SAMPLING AND ANALYSIS PLAN
AND WORK PLAN
FOR
PHASE II ENVIRONMENTAL SITE ASSESSMENT

Old Goodwill
Tamuning, Guam

October 2018

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EA Project No. 15672.01

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Acronyms and Abbreviations

°C	degrees Celsius
CASRN	Chemical Abstracts Service Registry No.
COC	chain-of-custody
DISID	Department of Integrated Services for Individuals with Disabilities
DL	detection limit
DOT	Department of Transportation
DQO	data quality objective
DU	decision unit
EA	EA Engineering, Science, and Technology, Inc., PBC
EPCRA	Emergency Planning and Community Right-to-Know Act
ESA	Environmental Site Assessment
Guam EPA	Guam Environmental Protection Agency
ID	identification
ISM	incremental sampling methodology
MDL	method detection limit
mg/kg	milligram(s) per kilogram
mg/L	milligram(s) per liter
NA	not applicable
NS	not stated
PAH	polycyclic aromatic hydrocarbon
PAL	project action limit
PCB	polychlorinated biphenyl
PPE	personal protection equipment
PQO	project quality objective
QA	quality assurance
QC	quality control
QL	quantitation limit
RCRA	Resource Conservation and Recovery Act
REC	recognized environmental condition

Acronyms and Abbreviations (Continued)

SAP	Sampling and Analysis Plan
SIM	selected ion monitoring
SOP	standard operating procedure
TCLP	Toxicity Characteristic Leaching Procedure
TPESL	Tropical Pacific Environmental Screening Level
TPH	total petroleum hydrocarbons
USEPA	United States Environmental Protection Agency
USDA	United States Department of Agriculture
UST	underground storage tank

1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) presents the sampling approach, rationale, analyses, and field procedures for the Phase II Environmental Site Assessment (ESA) at the Old Goodwill site, which is now the proposed Rehabilitation Center for Guam Department of Integrated Services for Individuals with Disabilities (DISID) (herein referred to as the "subject site"). The SAP will guide the technical and quality aspects of the field, laboratory, and reporting activities. This Phase II ESA will be conducted by EA Engineering, Science, and Technology, Inc., PBC (EA) on behalf of the Guam Environmental Protection Agency (Guam EPA) Contract Request for Proposal No. 2015-001, dated 13 April 2018.

The project scope of work consists of the following tasks:

- Mobilizing and performing site preparatory activities.
- Collecting soil and product samples including quality control (QC) samples from the subject site to evaluate the nature and extent of potential contamination.
- Analyzing the samples collected for the identified parameters of concern.
- Surveying sample locations and site features at the subject site.
- Performing data validation and assessment on the collected data.
- Preparing a Phase II ESA report detailing field activities, deviations from this SAP if any, and recommendations for additional studies or remedial actions, along with associated rough order of magnitude costs.

1.1 Site Name or Sampling Area

The common name for the overall subject site is the Old Goodwill site ("subject site"). The subject site is located off Route 1, Marine Corps Drive, in Tamuning, Guam (Figure 1).

1.2 Site Location

The subject site is located off Route 1, Marine Corps Drive, in Tamuning across from John F. Kennedy High School and is south of Kmart located within Tamuning, Guam. The subject site is 5.7 acres in size containing three different Lots, as shown in Figure 2. The subject site (Lot No. 5138-2-R3 NEW) was parceled out creating two more lots: Lot Nos. 5138-2-R3 NEW-1 & 5138-2-R3 NEW-2. During this time, a semi-concrete building was built creating the vocational rehabilitation center on Lot No. 5138-2-R3 NEW-1-R2. Two other buildings were then built on Lot No. 5138-2-R3 NEW-1-2 including a car wash shed on Lot No. 5138-2-R3 NEW-1-1. The eastern boundary of the subject site falls within Lot 5138-2-R3 NEW-1 R/W. Lot 5138-2-R3 NEW-1-1, Lot 5138-2-R3 NEW-1-2 and Lot 5138-2-R3 NEW-1 R/W is a subdivision of Basic Lot 5138-2-R3 NEW-1.

1.3 Responsible Agency

Guam EPA is the responsible agency for the Phase II ESA activities. This Phase II ESA includes site characterization, and preparation of a Phase II ESA report that includes recommendations and a rough order of magnitude cost estimate for solid waste removal, if necessary.

1.4 Project Organization

The project organizational chart (Figure 3) presents the list of project personnel related to the implementation of field activities described in this SAP.

2.0 BACKGROUND INFORMATION

The following section presents background information including a site description, operational history, summary of the findings of the previous investigation, physical setting, and the potential source(s) of contamination at the subject site.

2.1 Site Area Description

The subject site is a irregularly-shaped parcel and contains three concrete buildings (Figure 2). The subject site is bounded to the east and cannot be accessed due to the large boulders placed outside of the property. The remaining boundaries are unmarked and partially fenced.

2.2 Operational History

The subject site was transferred to the Government of Guam and was established as a rehabilitation center in March 1988. In May 1993, a 20-year lease agreement was made between the Guam Rehabilitation and Workshop Center Inc., also known as Department of Vocational Rehabilitation and the Government of Guam. The facility was utilized as a Sheltered Workshop Training Center which included bookbinding, small engine repair, packaging of earphone sets for the former Continental Airlines, and a ceramic pottery shop. In August 2006, the subject site was administratively transferred over to DISID and is now under the jurisdiction of the Government of Guam.

In addition, the subject site was historically utilized by the Guam Rehabilitation and Workshop Center in 1966 with the purpose of providing work activities for people with disabilities. Work activities included food services training, school furniture repair, book binding for the library, manufacturing ceramic pottery, cabinet making, picture framing, construction of shipping crates for the military, engine repair services, construction of shipping crates for the military, and manufacturing of local arts and crafts.

The subject site is currently unoccupied. There are fences surrounding the subject site, and illegal dumping has occurred along the road side adjacent to the parcel.

2.3 Previous Investigations

A Phase I ESA was conducted during the period of May 2018 through September 2018. The Phase I ESA included a combination of historical research activities, interviews, site observations, and database records searches that were used to develop an understanding of the history and current environmental condition of the subject site (EA, 2018a).

The following recognized environmental conditions (RECs) were identified (EA, 2018a):

- Further investigation such as a Phase II ESA is warranted to address the potential for impact to the subject site from the former building that was damaged by fire.

- Further investigation is warranted regarding the vent pipe to determine its origin and use.

2.4 Physical Setting

The subject site is located on the U.S. Geological Survey Hagatña topographic quadrangle map and is approximately 120 to 125 feet above mean sea level (United States Geological Survey, 1984). The topography of the site is relatively flat. Regional topography slopes north toward Tumon Bay.

The subject site is located within the Reef facies (Water & Environmental Research Institute, 2007) and is underlain by the Pliocene and Pleistocene-age Mariana limestone.

Review of the United States Department of Agriculture (USDA) Soil Survey of Guam, dated 1988, indicates that the subject site is underlain by soils of the Guam-Urban land complex. The Guam-Urban land complex is well drained with 0 to 3 percent slopes (USDA, 1998).

There are no surface water features on or near the subject site. Northern Guam has a permeable limestone that supports a groundwater aquifer. Groundwater is relatively deep between 160 and 180 feet below ground surface. The groundwater flows in a general northwesterly direction.

2.5 Environmental and/or Human Impact

The RECs defined in the Phase I ESA and described above in Section 2.3 have potential impact to not only the environment, but also to site workers and users of the subject site.

Groundwater is not considered a completed pathway for potential contaminants as the depth to water is relatively deep (160 to180 feet).

3.0 PROJECT DATA QUALITY OBJECTIVES

An integral part of the SAP is the formulation of the project quality objectives (PQOs). The PQOs incorporate the elements of the United States Environmental Protection Agency (USEPA) data quality objective (DQO) process, which in turn consists of a series of seven planning steps that are designated to ensure the type, quantity, and quality of the environmental data used in the decision making are appropriate for their intended application. The DQO process is outlined in the guidance document entitled "Guidance on Systematic Planning Using the Data Quality Objectives Process" (USEPA, 2006).

The PQOs for this site are defined by covering the following elements: (1) who will use the data, (2) what are the project action limits, (3) what will the data be used for, (4) what type and matrix of data are needed, (5) how "good" the data need to be in order to support the environmental decision, (6) how much data are needed, (7) where, when, and how should the data be collected/generated, (8) who will collect and generate the data, (9) how will the data be reported, and (10) how will the data be archived.

This section documents the seven-step DQO process used to develop the PQOs in this SAP as well as the data quality indicators.

3.1 State the Problem

Based upon the recommendations and RECs identified in the Phase I ESA (EA, 2018a), contamination may be present in surface soil at the subject site. Chemical data will be collected to confirm the presence or absence of contamination, and if the project-generated wastes are suitable for the planned disposal options.

3.2 Identify the Decision

The following section identifies how environmental data will be used to meet objectives and to solve the problem.

The following principal study questions and alternative action have been identified.

1) Do concentrations of contaminants of potential concern (COPCs) in soil pose unacceptable risk to human health or the environment?
   - Alternative 1: No. Recommend no further action with regards to COPCs in soil.
   - Alternative 2: Yes. Recommend further remedial action to address residual contamination.

2) Are the potential waste (soil and tank contents) hazardous?
   - Alternative 1: No. Dispose of waste as nonhazardous on island.

- Alternative 2: Yes. Disposal options will be identified for the waste (i.e., on-island or off-island disposal).

3.3 Identify Inputs and Sources to the Decision

This section identifies data and information necessary to answer the study questions. The primary type of information needed to support the project decisions include laboratory analysis of soil and tank contents samples collected from the project site.

3.3.1 Target Analytes and Action Levels

The target analytes for the soil samples are based upon the historical site activities and include polycyclic aromatic hydrocarbons (PAHs) and metals, as well as testing for waste characterization, as identified in the following tables. The laboratory results from the soil samples collected will be compared to Fall 2017 Tropical Pacific Environmental Screening Levels (TPESLs) for both unrestricted land use (residential) and commercial land use direct contact exposure scenarios to determine whether impacts exist at concentrations at or above levels found to be protective of human health and the environment. These screening levels were selected based upon consideration of the potentially complete exposure pathways.

The screening levels for the parameters used to characterize the project-generated wastes (total and TCLP parameters and waste characteristics) are based upon the requirements of the receiving disposal facility and 40 Code of Federal Regulations Part 261.24 for soil and Part 279.11 for used oil specifications. The product sample will be tested for ignitability, total petroleum hydrocarbons (TPH) fuel fingerprint, polychlorinated biphenyls (PCBs) as Aroclors, and metals.

Tables 1 and 2 summarize the testing parameters along with the appropriate analytical methods, screening limits, and laboratory detection limits.

Table 1. Contaminants of Potential Concern Analytical Reference Limits and Evaluation

Analyte	CASRN	PAL[(a)]		Laboratory Detection Limits	
		Residential	Commercial/ Industrial	MDL	QL
Polycyclic Aromatic Hydrocarbons by SW8270C SIM (mg/kg)					
Acenaphthene	83-32-9	40	40	0.00047	0.0050
Acenaphthylene	208-96-8	5.5	5.5	0.00033	0.0050
Anthracene	120-12-7	1.4	1.4	0.00040	0.0050
Benzo(a)anthracene	56-55-3	3.3	3.3	0.00030	0.0050
Benzo(a)pyrene	50-32-8	3.6	5.9	0.00040	0.0050
Benzo(b)fluoranthene	205-99-2	11	21	0.00050	0.0050
Benzo(g,h,i)perylene	191-24-2	35	35	0.0010	0.0050
Benzo(k)fluoranthene	207-08-9	39	39	0.00076	0.0050
Chrysene	218-01-9	30	30	0.00035	0.0050
Dibenzo(a,h)anthracene	53-70-3	1.1	9.6	0.0012	0.0050
Fluoranthene	206-44-0	29	29	0.00029	0.0050
Fluorene	86-73-7	31	31	0.00049	0.0050
Indeno(1,2,3-cd)pyrene	193-39-5	11	31	0.00048	0.0050
1-Methylnaphthalene	90-12-0	0.89	0.89	0.00045	0.0050
2-Methylnaphthalene	91-57-6	1.9	1.9	0.00033	0.0050
Naphthalene	91-20-3	3.1	3.1	0.00031	0.0050
Phenanthrene	85-01-8	23	23	0.00035	0.0050
Pyrene	129-00-0	41	41	0.00035	0.0050
Polychlorinated Biphenyls as Aroclors by SW8082 (mg/kg) for Product Sampling Only					
Aroclor 1016	12674-11-2	NA	NA	NS	0.50
Aroclor 1221	11104-28-2	NA	NA	NS	0.50
Aroclor 1232	11141-16-5	NA	NA	NS	0.50
Aroclor 1242	53469-21-9	NA	NA	NS	0.50
Aroclor 1248	12672-79-6	NA	NA	NS	0.50
Aroclor 1254	11097-69-1	NA	NA	NS	0.50
Aroclor 1260	11096-82-5	NA	NA	NS	0.50
Total PCBs	1336-36-3	1.2	9.7	NS	0.50
Metals by SW6010B/6020/7471A (mg/kg)					
Arsenic	7440-38-2	24	95	0.15	0.20
Barium	7440-39-3	1,000	2,5000	0.14	0.20
Cadmium	7440-43-9	14	74	0.050	0.10
Chromium	7440-47-3	1,100	1,100	0.10	0.20
Lead	7439-92-1	200	800	0.060	0.10
Mercury	7439-97-6	4.7	70	0.0085	0.040
Selenium	7782-49-2	78	1,200	0.10	0.20
Silver	7440-22-4	78	1,200	0.030	0.10

Analyte	CASRN	PAL[(a)]		Laboratory Detection Limits	
		Residential	Commercial/ Industrial	MDL	QL

CASRN = Chemical Abstracts Service Registry No. MDL = method detection limit
mg/kg = milligrams per kilogram NS = not stated NA = not applicable PAL = Project Action Limit
QL = quantitation limit SIM = selected ion monitoring
Notes:
(a) PALs refer to Tropical Pacific Environmental Screening Levels for direct contact exposure to soils for residential and commercial/industrial land use (Fall 2017) for shallow soil (≤3 meters below ground surface) and groundwater is a current or potential source of drinking water (Table A) (Guam EPA, 2017).
(b) Analytes shown in bold and highlight have a PAL lower than or equal to the MDL and/or QL.

Table 2. Waste Characterization Analytical Reference Limits and Evaluation

Analyte	CASRN	Regulatory Levels*	Laboratory Detection Limits	
			MDL	QL
TCLP Metals by SW1311/6010C/7470A (mg/L)				
Arsenic	7440-38-2	5.0	0.060	1.0
Barium	7440-39-3	100	0.012	5.0
Cadmium	7440-43-9	1.0	0.0025	0.050
Chromium	7440-47-3	5.0	0.0060	0.10
Lead	7439-92-1	5.0	0.012	0.50
Mercury	7439-97-6	0.20	0.00030	0.0020
Selenium	7782-49-2	1.0	0.065	0.20
Silver	7440-22-4	5.0	0.0042	0.10
Characteristics				
TPH Fuel Fingerprint	NS	NS	NA	NA
Ignitability (flashpoint)	NS	<140	NA	NA

* Regulatory levels for the Toxicity Characteristic (40 Code of Federal Regulations Part 261.24).
CASRN = Chemical Abstracts Service Registry No. MDL = method detection limit mg/L = milligrams per Liter
NS = not stated NA = not applicable QL = quantitation limit
TCLP = Toxicity Characteristic Leaching Procedure TPH = total petroleum hydrocarbons

3.3.2 Data Collection

The strategies for collecting data are presented in detail in Section 4.0. The following samples are planned:

- Incremental sampling methodology (ISM) surface (0-6 inches) soil samples will be collected at the subject site. The ISM sample method provides a reliable estimate of the average concentration of analytes across a DU. By collecting increment samples using a systematic-random approach, distributional heterogeneity within the DU is reduced. The ISM sample better represents chemical characteristics of the DU. The subject site will be divided into five DUs (Figure 4). One ISM surface soil sample will be collected at each DU. In addition a duplicate and triplicate sample will be collected at one of the DUs for

quality control assurance/quality control (QA\QC) and to ensure that the initial sample approach is representative of site conditions. Each ISM surface soil samples will be collected using a dedicated EasyDraw Syringe®. The ISM surface soil samples will be analyzed for PAHs (USEPA Method SW8270C using selected ion monitoring [SIM] mode) and metals (USEPA Methods SW6010B/6020/7471A).

If concentrations of metals in a specific DU soil sample exceed the TPESLs then the remaining soil sample that was already collected and sent to the laboratory for that DU will be used to perform further testing of waste characterization. That soil will be analyzed for TCLP metals.

- If a UST (containing product) is found on the subject site then a petroleum product sample will be collected from the UST and analyzed for ignitability, TPH-fuel fingerprint, PCBs (USEPA Method SW8082), and metals (arsenic, cadmium, chromium, and lead).

3.3.3 Data Users

The data users will include the regulatory authorities Guam EPA and USEPA Region 9, and the project stakeholders, which also include the nearby residents and users of the area.

3.4 Define the Study Boundaries

The study area boundaries and environmental media that require data acquisition to support project decisions are defined below.

The vertical boundary of the study area for soil sampling is bedrock. No temporal boundary exists since the concentrations of contaminants are not likely to change over the project duration. The horizontal boundary of the subject site is shown in Figures 2 and 4. Various environmental media at the subject site will be sampled and analyzed, including surface soil and petroleum product.

3.5 Develop a Decision Rule (If/Then Statements)

Rules defining the conditions that would cause the decision maker to choose among alternative response actions are stated below.

- If contaminant concentrations in the soil samples are below the TPESLs, then the extent of contamination has been identified. If the contaminant concentrations are at or above the TPESLs, then further investigation may be recommended.
- If the contaminant concentrations in waste characterization samples (soil [samples already collected and initially held by the laboratory] and product in UST, if applicable) are below the site-specific action levels, then the waste may be disposed of as nonhazardous. If contaminant concentrations are at or above the site-specific action levels, then alternative disposal options will be found for the waste (i.e., on-island or off-island disposal).

3.6 Specify Limits on Decision Error (Performance or Acceptance Criteria)

This section evaluates the consequences of making incorrect decisions and considerations and/or actions taken to mitigate decision error.

3.6.1 Decision Error and Potential Consequences

The acceptable limits for false positive or false negative decision errors will be based on evaluating the potential consequences of these decision errors (such as risks to human health and the environment or unnecessary expenditures for additional sampling) if specific contaminants are detected or are not detected above action levels.

Two potential decision errors could be made based upon interpreting the results of sampling and analytical data:

1. Concluding that concentration of a specific chemical at a sample location within an area is below the action level when it truly is at or above the action level.
2. Concluding that concentration of a specific chemical at a sample location within an area is greater than or equal to the action level when it truly is below the action level.

The consequences of the first error could result in unacceptable risk to human health or the environment because contaminants would be left onsite at concentrations at or above the action levels. The consequences of the second error would result in unnecessary expenditure, and diversion of resources that could be used for cleanup of other contaminated areas.

The consequences of the first error are deemed more serious because of the potential risk. The baseline condition, therefore, is established such that the contaminant concentration is truly greater than or equal to the action level. The baseline condition is defined as the null hypothesis (H_0). The alternative is defined as the alternative hypothesis (H_a). This may be summarized as follows:

H_0: [concentration] $\geq$ action level
H_a: [concentration] $<$ action level

A false positive error, also known as a Type I error, occurs when the null hypothesis is falsely rejected (*i.e.*, the sample data shows that the concentration of a chemical is below the action level when it actually is at or exceeds the action level). The measurement of the size of this error is called alpha (α), the level of significance. Alpha is expressed numerically as a probability or the tolerance for uncertainty.

A false negative error, also known as a Type II error, occurs when the null hypothesis is falsely accepted (*i.e.*, the sample data shows that the concentration of the chemical is at or above the action

level when it actually is below the action level). The measurement of the size of this error is called beta (β), or the complement of the power of the hypothesis test.

The tolerance limits for decision error have been established at α=5% or 0.05 for false positives and β=20% or 0.2 for false negatives.

The analytical data and sampling design performance will be statistically evaluated based on the detected contaminant concentrations at the subject site.

3.6.2 Sources of Error

Total study error potential is equally attributable to sampling and measurement error because of the steps and sample volume associated with the planned sample collection and analysis. Successfully managing the magnitude of total study error is the result of understanding the error sources, generating an appropriate sampling design, and choosing accurate measurement techniques. The approach used to manage study error for the planned sampling and analysis is discussed below.

- *ISM Soil Samples and Product Samples*—the source of decision error for these results are equally attributable to sampling or measurement error. This conclusion is based upon review of the sampling and analysis strategy. The sampling design uses the ISM sampling approach for soil. Analysis will be performed using the services of a National Environmental Laboratory Accreditation Program accredited laboratory with standard USEPA SW-846 methods.

The quality of sampling and analysis must be at a level that results in representative, precise, and reproducible data. The data generated will be sufficient for the intended use. "Good" data will be defined as data that are produced following the specified standard operating procedures (SOPs) and meeting the established criteria in this SAP, including precision, accuracy, comparability, representativeness, completeness, and sensitivity.

The analytical data that are not qualified or qualified but not rejected (R-flagged data) are deemed acceptable for project use. The project data will be assessed by the Project Chemist.

3.7 Optimize the Design for Obtaining Data and Conclusions

ISM sampling will be used for the soil samples collected at the subject site. This method is well suited to confirm if the RECs identified in the Phase I ESA are present at the subject site.

The ISM sampling approach is for sites that require representative results in clearly specified DUs and is beneficial for data reproducibility and as an initial screening tool; these descriptors apply to the project presented in this SAP. Further details on the ISM sampling approach are presented in Section 4.0.

The data will be presented in a Phase II ESA Report. The report will include a detailed narrative of each field activity and a summary of the sampling and analyses conducted. Site drawings, figures, survey coordinates, photographs documenting field activities, and disposal documentation for wastes that may be generated during the field investigation will be included as attachments to the report. The analytical data will be reported in summary tables to facilitate data analysis. The Phase II ESA Report will be comprehensive in nature and no additional sources of information will be necessary to capture the full extent of the field operations and data collected.

The electronic data deliverables and the laboratory data reports will be collected in project archives in existing electronic formats provided by the analytical laboratory. These will include the executable files delivered by the laboratory.

3.8 Summary of Data Quality Indicators

Precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity are the data quality indicators used to assess the data produced during the project. Each data quality indicator is described below, including a definition of the terminology and the referenced process for calculating the indicator.

3.8.1 Precision

Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance, percent difference, or range, in either absolute or relative terms. The QC measures for precision include field duplicates, laboratory duplicates, matrix spike, matrix spike duplicates, analytical replicates, and surrogates. The laboratory will perform matrix spike and matrix spike duplicate analysis at a rate of 1 set per 20 discrete surface soil samples.

Precision may be the result of one or more of the following: field instrument variation, analytical measurement variation, poor sampling technique, sample transport problems, or spatial variation (heterogeneous sample matrices). The field sampling design rationale and sampling techniques will be evaluated, and both field and analytical duplicate/replicate sample results will be reviewed to identify the cause of imprecision. The laboratory may be the source of error if poor precision is indicated in both the field and analytical duplicates/replicates. However, the sampling technique, field instrument variation, sample transport, and/or spatial variability may be the source of error if poor precision is limited to the field duplicate results.

3.8.2 Accuracy/Bias

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) that are

due to sampling and analytical operations. Examples of QC measures for accuracy include matrix spikes and laboratory control samples.

3.8.3 Representativeness

Representativeness is the measure of the degree to which data accurately and precisely represent a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition. Replicates will be collected for the ISM soil samples to verify the representativeness of the data collected. Additional scoping meetings and subsequent resampling may be needed in order to collect data that are more representative of a non-homogeneous site if field replicates precision checks indicate potential unacceptable spatial variability.

3.8.4 Comparability

Comparability is the degree to which different methods, data sets, and decisions agree or can be represented as similar. Comparability describes the confidence (expressed qualitatively or quantitatively) that two data sets can contribute to a common analysis and interpolation. The samples will be collected using SOPs and the analysis of samples will be performed using USEPA standardized methodology to meet the needs of the data users.

3.8.5 Sensitivity and Quantitation Limits

Sensitivity is the capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. Examples of QC measures for determining sensitivity include laboratory fortified blanks, method detection limit (MDL) studies, and low-level calibration standards.

The laboratory will establish a detection limit (DL), typically the MDL, using a scientifically valid and documented procedure. The MDL is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The DL is the laboratory's "best case" sensitivity for a given analytical method. The laboratory may establish MDLs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project using the statistical method presented in the 40 Code of Federal Regulations Part 136.

3.8.6 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under correct, normal circumstances. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. Completeness measures the

effectiveness in sample collection, analysis, and result reporting of the entire investigation, and is calculated on a per-analyte basis by the following equation:

$$\%Completeness = \frac{Number\ of\ valid\ results}{Number\ of\ possible\ results} \times 100$$

The numerator of this calculation becomes the number of possible results minus the number of possible results not reported for any instances of samples that could not be analyzed for any reason (holding time violations in which re-sampling and analysis were not possible, samples spilled or broken, etc.).

A completeness check will be done on the data generated by the laboratory. For this project, 90 percent of usable sample data is considered the minimal acceptance criteria for completeness; the goal is to achieve 100 percent completeness.

4.0 SAMPLING RATIONALE

Further investigation is warranted to address the potential for impact to the subject site from the former building which was damaged by fire and the vent pipe to determine its origin and use.

The number of samples to be collected at the subject site are summarized in Table 3 and discussed further below.

Table 3. Sample Summary

Parameter	Analytical Method	Sample Type	Field Samples(1)	Field Replicates
Surface ISM Soil Samples (DU1 through DU5)				
Polycyclic Aromatic Hydrocarbons	SW8270C SIM	ISM	5	2
RCRA Metals	SW6010B/6020/7471A	ISM	5	2
Waste Characterization				
Toxicity Characteristic Leaching Procedure Metals	SW1311/6010B/7470A	ISM	5	2
Petroleum Product Sample				
Ignitability	SW1010A	Grab	1	0
Polychlorinated Biphenyls as Aroclors	SW8082	Grab	1	0
Total Petroleum Hydrocarbons fuel fingerprint	SW8015B	Grab	1	0
Metals (arsenic, cadmium, chromium, and lead)	SW6010B	Grab	1	0

Notes:
NA = not applicable
ISM = incremental sampling methodology
SIM = selected ion monitoring
(1) Applies to samples collected using dedicated sampling equipment.

4.1 Incremental Sampling Methodology

ISM surface soil samples will be collected in each DU to assess if a potential impact to soil had occurred related to the subject site. Each DU will be divided into a 30 grid sub-sample location, and one increment of the sample will be collected from each of these locations. One ISM surface soil sample will be collected from each of the five DUs. In addition, a duplicate and triplicate sample will be collected at one of the DUs to ensure that the initial sample approach is representative of site conditions. The locations of the samples will be surveyed after the samples are collected as described in Section 6.5. Approximate locations of the proposed soil sample locations are shown in Figure 4.

The ISM surface soil samples will be analyzed for the following:

- PAHs
- Resource Conservation and Recovery Act (RCRA) metals

- TCLP metals (if soil sample metals results exceed TPESLs).

