

Appendix E

Standard Operating Procedure

SOP E.2
Dishwasher Steam Scrubber Operation

1.0 DISHWASHER AND STEAM SCRUBBER OPERATION

1.1 To prevent cross-contamination or carry-over, all organic extraction glassware is thoroughly washed in a Labconco Dishwasher steam scrubber.

2.0 Standard Operating Procedure:

2.1 As soon as possible, glassware that has come in contact with samples or standards should be rinsed with methylene chloride or the solvent last used in the glassware.

2.2 Soak the glassware in hot water with a non phosphate detergent such as Extran-300, to loosen and float particles.

2.3 Manually scrub the glassware with a pad or brush.

2.4 Hot water rinse the glassware to flush away any particles.

2.5 Recommended dishwasher settings:

2.5.1 Steam action on;

2.5.2 Power dry heat;

2.5.3 Fill detergent container with appropriate amount of detergent,

2.5.4 Close the detergent container and fill the extra containers as needed,

2.5.5 Set to appropriate cycle: i.e. plastic, glass 1, glass 2, rinse, etc.,

2.5.6 Push start switch to begin cycle; and,

2.5.7 After the dishwasher stops, inspect and separate glassware which visibly does not look clean and repeat the procedure again.

2.6 Once the glassware has been cleaned and visually inspected it is oven dried at 104 °C for a minium of 4 hours.

Appendix E

Standard Operating Procedure

SOP E.3
Manual Glassware Cleaning Procedure

1.0 MANUAL GLASSWARE CLEANING PROCEDURE

1.1 Certain glassware is incompatible with cleaning in the dishwasher steam scrubber; therefore, the following procedure will be followed.

2.0 Standard Operating Procedure

2.1 Remove surface residuals immediately after use. As soon as possible, glassware that has come in contact with organic sample and standards should be rinsed with methylene chloride or the same solvent used prior to washing; for inorganic analysis, soak in deionized water.

2.2 Soak the glassware in hot water with a non phosphate detergent such as Extran-300, to loosen and float particles.

2.3 Manually scrub the glassware with a pad or brush.

2.4 Hot water rinse the glassware to flush away any particles.

2.5 After washing, inspect and separate glassware which visibly does not look clean and repeat the procedure again.

2.6 Once the glassware has been cleaned and visually inspected, glassware for organic analysis is oven dried at 104 °C for a minium of 4 hours. Glassware for inorganic analysis does not need to be oven dried with the exception of TKN and ammonia glassware.

Appendix E

Standard Operating Procedure

SOP E.4
Sample Container Cleaning Procedures

1.0 SAMPLE CONTAINER CLEANING PROCEDURES

1.1 Cleaning procedures are practiced to minimize contamination from the containers in which they are stored.

2.0 Standard Operating Procedure

2.1 Policy

It is Alpha's policy not to reuse any sampling bottles or containers used in the collection of environmental field samples. All containers purchased by Alpha are purchased from manufacturers which follow the minimum EPA Level I Protocol prescribed cleaning protocols as outlined below.

2.2 Sample Bottle Material

2.2.1 If the analysis to be determined are organic in nature, the container should be made of borosilicate glass.

2.2.2 If the analytes are inorganic, the container should be polyethylene.

2.2.3 When both organic and inorganic substances are expected to be present, separate sample aliquots should be taken.

2.3 Cleaning Protocols

Commercially cleaned containers are used if cleaning procedures comply with EPA procedures as outlined below.

2.3.1 The procedures for cleaning glass and polyethylene containers and their caps for **EPA level I** protocol are as follows:

2.3.1.1 **Cleaning Procedure A Extractable Organic Compounds** (Glass wide mouth jars and amber Boston Rounds)

a) Scrub and wash bottles, teflon liner, and caps in laboratory grade, non-phosphate detergent;

b) Rinse three times with tap water;

c) Rinse with 1:1 nitric acid;

d) Rinse three times with ASTM type 1 organic free water;

e) Rinse with pesticide grade methylene chloride; and

f) Oven dry at 125° C, allow to cool to room temperature in an enclosed contaminant-free environment.

2.3.1.2 **Cleaning Procedure B Volatile Organic Compounds**
(40ml vials with septa and cap)

a) Wash glass vial in hot tap water using laboratory grade non-phosphate detergent;

b) Rinse three times with distilled water;

c) Rinse three times with ASTM type 1 deionized water;

d) Oven dry vials at 104°C for one hour;

e) Allow vials to cool to room temperature in an enclosed contaminant-free environment; and

f) Seal 40 ml vials with septa and cap.

2.3.1.3 **Teflon liners**

a) Wash with laboratory grade non-phosphate detergent;

b) Rinse with distilled water;

c) Rinse with acetone;

d) Rinse with Hexane;

e) Air dry;

f) Place liners in cleaned caps;

g) Heat to 104°C for two hours;

h) Allow to cool in contaminant-free environment; and

i) Cap cleaned bottles.

2.3.1.4 **Cleaning Procedure C Inorganic Metals**
(High Density Polyethylene (HDPE) bottles)

a) Wash polyethylene bottles in hot tap water with laboratory grade non-phosphate detergent;

b) Rinse with 1:1 nitric acid;

c) Invert and air dry in contaminant-free environment; and

d) Cap bottle.

Appendix E

Standard Operating Procedure

SOP E.5
Prevention of Sample Contamination

1.0 PREVENTION OF SAMPLE CONTAMINATION

1.1 The prevention of sample contamination or cross-contamination is an important element in maintaining sample integrity. Contamination can occur virtually anywhere, and therefore, strict adherence to standard operating procedures must be followed.

1.2 It is a primary goal of Alpha to ensure that the environmental conditions do not invalidate the results or adversely affect the required quality of any test measurement.

2.0 Standard Operating Procedure

2.1 Laboratory Facilities

2.1.1 Neighboring work areas in which there are incompatible activities are physically separated as a precaution to prevent cross-contamination. Physically separating work areas is a key component in producing accurate data results and includes such activities as volatile organic handling areas and semivolatile sample preparation.

2.1.2 The heating, ventilation and air-conditioning (HVAC) systems have been engineered to isolate incompatible activities as a precaution against cross-contamination. HVAC systems are designed to ensure the analytical rooms and volatile organic sample preparation rooms are under positive pressure by pulling the makeup air from the outside and not using indoor re-circulated air which may have become contaminated.

These types of roof vents are placed upwind from the extraction exhaust hood roof vents to prevent that source of potentially contaminated exhaust air from re-entering the laboratory as the outside make-up source.

There are other areas in the laboratory which needs to be under negative pressure to ensure the air in those laboratory facilities are continually being evacuated such as the extraction laboratories.

2.2 Field Sampling

2.2.1 Prevention of sample contamination begins in the field when samples are collected. Samples must be collected according to the procedures described in the FSP or a project specific plan.

2.2.2 Alpha maintains a sequestered supply of sample containers which are factory cleaned according to the procedures described by the USEPA "Specifications and Guidance for Contaminant-Free Sample Containers". Each case of sample container are identified with a lot number which can be traced back to an analytical report certifying its cleanliness.

2.2.3 Chemicals used to preserve environmental field samples, such as hydrochloric acid, are analyzed prior to its use to prevent possible cross-contamination.

2.3 Sample, Extract and Standard Storage

2.3.1 Samples are segregated from sample extracts and from standards to prevent possible cross-contamination or laboratory contamination. In addition, samples are segregated from one another depending upon factors such as matrix or the types of analysis requested and are maintained under refrigerated conditions.

Note: When pure products or samples are suspected of containing high concentrations of target analytes, they are isolated from other samples by placing them into sealed paint cans and storing those samples in specifically designated refrigerators to prevent any possible cross-contamination.

2.3.2 Extracts are segregated from samples and from standards to prevent possible cross-contamination or laboratory contamination. In addition, sample extracts are segregated from one another depending upon factors such as solvent or the types of analysis requested and are maintained under refrigerated conditions.

2.3.3 Standards are segregated from samples and from sample extracts to prevent possible cross-contamination or laboratory contamination. In addition, standards are segregated between types of analysis (e.g. volatile, semi-volatiles and metals) and are maintained under refrigerated conditions.

2.4 Sample and Standard Preparation

2.4.1 Extraction technicians adhere to SOP's regarding cleaning of glassware, operations of the dishwasher, and other practices used in the laboratory to prevent possible cross contamination or laboratory contamination.

2.4.2 Once the glassware is used, it is scrubbed and washed according to the written SOP's, and oven dried accordingly.

2.4.3 All glassware which is used during sample preparation, extraction or dilution, is rinsed multiple times with the solvent or acid used in the procedure prior to its use.

2.4.4 Syringes which may come in contact with the sample or with the sample extracts are thoroughly rinsed before and after use.

2.4.5 Glassware and syringes used in the preparation of standards and samples are segregated by analysis such as VOC, semi-volatile and metals.

2.4.6 Alpha buys the best solvents and acids available for low-level environmental analysis. Solvents and acids are maintained in an environment free of potential cross-contamination from air-borne analytes.

2.4.7 Water used in washing or the rinsing of VOC syringes, VOC sample preparation, metals and inorganic sample preparation or associated equipment is produced from our water system and documented to be free of any target analytes which the water is used for.

2.5 Sample Analysis

2.5.1 Analytical instruments are oven-cycled baked or cleaned after highly contaminated samples have been analyzed. Prior to the analysis of additional samples, a method blank or instrument blank is analyzed for proof that a clean analytical instrument was used and did not contribute to environmental sample contamination caused by potential analytical "carry-over".

2.5.2 Extraction personnel or staff members which have come from the extraction lab and need to enter the VOC analytical room, first must aerate themselves by walking outside prior to entering.

2.5.3 Storage blanks are used to determine if cross-contamination may have occurred. Several types of storage blanks are used to monitor possible contamination either from the lab or sources other than environmental contamination. These blanks are analyzed on an as-need basis for many of these storage blanks and on a regular frequency for the remainder. For a better understanding of the types of blanks analyzed, and how they are used to monitor contamination refer to SOP E.7 for details.

Appendix E

Standard Operating Procedure

SOP E.6
Standards Preparation Procedure

STANDARDS PREPARATION STANDARD PREPARATION PROCEDURE

1.0 Identification of Test Method

1.1 The preparation of standards and reagents are in support of the various analytical methods described in the Procedure Manual containing the analytical SOPs,

2.0 Applicable Matrix or Matrices

Not applicable

3.0 Method Detection Limit

Not applicable

4.0 Scope and Application

4.1 The preparation and maintenance of standards are essential elements for the determination of chemical analytes. The procedures for the preparation of all standards and subsequent dilutions must be consistent and standardized to minimize possible analytical errors.

4.2 This procedure includes all reportable target analytes for all methods. Many EPA standardized methods do not describe in full detail, the preparation of standards, compound stability, expiration dates, etc for the listed method analytes; however, each individual in-house analytical method SOP describes these procedures in detail.

4.3 This procedure is intended as a general guideline for the preparation and the documentation of standards and reagents and should be used in conjunction with the individual in-house method SOPs.

5.0 Summary of Method

5.1 Pure product "Neat" standards are prepared by measuring an appropriate mass or weight of the target analyte into Class A glassware and diluting to volume with the appropriate solvent. The net weight gain is calculated, and the concentration is determined.

5.2 Commercial and/or primary dilution standards may be further diluted (secondary dilutions) to a pre-determined concentration, by measuring an appropriate volume of the concentrated standard and diluting with solvent to the appropriate final volume.

5.3 All measuring and weighing devices including balances, syringes, auto-pipettors, volumetric glassware and/or manual volumetric dispensing devices are certified to accuracy and precision as described by NELAP.

6.0 Definitions

6.1 America Chemical Society (ACS) Grade Chemicals - Chemicals of the highest quality; which often exceeds the latest purity standards set by the American Chemical Society. ACS grade chemicals does not imply a standardized percent purity for all chemicals, but rather is a purity threshold established for individual compounds. ACS grade is the only universally accepted standard grade when evaluating chemicals.

6.2 Primary Dilution Standard - A solution prepared from the stock standard solution and diluted as needed to prepare a more dilute solution.

6.3 Reagent Grade Chemicals - Chemicals with purity that are typically equal to ACS grade. The grading of these types of chemicals are similar to that of ACS grade in that reagent grade does not imply a standardized percent purity for all chemicals; rather it is a purity threshold established for individual compounds and is typically less pure than ACS grade. This grade is suitable for analytical work and is more than adequate for general lab use.

6.4 Stock Standard Solution (SSS) - A concentrated solution containing method analytes prepared using reference material or purchased from a commercial source.

7.0 Interference

7.1 It is beyond the scope of this SOP to describe all potential sources of interference, but is a reminder to verify glassware, labware etc., are free from interferences when preparing standards and reagents.

7.2 Sources of Potential Contamination During Standard Preparation

There are several potential sources of contamination when preparing standards with this procedure.

7.2.1 Contaminated neat material is particularly a potential problem if the neat material is < 98% pure product. Since it is not "pure" product, the remaining non-target material, may be a source of contamination and must be evaluated carefully.

7.2.2 Contaminated dilution solvent.

7.2.3 Contaminated standard preparation glassware to include, syringes, volumetric flasks, weigh boats, etc.

7.2.4 Contamination by carryover can occur whenever high-concentration and low-concentration standards are prepared in sequence. To reduce the potential for

carryover, the standard preparation syringe and/or volumetric measuring containers should be rinsed between standard preparation with an appropriate solvent, typically the dilution solvent.

7.2.4.1 Syringe Rinsing

Clean syringes by flushing the syringe barrel approximately 7-10 times using the standard dilution solvent before and/or after the preparation of individual standards.

7.2.4.2 Volumetric-ware Rinsing

All volumetric glass and plastic ware must be scrupulously cleaned. Clean as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinse with tap water and reagent-free water. Drain the glassware and dry in an oven at 104°C for several hours or rinse with methanol if the labware is to be used for organic methods of analysis and/or 1:1 nitric acid if the labware is to be used for inorganic methods of analysis and drain. Store dry volumetric-ware in a clean environment.

Note: It is extremely important not to dry Class A glassware or plastic ware in ovens above this temperature, due to the potential change in internal volume from the heating and cooling cycles. Most Class A volumetric ware is certified to 1-2%, but is not warranted if heated above 104°C.

7.3 Sources of Potential Organic Contamination from Phthalate Esters

7.3.1 Common plastics contain varying amounts of phthalate esters and are easily extracted from these types of material during laboratory operations.

7.3.2 Interference from phthalate esters are best minimized by eliminating contact with any plastic material by using borosilicate glassware and checking all solvents and reagents for phthalate contamination.

7.4 Sources of Potential Inorganic Contamination

7.4.1 Zinc, and other metals may leach from the walls of plastic volumetric glassware.

7.4.2 Boron may leach from the walls of borosilicate glassware.

8.0 Safety

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible extent. A reference file of material safety data sheets is available. See Alpha's Laboratory Safety/Hazardous Communications Manual and Chemical Hygiene Plan for additional information and details.

9.0 Equipment and Supplies

9.1 Equipment

- Analytical and top loading balances, see balance SOP for details
- Manual Volumetric Dispensing Devices, see MVDD SOP for details

9.2 Supplies

- Syringes of various sizes,
- Pipettes of various sizes,
- Volumetric glass water of various sizes,
- ACS grade acids, and
- Pesticide grade solvents.

10.0 Reagents and Standards

10.1 Reagents

10.1.1 Acids - Ultra high-purity grade acids used in the preparation of standards must be used to avoid any potential contamination. Concentrated nitric and hydrochloric acids should be analyzed to determine the levels of impurity.

10.1.2 Reagent Water - Distilled or deionized water 17.8 Mohm or better, free of the analytes of interest. Water should contain particles no larger than 0.20 µm. This is commonly referred to as ASTM type I water (ASTM D1193).

10.1.3 Solvents - Organic solvents used in the preparation of standards must be of pesticide grade or better.

10.2 Standards

10.2.1 See the following tables for details.

11.0 Sample Collection, Preservation and Storage

11.1 See the following tables for standard storage and expiration.

Stability of Standards
Organic Methods of Analysis
Table E.6-1

Method	Expiration Stock Standard Solutions	Expiration Primary/Secondary Dilutions	Expiration Working Standards	Dilution Solvent
Chlorinated Pesticides 608/SM6630C/8081	Replace after 1 year, or manufacturer's expiration date, or sooner if QC indicates a problem. Store at ≤6°C in the dark	Replace after 6 months or manufacturer's expiration date, or sooner if QC indicates a problem. Store at ≤6°C in the dark	Replace after 6 months or manufacturer's expiration date, or sooner if QC indicates a problem. Store at ≤6°C in the dark	Hexane, iso-octane, acetone, methanol
PCB / Aroclor 608/SM6630C/8082	Replace after 1 year, or manufacturer's expiration date, or sooner if QC indicates a problem. Store at ≤6°C in the dark	Replace after 6 months or manufacturer's expiration date, or sooner if QC indicates a problem. Store at ≤6°C in the dark	Replace after 6 months or manufacturer's expiration date, or sooner if QC indicates a problem. Store at ≤6°C in the dark	Hexane, iso-octane, acetone, methanol
PCBs/Aroclors in Oil EPA-600/4-81/045	Method states: "...standards are non-labile and if maintained, can be stored indefinitely..." Clarification: will use expiration dates as described in 608/SM6630C/8082 above.			Hexane, iso-octane, acetone, methanol
TPH-Extractable 8015B/D-DRO	Replace after 1 year, or manufacturer's expiration date, or sooner if QC indicates a problem. Store at ≤6°C in the dark	Same as SSS	Same as SSS	Hexane
TPH-Purgeable 8015B/D-GRO	Replace after 6 months or manufacturer's expiration date, or sooner if QC indicates a problem. Store at -10 to -20°C with minimal head space.	Same as SSS	Prepare aqueous working standards daily with zero head space.	Methanol for SSS and dilutions. Organic free water for daily working standards.
Volatile Organics 624/8260B	Gases- stable for at least 1 week. Longer if drift is less than 20%. Liquids - stable for at least 6 months or manufacturer's expiration date or sooner if QC indicates a problem. Store at -10 to -20°C with minimal head space.	Same as SSS	Prepare aqueous working standards daily with zero head space.	Methanol for SSS and dilutions. Organic free water for daily working standards.
Semivolatile Organics 625/8270C	Replace after 1 year, or manufacturer's expiration date, or sooner if QC indicates a problem. Store at ≤6°C in the dark	Replace after 6 months or manufacturer's expiration date, or sooner if QC indicates a problem. Store at ≤6°C in the dark	Replace after 6 months or manufacturer's expiration date, or sooner if QC indicates a problem. Store at ≤6°C in the dark	Methylene chloride or other appropriate solvent.
SDWA Volatile Organics 524.2	Gases - stable for at least 1 week Liquids - stable for at least 4 weeks Should replace after 6 months or manufacturer's expiration date or sooner for all standards if comparison with check standards indicates a problem. Store at <0°C with minimal head space	Same as SSS	Prepare aqueous working standards daily with zero head space.	Methanol for SSS and dilutions. Organic free water for daily working standards.
Organic Acids	Replace after 6 months or sooner if QC indicates a problem. Store at ≤6°C in the dark	Replace after 6 months or sooner if QC indicates a problem. Store at ≤6°C in the dark	Replace after 1 week or sooner if QC indicates a problem. Store at ≤6°C in the dark	Reagent-free water

Stability of Standards
Inorganic and Wet-chemistry Methods of Analysis
Table E.6-2

Method	Expiration Stock Standard Solutions	Expiration Primary/Secondary Dilutions	Expiration Working Standards	Dilution Solvent
Conductivity 120.1/SM2510B	No method specified criteria. In-house criteria - if stored in sealed containers, replace after 1 year, or manufacturer's expiration date, or sooner if QC indicates a problem. Note: SSS are purchased as satchets. These satchets contain a small volume of standard sealed in an aluminum bag. Once a satchet is opened and used, it is discarded and not re-used due to potential degradation from atmospheric CO2.	Same as SSS	Same as SSS	Reagent grade water.
pH 150.2/SM4500H B/ 9040C/9045D	No method specified criteria. In-house criteria - replace after 6 months, or manufacturer's expiration date, or sooner if QC indicates a problem.	NA Same as SSS	No method specified criteria. In-house criteria - pour fresh working standards from SSS daily. Do not re-use due to potential degradation from atmospheric CO2.	NA
TS - SM2540B TDS - SM2540C TSS - SM2540D	No method specified criteria. In-house criteria - replace after 1 year, or sooner if QC indicates a problem.	No method specified criteria. In-house criteria - replace after 1 year, or sooner if QC indicates a problem.	No method specified criteria. In-house criteria - replace after 1 year, or sooner if QC indicates a problem.	Reagent grade water.
Turbidity 180.1/SM2130B	Method specifies 1 month if preparing in the lab. Does not specify commercial standards. In-house commercial standards - AMCO-AEPA manufacturer specifies 1 year or sooner if comparison to QC indicates a problem	NA Same as SSS	Method specifies 1 week if preparing in the lab. Does not specify commercial standards. In-house commercial standards - AMCO-AEPA manufacturer specifies 1 year or sooner if comparison to QC indicates a problem	NA
Acidity SM2310B	Method states a minim of 1 week for sodium carbonate used to normalize hydrochloric or sulfuric acid. Method has no other expiration date for standards or reagents. In-house criteria - replace after 6 months, or manufacturer's expiration date, or sooner if QC indicates a problem.	Same as SSS	Same as SSS	Reagent grade water.
Alkalinity SM2320B	Method states a minim of 1 week for sodium carbonate used to normalize hydrochloric or sulfuric acid. Method has no other expiration date for standards or reagents. In-house criteria - replace after 6 months, or manufacturer's expiration date, or sooner if QC indicates a problem.	Same as SSS	Same as SSS	Reagent grade water.
Oil and Grease 1664A	Replace after 6 months, or manufacturer's expiration date or sooner if QC indicates a problem. Store at room temperature in the dark.	Same as SSS	Same as SSS	Acetone

Stability of Standards
Inorganic and Wet-chemistry Methods of Analysis
Continued
Table E.6-2

Method	Expiration Stock Standard Solutions	Expiration Primary/Secondary Dilutions	Expiration Working Standards	Dilution Solvent
Ammonia SM4500NH3 B SM4500NH3 D	No method specified criteria. In-house criteria - replace after 6 months or manufacturer's expiration date or sooner if QC indicates a problem.	Same as SSS	No method specified criteria. In-house criteria - working standards should be prepared fresh daily or manufacturer's expiration date or sooner if QC indicates a problem.	Reagent grade water.
TKN SM4500Norg C SM4500NH3 D	No method specified criteria. In-house criteria -replace after 6 months or manufacturer's expiration date or sooner if QC indicates a problem.	Same as SSS	No method specified criteria. In-house criteria - working standards should be prepared fresh daily or manufacturer's expiration date or sooner if QC indicates a problem.	Reagent grade water.
Iron SM3500Fe D	One year or manufacturer expiration date prior to color development.	Same as SSS	Non-colored working standards are prepared fresh daily. Method specifies a 6 month criteria for color developed standards provided they are sealed and protected from light. In-house criteria - working color developed standards should be replaced after 1 month or sooner id QC indicates a problem.	Reagent grade water.
Hexavalent Chrome 7196A/SM3500Cr D	No method specified criteria. In-house criteria - replace after 1 year or manufacturer's expiration date or sooner if QC indicates a problem.	Same as SSS	No method specified criteria. In-house criteria - working standards should be prepared fresh daily.	Reagent grade water.
Chlorine SM4500Cl G	No method specified criteria. In-house criteria - replace after 1 year, or manufacturer's expiration date or sooner if QC indicates a problem.	Same as SSS	No method specified criteria. In-house criteria - working standards should be prepared fresh daily or manufacturer's expiration date or sooner if QC indicates a problem.	Reagent grade water.
Total Phosphorus 365.3//SM4500P E	No method specified criteria. In-house criteria - replace after 1 year or manufacturer's expiration date or sooner if QC indicates a problem.	Same as SSS	No method specified criteria. In-house criteria - working standards should be prepared fresh daily, but may be used up to 48 hours.	Reagent grade water.
Sulfide SM4500S D	No method specified criteria. In-house criteria - replace after 1 month or manufacturer's expiration date or sooner if QC indicates a problem.	Same as SSS	No method specified criteria. In-house criteria - working standards should be prepared fresh daily or manufacturer's expiration date or sooner if QC indicates a problem.	Reagent grade water.
COD 410.4/SM5220D	Replace after 3 months if no visible microbial growth, or manufacturer's expiration date or sooner if QC indicates a problem, refrigerate	Same as SSS	Same as SSS	Reagent grade water

Stability of Standards
Inorganic and Wet-Chemistry Methods of Analysis
Continued
Table E.6-2

Method	Expiration Stock Standard Solutions	Expiration Primary/Secondary Dilutions	Expiration Working Standards	Dilution Solvent
TOC SM5310C	No method specified criteria. In-house criteria - replace after 1 year or manufacturer's expiration date or sooner if QC indicates a problem. Store at ≤6°C.	Same as SSS	Same as SSS	Reagent grade water.
Metals 200.8/6020	No method specified criteria. In-house criteria - replace after 1 year or manufacturer's expiration date or sooner if QC indicates a problem.	Same as SSS	Method 200.2 - working CAL standards 2 weeks. Not specified for non-CAL standards. In-house criteria - replace after 1 year or manufacturer's expiration date or sooner if QC indicates a problem.	1-2% nitric acid and reagent grade water.
Anions 300.0/9056	Method states a minimum of 1 month when stored at 4°C. In-house criteria - replace after 1 year or manufacturer's expiration date or sooner if QC indicates a problem. Store at ≤6°C.	Same as SSS	Working standards 1 week. Working standards containing Nitrate, Nitrite and/or O-phosphate should be prepared fresh daily, but may be used up to 48 hours.	Reagent grade water.
Perchlorate 314.0	Replace after 1 year or manufacturer's expiration date or sooner if QC indicates a problem. Store at room temperature.	Same as SSS	Same as SSS	Reagent grade water.