4.2 Petroleum Product Samples

If the vent pipe leads to a UST found during exploratory activities, field scientist will determine if there is product within the UST. If there is product, one petroleum product sample is proposed for the subject site. The petroleum product sample will be collected from the UST and analyzed to determine waste characteristics for disposal options.

The waste characteristic petroleum product sample will be analyzed for the following:

- Ignitability
- PCBs
- Metals (arsenic, cadmium, chromium, lead)
- TPH fuel fingerprint.

5.0 REQUEST FOR ANALYSES

The following section describes the preparation and analysis that will be performed on the samples to be collected at the subject site.

The laboratory quality assurance (QA)/QC criteria have been reviewed and meet the project requirements. The laboratory QA Manual and SOPs for the analytical tests scoped within this project are provided in Appendix A, "Analytical Quality Assurance Manuals and Standard Operating Procedures."

5.1 Laboratory ISM Sampling Procedures

The ISM samples will be properly containerized and submitted to TestAmerica located in West Sacramento, California under standard chain-of-custody (COC) procedures for processing prior to analysis.

Each ISM sample will be air dried then sieved to less than 2 millimeters particle size. Sub-sampling may be accomplished with a sectorial splitter (also called a rotary riffle splitter), or a representative subsample may be hand collected by taking approximately 30 small increments from systematic random locations from the dried and sieved sample spread out in a thin layer. Sub-sampling is used to provide a representative laboratory subsample (and any laboratory replicates) for a single ISM sample, and to provide representative sub-samples for multiple analyses. The mass of sample needed for the subject analytical test or tests is used to determine the parameters for splitting the sample with the sectorial splitter, or in selecting the mass of each increment if hand collecting the sub-sample. It is critical that the entire mass of dried and sieved sample is used in the sub-sampling process.

The laboratory will verify that the maximum sub-sample mass for preparation and analysis of soil samples is used to reduce inherent sources of fundamental error. For analyses of fine particulates (e.g., <250 micrometers), a 1-gram sub-sample may be adequate to reduce fundamental error below 15 percent; however, if a larger mass may be reliably run by the method (e.g., 2 to 10 grams), a larger mass will be used to help reduce potential error.

5.2 Grab Sampling Procedure

A liquid grab sample (petroleum product) will be collected in the field from the UST, properly containerized and shipped to TestAmerica located in West Sacramento, California for analysis under standard COC procedures, if applicable.

5.3 Analytical Methods

The analytical methods to be performed for the planned samples are presented in Section 4.0.

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6.0 FIELD METHODS AND PROCEDURES

The following section describes the methods and procedures to be used during the Phase II ESA field investigation.

6.1 Standard Operating Procedures

The SOPs (EA, 2018b) to be used in support of this project are presented in Appendix B of this SAP and are listed below:

- EA SOP No. 001 - Sample Labels
- EA SOP No. 002 - Chain-of-Custody Form
- EA SOP No. 004 - Sample Packing and Shipping
- EA SOP No. 057 - Multi-Incremental Sampling (known as ISM Sampling).

6.2 Field Activities

6.3 Soil Samples - Incremental Sampling Methodology

ISM surface soil samples will be prepared by collecting a minimum of 30 small increments of soil from the specified DU using a dedicated EasyDraw Syringe® for each DU and combining these increments into a single sample, referred to as the ISM sample. Individual soil increments will weigh approximately 30 grams, with the field ISM sample weighing 900 grams and providing mass sufficient to minimize fundamental error for sample collection after sieving soil samples to the target particle size. Note that sieving of soil samples to the less than 2-millimeter particle size, will be performed in the laboratory during the sample preparation process by TestAmerica.

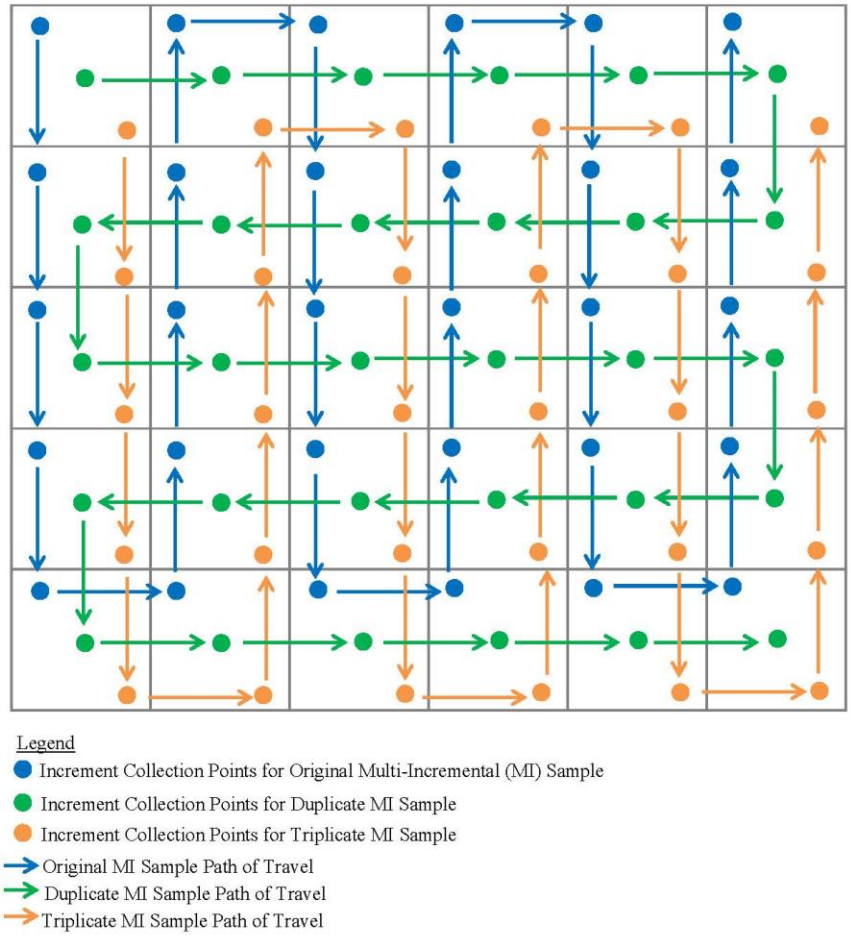
To collect the ISM sample, a systematic-random sample collection scheme will be utilized. Each DU will be divided into 30 equally sized sub-sample locations (Figure 5). The ends of each row and column will be marked with flags to help establish approximate lines for the collection of increments. Thirty individual increment samples will then be collected by collecting one ISM sample at the start of each rows and columns, if feasible. The exact distance does not need to be individually measured.

Replicate (one duplicate and one triplicate) ISM surface soil samples will be collected at a randomly selected DU. The duplicate and triplicate ISM surface soil samples from the same DU will be collected following a different path, as shown in Figure 5.

Individual incremental samples will be collected by using a dedicated EasyDraw Syringe® to collect a 30-gram increment that will then be transferred into a 1-gallon size bulk resealable bag (i.e., Ziplock™ bag) to produce the ISM sample. Soil samples will not include rocks, pebbles, or other non-soil material. It is not necessary to decontaminate the sampling tool between the increments within a DU.

Sample bags will be closed as soon as they are filled, placed on ice and chilled to 6 degrees Celsius (ºC), and processed for shipment to the laboratory. The laboratory will homogenize the sample from the resealable bag for analysis.

Figure 5. ISM Sampling in a Decision Unit



6.4 Petroleum Product Samples

Heavy equipment will be used to perform exploratory (excavation) activities to determine if the vent pipe previously identified on-site leads to a UST. If a UST is found during excavation activities, field scientist will determine if there is product within the UST by opening an existing fill port. If product is found in the UST, a liquid grab sample will be collected from a dedicated sampling bailer and placed in a wide-mouth glass jar with Teflon™-lined lid. Sample containers will be closed as soon as they are filled, placed on ice and chilled to 6ºC, and processed for shipment to the laboratory.

6.5 Equipment Decontamination

Dedicated/disposable equipment will be utilized for the sampling activities. Dedicated/disposable equipment intended for one-time use will not be decontaminated and will be packaged for appropriate disposal. Because dedicated equipment will be used during field sampling activities, it is anticipated that no rinsate water will be generated and no equipment blank will be collected.

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7.0 SAMPLE CONTAINERS, PRESERVATION, AND STORAGE

The recommended sample container type and volume, initial preservative, and holding times for the analytes that will be tested are shown in Table 4. The sample containers will be obtained from the analytical laboratory in pre-cleaned condition.

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Table 4. Sample Storage and Preservation Requirements

Parameter	Analysis Method(s)	Holding Time	Container(s)	Storage Requirements
Soil Samples				
PAHs	SW8270C SIM	14 days until extraction, 40 days to analyze extract	(1) 1-gallon size bulk resealable bag (i.e., Ziplock™ bag)	≤6°C
RCRA Metals	SW6010B/6020/ 7471A	180 days until analysis for metals with the exception of mercury (28 days)	(1) 1-gallon size bulk resealable bag (i.e., Ziplock™ bag)	≤6°C
TCLP Metals	SW1311/6010B/ 7470A	180 days until TCLP preparation for metals with the exception of mercury (28 days) 180 days to analyze TCLP leachate for metals with the exception of mercury (28 days)	(1) 1-gallon size bulk resealable bag (i.e., Ziplock™ bag)	≤6°C
Petroleum Product Samples				
PCBs as Aroclors	SW8082	14 days until extraction, 40 days to analyze extract	(1) 4-ounce wide-mouth glass jar with Teflon™-lined lid	≤6°C
Metals (arsenic, cadmium, chromium, and lead)	SW6010B	180 days until analysis for metals	(1) 4-ounce wide-mouth glass jar with Teflon™-lined lid	≤6°C
TPH Fuel Fingerprint	SW8015B	14 days until extraction, 40 days to analyze extract	(1) 4-ounce wide-mouth glass jar with Teflon™-lined lid	≤6°C
Ignitability	SW1010A	none	(1) 4-ounce wide-mouth glass jar with Teflon™-lined lid	≤6°C

Notes:
°C = degrees Celsius PAH = polycyclic aromatic hydrocarbon PCB = polychlorinated biphenyl RCRA = Resource Conservation and Recovery Act
TCLP = Toxicity Characteristic Leaching Procedure TPH = total petroleum hydrocarbons

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8.0 DISPOSAL OF RESIDUAL MATERIALS

The investigation-derived wastes generated during the proposed investigations may include:

- Used personal protection equipment (PPE)
- Dedicated sampling equipment.

Used PPE and dedicated sampling equipment will be placed in a trash bag and disposed of offsite in a municipal refuse dumpster. These wastes are not considered hazardous and shall be sent to the municipal landfill.

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9.0 SAMPLE DOCUMENTATION AND SHIPMENT

9.1 Field Notes

This section discusses required recordkeeping in the field, which will consist of the use of field logbook, photographs, and pre-printed forms.

9.1.1 Field Logbooks

The information recorded in field logbooks will document sample locations, sampling dates, sampling procedures, and names of field personnel responsible for conducting the sampling activities. Logbook entries will also include descriptions of the field activities. Logbooks will be bound with consecutively numbered pages. Each page will be dated, and the time of entry will be noted in military time. The entries will be legible, written in black ink, and signed by the individual making the entries. Language will be factual, objective, and free of personal opinions. At a minimum, the following information will be recorded, as appropriate, during sampling activities:

- Time of arrival/entry on site and time of departure
- Other personnel on site
- A brief summary of meetings or discussions with any potentially responsible parties, or representatives of any federal, state, or other regulatory agency
- Deviations from sampling plans, safety plans, and QA procedures
- Records of photographs
- Sample location and description
- Site sketch showing sample location and measured distances
- Sampler's name
- Date and time of sample collection
- Designation of sample
- Type of sample (matrix)
- Field observations and details important for laboratory analysis or integrity of samples (e.g., sediment grain size, rain, odors, etc.).

If a correction needs to be made to a field logbook, draw a single line crossing-out the error and place an initial of the person making the change.

9.1.2 Photographs

Photographs will be taken at representative sample locations and at other areas of interest on subject site. A photograph log will be maintained in the field logbook.

9.2 Sample Custody and Documentation

Sampling information will be recorded on a COC record and in a permanently bound field logbook. The entries will be legible and recorded in indelible black ink. The requirements presented in EA SOP No. 002 (2018b) (Appendix B) will be followed for completing the COC record.

9.3 Sample Identification

EA SOP No. 001 (2018b) (Appendix B) will be followed for sample labeling. A sample identification (ID) system has been developed to provide uniform classification and to assist project personnel to interpret data reports and field notes. Sample identification numbers will be affixed to each sample container and entered on the COC record. The sample number will uniquely identify the sample to a specified location.

Soil Description Sample

For example: OGW-S001

- The first three characters (OGW) represent the site name (Old Goodwill).
- The next character (S) represent a soil medium
- The next three digits (**001**) represent the sequential sample number.

Product Description Sample

For example: OGW-P001

- The first three characters (OGW) represent the site name (Old Goodwill).
- The next character (P) represent a petroleum product medium
- The next three digits (**001**) represent the sequential sample number.

For samples requiring multiple containers, a single sample number will apply to every container for that sample. The sample number, along with the date and time of sample collection, and the type of sample collected will be recorded in the field logbook, on the sample log sheet, and on the sample label affixed to every container and entered on the COC record.

9.4 Sample Packaging and Shipping

The laboratory will supply sample containers and appropriate preservation additives, if needed. On-site personnel will be responsible for ensuring that adequate sample containers are available for the work scheduled at the sample collection points. The sample containers will be bubble-wrapped, taped for shipping, and placed in coolers with ice and chilled to $\leq$6°C (not frozen) for transport to the laboratory after the appropriate labeling and COC records are completed. The procedures to be followed for sample packaging and shipping are presented in EA SOP No. 004 (2018b) (Appendix B).

Environmental samples from this project will be packaged and shipped in a manner that will ensure the safety and accountability of each sample, and all procedures will be in accordance with applicable federal and local requirements (i.e., USDA permit requirements for shipping soil samples). The persons packing and shipping environmental samples will review and be aware of state, federal, Department of Transportation (DOT), and International Air Transport Association regulations governing environmental and hazardous sample packaging. The person(s) shipping the samples is responsible for being in compliance with applicable packaging, labeling, and shipping requirements.

9.5 Chain of Custody

COC documentation is required for each sample to track collection, shipments, laboratory receipt, custody, and disposal. The COC record is preprinted with appropriate space for the applicable data to be entered.

Each individual who has the samples in their possession will sign the COC record. A sample is considered to be in custody under the following conditions:

- It is in actual possession or in view of the person who collected the sample
- It is locked in a secure area
- It is placed in an area restricted to authorized personnel.

Each sample will be assigned a unique sample ID number, which will be entered on the COC record. Samples to be transported to an off-site laboratory by a courier service will have the courier name and/or airbill number noted on the COC record. As a final step, custody seals are attached to the front and back of the lid of the shipping container. The samples in the cooler are checked against the COC record by laboratory personnel upon arrival at the laboratory. The samples in question will be segregated and field personnel will be immediately notified if discrepancies are noted. The person accepting the delivery will sign and date the COC record.

9.6 Sample Shipment

The samples will be transported or shipped to the analytical laboratory in insulated containers within the appropriate holding time and will be accompanied by a COC record that identifies the sample bottles, date and time of sample collection, and analyses requested. Shipped samples will be packaged and shipped in accordance with DOT standards with International Air Transportation Association requirements. The original COC record will be given to the lab with the samples and a copy will be retained in project records. Once received by the laboratory, a sample receipt and storage record will be generated.

10.0 QUALITY CONTROL

This section details the QC samples that are to be collected to support the sampling activities. Field QC is intended to provide an assessment of possible field contamination and assessment of field variability. The latter may include variability in sampling techniques and instrument variability.

10.1 Temperature Blanks

For each cooler that is shipped or transported to an analytical laboratory, a container filled with water will be included that is marked "temperature blank." This blank will be used by the laboratory to check the temperature of samples upon receipt.

10.2 Field Replicates

Field replicates will be taken for ISM sampling efforts. ISM field replicate samples will be collected at a one randomly selected DU location of the five DUs (DU1, DU2, DU3, DU4, and DU5).

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11.0 FIELD VARIANCES

As conditions in the field may vary, it may become necessary to implement minor modifications to sampling as presented in this plan. For example, a petroleum product sample will not be collected if there is no UST present or if a UST is present but contains no liquid in the UST. When appropriate, the client representative will be notified, and a written approval will be obtained before implementing the changes. Modifications to the approved plan will be documented in the Phase II ESA Report.

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12.0 FIELD HEALTH AND SAFETY PROCEDURES

A Site Health and Safety Plan has been prepared to minimize the threat of serious injury to workers engaged in sampling activities while performing site work. The Site Health and Safety Plan is presented in Appendix C.

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13.0 ENVIRONMENTAL PROTECTION PLAN

The following sections describe the environmental protective measures that will be used to control and correct conditions that may develop at the subject site during fieldwork associated with this project. Protective measures for this project will include erosion and sediment controls and work practices that minimize damage to site features and adjacent vegetation.

Shipping procedures that will prevent the accidental transportation of plants, insects, or animals will be used for soil samples shipped out of Guam. The samples will be shipped in sample coolers with a USDA foreign soil permit affixed that allows the selected laboratories to receive samples and the Contractor's USDA Compliance Agreement for the shipping of soil samples. Samples will be packed to comply with International Air Transportation Association requirements.

This Environmental Protection Plan provides site-specific information for the following:

- Emergency Planning and Community Right-to-Know Act (EPCRA) requirements
- Protection of natural resources (land, air, water, and fish and wildlife)
- Archeological and cultural resources preservation
- Erosion and sediment control
- Spill prevention.

This plan will be implemented in accordance with applicable federal, state, and local laws, regulations, and permits for protecting the environment.

13.1 Emergency Planning and Community Right-to-Know Act Reporting Requirements

Reporting requirements established for hazardous chemicals in accordance with the EPCRA regulation (USEPA, 2012), also known as Title III of the Superfund Amendments and Reauthorization Act of 1986, will be followed at the subject site. A complete list of hazardous chemicals used for this project, which will meet the EPCRA requirements, will be completed and maintained. The list will include the following:

- Trade and/or chemical name
- Chemical Abstract Service Registry Number
- Classification
- Reportable quantity
- Maximum volume at subject site
- Average daily volume at subject site
- Total volume throughout project.

Hazardous substances will be stored in accordance with applicable regulations.

13.2 Protection of Natural Resources

Project work will be performed within the subject site area shown on Figures 2 and 4. Except for designated work areas, the subject site will be preserved in its original condition. Field activities will be limited to the areas described in this SAP.

Intrusive activities will be conducted in a manner that minimizes impact and protects the surrounding areas from being disturbed. The following sections detail precautions to be taken to minimize impacts.

13.3 Protection of Land

During minimal intrusive activities, efforts will be made to minimize the impact to vegetation outside the designated work areas, storage areas, and access routes. Precautions taken to minimize the impact of field activities on the existing vegetation will include the following:

- Limit vehicle operation to designated roadways and predetermined routes
- Collect debris from work activities daily.

Based on available information, no species have been identified that require special protection at this time.

13.3.1 Protection of Air Resources

Every effort will be made to prevent and limit the spread of emissions and dust to the maximum extent possible to avoid creating a nuisance and/or hazard to workers and personnel in the surrounding area. This includes limiting and/or controlling emissions at their source.

The following steps and processes will be followed to control dust from field activities at the subject site:

- If dust is observed caused by wind, dust suppression using water will be implemented as an engineering control.
- To control engine exhaust from field activities at the subject site, field vehicles will be properly maintained to ensure that no unusually heavy emissions are generated during project activities and will be turned off when not in use.

13.3.2 Protection of Water Resources

Minimal intrusive activities will be conducted in a manner to prevent chemicals, fuels, oils, grease, and contaminated material from entering potential nearby surface water and to minimize infiltration of contaminants into groundwater. Erosion and sediment control measures will be implemented to minimize the potential for discharges of waste impacted storm water, if deemed

necessary during field conditions. Absorbent material will be available for quick response to a release of fluids other than clean water.

Should a leak occur, absorbents will be used to prevent runoff onto the surrounding area.

13.3.3 Protection of Fish and Wildlife Resources

Precautions will be taken during project site activities to minimize disturbances to fish and wildlife and their habitat adjacent to the subject site. Project personnel will minimize the actual work area as much as possible and practical to minimize the impacts to wildlife. Based on the Phase I ESA site assessment information, no species have been identified that require special protection at this time. No surface bodies of water are found near the subject site.

13.4 Archeological and Cultural Resources Preservation

There are no archeological or cultural resources believed to be located at the site, based on the Phase I ESA land use and background data for this project site. In the event that archaeological resources are encountered, measures will be taken to carefully preserve and immediately report these findings to the Government of Guam. If archaeological resources such as artifacts (e.g., stone tools), features (e.g., stone walls), deposits (e.g., sea shells and charcoal-stained soil), human bones, and other cultural remains are encountered, that portion of the work will immediately cease, and the Government of Guam will be immediately notified.

13.5 Spill Prevention

Measures will be taken to avoid spills. Spill control techniques will be used that may include temporary soil berms, surface cover mesh, and/or sandbags.

13.5.1 Contingency Plan

In the event of a spill, the Contractor will notify the authorities immediately.

The following actions/measures will be implemented during a spill response action:

- Take immediate measures to control and contain the spill using above mentioned equipment and materials
- Isolate and contain hazardous spill areas
- Deny entry to unauthorized personnel
- Do not allow anyone to touch spilled material
- Stay upwind
- Keep out of low areas

- Keep combustibles away from the spilled material
- Use water spray to reduce dust, as needed
- Perform cleanup activities as directed by using certified personnel
- If released from tanks, prevent discharge beyond site boundaries
- Any other actions as needed.

If a spill occurs on the ground:

- Dig up contaminated soil, placing it onto a layer of plastic.
- Cover the hole and pile of soil in a manner that will prevent any water from entering the covered area; the cover must be replaced at the end of each day
- Collect soil samples from the hole and the soil pile.
- Provide the results to Guam EPA.
- Guam EPA will review the sample results from the hole and advise if further excavation is needed or the hole may be filled with clean soil.
- Dispose of the stockpiled soil in accordance with Guam EPA based on the sample results.
- Provide Guam EPA a letter stating the disposition procedure and location of the soil.

If the spill occurs on pavement or asphalt:

- Place absorbent down to clean up the entire product.
- Place all the absorbent into an open top drum which meets all DOT requirements for the contents.
- Collect a sample of the spill residue and dispose of the material in accordance with Guam EPA requirements based on sample results.
- Provide Guam EPA with a letter stating the disposition procedure and location.

13.5.2 Notification of Spills and Discharges

Spill notifications under federal, state, and local regulation will be made immediately upon discovery of a spill or discharge. The Contractor will notify the appropriate authorities. A report, submitted no later than 24 hours after a release, will include the following items:

- Description of material spilled, including identity, quantity, and a copy of the waste disposal manifest
- Exact time and location of the spill, and the description of the area involved
- Containment procedures utilized

- Description of the cleanup procedures employed at the site, including disposal of spill residue
- Summary of the Contractor's communications with other agencies.