12.0 Quality Control

12.1 Isolation of Standards

12.1.1 Potential contamination and/or cross-contamination is a concern for all samples, sample extracts and standards. In order to minimize the potential damage from this type of problem, standards are stored and isolated in their own refrigerators independent from samples and sample extracts.

12.1.2 Standards are also isolated according to method of analysis. Typically, methods are designed to analyze a specific class of compounds or to analyze analytes of a common volatility range or other chemical characteristic. This provides a common separation of standards, samples and sample extracts by method of analysis.

12.1.3 Neat standards are additionally isolated from their common diluted standards of the same method. Neat standards, are stored and sealed in paint cans to further help minimize any potential problems due to cross-contamination of high concentration pure product standards.

13.0 Calibration and Standardization

13.1 Standardization

Standards made from neat material are considered a source of reference standard material used in the determination of various methods of analysis. If available, neat material is purchased as ACS grade and no purity correction is required. However, if the chemical purity is <98 % or ACS grade reference material is unavailable, then the next best grade of neat material or reagent grade is purchased. In these cases all standard material should be corrected for purity, when the purity is <98%.

14.0 Procedure

14.1 The standard preparation logbook is designed to maintain records on standards, reagents and reference material preparation. These records document the traceability of purchased stock and neat standards to the method of preparation, the standards lot number, the standards expiration date and the preparer's initials.

14.2 Identification of Standards, Solvents and Reagents

14.2.1 All containers of prepared standards and reference material are labeled with a unique identification and expiration date that unequivocally links that container with its associated standard and reference material documentation.

14.2.2 All reference and non-reference chemicals used in any of the methods of analysis and/or extraction procedures are given an in-house identification number for traceability.

14.2.3 Reagent identifications are segregated by type of method followed by a date, followed by a numerical identification for that day. For example:

FID110503-06 would indicate:

FID - identifies the section of the laboratory which uses GC/FID instruments and where the standard was made or reference material will be used,

110503 - indicates the date in which the standard was logged into the system or prepared, and

-06 - indicates the sixth standard or other reference material identified during that day.

14.3 Documentation of Reagents

14.3.1 After the reagents, such non-standard reference chemicals, have been given an identification they are further documented by entering their relevant information such as lot number, manufacturer, expiration date, etc. into to our Solvent and Chemical Log.

14.4 Documentation of Reference Material

14.4.1 Reference material such as commercially prepared standard mixes and neat material of individual compounds must be documented into Alpha's tracking system to insure proper identification and traceability of these materials.

14.4.2 Certificates of Analysis (C of A)

14.4.2.1 These types of reference material are typically accompanied with a C of A, which describes the compound or mix of compounds, a lot number, and purity or absolute concentrations from the manufacturer.

14.4.2.2 Upon receipt of the standards and C of As, the chemist or technician responsible for the preparation and traceability of that standard must date and initial all certificates. The standard is also given a standard identification, which follows that standard throughout its life in the laboratory.

14.4.2.3 If the reference material is received without the C of A, it is the responsibility of that analyst, or technician, to call the manufacturer and request this information. Often times this information can be downloaded from the manufacturer's web site.

14.4.2.4 Once the standard is logged in to the tracking system the C of A for that standard is then placed into the Reference Material Logbook, a 3-ring binder, and stored for historical reference.

14.5 Documentation of the Original Container

14.5.1 If standards, reagents or reference material are kept in the original containers (as provided by the manufacturer or vendor) the must be labeled with the expiration date in addition to the laboratory identification.

14.6 Preparation of Standards from Neat Material

14.6.1 Determine the concentration and volume of the secondary or working

standard required. Typically, stock standards are prepared at a concentration of ten to a hundred times the concentration required for the final working standard to allow for additional dilutions.

14.6.2 Prepare stock standards in the diluting solvent, or acid.

14.6.3 If a 10 ml volumetric flask is used, place approximately 9.8 ml of solvent in the tared volumetric flask.

14.6.4 Allow the flask to stand, un-stoppered, for approximately 5-10 minutes or until all wetted surfaces have dried.

14.6.5 Weigh the flask to the nearest 0.1mg and record and/or re-tare the balance.

14.6.6 Liquid Neat Material

Using a dedicated syringe or preferably disposable pipette, immediately add two or more drops of reference material to the flask, then re-weigh. The liquid must fall directly onto the solvent without contacting the neck of the flask.

14.6.7 Solid Neat Material

Using a disposable weighing boat or weighing paper, measure an approximate weight of the neat material and add directly into the tared flask.

14.6.8 Re-weigh the volumetric flask and record the net gain in weight.

14.6.9 Dilute to volume with the diluting solvent, then mix by inverting the flask several times.

14.6.10 Record all data in the Analytical Standard Log book.

14.6.11 Transfer the stock standard solution into a Teflon-lined screw cap vial or other appropriate long-term storage container.

15.0 Calculations

15.1 Calculation of Concentration

When secondary or working standards are prepared, final standard concentrations are determined as follow:

15.1.1 Determine the concentration and volume of the diluted standard required.

15.1.2 Calculate the amount of stock standard needed using the following equation:

$(V_f)(C_f) = (V_i)(C_i)$

Where:

V_f = Volume of final secondary/working standard,
C_f = Concentration of final secondary/working standard,
V_i = Volume of initial primary/stock standard, and
C_i = Concentration of initial primary/stock standard.

15.1.3 Add the appropriate solvent and dilute to volume, stopper, then mix by inverting the flask several times, and

15.1.4 Record all required information in the Analytical Standard Log book.

15.2 Calculation of Concentration Corrected for Purity

15.2.1 When standards are less than 98% pure, concentrations should be adjusted by calculating the correct concentration. For example if a neat standard were prepared at a target concentration of 1000 ug/mL and the standard was 96% pure the actual standard concentration would be:

1000 ug/mL x (0.96) or 960 ug/mL.

16.0 Method Performance

Not applicable.

17.0 Pollution Prevention

Minor amounts of solvents and/or acids are used with this procedure as the diluting reagent. The only other chemicals used in this procedure are the occasional use of neat materials and/or commercially prepared standard mixes used in the preparation of standard dilutions. All are used in extremely small amounts and pose no threat to the environment. However, all standards, solvents etc., must be disposed of properly according to our Hazardous Waste and Safety Manual.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Not applicable.

19.0 Corrective Actions for Out-of-Control Data

Not applicable.

20.0 Contingencies for Out-of-Control Situations

Not Applicable.

21.0 Waste Management

All expired standards and/or reagents must be disposed of properly as described in our Waste Disposal SOP. Reference Alpha Analytical's Sample Waste SOP.

Appendix E

Standard Operating Procedure

SOP E.7
Storage Blank Procedures

1.0 STORAGE BLANK PROCEDURES

1.1 VOC samples may be contaminated in a number of different ways. Several types of blanks are used to monitor various sources of potential contamination. Storage blanks are used to monitor possible VOC contamination contributed by the refrigerator or by cross-contamination from samples stored in the same refrigerator.

2.0 Standard Operating Procedure

2.1 Storage blanks are used to monitor possible VOC contamination. This potential for cross-contamination may occur by the out-gassing of highly contaminated samples through the container septum or sample containers that are contaminated on the outside of the container walls and subsequently contaminate the storage space.

VOC samples may also be contaminated by the refrigerant (Freon) used in refrigerators, by air-conditioning units or by a number of other means.

2.2 Storage blanks are prepared and placed in sample containers and treated as a sample. Storage blanks are not, however, transported to the sample site or exposed to any sampling activities.

2.3 It is the responsibility of the assigned person to prepare, store, and monitor storage blanks for potential contamination.

2.4 Refrigerator Storage Blank Preparation (Aqueous Refrigerators)

2.4.1 The refrigerator monitor prepares refrigerator storage blanks, by filling 40ml VOA vials with organic free water.

2.4.2 When making refrigerator storage blanks an additional blank is prepared as a QC check blank. This blank is prepared and analyzed to assure the original storage blanks are free from VOC contamination. The results of this analysis is documented in the Refrigerator Storage Blank Log Book.

2.5 Refrigerator Storage Blank Preparation (Soil Refrigerators)

2.5.1 The refrigerator monitor prepares refrigerator storage blanks, by filling 40ml VOA vials with 8 ml of methanol, mimicking the volume of methanol used in the soil extraction procedure.

2.5.2 When making refrigerator storage blanks an additional blank is prepared as a QC check blank. This blank is prepared and analyzed to assure the original storage blanks are free from VOC contamination. The results of this analysis is documented in the Refrigerator Storage Blank Log Book.

2.6 Quarterly Storage Blanks (Long Term)

2.6.1 Twelve refrigerator storage blanks, in triplicate for a total of thirty-six, are prepared at the beginning of the calendar year and placed in each designated refrigerator.

2.6.2 During the first week of each new quarter one of the three vials designated for that quarter is analyzed for VOCs.

2.7 Semi-monthly Water Storage Blanks (Short Term)

2.7.1 In addition to the quarterly storage blanks, three storage blanks are prepared at the beginning of each 2-week interval and placed in the designated VOC sample refrigerators to mimic the storage time for typical VOC samples. At the end of that two-week period, one of the three storage blanks is analyzed for VOCs.

2.8 Analytical Procedure

2.8.1 VOC storage blank results must be less than one half of the reporting limit for non-laboratory solvents and below the reporting limit for common lab solvents to be acceptable. Analytical results are documented in the Refrigerator Storage Blank Log Book.

2.9 Procedure for non-contaminated Storage Blanks

2.9.1 If VOCs are not found in a storage blank, then it can be determined the refrigerator has been clean of potential air-borne contaminants and no cross-contamination could have occurred for that time period.

2.9.2 If the semi-monthly storage blank indicates no contamination after the two-week storage period, another set of three storage blanks is prepared and placed in the designated refrigerator.

2.9.3 Semi-monthly Water Storage Blanks (Medium Term)

The two remaining original vials are also kept as a backup and purged from the refrigerator on the subsequent 2-week interval and are referred to as the semi-monthly medium term storage blanks.

2.9.4 If both the semi-monthly storage blank and quarterly blank indicates no contamination at the end of the year, another set of 12 quarterly storage blanks is prepared and placed in the designated refrigerator. The remaining original quarterly blanks are purged from the refrigerator.

2.10 Procedure for Contaminated Storage Blanks

2.10.1 If the semi-monthly storage blank indicates possible contamination, the second of the three semi-monthly storage blank vials is analyzed to confirm the presence of the contaminant.

2.10.2 If the second semi-monthly storage blank is confirmed positive, then a third stage of confirmation is performed.

2.10.3 This third stage consists of analyzing the previous months semi-monthly medium term storage blank as well as one of the quarterly storage blanks and potentially the activated carbon.

2.10.4 If the quarterly storage blank or the previous months semi-monthly medium term storage blank analysis is confirmed positive, then it should be considered that all samples within that 2 week period may be suspect for that analyte and appropriate actions should be taken.

2.10.5 Corrective Actions

2.10.5.1 Appropriate actions would be decontamination or decommissioning of the refrigerator.

2.10.5.2 If the refrigerator contained activated charcoal, it should be removed as a protection against additional cross-contamination while cleaning the refrigerator. A tray of new activated carbon is then placed back into the refrigerator.

2.10.5.3 All samples stored in the refrigerator which has been confirmed positive must be isolated to prevent any additional cross-contamination.

2.10.5.4 All reported sample data previously stored in the contaminated refrigerator during the associated time period, must be re-evaluated for the identified contaminant.

2.10.5.5 These samples require a case-by-case decision whether to submit an amended report to the client.

2.11 Housekeeping Procedures for VOC Refrigerators

2.11.1 A tray of activated carbon is placed in all VOC refrigerators to absorb any potential VOC contaminants within the refrigerator.

2.11.2 This carbon is replaced on an as-needed basis and the date of replacement is noted on the refrigerator temperature log.

2.11.3 If it was determined, by the analysis of the storage blanks, cross-contamination may have occurred, then the activated charcoal may be analyzed as another confirmation.

TABLE E.7-1
TABLE OF STORAGE BLANKS AND FREQUENCY

VOC Refrigerator	Bimonthly Analysis	Quarterly Analysis
SAR-1B	Every other Friday	Every 3 Months
SAR-5D	Every other Friday	Every 3 Months
SAR-8A	Every other Friday	Every 3 Months
SAR-10A	Every other Friday	Every 3 Months
SAR-11A	Every other Friday	Every 3 Months
SAR-12A	Every other Friday	Every 3 Months
SAR-14A	Every other Friday	Every 3 Months
SAR-15B	Every other Friday	Every 3 Months

Appendix E

Standard Operating Procedure

SOP E.8
A Practical Application Guide for Performing a Method Evaluation and/or Validation Demonstration to Include: a) Demonstration of Capabilities (DOC), b) Method Detection Limit(MDL), c) Limit of Detection (LOD) and d) Limit of Quantitation (LOQ) Studies

1.0 A Practical Application Guide for Performing a Method Evaluation and/or Validation Demonstration to Include: a) Demonstration of Capabilities (DOC), b) Method Detection Limit (MDL), c) Limit of Detection (LOD), and d) Limit of Quantitation (LOQ) Studies

2.0 Additional References

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

3.0 Demonstration of Capability

3.1 Initial Demonstration of Capability Study

3.1.1 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. Demonstration of Capability (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). In addition, for analytes which do not lend themselves to spiking, e.g, TDS, the DOC may be performed using quality control samples.

1) Clarification: "Significant change" refers to any change in personnel, instrument, test method, or sample matrix that potentially impacts the precision, accuracy, sensitivity and selectivity of the data (for example, a change in detector, column type, sample matrix, or a significant method revision). All new analysts, regardless of experience on that instrument in another laboratory, shall complete a demonstration of capability.

3.1.2 When an analyte is added to an existing accredited test method (i.e., a new target analyte) an initial evaluation is performed for that analyte.

3.2 Continuing Demonstration of Capability

Once the IDC has been performed a continuing demonstration of capability is performed annually and the documentation of continued proficiency by at least one of the following once per year:

i. acceptable performance of a blind sample (single blind to the analyst);

Note: successful analysis of a blind performance sample on a similar test method using the same technology, e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624 or 8260, would require documentation for one of the test methods.

ii. a new IDC study, or a study from a similar test method,

iii. at least four consecutive laboratory control samples with acceptable levels of precision and accuracy within the last 12 months, or

iv. if i-iii cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.

Note: Continuing demonstration of capabilities is continuously monitored during the normal course of sample analysis through the use of LCS data evaluation. Laboratory control samples are typically spiked and monitored at the same spike concentration and windows of acceptability as used for the IDC study. Therefore, analytical batch LCS data may also be used to satisfy this requirement.

3.3 Work Cells

A work cell is considered to be all individuals who see a sample through the complete process of preparation, extraction/digestion and analysis.

3.3.1 Individual Demonstration of Capability within the Work Cell

This work cell or "group" as a unit must meet the DOC criteria and the study must be documented for all individuals within that work cell. Even though the work cell operates as a "team," the demonstration of capability at each individual step in the sequence, as performed by each individual team member, remains of utmost importance, e.g., each team member must demonstrate capability in his/her area of responsibility in the sequence.

2) Clarification: It is not the intent to require each combination/permutation of work cell members to demonstrate group capability since DOC is for the individual only. Even though the work cell operates as a "team," the demonstration of capability at each individual step in the sequence, as performed by each individual analyst/team member, remains of utmost importance. For example, if multiple individuals contribute to a single analytical result (e.g., perform preparation, extraction, and analysis) and that result meets appropriate acceptance criteria, than all individuals have demonstrated their capability for that method of analysis in which they are a work cell member.

Work cells cannot be defined as a group within that team who perform the same step in the same process (for example, extractions for Method 8270), represented by one extraction chemist who has demonstrated capability for that step.

3.3.2 New Members within a Work Cell

3.3.2.1 When a work cell is used, and a member of a work cell changes, the new employee must work with experienced personnel in that area of the work cell where they are employed, e.g., a new analyst should work with an experienced analysts for initial training.

3.3.2.2 This new work cell must demonstrate acceptable performance through acceptable continuing performance checks such as laboratory control samples. This performance is documented and the preparation batch following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control samples, or the demonstration of capability must be repeated. In addition, if the entire work cell is changed or replaced, the work cell must perform a new complete DOC study.

3.4 DOC Study Training Documentation

Technical training of each member of the technical staff is kept up-to-date (on-going) by requiring each member to have on record, found in the training file, a certificate that they have read, understood, and are using the latest version of the QA Manual and SOPs which relate to his/her job responsibilities. This includes the DOC performance of individual analysts as well as all members of the work cell are documented through the use of the DOC Summary Data Form.

3.4.1 Certification Statement

The Certification of Initial/Continued Proficiency Requirements, Figure E.8-1, is used to document the completion of each demonstration of capability. A copy of the certification statement is retained in the personnel training records of each affected employee.

3.4.2 Supporting Data

Data applicable to the study does not need to be attached to the form, but is retained and available for review. The data includes such items as quantitation reports etc., necessary to reproduce the analytical results summarized in the Certification Statement is also retained.

3.5 DOC and Continuing DOC Study Organization

3.5.1 Data is reviewed, to verify the analytical method requirements associated with the study have been achieved.

3.5.2 Data packets should be prepared with the analytical sequence log, followed

by the sample data in chronological order. Batch QC summary sheets and extraction logs should also accompany this data packet. This helps organize the data for peer review.

3.5.3 A copy of the standards preparation logbook clearly documenting the source of the standards, and/or a brief summary of the stock standard solution, secondary solution, working standards, etc. should also be placed in a separate document packet.

Note: A typical level IV data package is more than adequate and may be used for complete study documentation.

3.6 Demonstration of Capability Study Procedure

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

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

3.6.2 The analytes are spiked into a volume of a clean quality system matrix sufficient to prepare four aliquots at the method specified concentration, or if unspecified, at a concentration of 1-4 times the Limit of Quantitation.

Note: See the section below describing the spike concentrations associated with the Limit of Detection and Limit of Quantitation.

3) Clarification: The limit of quantitation is synonymous with the reporting limit. This would suggest that the DOC study should be analyzed at a concentration of 1-4 times the reporting limit; while the NELAC standards also allow the use of LCS samples which are typically spiked at 5-10 times the reporting limit to be used. Therefore, a concentration of 1-10 times the reporting limit is acceptable.

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

3.6.4 Using all the results, calculate the mean recovery and the standard deviations of the population sample (n-1) for each parameter of interest.

4) Clarification: The average recovery (not individual recoveries), is compared to the accuracy limits that are either method specified or determined by the laboratory. For most cases this will be the laboratory established LCS window of acceptability.

Note: When it is not possible to determine the mean and standard deviation, such as for pH logarithmic values, the performance is assessed against established and documented criteria.

3.6.5 Compare the information from above to the corresponding acceptance criteria for precision and accuracy in the applicable test method or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of samples may begin. If any one of the parameters fails to meet the acceptance criteria, the performance is unacceptable for that parameter and the test needs to be repeated.

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

3.6.6.1 Beginning with (3.6.3) above, repeat the test for all parameters that failed to meet the acceptance criteria.

3.6.6.2 Repeated failure, however, generally confirms a problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with (3.6.3) above.

4.0 Test Method Evaluation

4.1 Evaluation for Standardized Methods

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

4.2 Evaluation for Laboratory Developed and Non-standardized Methods

4.2.1 The introduction of a laboratory developed or non-standardized test method typically requires an enormous amount of planning and is assigned to a member of the technical staff who is equipped with the adequate resources to carry out the duties of method development and final evaluation. As method development proceeds, the modifications, changes, experiments, etc. are communicated to all involved personnel, in order to keep them abreast of those changes.

4.2.2 In the event that analyses must be conducted for compounds for which no reliable method exists, or when it is necessary to use methods not covered by standard method procedures, the method goals are discussed and agreed upon

with the client in order to fulfill the client's requirements. Both laboratory developed and non-standardized methods should be appropriately validated before use.

4.2.3 As part of the method development process and to ensure continuous quality of data, QC criteria must be proposed and established that is consistent with similar methods or technology. At a minimum, the method development process must address these QC requirements:

- Calibration;
- Interference and/or contamination;
- Analyte identification;
- Selectivity;
- Sensitivity;
- Precision; and
- Accuracy.

4.2.4 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.

4.3 Method Detection Limit (MDL) Study

4.3.1 The MDL is defined as an estimate of the minimum amount of a substance that an analytical process can reliably detect. An MDL study is analyte and matrix specific and may be laboratory dependent.

Note: This study is also known simply as a Detection Limit (DL) study and may be used interchangeably; however, some organizations suggest these are slightly different. They would identify a DL study as verifying the MDL study.

4.3.2 One way to establish the MDL, is to perform an MDL study defined as, the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. An MDL study is generally conducted as follows:

a) A properly conducted MDL study requires that all sample processing steps be included in the determination of the limit of detection.

b) An MDL study is not required for any component for which spiking solutions or quality control samples are not available such as temperature.

c) An MDL study is not required when test results are not reported to the MDL concentration but reported above the MDL and within the working-range of the instrument calibration.

Note: When a annual MDL study is not performed, data cannot be reported below the LOQ.

4.3.3 MDL Study Frequency

4.3.3.1 Appendix D.1.2.1 of the System 5 NELAC standards states the following:

A LOD (MDL) study is NOT REQUIRED for a test method when the test results are not reported outside of the calibration range.

5) Clarification: Since MDL studies (non-SDWA methods of analysis) are not required under the NELAC standards, DOD requires the method sensitivity to be determined and verified by conducting quarterly a LOD study.

4.3.3.2 NELAC also requires the MDL study be conducted by the protocol in the mandated test method or applicable regulation, e.g., 40 CFR, Part B, Appendix B.

6) Clarification: Alpha follows the procedures as described in 40 CFR, Part 136, Appendix B, Definition and Procedure for the Determination of the Method Detection Limit.

4.3.4 MDL Study Procedure

4.3.4.1 The MDL study is not conducted using real world samples, but using an 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). This is the same matrix as used for the preparation of the method blank or laboratory control sample, e.g., reagent grade water for water matrices, or Ottowa sand, sodium sulfate or Teflon chips for soil matrices.

4.3.4.2 Cleanup Procedures

MDL studies are generated using the preparatory and cleanup procedures routinely used on sample preparation and analysis.

4.3.4.3 MDL Study Frequency

A MDL study is performed initially for all compounds of interest in

each test method. A MDL study should be determined each time there is a change in the test method that affects how the test is performed or when a significant change in instrumentation occurs that affects the sensitivity of the analysis.