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14.0 REFERENCES

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Figures

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Legend

Project Location

COORDINATE/REFERENCE: WGS_1984_UTM_Zone_55N: IMAGERY 2016

EA Engineering, Science, and Technology, Inc., PBC
1001 Army Drive, Suite 103,
Barrigada, 96913-1402
Telephone: (671) 646-5231
Facsimile: (671) 646-5230

Project No/TO.:RFP No. 2015-001
Guam EPA Brownfields 2017
Phase II ESAs

Figure 1
Site Vicinity Map

Drawing No.	Date	Drawn By	EA Project No.
Fig 1_Project Site Location	08/15/2018	TChargualaf	1567201

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COORDINATE/REFERENCE: WGS_1984_UTM_Zone_55N: IMAGERY 2016

Legend

- Project Site Location
- Roadway

370 185 0 370 740 Feet

N

EA Engineering, Science, and Technology, Inc., PBC
1001 Army Drive, Suite 103,
Barrigada, 96913-1402
Telephone: (671) 646-5231
Facsimile: (671) 646-5230

Project No/TO.:RFP No. 2015-001
Guam EPA Brownfields 2017
Phase I ESAs

Figure 2
Project Site Location

Drawing No.	Date	Drawn By	EA Project No.
Fig 1_Project Site Location	08/15/2018	TChargualaf	1567201

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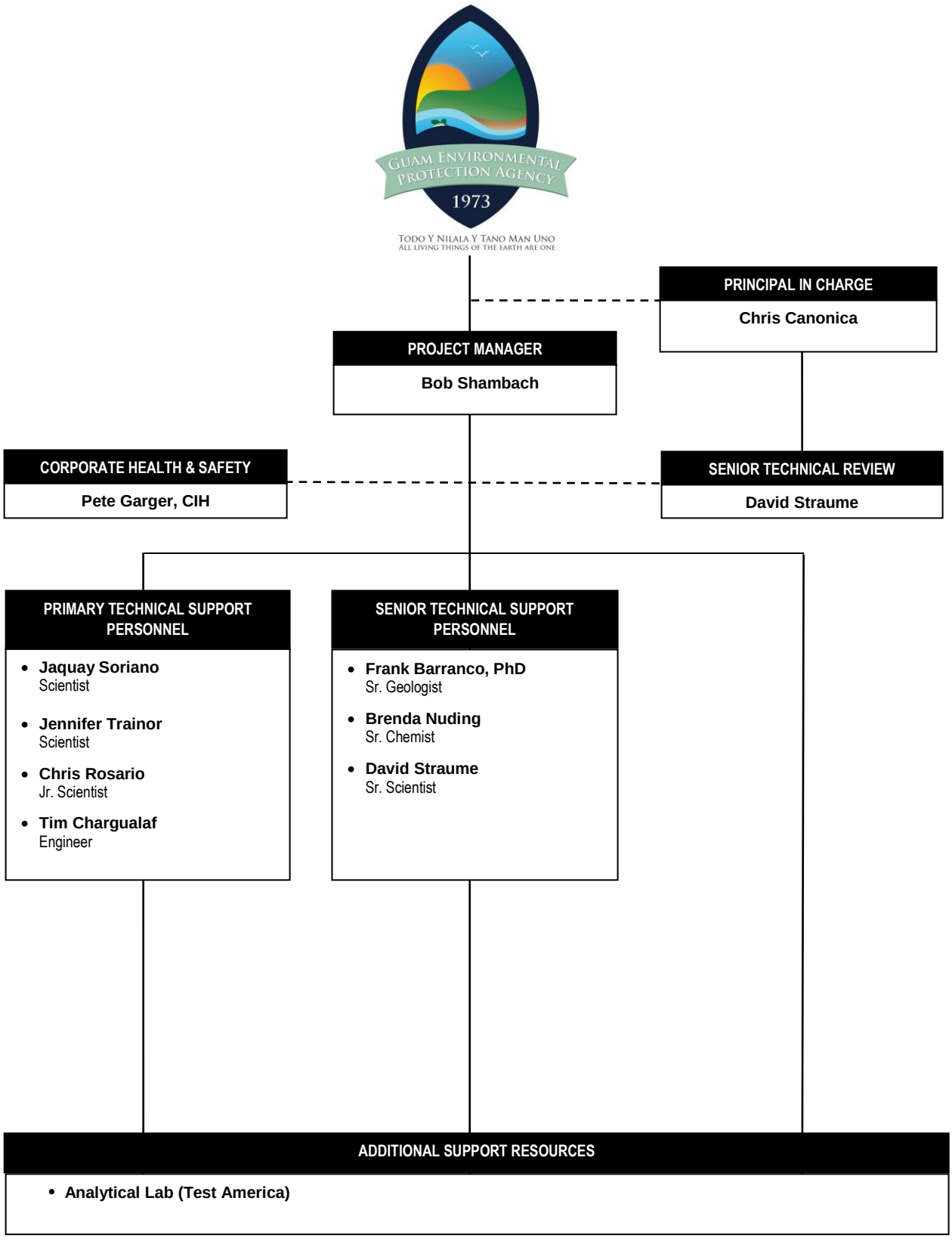
FIGURE 3. PROJECT ORGANIZATIONAL CHART



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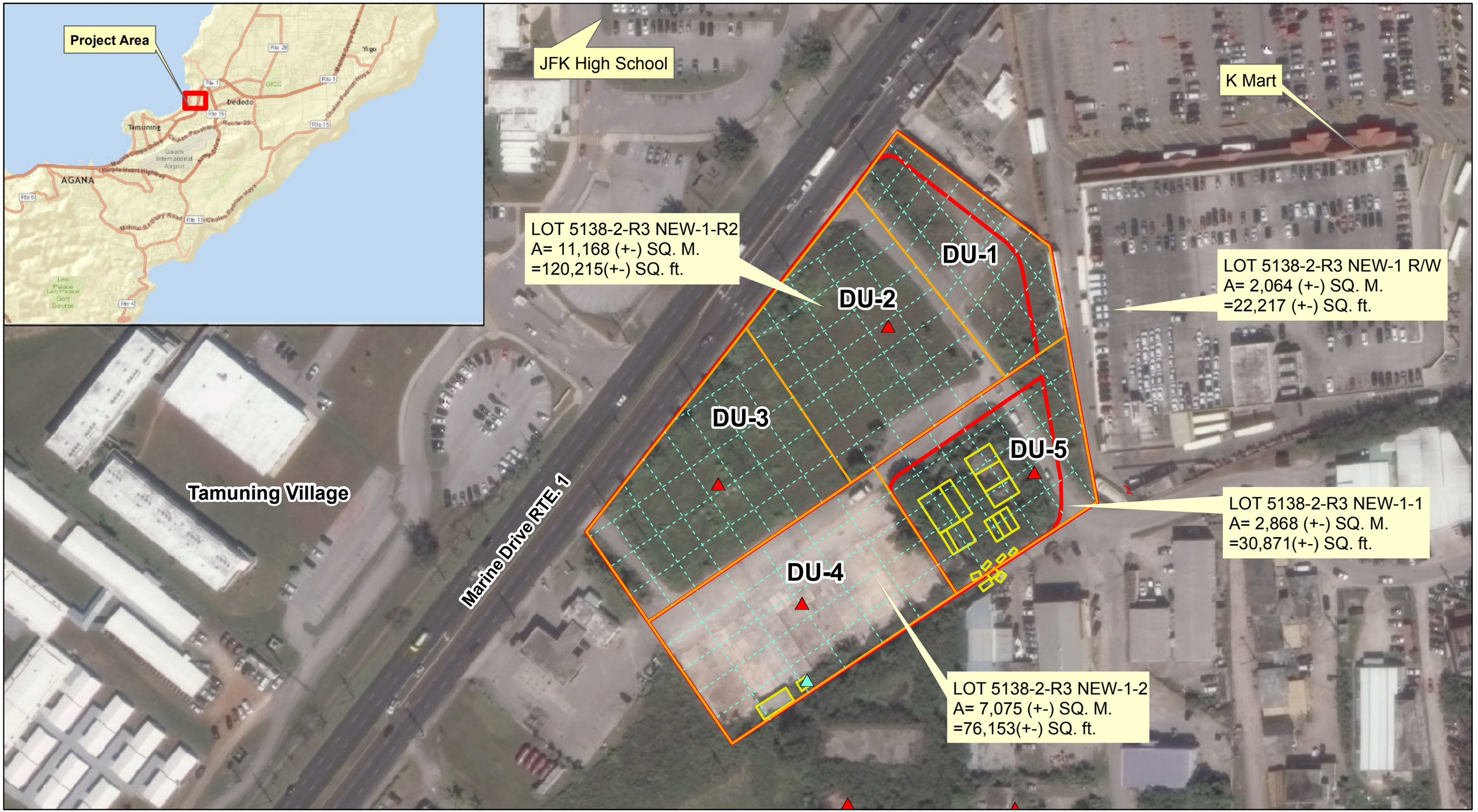

COORDINATE/REFERENCE: WGS_1984_UTM_Zone_55N: IMAGERY 2016

Legend

- Proposed Petroleum Product Sample
- Incremental Sampling Methodology (ISM)- Each DU consist of 30 surface grab samples collected to make 1 composite ISM
- PhaseIIGrid
- DecisionUnits
- SiteFeatures
- Survey Boundary Lines

110 55 0 110 220 Feet

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		Figure 4 **Proposed Sample Location Map**	
Drawing No. Fig 1_Project Site Location	Date: 08/15/2018	Drawn By: TChargualaf	EA Project No. 1567201

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Appendix A

Analytical Quality Assurance Manual *(Compact Disc)*

and

Standard Operating Procedures

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Title: Gas Chromatographic Analysis of Total Petroleum Hydrocarbons [Methods AK 102/103, SW846-8015B, SW846-8015C, SW846-8015D, CA-LUFT]

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1. SCOPE AND APPLICATION

1.1. This SOP describes procedures to be used for determination and/or quantitation of semivolatile hydrocarbon mixtures that elute between n-C8 (octane) and n-C36 (hexatriacontane). Extension of the chromatographic run to include to n-C44 (tetratetracontane) may be performed as a client-specific measure. This SOP is applicable to extracts that are prepared according to the appropriate sample extraction SOPs (WS-OP-0004).

1.2. Table 1 lists hydrocarbon fuel mixtures that are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs are based on the low level standard and the sample preparation concentration factors. Matrix interferences or limited sample volume may result in higher RLs than those listed.

1.3. This method is designed to measure mid-range petroleum products such as Diesel #2 or motor oil utilizing methods AK102/103, SW846-8015B and SW846-8015D. As of the September 2012 release of the leaking Underground Fuel Tank Guidance Manual, CA_LUFT samples are to be run per SW846-8015B protocols and criteria.

1.4. When undertaking projects for Department of Defense (DoD) or the Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 “Federal Program Requirements” must be checked and incorporated

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts for hydrocarbon fuel mixtures. The extracts are injected onto the column and separated and detected by flame ionization detection. Quantitation is by the external standard method.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID).

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the flame ionization detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3. Other organic compounds including animal and vegetable oil and grease, chlorinated hydrocarbons, phenols and phthalate esters are measurable under the conditions of this method. As defined in the method, the unknown hydrocarbon results include these compounds.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including silica gel cleanup. A silica gel cleanup can remove most of the polar organic interferences. These cleanup procedures are included in SOP WS-OP-0004.

4.5. Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank to check for carryover.

4.6. Petroleum products are complex mixtures of compounds derived from crude petroleum, and different products may have overlapping boiling ranges and chromatograms. The products are also subject to degradation in the environment with consequent changes in the chromatographic profile. When such changes are considerable, it may not be possible to positively identify the parent mixture.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Nitrile gloves should be used when working with solvent or extract containers. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

5.1.2. Ensure that all instrument exhaust vents and lines are properly connected to either a laboratory vent or an appropriate filter. Instruments may not be vented to the working environment.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable. All preparation of standards and dilutions shall be performed inside an operating fume hood. All samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride (2-1-0)	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

6.1. Gas Chromatograph - Agilent model 6890N or equivalent.

6.1.1. Autosampler – Agilent model 7673, 7683B or equivalent.

6.1.2. Operating System – Agilent MSD ChemStation D.02-00-275 or equivalent.

6.1.3. Software version –Chrom Peak Review, Version 2.1. or equivalent.

6.2. Preventive and routine maintenance is described in Table 9 of this SOP.

6.3. Refer to Table 3 for analytical column and conditions.

6.4. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Neat Standard Materials

Hydrocarbon fuels are generally available as stock solutions from commercial vendors. In rare instances when they are not available from vendors, neat materials may be acquired directly from the client. Such neat materials expire following a period of 3 years from the date of receipt, or sooner if problems such as pattern degradation or linearity loss occur.

7.2. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Standard solutions are stored at 0 - 6°C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Stock standard solutions must be replaced after six months (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased), or the manufacturer's expiration date, whichever is sooner. Stocks may be replaced sooner if comparison with check standards prepared from an independent source indicates a problem.

7.3. Calibration Standards

Calibration standards are prepared as dilutions of the stock standards. Surrogate standards are used as specified. Calibration solutions must be refrigerated at 0 - 6°C and protected from light. The standards are given a 6 month expiration date from the time of preparation, or the expiration date of the stock solution, whichever is sooner.

Standards may be replaced sooner if there is reason to believe that the standard has degraded or concentrated or if comparison with check standards indicates a problem. Refer to Table 3 for details of calibration standards.

7.4. Surrogate Standards

7.4.1. Ortho-terphenyl is the surrogate standard. Refer to Tables 4 and 6 for details of the surrogate standard.

7.4.2. The surrogate n-triacontane-d62 is required for many QSM programs and may be used as a surrogate for AK103 (RRO) analysis. Refer to Tables 4 and 6 for details of this surrogate standard.

7.5. Alkane Range

A single level standard containing the even-numbered alkanes from n-C8 to n-C40, n-C19 and n-C25, is used to set retention time windows for fuel identification and quantitation. This may be made from multiple commercially available standards. The alkane range solution has an expiration of one year from date of preparation or the manufacturer's expiration of the solution(s) from which it is diluted, whichever is earlier. Refer to Table 5 for details of the alkane range standard.

7.6. Gases for carrier, make-up, and detector fuel: hydrogen, and nitrogen, and air.

7.7. Quality Control (QC) Standards

QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They may be made from the same stock as the calibration standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Semivolatile extracts must be refrigerated at 0 - 6°C. Aqueous samples must be extracted within 7 days of collection and solid samples within 14 days of collection. Extracts must be analyzed within 40 days.

8.2. Aqueous samples for analysis by AK102 must be preserved to pH < 2 and extracted within 14 days of collection. Solid samples for both AK methods must be extracted within 14 days of collection. All samples for both AK methods must be analyzed within 40 days of collection (not extraction).

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection

limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is the analysis of a single point calibration standard.

9.2. Quality Control Batch

The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike/matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify particular samples for MS/MSD, the batch may contain multiple MS/MSDs. See policy WS-PQA-003 for further definition of the batch.

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC Program document (WS-PQA-003) for further details of the batch definition.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). Refer to policy WS-PQA-003 for more details.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction). Surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.3.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into TALS or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.3. Refer to the QC Program document (WS-PQA-003) for further details of control limits.

9.3.4. Refer to Table 10 for AK 102/103 recovery requirements for surrogates and laboratory control samples (LCS/LCSD) and RPD (Relative Percent Deviation).

9.4. Surrogates

Surrogate recoveries in field samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem. Repreparation is not necessary if there is obvious chromatographic interference.
- The decision to reanalyze or flag the data should be made in consultation with QA and the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.4.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.4.2. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.5. Method Blanks

One method blank must be processed with each preparation batch. The method blank consists of reagent water for aqueous samples. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher. Certain programs, such as DOD QSM, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the reporting limit.

9.5.1. Re-preparation and reanalysis of any samples with reportable concentrations of analytes less that 10 times the value found in the method blank is required

unless other actions are agreed with the client.

9.5.2. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported. This must be documented in the NCM program.

9.5.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all positive results in associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.5.4. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.6. Laboratory Control Samples (LCS)

9.6.1. If insufficient sample is available to process a MS/MSD, then a LCS/LCSD pair musty be processed. The LCS/LCSD pair is then evaluated according to the MS/MSD RPD criteria. Use of a LCS/LCSD pair in place of a MS/MSD must be documented.

9.6.2. For each batch of samples, analyze an LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS/ may also contain the full set of analytes. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur.

9.6.2.1. LCS compound lists are included in Table 6.

9.6.2.2. For AK Methods (AK102; AK103) prepare and analyze an LCS Duplicate sample as well as an LCS sample for each component of interest (DRO, RRO) for each analytical batch.

9.6.3. If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur:

- Check calculations,
- Check instrument performance,
- Reanalyze the LCS, and if still outside of control limits,
- Evaluate the data, and/or
- Re-prepare and reanalyze all samples in the QC batch.

9.6.4. With the exception of QSM 5.0 projects, data may be reported with an anomaly in the following cases:

- The LCS recoveries are high and the analyte of concern is not detected in field samples,
- All requested target analytes are within control, but other LCS compounds are out of control.

9.6.5. The analyst should evaluate the anomalous analyte recovery for possible trends.

9.6.6. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.

9.6.7. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS/LCSD is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6.8. Refer to the QC Program document (WS-PQA-003) for further details of the corrective action.

9.7. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 6. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits.

9.7.1. If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The first corrective action is to evaluate the MS/MSD for any systemic issues such as mislabeling, incorrect spiking, bad injection, etc. If the failure is due to these issues it must be re-injected or re-extracted depending on the situation. If no systemic problem is found corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.

9.7.2. If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

9.7.3. If a MS/MSD is not possible due to limited sample, then a LCS duplicate may be analyzed if desired by the client.

9.7.4. The MS/MSD must be analyzed at the same dilution as the unspiked sample,

unless the matrix spike components would then be above the calibration range.

9.7.5. If the amount of an analyte found in the unspiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply and recoveries are not evaluated. Other analytes in the MS and MSD must still be reported. If TALS has appropriately flagged the data, an NCM is not necessary. Otherwise, file an NCM stating that the 4X rule was applied.

9.8. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS may be processed, if precision data is required by the client. The LCS pair is then evaluated according to the MS/MSD RPD criteria. Use of an LCS pair in place of an MS/MSD must be documented using the NCM module in TALS.

9.9. Initial Calibration Verification (ICV) -- An ICV must be run after all points to be used in the curve have been run and before any samples are run. The ICV is from a certified second source and should be at a concentration roughly at the midpoint of the calibration. All analytes to be reported must be in the ICV.

Each compound of the second source calibration must be within the criteria below when evaluated with respect to the calibration curve:

Method/Program	%D from expected value
Method 8000B/8015B	± 15%
Method 8000C/8015C/8015D	± 20%
DOD/DOE QSM	± 20%
AK102/103	± 25%
CALUFT	± 20%

Corrective actions for the ICV include:

- Rerun the ICV
- Remake or acquire a new ICV
- Evaluate the instrument conditions
- Evaluate the Initial Calibration Standards

9.10. Certain clients may require specific project or program QC which may supersede these method requirements. The analyst should check the project notes for the job in TALS before running samples.

9.11. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the QA Manager.

9.12. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. Initial Calibration

10.1.1. External calibration is used. Prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in Table 4.

10.1.2. The alkane range standard should be run with the initial calibration. If the initial calibration consists only of add-on mixtures, and the current diesel fuel calibration standards indicate that no peak shifting has been occurring, analysis of the alkane standard may be waived.

10.1.3. Quantitation of hydrocarbon mixtures

10.1.3.1. Starting and ending retention times for quantitation are determined for each fuel. The peak areas between the starting and ending times are summed and used to generate a response factor. This factor is used to quantitate sample results, and depending on the client requirements, this factor may be applied to the same retention range as the standard, or to a different range.

10.1.3.2. See Table 2 for details of the commonly used retention and quantitation ranges.

10.1.3.3. When setting starting and ending retention times based on Table 2 ranges (or other commonly used ranges), the start of the range should be set ± 0.1 minute before the first marker peak and end ± 0.1 minute before the last marker peak. The retention times of the apexes of the marker peaks should not be used as the retention time windows for the fuels.

10.1.4. Analyze an ICV (Section 9.9) immediately following the calibration curve and before field sample analysis. This verification standard is used as a quality control check to verify the accuracy of the calibration.

10.2. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, or changing PID lamps or FID jets, and any changes in instrument operating parameters, including gas flows, detector temperatures, oven temperatures, etc.

10.3. With the exception of instances detailed in Policy CA-Q-P-003, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve.

10.4. Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

Equation 1

$$Calibration\ Factor\ (CF) = \frac{Area\ of\ Peak(s)}{Concentration\ of\ Standard\ (ng/ml\ or\ ug/ml)}$$

Due to the nature of hydrocarbon fuels, peak height may not be used for quantitation. Variations on this formula are permitted so long as the calculations between standards and samples remain consistent.

10.5. Average response factor or linear regression may be used to fit the data. Linear regression may be used if the calibration range is sufficiently narrow so that a straight line will fit the calibration points and r>0.995. Average response factor may be used if the %RSD of the response factors or calibration factors is ≤ 20%.

10.5.1. Average response factor

10.5.1.1. The average response factor may be used if the percent relative standard deviation (%RSD) of the response factors is ≤ 20%.

10.5.1.2. The equation for average response factor is:

Equation 2

$$\text{Average response factor} = \frac{\sum RF_{1-n}}{n}$$

Where: n = Number of calibration levels

$\sum RF_{1-n}$ = Sum of response factors for each calibration level

10.5.2. Linear regression

10.5.2.1. The linear fit uses the following function:

10.5.2.2. **Equation 3**

$$Concentration = A + BR$$

Where: A = Intercept
B = Slope
R = Response
Correlation coefficient r>0.995

10.6. Evaluation of calibration curves

10.6.1. The percent relative standard deviation (%RSD), or percent relative standard error (%RSE) for non-average response factor calibration models, from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.6.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level.

10.6.2.1. The percent relative standard error (%RSE) is calculated as follows:

Equation 4

$$\%\ RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{Ci - PCi}{Ci}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve

P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)

C_i = True concentration for level i

PC_i = Predicted concentration for level i

Note that when average response factors are used, this equation gives the same value as the %RSD of the response factors.

10.6.2.2. Percent relative standard deviation is calculated as follows:

Equation 5

$$\%\ RSD = 100\% \times \frac{\sqrt{\frac{\sum_{i=1}^{N}\left[RF_i - \overline{RF}\right]^2}{N-1}}}{\overline{RF}}$$

Where:

N = Number of points in the curve

RF_i = Response factor calculated for level i

$\overline{RF}$ = Average response factor

10.7. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response must be less than ± the reporting limit for the analyte.
- Relative standard deviation (or RSE) of the calibration points from the curve used must meet the criteria below:

Method/Program	RSD
Method 8000B/8015B	20%
Method 8000C/8015C/8015D	20%
DOD/DOE QSM	20%
AK102/103	25%
CALUFT	20%

- In some cases, (normalized) %RSD may not be appropriate (i.e., curve fits without 1/Concentration^2 weighting). In this case, the correlation coefficient (r-squared) may be used as an alternative to the %RSD, and must be greater than 0.990.

Note: The normalized %RSD is superior to the Correlation Coefficient (r) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result a curve may have very good correlation coefficient (>0.995), while also having >100% error at the low point.

10.7.1. Weighting of data points

In a linear calibration fit, the points at the lower end of the calibration have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

NOTE: Prior to accepting an initial calibration curve, it is highly recommended to recalculate the calibration levels using the new calibration response factors. Then calculate the % difference for every analyte and every level of the calibration and compare to the criterion of ≤20% from the true concentration. If the curve fails this criteria consult the department manager about whether to re-do the calibration.

10.7.2. Non-standard analytes are sometime requested. For these analytes, it is acceptable to analyze a single standard for pattern identification daily, rather than a five point initial calibration, provided that the client agrees with this strategy.

10.7.2.1. Positives which match the pattern of the non-standard analyte may be quantitated based on a standard reference if the elution range is similar and the client agrees to this. Single point ICAL is analyzed once per week. The CCV is analyzed daily (once every 12 hours and/or once for every 20 sample sequence).

10.7.2.2. Otherwise, a five-point initial calibration must be run and samples with positives must be reanalyzed for quantitation. If the analyte is not detected, the non-detect may be reported and no further action is necessary.

10.8. Integration

10.8.1. When evaluating the initial calibration, also evaluate hydrocarbon pattern integration.

10.8.1.1. The integration baseline should run from the point in the chromatogram prior to peaks to a region following the mixture's peaks in a fairly straight line.

10.8.1.2. If there is unresolved material in the middle of the chromatogram, the area under the "hump" should also be integrated.

10.8.2. The default integration parameters generated as a result of evaluating the initial calibration should remain in effect until the initial calibration is reanalyzed.

10.9. Calibration Verification (CCV)

10.9.1. The working calibration curve or RF must be verified by the analysis of a mid-point calibration standard. For Method 8015B, 8015D and AK102/103 the CCV must be run at the beginning, after every 20 field samples or 12 hours, whichever is more frequent, and at the end of the analysis sequence. Specific details may be found in method 8000C section 11 and paragraph 9.2.2. For QSM 5.0 work the CCV must be run at the beginning, after every 10 field samples or 12 hours, whichever is more frequent, and at the end of the analytical sequence.

10.9.2. The center of each retention time window for surrogate compounds is updated with each CCV. For fuel standards, the fuel's elution profile is compared to the initial calibration to verify that shifting has not occurred since initial calibration. If an alkane range standard has been analyzed, it is checked to verify that shifting has not occurred since the retention time windows for the fuels were set.

10.9.3. It may be appropriate to analyze a mid-point standard more frequently than

every 20 samples (or 12 hours, whichever is more frequent). If these continuing calibration standards are analyzed, requirements are the same.

10.9.4. Each compound within the CCV must be within the criteria below when evaluated with respect to the calibration curve.

Method/Program	%D from expected value
Method 8000B/8015B	± 15%
Method 8000C/8015C/8015D	± 20%
DOD/DOE QSM	± 20%
AK102/103	± 25%
CALUFT	± 20%

If these criteria are exceeded, then corrective actions must be implemented. Typically, samples analyzed after the nonconforming CCV are reanalyzed. Samples analyzed prior to the nonconforming CCV are also reanalyzed, unless the samples are non-detect and the CCV is exhibiting an elevated response. A non-conformance memo must be filed in this instance. Refer also to Section 10.9.6

NOTE: Samples analyzed under DOD QSM version 5 must have passing CCVs both before and after them.

10.9.4.1. % Difference calculation

Equation 6

$$\% \text{ Difference} = \frac{|\text{Expected value} - \text{Calculated value}|}{\text{Expected value}} \times 100\%$$

10.9.5. It is not necessary to run a continuing calibration standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.9.6. If the analyst notes that a CCV has failed and can document the reason for failure (e. g. no purge, broken vial, carryover from previous samples, etc) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV then the preceding samples have not been successfully bracketed but analysis may continue. For corrective actions required for QSM projects see WS-PQA-021.

10.9.7. In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed the first will serve as the bracketing standard for the preceding samples and the last will serve as the CCV for the following samples.

10.9.8. If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run.

10.9.9. CCV analysis for Methods AK102.0 and AK103.0 (State of Alaska) analysis includes analysis of the alkane range standard to verify retention times.

Certain programs, such as those falling under the DOD/DOE/QSM, require that the alkane range standard be run daily to verify retention times.

10.10. Refer to Table 3 for details of GC operating conditions.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Extraction

The extraction procedure is described in SOP WS-OP-0004.

11.3. Cleanup

Cleanup procedures are described in WS-OP-0004.

11.4. Suggested gas chromatographic conditions are given in Table 3. Actual gas chromatographic conditions can be found in the maintenance logs.

11.5. Instrument Bleed Profile

Because the extractable hydrocarbon analysis is subject to integration difficulties as a result of column bleed and late-eluting compounds, it is advisable to subtract the instrument bleed profile from every injection by performing a column comp. Column comps should be renewed when there is an obvious change in the instrument's column bleed profile. Only instruments runs performed without injection of solvent and where the chromatogram shows no obvious peak may be use for column comps. When a column comp is performed, it needs to be documented in the instrument's maintenance log.

11.6. Analytical Sequence

An analytical sequence starts with an initial calibration or a calibration verification (CCV), performed as described in Sections 10.1 and 10.9.

11.6.1. If there is a break in the analytical sequence of greater than 2 hours, the sequence must be restarted with a DCM blank.

11.7. Retention Time Windows - Single Peaks

11.7.1. Retention time windows must be determined for all single peak analytes (including surrogate compounds). Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.7.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows for surrogate compounds are updated with the mid-point of the initial calibration and each daily calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.7.3. If the retention time window as calculated above is less than ± 0.03 minutes, use ± 0.03 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.7.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.7.5. The retention times of all surrogate compounds in each continuing calibration must be within the retention time windows established by the daily calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed.

11.8. Retention Time Windows - Multiple Peak Standards

Because multiple peak hydrocarbon mixtures are quantitated based on the total area between two retention times, the retention time windows that are generated are also used to determine the times used for area summing.

Retention time windows must be determined for each hydrocarbon mixture to be analyzed.

11.8.1. Make an injection of the alkane range standards once per day over a three day

(72 hour) period. Calculate and tabulate the standard deviation of the three retention times for each peak in the mixture. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.8.2. Hydrocarbon mixture summing windows are the first peak minus three times the standard deviation for that peak to the last peak plus three times the standard deviation for that peak. For example, for a fuel quantitated between n-C10 and n-C24, the peak summing window is from the retention time for n-C10 minus three times the standard deviation for n-C10 to the retention time for n-C24 plus three times the standard deviation for n-C24.

11.8.3. For mixtures which use an alkane which is not present in the carbon range reference, the retention time of the alkane may be determined by linear extrapolation, and the retention time window of the alkane following the one not present may be used to set peak summing windows. An example of this instance is JP-4 in the extractable hydrocarbons test, which uses n-C13 as the end of the summing window.

11.8.3.1. Linear Interpolation of Retention Times

Equation 7

$$RT_{Int} = RT_{Earlier} + \frac{(RT_{Later} - RT_{Earlier})}{2}$$

Where:

RT_{Int}	=	Interpolated Retention Time (for n-C13 in the example above)
$RT_{Earlier}$	=	Retention Time of earlier peak (for n-C12 in the example above)
RT_{Later}	=	Retention Time of later peak (for n-C14 in the example above)

11.8.4. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows for the alkane range peaks are updated with the alkane range that is run with new initial calibrations. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.8.4.1. Retention time windows for hydrocarbon mixtures are updated at the same time as those for the alkane peaks.

11.8.4.2. If an alkane range is not run with the initial calibration, retention times and summing windows may not be updated.

11.8.5. If the retention time window for an alkane peak as calculated above is less

than ± 0.03 minutes, use ± 0.03 minutes as the retention time window.

11.8.6. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Because quantitation of hydrocarbon mixtures is dependent on the peak summing window, calibration data evaluation and sample analysis cannot continue until the alkane retention time windows are established.

11.8.7. Because quantitation of fuels is based on the total area between two set retention times, each fuel standard's elution profile (peak pattern with respect to retention times denoting start and end of quantitation) in each continuing calibration must be similar to the profile at the time of initial calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed.