Note: See the clarification box for "significant change" as it applies to both the DOC and MDL studies.

4.3.4.4 Number of Replicates

It is recommended to analyze a minimum of ten replicates containing the analytes of interest at the selected concentrations over the course of several days.

7) Clarification: As stated in 40 CFR 136B, the MDL study shall be determined using a minimum of seven replicates. In addition, several regulatory agencies require that if more than seven replicates are processed, data cannot be excluded, unless exclusion is supported with sound, documented technically based justification (e.g., Dixon Outlier Test). The state of California does not agree with this and will not allow exclusion of any data regardless of reason, (e.g., outlier tests are not allowed) and/or number of replicates.

4.3.4.5 Spike Source

A Quality Control (QC) standard is obtained from a source independent from the source used in the instrument calibration. If a completely independent source cannot be obtained, then as a last resort, the QC sample may be prepared using stock standards that are independently prepared from those used in the instrument calibration.

8) Clarification: Since the MDL study samples must be prepared from a source independent from the calibration source; it may be wise to use the same independent second source for the Initial Calibration Verification (ICV), DOC study and MDL study.

4.3.4.6 Spike Concentration

Determine the MDL spike concentration that corresponds to the following criteria:

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

4.3.4.7 Calculate the Standard Deviation (SD) of each of the analytes.

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

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

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, Table E.8-2.

4.3.4.9 Calculate the MDL for each analyte and matrix.

4.3.5 MDL Evaluation (DL Study)

4.3.5.1 If the calculated MDL is higher than the spike concentration, then the study should be repeated at a higher concentration.

4.3.5.2 Compare calculated MDL results to ensure they are at or below the Limit of Quantitation (LOQ).

4.3.5.3 Verify the calculated MDL with a LOD spike, see LOD section below.

9) Clarification: Method 8000B requires the RL/LOQ to be established at or above the lowest calibration point established during the initial calibration curve. This should be further refined and supported by establishing LOQs that have calculated MDLs at or below this limit.

4.3.5.4 Compare the ratio between the mean recovered concentration and the calculated MDL. This ratio should be between 1 and 5 for reagent water matrices and 1 and 10 for other matrices. If the calculated MDL is less than 1/10 of the spike concentration, then the study should be repeated at a lower concentration.

4.3.5.5 MDL Study Data From Multiple Instrument

4.3.5.5.1 If multiple MDL results are generated from multiple instruments with identical configurations, then the highest MDL among those should be used in reporting data from all of those instruments. If a lower MDL is reported for specific samples, then the samples must have been run on that specific instrument on which the lower MDL was generated.

4.3.5.5.2 If multiple instruments with identical configurations are used, then conduct a MDL study on at least one of

the instruments and confirm the attainability of that MDL on all instruments by using a LOD verification check sample. The LOD verification must be performed quarterly on every instrument.

Note: When confirming the MDL using only a LOD verification, the concentration of the LOD verification should be no greater than 3 times the MDL for single analyte tests and 1-4 times the MDL for multiple analyte tests. This is a different proven than when performing the base MDL study.

4.3.6 MDL Study Documentation

All associated supporting data, such as quantitation reports etc., necessary to reproduce the analytical results summarized in the DOC Certification Statement is also retained.

4.4 Limit of Detection (LOD) also referred to as an MDL Verification Check or a DL Study

4.4.1 Definition - An MDL verification procedure used to estimate of the minimum amount of a substance that an analytical process can reliable detect. An LOD is analyte and matrix specific and may be laboratory dependent.

10) Clarification: DoD clarifies this definition as: "The smallest amount of concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%". This is essentially the definition for an MDL study.

4.4.2 Purpose - To establish an analytical concentration that an analytical procedure can reliably detect. This is analyte, matrix and method specific.

4.4.3 Frequency

4.4.3.1 Analyze an LOD immediately following the MDL study to validate the statistics of the MDL study.

4.4.3.2 If an annual MDL study is not performed, LOD verification checks should be performed quarterly.

Note: An LOD verification and/or quarterly LODs are not required when test results are not reported outside of the calibration range.

11) Clarification: DoD requires the LOD to be verified quarterly, regardless of any other regulatory requirement.

4.4.4 Procedure

4.4.4.1 Determine the method LOD for each target analyte of concern in a clean quality system matrix.

4.4.4.2 All sample processing steps must be included in the determination of the LOD.

4.4.4.3 Spike the LOD verification as follows:

a) for singe analyte methods - spike at a concentration of 2 to 3 times the LOD (MDL)

b) for multiple analyte methods - spike at a concentration of 1 to 4 times the LOD (MDL)

4.4.4.4 The LOD verification must be performed on every instrument that is used to analyze and report data.

Note1: An LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperatrure.

Note 2: An LOD study is not required for any component when test results are not reported to the LOD/ (MDL) (versus the Limit of Quantitation or reporting limit).

Note 3: When a LOD study is not performed (MDL) and/or quarterly LOD, analytical data cannot be reported below the LOQ.

4.4.5 Acceptance Criteria

4.4.5.1 The LOD and MDL study is confirmed by the qualitative identification of the analytes in a LOD verification sample.

12) Clarification: DoD clarifies the LOD acceptance criteria as meaning: the apparent signal to noise ratio at the LOD concentration must be at least three and the results must meet all method requirements for analyte identification (e.g. ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is three standard deviations greater than the mean method blank concentrations.

4.4.6 Corrective Action - If the LOD verification fails, additional LOD verification checks may be performed at a higher level to set the MDL higher, or the MDL study should be re-analyzed.

4.5 Limit of Quantitation (LOQ) also referred to as the Reporting Limit (RL)

4.5.1 Definition - The minimum levels, concentrations or quantities of a target analyte that can be reported with a specified degree of confidence.

13) Clarification: DoD clarifies this definition as: "The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard".

4.5.2 Purpose - To define the lowest level of reporting within a specified degree of confidence.

4.5.3 Frequency - The LOQ is verified annually.

Note: The LOQ does not have to be verified annually, if the LOQ is reported at or above the lowest calibration point.

14) Clarification: DoD requires the LOQ to be verified quarterly, regardless of any other regulatory requirement.

4.5.4 Procedure

4.5.4.1 The LOQ study is not required for components or methods for which spiking solutions are not available or inappropriate, e.g. pH.

4.5.4.2 The LOQ is verified by successful analysis of a QC sample spiked with the target analytes of concern in each quality system matrix at a concentration of 1 to 2 times the claimed LOQ.

4.5.4.3 If a client requires a LOQ/RL below the established LOQ,

method modification is required or the client will be required to accept the established LOQ as the lowest technically valid value that can be proved.

4.5.4.4 The verification of the LOQ is not required if the method precision and accuracy is evaluated at the LOQ, e.g., if the LCS and routinely spiked, analyzed and evaluated at this level.

4.5.5 Acceptance Criteria

4.5.5.1 If the recovery of the LOQ verification check standard is within established test method acceptance criteria, or client data quality objectives for accuracy, than the LOQ is acceptable.

4.5.6 Corrective Action

4.5.6.1 If analytes are reported below the established LOQ, they should be flagged as follows:

a) If the analyte is reported (detect or non-detect) down to one-half of the LOD, the data is flagged with a J.

b) If the analyte is reported as a non-detect down to the LOD, the data is flagged with a U.

c) If the analyte is detected between the LOD and LOQ, the data is flagged with a J.

5.0 Outlier Test

Even though CA NELAP does not allow the use of the Dixon Outlier test it is included for reference for use in other programs.

5.1 Often in a series of measurements, one or more of the results will differ greatly from the other values. Theoretically, no data results should be rejected, because it may indicate either a faulty technique that casts doubt on all results or the presence of a true variant in the distribution. In practice, reject the result of any analysis in which a known error has occurred (data rejection must be supported with proof of the error). In environmental analysis, extremely high and low concentrations of contaminants may indicate the existence of areas with problems or areas with no contamination, so they should not be rejected arbitrarily.

5.2 If a set of data is ordered from low to high, and the average and standard deviation are calculated, then suspected high or low outliers can be tested by the following procedure. First calculate the statistic T:

$T = (X_H - X_{AV})/S$ for a high value, or

$T = (X_{AV} - X_L)/S$ for a low value.

5.3 Compare the value of T to the Outlier Statistics Table E.8-1 at the 1% level of significance. If the calculated T is larger than the table value for the number of measurements, n, then the X_H or X_L is an outlier at that level of significance.

Dixon
Outlier Statistics Table

Number of Measurements n	Critical Value 1%
3	1.15
4	1.49
5	1.75
6	1.94
7	2.10
8	2.22
9	2.32
10	2.41
12	2.55
14	2.66
15	2.71
16	2.75
18	2.82
20	2.88
30	3.10

Table E.8-1

Demonstration of Capability
Certification Statement

Date:_____
Alpha Analytical, Inc.
255 Glendale Ave Ste21
Sparks, NV 89431-5778

Analyst Name:_____
Matrix:_____
Method #:_____
SOP #/Rev #:_____

Parameters: MDL/IDC

We, the undersigned, CERTIFY that:

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

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

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

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

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

_____	_____	_____
Analyst	Signature	Date
_____	_____	_____
Quality Assurance Officer	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True:	Consistent with supporting data.
Accurate:	Based on good laboratory practices consistent with sound scientific principles/practices.
Complete:	Includes the results of all supporting performance testing.
Self-explanatory:	Data properly labeled and stored so that the results are clear and require no addition explanation.

Figure E.8-1

TABLE OF STUDENT'S t VALUES AT THE 99 PERCENT CONFIDENCE LEVEL		
NUMBER OF REPLICATES	DEGREES OF FREEDOM (n-1)	$t_{n-1, .99}$
4	3	4.541
5	4	3.747
6	5	3.365
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
12	11	2.718
13	12	2.681
14	13	2.650
15	14	2.624
16	15	2.602
17	16	2.583
18	17	2.567
19	18	2.552
20	19	2.539

Table E.8-2

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	$(\frac{s}{\bar{x}})\ x\ 100$	Relative standard deviation, adjusts for magnitude of observations	Assess precision for replicate results
Percent Difference	% D	$\frac{x_i - x_2}{x_1}\ x\ 100$	Measure of the difference of 2 observations	Assess accuracy
Relative Percent Difference	RPD	$\left(\frac{(x_1 - x_2)}{(\frac{x_1 + x_2}{2})}\right)\ x\ 100$	Measure of the variability that adjusts for the magnitude of observations	Assess total and analytical precision of duplicate measurements
Percent Recovery	% R	$\left(\frac{x_{measured}}{x_{true}}\right)\ x\ 100$	Recovery of spiked compound in pure matrix	Assess accuracy
Percent Recovery	% R	$\frac{\text{Value of Spiked Sample} - \text{Value of Unspiked Sample}}{\text{Value of Added Spike}}\ x\ 100$	Recovery of spiked compound in sample matrix	Assess matrix effects and total precision

x = Observation (concentration)n = Number of observations

Table E.8-3

Appendix E

Standard Operating Procedure

SOP E.9
A Practical Application Guide for Performing Manual and Automated Integration Routines

1.0 A PRACTICAL APPLICATION GUIDE FOR PERFORMING MANUAL AND AUTOMATED INTEGRATION ROUTINES

2.0 General Chromatographic Principals and Goals when Performing Manual Integrations

2.1 The goal of analytical chromatography is to separate sample constituents within a reasonable time. Baseline resolution of each target analyte from co-extracted materials provides the best quantitative results but is not always possible to achieve.

2.2 Some analytical procedures list analytes that may not all be resolved from one another. Therefore, while each of these methods is suitable for the listed compounds, they may not be suitable to measure the entire list in a single analysis. In addition, some methods include compounds that are isomers or closely related compounds which are well-known as co-eluting or are not completely separable. In these instances, the results should be reported as the sum of the two (or more) compounds.

2.3 This application procedure is written as a guide to help aid analysts in determining if there is acceptable baseline resolution and peak shape and that appropriate integration routines are being used. It is also the purpose of this policy to give analysts guidance in how to consistently integrate; how to produce accurate quantification, and how to handle data in situations where accurate quantification is difficult, such as coeluting isomeric pairs. The importance of the analysts' experience in performing these types of anlayses to the ultimate success of the methods can not be overemphasized.

2.4 It is not the intent of this application procedure to provide guidelines for all possible case scenarios when less than perfect integration is achievable. However, this application procedure is written such that, when doubt exists regarding integration, then the analyst must use the philosophy of consistent integration techniques that are also compatible with the guidelines of Alpha's Ethics Policy.

2.5 This application procedure is written as a philosophical approach to help aid analysts' decision making procedures in the integration of chromatograms.

3.0 Manual Integration Policy

3.1 There are no mandated rules that can be applied to the practice of integration. However, it is Alpha's policy to produce analytical data using the automated and manual integration practices, in a manner that is:

- Non-arbitrary integration - standards, control samples, and client samples are all integrated using consistent integration practices.
- Rational integration - data can be backed up with the reason for a particular integration practice.

3.2 This policy is applicable to all data produced from environmental analyses performed by GC, GC/MS, HPLC, IC or any other instrument capable of producing a chromatogram used to report quantitative data.

3.3 This policy is not intended to teach chromatography, but rather provides guidance when chromatographic difficulties present itself.

3.4 This policy is independent of any software.

3.5 This policy is consistent with the EPA's procedures and methods for environmental analyses, good laboratory practice and Alpha's ethics policy conducive to producing consistent, scientifically valid and defensible data for use by any end user.

4.0 Manual Integration Guidelines and Procedures

4.1 Consistent Verses Inconsistent Integration

4.1.1 Individual analytes in the initial calibration standards should be integrated consistently, (i.e. the same integration technique), with calibration points within the initial calibration curve regardless of concentration.

4.1.2 Individual analytes in the calibration verification standards should be integrated using the same integration technique, for that analyte, as used with that initial calibration.

4.1.3 Individual analytes in samples should be integrated using the same integration technique, for that analyte, as used with that initial calibration.

Generally, samples should be integrated in a manner consistent with the calibration standards. However, because of matrix effects, interferences, and compounds at varying concentrations, it is not always possible nor appropriate to integrate samples exactly the way the calibration standards were integrated. In calibration standards, each compound is usually at the same concentration as every other compound in the standard. In a sample, each compound will probably have a different concentration. Therefore, an integration technique that was accurate for a calibration standard may not be accurate for a sample. The analysts' experience, detailed examination, and good judgement is required in these cases.

4.2 Poor Instrument Chromatography Verses Nasty Samples

4.2.1 When establishing and evaluating a set of instrument parameters and conditions for an analytical system the analyst has a primary responsibility to ensure adequate analyte sensitivity, resolution, lineartiy, and quantitation. The analyst should ask themselves these fundamental questions:

- Is the low standard response so small that it does not produce a three to one signal to noise ratio?
- Does the separation between compounds become worse as the concentration increases?
- Is there column overloading at the higher concentrations?
- Is the instrument linear throughout the whole curve?
- Is the column capacity sufficient for the amount of material being analyzed?

If this is not the case, the analyst must find and resolve the problem before proceeding with sample analysis.

4.3 Documentation of Manual Integrations

4.3.1 Data Files

4.3.1.1 Software generated automatic integrations must be supported by sound integration routines and parameters. These integration routines are documented and supported by the method file saved in support of the final analytical data. When software generated integration routines need to be adjusted; then the manual integrations must be based upon industry accepted chromatographic integration practices.

4.3.1.2 This procedure does not imply that an additional electronic data file needs to be kept; however, it does imply the final data file must be fully supported electronically.

4.3.2 Chromatograms

4.3.2.1 Manual integrations performed on initial calibrations should be documented to include a complete audit trail for those manipulations (i.e., the chromatograms obtained before and after the manual integration should be retained to permit reconstruction of the results).

The analyst performing the manual integrations must sign and date each chromatogram and document the rationale for performing manual integrations (an electronic signature is acceptable that is to say if the analyst name or letter abbreviation is included on those chromatograms).

4.3.2.2 The "before" data file chromatogram needs to be printed in such a manner to visually verify the manual manipulation of peaks and/or a notation of rationale. This does not imply each and every peak should be printed out on individual pages, but it is a strong suggestion that a "clear picture speaks a thousand words." The principal purpose of printing a "before" and "after" chromatogram is to develop and track a complete audit trail of all manual integrations.

Note: Records for manual integrations may be maintained electronically as long as all requirements, including signature requirements, are met and the results can be historically reconstructed.

4.4 Manual Integration of Multi-response Analytes

4.4.1 Several analytes such as diesel, gasoline, toxaphene, technical chlordane and PCBs are considered multi-response analytes. Such analytes and their associated methods require an integration routine encompassing an extremely wide retention time window. For these types of compounds manual integration is often required with each chromatographic run as the software may be incapable of making these types of integration decisions. Therefore, the documentation of manual integrations with before and after chromatograms and the justification of those manul integrations are not required; rather good chromatographic judgment is required by the analyst.

4.5 The Number of Times an Analyte may be Integrated and the Associated Documentation

4.5.1 Manual integrations should only be integrated one time. However, occasionally a second manual integration is required. These types of integrations are documented with the normal description justifying the reason for the manual integration.

4.5.2 When more than two manual integrations are performed on any one analyte it must be supported with a description of why three integrations were required in addition to the primary reason for the manual integration to begin with.

4.5.3 If more than three manual integrations are warranted, the integration must be initialed by the analysts direct supervisor.

4.6 State and/or Project Specific Manual Integration Documentation Requirements

4.6.1 Manual integrations of all data in support of programs which require manual

integration documentation must be performed in such a manner as to produce a complete audit trail. Audit trails are most easily produced by printing a "before" and "after" chromatogram and initialing and dating the printouts.

4.7 In summary, there are two situations which may trigger the printing of a before and after chromatogram when manual integrations have been performed and they are:

a) initial calibrations; and

b) sample and batch QC data.

In addition, there may be three situations which trigger the documentation of these chromatograms to produce a complete audit trail and they are:

a) initial calibrations;

b) sample and batch QC data; and,

c) when more than one manual integration has been performed on a single analyte.

4.8 Documenting Manual Integrations using Standardized Peak Nomenclature

Reasons to perform manual integrations are documented using the mnemonic scheme described as follows:

Mce	**M**anual integration due to **c**o-**e**lution (target and/or non-target analyte)
Mpnfi	**M**anual integration **p**eak **n**ot **f**ully **i**ntegrated (typically due to peak tailing, small peaks near the baseline, etc.).
Mpoi	**M**anual integration **p**eak **o**ver **i**ntegrated (typically due to negative peak, or a dip in the baseline, etc.)
Mmp	**M**anual integration **m**issed **p**eak or peak simply not integrated.
Mipi	**M**anual integration **i**ncorrect **p**eak **i**ntegration.
Mrts	**M**anual integration **r**etention **t**ime **s**hift
Mcsm	**M**anual integration **c**omplex **s**ample **m**atrix
Mcd	**M**anual integration **c**hromatographic **d**egradation (creates a situation where the software routine, instrument needs to undergo maintenance).
Mad	**M**anual integration **a**nalyst **d**iscrection

5.0 Chromatography Guidelines and Evaluation Procedures

5.1 Peak Definition

A peak is defined as a pictorial representation of the response of the compound where the response of the compound is at least three times the background noise. Background noise can be from the instrument electronics, solvent, sample matrix, mobile phase, gases, column bleed, etc.

5.2 Peak Shape

5.2.1 Ideal peak shape is a bell curve or Gaussian peak shape. The perfect peak should be symmetrical with a round top. The compound signal should take a few seconds to rise and fall. The rise and fall of a signal too quickly would generally be indicative of a noise spike.

5.2.2 Due to detectors, matrix and compound polarity, peaks may become broadened, have shoulders or tailing. These are all acceptable as long as it is not indicative of co-elution. Some methods have specific criteria as to how much of these imperfections are allowable. Judgement is necessary as to what is excessive. An analyst should base the judgement of current chromatography on historical instrument performance.

5.3 Baseline Resolution

5.3.1 The best case chromatography is an ideally shaped peak residing on a flat baseline completely resolved from any other peak or interference. Baseline integration is a line drawn from each endpoint of the peak, baseline to baseline, including the entire area under the curve of the peak (Figure E.9-1). This is appropriate when the quantification will be based on peak area.

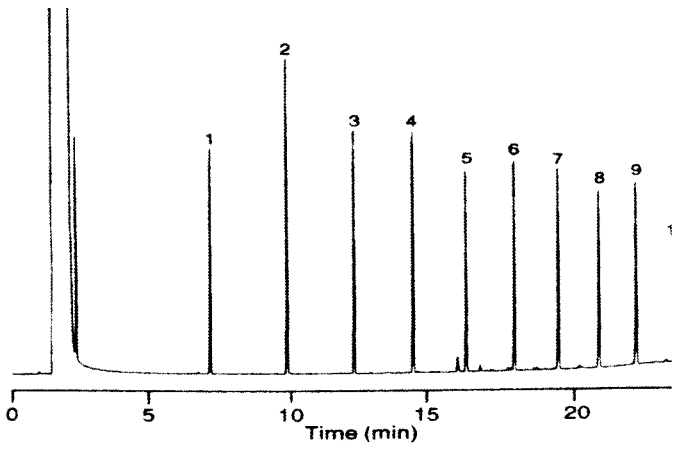


Figure E.9-1

6.0 Integration Techniques

6.1 Irregular Peak Shape

When there is an irregular peak shape first ensure that it is not excessive and indicative of an instrument problem or column overload. Then ensure this is not due to any co-elution problems from either target compounds or the matrix. If neither, one of the above, is the reason for the irregular peak shape, then the general guideline is to integrate the entire peak (Figure E.9-2).

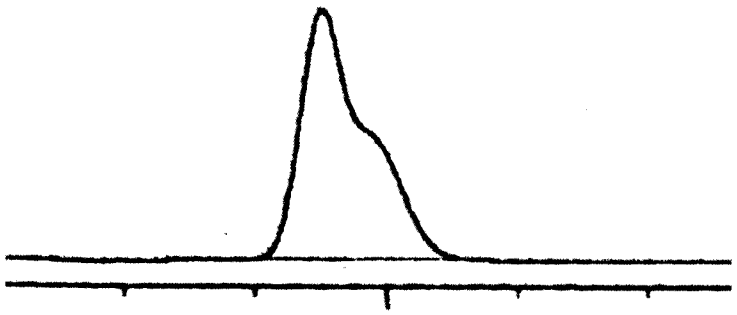


Figure E.9-2

6.2 Less than 100% Baseline Resolution

6.2.1 Dropping a Perpendicular

This integration technique is used when two compounds partially co-elute and there is a discernable valley between the two peaks. A perpendicular line is drawn from the valley between the two peaks to the baseline.

This technique assumes that both compounds have symmetrical peak shape and equal detector response, such as in the case of isomeric pairs. If this assumption is correct, then the area contributed from one compound as compared to the other compound will be the same and the quantification will be equivalent to 100% baseline resolved. Usually this is not the case, which means the detector has different sensitivity, peak shape and amount of resolution or coelution must be carefully evaluated before using this technique.

Several methods state that acceptable resolution is achieved for structural isomers if the height of the valley between the two isomeric peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as an isomeric pair. The method formula for acceptable resolution is V< 0.25(P1+ P2). Where V is the height of the valley between the peaks; P1 is the left peak and P2 is the right Peak (Figure E.9-3).

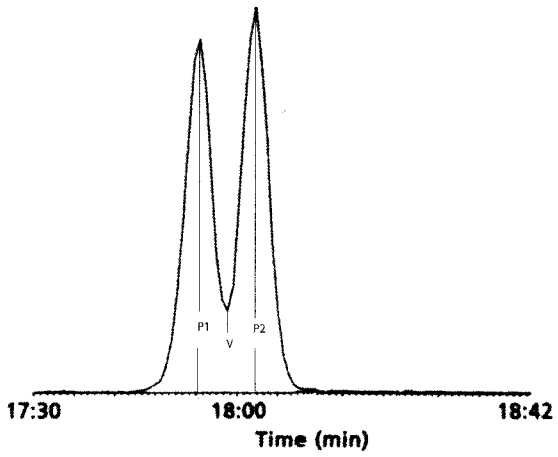


Figure E.9-3.

A second approach used by many methods in determining adequate resolution between two peaks is defined by the equation:

$$R = \frac{t}{w}$$

where t is the difference in elution times between the two peaks and w is the average peak width, at the baseline of the two peaks. Resolution is adequately resolved when R> 1.0 using this formula (Figure E.9-4).

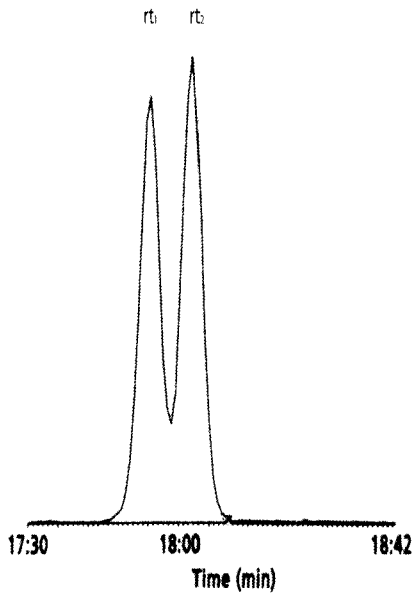


Figure E.9-4

6.2.2 Structural Isomers

While structural isomers represent the best conditions under which to integrate peaks by dropping a perpendicular, this criteria can be applied to all coeluting peaks. If in the analyst's judgement adequate resolution cannot be achieved than this technique should not be used. As an alternative, either integrate the two peaks together and report them with a footnote as coeluting peaks or do further method development to achieve better separation.

6.3 In GC/MS methods where chromatograms of a single quantitation ion are used, more unique ions that have adequate intensity should be considered for cases of close compound elution.

6.4 Valley to Valley and Baseline to Valley Integration

6.4.1 The valley to valley technique involves drawing the integration line from the valley between two peaks to another valley between two peaks.

6.4.2 Baseline to valley integration is drawing the line from the baseline at one end of the peak to the valley between two peaks at the other end of the peak being integrated.

6.4.3 The weakness of these two techniques occurs with the quantitation of samples that have a calibration produced where peak integration was produced on a flat baseline as compared to samples which do not exhibit the same flat

baseline attribute, and thus may under quantitate or over quantitate actual sample concentration.

The following examples attempt to explain the weakness of this integration technique.

6.4.3.1 Two Peaks with similar and non-similar response in the initial calibration

6.4.3.1.1 Two peaks have similar detector response such that standards would be integrated by dropping a perpendicular where less than 100% baseline resolution occurs (Figure E.9-5).

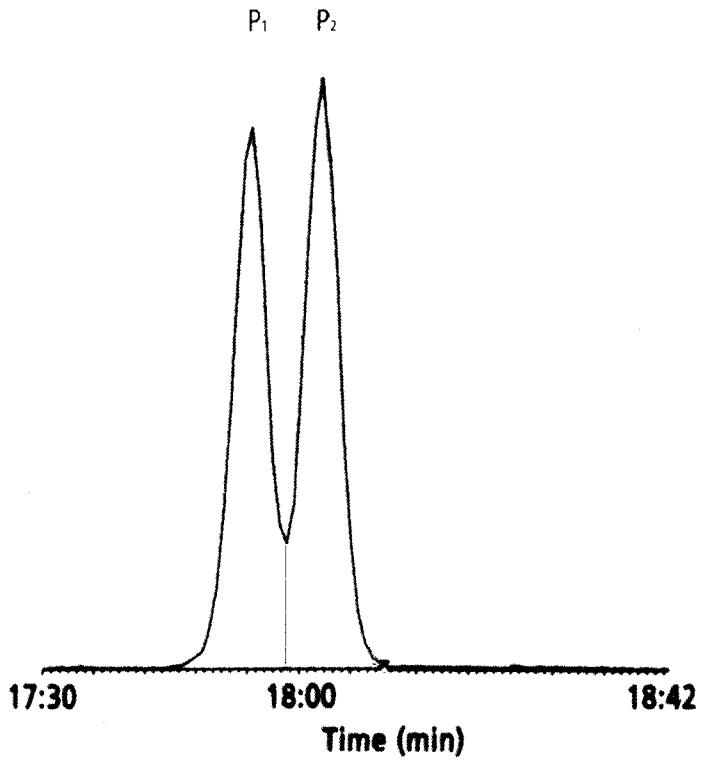


Figure E.9-5.

6.4.3.1.2 In this situation peak #1 (P1) has a greater signal intensity or response than peak #2 (P2) (Figure E.9-6.).

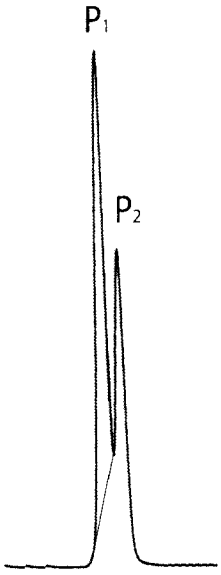


Figure E.9-6

6.4.3.1.3 If P1 is integrated baseline to valley, then P1 will be under quantitated and P2 will be over quantitated. The percentage error associated with P1 is less than the error associated with P2. This may be significant if P2 is at or slightly above the reporting limit where the error could be as much as or greater than 100% or a factor of 2 of the actual concentration. It may be appropriate to drop a vertical (Figure E.9-7) or to integrate valley to baseline (Figure E.9-8).

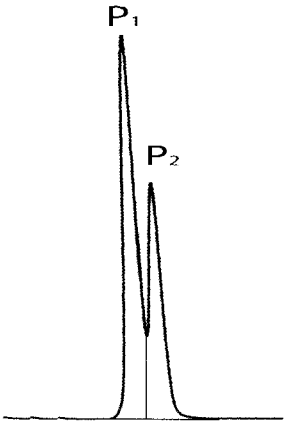


Figure E.9-7.

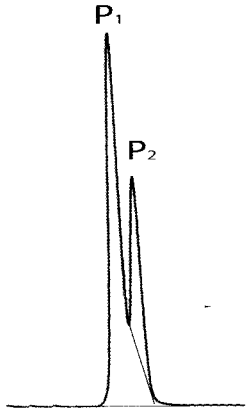


Figure E.9-8

6.4.3.1.4 The weakness of integrating valley to baseline (Figure E.9-8) are the same problems as discussed in the following section, peak skimming; namely, excluding area from P2 thought to be contributed by the larger peak. Normally peak skimming is not a recommended practice since peak tailing is very inconsistent. Therefore, it may be most appropriate to drop a vertical line and integrate.

6.4.3.1.5 Two peaks have similar detector responses such that standards would be integrated by dropping a perpendicular where less than 100% resolution occurs. The same situation as indicated in Figure E.9-5.

6.4.3.1.6 In this situation P2 has a greater signal response than P1 (Figure E.9-9).

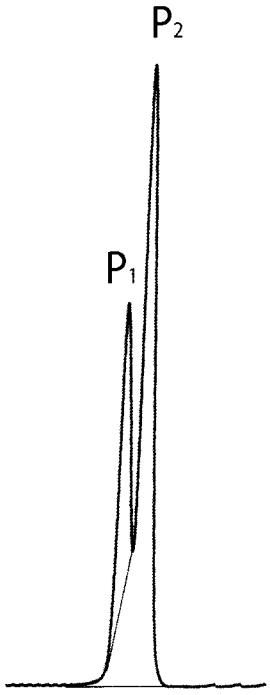


Figure E.9-9

6.4.3.1.7 If P1 is integrated baseline to valley (Figure E.9-9), then the same problems associated with Figure E.9-8 exists. Area is being excluded from P1 thought to be contributed by P2. Secondly the error associated with P1 will be significant if it is at or slightly above the reporting limit.

6.4.3.1.8 If P1 is integrated valley to baseline (Figure E.9-10) then P1 will be over quanitated and P2 will be under quantitated and it may be more appropriate to drop a vertical integration line.

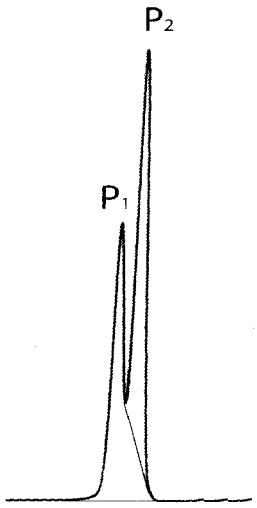


Figure E.9-10

6.4.3.2 Two Peaks with dissimilar response in the initial calibration

6.4.3.2.1 The two peaks have widely differing detector responses such that the standards would be integrated by dropping a perpendicular where less than 100% baseline resolution occurs (Figure E.9-11.).

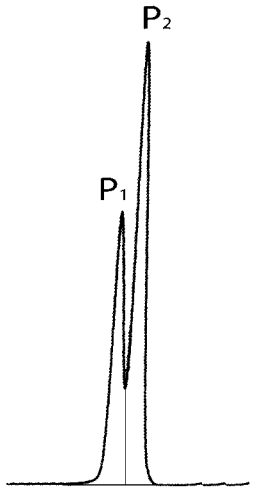


Figure E.9-11.

6.4.3.2.2 The integration scenarios detailed in example #1 and example #2 also apply to this situation. It is up to the analysts best judgment in determining the best integration techniques in order to minimize the potential quantitation errors.

6.4.3.3 The two peaks have similar or widely varying detector responses such that the standard would be integrated by dropping a perpendicular where less than 100% baseline resolution occurs.

In this situation either P1 or P2 as integrated in the standard is not found in the sample. Regardless of integration techniques used the target analyte will be under quantitated due to the area in the standard that was less than 100% baseline resolved. In this situation it is important to understand the significance of the error associated with the situation and overall impact of the data.

6.4.4 Because of these limitations, valley to valley and baseline to valley integration techniques are recommended only for instances where the baseline is shifting or matrix interferences are inherent in the analysis.

6.4.5 To help determine where the baseline is in a calibration standard, analyze a method blank. This should give a good indication of how the baseline is shifting throughout the run due to inherent system characteristics. When the calibration standards are analyzed, draw the baseline in the same general shape and slope as it appears on the method blank. Do not forget to take into account the additional baseline rise or shifting that the compound is causing.

6.5 Peak Skimming

Peak skimming is used when one peak elutes on the tail of another, larger peak (Figure E.9-12). Generally, the analyst would draw the integration line that defines where and how long the tail of the larger peak is. This excludes area from the smaller peak thought to be contributed by the larger peak. Normally peak skimming is not a recommended practice since peak tailing is very inconsistent. The analyst should try to achieve better resolution; however, this is not always practical.

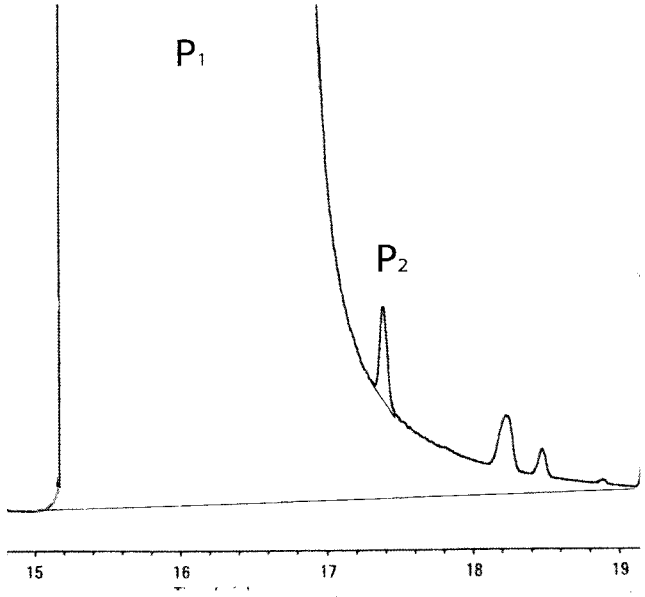


Figure E.9-12.

7.0 Inappropriate and Unacceptable Integration Practices and Techniques

7.1 The following scenarios constitute an ethics and fraud violation when integrating analytical data:

a) No manual integration will be performed exclusively on QC samples in order to meet QC acceptance criteria. Manual integration is acceptable as long as consistency rules are applied to all standards and samples, and there is a reason why it is necessary.

b) Under no circumstances is peak shaving allowed for the sole purpose for obtaining QC acceptance.

7.2 Peak Shaving

Peak shaving is integrating in a manner to exclude part of the peak response. Omitting portions of a peak or drawing the integration further into the peak is an unacceptable practice.

7.3 Adding Area Under the Baseline

This involves creating a peak from area under the baseline or adding to the response of a peak by taking additional area from under the baseline. In analysis where system behavior or matrix effects make it difficult to determine where the baseline is, some discretion is to be used to avoid adding or subtracting area which is not due to the compound's response.

7.4 Inadequate Resolution

7.4.1 Adding area from another peak often occurs with partial coeluting compounds which exhibit poor resolution. Even if the analyst knows a compound is present, as in the case of standards, and the irregular peak shape is due to this compound, the analyst should not artificially create a separation between two peaks which do not have adequate resolution.

7.4.2 As mentioned earlier, either integrate the two peaks together and report them as coeluting peaks or perform additional method development to achieve better baseline resolution.

7.4.3 Samples which exhibit matrix interference may produce target analytes with irregular peak shapes produced by the presence of non-target analytes. In this case, either integrate the compound and its' interferent together, or if a discernable valley exists or shoulder exists, drop a perpendicular.

7.5 Negative Spikes or Peaks

7.5.1 A negative spike or peak occurs when matrix interferences cause a sudden drop then increases in the baseline. In general the analyst should improve chromatography so that all target compounds are resolved from the negative spike. For some analyses this is very difficult. This problem should be corrected by getting the analyte to move away from this region then integrate the peak normally. If this occurs in a sample but not in the standards, integrate as best as possible.

Laboratory Ethics/Fraud Prevention Program for Manual Integration

The following scenarios constitute an ethics and fraud violation when integrating analytical data:

a) No manual integration will be performed exclusively on QC samples in order to meet QC acceptance criteria. Manual integration is acceptable as long as consistency rules are applied to all standards and samples, and there is a reason why the manual integration is necessary.

b) Under no circumstance is peak shaving allowed for the sole purpose for obtaining QC acceptance.

c) Under no circumstance may an analyst add area under the baseline of a peak, in an effort to create additional peak area.

These are specific examples of fraudulent and unethical manual integration practices, and are not intended to be a comprehensive list; rather this list is intended to give the analyst an idea of the types of unethical behavior that will not be tolerated.

I the undersigned, CERTIFY, that:

I have read, acknowledged and understand the personal ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions regarding manual integrations.

Employee Name (Print)	Signature	Date

Appendix E

Standard Operating Procedure

SOP E.10
Preparation of Reagent Grade Water

1.0 Preparation of Reagent Grade Water

1.1 The preparation of reagent grade water is an essential element in the success of our laboratory. Reagent grade water is used in many critical and non-critical areas of the laboratory during the production and analysis of environmental samples. The use of reagent grade water in the critical areas of the laboratories requires this water to be of such quality as to not interfere with the analysis of target analytes at the method reporting limits.

1.2 References

1.2.1 Method 1080 (Reagent Water): Standard Methods for the Examination of Water and Wastewater, 20th edition, 1998.

1.2.2 American Society for Testing and Material, ASTM, Volume 11.01, Section DH93-91, 1992.

2.0 Standard Operating Procedure

2.1 Introduction

2.1.1 One of the most important aspects of chemical and biological analysis is the preparation of reagent grade water. The quality of water required is related directly to the analysis being made. Reagent grade water is used for several critical areas in the final production of analytical data. Some of these critical areas include such things as:

a) dilution of chemical reagents;

b) dilution of samples requiring direct analysis;

c) analytical instrument rinse water; and

d) preparation of Quality Control samples such as:

- method blanks,
- laboratory control samples,
- trip blanks,
- field blanks,
- equipment/rinsate blanks, etc.

2.1.2 ASTM Type I ultra-pure water is produced by purifying tap water using a series of water clean-up procedures. Tap water is first treated through a reverse osmosis system; the water is subsequently stored in a 100 L reservoir and finished by treating this water with a deionization unit.

2.1.3 The RO-pure Infinity reverse osmosis system is a fully automated system providing up to 600 liters per day of ASTM Type I water when used in combination with the NANO-pure Infinity deionization unit.

2.2 Definition

2.2.1 Adsorption - a process used to remove chlorine and organic impurities. It is accomplished typically with granular activated carbon. In general, organic adsorption efficiency is inversely proportional to solubility and may be inadequate for the removal of low-molecular-weight, polar compounds.

2.2.2 ASTM Type Water - water that has the following water quality characteristics:

	Type I	**Type II**	**Type III**	**Type IV**
Electrical conductivity, Max *u*s/cm at 25°C	0.056	1.0	0.25	5.0
Electrical resistivity, Min MΩ at 25°C	18.0	1.0	4.0	0.2
pH at 25°C	*	*	*	5.8-8.0
Total Organic Carbon (TOC) Max, ug/L	100	50	200	no limit
Sodium, Max, ug/L	1	5	10	50
Chlorides, Max, ug/L	1	5	10	50
Total silica, Max, ug/L	3	3	500	no limit

Note: * The measurement of pH in Type I, II and III reagent water has been eliminated from this specification because these grades of water do not contain constituents in sufficient quantity to significantly alter the pH.

2.2.3 Ion exchange - a process in which water is prepared by passing feed-water through a mixed-bed ion exchanger, consisting of a strong anion and strong cation resins mixed together.

2.2.4 Reagent water - water with no detectable concentrations of the compounds or elements to be analyzed at the detection limit (typically established at one-half of the reporting limit) of the method of analysis and free of substances that may interfere with those analytical methods.

2.2.5 Reverse osmosis - a process in which water is forced under pressure through a semipermeable membrane removing a portion of dissolved constituents and suspended impurities.

2.3 System Description

2.3.1 Reverse Osmosis

2.3.1.1 The ROpure Infinity reverse osmosis system uses a thin micro-porous surface that rejects impurities, but allows water to pass through. The membrane rejects the following:

Constituent	Rejection Rate
• Monovalent ions	90-95%
• Polyvalent Ions	95-99%
• Inorganic solids	85-95%
• Organic solids	>99% (with a mol. weight > 300)
• Dissolved gasses	0%
• Microorganisms	>99% (bacteria and viruses)

This system is capable of producing up to 30 liters per hour of reagent grade water. This system is also microprocessor controlled which can allow automatic operation up to 24 hours a day. The RO membranes are automatically flushed for 10 minutes every 4 hours, eliminating contaminant buildup on the membranes.

2.3.1.2 Reject Water

A large percentage (50-98%) of the feed water does not pass through the membrane but flows across the membrane surface, constantly cleaning the membrane and carrying the inorganic and organic solids to drain.

2.3.1.3 Feed Water Pressure

The purity of the product water depends on the purity of the feed water. During the reverse osmosis process feed water pressure affects both the quantity and purity of reverse osmosis product water. Lower feed water pressure causes lower product flow rate and lower product purity. Feed water pressure should operate in the range of 30 - 100 psi.

2.3.1.4 RO Pretreatment System

The RO-Pure Infinity uses a thin film composite membrane to produce reagent grade water. The thin film membrane may be damaged by free chlorine; therefore, a pretreatment system using activated carbon is used to remove the free chlorine.

2.3.1.5 Feed Water Temperature

RO membrane performance is based on feed water temperature of 25°C (77°F). For every 1°C below 25°C product water quantity is reduced 3%. Therefore, a mixing valve (hot and cold) is used to maximize water production at a feed water temperature of 25°C/77°F.

2.3.2 Storage Reservoir

2.3.1 The reverse osmosis system requires storage to maintain back pressure on the RO membrane and also provides a large quantity of water on demand. This system uses a 100 L reservoir with an automatic shut-off float valve designed for automatic hands free water production.

2.3.3 Deionization

2.3.3.1 The NANO-pure Infinity system is designed to deliver up to 1.5 L/min of deionized water with resistivities of up to 18.3 MΩ/cm and less then 1 ppb organics. This system uses 4 distinct steps to produce final product water. They are as follows:

a) a pretreatment adsorption cartridge,
b) a mixed bed deionization cartridge,
c) a finish grade organic adsorption cartridge, and
d) a final membrane filtration.

2.3.3.2 Pre-deionization Adsorption Cartridge

An pre-deionization adsorption cartridge made of activated carbon on a unique macroreticular resin is used to remove organics, chlorine, colloids, and some bacteria from the feed-water. This activated carbon cartridge is used to extend the life of the deionization cartridge. The use of two different carbons promotes the removal of both large molecular weight and smaller volatile organics, providing for lower overall TOC values in the final product water.

2.3.3.3 Mixed Bed Deionization Cartridge

Ions are removed from water as the water passes through the ion exchange resin beds. In the generated form, cation resin contains hydrogen ions on its surface which are exchanged for positively charged ions. Anion resin contains hydroxide ions on its surface which are exchanged with negatively charged ions. The final product of these two exchanges form water molecules.

2.3.3.4 Membrane Filtration

Final product water is filtered using an in-line 0.2 micron membrane filter to remove bacteria or particles that may have passed through the other cartridges.

2.4 Cartridge and Membrane Installation

2.4.1 RO-pure Infinity Reverse Osmosis System

2.4.1.1 To replace the cartridges or membranes first disconnect the unit from the power supply. Open the front door and disengage the cartridge hold-down bracket by pulling it out and up.

2.4.1.2 Prefilter Installation

The prefilter is installed to remove particulates from the feed-water which could cause damage to the RO membrane. Install the prefilter cartridge as follows:

2.4.1.2.1 Remove a new prefilter (PN D9004) and wet the o-rings on both end caps.

2.4.1.2.2 Insert the upper end cap into the upper farthest left position of the two cartridge end cap sockets until it bottoms out in the connector.

2.4.1.2.3 Lower the prefilter and insert the lower end cap into the lower socket until it is firmly seated.

2.4.1.3 Pretreatment Carbon Cartridge

The pretreatment carbon cartridge is designed to remove chlorine from the feed-water which could cause damage to the RO membrane. Install the pretreatment carbon cartridge as follows:

2.4.1.3.1 Remove a new pretreatment carbon cartridge (PN D9005) and wet the o-rings on both end caps.

2.4.1.3.2 Insert the upper end cap into the upper position immediately right of the prefilter until it bottoms out in the connector.

2.4.1.3.3 Lower the carbon cartridge and insert the lower end cap into the lower socket until it is firmly seated.

2.4.1.3.4 With both the prefilter and pretreatment carbon cartridges installed, reposition the cartridge hold-down bracket.

2.4.1.4 Membrane Protection System (MPS) Installation

The MPS is a clear plastic bag containing organophosphate powder. This powder is combined with water and pumped through the RO system to eliminate scale buildup on the membrane surface. After feed-water flows through the prefilter and pretreatment carbon cartridges, a pump injects 4-8 ppm of the powder-water mix into the water flowing through the unit before it reaches the RO membranes. Install the MPS cartridge as follows:

2.4.1.4.1 Remove the lid on the new MPS cartridge (PN CM900X1) and add approximately 1L of deioninzed water and agitate.

2.4.1.4.2 Close the lid, and locate and reattach the MPS bag using the quick disconnect fitting.

2.4.1.4.3 Carefully place the bag upside down in the MPS holder.

2.4.1.5 Membrane Installation

This is a 2 membrane system used for the production of reverse-osmosis water. The membranes are installed as follows:

2.4.1.5.1 Place the first membrane cartridge in the right rear of the system cabinet. Ensure that the center connector (product water) points to the left and the offset connector (reject water) points to the rear of the unit.

2.4.1.5.2 Locate the tubing labeled Product and attach to the appropriate connector. Locate the Reject tubing and attach this tubing to the Reject connector.

2.4.1.5.3 Place the second membrane in the front of the fist membrane and install as mentioned above.

2.4.2 NANO-pure Infinity Deionization System

2.4.2.1 To replace the cartridges first disconnect the unit from the power

supply. Open the front door and disengage the cartridge hold-down bracket by pulling it out and up.

2.4.2.2 Cartridge Installation

2.4.2.2.1 Install the cartridges in the following order from left to right:

Position 1)	Pretreatment	PN D50251
Position 2)	Ultrapure	PN D50253
Position 3)	Ultrapure	PN D50253
Position 4)	Organic Free	PN D50252

A kit consisting of all four cartridges can be purchased using part number PN D50254.