11.8.7.1. Compare elution profiles by utilizing the data system's capability to overlay chromatograms on-screen, and overlay the mid-level standard from the calibration curve with the daily calibration.

11.8.7.2. Alternatively, compare elution profiles by overlaying hard-copy chromatograms that are at the same scale, and comparing them.

11.8.7.3. Certain methods may require that the alkane standard be run prior to each analytical batch, and that retention times for this standard are evaluated. Such instances are detailed in the appendices.

Note: When exaggerated shifting of surrogate compounds occurs in samples, quantitation of any fuel mixture, unknown or otherwise, is likely to be adversely affected. Retention time shifting can be a result of instrument gas leaks, poor temperature control at the instrument, poor laboratory temperature control, or matrix effects. It is highly recommended that the cause is determined and samples reanalyzed when retention time shifting occurs.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Identification

Tentative identification occurs when a peak pattern matching the reference standard is found within the elution window for a fuel, at a concentration above the reporting limit, or above the MDL if J flags are required.

12.1.1. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

Note: In cases of extremely oily samples, it may be advisable to dilute samples such that the analyte concentration is in the lower half of the calibration range. Such deviations should be noted and filed with the project.

12.3. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the peaks in the Level 3 standard, then the sample should be reanalyzed at a more concentrated dilution.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12.4. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Calculations

Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.5.1. Aqueous Samples (assuming average response factor calibration)

Equation 8

$$Concentration\ (\mu g / L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_s)}$$

Where:

A_x = Response for the analyte in the sample

D_f = Dilution factor

V_t = Volume of total extract, mL

V_s = Volume of sample extracted or purged, mL

CF = Calibration factor, area/ng/mL or area/µg/mL, Section 10.1

12.5.2. Non-aqueous Samples (assuming average response factor calibration)

Equation 9

$$Concentration(\mu g/kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$D = \frac{100 - \%Moisture}{100}$ (D = 1 if wet weight is required)

Note: The percent moisture correction may occur following entry into a LIMS database instead of at the instrument.

A_x, V_t, D_f, and CF are as defined in Equation 8

12.6. Identification of Multi-component Analytes

12.6.1. Chromatograms are evaluated only for the fuel list specified by the client. "Fingerprinting" of materials is not normally performed beyond the specified list.

12.7. Evaluating Degraded Diesel #2

12.7.1. All fuels exposed to the environment are subject to a variety of weathering and degradation agents, and therefore they may not be identified after a period of time in the environment. To aid in the identification of degraded Diesel #2, four criteria should be evaluated. If at least two of the four criteria are met, but the profile is not a good match for the diesel standard, then it can be considered a degraded diesel fuel and reported as diesel fuel, with a footnote denoting that it is degraded.

12.7.2. Criteria for evaluating degraded Diesel Fuel #2

- The OTP peak (about C-19) is distinctly on the downslope of the unresolved material.
- All alkane peaks from n-C10 to n-C23 are visible.
- The envelope of unresolved material returns to the baseline between n-C23 and n-C26.
- The baseline rise for the unresolved material starts by n-C11 and the early end from n-C10 to n-C13 is present.

12.8. Surrogate recovery results are calculated and reported for Ortho-terphenyl (OTP). When this analysis is performed in support of method AK103, results for n-triacontane-d62 must also be reported. In some client specific cases, other surrogate compounds may also be used. Due to the nature of hydrocarbon fuel analysis, surrogate recovery may be elevated beyond the upper control limit as a result of area contribution from a fuel analyte. In these instances, report the recovery, and narrate the elevated recovery in the case narrative.

12.9. When reporting Diesel Range Organics (DRO), no Unknown Hydrocarbon is evaluated. If there is a concentration above the reporting limit in the DRO range, then there is DRO present.

12.10. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

Equation 10

$$\% \text{ Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100$$

12.11. Reporting Requirements

12.11.1. Results are reported as described in the method appendices.

12.11.2. Reporting limits are listed in the method specific appendices.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for

determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits referenced in Table 7 (Performance limits, four replicate initial demonstration of capability).

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Autovials contaminated with methylene chloride. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After the autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methylene chloride to evaporate into the atmosphere. The satellite collection

containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996. Section 8000B

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV, March 2003. Section 8000C

16.3. Leaking Underground Fuel Tank Guidance Manual, State of California, September, 2012

16.4. Underground Storage Tanks Procedures Manual, Alaska Department of Environmental Conservation, November 7, 2002; Appendix D

16.5. Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997

16.6. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW 846, 3rd Edition, Final Update III, December, 1996; Method 8015B

16.7. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW 846, 3rd Edition, Final Update IV, March 2003; Method 8015D (Revision 4, June 2003)

16.8. Alaska Department of Environmental Conservation, April 8, 2002; Methods AK102.0 and AK103.0

17. METHOD MODIFICATIONS

17.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2. ADEC methodology specifies that standards and extracts are stored at –10 to –20°C. This SOP used 4 ± 2°C as the requirement. This temperature range is consistent with other SOPs at this facility.

18. ATTACHMENTS

18.1. Table 1 – Standard Analyte List and Reporting Limits

18.2. Table 2 – Extractable Petroleum Hydrocarbon Quantitation Ranges

18.3. Table 3 – Parameters and Recommended Conditions

18.4. Table 4 – Calibration Levels µg/mL

18.5. Table 5 – Components of Alkane Range Standard

18.6. Table 6 – LCS/Matrix Spike and Surrogate Spike Levels µg/L or mg/kg

18.7. Table 7 – Performance Limits, Four Replicate Initial Demonstration of Capability

18.8. Table 8 – AK102/103 Recovery Requirements

18.9. Table 9 – Recommended Preventative Maintenance

19. REVISION HISTORY

19.1. WS-GC-0007, Revision 6.4, Effective 03/06/2018

19.1.1. Section 1.1, changed "n-C40 (tetracontane)" to n-C44 (tetratetracontane)

19.1.2. Updated Table 4 to reflect current calibration levels.

19.1.3. Updated Table 5 to reflect the current components of the alkane range standard.

19.1.4. Editorial changes.

19.2. WS-GC-0007, Revision 6.3, Effective 03/08/2017

19.2.1. Changed footnote 2 at end of Table 1 to read, "The method specifies that the peak summing window must begin at the start of the n-C25 peak and stop at the end of the n-C36 peak."

19.2.2. Editorial changes.

19.3. WS-GC-0007, Revision 6.2, Effective 11/18/2016

19.3.1. Section 11.5, changed to "Because the extractable hydrocarbon analysis is subject to integration difficulties as a result of column bleed and late-eluting compounds, it is advisable to subtract the instrument bleed profile from every injection by performing a column comp. Column comps should be renewed when there is an obvious change in the instrument's column bleed profile. Only instruments runs performed without injection of solvent and where the chromatogram shows no obvious peak may be use for column comps. When a column comp is performed, it needs to be documented in the instrument's maintenance log."

19.3.2. Editorial changes.

19.4. WS-GC-0007, Revision 6.1, Effective 08/05/2016

19.4.1. Table 3 – Updated column type.

19.4.2. Added Section 9.6.1 – "If insufficient sample is available to process a MS/MSD, then a LCS/LCSD pair musty be processed. The LCS/LCSD pair is then evaluated according to the MS/MSD RPD criteria. Use of a LCS/LCSD pair in place of a MS/MSD must be documented. "

19.4.3. Editorial changes.

19.5. WS-GC-0007, Revision 6.0, Effective 7/10/2015

19.5.1. Updated Copyright information on Title Page.

19.5.2. Section 10.5 - Added linear fit correlation coefficient: r>0.995.

19.5.3. Revised Section 10.7.2.1 - Single point ICAL is analyzed once per week. The CCV is analyzed daily (once every 12 hours and/or once for every 20 sample sequence)."

19.5.4. Section 10 9.1 – Included Method AK102/103 to list of methods requiring a CCV to be run at the beginning, after every 20 field samples or 12 hours, whichever is more frequent, and at the end of the analysis sequence.

19.5.5. Deleted Section(s) 12.1.1 and 12.6.2 – reporting J values for unknown hydrocarbons. Unknowns are no linger reported for these methods.

19.5.6. Updated Table 1 to change RL's for 8015B/8015C/8015D and to include DOD 5 RL's

19.5.7. Editorial changes.

19.6. WS-GC-0007, Revision 5.9, Effective 11/21/2014

19.6.1. Removed Section 10.9.3 – "The mid-level calibration standard used at the start of the analysis sequence must be a different concentration than the mid-level calibration standard used for subsequent calibration verifications. Recommended concentrations are noted in Table 4."

19.6.2. Editorial changes.

19.7. WS-GC-0007, Revision 5.8, Effective 06/27/2014

19.7.1. Deleted Section 7.4.3 – "Samples to be run in accordance with CA LUFT methodology are spiked with capric acid as a reverse surrogate added prior to silica gel cleanups. Detection of this surrogate in the final extract indicates the presence of water and/or an overloaded cleanup column" as this procedure is not being used at TestAmerica Sacramento.

19.7.2. Revised Section 7.7 - "QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They may be made from the same stock as the calibration standards.

19.7.3. Editorial changes.

19.8. WS-GC-0007, Revision 5.7, Effective 10/21/2013

19.8.1. Removed all references to NWTPH methods as they are no longer offered by the laboratory. This includes edits to the Title, Section 1.3, and removal of Section 8.2, Section 12.10, 16.9 and 17.5.

19.8.2. Updated to reflect the current data processing and LIMs system, which permits easy use of multiple program acceptance criteria for QC elements. Consequently, Sections 17.5 and 17.6 have been removed, as the less stringent AK criteria have been programmed into the data processing system for use with AK samples.

19.8.3. Replaced/Updated CA-LUFT references to reflect the September 2012 manual. Section 17.2 and 17.3 have been removed, Section 16.3 has been revised.

19.8.4. Section 4.4, added, "A silica gel cleanup can remove most of the polar organic interferences." in the middle of the paragraph.

19.8.5. Section 6, updated the data processing software to the current Chrom software, and changed the reference for preventative maintenance to Table 9 of this SOP.

19.8.6. Added Section 7.4.3 (Capric acid reverse surrogate).

19.8.7. Section 7.5 and Table 5, added n-C19.

19.8.8. Added Table 9

19.8.9. Changed all references to QuantIMS to TALS.

19.8.10. Added corrective actions to Section 9.7.1.

19.8.11. Removed Section 12.11.3, as the information is now all stored and available in

the LIMS and data processing software to the data reviewer.

19.8.12. Updated Section 9.9 to include criteria for all method variations covered by this SOP, as well as to clarify when the ICV must be analyzed.

19.8.13. Updated Section 10.7 and 10.9.5 criteria, added the note following Section 10.7.1, and updated Section 10.9.1 frequency.

19.8.14. Added references to SW-846 Update IV methods (8000C, 8015D).

19.8.15. Editorial revisions

19.9. WS-GC-0007, Revision 5.6, Effective 05/31/2013

19.9.1. Changed Clouseau references to 'NCM module in TALS'.

19.9.2. Replaced footer in Table I to reference reduced volume extractions.

19.9.3. Removed footnotes following Table 4.

19.9.4. Editorial changes.

19.10. WS-GC-0007, Revision 5.5, Effective 09/14/2012

19.10.1. Removed Table 8 and Table 9 due to TALS implantation.

19.10.2. Remove Section 12.8 "Reporting Unknown Materials as this descriptor is not used with TALS/Chrom reporting.

19.10.3. Editorial changes

19.10.4. Editorial revisions

Table 1 Standard Analyte list and Reporting Limits					
Fuel	Quantitation Range	(8015B / 8015C / 8015D)		DOD 5	
		Water (µg/L)	Soil (mg/kg)	Water (µg/L)	Soil (mg/kg)
Diesel Fuel #2	n-C10 to n-C24	50	1	50	2
Additional Fuels					
Motor Oil (10W-40)	n-C19 to n-C36	500	5	500	20
JP-4	n-C8 to n-C13	50	1		

	Sample Vol.	Final Vol.
Water	1000 mL	3 mL
Reduced Volume Extraction (LVI/RVE)	250 mL	2 mL
Soil/Solid	30 g	3 mL
Low Level Soil/Solid by EPA 3586 (MAE)	15 g	2 mL

Table 2 Extractable Petroleum Hydrocarbon Quantitation Ranges.			
Regulatory Method	Component	Quantitation Range	
EPA 8015B EPA 8015D	Diesel Fuel	n-C10	n-C28
	Mineral Oil	n-C15	n-C40
	Mineral Spirits	n-C8	n-C13
	JP-4	n-C8	n-C13
	JP-5	n-C8	n-C16
	JP-8	n-C8	n-C18
	Hydraulic Oil	n-C20	n-C40
	Kerosene	n-C8	n-C16
	Fuel Oil #6	n-C10	n-C16
	Stoddard Solvent	n-C8	n-C13
CA-LUFT (Default)	Diesel Range Organics (DRO) Oil Range Organics (ORO)	n-C12 n-C23	n-C22 n-C32
AK102.0[1] AK103.0[2]	DRO RRO	n-C10 n-C25	n-C25 n-C36

[1] *The method specifies that the peak summing window must start at the start of the n-C10 peak and end at the start of the n-C25 peak.*

[2] *The method specifies that the peak summing window must begin at the start of the n-C25 peak and stop at the end of the n-C36 peak.*

Table 3	
Parameter	**Recommended Conditions**
Injection port temp	250°C
Detector temp (FID)	320°C
Injection	2µL
Injection Type/Splitter	Splitless, 0.3 min purge time, 20 mL/min purge flow
Column	RTX-5MX, 30m x 0.32 mm, 0.25 µm (or equivalent)
Temperature program	50°C for 2.0 min, 15°C/min to 320°C, 3 min hold
Flow Program	5 mL/min for 2.0 min, 2 mL/min to 12 mL/min, hold to end of run
Gas Saver	On, flow = 20 mL/min
Make up gas flow mode	Makeup + column flow = constant, set for 50 mL/min
Carrier Gas	Hydrogen
Make-up Gas	Nitrogen
Fuel Gas	Hydrogen, 40 mL/min
Oxidant Gas	Air, 450 mL/min

Table 4							
Calibration Levels µg/mL							
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**
Diesel Fuel	10	50	100	300	500	1000	2000
Motor Oil (10W - 40)	50	100	300	500	800	1200	1500
JP-4	10	50	100	250	500	1000	
o-terphenyl (surrogate)	1	5	10	15	20	25	30
n-triacontane-d62 (surrogate)	1	5	10	15	20	25	30

Table 5	
Components of Alkane Range Standard[1]	
Component	**Approximate Concentration (µg/mL)**
n-C10 (decane)	5
n-C11 (undecane)	5
n-C12 (dodecane)	5
n-C13 (tridecane)	5
n-C14 (tetradecane)	5
n-C16 (hexadecane)	5
n-C18 (octadecane)	5
n-C19 (nonadecane)	5
n-C20 (cosane)	5
n-C22 (docosane)	5
n-C24 (tetracosane)	5
n-C25 (pentacosane)	5
n-C26 (hexacosane)	5
n-C28 (octacosane)	5
n-C30 (triacontane)	5
n-C32 (dotriacontane)	5
n-C34 (tetratriacontane)	5
n-C36 (hexatriacontane)	5
n-C38 (octatriacontane)	5
n-C40 (tetracontane)	5
n-C42 (n-Dotetracontane)	5
n-C44 (tetratetracontane)	5

[1] Standard is used only for setting retention times; therefore, exact concentrations are not necessary. A concentrated mixture of n-alkanes from n-C10 through n-C40 is available from Ultra Scientific. Its use is recommended.

Table 6		
LCS/Matrix Spike and Surrogate Spike levels µg/L or mg/kg		
	Aqueous	**Soil**
Diesel Fuel	300	10
o-Terphenyl (Surrogate)	40	1.33
Motor Oil	900	30
n-Triacontane-d32	50	1.67

Table 7		
Performance limits, four replicate initial demonstration of capability		
Compound	**Initial demonstration, mean recovery limits**	**Initial demonstration, RSD limits**
Diesel Fuel	50-150	25
Motor Oil (10W - 40)	50-150	25
JP-4	50-150	25
Other Add-on Fuels	50-150	25

Table 8	
AK102/103 Recovery Requirements	
Requirement	**Criteria**
Surrogate recovery in samples	50-150%
Surrogate recovery in CCV	60-120%
Surrogate recovery in LCS/D	60-120%
LCS Recovery	75-120% (Diesel); 60-120% (AK-103)
LCS/LCSD RPD	Less than or equal to 20%

Table 9	
Recommended Preventative Maintenance for GC-FID	
Maintenance	**Frequency**
Replace septum. Clean injector port Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed. Replace or repair flow controller if constant gas flow cannot be maintained. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace igniter. Replace fuse. Reactivate external carrier gas dryers. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents. Check inlets, septa.	As Needed
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Daily (when in use)

Title: Acid Digestion of Aqueous Samples by SW846 and MCAWW [Methods 3005A, 3010A, 200.7-4.4, 200.8-5.4]

Approvals (Signature/Date):

Robert Hrabak 3/30/17 Technical Manager Date	Joe Schairer 3/30/17 Health & Safety Manager / Coordinator Date
Lisa Stafford 4/20/2017 Quality Assurance Manager Date	Crystal Pollock 4.24.17 Laboratory Director Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP – method 6010B, 6010C, 200.7-4.4), and Inductively Coupled Plasma Mass Spectrometry (ICPMS – method 6020, 6020A) using SW846 3005A/3010A, 200.8-5.4, and 200.7-4.4 sample preparation protocols.

1.2. This SOP provides procedures applicable to the preparation of total recoverable, dissolved, and total metals in ground water, surface water, saline water, and wastewater. It also applies to samples prepared by TCLP extraction.

1.3. Tables I and II show a list of the analytes that are analyzed at the Sacramento facility and that are evaluated relative to method performance per NELAC criteria.

2. SUMMARY OF METHOD

2.1. Method 3005A is applicable to the preparation of total recoverable metals and dissolved metals by ICP and ICPMS. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to the original volume.

2.2. Method 3010A is applicable to the preparation of total metals for ICP analysis. A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary) and brought up to the original volume.

2.3. Method 200.7-4.4 and Method 200.8-5.4 are applicable to the preparation of total recoverable metals by ICP and ICPMS. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to the original volume.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Dissolved Metals: Metals present in a sample after passing through a 0.45 um membrane filter. The sample should not be preserved prior to filtration. Once filtered, the sample must be preserved to pH <2 with the addition of concentrated nitric acid.

3.4. Total Metals: metals present in an unfiltered, preserved sample followed by more aggressive digestion with nitric and hydrochloric acids (method 3010A).

3.5. Total Recoverable Metals: metals present in an unfiltered or dissolved sample following treatment with hot, dilute acid (method 3005A).

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include metallic or metal-containing lab ware, wearing improper gloves, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix A for additional contamination control guidelines.

4.3. Boron and silica from the glassware will migrate into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.7. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample.

4.8. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate

safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Vinyl gloves provide adequate protection against the chemicals and reagents typically used during this process. However, if any organic solvents are used, or any sample matrix contains organic solvents, only nitrile gloves should be used.

5.1.2. The use of vacuum systems during filtration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.3. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.4. The acidification of samples containing reactive materials may result in the release of toxic gasses, such as cyanides or sulfides. Acidification of samples must be done in a fume hood.

5.1.5. When digesting samples on a hot plate or digestion block, heat protective gloves and/or hot tongs must be used when handling containers. Ensure that digestion containers are arranged safely and are not overcrowded.

5.1.6. Filtering, rinsing, and adjusting the volume of samples (when necessary) creates a significant risk of ergonomic injuries due to repetition and use of excessive force. Analysts performing these tasks must ensure that they do not perform any single task like this for more than 30 minutes without taking an ergonomic break or working on a different task. Analysts with smaller hands should ensure that they use 500 mL squirt bottles rather than 1 liter bottles, or use varying techniques (other than squirt bottles) for rinsing and volume adjustment when there are numerous samples to be processed.

5.1.7. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.1.8. Acid washing of glassware is classified as a high-risk activity. A face shield must be worn over safety glasses or safety goggles during this process.

5.1.9. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and

reagents are being handled.

5.1.10. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred, prepared, digested and cooled in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.11. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Digestion block capable of maintaining a temperature at approximately 95°C.

6.2. Calibrated thermometers that cover a temperature range of 0-200°C.

6.3. Disposable graduated block digestion tubes.

6.4. Whatman No. 41 filtration paper.

6.5. Bottle-top dispensers.

6.6. Adjustable air displacement pipettes.

6.7. pH indicator strips (pH range 0 - 14).

6.8. Polyethylene storage bottles.

6.9. Vacuum flask with filtration assembly.

6.10. Vacuum pump.

6.11. 0.45 um membrane filters.

7. REAGENTS AND STANDARDS

7.1. Use reagent grade chemicals in all tests. “Certificates of Analysis” should be supplied with all chemicals purchased. If not supplied, contact the vendor. When received, label the certificate and the reagent container with the receipt date. Reagent containers also need to be labeled with the opened and expiration dates.

7.2. Reagent water is produced by a Millipore nanopure system (SOP WS-QA-0014). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.3. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as certified custom solutions (Tables I, II). All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. All stock standards must be labeled with the date of receipt and the date the standard is opened.

7.4. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the

custom solution, the lab must purchase a solution from a designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.5. Nitric acid, concentrated: Analytical reagent grade

7.6. Hydrochloric acid, concentrated: Analytical reagent grade

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Samples submitted for "total recoverable" or "total" metals analysis should be preserved in the field to a pH <2 with the addition of HNO_3 after sampling. Prior to sample preparation every sample must be tested for proper preservation using pH paper.

8.3. For dissolved metals analysis, the samples should be filtered through a 0.45 um filter prior to preservation. Filtration must be done in the field or within 24 hours of collection. For filtration done by the laboratory the sample must be preserved with HNO_3 immediately following filtration.

8.4. Filtration and preservation done by the lab analyst must be documented by a NCM in the prep batch.

Note: If a sample being analyzed for dissolved metals is found to contain sediment the analyst should contact their supervisor or group leader. The client should be notified of the problem to decide how to treat the sample.

9. QUALITY CONTROL

9.1. Batch - A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards. A batch must be prepared within the same time frame. A method blank (MB) and a laboratory control sample (LCS) or duplicate control sample (LCS/LCSD) must be prepared as a part of every batch. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/SD), or in some instances a sample/sample duplicate. An analysis batch must include all QC samples, however they do not contribute to the maximum of 20 samples. See policy WS-PQA-003 of the Quality Control Program for more details.

9.2. One method blank (MB) must be prepared for every 20 samples. A method blank consists of reagent water processed through all of the steps, and at the same time as the associated samples. If a method blank exceeds +/- the reporting limit for a given analyte than the samples associated with that batch must be re-prepared. The exception

is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo. For samples requiring laboratory filtration and preservation, the method blank must also be filtered and preserved. See policy WS-PQA-003 of the Quality Control Program for further details.

9.3. A laboratory control sample (LCS) must be prepared for every 20 samples. A LCS consists of reagent water spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-prepared except when a LCS exhibits high recovery. For such a case, those samples with analyte concentrations less than the reporting limit can be reported. All corrective actions must be documented on a Non-conformance memo. See policy WS-PQA-003 of the Quality Control Program for further details.

9.3.1. For methods 6010C and 6020A, a Low Level Control Sample (LLCS) must be prepared quarterly. The LLCS is spiked at the Reporting Limit (RL).

9.4. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be prepared with every process batch of similar matrix, not to exceed twenty (20) samples. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Re-extraction of the blank, LCS, selected field samples, and the MS/MSD may be required after evaluation and review of the MS/MSD results.

Note: Samples identified as field blanks, equipment blanks, or trip blanks should not be used for sample/sample duplicate nor MS/MSD analysis.

9.5. A duplicate control sample (LCS/LCSD) may be prepared when insufficient volume is provided to process a sample/sample duplicate or MS/MSD pair if required by client or program. The LCS and LCSD are evaluated independently for acceptance. See policy WS-PQA-003 “Quality Control Program” for further details.

10. CALIBRATION

10.1. A digestion vessel filled with reagent water is monitored for temperature for each digestion block. The analyst is to monitor this temperature for accuracy throughout the digestion and record the initial and final temperatures on the sample digestion log.

10.2. All air displacement pipettes must be verified over their range of use periodically as described in SOP WS-QA-0004.

10.3. All volumetric digestion vessels must be verified according to the frequency and procedures outlined in SOP WS-QA-0004. If the analyst suspects a vessel's reliability, a new verification of vessel may be required.

10.4. Pump-style dispensers for non-quantitative reagents (acids, hydrogen peroxide, etc.) are not subject to the periodic verification described in SOP WS-QA-0004. The amounts of nitric acid, hydrochloric acid, and peroxide specified in this SOP are considered nominal, rather than exact, values.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. All preparation procedures must be carried out in a properly functioning hood.

11.3. All samples are to be checked out and back into sample control with the chain of custody documentation filled out completely. Samples are to be returned to the sample control area once all of the digestions have been initiated.

11.4. Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be accurate and legible. Always set the samples up on the sample cart in the order with which they are to be dispensed. Double-check the sample bottle IDs, once prior to pouring them into the digestion vessels and before they are returned to the sample cart.

11.5. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/plant) are usually logged in with solid test codes. When initiating preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge-like, organic liquid, lots of sediment etc.) contact the lab supervisor or project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.6. Always read the QAS for every project prior to establishing batch associations. This is meant to assure that all client requirements are satisfied and it also aids in creating

more efficient batches. Setting up batches to minimize QC and meet all client requirements is a skill that must be developed by all new personnel with the aid of skilled sample preparation and analytical staff.

11.7. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.8. The following procedure must be followed for all aqueous sample preparations:

11.8.1. Use a small aliquot of each sample and measure the pH with pH indicator strips. If a bottle has a "X" on the top, it has already been tested for preservation and does not require this step.

11.8.1.1. For samples with pH < 2 put an "X" on top of the bottle indicating the samples were properly preserved. For these samples, record pH < 2 on the preparation log.

11.8.1.2. For samples with pH > 2, notify the project manager immediately so they can contact the client. If the client requests the sample be preserved by the laboratory, add HNO_3 in 1.0 mL aliquots until the sample remains at pH < 2 for at least 10 minutes. Let the sample stand for 24 hours and re-test the pH prior to preparation. As long as the pH < 2, the sample can be digested. A non-conformance memo must be created with an account of the anomalous event and the corrective action. Put a "X" on top of the bottle indicating the sample was properly preserved.

11.8.2. Mix the sample by shaking the container and pour the aliquot immediately. Shake the container between pouring the sample, MS, and MSD.

11.8.3. Measure and transfer 50 mL of each sample into a graduated disposable digestion tube.

11.8.4. Measure two extra aliquots of the sample selected for the MS/MSD analysis.

11.8.5. Measure and transfer 50 mL of reagent water into digestion tubes for the method blank, LCS and LCSD (if required).

11.8.6. Using an adjustable volumetric air displacement pipette spike the LCS, LCSD, and MS/SD with the appropriate spiking solution/s shown in Tables I and II. ICP spikes require 0.5 mL of spiking solution and ICPMS spikes are spiked with 100 µL of spiking solution. A chemist trained in metals preparation and/or analysis must always witness the spiking procedure.