2.4.2.2.2 Wet the o-rings with water on both cartridge nipples.

2.4.2.2.3 Install the appropriate cartridge in the correct position by pressing the top cartridge end cap into the upper socket until it bottoms out.

2.4.2.2.4 Lower the cartridge and insert the lower end cap into the lower socket until it is firmly seated.

2.4.2.2.5 Replace the cartridge hold-down bracket.

2.5 System Operation

For complete system operation see the operating manual.

Appendix E

Standard Operating Procedure

SOP E.11
Sub-sampling and Sample Compositing Procedure

1.0 Sub-sampling and Sample Compositing Procedures

1.1 When samples are received in greater volumes or numbers than are required for final analyses, laboratory sample mass reduction is necessary. There are two primary approaches for preparing samples for analysis: 1) sub-sampling the original discrete sample in order to prepare the sample for final analysis; or 2) compositing multiple discrete samples by sub-sampling each individual sample and physically combing the individual sub-samples into a single final composite sample to be analyzed. This procedure describes the steps necessary to sub-sample or to composite discrete individual samples to ensure a representative sample is obtained for analysis and to ensure the procedure was properly documented.

2.0 References

2.1 Method EPA/600/R-03/027: Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples, USEPA, Office of Research and Development, November, 2003.

2.2 Method ASTM D 6323- 98: Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities, ASTM Standards, Vol II.04, 1998.

3.0 Standard Operating Procedure

3.1 Introduction

3.1.1 Most analytical methods require a sample preparation procedure prior to the final analysis. These sample preparation procedures sometimes require the extraction chemist to sub-sample (i.e. take a smaller aliquot) the original grab sample. When sub-sampling is necessary, the goal is to obtain a representative sample as described in the references above.

To avoid preparing a non-representative sample, it is not appropriate to target a specific weight during the sample preparation procedure (e.g., it is inappropriate to manipulate the sample material so the sample aliquot weighs exactly 1.00 g ± 0.1g). However, each method has an analytical accuracy that can be determined at its reporting limit that may be used when preparing sub-sample aliquots. That is to say, if a method has a reporting limit of 10 mg/Kg and the sub-sample can be taken such that 10 g ± 0.4 g will produce the same data results, then an aliquot of 9.6 to 10.4 grams can be recorded as 10 grams.

3.1.2 As these documents note, representativeness is a matter of scale. Fortunately for water and virtually all soil samples received for environmental analysis, the sample collection bottles, and those containers used for sub-sampling devices greatly exceed the minimum interior diameter of 3 times the largest particles, once extraneous material has been removed from the samples.

3.1.3 For soil samples, multi-phase samples and samples containing extraneous material, sub-sampling can be the major source of error in the measurement process, so great care must be used in applying these procedures to obtain a representative sample.

3.2 Multi-phase Samples

3.2.1 Inspect the sample and determine if the sample consists of a single matrix or if multiple matrices are encapsulated in the single discrete grab sample.

3.2.2 It is Alpha's policy to analyze and report a single discrete sample per matrix and to not prepare and analyze multi-phase samples as a single discrete sample. Because each matrix must be analyzed separately, a determination of which matrix or matrices to be analyzed must be made. If multiple matrices are included perform the following procedure:

- Scrutinize the chain-of-custody and determine if the client has provided further guidance;
- If no guidance is given on the COC, the client should be called to clarify exactly which matrix or matrices the client wishes to have analyzed. This information must be documented in the client file; and,
- If a particular matrix is requested, the sample is decanted into discrete containers isolating the various matrices and the requested matrix is stored for final sample preparation.

3.3 Water Sub-sampling Procedures (excluding VOCs)

3.3.1 Discrete water samples are typically collected in 1-L glass or plastic containers. Samples are visually inspected to determine if there are extraneous materials that should not be included as an integral part of the sample matrix. Extraneous materials may include such things as twigs, leaves, worms, etc., which obviously are not an integral part of the matrix to be extracted/digested and analyzed. The extraneous material is removed from samples before sub-sampling and preparation for analysis.

3.3.2 Inspect the sample and determine if filtering is required. Colloidal suspensions typically associated with high dissolved solids often times interfere with the organic solvent extraction procedures and may need to be filtered prior to the extraction. Refer to the particular analytical and sample preparation procedures for specific filtering requirements. The only universal exception which prohibits the filtering of any sort is oil and grease, sulfides, and ammonia analysis.

3.3.3 Once the sample matrix has been inspected, and prepared, the entire grab sample should be vigorously shaken in an effort to completely homogenize the sample constituents.

3.3.4 The sample is now ready for sub-sampling. This is most easily accomplished by decanting the required volume in a volumetric glass cylinder.

3.3.4.1 Once samples have been sub-sampled into a secondary container, they must never be poured back into the original sampling container, preventing a possibility of contamination.

3.3.4.2 If samples have been decanted back into their original container, they should be considered compromised and not used in any further preparation or analysis.

3.3.4.3 Sample volumes are then recorded and the sample preparation procedure may commence.

3.4 Soil Sub-sampling Procedures (excluding VOCs)

3.4.1 Discrete soil samples are typically collected in 250 ml wide mouth glass containers or occasionally in brass sleeves. Samples are visually inspected to determine if there are extraneous material that should not be included as an integral part to the sample matrix. Extraneous material may include such things as twigs, leaves, rocks, etc which obviously are not an integral part of the matrix to be extracted/digested and analyzed. The extraneous material is removed from samples before sub-sampling and preparation for analysis.

3.4.2 Inspect the sample and determine if the sample consists of a single solid matrix or if the matrix is a sludge.

3.4.3 Sludge Sub-sampling Procedures

It is Alpha's policy to analyze and report a single discrete sample matrix and to not prepare, analyze and report a multi-phase sample as a single discrete sample.

3.4.3.1 Inspect the sample and determine if the sample consists of a single matrix or if multiple matrices are encapsulated in the single discrete grab sample.

3.4.3.2 If the sample is defined as a sludge (i.e. solid matrix that has the viscosity properties of a liquid), then the following procedures are followed:

- Scrutinize the chain-of-custody and determine if the client has provided further guidance;
- If no guidance is given on the COC, the client should be called to clarify exactly which matrix or matrices the client wishes to have analyzed. This information must be documented in the client file; and,
- If the supernatant is requested, the liquid matrix is decanted and isolated for final sample preparation.
- If the sludge is requested, the supernatant is decanted from the sludge and the sludge is isolated for final sample preparation.

3.4.4 Sub-sampling Wide Mouth Sample Containers

3.4.4.1 Once the sample matrix has been inspected, and isolated, the entire grab sample should be stirred with a clean, non-reactive spatula, or glass stirring rod, in an effort to completely homogenize the sample constituents. Samples should be homogenized until the texture and color appear to be uniform.

3.4.4.2 Sub-sampling is most easily accomplished by scooping, with a clean spatula, a portion of the well-mixed solid matrix into a receiving container.

3.4.4.3 Once samples have been sub-sampled into a secondary container, they must never be placed back into the original sampling container, preventing a possibility of contamination.

3.4.4.4 If samples have been decanted back into their original container, they should be considered compromised and not used in any further preparation or analysis

3.4.4.5 Sample weights are then recorded and the sample preparation procedure may commence.

3.4.5 Sub-sampling Brass Sleeves

3.4.5.1 Sample collection using brass sleeves involves pounding the brass sleeve into the ground or collecting the sample from a split spoon sample coring device. This collection procedure prohibits the sample preparation person from easily extruding the sample from the brass sleeve without enormous effort or

possible contaminating the sample. Therefore, samples collected in brass sleeves typically cannot be completely homogenized prior to sample preparation but are sub-sampled and prepared as follows:

- Open the sample container and discard the first 0.25 to 0.5 inch of material. This material should not be used due to possible contamination during the sampling event and contact with the sample lid;
- Proceed with weighing the required sample amount needed to perform the extraction or digestion;
- If a higher degree of homogenization is required then weigh a portion of material approximately 2-3 times the amount required for sample preparation procedure;
- Perform this same procedure on the opposite side of the brass sleeve and combine the two sub-samples;
- Stir with a clean, non-reactive spatula, or glass stirring rod, in an effort to completely homogenize the sample constituents. Samples should be homogenized until the texture and color appear to be uniform; and
- Proceed with weighing the required sample amount needed to perform the extraction or digestion.

3.5 Sample Compositing Procedures

3.5.1 Compositing is a method of combining several samples of a specific sample matrix for a single chemical analysis. The single chemical analysis of a composite sample results in an averaging of the concentrations of its individual component samples.

3.5.2 Composite sampling and analysis can substantially reduce analytical costs by reducing the number of required analysis. A composite sample is typically produced by the physical mixing of individual samples and subsequently analyzing this as a single sample. By selecting the appropriate composite sample size and retesting individual samples, the composite sample may reveal the same information as would otherwise require many more analyses.

3.5.3 Prepare composite samples using equal volumes or weights of each single discrete sample. Sub-sampling of individual discrete samples should be completed as described below.

3.5.4 Mix the composite sample throughly by stirring with a clean, non-reactive spatula, or glass stirring rod, in an effort to completely homogenize the sample constituents. Samples should be homogenized until the texture and color appear to be uniform. For water samples, vigorously shake the final sample composite or stir to homogenize the resultant sample.

3.5.5 Ideally, when preparing a composite sample, each sub-sample comprising the composite sample should be of the same weight or volume. This would limit the error associated with the compositing procedure to the same final analytical error expressed on the final analytical report as significant figures.

Secondly, analytical methods of analysis vary greatly in significant figures and concentration units. Therefore, to decrease the systemic error produced through sample compositing a higher degree of homogenization is required.

Soil samples inherently have a larger degree of potential sampling error associated with them when compared to liquid samples due in most part to particle size distribution. This is most easily overcome by sub-sampling a larger portion of material, approximately 2-5 times the amount required for the sample preparation, thereby decreasing the overall error. Liquid samples are simply proportioned mathematically to the overall volume required.

There are no universal rules applied to the error margin involved with sample compositing due to the complexity of the overall analytical procedures etc. Therefore, a general guideline to minimize the potential sub-sampling error for soils would be to weigh a sample aliquot to ± 5% of the final target weight. Conversely a general guideline for liquids of ± 2% sub-sampling error should be used when preparing composite samples.

Note: Volumetric cylinders have a volumetric error of ± 2% (Class A) and are acceptable to use when preparing liquids for compositing with the exception of VOCs.

3.5.6 The single most important compositing rule is NEVER mix the entire discrete samples into a single composite sample. Sample weights and volumes will probably not be the same, thereby introducing biased analytical results, and secondly, no additional sample is left to analyze the individuals as discrete samples.

SVOC Soil Compositing Table
Targeting a Overall Weight
Two Times Extraction Requirement
Table E.11-1

Number of composite samples	Sub-sample aliquot (±5% error)	Target weight of individual sub-samples	Final overall Target weight	Weight required for analysis
5	3.8 - 4.2	4.0 g	20 g	10 g
4	4.8 - 5.3	5.0 g	20 g	10 g
3	6.3 - 7.0	6.7 g	20 g	10 g
2	9.5 - 10.5 g	10 g	20 g	10 g

SVOC Soil Compositing Table
Targeting a Overall Weight
Five Times Extraction Requirement
Table E.11-2

Number of composite samples	Sub-sample aliquot (±5% error)	Target weight of individual sub-samples	Final overall Target weight	Weight required for analysis
5	9.5 - 10.1	10.0 g	50 g	10 g
4	11.9 - 13.1	12.5 g	50 g	10 g
3	15.9 - 17.5	16.7 g	50 g	10 g
2	23.8 - 26.3	25 g	50 g	10 g

SVOC Water Compositing Table
Table E.11-3

Number of composite samples	Sub-sample aliquot (±2% error)	Target volume of individual sub-samples	Final overall Target volume	Volume required for analysis
5	98 - 102 ml	100 ml	500 ml	500 ml
4	122 - 128 ml	125 ml	500 ml	500 ml
3	163 - 170 ml	167 ml	500 ml	500 ml
2	245 - 255 ml	250 ml	500 ml	500 ml

3.6 VOC Preparation

The preparation of VOCs are of great concern when sub-sampling or preparing samples for compositing. The compositing of VOCs is not recommended because the compositing process itself provides a mechanism for the contaminants to escape into the atmosphere. Consequently, composite samples may underestimate the amount of VOCs actually present in the sample.

If VOC samples are to be sub-sampled or composited, then the following SOP is appropriate, with the following caveats:

3.6.1 Soil samples must not be mixed or shaken to homogenize the matrix at any point with the exception of shaking or vortexing the sample once it has been extracted with methanol.

3.6.2 VOC sub-sampling or compositing must take precedence over all other analysis, to minimize their potential loss into the atmosphere, as well as minimizing the potential for contamination by lab solvents which are also VOC analytes.

3.6.3 Once the sample container has been opened, speed is of paramount importance, DO NOT DELAY in any aspect of preparing the sample for final analysis. Because of the fragile nature of these compounds, samples should be subsampled mathematically to the overall volume or weight required for the analysis.

3.6.4 As discussed previously, there are no universal rules applied to the error margin involved with sample compositing due to the complexity of the overall analytical procedures etc. Therefore, a general guideline for soils of ± 5% sub-sampling error of the final weight should be used when preparing composite samples. Conversely a general guideline for VOC liquids of ± 2% sub-sampling error should be used when preparing composite samples.

VOC Soil Compositing Table
Table E.11-4

Number of composite samples	Sub-sample aliquot (±5 % error)	Target weight of individual sub-samples	Weight required for analysis
5	3.8 - 4.2	4.0 g	20 g
4	4.8 - 5.3	5.0 g	20 g
3	6.3 - 7.0	6.67g	20 g
2	9.5 - 10.5	10 g	20 g

VOC Water Compositing Table
Table E.11-5

Number of composite samples	Sub-sample aliquot (±2% error)	Target volume of individual sub-samples	Volume required for analysis
5	8.4 - 8.8	8.6 ml	43 ml
4	10.5 - 11.0	10.8 ml	43 ml
3	14.0 - 14.6	14.3 ml	43 ml
2	21.1 - 21.9	21.5 ml	43 ml

Appendix E

Standard Operating Procedure

SOP E.12
A Practical Application Guide for Performing Instrument Calibration, Calibration Model Determination and Calibration Software Verification

A Practical Application Guide for Performing Instrument Calibration, Calibration Model Determination and Calibration Software Validation

1.0 Initial Calibration (IC)

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

This document 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.

If more stringent standards or requirements are included in a mandated test method or by regulation, those procedures will take precedence to ensure those requirements are met. If it is not apparent which standard is more stringent, then the requirements of the regulation or mandated test method are followed.

1.1 Definition

1.1.1 Calibration - A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and corresponding values realized by standards.

a) In calibration of support equipment the values realized are established through the use of Reference Standards that are traceable to the International System of Units (SI), i.e., (ug/kg or ug/L etc).

b) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

1.1.2 Calibration Curve (Model) - The graphical relationship which represents a mathematical function between the known values, such as concentrations, of a series of calibration standards and the instrument response.

1.1.3 Calibration Method - A defined technical procedure for performing a calibration.

1.1.4 Calibration Range - The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals which used a single-point calibration, the low level calibration standard and

the high standard established during the linear dynamic range study defines the calibration range for this situation.

1.1.5 Calibration Standard - A substance or reference material used to calibrate an instrument.

1.2 Purpose - To establish a calibration curve for the quantification of the analytes of interest. The initial calibration standards/curve define the working range for the individual target analytes.

1.3 Frequency - In order to perform quantitative measurements, the relationship between the response of the instrument to a known sample analyte concentration must be established prior to sample analysis.

1.4 Procedure - Initial calibration procedures are performed using reference standards with known values for selected concentration points defining the initial calibration range. These initial calibration points are measured and an initial calibration is constructed using various mathematical function describing the calibration curve.

1.4.1 Calibration Curve Basic Construction

1.4.1.1 The most commonly constructed calibration curve used in analytical chemistry is a two variable calibration curve. The two variables are as follows:

a) Independent - the one we set (e.g. concentration - X axis); and

b) Dependent - the one we measure (e.g. response - Y axis).

In reality both variables are somewhat considered independent. In developing a calibration curve for sample quantitation, the primary goal is to develop a calibration model to relate the two variables.

1.4.1.2 The most common approach used to develop a calibration model is to:

a) prepare a series of known analyte standards;

b) hold all other factors constant (e.g., injection volume);

c) measure the response; and,

d) develop a calibration model (calibration curve).

1.4.1.3 It must be stressed that the analyte response after calibration may actually rely on a number of factors that may affect sample quantitation such as:

a) matrix,
b) interfering analytes,
c) random errors,
d) sample preparation, and
e) sample calibration.

Therefore, the relationship between the analyte and its response is a function of the entire method.

1.4.2 Calibration Range

1.4.2.1 All instruments have a range in which the relationship between the analyte response and the analyte concentration may be determined; however, for this relationship to be included into a calibration curve, method calibration technical criteria must be met for both calibration points and target analtyes.

1.4.2.2 For most methods the calibration range must satisfy the following minimum requirements as follows:

a) The lowest concentration calibration standard that is analyzed during an initial calibration establishes the lowest possible, non-qualified, method quantitation limit based on the final volume of the extract or sample.

b) The other concentrations should define the working range of the detector or should correspond to the expected range of the concentrations found in actual samples that are also in the working range of the detector.

c) For each analyte, at least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project, which may include establishing compliance with a regulatory or action limit.

d) Given the large number of analytes, and varying range of analyte linearity, it may be necessary to prepare more than one initial calibration. However, each initial calibration must be analyzed with the minimum method required calibration points and evaluated as separate calibrations.

1.4.2.3 When a calibration curve is constructed and the calibration range defined; there are several primary limiting points which help define both the calibration curve and the calibration range. They are as follows:

a) Limit of Quantitation (LOQ)- The minimum levels, concentrations, or quantities of a target analyte that can be reported with a specified degree of confidence.

- SW846 requires Reporting Limits (RL) to be established at or above the lowest calibration point in a calibration curve.
- The LOQ and RL are used by our laboratory synonymously.

1) Clarification: DoD clarifies this as the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ must be set at or above the concentration of the lowest initial calibration standard. (This is the same as NELAC).

b) Method Detection Limit (MDL) - 40 CFR Part 136 Appendix A states the following: "The Method Detection Limit (MDL) is 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 a statistical determination which may or may not be at a concentration that can be measured. This is not factored into the MDL study but is clearly mis-stated in the overall definition.
- Some regulatory agencies are beginning to recognize this fact by requiring laboratories to verify the calculated MDL.

  This is typically accomplished by analyzing a QC sample at two to four times the MDL (also known as the LOD or a DL study).

c) 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.

- In certain situations, this may be the same concentration as the statistically determined MDL, but not necessarily.

  Some regulators/auditors believe the LOD and the MDL may be used interchangeably. This is not true as the process of determining an MDL and an LOD are quite different. None-the-less they are closely related.

- The LOD is typically a statistically determined concentration point most often below the lowest calibration point and in this case would not be included in the calibration curve and/or calibration range.

- If the MDL verification standard (also known as the LOD or DL study) has a signal to noise ratio greater than three, the MDL is recognized as valid. If not the MDL study needs to be re-conducted at a higher concentration.

2) Clarification: DoD clarifies this as the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

d) Range of Linearity (ROL)- The range of concentration points which define both the upper and lower boundaries which exhibit a proven linear relationship. The ROL is the range in which method analytes may be quantitated. Compounds outside of the ROL cannot be quantitated without further concentration or dilution or reporting with footnotes.

- SW846 would define this as the upper and lower calibration points in the established calibration curve, regardless of calibration model used for the initial calibration.

- Other methods, notably methods 300.0 and

200.8 would define this as a concentration point above which the linear relationship does not exist at a defined limit of acceptability.

For example, if an aluminum standard at a concentration of 1000 mg/L produced a quantitated value of 1256 mg/L it would not be considered linear at that particular concentration. The criteria used to establish calibration linearity is generally defined at ± 10%. Therefore, in the case cited above, the method would require aluminum to have a quantitated value no larger than 1100 mg/L to be acceptable.

14.2.4 In reviewing these limiting points of calibration, detection and reporting limit, it should become obvious that the calibration range which may be used to quantify and report data is defined by:

a) the lower limit as the LOQ/RL, and

b) the upper limit as the upper ROL.

This is stated in our analytical SOPs as follows:

Calibration standards must cover the working range of the instrument with the low level standard at or below the reporting limit. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration.

1.4.2.5 The extrapolation of the calibration to concentrations above or below those of the actual calibration standards is not appropriate and may lead to significant quantitative errors regardless of the calibration model chosen. Therefore, it may be necessary to prepare calibration standards that cover concentration ranges that are appropriate for specific projects or types of analyses.

1.4.3 Calibration Models

1.4.3.1 All calibration models must be continuous. A curve is continuous when it has consecutive numerical values along the function, whether increasing or decreasing without having breaks, i.e., standards cannot be randomly removed in the IC. In addition, the calibration model must also be monotonic, such that all tangent lines of the points on the

calibration curve have either only positive or negative slopes, i.e., parabolic curves are not allowed.

1.4.3.2 Types of Calibration Models

There are three primary models for calibration curves that can be considered for instrument calibration, Linear, Quadratic or non-linear and Power. For environmental analysis we are interested in only two of the three.

a) Linear $Y = a + bX + \varepsilon$

b) Quadratic $Y = a + bX + c(X^2) + \varepsilon$

Where:

Y = instrument response;
X = known concentration value of a reference standard;
a,b,c = coefficients to be determined; and
ε = a measurement error also known as the residual.

1.4.3.3 Special Calibration Cases

There may be a special case in which a multipoint calibration is not required, e.g., single-point-calibration, where the calibration is proven to exhibit a linear response. That is: a = 0, and b = 1.

1.4.3.4 Choosing A Calibration Model

The choice of choosing a specific calibration model should be made in one of two ways.

1.4.3.4.1 The first is to begin with the simplest approach, the linear model through zero, and progressing through other options until the calibration acceptance criteria are met.

1.4.3.4.2 The second approach is to use *priori knowledge* of the detector response to choose the calibration model.

3) Clarification: It is the intent of SW-846 to prioritize the use of IC mathematical models. The first is to begin with the simplest approach, the linear model through zero, and progressing through other options until the calibration acceptance criteria are met. However, it is also acceptable to directly use *priori knowledge* of the detector response to choose the calibration model.

1.4.3.5 Linear Calibrations Through Zero
(Average Response Factor)

1.4.3.5.1 The linear calibration model is widely applied to many instrument calibration procedures because it has several advantages over the more complicated quadratic model. A couple of these advantages are as follows:

a) the computation of coefficients and standard deviations are easy;

b) the correction for bias is easy;

c) there is often a theoretical basis for the model (i.e., a stoichmetric relationship exists between color intensity and concentration.

1.4.3.5.2 The linear calibration model equation or mathematical function is described as:

$$Y = a + bX + \varepsilon$$

and is often written as:

$$Y = mX + b$$

where:

Y = instrument response;
X = known value of a reference standard;
m = the slope of the curve; and
b = the y intercept.

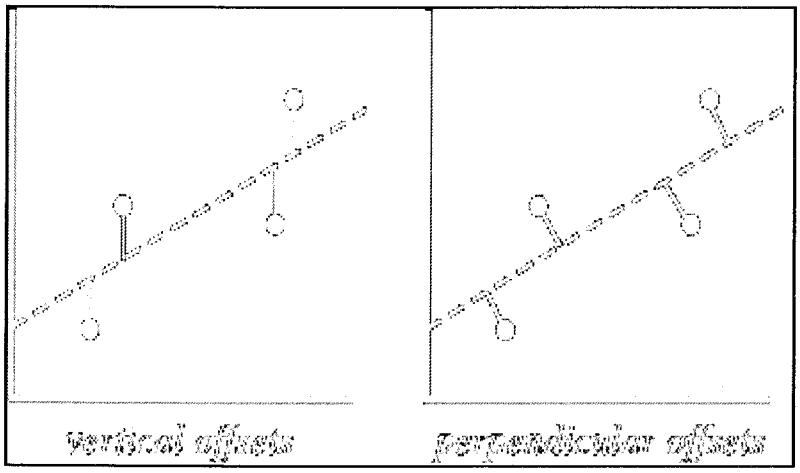
Therefore, if b = 0, the equation can be rewritten as:

$$Y = mX$$

In this case, the average calibration factor is used to construct a calibration curve.

1.4.3.6 Linear Calibration not Forced Through Zero
(Least Squared (LS) Regression)

1.4.3.6.1 Least squared regression analysis used for curve fitting is a mathematical procedure for finding the best-fitting curve to a given set of points by minimizing the sum of squares of the offsets or "residuals" of the points from the curve. The sum of the squares of the residuals is used instead of the residual absolute values because this allows the residuals to be treated as a differentiable quantity. However, because squares of the residuals are used, outlying points can have a disproportionate effect on the curve fit, a property which makes this mathematical model non desirable in some situations.



1.4.3.6.2 In practice, the vertical offsets from a line are almost always minimized instead of the perpendicular residuals. This provides the following advantages:

a) a fitting function for the independent variable X that estimates y for a given x;

b) allows uncertainties of the data points along the x and y axes to be incorporated more simply than a perpendicular residual;

c) allows for a much simpler analytical and mathematical form for the curve fitting parameters than would be obtained using a curve fit based on the perpendicular residual function; and,

d) most importantly, the vertical residual curve fitting technique is easily generalized from a

best fit least squared regression line to a best fit polynomial when the sums of vertical distances are used.

1.4.3.6.3 The linear model is most commonly written as:

$$Y = mX + b + \varepsilon$$

In this case b is not equal to 0 and ε or the residual becomes extremely important.

1.4.3.6.4 The linear calibration model is the most common straightforward method for instrument calibration, however, this calibration model cannot compensate for detector non-linearity. Therefore, the following assumptions are made when using the method of least squares:

a) for each fixed X-value the variable Y is normally distributed with a constant variance; and,

b) the n data points are independent of one another.

It must be noted that when the Y value is not normally distributed with a constant variance, then the mathematical function is describing a non-linear relationship. In this case, the least squared regression equation for calibration should not be used and a second or third order calibration model such as a quadratic equation should be considered.

1.4.3.7 Quadratic Calibration Models

14.3.7.1 The quadratic calibration model is written as:

$$Y = a + bX + c(X^2)$$

14.3.7.2 In this case, the concentration of unknown samples is calculated by solving the equation for X using the classical "quadratic formula", namely:

$$X = \frac{-b \pm SQRT(b^2-4*a*(c-Y))}{2a}$$

Where:

Y = instrument response; and

a,b,c = the three coefficients from the quadratic fit.

Note: Initial calibrations using quadratic curve fitting equations can only use the positive root of this function.

1.4.4 Calibration Mathematics

1.4.4.1 Calibration curve fitting mathematics can be greatly simplified by the use of linear algebra. It is not the intent to study or define the principles of this area of mathematics but to simply state that curve fitting for both least squared regression and quadratic regression equations can be solved for by the use of linear algebra.

1.4.4.2 Eigenvalues, eigenvectors, identity matrices, and inverted matrices can be used to solve both the least squared and the quadratic formulas. The use of linear algebra is used to produce a mathematical matrix array to solve for the coefficients simultaneously as the identity matrix or inverse matrix array as follows:

Matrix Algebra Calculation Array							
Mathematical Matrix Array to solve x(a), x(b) and (c) simultaneously							
A Matrix				X Matrix		C Matrix	
n	ΣX	ΣX^2	*	c	=	ΣY	
ΣX	ΣX^2	ΣX^3		b		ΣXY	
ΣX^2	ΣX^3	$\Sigma X4$		a		ΣX^2Y	

1.4.4.3 Weighting

1.4.4.3.1 The use of matrix algebra also makes possible the use of weighting linear and polynomial regression equations. However, when applying weighing factors to calibration curves, it is important to understand

what the influences of weight factors are as a weighted calibration curve is calculated. A weight factor influences the weight of every data point in the calibration curve in relation to its contribution to the sum of squares.

1.4.4.3.2 Typically, there are two common types of weighting routines used by most data acquisition and calibration routines, and they are:

a) inverse concentration, and
b) inverse squared concentration.

1.4.5 Calibration Methods

1.4.5.1 Initial calibration points for the individual target analytes are prepared and analyzed using a minimum of three points for 600 series organic methods and a minimum of five points for 8000 series organic methods covering the expected working range of the instrument and is used to construct a calibration curve.

1.4.5.2Once the calibration standards have been analyzed, a functional relationship is established between the values of the standards and the corresponding measurements. The two primary techniques used to perform initial calibrations are called internal and external calibrations.

An internal calibration procedure is used for GC/MS and ICP/MS methods of analysis and an external calibration procedure is generally used for most other methods of analysis.

1.4.5.3 External Calibration

The external standard calibration technique involves comparison of instrument responses form the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to the peak areas (or peak heights) of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF).

$$CF = \frac{\text{Peak Area (or Height) of the compound in the Standard}}{\text{Mass of the compound injected}}$$

1.4.5.4 Internal Calibration

The internal standard calibration technique involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection.

The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as the relative response factor in some methods.

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

Where:
A_s = Peak area of the analyte
A_{is} = Peak area of the internal standard
C_s = Concentration of the analyte in ug/L
C_{is} = Concentration of the internal standard in ug/L

1.5 Acceptance Criteria

The following general criteria are those taken from SW846 Method 8000, Organic Analysis to be used for general illustrative information only. The analyst must use the method criteria to evaluate their specific situation when performing an initial calibration which is described in detail in the individual analytical SOPs.

1.5.1 Linear Calibrations Through Zero
(Average RF/CF Calibration)

As described above, the calibration factor or response factor is a measure of the slope of the calibration curve and assumes that the curve passes through the origin. Under ideal conditions, the calibration factors will not vary with the concentration of the standard that is injected into the instrument. In practice, some variation is to be expected. This variation is measured as the relative standard deviation.

1.5.1.1 Relative Standard Deviation

If the RSD calculated from the RF/CF is generally less then or equal to 15% for GC/MS methods and less then or equal to 20% for GC methods, then the response of the instrument is considered linear and the average response factor can be used to determine sample results (e.g., linear forced through zero).

Linearity through zero is a statistical assumption and is not a rationale for reporting results below the calibration range demonstrated by the analysis of standards.

1.5.2 Linear Calibrations Not Forced Through Zero (Least Squared (LS) Regression)

1.5.2.1 Forcing The Origin

This calibration model will not allow the calibration line to be forced through the origin, i.e., do not set the intercept as 0, and do not include the origin (0,0) as a calibration point.

1.5.2.2 Relative Standard Deviation

If the RSD calculated from the RF/CF is generally greater then 15% for GC/MS methods or greater then 20% for GC methods, then linearity through the origin cannot be assumed.

Note: At the discretion of the analyst, this approach also may be used for analytes that do meet the minimum RSD limits where a average CF/RF model could be used.

1.5.2.3 Weighting

The regression calculations attempt to minimize the sum of squares, hence the name least squared regression.

Weighting the sum of the squares may significantly improve the ability of the least squared regression to fit the linear model of the data. The mathematics used in the least squared regression model has a tendency to favor numbers of larger values over numbers of smaller values. Thus the regression curves that are generated will tend to fit points that are at the upper calibration levels better than those points at the lower calibration. To compensate for this, a weighting factor which reduces this tendency can be used as described above.

1.5.2.4 Correlation Coefficient

The linear regression calculation will generate a weighted correlation coefficient (r) that is a measure of the "goodness of fit." A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99.

3) Clarification: DoD requires the correlation coefficient to be ≥ 0.995. The method and NELAP only require a (r) value of 0.99.

1.5.3 Non-Linear Calibration (Quadratic)

In situations where the instrument response does not follow a linear model over a sufficiently wide working range, or when the other calibration models have not met the acceptance criteria, a non-linear calibration model may be used.

Note: The option for non-linear calibration models may be necessary to achieve low detection limits or to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibrations to be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance.

1.5.3.1 Forcing The Origin

This calibration model will not allow the calibration line to be forced through the origin, i.e., do not set the intercept as 0, and do not include the origin (0,0) as a calibration point.

1.5.3.2 Relative Standard Deviation

If the RSD calculated from the RF/CF is generally greater then 15% for GC/MS methods or greater then 20% for GC methods, then linearity through the origin cannot be assumed.

Note: At the discretion of the analyst, this approach also may be used for analytes that do meet the minimum RSD limits where a average or a LS calibration model could be used.

1.5.3.3 Weighting

Weighting in this type of calibration model may significantly improve its accuracy.

1.5.3.4 Coefficient of Determination

The curve fitting mathematics uses a form of least squares to minimize the coefficients of the polynomial to determine the polynomial that best fits the data. The "goodness of fit" of curve fitting equations is evaluated by calculating the weighted Coefficient of Determination (COD). Under ideal conditions, with a "perfect fit" of the model to

the data, the coefficient of determination will equal 1.00. In order to be an acceptable nonlinear calibration, the COD must be ≥0.99.

1.5.3.5 Some of the disadvantages of the use of quadratic and higher order polynomials are:

a) they require more reference standards to capture the region of curvature and define the range of calibration; and

b) the correction for bias is more complicated than for the linear model and may not be well understood,

A plot of the data is always recommended, but is not sufficient for identifying the correct model for the calibration curve. Instrument response may not appear to be non-linear over a large calibration range. Calibration models must be justified by the RSD of the RF or the CF.

In addition, test the quadratic calibration model with low level standards quantitated against itself and/or method blanks to understand the significance of the quantitation as it approaches zero and/or the reporting limit.

1.6 Corrective Action

Given the large number of analytes in some methods of analyses, it is likely that some analytes may exceed the method acceptance criteria for the RSD, correlation coefficient, or coefficient of determination. In those instances, the following steps are recommended.

1.6.1 Corrective Actions Requiring No Instrument Maintenance

1.6.1.1 If the RSD for one or more analytes exceed the method criteria, then the following steps are recommended but not required:

1.6.1.1.1 If the RSD appears to be associated with a single standard, that one standard may be re-analyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

1.6.1.1.2 Narrow the calibration range by eliminating one or more of the calibration standards producing a narrower calibration range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analysis.

Note Changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method.

Note: As previously noted, the method reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Hence, narrowing the calibration range by changing the concentration of the lowest standard will, by definition, change the method quantitation limit.

4) Clarification: If linearity can be achieved by either replacing an aberrant standard, or using a narrower calibration range, then that decision must be clearly documented. If a standard is replaced, then the entire standard and all CF associated with that standard must be replaced.

1.6.2 Corrective Actions Requiring Instrument Maintenance

1.6.2.1 If the minimum RSD, correlation coefficient or coefficient of determination is not met, the system must be evaluated and corrective action taken before sample analysis begins. Possible problems include standard degradation, injection port inlet contamination, contamination at the front end of the analytical column, active sites in the column or chromatographic system or degradation and/or contamination of the detector.

1.6.2.2 Possible corrective actions include cleaning or replacing the injector liner, seal and/or capillary column, and detector and then repeat the initial calibration. If no source of the problem can be determined after corrective actions have been taken, a new initial calibration curve must be generated. These criteria must be met before sample analysis begins.

1.6.3 It is possible for different analysts to produce instrument measurements with variances that differ between analysts on the same instrument. Small differences among analysts can be expected as part of the imprecision of the measurement process, but large systematic differences among analysts require resolution. These differences of imprecision can usually be traced back to systematic problems such as the preparation of standards etc.

1.6.4 Once established, the calibration procedure relies on the instrument continuing to respond in the same way over time. If the system drifts or takes

unpredictable excursions, the calibrated values may not be properly corrected for bias, and may further degrade the accuracy of the measurements. To assure that future measurements are being produced correctly the calibration curve is verified on a regular frequency with calibration verification standards.

5) Clarification: The ICV using a second source standard is not required by any of the established methods. However, NELAP has established the use of an ICV, but has not established acceptance criteria. Since the ICV is from a second source standard, additional variability is inherent in the quantitation of this standard. Therefore, the ICV should generally meet the CV criteria.

2.0 Calibration Model Software Validation

2.1 A primary consideration for employing any calibration model is determining a procedure to identify and validate the model used for the calibration curve. The statistical criteria used to validate the calibration model has already been discussed. These criteria are generally established and stated in the individual methods of analysis; however what is not discussed in most analytical methods are the validation criteria for assessing the data acquisition and calibration software.

2.2 It is a general established criteria that any data acquisition and quantitation software must display sample data in a format that would allow the analyst to verify the sample quantitation (concentration) report against the initial calibration regardless of calibration model.

2.3 The primary software programs used in the laboratory for data acquisition and sample quantitation are as follows:

a) Agilent Technologies, Chemstation (used for GC, GC/MS and ICP/MS);

b) Dionex, Chromeleon (used for anions and perchlorate analysis); and

c) Baush and Laumb, Vision-light (used for spectrophotometric analysis.

2.4 Chemstation Manual Software Validation Procedure

The following examples below illustrate the software validation of a single analyte for one type of calibration model. Software validation is not typically performed in this fashion, but rather by an externally prepared software validation program, specifically coded to validate data acquisition and sample quantitation software.

2.4.1 SVOC (Internal Calibration Procedure)

The table presented below represents the quadratic equation [$a(x*x) + bx + c$)] determined by the Agilent Chemstation for the compound 2,4-dinitrophenol.

This equation has been used to verify and validate the quantitation determined for this compound as tabulated below.

This compound was quantitated against a quadratic equation displayed at the bottom of the compound's calibration curve. Since this is an internal standard calibration procedure, response ratios are used to verify the Amount Ratios.

The response ratios are simply determined by:

$$\frac{\text{Response of the analyte}}{\text{Response of the associated internal standard}}$$

This value can be used to verify the concentration on the quantitation report.

The concentration value displayed on the quantitation report can be converted to a Amount Ratio simply by:

$$\frac{\text{Concentration of the analyte on the quantitation report}}{\text{Concentration of the associated internal standard}}$$

If these two values agree than the calibration model and software quantitation has been verified. This same decision-tree-procedure is used regardless of calibration model.

2,4-Dinitrophenol	Quadratic equation			R = [.0399 * (a*a)] +(.181*a) - .0449				
Std Conct.	0.5 ng/ul	0.75 ng/ul	1 ng/ul	1.5 ng/ul	2 ng/ul	2.5 ng/ul	3.0 ng/ul	4.0 ng/ul
Injection Vol	20 ul	20 ul	20 ul	20 ul	20 ul	20 ul	20 ul	20 ul
mass on column	10 ng	15 ng	20 ng	30 ng	40 ng	50 ng	60 ng	80 ng
Amt Ratio Cont (mass)/Cont IS	0.250	0.375	0.500	0.750	1.00	1.25	1.50	2.00
Response Std	4353	13038	22055	43054	66929	92647	130286	158290
Response IS (Acenapthene)	452336	425018	418961	411835	406809	380772	381953	338524
Response Ratio Rsp(std)/Rsp(IS)	0.00962	0.0306	0.0526	0.1045	0.1645	0.2431	0.3411	0.4675
R= (manual calculation) (e.g., a = .25, .375 etc) (Theoretical Response Ratio calculated to best fit quadratic curve)	0.00284	0.0285	0.0556	0.1133	0.1760	0.1820	0.2275	0.4767
concentration on quantitation report	11.34 ng	15.39 ng	19.46 ng	28.53 ng	38.21 ng	49.92 ng	63.22 ng	78.89 ng

Amt Ratio calculated from quantitation report (e.g. 11.34/40 = 0.2835)	0.2835	0.38475	0.4865	0.71325	0.95525	1.248	1.5805	1.97225
Response Ratio calculated from Amt Ration from the Qt report inserted into the quadratric equation.	0.00962	0.0306	0.0526	0.1045	0.1645	0.2431	0.3408	0.4673
Verified	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

2.4.2 VOC

2.4.2.1 Software validation of a quadratic calibration model (Internal Calibration Procedure)

The table presented below represents the quadratic equation [a(x*x) + bx + c)] determined by the Agilent Chemstation for the compound Chloroethane. This equation has been used to verify and validate the quantitation determined for this compound as tabulated below.

This same decision-tree-procedure is used regardless of calibration model as described below.

Chloroethane	Quadratic equation			R = [-0.0101 * (a*a)] +0.152*a) - 0.000109			
Std Conct.	0.25 ug/L	0.50 ug/L	1.0 ug/L	2.0 ug/L	4.0 ug/L	8.0 ug/L	16 ug/L
Amt Ratio "a" Cont (mass)/Cont IS	0.0250	0.05	0.1	0.2	0.4	0.8	1.6
Response Std	2616	4975	8787	12919	40853	72905	138320
Response IS	617903	622915	633385	626190	597915	622710	644054
Response Ratio Rsp(std)/Rsp(IS)	0.004	0.008	0.014	0.021	0.068	0.117	0.215
"R" determined from quadratic using "a"	0.004	0.007	0.015	0.030	0.059	0.115	0.217
concentration on quantitation report (ug/L)	0.29	0.53	0.93	1.38	4.65	8.16	15.82
Amt Ratio calculated from quantitation report (e.g. 0.29/10 = 0.029)	0.029	0.053	0.093	0.138	0.465	0.816	1.582
Response Ratio "R" calculated from Amt Ration from the Qt report inserted into the quadratric equation.	0.004	0.008	0.014	0.21	0.068	0.117	0.215
Verified	Yes	Yes	Yes	Yes	Yes	Yes	Yes

2.4.2.2 Software validation of a linear regression calibration model (Internal Calibration Procedure)

The table presented below represents the linear regression equation (aX + b)] determined by the Agilent Chemstation for the compound 1,1,2,2-Tetrachloroethane. This equation has been used to verify and validate the quantitation determined for this compound as tabulated below.

1,1,2,2-tetrachloroethane	Linear Regression equation			R = [0.492*a) - 0.00356				
Std Conct.	0.25 ug/L	0.50 ug/L	1.0 ug/L	2.0 ug/L	4.0 ug/L	8.0 ug/L	16 ug/L	32 ug/L
Amt Ratio "a" Cont (mass)/Cont IS	0.0250	0.05	0.1	0.2	0.4	0.8	1.6	3.2
Response Std	2477	5280	11718	21909	48065	87603	189127	402829
Response IS	242481	246435	253070	248662	237135	245818	248731	260591
Response Ratio Rsp(std)/Rsp(IS)	0.010	0.021	0.046	0.088	0.203	0.356	0.760	1.55
"R" determined from linear equation using "a"	0.009	0.021	0.046	0.095	0.193	0.390	0.784	1.57
concentration on quantitation report (ug/L)	0.28	0.51	1.01	1.86	4.19	7.31	15.51	31.47
Amt Ratio calculated from quantitation report (e.g. 0.28/10 = 0.028)	0.028	0.051	0.101	0.186	0.419	0.731	1.551	3.147
Response Ratio "R" calculated from Amt Ration from the Qt report inserted into the quadratric equation.	0.010	0.022	0.046	0.088	0.203	0.356	0.760	1.54
Verified	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

2.4.2.3 Software validation of a linear through zero (average RF) calibration model (Internal Calibration Procedure)

The table presented below represents the linear equation (aX) determined by the Agilent Chemstation for the compound Toluene. This equation has been used to verify and validate the quantitation determined for this compound as tabulated below.

Toluene	Linear Average RF equation			R =(3.35*a)				
Std Conct.	0.25 ug/L	0.50 ug/L	1.0 ug/L	2.0 ug/L	4.0 ug/L	8.0 ug/L	16 ug/L	32 ug/L
Amt Ratio "a" Cont (mass)/Cont IS	0.0250	0.05	0.1	0.2	0.4	0.8	1.6	3.2
Response Std	19818	41005	85866	161992	338446	621027	1407233	2869298
Response IS	242481	246435	253070	248662	237135	245818	248731	260591
Response Ratio Rsp(std)/Rsp(IS)	0.082	0.166	0.339	0.651	1.43	2.53	5.66	11.01
"R" determined from linear equation using "a"	0.084	0.168	0.335	0.670	1.34	2.68	5.36	10.72
concentration on quantitation report (ug/L)	0.24	0.50	1.01	1.94	4.26	7.54	16.88	32.86
Amt Ratio calculated from quantitation report (e.g. 0.24/10 = 0.024)	0.024	0.05	0.101	0.194	0.426	0.754	1.688	3.286
Response Ratio "R" calculated from Amt Ration from the Qt report inserted into the quadratric equation.	0.080	0.168	0.338	0.650	1.43	2.53	5.65	11.01
Verified	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

2.5 Chemstation Metals Manual Software Validation Procedure

2.5.1 Sample concentrations may be verified by the use of the Counts Per Second (CPS) information displayed on the calibration and final sample data quantitation reports. This data used in conjunction with the initial calibration equation report may be used to verify the calculated concentrations on the final sample quantitation report.

Example:

Scandium initial calibration equation using the weighted linear regression equation is a follows: (This changes for each new initial calibration, ie. daily).

$Y = (1.642E+0 * X) + 4.195E-2$

Therefore:

$$X = \frac{Y-(4.195E-2)}{1.642E+0}$$

The sample concentration may be verified as follows:

Y = Sample counts for Scandium is 84.9575

Therefore:

X = 51.70 ug/L which equals the concentration on the sample quantitation report.

2.6 Dionex Manual Software Validation Procedure

2.6.1 Software validation of a quadratic calibration model (External Calibration Procedure)

The table presented below represents the quadratic equation [a(x*x) + bx + c)] determined by the Dionex software for the compound alpha-BHC. This equation has been used to verify and validate the quantitation determined for this compound as tabulated below.

This same decision-tree-procedure is used regardless of calibration model as described below.

α-BHC	Quadratic equation		Amount (concentration) = [5.746319xE-15 * (a*a)] +(1.510110 x E-07*a) + (-0.0010)							
Std Conct (ug/ml)	0.0005	0.001	0.03	0.05	0.06	0.09	0.12	0.15	0.18	0.20
α-BHC response	9307	8683	203684	348143	393666	588711	793844	941483	1143017	1280581
Conct. From QT report in ug/L	0.0004	0.0003	0.0300	0.0522	0.0593	0.0899	0.1225	0.1462	0.1791	0.2018
Calluated Conct from equation	0.0004	0.0003	0.0300	0.0522	0.0593	0.0899	0.1225	0.1462	0.1791	0.2018
Verified	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

2.6.2 Software validation of a Linear calibration model (External Calibration Procedure)

The table presented below represents the linear equation [(ax + b)] determined by th Dionex software for the compound alpha-BHC. This equation has been used to verify and validate the quantitation determined for this compound as tabulated below.

α-BHC	Linear equation Amount (concentration) = (1.557 x E-07*a) + (0)									
Std Conct (ug/ml)	0.0005	0.001	0.03	0.05	0.06	0.09	0.12	0.15	0.18	0.20
α-BHC response	9307	8683	203684	348143	393666	588711	793844	941483	1143017	1280581
Conct. From QT report in ug/L	0.0014	0.0014	0.0317	0.0542	0.0613	0.0917	0.1236	0.1466	0.1780	0.1994
Calluated Conct from equation	0.0014	0.0014	0.0317	0.0542	0.0613	0.0917	0.1236	0.1466	0.1780	0.1994
Verified	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

Appendix F

Software Quality Assurance Plan (SQAP)

Appendix F

Standard Operating Procedure

SOP F.1
Software Quality Assurance Plan (SQAP)

1.0 SOFTWARE QUALITY ASSURANCE PLAN (SQAP)

1.1 The generation, compilation, and reporting of electronic data is a critical component of Alpha's laboratory operations. In order to generate data of known and acceptable quality, the quality assurance procedures and quality control practices for electronic data systems have been developed to be comparable in sophistication and intent to other elements of Alpha's QA program. Sections 8.1 through 8.11 of EPA Document 2185-Good Automated Laboratory Practices has been used as the foundation for the development and implementation of Alpha's SQAP.

1.2 Alpha has developed and implemented a Software Quality Assurance Plan (SQAP) that addresses requirements and responsibilities for QA activities related to the LIMS, local software, instrumentation software, and other systems related to the generation, compilation, reporting of data, or other supporting information. It is the responsibility of the LIMS Administrator to implement the SQAP and to ensure that all software and QA functions are being properly documented. The LIMS Administrator is also a part of the QA Team and reports on a regular basis to the QA Officer. The SQAP describes polices and practices for the development, modification, maintenance, archival, and use of computer software.