11.8.7. Using an adjustable volumetric air displacement pipette, spike the LLCS with the appropriate spiking solution/s shown in Tables 1.1 and 2.2. ICP spikes

require 0.5 mL of spiking solution and ICPMS spikes are spiked with 0.5 mL of spiking solution. A chemist trained in metals preparation and/or analysis must always witness the spiking procedure.

11.9. Proceed to the appropriate section for the desired method as follows:

Method 3005A, 200.8-5.4	11.10
Method 3010A, 200.7-4.4	11.11

11.10. Method 3005A - preparation for total recoverable or dissolved metals analysis by ICP or ICPMS (see Figure 1).

11.10.1. To each digestion tube, add 1 mL of concentrated HNO_3.

11.10.2. For ICP (method 6010B, 200.7-4.4) digests, add 2.5 mL of concentrated HCl. For ICPMS (method 6020, 200.8-5.4) digests, add 0.5 mL of concentrated HCl.

11.10.3. Heat at 90 - 95°C on a block digester until the volume is reduced to between 15 and 20 mL. Monitor the temperature regularly to assure proper heating.

Note: Do not allow samples to boil or go dry. Doing so will result in the loss of analytes and the samples must be re-prepared.

11.10.4. Cool the digestion tubes in a fume hood.

11.10.5. Adjust the final volume to 50 mL with reagent water. The sample is now ready for analysis

11.10.6. If insoluble material is present in the digestates, filter through Whatman 41 filter paper.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Warning: Filtering, rinsing, and adjusting the volume of samples (when necessary) creates a significant risk of ergonomic injuries due to repetition and use of excessive force. Analysts performing these tasks must ensure that they do not perform any single task like this for more than 30 minutes without taking an ergonomic break or working on a different task. Analysts with smaller hands should ensure that they use 500 mL squirt bottles rather than 1 liter bottles, or use varying techniques (other than squirt bottles) for rinsing and volume adjustment when there are numerous samples to be processed.

Warning: The use of vacuum systems during filtration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked,

rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

11.11. Method 3010A - preparation for total metals analysis by ICP (See Figure 2).

11.11.1. To the sample digestion tubes, add 1.5 mL of concentrated HNO_3.

11.11.2. Heat at approximately 95°C on block digesters until volume is reduced to between 15-20 mL. Monitor the temperature regularly to assure proper heating.

Note: Do not allow samples to boil or go dry. Doing so will result in the loss of analytes and the samples must be re-prepared.

11.11.3. Cool the digestion tubes in a fume hood to ambient temperature.

11.11.4. Add another 1.5 mL portion of concentrated HNO_3.

11.11.5. Continue heating on the block digester for 20-25 minutes while ensuring that no portion of the bottom of the digestion tubes are allowed to go dry.

Note: If any sample shows a significant release of brown fumes while heating, add one more 1.5 mL aliquot of concentrated HNO_3 and heat for approximately 10 minutes.

11.11.6. Cool the digestion tubes in a fume hood to ambient temperature.

11.11.7. Add 2.5 mL of concentrated HCl.

11.11.8. Heat for an additional 15 minutes to dissolve precipitate or residue.

11.11.9. Cool the digestion tubes in a fume hood.

11.11.10. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

11.11.11. If insoluble material is present in the digestates, filter through Whatman 41 filter paper.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Warning: Filtering, rinsing, and adjusting the volume of samples (when necessary) creates a significant risk of ergonomic injuries due to repetition and use of excessive force. Analysts performing these tasks must ensure that they do not perform any single task like this for more than 30 minutes without taking an ergonomic break or working on a different task. Analysts with smaller hands should ensure that they use 500 mL squirt bottles rather than 1 liter bottles, or

use varying techniques (other than squirt bottles) for rinsing and volume adjustment when there are numerous samples to be processed.

Warning: The use of vacuum systems during filtration presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

12. CALCULATIONS/DATA REDUCTION

Not Applicable.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Acidic waste generated by the extraction and unused acidic digestate containing nitric and hydrochloric acid. This acidic liquid is consolidated into a plastic acid waste drum. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Contaminated disposable materials such as plastic vials, pipettes, and filters used during sample preparation and digestion. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A, 3010A.

16.2. Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983, 1994. Methods 200.7, Revision 4.4, 1994; 200.8, Revision 5.4, 1994.

16.3. WS-MT-0003, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010B and Method 200.7.

16.4. WS-MT-0001, Analysis of Metals by Inductively coupled Plasma/Mass Spectrometry.

17. METHOD MODIFICATIONS

17.1. Modifications applicable to SW-846 reference methods.

17.1.1. The totals sample volume has been reduced from 100 mL to 50 mL to accommodate hot block digestion.

17.2. Modifications Specific to Method 3005A

17.2.1. Method 3005A is being used for the preparation of "total recoverable" metals in drinking waters.

17.2.2. The amount of HCl added is reduced to 0.5 mL for ICPMS digests.

17.3. Modifications Specific to Method 3010A

17.3.1. Section 11.12.2 of this SOP requires the sample be reduced to a volume of 15 – 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL but also states that no portion of the bottom of the digestion tube should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the digestion tube so as to prevent the loss of critical analytes through volatilization.

17.3.2. The scope of 3010A has been expanded to include silver based on comparison studies with 7760A. Method 3010A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water and TCLP leachate) up to a concentration of 1 ppm silver.

17.4 Modifications Specific to Method 200.7-4.4

17.4.1 The initial sample volume has been reduced from 100 mL to 50 mL to accommodate hot block digestion.

17.4.2 The addition of 2 mL (1:1) nitric acid and 1.0 mL of (1:1) hydrochloric acid has been changed to 1 mL of nitric acid and 2.5 mL of hydrochloric acid.

17.5 Modifications Specific to Method 200.8-5.4

17.5.1 The initial sample volume has been reduced from 100 mL to 50 mL to accommodate hot block digestion.

17.5.2 The addition of 2 mL (1:1) nitric acid and 1.0 mL of (1:1) hydrochloric acid has been changed to 1 mL of nitric acid and 0.5 mL of hydrochloric acid.

18. ATTACHMENTS

18.1. Figure 1 — Method 3005A Flow Diagram

18.2. Figure 2 — Method 3010A Flow Diagram

18.3. Table 1 — Analyte List Certified Under NELAC Criteria: Method 6010B and Method 200.7

18.4. Table 1.1 — Analyte List Certified under NELAC Criteria: Method 6010B – Low Level Control Sample (LLCS)

18.5. Table 2 — Analyte List Certified Under NELAC Criteria: Method 6020 and Method 200.8

18.6. Table 2.1 — Analyte List Certified Under NELAC Criteria: Method 6020 - Low Level Control Sample (LLCS)

18.7. Appendix A — Contamination Control Guidelines

19. REVISION HISTORY

19.1. WS-IP-0001, Revision 5.6, Effective 04/25/2017

19.1.1. Section 9.3.1, changed, “For methods 6010B and 6020A a Low Level Control Sample (LLCS) must be prepared for every 20 samples” to “For methods 6010C and 6020A a Low Level Control Sample (LLCS) must be prepared quarterly.”

19.1.2. Section 11.8.2 changed “SD” to “MSD.”

19.1.3. Editorial changes.

19.2. WS-IP-0001, Revision 5.5, Effective 08/26/2016

19.2.1. Added Section 10.4 – “Pump-style dispensers for non-quantitative reagents (acids, hydrogen peroxide, etc.) are not subject to the periodic verification described in SOP WS-QA-0004. The amounts of nitric acid, hydrochloric acid, and peroxide specified in this SOP are considered nominal, rather than exact, values.”

19.2.2. Editorial changes.

19.3. WS-IP-0001, Revision 5.4, Effective 12/12/2014

19.3.1. Inserted Section 2.3 – “Method 200.7-4.4 and Method 200.8-5.4 are applicable to the preparation of total recoverable metals by ICP and ICPMS. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to the original volume.

19.3.2. Inserted Section 17.4 – “Modifications Specific to Method 200.7-4.4:”

19.3.3. Inserted Section 17.5 – "Modifications Specific to Method 200.8-5.4"

19.3.4. Editorial changes.

19.4. WS-IP-0001, Revision 5.3, Effective 03/28/2014

19.4.1. Inserted Section 9.3.1 - For methods 6010B and 6020A a Low Level Control Sample (LLCS) must be prepared for every 20 samples.

19.4.2. Appended Section 9.3.1 – "The LLCS is spiked at the Reporting Limit (RL)"

19.4.3. Section 11.8.8.2, removed from the paragraph, "Record "AF" for "Anomaly Filed" on the digestion log."

19.4.4. Inserted Section 11.8.7 – "Using an adjustable volumetric air displacement pipette spike the LLCS with the appropriate spiking solution/s shown in Tables 1.1 and 2.2. ICP spikes require 0.5 mL of spiking solution and ICPMS spikes are spiked with 0.5 mL of spiking solution. A chemist trained in metals preparation and/or analysis must always witness the spiking procedure."

19.4.5. Inserted Tables 1.1 and 2.1.

19.4.6. Editorial changes.

19.5. WS-IP-0001, Revision 5.2, Effective 10/31/2012

19.5.1. Removed all references to Methods 200.7 and 200.8 from SOP.

19.5.2. Editorial changes.

19.6. WS-IP-0001, Revision 5.1, Effective 10/02/2009

19.6.1. Changed Section 11.8.6 "… ICPMS spikes are spiked with 1.0 mL…" to "…ICPMS spikes are spiked with 100 µL".

19.6.2. Changed "Table 2 - Analyte List Certified Under NELAC Criteria, Method 6020" Source from Spex XCAL-45 to Spec XCAL-58 and each of the Initial Concentrations except Hg are increased by a factor of 10x.

19.7. WS-IP-0001, Revision 5, Effective 09/15/2008

19.7.1. Updated to TestAmerica format.

Figure 1
Method 3005A

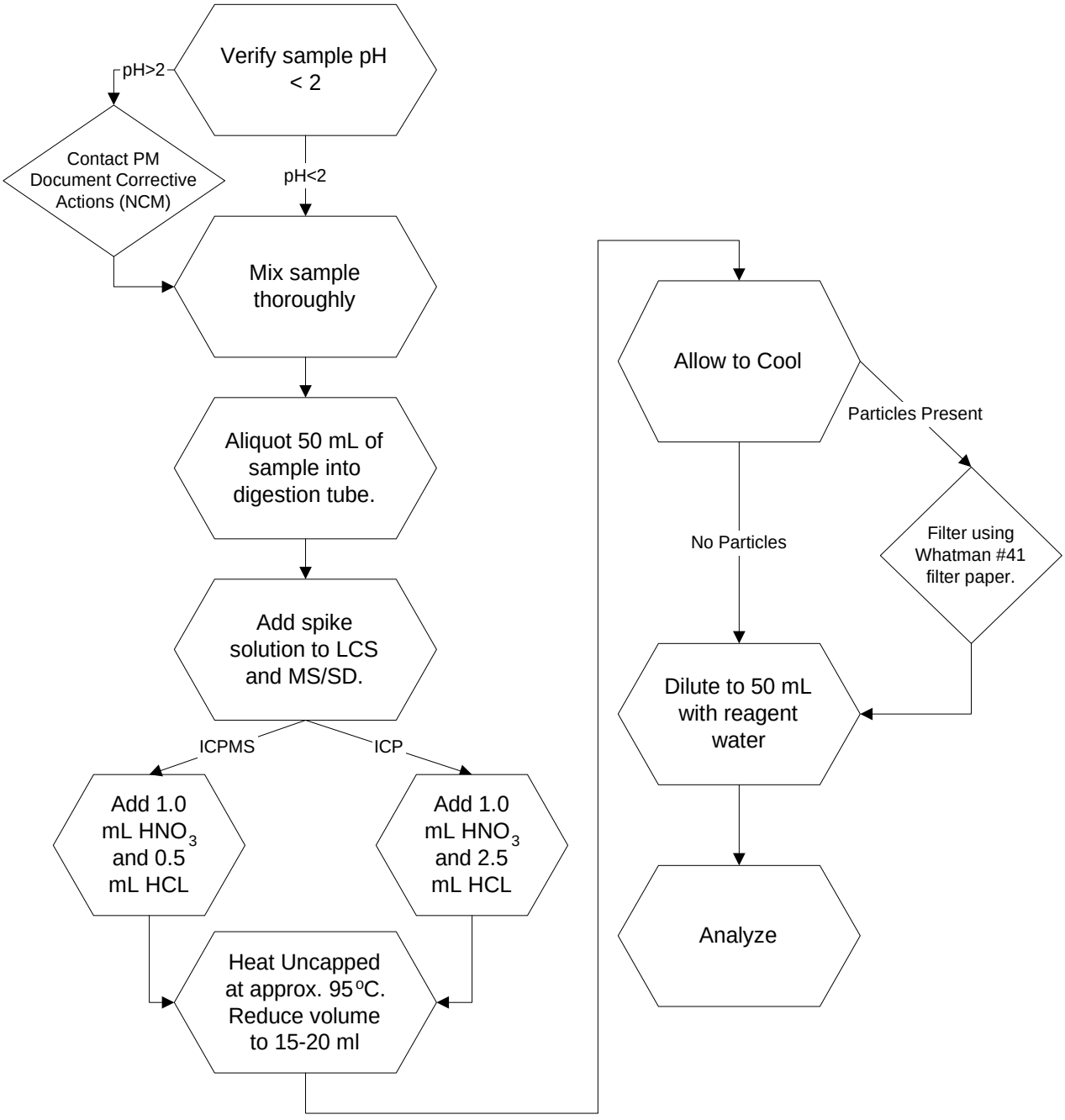

Figure 2

Method 3010A

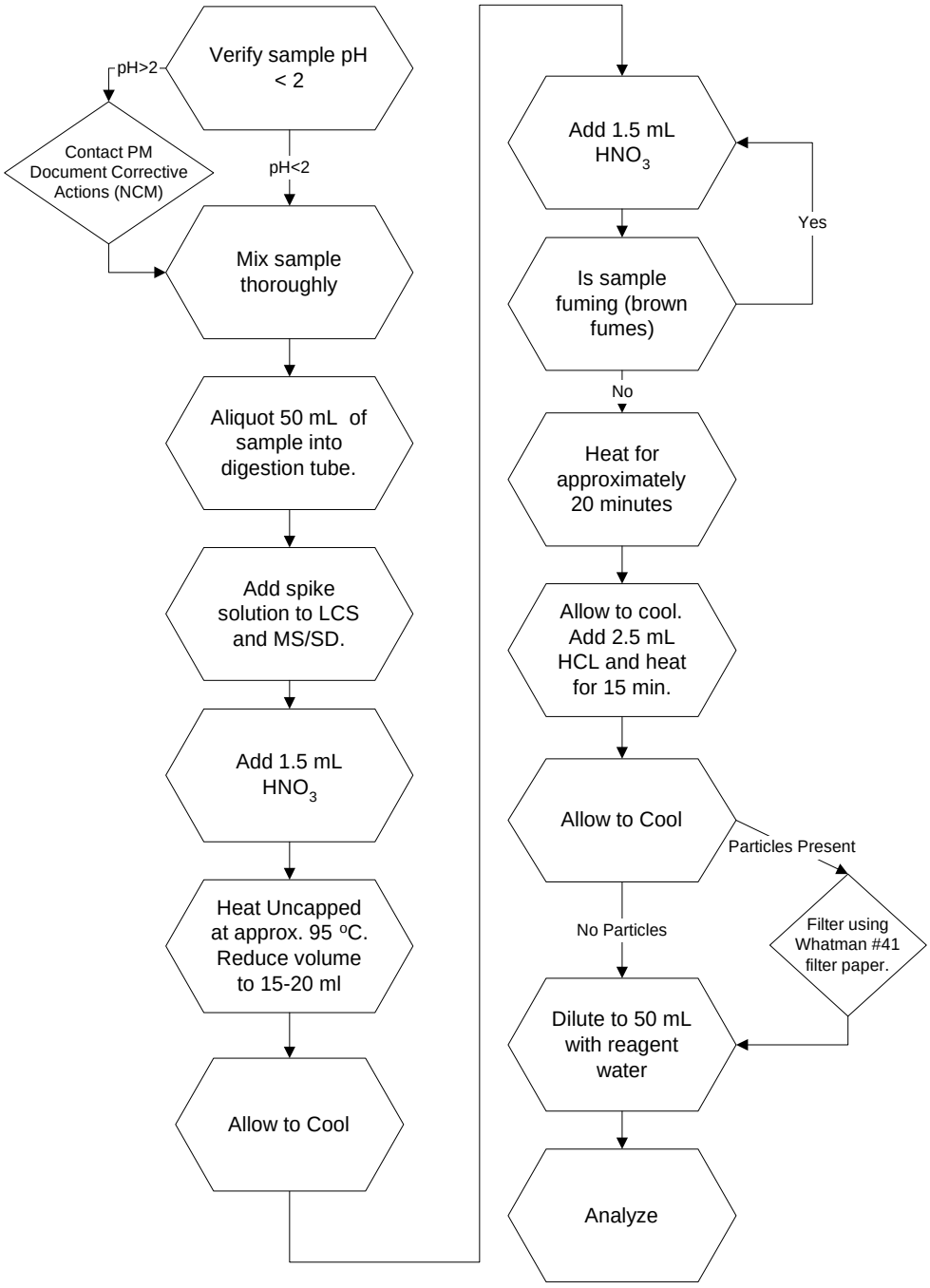

TABLE 1.

Method 6010B & Method 200.7

Element	Symbol	Initial Conc mg/L	Final Conc mg/L	Source
Aluminum	Al	200	2.0	6010-SPIKE-1
Antimony	Sb	50	0.5	6010-SPIKE-2
Arsenic	As	200	2.0	6010-SPIKE-1
Barium	Ba	200	2.0	6010-SPIKE-1
Beryllium	Be	5.0	0.05	6010-SPIKE-1
Boron	B	100	1.0	6010-SPIKE-1
Cadmium	Cd	5.0	0.05	6010-SPIKE-1
Calcium	Ca	5000	50	6010-SPIKE-1
Chromium	Cr	20	0.2	6010-SPIKE-1
Cobalt	Co	50	0.5	6010-SPIKE-1
Copper	Cu	25	0.25	6010-SPIKE-1
Iron	Fe	100	1.0	6010-SPIKE-1
Lead	Pb	50	0.5	6010-SPIKE-1
Lithium	Li	100	1.0	6010-SPIKE-1
Magnesium	Mg	5000	50	6010-SPIKE-1
Manganese	Mn	50	0.5	6010-SPIKE-1
Molybdenum	Mo	100	1.0	6010-SPIKE-2
Nickel	Ni	50	0.5	6010-SPIKE-1
Phosphorous	P	1000	10	6010-SPIKE-1
Potassium	K	5000	50	6010-SPIKE-1
Selenium	Se	200	2.0	6010-SPIKE-1
Silicon	Si	1000	10	Inorganic Ventures
Silver	Ag	5	0.05	6010-SPIKE-2
Sodium	Na	5000	50	6010-SPIKE-1
Strontium	Sr	100	1.0	6010-SPIKE-1
Sulfur	S	1000	10	6010-SPIKE-2
Thallium	Tl	200	2.0	6010-SPIKE-1
Tin	Sn	200	2.0	6010-SPIKE-2
Titanium	Ti	100	1.0	6010-SPIKE-2
Vanadium	V	50	0.5	6010-SPIKE-1
Zinc	Zn	50	0.5	6010-SPIKE-1

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE 1.1 : Method 6010B.

Low Level Control Sample (LLCS)

Element	Symbol	Initial Conc mg/L	Final Conc mg/L	Source
Aluminum	Al	0.5	0.005	Intermediate
Antimony	Sb	20	0.2	Intermediate
Arsenic	As	4	0.04	Intermediate
Barium	Ba	2	0.02	Intermediate
Beryllium	Be	0.3	0.003	Intermediate
Boron	B	20	0.2	Intermediate
Cadmium	Cd	0.3	0.003	Intermediate
Calcium	Ca	50	0.5	Intermediate
Chromium	Cr	1	0.01	Intermediate
Cobalt	Co	1	0.01	Intermediate
Copper	Cu	1.5	0.015	Intermediate
Iron	Fe	10	0.1	Intermediate
Lead	Pb	1	0.01	Intermediate
Lithium	Li	2	0.02	Intermediate
Magnesium	Mg	50	0.5	Intermediate
Manganese	Mn	1	0.01	Intermediate
Molybdenum	Mo	2	0.02	Intermediate
Nickel	Ni	1	0.01	Intermediate
Phosphorous	P	50	0.5	Intermediate
Potassium	K	100	1	Intermediate
Selenium	Se	4	0.04	Intermediate
Silicon	Si	50	0.5	Intermediate
Silver	Ag	0.5	0.005	Intermediate
Sodium	Na	50	0.5	Intermediate
Strontium	Sr	5	0.05	Intermediate
Sulfur	S	5	0.05	Intermediate
Thallium	Tl	3	0.03	Intermediate
Tin	Sn	2	0.02	Intermediate
Titanium	Ti	1	0.01	Intermediate
Vanadium	V	2	0.02	Intermediate
Zinc	Zn	2	0.02	Intermediate

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met

TABLE 2

Method 6020 & Method 200.8 Spiking

Element	Symbol	Initial Conc mg/L	Final Conc mg/L	Source
Aluminum	Al	500	1.0	TACA-1
Antimony	Sb	25	0.05	TACA-2
Arsenic	As	100	0.2	TACA-1
Barium	Ba	100	0.2	TACA-1
Berylliu m	Be	100	0.2	TACA-1
Boron	B	500	1.0	TACA-1
Cadmium	Cd	100	0.2	TACA-1
Calcium	Ca	500	1.0	TACA-1
Chromium	Cr	100	0.2	TACA-1
Cobalt	Co	100	0.2	TACA-1
Copper	Cu	100	0.2	TACA-1
Iron	Fe	500	1.0	TACA-1
Lead	Pb	100	0.2	TACA-1
Lithium	Li	100	0.2	TACA-1
Magnesium	Mg	500	1.0	TACA-1
Manganese	Mn	100	0.2	TACA-1
Mercury	Hg	1.0	0.005	Inorganic Ventures
Molybdenum	Mo	100	0.2	TACA-2
Nickel	Ni	100	0.2	TACA-1
Phosphorous	P	500	1.0	TACA-1
Potassium	K	500	1.0	TACA-1
Selenium	Se	100	0.2	TACA-1
Silver	Ag	25	0 .05	TACA-1
Sodium	Na	500	1.0	TACA-1

Strontium	Sr	100	0.2	TACA-1
Thallium	Tl	25	0.05	TACA-1
Tin	Sn	100	0.2	TACA-2
Titanium	Ti	100	0.2	TACA-2
Uranium	U	100	0.2	TACA-1
Vanadium	V	100	0.2	TACA-1
Zinc	Zn	100	0.2	TACA-1

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE 2.1 —Method 6020

Low Level Control Sample (LLCS)

Element		Symbol		Source
		Initial Conc mg/L	Final Conc mg/L	
Aluminum	Al	10	0.1	Intermediate
Antimony	Sb	0.6	0.006	Intermediate
Arsenic	As	0.3	0.003	Intermediate
Barium	Ba	0.15	0.0015	Intermediate
Beryllium	Be	0.1	0.001	Intermediate
Boron	B	5	0.05	Intermediate
Cadmium	Cd	0.15	0.0015	Intermediate
Calcium	Ca	10	0.1	Intermediate
Chromium	Cr	0.5	0.005	Intermediate
Cobalt	Co	0.3	0.003	Intermediate
Copper	Cu	0.3	0.003	Intermediate
Iron	Fe	10	0.1	Intermediate
Lead	Pb	0.2	0.002	Intermediate
Lithium	Li	1	0.01	Intermediate
Magnesium	Mg	10	0.1	Intermediate
Manganese	Mn	0.6	0.006	Intermediate
Mercury	Hg	0.002	0.00002	Intermediate
Molybdenum	Mo	0.3	0.003	Intermediate
Nickel	Ni	0.3	0.003	Intermediate
Phosphorous	P	5	0.05	Intermediate
Potassium	K	10	0.1	Intermediate
Selenium	Se	0.3	0.003	Intermediate

Silver	Ag	0.1	0.001	Intermediate
Sodium	Na	10	0.1	Intermediate
Strontium	Sr	2	0.02	Intermediate
Thallium	Tl	0.15	0.0015	Intermediate
Tin	Sn	1	0.01	Intermediate
Titanium	Ti	0.5	0.005	Intermediate
Uranium	U	0.1	0.001	Intermediate
Vanadium	V	1.2	0.012	Intermediate
Zinc	Zn	1.2	0.012	Intermediate

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

APPENDIX A.

CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water. Disposable lab ware should be used whenever possible.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Title: Acid Digestion of Soils, SW-846 Method 3050B [Method EPA 3050B]

Approvals (Signature/Date):

for 8.16.16	8/16/16
Robert Hrabak Technical Manager Date	Joe Schairer Health & Safety Manager / Coordinator Date
8/23/2016	8.16.16
Lisa Stafford Quality Assurance Manager Date	Crystal Pollock Laboratory Director Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soils, sediments, wastes, and other miscellaneous solid samples for the analysis of metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Inductively Coupled Plasma Atomic Emission Mass Spectroscopy (ICP/MS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICP/MS for the elements listed in Tables I and II. Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve most all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment

2. SUMMARY OF METHOD

A representative 1 g to 2 g (wet weight) portion of sample is digested in nitric acid, hydrogen peroxide, and hydrochloric acid. The extractions are then filtered and diluted to 100 mL.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing lab ware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix A for additional contamination control guidelines.

4.3. Boron from borosilicate glassware will leach into the sample solution during and following sample processing. For critical low level determinations of boron only quartz and/or plastic lab ware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric media.

4.7. Specific analytical interferences are discussed in each of the determinative methods.

4.8. Available silica is lost during the preparation and therefore silica is not analyzed in solid matrices.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Opening, closing, filtering, rinsing, and adjusting the volume of samples (when necessary) creates a significant risk of ergonomic injuries due to repetition and use of excessive force. Analysts performing these tasks must ensure that they do not perform any single task like this for more than 30 minutes without taking an ergonomic stretch break, or working on a different task. Analysts with smaller hands should ensure that they use 500 mL squirt bottles rather than 1 liter bottles, or use varying techniques (other than squirt bottles) for rinsing and volume adjustments when there are numerous samples to be processed. There are numerous wrenches and tools available to reduce the impact of opening large numbers of sample containers or digestion tubes.

5.1.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.3. The acidification of samples containing reactive materials may result in the release of toxic gasses, such as cyanides or sulfides. Acidification of samples

must be done in a fume hood.

5.1.4. When digesting samples on a hot plate or digestion block, heat protective gloves and/or hot tongs must be used when handling containers. Ensure that digestion containers are arranged safely and are not overcrowded.

5.1.5. Acid washing of glassware is classified as a high-risk activity. A face shield must be worn over safety glasses or over safety goggles during this process.

5.1.6. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Vinyl and nitrile gloves all provide satisfactory protection against the reagents used in this process. However, if any organic solvents are used, or any sample matrix contains organic solvents, only nitrile gloves should be used.