Appendix F

Standard Operating Procedure

SOP F.2
Computer Software Operations

1.0 COMPUTER SOFTWARE OPERATIONS

1.1 This standard operating procedure outlines and defines the techniques, operations, maintenance, and security when dealing with any of the various electronic data collection, processing, and archival devices present in Alpha's facilities.

2.0 Standard Operating Procedure

2.1 In general the computer department is responsible for all software loading, upgrades, coding changes, debugging and hardware/software retirements.

The following general policies apply to these functions:

a) Only authorized, trained personnel are allowed to perform these functions;

b) No unauthorized software is to be loaded on any company computer;

c) Only authorized, trained personnel are allowed to use company connections to the network or internet; and,

d) No hardware, software, or raw data is to be removed from the laboratory without written authorization.

2.2 If available in the instrument software, all electronic tracking and audit functions must be enabled such as when manual integrations are performed by chemstation an "m" appears next to the analyte which was manually integrated. This function can be turned off; however, for our operations these types of audit functions should never be turned off or inactivated.

2.3 Definitions

Alpha - a specific MSAccess2003 database containing laboratory data.

Khemia - software program utilizing MSAccess2003 security formats for the Omega and Alpha databases.

Laboratory Information Management System (LIMS) - a general term used to describe the entire in-house network.

Network - the interconnection of various PCs and servers.

Omega - a specific MSAccess2003 database containing templates for queries, reports and forms. This term is also used as a general grouping of the interconnected Khemia, Omega, and Alpha database structure.

2.4 Safety

2.4.1 General

When dealing with any of the equipment in this facility, assume that hazardous conditions are present in the form of mechanical, electrical, or chemical situations. Complete a new assessment of the potential hazards of any equipment prior to undertaking any interactions with this equipment. This assessment should be made each time the equipment has been out of sight. There should also be a continual scanning of the equipment during use to ensure that no new situations have occurred or been overlooked. If any hazardous conditions are present, appropriate precautions should be undertaken to limit the user's interactions with these conditions.

2.4.2 Mechanical Hazards

2.4.2.1 Temperature

Most of the equipment present in this facility contains zones which are used to heat or cool. Either of these conditions can cause severe burns with only momentary contact. If it is necessary to work around these zones without bringing them to room temperature, the use of protective devices should be used to limit the possibility of contacts.

2.4.2.2 Pinching and Crushing

Various portions of the equipment in this facility use automated robotics. Due to automated movements of these devices severe damage can occur. Shielding should be used whenever it is necessary to work around these devices without disabling the automated processes. Extreme caution is to be taken to ensure that the automated processes are not accidently started.

2.4.2.3 Weight

Training has been given to all employees regarding the appropriate techniques to use when moving or lifting heavy equipment. These should be followed without exception.

2.4.2.4 Ergonomics

Much of the equipment is located in areas that are not easily accessible for maintenance. If it is not possible to disconnect and move the equipment to a more accessible area, extreme care should be taken to limit the possibility of injury due to falls, strains, or space constraints.

Also, care should be taken to ensure weight limits are not exceeded that may be present when climbing on or around the equipment, especially when dealing with tables and ladders.

2.4.2.5 Dust

Due to the area in which we are located, dust is quickly accumulated in all of the facility's equipment. This dust creates a potential for blinding or ingestion. Before working on any piece of equipment it is recommended that the dust be removed with a vacuum. If a pressurized container is to be used to remove the dust, extreme care should be taken that items are not propelled into the nose, mouth, or eyes.

2.4.3 Electrical Hazards

2.4.3.1 Dangerous currents are present in most of the equipment in this facility. Extreme care should be taken to ensure that the operator does not come into contact with any source of electricity. Whenever dealing with electrical areas, it is strongly recommended that all sources of current be removed. When systems have been unplugged from their current source, care should be taken that they are not accidentally reconnected. Lockout tags or plastic ties can be used to alert the need for the source to remain disconnected.

Current can still exist after the source has been disconnected! Allow ample time for capacitors to discharge prior to maintenance.

2.4.3.2 Static electricity is an ever present consideration. The discharge of static through the equipment components can cause failures. It is strongly recommended that static wrist bands be worn whenever electronic components are being maintained.

2.4.3.3 Liquids of various types are used throughout our facility. Many of these are excellent conductors of electricity. Do not work on electrical equipment that is wet without removing any potential source of current.

2.4.4 Chemical Hazards

2.4.4.1 This facility utilizes many chemicals that are of an extreme hazardous nature. Read the MSDS for each chemical present in the work area prior to starting work. Especially note any potential situations that might affect the work area such as flammability and contact thresholds.

2.4.4.2 When dealing with any cleaning solutions, be aware of their potential effects on the surrounding equipment. Many of the cleaning solutions are potential contaminants in our testing procedures.

2.5 Software Documentation

Computer software developed by Alpha and commercial off-the shelf computer software program validation studies are conducted in a traceable and planned fashion to ensure all major programing activities such as, coding changes and debugging can be documented.

2.5.1 Software Inventory/Coding Modification Logbooks

Three-ring logbooks contain records documenting laboratory controlled software programs and their revisions used by Alpha. These commercially purchased software products have their own logbook.

2.5.1.1 Omega Database

Two logbooks are kept for changes made to the omega database. One of these logbooks is maintained for documenting coding and structure modifications and the other logbook is used for data and data quality objective changes made to the omega database.

2.5.1.2 Chemstation

A single logbook is maintained documenting both common coding changes and individual system (instrument) code changes made to the chemstation software.

Appendix F

Standard Operating Procedure

SOP F.3
Data Collection and Storage

1.0 DATA COLLECTION AND STORAGE

1.1 This procedure is used to standardize the naming of subdirectories used to collect and store computer data for consistency throughout the laboratory.

2.0 Standard Operating Procedure

2.1 Data Collection

All data collection, interpretation, and corrections are made in a manner consistent with the practices and protocols outlined in the various Standard Operating Procedures governing these areas.

2.2 HP Chemstation / Enviroquant

2.2.1 Data File Nomenclature

YYMMDD## where:

YY - the YEAR that the data file was created.
MM - the MONTH that the data file was created.
DD - the DAY that the data file was created.
- a sequential NUMBER indicating the order in which the data file was analyzed and resets with each sequence to 01.

Thus a data file that was created on 01FEB11, and was the third in that sequence would be: 11020103.D.

2.2.2 Data File Storage

2.2.2.1 Each PC has its own harddrive (C: or D:) location for the storage of the Chemstation/Enviroquant files. Within this harddrive, all of the Chemstation/Enviroquant files are stored under the subdirectory HPCHEM or MSDCHEM.

2.2.2.2 Within this subdirectory, the METHODS, SEQUENCES, and DATA, have their own subdirectories that exist in a subdirectory named after the specific instrument (e.g.,C:\HPCHEM\MS15\DATA\).

2.2.2.3 Sequences should be named the same as the data files, but without the sequential number indicating order (e.g., 11201.S).

2.2.2.4 DATA should be stored in its own data subdirectory that is named the same as the sequence within which it was collected. This additional

subdirectory exists within the DATA subdirectory and helps to create "packets" of related data that are easier to locate, archive, and retrieve (e.g., C:\HPCHEM\MS15\DATA\110201\11020103.D).

2.3 Long Term Data File Storage and Archival

2..31 Each month's data subdirectories are compiled into a single subdirectory named for the month and year (e.g., Feb 11).

2.3.2 Each month, the previous month's data is archived on dual DVDs, one of which is kept offsite.

2.3.3 As space permits, previous data is kept on each PC, with the oldest data being removed first.

2.3.4 Each day, the entire, non-monthly archived , contents of each system's data collection software is copied onto the main server. Each subsequent day's archive is stored separately in folders on the server. Mid-month, the previous month's archives are deleted.

Appendix F

Standard Operating Procedure

SOP F.4
Data File Uploading Procedures

1.0 DATA FILE UPLOADING PROCEDURES

1.1 All data produced by analytical instruments must be formatted in a way the Omega software can parse-out information that is critical to the final compilation, reduction and reporting of data.

2.0 Data File Downloading into Omega Formats (Chemstation Data)

2.1 For correct interfacing between the Chemstation/ Enviroquant text file and the Omega database, the following information must be present in specific areas on the quantitation report. This interface needs to have the specified information in the exact position noted below. Each field must contain the exact number of characters. Use blank spaces for empty characters. If too few or too many spaces are present, the file will not upload. Any information after the sample ID on the sample line is ignored as far as Omega is concerned.

2.2 The positions are those as seen when in the sequence table or when editing the file information from the quantitation screen.

Field	Example	Quant report Line	Start Position	Length
Sample Type	SAMP	Sample	1	5
Sample ID	05021422-23AMSD	Sample	6	16
VOC or BNA Batch ID	MS06W0625A	Misc	1	10
VOC or BNA Test Name	VOC_W	Misc	11	6
VOC or BNA PQL Multiplier	1.000	Misc	17	6
TPH Batch ID	MS06W0625B	Misc	23	10
TPH Test Name	TPH/P_W	Misc	33	8
TPH PQL Multiplier	1.000	Misc	41	5
Alcohol Batch ID	08460	Misc	1	6
Alcohol Test Name	Alcohol_W	Misc	7	10
Alcohol Multiplier	1.000	Misc	17	6

If there are no TPH-Purgeable batches associated with the data file, these fields can be left blank or used for other information.

2.2.1 Field — Explanations

Sample Type — use **SAMP** for all client samples

use **MBLK** for all Method Blanks (not needed for cleanout blanks)
use **DUP** for all sample Duplicates (not fortified/spiked)
use **LCS** for all Laboratory Control Samples and Laboratory Fortified Blanks
use **LCSD** for all LCS Duplicates and LFB Duplicates
use **MS** for all Matrix Spikes
use **MSD** for all MS Duplicates

Sample ID

Client ID & Sample ID & Fraction (must be identical to that listed on our work-order sheet.)
(e.g. GMT09020143-06A)
(e.g. GMT09020143-06ADUP)
(e.g. GMT09020143-06AMS)
(e.g. GMT09020143-06AMSD)

Batch ID

Instrument number and Matrix and Month and Day and Letter of the batch associated with the VOC samples (e.g. MS09W0214B) or prep batch ID associated with SEMIVOA samples. (e.g. 12345). This must be identical to that listed on our work-order sheet.

Test Name

- PNA_SIM_S
- PNA_SIM_W
- TPH/P_W
- TPH/P_S
- TPH/P_A
- VOC_W
- VOC_S
- VOC_A
- BNA_W
- BNA_S
- ALCOHOL_W
- ALCOHOL_S

PQL Multiplier

The amount by which the report's standard reporting limit needs to be raised. Note that this may not necessarily be the instrument multiplier.

2.3 When all of the reportable runs have been reviewed, run a DoList selecting MACRO and then OMEGA. This will dump the selected files onto the floppy in the A: drive, in a text format that is readable by Omega. This floppy should then be included with the hard copies of the QC data.

2.3.1 Include the associated sample with the MS/MSD on the QC floppy (as the LIMS system needs to reference the MS/MSD to its parent sample).

2.3.2 The sample associated with the MS/MSD must be logged-in for the associated analytical test.

3.0 Hard Copy, Non-automated, and Non-excel Data

3.1 This type of data is stored in the work order folders and are manually entered into Omega by the report writing department.

Appendix F

Standard Operating Procedure

SOP F.5
Electron Diskette Deliverables (EDDs)

1.0 ELECTRONIC DISKETTE DELIVERABLES (EDDs)

1.1 Many of our clients have a need to incorporate their data into a variety of databases for a multitude of end uses. In an effort to help our clients decrease the need for manually entering the hard copy results received from our facility, we provide a variety of electronic data formats.

1.2 There are generally no industry standard formats that are required by our clients; therefore, Alpha maintains the ability to create and structure a specific format for each client. These formats range from a simple text listing of the analytes and their results, through a typical excel format of the full sample and QC data package, to a six file package of the laboratory EDF (GeoTracker) result tables.

1.3 The electron transmission of results must be conducted to ensure that all reasonable steps have been taken to preserve sample and client confidentiality.

2.0 STANDARD OPERATING PROCEDURE

2.1 EDD Format Specification

Clients who request EDDs should provide a specific format and nomenclature for their data. Specific coding has been added to Omega to insure consistency in the EDD creation.

2.2 Data Inclusions

Some clients require QC in their EDDs, while others do not. If required, QC is entered by the reporting department prior to EDD creation.

2.3 EDD Creation

The following description briefly describes the essential elements for the creation of an electronic data deliverable.

2.3.1 Choose the EDD REPORTS option from the main categories in omega.

2.3.2 Under WORK-ORDER, enter the desired work-order. If QC is required use the bottom of the form to double check for errors or omissions prior to creating the EDD.

2.3.3 Click the OK button.

2.3.4 Use the EDD DATE UPDATE button to update the date that the EDD is emailed.

2.3.4.1 Enter the work-order number and then the ENTER key.

2.3.4.2 Repeat until all work-orders have been entered.

2.3.4.3 Click CLOSE to exit.

2.3.5 The unfinished files are located in the L:\Elec_DD\<ClientID>\ subdirectory (this is subject to change).

2.3.6 Open each Excel file and reformat the column widths, margins, and page settings.

2.3.6.1 Widths = size to fit (autofit).

2.3.6.2 Margins - top (0.5), bottom (0.25), left (0.25) and right (0.25).

2.3.6.3 Page settings (depends on the client format):

- Landscape,
- Fit to 1 page wide.

2.3.7 Check the number of rows against the number of expected line items (analytes x samples).

2.3.8 Spot check results with excel entries.

2.3.9 Save each Excel file in an Excel file format.

2.3.10 Email the files to the address listed in L:\Elec_DD\EDD_LIST.XLS (this is subject to change). Include a copy of the Email in the data folder.

2.3.11 Place the completed work-order folders in the scanning department file bins next to the outside door in the fax room.

2.4 File Transfer Protocol (FTP) Data Access

2.4.1 The FTP file server is a stand-alone PC that is behind Alpha Analytical's Firewall.

2.4.1.1 Individual file extensions can be included or excluded from being transferred into or out of the FTP file server.

2.4.2 The FTP file server can only be accessed from the "outside" through Alpha Analytical's IP wed address.

2.4.2.1 The IP wed address is: 64.42.17.198

2.4.2.2 Data is managed on the FTP file server through an Alpha Analytical account and password.

2.4.2.3 Client access is limited to READ only.

2.4.2.4 An example of the FTP software (freeware) is found at WS_FTP from www.ipswitch.com

2.4.3 The FTP file server cannot access Alpha Analytical's main server array.

2.4.4 Individual client file folders, accounts, and passwords are utilized to limit access to data on the FTP file server.

2.4.5 Client data on the FTP file server is only a copy of the data available on Alpha Analytical's main server array.

2.4.6 Clients are notified b email when data is added to their access account areas.

Appendix F

Standard Operating Procedure

SOP F.6
MSAcess2003 DATABASES

1.0 MSAccess2003 DATABASES

1.1 The Access databases are used to correlate, format and store the various data from our client's requests. These include (but are not limited to) chain-of-custodies, bottle orders, quotes, test preparation, analytes, sample prep, analysis, reports, QC, and EDDs. The Omega database consists of the templates, formats, and structures, for the various tables, forms, and reports. The Alpha database contains all of the data utilized by the Omega database.

2.0 STANDARD OPERATING PROCEDURE

2.1 Omega Database

2.1.1 The Omega database should be renewed on each working day through the double-clicking of the UPDATE icon located on the desktop of each PC. By updating on a daily basis, all of the various PCs will be utilizing the most recent changes to these tables, forms, and reports.

2.1.2 No one other than those authorized should make any coding changes to the databases. The structural integrity of the Omega database is so interwoven, that seemingly minor changes can have massive effects.

Unless specifically requested, only the LIMS administrator is authorized to make any changes.

2.1.3 Request forms are available if changes are desired to the Omega database. If approved, these changes will be incorporated in the next version of the update (see F.6.2.1.1 above).

2.1.4 Any changes to this database are only saved on the local PC. These changes must then be copied over to the server. Extreme care must be taken to insure that a copy of the most recent version is present locally prior to making any changes, otherwise it is possible to overwrite previous changes with an older version.

2.2 Alpha Database

2.2.1 This database contains the actual laboratory generated data. This data consists of not only analytical data, but also all of the information contained within substructures, such as laboratory personnel, analyte names, tests, client information, etc.. This database exists on the server and is therefore accessible to all areas of the laboratory simultaneously.

2.2.2 Due to the intricate interlacing of the information in this database, changes to any portion affects the entire database. Personnel must only utilize those areas

of this database in which they have been trained, and then assure to the best of their ability that the information they enter is correct. Since this information is utilized by all of the laboratory, any changes made to information already present may adversely affect other areas of the laboratory. Any changes made to information already present in the system should be closely examined for those other laboratory areas affected, and the personnel in those areas notified as to the changes.

2.2.3 Since this is a shared database, only one current copy exists. Any data deleted from this database is not retrievable! Only those personnel trained and authorized should perform any data deletions, and then only after careful scrutiny.

A copy of the Alpha database is made by the LIMS Administrator daily. If major problems occur, the copy can be restored, however this will remove any data added or changed since the copy was created.

2.2.4 Due to the shared status of this database, data corruptions occasionally occur for various reasons. These corruptions can be limited through the simple process of returning to the main menu as soon as possible whenever the current task or set of tasks is completed. If the system is not being used for several minutes, then return to the main menu. This action will release resources that might be needed by another area of the laboratory as well as prevent those resources from being corrupted in case of local PC malfunction. The system should never be allowed to stand idle for extended periods with any screen other than the main page present on the PC monitor.

2.3 Database Corruption

Database repair should only be undertaken by the LIMS Administrator or a designated person.

2.3.1 Repair

2.3.1.1 Copy the Alpha.mdb database from S:\Alpha.mdb to the local drive. (This is done to increase the speed of the repair process).

Caution: Be certain that all of the PCs have closed Omega prior to the copy. If any of the PCs are active in the database then the repair process will truncate the tables that were active, causing ALL of the data in those tables to be lost!

2.3.1.2 Open MSAccess without any open databases.

2.3.1.3 Under TOOLs click UTILITIES, then REPAIR and choose the Alpha.mdb database that was copied to the local drive.

2.3.1.4 After the repair is completed, COMPACT the database.

2.3.1.5 If any ERRORS have occurred, then repeat the process using the daily archived database.

- All data and changes entered into the database that day will have been lost and will need to be redone.
- Sometimes, compacting a corrupted database will allow a repair to proceed without errors.
- Sometimes, decompiling will fix a corruted database.

2.3.1.6 After compacting the repaired database, copy the file back to S:\Alpha.mdb.

2.3.2 Compact

2.3.2.1 Open the MSAccess without any open databases.

2.3.2.2 Copy the database from S:\ to the local drive.

Note: This will increase the speed of the compaction.

Caution: Be certain that the pathways are correct. Multiple copies of the same database from different time periods are present throughout the network.

2.3.2.3 Under FILE click UTILITIES, then COMPACT and choose the correct database on the local drive.

2.3.2.4 Compact the file into itself (i.e. - save the file under the same name and location).

2.3.2.5 Copy the file back into the S:\ directory, overwriting the original.

Appendix F

Standard Operating Procedure

SOP F.7
Data Archiving

1.0 DATA ARCHIVING

1.1 Archiving data is a major component or our record keeping system. The record keeping system is designed to allow for the historical reconstruction of all laboratory activities that produced the analytical data. The goal of the record keeping system is to ensure laboratory data is stored and archived for possible future use in a traceable, planned and orderly manner to facilitate access, ease of use, and security.

2.0 STANDARD OPERATING PROCEDURE

2.1 Analytical Instruments

On a daily basis, in-house software automatically copies all instrument data to a backup server. These daily backups are kept until after the monthly archiving to DVDs are completed. In case of data loss, (at the PC due to various reasons,) the original data can be restored.

On a monthly basis, the system administrator will archive any data files greater than one month old. These files are transferred to dual DVDs for both local and offsite storage. The archived data also remains on the individual PCs as hard drive room permits. When hard drive room is necessary, the oldest archived files are removed first.

2.1.1 In preparation for archiving, the system administrator compiles the various monthly data, method, and sequence files to create a monthly archival subdirectory on each analytical PC.

2.1.1.1 Within the Archive subdirectory, create a new folder called by the month and year of the data (e.g. FEB11).

2.1.1.2 Move the data and sequences into this new folder.

2.1.1.3 Drag (copy) the methods into this new folder.

2.1.2 This archive subdirectory is then copied across the network to the server via the in-house archiving software.

2.1.3 The monthly archive subdirectories are then copied onto DVDs.

2.1.3.1 Copy the archived data from the backup server to the archive PC.

2.1.3.2 Insert a new DVD into the R-RW device (LIMS Administrator's archive PC).

2.1.3.3 Click on the Creator Classic icon.

2.1.3.4 Enlarge the subdirectories in the upper screen such that the desired monthly folder is present on the right side.

2.1.3.5 Drag (copy) the monthly folder down to the right side of the lower screen. If multiple instruments, drag all of the desired instrument folders, not the monthly. Each DVD holds 6.4 Gb. The amount of the selected files is listed at the bottom of the screen. Do not exceed the 6.4 Gb limit.

2.1.3.6 Double click on the UNTITLED on the left bottom screen, and name the DVD with the month and year.

2.1.3.7 Click on the DISC tab at the bottom left, and then on the red RECORD button.

2.1.3.8 When the DVD is finished, the DVD will eject.

2.1.3.9 Insert a new DVD and restart at for the duplicate DVD.

2.1.4 Each DVD is labeled with the month and year

2.1.4.1 Use only felt pens (Sharpie) to write on the DVDs.

2.1.4.2 Label each DVD and jewel box with "RAW DATA," and the month and year.

2.1.5 Store the in-house copies in the DVD bins, and send the duplicates to offsite storage.

2.1.6 Data retrieval is accomplished by copying the desired files from the DVD back onto the archive PC and changing the properties (removing the read only check mark after double right clicking on the file or files). The files are then moved to the backup server where they can be uploaded onto the instrument PC.

2.1.7 Due to various circumstances, the data on the new DVDs may be corrupt. To ensure that a good copy is present, the data from the DVDs is copied to another PC and random files are checked for data integrity.

2.2 Omega

The Omega databases are backed up daily by the in-house archive software in the same manner as the instrument data.

The Omega databases are backed up monthly by the LIMS Administrator for offsite archiving.

2.3 Administration Files

The financial files (BUSINESSWORKS) are backed up nightly, weekly, and monthly with nightly and monthly archived files stored offsite.

2.4 Server

The entire server contents are backed up nightly, weekly, and monthly with monthly archives stored offsite.

2.5 Accessing Archived Data

2.5.1 Occasionally, there is a need to review data that is no longer on the local instrument PC that has been archived.

2.5.2 The LIMS Administrator maintains the original instrument data archived onto DVDs. In the event access to a DVD containing analytical data is required, that access is controlled by the LIMS Administrator.

2.5.3 Upon request, a copy of the required data is made on a CD from the original DVD. The original DVD never leaves the LIMS Administrator's controlled area.

Appendix F

Standard Operating Procedure

SOP F.8
PC/Server Integrity and Software Validation

1.0 PC/SERVER INTEGRITY AND SOFTWARE VALIDATION

1.1 PC/Server hardware and software must be used in an environment that promotes data integrity. All software used for acquisition, reduction and reporting of data is validated and verified prior to use.

2.0 STANDARD OPERATING PROCEDURE

2.1 PC/Server Data Integrity Procedures

2.1.1 Software Dos and Don'ts

a) All personnel should only use those programs in which they have been trained or have prior experience;

b) No unauthorized or unlicensed software is to be loaded on any company computer;

c) Only authorized, trained personnel are allowed to use company connections to the network or internet;

d) No hardware, software, or raw data is to be removed from the main offices without written authorization;

e) No unauthorized removal or modification of any software programs, functions, or coding;

f) Document all error messages or undocumented features (bugs); and,

g) No unauthorized modifications to PC settings.

2.1.2 Hardware Dos and Don'ts

a) Only trained, authorized personnel should attempt any hardware installations, repairs, or modifications; and

b) No unauthorized hardware is to be attached to any PC or to the LIMS network.

2.2 Software Validation

2.2.1 Software validation and verification activities are performed to ensure the software adequately and correctly performs all intended functions, and to ensure the software does not perform any unintended functions.

2.2.2 Software validation and verification are performed jointly by the computer QA unit and the end PC user.

2.2.3 To evaluate the technical adequacy of software, testing activities are performed to assure the software produces correct results for the test case. Test case results are then compared to results from alternate methods, such as manual computation of results or use of another program to process the same data file.

2.2.4 Once software has been validated, then continuing maintenance of the baseline software is produced during periods of use. As deemed appropriate, this may require removal of latent errors, or adoptions to changes in the operating environment. On a schedule determined by the LIMS administrator, baseline systems are checked for corruption.

2.3 Software and Data Security

The following security controls are established to permit authorized access and to prevent unauthorized access to software systems and data files:

2.3.1 All personnel should only use their own individual accounts when logging onto the PCs, server, or Omega.

2.3.2 All personnel should log off of the system whenever they are away from their station for extended periods.

2.3.2.1 In place of logging out, the use of password secured screen savers is adequate for normal working hours.

2.3.2.2 Instruments may have their own log-in identifications with limited server and LIMS access.

Appendix F

Standard Operating Procedure

SOP F.9
Sample Login

1.0 SAMPLE LOG IN (Computer Entry)

1.1 The following SOP is established to maintain uniformity, clarity and consistency when inputting information into the lab database upon sample receipt. This computerized Sample Log In SOP is implemented with all the provision established in the main Sample Log In SOP.

2.0 STANDARD OPERATING PROCEDURE

2.1 To enter Omega from the main computer menu, double click on the Omega icon. When the log-in prompt appears, type access name and click the "O.K."button. This will take you to the main Omega II menu. From this menu, double click on "Work Orders."

2.2 To add a work order, double click the "Add" key in the lower left-hand corner of the screen. Amend the work order number to correspond with the correct sequence:

SCO: 1-19
Assistant SCO: 20-39
Assistant SCO: 40-59
Assistant SCO: 60-79

2.3 In the "Client ID" box, enter the client's three letter code, then double click the "Client Information" field. The company name, contact person, address, phone and FAX numbers will appear automatically.

Amend the "TAT" (turn-around-time) box as needed for TAT"s shorter than ten days. Add an "Order name" if applicable. Click the box and type the name. Add "Report Attention", COC #, sampled by, cooler temp. and comments.

2.4 Double click the "Received" box. A calendar will appear with today's date selected. Double click the proper date. The computer will automatically enter the correct date. Double click the "Date Due" box. The computer will automatically count ahead the numbers of days selected in the TAT box (excluding weekends).

Add any additional comments in the "Comments" box by clicking once in the box, then start typing. Input information such as:

- Real ice present or not,
- Samples frozen or not,
- Security seals present or not,
- Rush Turn Around Time (TAT) sample,
- California samples, etc.

in the comments section. If the client would like to give a purchase order number,

click the"Invoice Info" button at the top of the screen and enter the Purchase Order number.

2.5 Sample Log-In Procedure

2.5.1 Click once in the "Client Sample ID" box. Type in the ID requested by the client.

2.5.2 TAT will automatically appear.

2.5.3 Enter the sample "Collection Date." This should be written on the sample label - otherwise, ask the client. Double space and enter the time sampled in military time.

2.5.4 Enter the sample " Matrix." This can be typed by hand, or by clicking the down arrow adjacent to the matrix box and then selecting the proper matrix (soil, aqueous, etc.). Enter the bottle (container) type the list provided.

2.5.5 Enter the number of containers which belong to the sample currently being entered.

2.5.6 For drinking water, some clients will give Public Water System numbers. This number can be typed in the "PWS/DWS" box in the field data page. A Sample Site ID and System ID can also be added at the client's request in the proper box.

2.5.7 Add any extra comments.

2.6 Click once in the "Test Group" box. Enter the proper test, then click the down arrow next to the box to view the list of possible choices. Tests are segregated by state and test method.

2.6.1 For each *new* sample, click the "Add Sample" and repeat the sample log-in process, starting with a new client ID. Many samples will have Trip Blanks included. Treat the Trip Blank as a separate sample. For Client Sample ID type "Trip Blank." The TAT will be the same as for the corresponding samples. The collection date is listed as the earliest sample date present in the work order. Trip Blanks are not analyzed unless requested by the client. Therefore, under Test Groups type "Hold ". This will tell the analysts that the sample should not be run.

2.6.2 When all of the relevant tests are entered, click the "out-the-door" button at the lower right-hand corner of the screen (picture of an open door with an arrow pointing into it).

2.7 To print the Chain of Custody, click the WO COC button at the top of the screen.

2.8 To print the analyte lists, click on the PRINT TEST Button at the top of the screen. Enter the sample ID (e.g. 02) of the sample with the test parameters that are to be printed. Use a "*" to print all of the test codes, unless a specific test is desired for printing.

2.8.1 These analyte lists are to be included in each of the appropriate test file folders (e.g. the VOC_W list goes into the VOC file folder).

Appendix F

Standard Operating Procedure

SOP F.10
Sample Preparation Omega SOP

1.0 SAMPLE PREPARATION OMEGA SOP

1.1 The following SOP is established to maintain uniformity, clarity and consistency when inputting sample preparation data. This computerized sample preparation SOP is implemented with all the provisions established in the main Sample Preparation SOP.

2.0 STANDARD OPERATING PROCEDURE

2.1 From the Windows desktop, Click on the OMEGA Icon

2.1.1 Every morning, before opening Omega, the UPGRADE icon should be clicked to ensure that the newest version of the Omega software is being used.

2.1.2 The Alpha screen will appear, press enter.

2.1.3 Click on the SAMPLE PREP button.

2.2 To add samples to a previously existing batch:

2.2.1 Scroll down the left list and double click on the desired prep batch.

2.2.2 Click on the SAMPLE ID column and arrow down to the bottom of the list

2.2.3 Enter the sample ID in the first blank line (Only the numbers followed by the letter A).

Note: This can be accomplished by either typing the sample ID or using the user select option.

2.2.3.1 Make sure that the sample ID matches that on the work-order (e.g. : 11021401-01A).

2.2.3.2 If a Client ID and Client Sample ID do not appear after moving out of that cell, then double check the sample ID. If it still does not appear, hand enter the correct information.

2.2.3.3 For MS, MSD, and DUP samples use the parent sample ID followed by the type of QC (e.g. : 11021401-01AMS or 11021401-01AMSD or 11021401-01ADUP).

2.2.3.4 There should be no more than twenty (20) samples in a batch (not including QC).

2.2.4 Fill in the sample volumes and/or weights used for the extraction.

2.3 To create a new batch

2.3.1 Click on the ADD button,

2.3.2 Choose the correct PREP CODE,

2.3.3 Enter the TECHNICIAN'S name,

2.3.4 Start adding samples (see F.10.2.2 above)

2.3.4.1 The system automatically adds a MBLK and LCS/LCSD set. If the LCSD is not required, Click on the far left of the LCSD row to highlight the entire line. Use the DELETE key to remove the entry.

2.3.4.2 A MS/MSD set will need to be added to each batch.

2.4 Click on the REAGENTS/SPIKES tab to enter the surrogate spike data

2.4.1 Enter the IDs from the spike mix containers. If the ID is not present on the list, also enter the spike name in the next cell.

2.4.2 Enter the correct sample type with which the spike is associated.

2.4.2.1 If the same volume and spike mix is used, then:

2.4.2.2 Use SAMP with those spike mixes which are added to everything (e.g. Internal Standards and Surrogates).

2.4.2.3 Use LCS for the mix associated with all of the LCS, LCSD, MS, and MSD QC.

2.4.2.4 If different volumes or mixes are used, add a separate line for each combination.

2.4.3 Enter the correct volume of spike added to each individual sample.

2.5 Click on the VIEW button, to return to the sample list.

2.6 Click in the first SPK ADDED cell and begin to add the spike information.

2.6.1 Enter the line number(s) from the associated reagent/spike table.

2.6.1.1 For example, if the LCS was spiked with three different spikes (IS, Surrogate, and Spike) that corresponded to the first three lines of the spike table, then the line numbers would be 1,2,3.

2.6.1.2 Either spaces or commas may be used as delineators between the different mixes.

2.6.2 If the same reagent is used throughout the entire batch, no notation is necessary for each sample.

2.6.3 If different reagents are used, notate the reagent line using alpha characters (A,B,C,etc.)

2.7 Any deviations from the normal methodology are to be entered in the comments line of the associated sample.

2.7.1 These deviations include such things as different initial or final volumes, spike problems, filtration problems, emulsions, spilled sample, etc.

2.7.2 If the samples are aqueous, measure the sample pH and add this value to the pH column.

2.8 Check all fields before printing.

2.8.1 Before printing, double click on the 2nd prep date line to complete the status of the preparation batch.

2.8.2 After all of the associated information is entered, click on the PRINT button.

2.8.3 Review all of the information on the print out. If everything is accurate initial and date the batch report.

2.8.4 Include a copy of the hard copy batch report in each of the associated work-order folders.

2.8.5 Place the original preparation batch report in the appropriate extraction batch binder.

Appendix G

Laboratory Ethics/Fraud Prevention and Data Integrity Program

Laboratory Ethics/Fraud Prevention and Data Integrity Program

I the undersigned, CERTIFY, that:

I have read, acknowledged and understand the personal ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions.

This includes, data integrity and/or data authentication issues such that the analytical process can be completely reviewed by recreating the paper trail.

Employee Name	Signature	Date
Senior Management Name	Signature	Date

1.0 Laboratory Ethics, Fraud Prevention and Data Integrity Program

1.1 The QA Program includes the components necessary to achieve acceptable data and assumes that personal behavior is ethical. Analytical methods, procedures and EPA programs do not set standards for ethical behavior in the laboratory and assumes that work being conducted in an analytical chemistry laboratory is of the highest integrity. This program is established to communicate to all employees what constitutes expected conduct, ethical behavior, unethical behavior and fraudulent behavior.

1.2 It is Alpha's policy to establish and maintain a Laboratory Ethics, Fraud Prevention and Data Integrity program. There are four required elements within our data integrity system. These are: 1) data integrity training, 2) signed data integrity documentation for all laboratory employees, 3) in-depth, periodic monitoring of data integrity, and 4) a data integrity SOP.

1.3 Data integrity training is provided as a formal part of new employee orientation and is conducted on an annual basis for all current employees.

2.0 Definitions

2.1 Data integrity - the act of conducting data analysis under a program that documents data analysis without any type of data manipulation, falsification or misrepresentation of the actual data results.

2.2 Ethical Behavior - behavior that conforms to accepted professional standards of conduct. Unethical behavior therefore is behavior not conforming to these standards of conduct.

2.3 Ethics - a set of moral principles or a code of right and wrong.

2.4 Fraud - an intentional act of deceit that may result in termination or legal prosecution.

2.5 Improper Actions - deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional.

2.6 Integrity - moral soundness, or honesty.

2.7 Laboratory Fraud - the deliberate falsification, with intent to conceal analytical or quality assurance results.

2.8 Unethical or Illegal Actions - the deliberate falsification of analytical or quality assurance results, where method or contractual requirements are made to appear acceptable.

3.0 Objectives

3.1 The objective of our Ethics, Fraud Prevention and Data Integrity Program is to prevent fraudulent or unethical employee behavior and to ensure ethics violations do not occur. The impact of unethical behavior and fraud could be devastating to our laboratory, our employees, as well as to the data users.

3.2 An effective and rigorous ethics program has been established in conjunction with our laboratory QA program to better ensure that employees act ethically and within the bounds of these programs.

3.3 The objectives of the Ethics and Fraud Prevention program have been established, and are carried out by the annual discussion and documented training of the following critical elements:

- Ethics education and discussion,
- Awareness and prevention of unethical or fraudulent acts, and
- Establishment of policies to prevent and handle problem situations.

3.4 Data integrity training is focused on the primary objective of creating an environment that fosters professional and scientific honesty.

4.0 Training Topics

4.1 Training topics are documented in the individual personnel training documents. Key topics covered include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting,
- How and when to report data integrity issues,
- Record keeping,
- Discussion of data integrity procedures,
- Data integrity training documentation, and
- In-depth data monitoring and data integrity procedure documentation.

4.2 The employees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, debarment or civil/criminal prosecution.

5.0 Laboratory Ethics/Fraud Prevention and Data Integrity Program

5.1 Examples of Inappropriate Practices

The QAM is written as a set of policies and procedures that defines what laboratory

personnel are required to do; however, the following policies are written to ensure that employees are educated as to what they are not allowed to do. The following table lists specific examples of unacceptable laboratory practices and our policy regarding each issue.

Unacceptable Laboratory Practice	**Laboratory Policy**
1. Dry Labbing Making up data such as creating data for an analysis that was not performed or creating information that is not true or was not collected.	Analytical results for all sample and Quality Control must be based on actual analysis that were performed. Documented data must match actual data (Data Integrity). Sampling information must be based on actual sampling events.
2. Time Traveling Resetting the internal clock on an instrument to make it appear that a sample was analyzed within a specified holding time when in fact it was not. Alternatively, changing the actual time or recording a false time to make it appear that holding times were met, or changing the time for sample collection, extractions or other steps to make it appear that they were performed at the correct time when in fact they were not.	The recorded date and the time of collection, preparation or analysis must match the actual date and time that the action was performed. Samples exceeding holding times are reported in a footnote or case narrative.
3. Improper Peak Integrations Improper baseline manipulation such as artificially subtracting (peak shaving) or adding (peak enhancing) peak area to produce an erroneous area that forces data to meet specific QC criteria when in fact the criteria were not met.	Instrument peaks must be consistently integrated and reporting according to proper techniques, generally baseline to baseline, valley to valley or a combination of the two. Peak area cannot be subtracted or added to force the data to meet specified criteria. Preventative or corrective action must be taken on instrument data not meeting required criteria.
4. File Substitutions Substituting previously generated runs from a non-compliant calibration or QC run to make it appear that an acceptable run was performed when in fact it was not.	All data must be generated and reported for actual analysis performed. Reported dates and times for all analysis must match actual dates and times. Substitution of files is not permitted.
5. Alteration of Analytical Conditions Improperly altering analytical conditions, such as changing instrument conditions for sample analysis from that used for standard analyses. Also using different procedures to process standards data or standard concentrations other than those used for samples.	All sample analyses must be performed under the same conditions as those used for standard analyses. All standard data must be processed by the same procedures as those used for processing sample data.

Unacceptable Laboratory Practice	Laboratory Policy
6. Improper Calibration a) Performing more than two calibrations or altering a calibration by eliminating one or more points, until one analysis barely meets criteria, rather than taking needed corrective action after the second failed analysis, and not documenting or retaining data for the other unacceptable data. b) Using the incorrect initial calibration to make calibration verification data appear to be acceptable when in fact it was not acceptable when compared to the correct initial calibration. c) Randomly discarding points in the initial calibration to force the calibration to meet the acceptance criteria. d) Discarding points from a Limit of Detection (LOD) study to force the calculated LOD to be lower than the actual value.	a) All calibration and QC data associated with sample analysis must be documented. Preventative or corrective action must be taken and documented if calibration and/or other QC criteria were not met. b) Acceptance of calibration verification data must be based in the correct initial calibration c) Calibration points can be rejected for inclusions in the calibration curve if a known or suspected error was made. When multiple target analytes are included in each calibration standard it may become necessary to discard selected upper or lower points for individual target analytes. Points can be discarded at the upper end of the curve if the linear range of the detector has been exceeded. For this case samples must be diluted that exceed the highest point of the calibration curve. Points can be discarded at the lower end of the curve if the detector is not producing a response. For theses cases reporting limit must be adjusted accordingly. d) Calibrations used for the production of samples must also be used to produce the LOD study data.
7. Misrepresenting Samples, QC Samples and Spikes a) Unjustified dilution of samples. b) Adding SV surrogates after sample extraction rather than prior to sample extraction. c) Reporting post-extracted spikes or duplicates as re-extracted spikes and duplicates. d) Not preparing or analyzing method blanks and Laboratory Control Samples (LCS's) the same way that samples are prepared and analyzed in order to make it appear that method blank or LCS results are acceptable when in fact they may not be.	Samples must be prepared, analyzed and reported according to appropriate procedures. a) Samples should be diluted for the sake of reporting non-detects. b) Surrogates must be added prior to sample extraction. c)Post extracted spikes and duplicates must be reported as post extracted and must not be misrepresented as pre-extracted spikes and duplicates. d) Method blanks and LCS's must be prepared and analyzed the same way that samples are prepared and analyzed. QC results outside of acceptance criteria are reported with a footnote or a case narrative.
8. Deletions of Non-Compliant Data Intentional deletion or non-recording of non-compliant data to conceal the fact that analyses such as calibration or QC were non-compliant.	All data associated with sample analysis, including any out of control events or non-compliant data must be documented and retained. Preventative or corrective action must be taken and documented for non-compliant data.

Unacceptable Laboratory Practice	Laboratory Policy
9. Manipulation of Computer Software Unwanted manipulation of computer software to force calibration or QC data to meet criteria.	Computer manipulation is allowed only for warranted reasons and any manipulation should be minimal and traceable.
10. Concealment of a Known Problem a) Concealing a known analytical or sample problem from laboratory management. b) Concealing a known improper or unethical behavior or action from laboratory management and failing to report the occurrence of a prohibited practice or known improper or unethical act to laboratory management.	a) Any knowledge of analytical or sample problems must be communicated to laboratory management and the client. b) Any knowledge of unethical behavior or actions must be fully communicated to laboratory management.

5.2 Most individuals do not personally gain from committing an unethical act except to relieve some pressure they feel, whether it is real or perceived. Therefore, education and communication are key elements for our program.

6.0 Relevant Criminal Laws

6.1 Unethical Behavior

An unethical action becomes a fraudulent act when the law is violated. For example, it is unethical if an analyst changes the instrument clock to make samples appear to be analyzed within holding time, when in fact they were not. It is also unethical to manipulate instrument calibration or QC samples to make the calibration or QC analysis meet an acceptance limit, when in fact the actual data was not acceptable.

6.2 Fraudulent Acts (wire fraud / mail fraud)

6.2.1 It becomes a fraudulent act when the falsified data is faxed, mailed or e-mailed. Faxing or mailing false information is an example of wire fraud or mail fraud, respectively, and the person or organization that does so could be charged with wire fraud or mail fraud, as well as making false statements if the work was done under a government contract.

6.2.2 The following is a list of relevant criminal laws all of which can result in substantial fines and possible imprisonment.

- False Claims - 18 U.S.C § 287
- False Statements - 18 U.S.C § 1001
- Mail Fraud - 18 U.S.C § 1341

- Wire Fraud - 18 U.S.C § 1343
- Conspiracy - 18 U.S.C § 371
- Mis-prison (concealment) of Felony - 18 U.S.C. § 4

7.0 Possible Penalties for Environmental Crimes

7.1 Laboratories can face these types of legal action for breaking the law:

- Administrative action - punishment which can result in debarment or probation;
- Civil Action - punishment which can result in large fines;
- Criminal Action - punishment which can result in prison sentences for company officials.

7.2 Individual who commit an unethical act and/or break the law can face the following types of action:

- Disciplinary actions up to and including termination from the job;
- Civil actions - punishment which can result in large fines;
- Criminal action - punishment which can result in prison sentences and/or probation sentences.

8.0 Zero Tolerance Policy

8.1 Alpha Analytical has a zero tolerance policy on unethical activities or fraudulent behavior. Unethical behavior includes, but is not limited to, the **intentional** falsification of the following:

- Data or records,
- Professional credentials,
- Employment records,
- Sampling or sample handling records,
- Laboratory worksheets,
- Analytical logbooks,
- Instrument settings,
- Sample results, and
- Laboratory analytical reports.

9.0 Data Integrity

9.1 One of our principal managerial guiding philosophies is to create and foster a work place culture that promotes professional and scientific honesty. Management

acknowledges its support of these issues by:

a) upholding the spirit and intent of all data integrity procedures, and

b) effectively implementing the specific requirements of the procedures.

9.2 Laboratory analysis is a human endeavor which requires a vast amount of professional decision making. Oftentimes these decisions are not black and white issues. Therefore, the documentation trail created during sample analysis is a critical data integrity element. Data integrity requires this paper trail to be complete in order for the data to be properly reviewed, evaluated and authenticated.

Analysts are encouraged to document non-perfect data results or instrument/sample abnormalities by a written narrative. These narratives, can be described as footnotes or other annotations that are written on data quantitation reports, corrective action reports, calibration summaries etc.

It is a laboratory policy to document clearly how all analytical results were obtained and to supply to the data user all relevant information.

9.3 Data integrity discussion items are the key sections found in the QA Manual, Volume I. Many of these items are expounded upon with additional detailed standard operating procedures typically found in QA Manul, Volume II. Data integrity training covers many topics and is continuously trained upon.

10.0 REPORTING

10.1 Alpha has a "no-fault"or what is commonly known as a "whistle-blower" reporting policy that encourages laboratory personnel to report suspected improper, unethical, or illegal activities, without fear of retribution. This no-fault policy is established to assure personal and scientific confidentiality and to provide a receptive environment in which all employees may privately discuss ethical issues or report items of ethical concern, if unethical activities or fraudulent behavior is suspected by a co-worker.

10.2 Laboratory management, to include the Laboratory Director, Laboratory Manager and QA Officer are the designated Data Integrity Officers to whom laboratory personnel may confidentially report suspected instances of improper, unethical, or illegal activities.

11.0 INVESTIGATIONS

11.1 If unethical activities or fraudulent behavior is discovered as a result of an internal audit or other discovery mechanism, a complete and comprehensive review is conducted by management.

11.2 The followup investigation, of these issues are handled in a confidential manner until such time as a follow-up evaluation, or other appropriate actions have been completed and the issues clarified.

11.3 All investigations are documented, and any investigation that results in findings of inappropriate activities will include disciplinary actions involved, corrective actions taken, and appropriate notifications of clients. The documentation of these investigations and actions taken are maintained for a minimum of five years.

Appendix H

State Certifications and Parameters of Analysis

State Certifications and Parameters of Analysis

H.1 The individual parameters and/or methods certified by each of the various state agencies are kept on file by the QA Officer.

H.2 Home State Certification

State	Certification Category	Status
Nevada Nevada Department of Conservation and Natural Resources Division of Environmental Protection Certification # NV00016	Waste Water Hazardous Waste Safe Drinking Water	Certified Certified Certified

H.3 Additional State Certifications

State	Certification Category	Status
Arkansas Arkansas Department of Environmental Quality Laboratory Certification Program Certification # 08-0622	Waste Water Hazardous Waste Safe Drinking Water	Certified Certified
California ELAP California Department of Public Health Environmental Lab Accreditation Program Branch Certification # 2019	Waste Water Hazardous Waste Safe Drinking Water	Certified Certified Certified

State Certifications and Parameters of Analysis

Additional State Certifications

State	Certification Category	Status
Kansas Kansas Department of Health and Environment Division of Environment Certification # E-10308	Waste Water Hazardous Waste Safe Drinking Water	Certified Certified Certified
Oregon Oregon Environmental Laboratory Accreditation Program Cert. # NV200001-006 Cert. # NV300001-007 (TPH only)	Waste Water Hazardous Waste Safe Drinking Water	Certified Certified Certified
Washington Washington Department of Ecology Certification #C21	Waste Water Hazardous Waste Safe Drinking Water	Certified Certified

National Primary Authority

ACCREDITED IN ACCORDANCE WITH nelac

Certification Accrediting

H.4 NELAP Certification

Primary Accrediting Authority	Certification Category	Status
California NELAP California Department of Public Health Environmental Lab Accreditation Program Branch Certification # 01154CA	Waste Water Hazardous Waste Safe Drinking Water	Certified Certified Certified

4.5 Department of Defense (DOD)

Accrediting Authority	Certification Category	Status
DoD-ELAP ACLASS ANSI-ASQ National Accreditation Board Certification # ADE-1426	Waste Water Hazardous Waste Safe Drinking Water	Certified Certified Certified