5.1.7. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred, prepared, digested and cooled in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.8. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and very irritating to the respiratory tract, eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Hot block digestion unit capable of maintaining a temperature of 90-95°C.

6.2. Digestion tubes: graduated volume relative to type of hot block.

6.3. Thermometer that covers a temperature range of 0-200°C.

6.4. Whatman No. 41 filter paper.

6.5. Wooden tongue depressors or stainless steel spatulas.

6.6. Top-loading balance capable of accurately weighing to the nearest 0.01 g.

6.7. Disposable watch glasses.

6.8. Bottle-top dispensers.

6.9. Calibrated adjustable air displacement pipettes.

6.10. 4 oz graduated snap cap containers for final transfer of digestions.

6.11. Teflon® boiling chips.

7. REAGENTS AND STANDARDS

7.1. Use reagent grade chemicals in all tests. "Certificates of Analysis" should be supplied with all chemicals purchased. If not supplied, contact the vendor. When received, label the certificate and the reagent container with the receipt date. Reagent containers also need to be labeled with the opened and expiration dates.

7.2. Reagent water is produced by a Millipore nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.3. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as certified custom TestAmerica solutions (Tables I, II). All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. All stock standards must be labeled with the date of receipt and the date the standard is opened.

7.4. The LCS and MS samples must contain all the elements designated for analysis. If a non-routine element is required that is not contained in the custom TestAmerica solution, the lab must purchase a solution from a designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.5. Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.6. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.7. 1:1 Hydrocloric acid: add 500 mL of concentrated HCl to 500 mL of reagent water.

7.8. 30% Hydrogen peroxide (H_2O_2), reagent grade.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Soil samples do not require preservation but must be stored at 0 to 6°C until the time of analysis.

9. QUALITY CONTROL

9.1. Batch - A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards. A batch must be prepared within the same time frame. A method blank (MB) and a laboratory control sample (LCS) or duplicate control sample (LCS/LCSD) must be prepared as a part of every batch. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/SD), or in some instances a sample/sample duplicate. An analysis batch must include all QC samples, however they do not contribute to the maximum of 20 samples. See policy WS-PQA-003 of the Quality Control Program for more details.

9.2. One method blank (MB) must be prepared for every 20 samples. A method blank consists of boiling chips processed through all of the steps, and at the same time as the associated samples. If a method blank exceeds +/- the reporting limit for a given analyte than the samples associated with that batch must be re-prepared. The exception is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo. For DOD projects, the MB criteria is less than ½ the reporting limit. See policy WS-PQA-003 of the Quality Control Program for further details.

9.3. A laboratory control sample (LCS) must be prepared for every 20 samples. A LCS consists of boiling chips spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-prepared except when a LCS exhibits high recovery. For such a case, those samples with analyte concentrations less than the reporting limit can be reported. All corrective actions must be documented on a Non-conformance memo. See policy WS-PQA-003 of the Quality Control Program for further details.

9.4. A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair must be prepared with every process batch of similar matrix, not to exceed twenty (20) samples. MS/MSD pairs are aliquots of a selected field sample spiked with all of the analytes of interest at known concentrations. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Re-extraction of the blank, LCS, selected field samples, and the MS/MSD may be required after evaluation and review of the MS/MSD results.

9.5. A duplicate control sample (LCS/LCSD) may be substituted when insufficient volume is provided to process a MS/MSD pair as required by client or regulatory requirements. The LCS and LCSD are evaluated independently for acceptance. See policy WS-PQA-003 of the Quality Control Program for further details

9.6. Quality Assurance Summaries: certain clients may require specific project or program QA information that supersedes the SOP requirements. Quality Assurance Summaries (QAS) should be developed by the PM's to address these requirements. Sample preparation analysts are required to read and print each QAS for every project they prepare. These are transferred to the instrument analysts with the sample digestions.

10. CALIBRATION

10.1. A digestion vessel filled with reagent water is monitored for temperature for each digestion block. The analyst is to monitor this temperature for accuracy throughout the digestion and record the initial and final temperatures on the sample digestion log.

10.2. All air displacement pipettes must be calibrated over their range of use at least monthly. If the analyst suspects a spiking volume problem, calibration may be required more often. See SOP WS-QA-0004 for pipettor calibration verification procedures.

10.3. All volumetric digestion vessels must be calibrated according to the frequency and procedures outlined in SOP WS-QA-0004. If the analyst suspects a vessel's reliability, calibration may be required more often.

10.4. Pump-style dispensers for non-quantitative reagents (acids, hydrogen peroxide, etc.) are not subject to the periodic verification described in SOP WS-QA-0004. The amounts of nitric acid, hydrochloric acid, and peroxide specified in this SOP are considered nominal, rather than exact, values.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. All preparation procedures must be carried out in a properly functioning hood.

11.3. All samples are to be checked out and back into sample control with the chain of custody documentation filled out completely. Samples are to be returned to the sample control area once all of the digestions have been initiated.

11.4. Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be accurate and legible. Always set the samples up on the sample cart in the order with which they are to be dispensed. Double-check the sample bottle IDs, once prior to aliquotting them into the digestion vessels and before they are returned to the sample cart.

11.5. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludge and tissues (animal/plant) are usually logged in with solid test codes. When initiating preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as solid but it appears more like a liquid, contact the department manager or project manager for further instructions.

11.6. Always read the QAS for every project prior to establishing batch associations. This is meant to assure that all client requirements are satisfied and it also aids in creating more efficient batches. Setting up batches to minimize QC and meet all client requirements is a skill that must be developed by all new personnel with the aid of skilled sample preparation and analytical staff.

11.7. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.8. Preparation of Soils, Sediments and Sludges for Analysis by ICP and ICP/MS.

11.8.1. SOP WS-QA-0018 must be followed to properly homogenize and aliquot solid samples.

11.8.2. Weigh out a 1.00 – 2.00 g sample aliquot into a graduated hot block tube using a wooden tongue depressor.

11.8.2.1. A 1.00 g +/- 5% aliquot is normal for samples that show no apparent free liquid phase or glistening from an aqueous phase.

11.8.2.2. A 1.4 g – 1.5 g aliquot should be used for samples with a muddy, glistening texture or small amount of free liquid.

11.8.2.3. A 2.0 g +/- 5% aliquot should be used for samples with a notable free liquid phase that does not mix into a structured muddy texture.

11.8.3. When sub-sampling for the MS/MSD sample aliquots, first transfer the sample per section 11.9.1 into a 50 mL polystyrene beaker and mix thoroughly before weighing the three replicates into the graduated hot block tubes.

WARNING: Opening, closing, filtering, rinsing, and adjusting the volume of samples (when necessary) creates a significant risk of ergonomic injuries due to repetition and use of excessive force. Analysts performing these tasks must ensure that they do not perform any single task like this for more than 30 minutes

without taking an ergonomic stretch break, or working on a different task. Analysts with smaller hands should ensure that they use 500 mL squirt bottles rather than 1 liter bottles, or use varying techniques (other than squirt bottles) for rinsing and volume adjustments when there are numerous samples to be processed. There are numerous wrenches and tools available to reduce the impact of opening large numbers of sample containers or digestion tubes.

11.8.4. Weigh out approximately 1g of Teflon® boiling chips into digestion tubes for MB and LCS/LCSD. For ICPMS, spike 0.2 mL of spiking solution into the LCS/MS/MSD. For ICP, use 1.0 mL of spiking solution. Tables I and II show the appropriate solutions to use and their concentrations. The final spiking levels are also listed.

11.8.5. Add 5 mL of reagent water to all of the digestion vessels.

11.8.6. Add 5 mL of concentrated HNO_3 and heat the samples to 90-92ºC and reflux for 10 minutes without boiling. For ICPMS only, add 5 mL of 1:1 HCl prior to heating.

11.8.7. Heat the samples to 90-92ºC and reflux for 10 minutes without boiling. Allow the samples to cool.

Note: Do not allow samples to boil or go dry during any part of the digestion. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.8.8. Add 5 mL of concentrated HNO_3 for both ICP and ICPMS.

11.8.9. Reflux at 90-92°C for 30 minutes. Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.

11.8.10. If brown fumes are observed, add concentrated HNO_3 in 5 mL aliquot with 30 minute refluxing cycles at 90-92º until no more fumes are observed.

11.8.11. Allow the sample to evaporate to approximately 5 mL while ensuring that no portion of the bottom of the tube is allowed to go dry. Do not heat the samples for more than 2 hrs. Allow the samples to cool.

11.8.12. Add 2 mL of reagent water and 3 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

Note: If the samples are very dark or look organic in nature, add the 30% H_2O_2 in 1 mL aliquots with short heating cycles to assure sample is not lost due to excessive effervescence.

11.8.13. Heat the samples for 15 minutes. Allow the samples to cool.

11.8.14. Add 3 mL H_2O_2 and continue heating at 90-92ºC until the volume is reduced

to approximately 5 mL. Do not exceed 2.0 hrs.

11.8.15. If the samples are being prepared for ICP analyses, add 5 mL of concentrated HCl and reflux for an additional 15 minutes without boiling.

11.8.16. If the samples are being prepared for ICPMS, add 5 mL of 1:1 HCl and reflux for an additional 15 minutes without boiling.

11.8.17. Allow the samples to cool and filter through Whitman #41 filter paper directly into 4 oz graduated snap cap containers. Dilute to a final volume of 100 ml with reagent water.

11.9. Filtration procedure:

11.9.1. Carefully pour the contents of each of the hot block digestion tubes into folded filter paper on top of the 4 oz containers. Be careful not to lose any of the contents through splashing.

11.9.2. Allow the contents to flow through the filter paper. Rinse each of the hot block tubes twice through the filter paper with reagent water.

11.9.3. Using reagent water, rinse each of the filter papers three times allowing the contents to filter thoroughly between rinses.

11.9.4. Adjust the final volume of the digestates to 100 mL with reagent water.

12. CALCULATIONS/DATA REDUCTION

Not Applicable.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This

requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Acidic waste generated by the digestion and unused acidic digestate containing nitric and hydrochloric acid. This acidic liquid is consolidated into a plastic acidic waste drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Contaminated disposable materials such as plastic vials, pipettes, and filters used during sample preparation and digestion. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996. Method 3050B.

16.2. WS-PQA-003: Quality Control Program

16.3. WS-QA-0004: Calibration of Autopippetors, Autodispensers, and Volumetric Containers

16.4. WS-QA-0018: Subsampling

17. METHOD MODIFICATIONS

17.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2. Samples are not centrifuged, but always filtered with Whatman #41 filter paper.

17.3. Samples are not sieved prior to weighing unless special instructions are given.

17.4. 5.0 mL of 1:1 HCl is added to the ICPMS digestions during the initial addition of reagents as studies have shown this increases the performance of silver analysis.

17.5. Temperature conditions of 95ºC ± 5ºC have been modified to 90-92ºC due to loss of silver at higher temperatures.

17.6. During the second step of H_2O_2 addition, 3.0 mL is added to all standards and samples instead of the steady additions of 1.0 mL aliquots.

17.7. For the final addition of HCl to the ICP aliquots, 5.0 mL of concentrated HCl is added instead of 10 mL.

17.8. 5.0 mL of 1:1 HCl is added to the final preparation phase for ICPMS to increase the performance of silver.

18. ATTACHMENTS

18.1. Figure 1 — Method 3050B (Modified) Flowchart

18.2. Table 1 — Method 6010B Spiking

18.3. Table 2 — Method 6020 Spiking

18.4. Appendix 1 — Contamination Control Guidelines

19. REVISION HISTORY

19.1. WS-IP-0002, Revision 5.4, Effective 08/19/2016

19.1.1. Added Section 10.4 – "Pump-style dispensers for non-quantitative reagents (acids, hydrogen peroxide, etc.) are not subject to the periodic verification described in SOP WS-QA-0004. The amounts of nitric acid, hydrochloric acid, and peroxide specified in this SOP are considered nominal, rather than exact, values."

19.1.2. Section(s) 11.8.5, 11.8.6, 11.8.8, 11.8.10, Note following 11.8.12, 11.8.14, 11.8.15 and 11.8.16 – removed decimal place after volumes indicating that measurements are approximate and not precise.

19.1.3. Editorial changes.

19.2. WS-IP-0002, Revision 5.3, Effective 01/19/2012

19.2.1. Section 6.10: "Teflon boiling chips".

19.2.2. Section 11.8.4: added "Weigh out approximately 1.00g of Teflon® boiling chips into digestion tubes for MB and LCS/LCSD."

19.3. WS-IP-0002, Revision 5.2, Effective 12/17/2010

19.3.1. Section 11.8.4: Changed ICPMS spiking level from 2.0 mL to 0.2 mL.

19.3.2. Updated Table II Source and Initial Conc. Levels for all analytes.

19.4. WS-IP-0002, Revision 5.1, Effective 10/19/2009

19.4.1. Revise Section 11.8.4 to read, "For ICPMS, spike 2.0 mL of spiking solution into the LCS/MS/MSD. For ICP, use 1.0 mL of spiking solution. Tables I and II show the appropriate solutions to use and their concentrations. The final spiking levels are also listed."

19.4.2. Revise Section 11.8.5 to read, "Add 5.0 mL of reagent water to all of the digestion vessels."

19.4.3. Add Section 11.9.4 to read, "Adjust the final volume of the digestates to 100 mL with reagent water."

19.4.4. Revised the flow chart (Figure 1) to reflect the changes above.

19.5. WS-IP-0002, Revision 5, Effective 09/30/2008

19.5.1. Updated to current TestAmerica format.

19.5.2. Added HCl to last acid addition for ICPMS analysis.

19.5.3. Change volumes to approximately 5 mL from 5-10 mL for reduction steps.

19.6. WS-IP-0002, Rev. 4, Effective 9/11/07

19.6.1. The SOP format was updated to TestAmerica format.

19.6.2. Reference to Silica was removed.

Figure 1: Method 3050B (modified) Flowchart

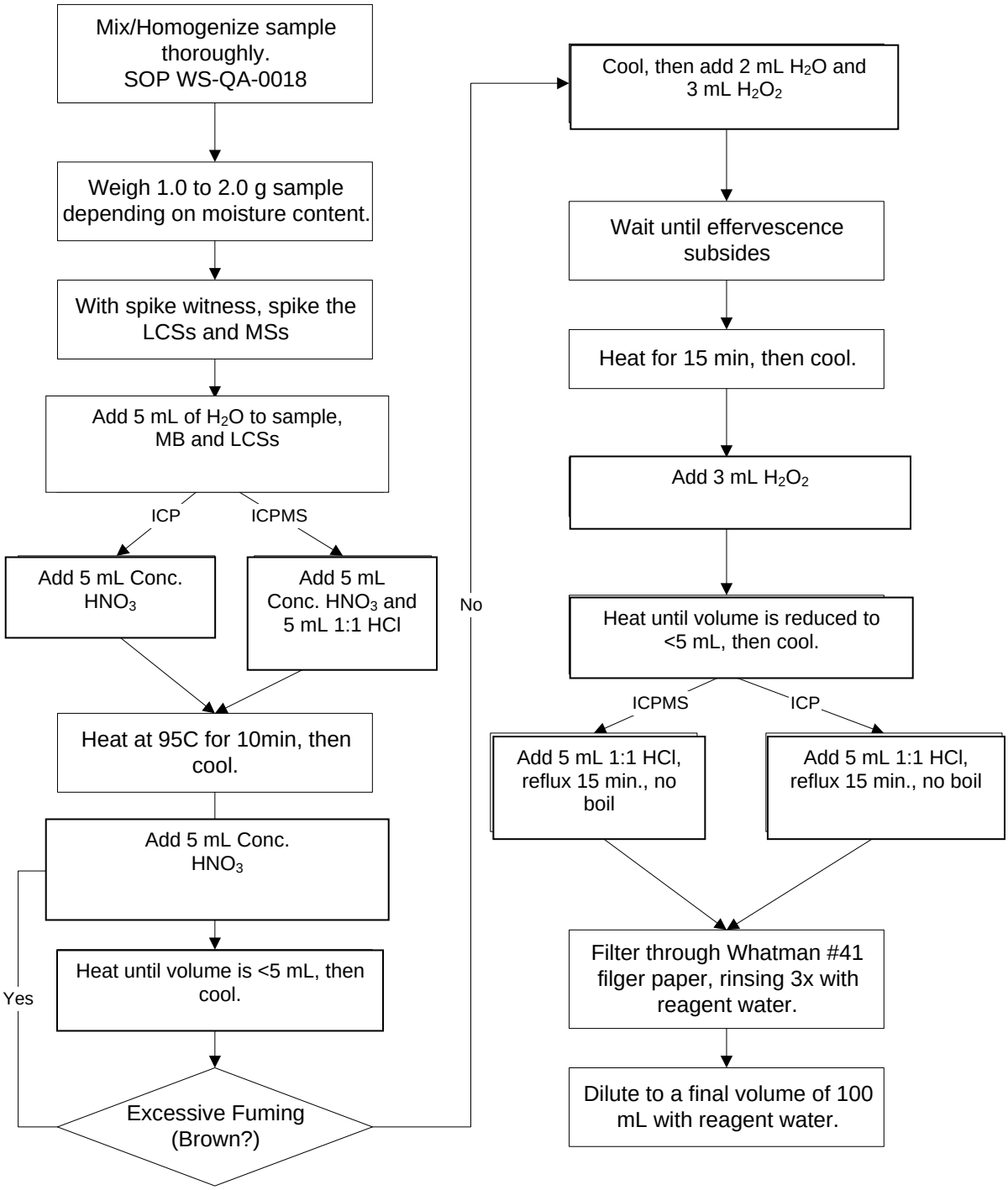

TABLE I Method 6010B Spiking				
		LCS/MS/SD Spiking		
Element	Symbol	Initial Conc. mg/L	Final Conc. mg/Kg	Source
Aluminum	Al	200	200	Spex XCAL-36
Antimony	Sb	50	50	Spex XCAL-36
Arsenic	As	200	200	Spex XCAL-36
Barium	Ba	200	200	Spex XCAL-36
Beryllium	Be	5.0	5.0	Spex XCAL-36
Boron	B	100	100	Spex XCAL-38
Cadmium	Cd	5.0	5.0	Spex XCAL-36
Calcium	Ca	5000	5000	Spex XCAL-36
Chromium	Cr	20	20	Spex XCAL-36
Cobalt	Co	50	50	Spex XCAL-36
Copper	Cu	25	25	Spex XCAL-36
Iron	Fe	100	100	Spex XCAL-36
Lead	Pb	50	50	Spex XCAL-36
Lithium	Li	100	100	Spex XCAL-38
Magnesium	Mg	5000	5000	Spex XCAL-36
Manganese	Mn	50	50	Spex XCAL-36
Molybdenum	Mo	100	100	Spex XCAL-36
Nickel	Ni	50	50	Spex XCAL-36
Phosphorous	P	1000	1000	Spex XCAL-38
Potassium	K	5000	5000	Spex XCAL-38
Selenium	Se	200	200	Spex XCAL-36
Silver	Ag	5	5	Spex XCAL-36
Sodium	Na	5000	5000	Spex XCAL-38
Strontium	Sr	100	100	Spex XCAL-38
Sulfur	S	1000	1000	Spex XCAL-38
Thallium	Tl	200	200	Spex XCAL-36
Tin	Sn	200	200	Spex XCAL-36
Titanium	Ti	100	100	Spex XCAL-36
Vanadium	V	50	50	Spex XCAL-36
Zinc	Zn	50	50	Spex XCAL-36

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE II
Analyte List Certified Under NELAC Criteria: Method 6020

Element		Symbol		Source
		Initial Conc. mg/L	Final Conc. mg/L	
Aluminum	Al	500	1.0	TACA-1
Antimony	Sb	100	0.05	TACA-2
Arsenic	As	100	0.2	TACA-1
Barium	Ba	100	0.2	TACA-1
Beryllium	Be	100	0.2	TACA-1
Boron	B	100	1.0	TACA-1
Cadmium	Cd	100	0.2	TACA-1
Calcium	Ca	500	1.0	TACA-1
Chromium	Cr	100	0.2	TACA-1
Cobalt	Co	100	0.2	TACA-1
Copper	Cu	100	0.2	TACA-1
Iron	Fe	500	1.0	TACA-1
Lead	Pb	100	0.2	TACA-1
Lithium	Li	100	0.2	TACA-1
Magnesium	Mg	500	1.0	TACA-1
Manganese	Mn	100	0.2	TACA-1
Molybdenum	Mo	100	0.2	TACA-2
Nickel	Ni	100	0.2	TACA-1
Phosphorous	P	500	1.0	TACA-1
Potassium	K	500	1.0	TACA-1
Selenium	Se	100	0.2	TACA-1
Silver	Ag	25	0 .05	TACA-1
Sodium	Na	500	1.0	TACA-1
Strontium	Sr	100	0.2	TACA-1
Thallium	Tl	25	0.05	TACA-1
Tin	Sn	100	0.2	TACA-2
Titanium	Ti	100	0.2	TACA-2
Uranium	U	100	0.2	TACA-1
Vanadium	V	100	0.2	TACA-1
Zinc	Zn	100	0.2	TACA-1

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

Appendix I

CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Auto sampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Title: Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure [Methods 1311 & 1312]

Approvals (Signature/Date):

Robert Hrabak, Technical Manager — Date: 5/8/18	Joe Schairer, Health & Safety Manager / Coordinator — Date: 5/8/18
Lisa Stafford, Quality Assurance Manager — Date: 5/7/2018	Crystal Pollock, Laboratory Director — Date: 5.8.18

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1. SCOPE AND APPLICATION

1.1. This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW846 Method 1311, to waste materials. The Toxicity Characteristic (TC) of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a waste. The TC rule utilizes the TCLP method to generate the leachate under controlled conditions which were designed to simulate leaching through a landfill. The specific list of TC analytes and regulatory limits may be found in Appendix A.

1.2. This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in appendix A. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP they are distinguished within this SOP.

1.3. The procedure is applicable to liquid, solid, and multiphase wastes.

1.4. If a total analysis of the waste demonstrates that individual analytes are not present, or that they are present at low concentrations, the procedure may not need to be run. If the total analysis results indicate that TCLP may not be required, the decision to cease TCLP analysis should be remanded to the client.

1.5. The department manager, project manager and client should be notified when leachates with high analyte concentrations are encountered.

1.6. Volatile organic analysis of the leachate obtained using a bottle extraction, normally used for extractable organics and metals, can be used to demonstrate that a waste is hazardous, but only the Zero Headspace Extraction (ZHE) option can be used to demonstrate that the concentration of volatile organic compounds is below regulatory limits. ZHE is not performed by TestAmerica Sacramento and is not included in this SOP.

2. SUMMARY OF METHOD

2.1. For liquid wastes that contain less than 0.5% dry solid material, the waste, after filtration through 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP leachate.

2.2. For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is extracted with an

amount of extraction fluid equal to 20 times the weight of the solid phase. For TCLP, the type of extraction fluid employed for extraction of non-volatile analytes is a function of the alkalinity of the solid phase of the waste. For SPLP, the extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil.

2.3. If compatible (i.e., multiple phases will not form when combined), the initial liquid phase of the waste is added to the liquid leachate and these are prepared and analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Oily wastes may present unusual filtration and drying problems. As recommended by EPA (see Figure 2), oily wastes will be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed. This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter.

4.2. Wastes containing free organic liquids (i.e., those with separable non-aqueous liquid phases) will be assumed to be 100% liquid and totals analysis will be performed to determine if the waste exceeds TCLP limits.

4.3. Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks as described in the determinative SOPs.

4.4. Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.

4.5. Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for metals.

4.6. The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled. The temperature must be maintained at 23 ± 2°C during extraction.

4.7. Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Rotary agitation apparatus is used in this procedure. Certain types of samples may break the glass jars used. For these samples extra caution may be necessary. This might include plastic or polyethylene overwraps of the glass jars. Use of either Teflon or high density polyethylene jars (depending on the type of extraction being performed) will also substantially reduce the possibility of pressure cracking or breaking the bottle.

5.1.2. During sample rotation, pressure may build up inside the bottle. Periodic venting may be necessary to relieve this pressure.

5.1.3. Due to the potential for ignition and/or flammability, do not attempt to dry non-aqueous liquid samples in an oven.

5.1.4. Preparation of sodium hydroxide solutions produces considerable amounts of heat. Use plastic containers to mix this solution if possible. If glass containers are used, they must be free of any cracks or irregularities.

5.1.5. The use of vacuum systems during the filtering process presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced. Staff members performing vacuum operations must read the appropriate Section of the Corporate Safety

Manual before starting work.

5.1.6. The TCLP leaching process uses acidic fluids. Samples that contain cyanide may release hydrogen cyanide gas during the leaching process. Leaching containers must only be opened in a fume hood in order to safely disperse any toxic vapors that have formed.

5.1.7. All glassware must be inspected before use. Chipped, cracked or broken glassware must be removed from service immediately. This glassware must either be sent out for repair or discarded.

5.1.8. Acid washing of glassware is classified as a high-risk activity. A face shield must be worn over safety glasses or safety goggles during this process.

5.1.9. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide satisfactory protection.

5.1.10. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.11. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials Section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Extraction vessels

6.1.1. For metals - either borosilicate glass jars (2.2 L, with Teflon lid inserts) or 2.2 L high density polyethylene (Nalgene or equivalent) bottles may be used.

6.1.2. For non-volatile organics - borosilicate glass or Teflon bottles may be used.

6.2. Borosilicate glass fiber filters, 0.6 - 0.8 µm (Whatman GF/F 12.5 cm, 0.7 µm or equivalent). When analyzing for metals, wash the filters with 1 N nitric acid and de-ionized water prior to use. Glass fiber filters are fragile and should be handled with care.

6.3. Rotary agitation apparatus, multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see figure 1). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.

6.4. Top loading balance, capable of 0 - 4000 ± 0.01g (all measurements are to be within ± 0.1 grams).

6.5. pH meter and probe capable of reading to the nearest 0.01 unit, and with automatic temperature compensation (accuracy measurement to be within ± 0.05 pH units)

6.6. Magnetic stirrer/hotplate and stirring bars.

6.7. Glass jars, 2.2 L, with Teflon lid-inserts.

6.8. Nalgene plastic or Teflon bottles, 2.2 liters.

6.9. Vacuum pump.

6.10. Drying oven.

6.11. Buchner funnel and filter flask.

6.12. Miscellaneous laboratory glassware and equipment.

6.13. Digital hi-lo thermometer capable of monitoring room temperature.

7. REAGENTS AND STANDARDS

7.1. Reagent water for non-volatile constituents must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Hydrochloric acid, 1 N: Carefully add 83 mL concentrated reagent grade HCl to 800 mL reagent water in a 1 L beaker, cool and dilute to 1 liter with reagent water. Stir with magnetic stir bar or glass rod.

7.3. Nitric acid, 1 N: Carefully add 64 mL concentrated reagent grade HNO_3 to 800 mL of reagent water in a 1 L beaker, cool and dilute to 1 liter with reagent water. Stir with magnetic stir bar or glass rod.

Warning: Always add acid to water, never add water to acid.

7.4. Sodium hydroxide, 1 N: Carefully add 40 g reagent grade NaOH pellets to 800 mL reagent water, stir until the pellets are completely dissolved, cool and dilute to 1 liter with reagent water. CAUTION: Heat is generated during this process.

7.5. Acetic acid, glacial: concentrated, reagent grade liquid.

7.6. Miller Analytical TCLP Extraction Fluid Concentrate: Glacial Acetic Acid (Part 1 of 2); 40% Sodium Hydroxide Solution (Part 2 of 2).

7.7. pH calibration solutions: buffered to a pH of 2, 4, 7, and 10. Commercially available. Fresh buffer solution must be used each day of analysis. Refer to SOP WS-WC-0028 "Determination of Alkalinity, Conductivity, pH and Fluoride".

7.8. TCLP leaching fluids

7.8.1. General comments

7.8.1.1. The pH of both solutions listed below shall be checked prior to use and the pH probes must be calibrated prior to use with the accuracy measured within ± 0.05 pH units.

7.8.1.2. The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared.

7.8.1.3. Additional volumes of extraction fluids listed above may be prepared by multiplying the amounts of acetic acid and NaOH by the number of liters of extraction fluid required.

Note: *If using Environmental Express TCLP Extraction Fluid Concentrate, skip sections 7.8.2 and 7.8.3 and proceed to Section(s) 7.8.4 and 7.8.5.*

7.8.2. TCLP fluid #1: Carefully add 5.7 mL glacial acetic acid and 64.3 mL of 1 N NaOH to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water. Pour into 20L Nalgene® carboy and mix well. When correctly prepared, the pH of this solution is 4.93 ± 0.05. If

7.8.3. TCLP fluid #2: Carefully add 5.7 mL glacial acetic acid to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water. Pour into 20L Nalgene® carboy and mix well. When correctly prepared, the pH of this solution is 2.88 ± 0.05.

7.8.4. TCLP fluid #1 is prepared using 1 vial of concentrate (part #E1002) dilute to 2 liters with reagent water. To ensure a quantitative transfer, pour solution into a 20 L Nalgene Carboy and rinse the vial with the prepared 2L solution. When properly prepared, the pH of the solution is 4.93+/- 0.05.

7.8.5. TCLP fluid #2 is prepared using 1 vial of concentrate (part #E2002) dilute to 2 liters with reagent water. To ensure a quantitative transfer, pour solution into a

20 L Nalgene Carboy and rinse the vial with the prepared 2L solution. When properly prepared, the pH of the solution is 2.88+/- 0.05.

7.9. SPLP leaching fluids

7.9.1. Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated HNO_3 to 500 mL reagent water. Cap and shake to mix well.

Warning: Always add acid to water, never add water to acid.

7.9.2. Sulfuric/nitric acid (60/40 weight percent mixture) H_2SO_4/HNO_3. Measure about 50 ml nanopure water into a 100 ml snap cup. Cautiously mix 3 g of concentrated sulfuric acid (reagent grade) with 2 g of concentrated nitric acid (reagent grade) into the cup. Bring up to 100 ml, close cap and mildly shake.

Warning: Always add acid to water, never add water to acid.

7.9.3. SPLP solutions are unbuffered and exact pH may not be attained. The pH of TCLP and SPLP fluids shall be checked prior to use. If not within specifications, the fluid shall be discarded and fresh fluid prepared.

7.9.4. SPLP fluid #1: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 4.20 ± 0.05 This fluid is used for soils from sites that are east of the Mississippi River and for wastes and wastewaters.

7.9.5. SPLP fluid #2: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05. This fluid is used for soils from sites that are west of the Mississippi River.

7.9.6. SPLP fluid #3: This fluid is reagent water and is used for leaching of volatiles. Additionally, any cyanide-containing waste or soil is leached with fluid #3 because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples being analyzed for non-volatile organic compounds should be collected, tumbled, and stored in glass containers with Teflon lid liners. Samples being analyzed for perfluorinated compounds (PFAS) should be collected, tumbled and stored in polyethylene containers. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation.

8.2. Samples being analyzed for metals can be collected in either glass or polyethylene containers.

8.3. Samples should be refrigerated to 4 ± 2 º C unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

8.4. The minimum TCLP sample collection size is determined by the physical state or states of the waste and the analytes of concern. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing. For aqueous samples containing between 0.5 and 10% solids, several kilograms of sample are required to complete the analyses. The general minimal requirements when the samples are 100% solids is 1 - 32 oz jar. Low-density sample materials, such as rags or vegetation, will require larger volumes of sample. For liquid samples (less than 50% solids), minimum requirements are 2 - 32 oz jar. If matrix spike or duplicate control samples are requested, additional sample volume is required. If sufficient sample volumes were not received, analyses cannot be started and the client should be notified as soon as possible.

8.5. TCLP leachates should be prepared for analysis and analyzed as soon as possible following extraction. Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid to a small aliquot of the leachate, then the remaining portion of the leachate shall not be acidified and the leachate shall be analyzed as soon as possible. All other leachates should be stored under refrigeration (4 ± 2 °C) until analyzed.

8.6. Samples are subject to appropriate treatment within the following time periods:

Table 1 - Holding Times (days)

Parameter	Collection to TCLP Extraction	TCLP Extraction to Preparative Extraction	Preparative Extraction to Determinative Analysis	Total Elapsed Time
Semi-volatiles:	14	7	40	61
Mercury:	28	N/A	28	56
Other Metals:	180	N/A	180	360

Note: *The initial holding time is measured from date of collection to date TCLP extraction started. (This should be the TCLP extraction date in TALS.) Semi-volatile method prep holding time is measured from the day TCLP extraction is complete to the start of preparative extraction. Subsequent analysis holding times are measured from the date extraction (TCLP or method prep) starts. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit. The Total Elapsed Time is to be used as guidance. If preps are initiated at the last possible moment of a holding time, the elapsed times may be exceeded.*

9. QUALITY CONTROL

9.1. Quality control batch (QC Batch) - WS-PQA-003 defines a QC Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same procedures, reagents and standards within the same time period. The same lot of reagents must be used within a batch. A minimum of one TCLP extraction blank (Method Blank), one Matrix Spike (MS), and one Matrix Spike Duplicate (MSD) will be prepared with each TCLP leachate batch. A leachate LCS is not required by the method.

9.2. Batching samples - Groups of samples of different waste types (e.g., wastewater treatment sludge and contaminated soil, etc.) must be batched separately. Different method blanks and matrix spike sample should be prepared with each waste type (not to exceed 20 samples).

9.3. TCLP extraction blanks - A minimum of one blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of samples extracted in a particular vessel type. The blanks are generated in the same way as the samples (i.e., blanks will be tumbled and filtered with the samples).

9.4. Matrix spike/matrix spike duplicate (MS/MSD) - Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. An MS/MSD pair is required with each batch of 20 or fewer samples. Matrix spike solutions are to be added after filtration of the TCLP leachate. Spike solutions are not to be added prior to the TCLP leaching. For metals, matrix spike solutions are to be added before preservation with nitric acid. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible.

9.4.1. The use of internal calibration or alternate methods may be needed when the recoveries of matrix spike analytes are below expected performance (see Section 9.6.2).

9.4.2. Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.5. Corrective actions

9.5.1. Consult the TestAmerica Sacramento QC Program and individual analysis SOPs for corrective action for blanks (see WS-PQA-003, Quality Control Program).

9.5.2. Method of Standard Additions (MSA) shall be used for metals if all of the following conditions are met:

9.5.2.1. Recovery of the analyte in the matrix spike is not at least 50%,

9.5.2.2. The concentration of the analyte does not exceed the regulatory level.

9.5.2.3. The concentration of the analyte measured in the sample is within 20% of the appropriate regulatory level.

9.5.3. If the matrix spike recovery is 5% or less due to dilution or matrix interference, contact the project manager and client for guidance. The client should also be contacted prior to initiation of any MSA steps. Refer to the individual analysis SOPs for details on how to perform MSA analysis.

10. CALIBRATION

10.1. Calibrate the pH meter with fresh buffer solutions each day of use in accordance with the pH SOP WS-WC-0044. The accuracy of the pH meter is to be within ± 0.05 pH units for the ICV measurement.

10.2. Calibrate the balance prior to use with verified weights that bracket the sample aliquot.

10.3. The Hi/Lo thermometer used to monitor the room temperature shall be calibrated quarterly.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Preliminary sample evaluation (refer to flow chart #1, Appendix D)

11.2.1. Preliminary TCLP evaluations (percent solids, particle size, selection of extraction fluid, and fluid/leachate compatibility) are required. This aliquot may also undergo the actual TCLP or SPLP extraction for non-volatiles ONLY IF it has NOT been oven dried. If the solid portion is oven dried, a separate aliquot must be used for the actual leaching procedure.

11.2.2. Consult the holding times for the appropriate tests (Section 8.6) and prioritize extractions such that holding times are not exceeded.

11.2.3. Determine the total volume of TCLP leachate (solid phase leachate + liquid filtrate) that needs to be generated for analysis according to the following:

Table 2 - Minimum Required Leachate Volume

Analysis	Minimum (mL)	Recommended (mL)	MS/MSD (mL)
Semi-volatiles (8270)	200	400	800 (600 min.)
Pesticides	200	400	800 (600 min.)
Additional Organic	200	400	800 (600 min.)
Metals	150	150	150
PFAS	250	500	800 (800 min.)

11.2.3.1. SPLP - similar volumes are required for metals. If semivolatiles or pesticides are required, a full 1 L volume must be prepared for each test requested.

11.2.3.2. For TCLP and SPLP samples used for matrix spike and matrix spike duplicate analysis, three times the listed volumes are required.

11.2.4. Sample description (enter data on worksheet 1)

11.2.4.1. Record the number of phases observed in the sample. It is common that when more than one container of multi-phasic materials is received from the field, each container will show different amounts of each phase.

11.2.4.1.1. If the sample has multiple phases and is received in more than 1 bottle then the contents of each bottle should be combined in a single larger container prior to processing the sample further. If this is not possible, then the alternate procedure described in the following Section should be used.

11.2.4.1.2. Properly record the relative amounts of each phase by measuring the depth of the layers in each container after the contents have been allowed to settle. Determine the combined volume of each phase for all containers. Then mark the phase composition on a single container, mix thoroughly to obtain a representative subsample, and accurately measure the phase composition according to the following procedure. The two sets of values (combined

volumes per phase and phase composition for one container) are used to determine the correct volume/mass adjustments on the final result.

11.2.4.2. Solid - record the visible presence of a solid material heavier than water. If the sample contains more than one solid phase (e.g., wood and sediment mixed with water), attach additional documentation to worksheet 1.

11.2.4.3. Liquid - record the number of liquid phases observed in the sample according to apparent density. It may be impossible to distinguish apparent density if only one liquid phase is observed and there is no indication on the COC form. If this is the case, record it as aqueous material and let the subsequent analytical record show if the liquid is organic.

11.2.5. Percent solid phase (enter data on worksheet 1)

11.2.5.1. Determine type of filtration apparatus needed

11.2.5.1.1. If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., it is 100% solid), then proceed to ection 11.2.6 (particle-size reduction).

11.2.5.1.2. If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (<10%) or a highly granular, liquid containing waste vacuum filtration may be used.

11.2.5.1.3. If the sample is viscous (sludge or has high solids content), use pressure filtration.

11.2.5.2. Weight of filter - Measure and record this value before loading the filter into the filter holder (worksheet 1).

11.2.5.3. Weight of subsample and filtrate for percent solids measurement

11.2.5.3.1. Assemble the filtration apparatus (use blunt forceps to handle the 0.6 to 0.8 μm filter membrane).

11.2.5.3.2. Homogenize the waste, transfer a minimum of a 100 g subsample to the weighing vessel. Measure and record the gross weight (worksheet 1).

11.2.5.3.3. Measure and record the tare weight of the filtration vessel (worksheet 1).

11.2.5.3.4. Transfer the sample to the filtration device attempting to spread the waste sample evenly over the surface of the filter. Measure and record the tare weight of the empty weighing vessel and any residual sample (worksheet 1).

11.2.5.3.5. Calculate and record the net weight of sample used for testing (worksheet 1).

11.2.5.4. Filtration for percent solids

11.2.5.4.1. Slowly apply gentle pressure or vacuum of 10 psi to the filtration apparatus. Allow the sample to filter until no significant additional liquid has passed through the filter during a 2 minute period.

Warning: Use of a vacuum creates a risk of implosion when using glass vessels. Thoroughly inspect all glass vessels prior to each use. Do not use any glass containers that are scratched, chipped, cracked or scraped. Send out for repair or discard.

11.2.5.4.2. Repeat previous step by increasing the pressure in 10 psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a 2 minute period. Do not exceed 50 PSI due to potential filter rupture.

***Note**: Some samples will contain liquid material that can not be filtered (e.g., oil). Do not attempt to filter the sample again by exchanging filters. Viscous oils or any wastes which do not pass through the filter are classified as a solid.*

11.2.5.4.3. Remove the filtrate collection vessel, weigh and record the gross weight (worksheet 1).

11.2.5.4.4. Calculate and record the net weight of filtrate (worksheet 1). This result will be used in the percent solids calculation.

11.2.5.4.5. Pour the filtrate into a graduated cylinder. Measure and record the volume of the aqueous phase (worksheet 1). Measure and record the volume of any organic phase (worksheet 1). If more than one organic phase is observed, enter “see below” and provide a description at the bottom of worksheet 1. These results will be used in the final sample calculations.

11.2.5.4.6. Retain the filtrate for use in Section 11.2.8 (determination of filtrate/extraction fluid compatibility), and for possible recombination with the filtrate obtained in Section 11.3.

11.2.5.5. Percent of wet solids

11.2.5.5.1. Calculate the total weight of wet solids using equation on worksheet 1 and record the result.

11.2.5.5.2. Calculate the weight percent of wet solids using equation on worksheet 1 and record the result.

11.2.5.5.3. If the percent wet solids result is ≥ 0.5% and < 5.0%, and it is noticed that a small amount of the aqueous filtrate is entrained in the wetting of the filter, proceed to Section 11.2.5.7 to complete the percent solids measurement on a dry-weight basis.

Note: *If obviously oily (non-aqueous) material is entrained on the filter, do not dry the filter; proceed to Section 11.2.6 (particle-size reduction).*

11.2.5.5.4. If the percent wet solids result is greater than 5.0%, proceed to Section 11.2.6 (particle-size reduction).

11.2.5.5.5. If the percent wet solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is equivalent to the final leachate.

11.2.5.6. Weight percent of dry solids (Worksheet 2). Skip this step for oily samples.

Note*: These steps are required only if it is noticed that a small amount of the filtrate is entrained in wetting of the filter <u>and</u> the percent wet solids content is ≥ 0.5% and < 5.0 %.*

11.2.5.6.1. Remove the filter with the wet solids from the filtration apparatus.

11.2.5.6.2. Dry the filter and solid phase at 100 ± 20 ° C.

11.2.5.6.3. Remove the filter from the oven and allow to cool in a desiccator.

11.2.5.6.4. Weigh and record the gross dry weight (worksheet 1).

11.2.5.6.5. Repeat the drying step. Weigh and record the second gross dry weight (worksheet 1). If the two weights do

not agree within 1%, perform additional drying and weighing until successive weighings agree within 1%.

11.2.5.6.6. Calculate the weight percent of dry solids using equation on worksheet 1 and record the result.

11.2.5.6.7. If the dry solids result is ≥ 0.5% and the sample will be extracted for non-volatile constituents, proceed to Section 11.2.6 (particle size reduction) using a fresh wet portion of waste.

11.2.5.6.8. If the percent solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is the TCLP leachate. Proceed to Section 11.2.8 (determination of filtrate/leachate compatibility) to determine whether or not the material is a non-aqueous, immiscible liquid.

11.2.6. Particle-size reduction for fluid selection (enter data on worksheet 1)

11.2.6.1. The subsample used for fluid selection must consist of particles less than 1 mm in diameter (versus the less than 1 cm requirement for the material used for the actual extraction). The method requires a smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full extraction. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.

11.2.6.2. Surface area exclusion - size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram (for materials such as cloth or paper). Enter “N” for no particle size reduction performed on worksheet 1.

11.2.6.3. If the sample contains particles greater than 1 mm in diameter, crush, cut, or grind the solids to the required size. E. Enter “Y” for particle size reduction performed on worksheet 1

11.2.6.4. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick).

11.2.7. Determination of appropriate extraction fluid (worksheet 1)

11.2.7.1. If the solid content is greater than or equal to 0.5%, and if the sample is being analyzed for metals or nonvolatile organic compounds, the type of leaching solution must be determined.

11.2.7.2. Follow times, temperature, and particle size specified in this Section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.

11.2.7.3. For SPLP, refer to Section 7.8 for fluid selection. Matrix type must be specified by the client. Check special instructions or see the project manager, then put a check mark by the fluid type selected (worksheet 1).

11.2.7.4. The TCLP leaching fluid for all volatiles is fluid #1.

11.2.7.5. For TCLP leach fluid determination for non-volatile analytes, continue with the following steps.

11.2.7.6. Calibrate the pH meter with fresh buffer solution in accordance with the pH SOP (see WS-WC-044, EPA Method 9045B pH soil).

11.2.7.7. Weigh out a 5.0 ± 0.1 g subsample (less than 1 mm particle size) of the solid phase into a 250-mL beaker. If 5.0 grams not used, enter the actual weight.

11.2.7.8. Add 96.5 ± 1.0 mL of reagent water, cover with a watchglass, and stir for 5 minutes on a stirrer or shaker. If a different volume used, enter the actual volume on worksheet 1.

11.2.7.9. Measure and record the sample pH (worksheet 1).

***Note**: To avoid damaging the pH probe when organic liquid is present, use narrow range pH indicator paper.*

11.2.7.10. If the pH is less than or equal to 5.0, use fluid #1 and proceed to Section 11.2.8 (fluid compatibility).

11.2.7.11. If the fluid pH is greater than 5.0, add 3.5 mL 1 N HCl, cover with a watchglass. Slurry the sample briefly then heat at 50 º C for 10 minutes.

***Note**: The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.*

11.2.7.12. Cool to room temperature.

11.2.7.13. Measure and record the pH immediately after the sample has reached room temperature (worksheet 1).

11.2.7.13.1. If the pH is less than or equal to 5.0, use fluid #1.

11.2.7.13.2. If the pH is greater than 5.0, use fluid #2.

11.2.8. Determination of filtrate/extraction fluid compatibility for multiphase samples (skip this step for SPLP extractions)

11.2.8.1. Place 5 mL of the appropriate leaching fluid (determined in the previous step) into a 20-25 mL vial.

***Note**: Use fluid type # 1 if simply testing the filtrate for a sample with less than 0.5% solids.*

11.2.8.2. Add 5 mL of the initial filtrate, cap and shake.

11.2.8.3. If the phases are miscible, the initial filtrate and solid phase leachate will be physically recombined upon completion of the leachate generation.

11.2.8.4. If the phases are not miscible. The initial filtrate and the solid phase leachate will be prepared and analyzed separately and the results mathematically combined (see Section 12.1.4).

11.3. Bottle extraction procedure for non-volatile constituents: semi-volatiles, pesticides, herbicides, metals (refer to flow chart #2, Appendix D)

11.3.1. All masses should be recorded to the nearest 0.1 g.

11.3.2. The aliquot used in the preliminary evaluation may be used for this procedure ONLY if it was not oven dried. If the sample is 100% solid or if the preliminary aliquot was not oven dried, proceed directly to Section 11.2.6 (particle size reduction). If the preliminary evaluation aliquot was oven dried then, using a fresh aliquot of sample, continue as described in Sections 11.3.3 through 11.3.6.

11.3.3. Examine the sample and determine the type of filtration to employ per Section 11.2.5.1.

11.3.4. Repeat the steps outlined in Sections 11.2.5.3 through 11.2.5.5.3.

11.3.5. Determine and record the volume (mass) of the initial filtrate. Cover with aluminum foil and retain for use as defined in Section 11.3.18.

11.3.6. Determine and record the “solid” phase mass by subtracting the mass of the liquid filtrate from the mass of the subsample.

11.3.7. Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e., capable of passing through a 9.5 mm, 0.375 inch, standard sieve). Size reduction is not required if the sample surface area is greater than or equal to

3.1 cm^2 per gram. Record this on worksheet 1.

11.3.7.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding should be subcontracted out for particle size reduction. Contact PM. Note that size reduction to fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

11.3.8. Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 11.2.3.

11.3.9. Divide the total volume of solid phase leachate required by 20 to determine the mass of solid phase required for leaching. Round this mass UP to the nearest 5g.

11.3.10. Weigh the required mass of solid phase into an appropriate bottle (plastic for PFC and Metals only, glass for all other organics and metals (if Boron is not needed)) and **slowly** add 20 times its mass of appropriate leaching fluid as determined under Section 11.2.7 (If less than 100 g of sample is available, contact PM immediately). Record the weight of the sample aliquoted for the extraction on worksheet 1, and the amount of extraction fluid added.

11.3.11. ISM (Incremental Sampling Method) samples are pre-weighed at 5.0 g for pH preliminary and 100 g for the leach.

***Note**: If less than 100 g of sample is available, contact PM immediately.*

11.3.12. **WARNING: Ensure any effervescence has stopped** before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. The temperature of the room shall be 23 ± 2°C. Reset the digital Hi-Lo thermometer in the TCLP room prior to starting extraction. The room temperature and time shall be checked at both the start and end of the extraction and the time recorded on worksheet 1.

WARNING: As agitation continues, pressure may build up within the bottle for some types of wastes. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.

11.3.13. Remove the bottle and filter the sample using vacuum or pressure filtration by filtering through a new glass fiber filter as discussed in Sections 11.2.5.6. For final filtration of the TCLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters must be acid washed if metals are to be determined (see Section 6.3). The entire sample need not be filtered;

however, sufficient volume should be generated to support the required analyses. Record the date and time the filtration is completed on worksheet 1.

11.3.14. If the waste contained no initial filtrate, this solution from 11.3.12 is defined as the TCLP leachate.

11.3.15. If the waste did yield an initial filtrate, consult the worksheet for initial filtrate/leachate compatibility. If they are compatible, they are to be combined in the correct proportions and mixed well. This combined solution is defined as the TCLP leachate.

11.3.16. If the individual phases are NOT compatible, they are to be prepared and analyzed separately and the results combined mathematically. See Section 12.1.4.

11.3.17. Measure and record the pH of the TCLP leachate on worksheet 1. (Do not attempt to measure the pH of oily samples as the probe may be rendered inoperable.)

11.3.18. Prepare subsamples for metals for MS/MSD quality control testing using the appropriate TCLP spiking solution (do not spike for organics). Refer to the appropriate determinative SOPs for further guidance on the spike components, levels and action criteria.

11.3.19. Immediately preserve the leachate as follows:

Metals	pH < 2 w/50% HNO_3 for non-oils (do not acidify oils)
All others	Refrigerate to 4 ± 2 ^{o}C

***Note**: Refer to Section 8.6 if precipitation occurs upon preservation.*

11.3.20. Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis with copies of the TCLP preparation worksheets.

12. CALCULATIONS/DATA REDUCTION

12.1. Calculations

12.1.1. Calculation of Percent Wet Solids:

$$\text{Percent Wet Solids} = 100 \left(\frac{\text{Mass, "soid" phase}}{\text{Mass, initial subsample}} \right)$$

12.1.2. Calculation of weight of extraction fluid to use:

$$Weight\ of\ Extraction\ Fluid = \frac{20\ X\ (\%\ wet\ solid)\ X\ (weight\ of\ waste\ to\ be\ extracted)}{100}$$

12.1.3. Calculation of volume of initial filtrate phase to recombine with solid phase leachate:

$$Volume\ of\ Filtrate\ Recombination = \left(\frac{Weight\ of\ solid\ leached}{Total\ weight\ of\ solids}\right)\left(\frac{Leachate\ recovered}{Fluid\ added}\right)(Volume\ of\ initial\ aqueous\ filtrate)$$

12.1.4. Mathematical recombination of analytical results:

$$Final\ Analyte\ Concentration = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

V_1 = total volume of the initial filtrate phase (L).
C_1 = analyte concentration in initial filtrate phase (mg/L).
V_2 = volume of the theoretical solid phase leachate (L).
C_2 = analyte concentration in solid phase leachate (mg/L).

12.2. Reporting requirement

12.2.1. Follow these reporting conventions for multi-phase samples:

12.2.1.1. If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.

12.2.1.2. If both phases are “ND,” Not Detected, the recombined result is “ND,” and the reporting limit is calculated from the reporting limit for each phase.

12.2.1.3. If one phase is “ND” and the other phase has a positive result, use the reporting limit for the “ND” phase and the positive value for the other phase to calculate the combined result. The combined reporting limit is based on the reporting limit for both phases. If the combined result is less than the combined reporting limit, then supply a footnote to indicate that “a positive result was detected below the calculated detection limit.”

12.2.2. Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

12.2.3. For limits and significant figures, consult the appropriate analytical methods.

12.2.4. Anomalies - all anomalies observed during the leach procedure must be noted on the worksheet and in Clouseau (see WS-QA-0023). Some examples of such anomalies are:

12.2.4.1. Sample was monolithic - subsample was obtained by crushing, cutting, grinding, sawing, etc.

12.2.4.2. Insufficient sample - less than the required 100 g minimum was available.

12.2.4.3. Multiple phases - "X" phases were present.

12.2.4.4. Sample was oil - single phase.

12.2.4.5. Sample contained liquid which did not filter under test conditions.

12.3. Review requirement

12.3.1. Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation form was properly documented and routed.

12.3.2. If Total analysis results are available, those results may be compared with the TCLP analysis results according to the following:

$$Total \geq 20 \times TCLP$$

***Note**: Assumes the sample is 100% Solids.*

12.3.3. Total constituent analysis results can be used to demonstrate the TCLP protocol is unnecessary. In performing a TCLP analysis, there is a 20:1 dilution of the original sample with the leaching solution. Thus, if the "total constituent" result is less than 20 times the TC level, it is impossible for the leachate to "fail" and the TCLP does not need to be performed. For example, the TC level for lead is 5.0 mg/L (ppm). Therefore, if a sample of lead-contaminated soil contains less than 100 ppm total lead, a TCLP test need not be run for lead.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for

determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department. MDLs are not performed on leachates.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the mid range of the calibration curve.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Unused acidic extraction fluid/leachate. Pour the fluid into a blue plastic acid waste drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the acid waste drum to the waste collection area for shipment.

15.2. Leached out solids. After pouring off any unused fluids, collect the leached solids into a contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut

and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Facility Specific SOPs

16.1.1. WS-PQA-003, Sacramento QC Program

16.1.2. WS-QA-0041, Calibration and Calibration Check of Balances

16.1.3. WS-QA-0023, NCMs and Corrective Action System

16.1.4. WS-WC-0044, pH of Soils and Wastes

16.2. Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update I.

16.3. Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, November 1992, SW-846 Proposed Update II.

16.4. Related Documents

16.4.1. Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990.

16.4.2. Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990.

16.4.3. Technical Background Document and Response to Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April 1989.

17. METHOD MODIFICATIONS

17.1. Modifications/Interpretations from Reference Methods

17.1.1. Section 11.2: Preliminary Evaluations. Section 7.1 of the source method states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction." Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., < 100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). Samples which have been subjected to the oven drying step may not

be used for TCLP extraction because solid phase degradation may result upon heating.

17.1.2. Section 11.2.5.: Percent Solids Determination. Section 7.1.2 of the source method indicates that "if the percent wet solids is ≥ 0.5% and it is noticed that a small amount of the filtrate is entrained in wetting of the filter" that the filter should be oven dried to determine percent dry solids ". Drying of oil or organic matrices can be hazardous and inappropriate. Additionally, it may be impossible to achieve a constant weight when performing this step. Due to safety concerns, if obviously oily or heavy organic matrices are entrained on the filter, the filter is not oven dried.

17.1.3. Section 11.2.8: Preliminary Determination of Filtrate/Extraction fluid Compatibility. Section 7.2.13 of the source method provides no guidance as to how to make this determination. As a result, the procedure herein was developed and incorporated into the Preliminary Determinations Section.

17.1.4. Section 9.1: TCLP Extraction Blanks. Section 8.1 of the source method states that a minimum of one blank for every 20 extractions "...that have been conducted in an extraction vessel." Sacramento has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e., Bottle or ZHE) per leach fluid used.

17.1.5. Section 11.2.7: Determination of Appropriate Extraction fluid. Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that fluid #1 must be used if the pH is less than or equal to 5.0.

17.1.6. Section 9.5: QA/QC - Matrix Spikes. Section 8.2 of the source method states, "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch." Further, Section 8.2.3 of the source method also states, "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist." The standard Sacramento QC Program is designed to address the performance monitoring of analytical methodology through the LCS program. A minimum of one MS and MSD will be prepared for each TCLP leachate batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results have immediate bearing only on the specific sample spiked and not all samples in the batch.

17.1.7. Section 8.2.2 of the source method states that "In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level." The method also states, "If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of

the analyte concentration but may not be less than five times the method detection limit". For several analytes, spiking at the regulatory level is inappropriate to the range of analysis afforded by the determinative methods. Due to the wide range in these levels, Sacramento spikes at the levels specified in the determinative SOPs.

18. ATTACHMENTS

18.1. Table 1 – Holding Time (days)

18.2. Table 2 – Minimum Required Leachate Volume

18.3. Table 3 - Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)

18.4. Attachment 4 – Instructions for Preparing TCLP Fluid #1

18.5. Attachment 5 – Instructions for Preparing TCLP Fluid #2

18.6. Figure 1 – Rotary Agitation Apparatus

18.7. Figure 2 - US Environmental Protection Agency Memorandum #35, Page 1

18.8. Example Worksheet 1 – Page 1

18.9. Example Worksheet 1 – Page 2

18.10. Example Worksheet 2 – Calculation of Weights of Percent Dry Solids

18.11. Flow Chart 1 – Preliminary Sample Evaluation (Section 11.2)

18.12. Flow Chart 2 – Bottle Extraction – Non Volatile Constituents (Section 11.3)

19. REVISION HISTORY

19.1. WS-IP-0004 Revision 3.7, Effective 05/09/2018

19.1.1. Removed Section 5.1.6, "Gas-pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized. Do not open equipment that has been pressurized until it has returned to ambient pressure."

19.1.2. Note after 7.8.1.3 revised to, "If using Environmental Express TCLP Extraction Fluid Concentrate, skip sections 7.8.2 and 7.8.3 and proceed to Section(s) 7.8.4 and 7.8.5."

19.1.3. Section 7.8.4 revised to, "TCLP fluid #1 is prepared using 1 vial of

concentrate (part #E1002) dilute to 2 liters with reagent water. To ensure a quantitative transfer, pour solution into a 20 L Nalgene Carboy and rinse the vial with the prepared 2L solution. When properly prepared, the pH of the solution is 4.93+/- 0.05."

19.1.4. Section 7.8.5 revised to, "TCLP fluid #2 is prepared using 1 vial of concentrate (part #E2002) dilute to 2 liters with reagent water. To ensure a quantitative transfer, pour solution into a 20 L Nalgene Carboy and rinse the vial with the prepared 2L solution. When properly prepared, the pH of the solution is 2.88+/- 0.05."

19.1.5. Section 8.1 revised to, "Samples being analyzed for non-volatile organic compounds should be collected, tumbled, and stored in glass containers with Teflon lid liners. Samples being analyzed for perfluorinated compound (PFAS) should be collected, tumbled and stored in polyethylene containers. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation."

19.1.6. Updated Table 2 values and added data for PFAS.

19.1.7. Added Section 11.3.11, "ISM (Incremental Sampling Method) samples are pre-weighed at 5.0 g for pH preliminary and 100 g for the leach."

19.1.8. Section 11.3.10 revised to, "Weigh the required mass of solid phase into an appropriate bottle (plastic for PFAS and Metals only, glass for all other organics and metals (if Boron is not needed)) and **slowly** add 20 times its mass of appropriate leaching fluid as determined under Section 11.2.7 (If less than 100 g of sample is available, contact PM immediately). Record the weight of the sample aliquoted for the extraction on worksheet 1, and the amount of extraction fluid added."

19.1.9. Added note after Section 11.3.11, "If less than 100 g of sample is available, contact PM immediately."

19.1.10. Throughout SOP, undated references to "QUANTIMS" to "TALS".

19.1.11. Updated Example #1, "Worksheet 1" to current version.

19.1.12. Removed revision history prior to 2014, it can be found in previous versions of this SOP.

19.1.13. Editorial changes.

19.2. WS-IP-0004 Revision 3.6, Effective 1/31/14

19.2.1. Added language in section 6.5 to state accuracy measurement of pH meter is to be within ± 0.05 pH units.

19.2.2. Revise section 7.8.1.1 to state the TCLP solutions shall be checked prior to use and that the accuracy measurement of the pH meter is to be within ± 0.05 pH units.

19.2.3. Revised the language in section 7.9.3 from “should’ to “shall”.

19.2.4. Added language in section 10.1 to calibrate the pH meter with fresh buffer solutions each day of use with the accuracy within ± 0.05 pH units for the ICV.

19.2.5. Added section 10.2 for calibration verification of the balance with verified weights each day of use.

19.2.6. Added section 10.3 for calibration verification of the Hi/Lo thermometer quarterly.

19.2.7. Editorial Changes

Table 3 - Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)

Contaminant	mg/L
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresols	200.0
m-Cresols	200.0
p-Cresols	200.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

Attachment 1 – Instructions for Preparing TCLP Fluid #1

Instructions for preparing TCLP Fluid #1

- Add 500mL reagent water to a 1 Liter graduated cylinder
- Carefully add 5.7mL glacial acetic acid
- Carefully add 64.3mL 1 N NaOH
- Dilute to final volume of 1 Liter with reagent water
- Pour into 20L Nalgene® carboy and mix well
- Check pH with a properly calibrated pH meter
- When correctly prepared, the pH of this solution is **4.93 ± 0.05 (4.88 – 4.98)**

Other volumes of TCLP Fluid #1 may be prepared following the table below.

Fluid #1

Final Volume, Liter	HOAc (Glacial Acetic Acid), mL	1N NaOH, mL	DI-Water, mL
1	5.7	64.3	930
1.5	8.7	96.5	1395
2	11.4	128.6	1860
2.5	14.3	160.7	2325
3	17	193	2790
3.5	20	225	3255
4	22.8	257.2	3720

pH of TCLP Fluid #1 is 4.93 ± 0.05 (4,88 – 4.98)

pH of TCLP Fluid #1 is to be checked prior to each use to ensure proper pH
If Fluid is not within specifications, discard and prepare fresh extraction fluid

Facility Distribution No. _____ **Distributed To:_____**

Attachment 2 – Instructions for Preparing TCLP Fluid #2

Instructions for preparing TCLP Fluid #2

- Add 500mL reagent water to a 1 Liter graduated cylinder
- Carefully add 5.7mL glacial acetic acid
- Dilute to final volume of 1 Liter with reagent water
- Pour into 20L Nalgene® carboy and mix well
- Check pH with a properly calibrated pH meter
- When correctly prepared, the pH of this solution is **2.88 ± 0.05 (2.83 – 2.93)**

Other volumes of TCLP Fluid #2 may be prepared following the table below.

Fluid #2

Final Volume, Liter	**HOAc (Glacial Acetic Acid), mL**	**DI-Water, mL**
1	5.7	994.3
1.5	8.7	1491.3
2	11.4	1988.6
2.5	14.3	2485.7
3	17	2983
3.5	20	3480
4	22.8	3977.2

pH of TCLP Fluid #2 is 2.88 ± 0.05 (2.83 – 2.93)

pH of TCLP Fluid #2 is to be checked prior to each use to ensure proper pH
If Fluid is not within specifications, discard and prepare fresh extraction fluid

Facility Distribution No. _____ **Distributed To:_____**

Figure 1 - Rotary Agitation Apparatus

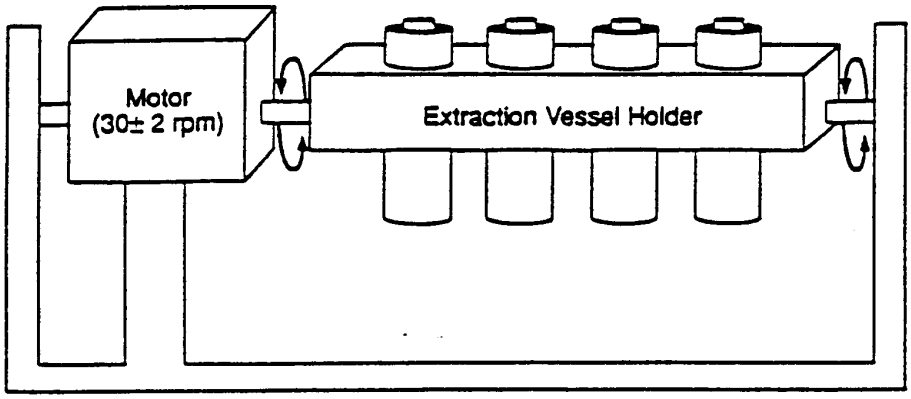

Figure 1. Rotary Agitation Apparatus

Figure 2 - US Environmental Protection Agency Memorandum #35, Page 1

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 35

DATE: June 12, 1992

SUBJECT: Notes on RCRA Methods and QA Activities

From: Gail Hansen, Chief
Methods Section (OS-331)

This memo addresses the following topics:

- o 1992 Symposium on Waste Testing and Quality Assurance
- o SW-846 Update
  - Final Rule for January 23, 1989 Proposed Rule
  - Notice, Proposed Rulemaking for the Second Update to the Third Edition
- o Chlorofluorocarbon 113 (CFC-113) Solvent Replacement Update
- o Environmental Monitoring Methods Index (EMMI)
- o Sampling Work Group Formation
- o MICE Update
- o Oily Waste Analysis
- o Electronic SW-846 Availability.

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Figure 2 - US Environmental Protection Agency Memorandum #35, Page 10

Oily Waste Analysis

One of the most frequently asked questions on the MICE Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

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Example Worksheet 1 – Page 1

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Balance ID:_____
Hot Plate ID:_____
Thermometer ID:_____

TestAmerica Sacramento
SPLP / TCLP Worksheet
(Circle Method)

Analyst:_____ Pre-Prep Batch:_____ Date: _____ Reviewed:_____
Room Temp: Start _____ End _____ Minimum_____ Maximum_____ Temp. Within **23°C ± 2?** Y / N; Date: _____
RT Thermometer ID:_____ *If room temperature exceeds requirement* (23± 2) **STOP**, *notify PM and QA immediately.*

A. SAMPLE DESCRIPTION	A	B	C	D	E	F	G	H	I	J	K
Number of Phases											
1. Solid											
2. Liquid											
B. PERCENT SOLID PHASE (WET/DRY) <SEE ATTACHED FORM(S)>											
C. EXTRACTION FLUID DETERMINATION											
1. Particle size reduction? Y/N											
2. Sample weight, check if 5.0 ± 0.1g											
3. Vol. of water, check if 96.5 ± 1.0mL											
4. Initial pH after 5 minutes mixing time											
5. If pH>5, check if 3.5mL 1N HCl added											
6. Check if heated for ten minutes at 50C											
7. Secondary pH(at room temperature)											
D. SELECTION OF EXTRACTION FLUID											
1. Check if pH C4 or C7 is <5, use Extraction fluid #1 4.93 ± 0.05 (pH of Fluid#1_____) Fluid Lot:_____ pH Meter ID:											
2. Check if pH C4 or C7 is >5, use Extraction fluid #2 2.88 ± 0.05 (pH of Fluid #2 _____) Fluid Lot: _____ pH Meter ID:											
3. SPLP: EAST: pH 4.2 ± 0.05 WEST: pH 5.0 ± 0.05 CN Soils: use DI Water Circle Fluid Used, pH _____ Fluid Lot: _____ pH Meter ID:											

A = _____ B= _____ C=_____
D = _____ E= _____ F=_____
G = _____ H= _____ I =_____
J = _____ K= _____

Example Worksheet 1 – Page 2

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Pre-Prep Batch:_____

TestAmerica Sacramento
Leachate Worksheet

BALANCE ID_____

A. SAMPLE DESCRIPTION	A	B	C	D	E	F	G	H	I	J	K
E. DETERMINATION OF SAMPLE SIZE											
1. Particle size reduction? Y/N											
2. Amount of dry solids (100g minimum)											
3. Amount of multi-phasic sample											
a. weight of material											
b. weight of filtrate											
c. Weight of solid material											
F. DETERMINATION OF AMOUNT OF EXTRACTION FLUID											
1. For dry solids (20x sample weight)											
2. For multi-phasic samples											
G. RECORD OF LEACHATE TEST (18 ± 2 HOURS)											
1. Extraction start date & time : _____											
2. Rotator: _____RPMs _____30±2											
3. Extraction stop date & time: _____											
4. Filtration start date & time: _____											
5. pH of final leachate											
6. Volume of filtrate											

1. For semi-volatiles, metals, and pesticides/herbicides, the theoretical amount of multi phasic waste necessary to yield a 100g sample is given by:
   Amount of multi-phasic material = 10,000/wt% wet solids
2. The amount of extraction fluid needed to extract the solid material from the filtered multi-phasic waste is given by:
   Amount of extraction fluid = 20(wt of material filtered – wt of filtrate)

NOTE: Use the min/max digital thermometer to record the room temperature in ºC. The minimum and maximum temperature on the digital thermometer must be reset prior to use. This is done by pressing the reset buttons for both on the back of the digital thermometer.

Example Worksheet 2

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica West Sacramento
Calculation of Weight of Percent Dry Solids

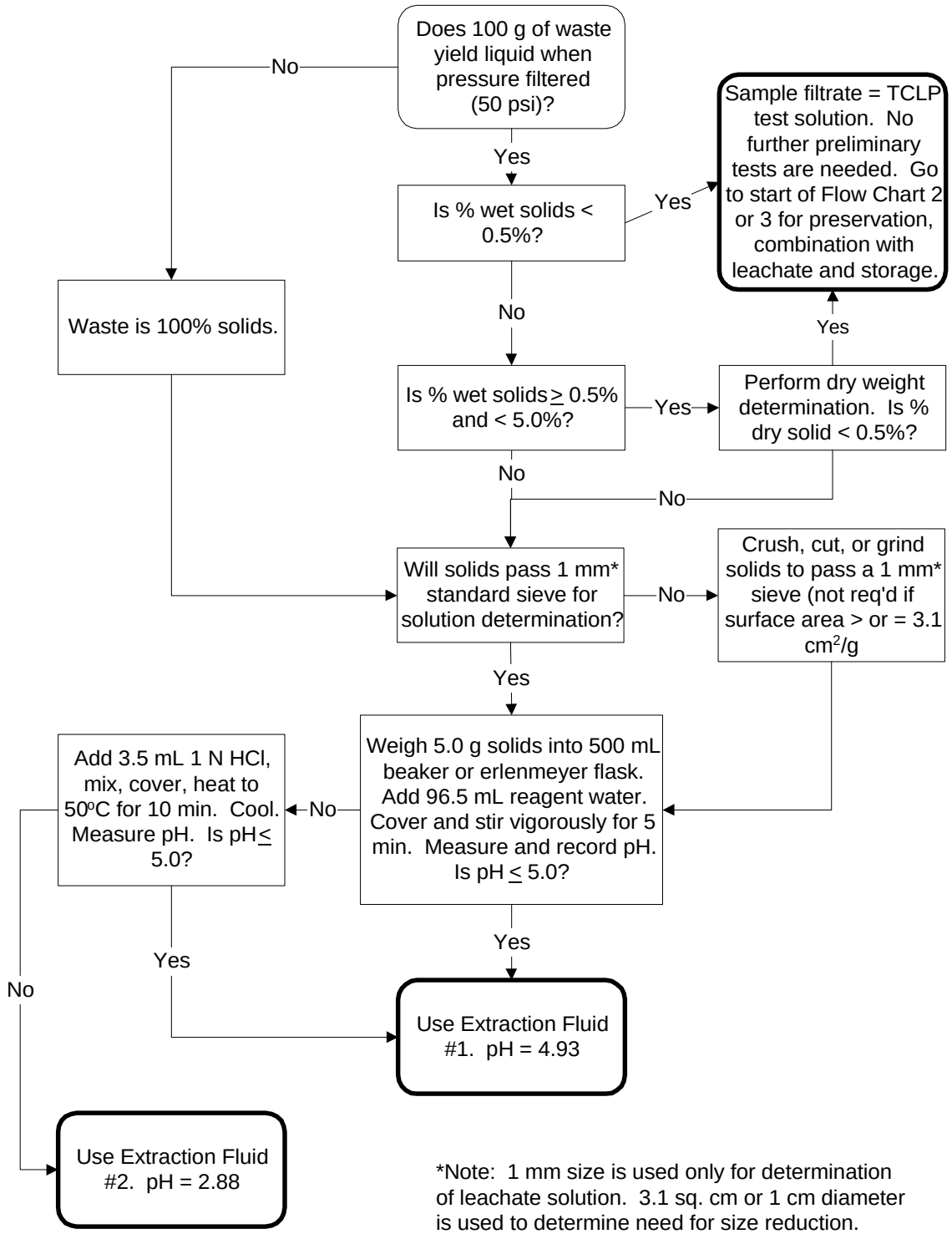
Analyst: _____ Reviewed: _____
Date: _____ Date: _____
Balance: _____ Pre-Prep Batch: _____

Samples are dried in an oven overnight, then equilibrated in a desiccator for 1 hour prior to weighing. Dry samples are weighed a second time after an additional 2 hours of drying followed by 1 hour of equilibration in a desiccator.

SAMPLE ID	A Filter Weight	B Subsample Weight	First			Second		
			C Wt of Filter + Dry Solid	D Wt of Dry Solid (C-A)	E % Dry Solids (D/B) * 100	F Wt of Filter + Dry Solid	G Wt of Dry Solid (F-A)	H % Dry Solids (G/B) * 100

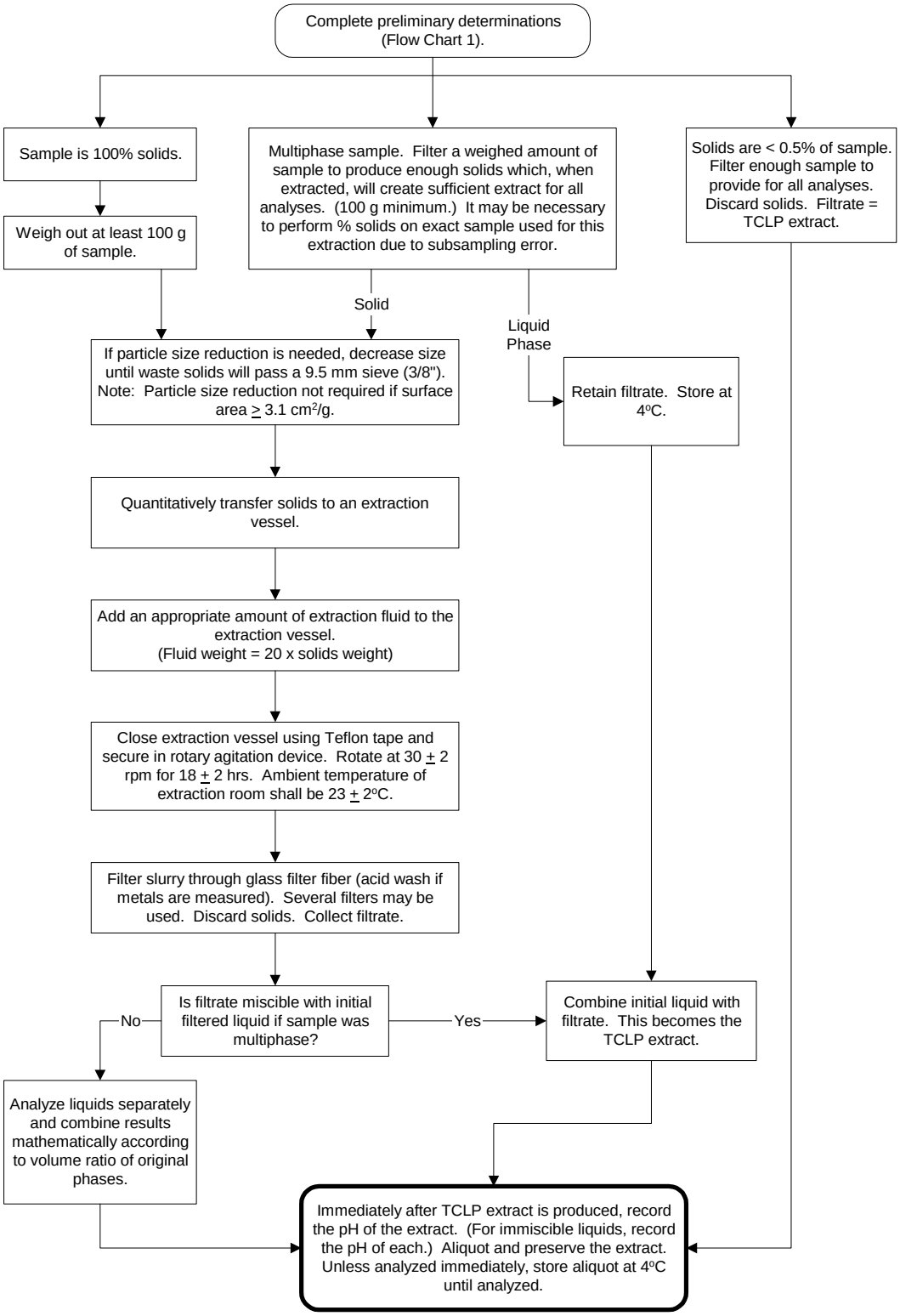
q:\forms\qa-522 tclp %dry solids.doc KS 12/10/07

Flow Chart 1. Preliminary Sample Evaluation (Section 11.2)



*Note: 1 mm size is used only for determination of leachate solution. 3.1 sq. cm or 1 cm diameter is used to determine need for size reduction.

Flow Chart 2. Bottle Extraction, Non-Volatile Constituents (Section 11.3)



Title: Determination of Polycyclic Aromatic Hydrocarbons (PAH) by GC/MS-SIM Internal Standard Technique [Method 8270C & 8270D]

Approvals (Signature/Date):

Victoria Nihart 6/19/18
Victoria Nihart Date
Department Manager

Joe Schairer 6/20/18
Joe Schairer Date
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1. SCOPE AND APPLICATION

1.1. This method is suitable for the analysis of PAHs in solid, aqueous, and oily matrices by Method 8270C SIM and 8270D SIM analysis. Refer to Table 1 for a list of individual PAHs determined by this method.

1.2. The standard reporting limit (SRL) of this method for determining an individual compound is approximately 5ug/kg (wet weight) for soil/sediment samples, 50ng/L for groundwater samples, and 500ug/kg or higher for oily matrices samples. Refer to TALS for specific SRLs. Reporting limits will be proportionately higher for samples that require dilution.

1.3. This method is restricted to use by, or under the supervision of, analysts experienced in the use of capillary column gas chromatography/mass spectrometry. Because of the toxicity of the materials known or believed to contain PAH, certain precautions must be taken to prevent exposure to the analyst or to others.

1.4. When undertaking projects for Department of Defense (DoD) and or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. The analytical method is gas chromatography combined with internal standard mass spectrometry. This entails the addition of extraction surrogates to all samples in known quantities, matrix-specific extraction of the sample with appropriate organic solvents, and analysis of the processed extract for PAHs using high-resolution capillary column gas chromatography coupled with low mass spectrometry (GC/MS) in selective ion monitoring mode (SIM). Analyte concentrations are calculated using the internal standard technique.

3. DEFINITIONS

3.1. Large Volume Injection (LVI) – An injection into a gas chromatograph that is larger than the typical 1 or 2µL injection used for hot vaporizing injectors.

3.2. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.3. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Interferences may be caused by contaminants in solvents, reagents, sorbents, glassware and other sample processing hardware that may lead to discrete artifacts and/or elevated backgrounds at the ions monitored. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

4.2. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1. Specific Safety Concerns or Requirements

5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile gloves must be used.

5.1.2. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.3. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.4. Ensure that all instrument exhaust vents and lines are properly connected to either a laboratory vent or an appropriate filter. Instruments may not be vented to the working environment.

5.2. Primary Material Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. See SOP WS-OP-0001 or WS-OP-0006 for details of sample preparation equipment and supplies.

6.2. GCMS Analytical System

6.2.1. Gas Chromatograph/Mass Spectrometer (GC/MS) – This method uses GC instrumentation manufactured by Agilent, Model 6890, or equivalent, and a Mass Spectrometer manufactured by Agilent, Model 5973 MS, or equivalent. The GC injection port must be designed for capillary columns. Splitless injection is recommended. See Table 8 for the recommended column for this analysis.

6.2.2. Mass Spectrometer - The mass spectrometer (MS) must be capable of operation in the Selective Ion Monitoring mode at a resolving power of 1 amu. Electron impact ionization must be used. The mass spectrometer must be capable of monitoring all of the ions listed in each of the four SIM descriptors (Table 9) with a total cycle time of 1 second or less.

6.2.3. GC/MS Interface

Any gas chromatograph to mass spectrometer interface may be used as long as it gives acceptable calibration response for each analyte of interest at the desired concentration and achieves the required tuning performance criteria.

To achieve maximum sensitivity, the exit end of the capillary column should be placed in the mass spectrometer ion source without being exposed to the ionizing electron beam.

6.3. Data Acquisition System

A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and plot a Selected Ion Current Profile (SICP), a plot of the abundances of the selected ions versus time or scan number. Software must also be available to integrate, in any SICP, the abundance between specified time or scan-number limits. The data system must provide hard copies of individual ion chromatogram's for selected gas chromatographic time intervals..

6.3.1. Acquisition software: ChemStation Software version D.

6.3.1.1. The system provides the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

6.3.2. Data Processing Software: Chrom version. 2.1

6.3.2.1. The software can search any GC/MS data file for ions of a specific mass and can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). .

6.3.2.2. The software allows integrating the abundances in any EICP between specified time or scan-number limits.

6.3.2.3. The most recent version of the NIST Mass Spectral Library is recommended.

7. REAGENTS AND STANDARDS

7.1. See WS-OP-0001 or WS-OP-0006 for details of reagents related to sample preparation.

7.2. Stock Standard Solution

Standard solutions can be prepared from pure standard materials or purchased as certified solutions (see analyte lists in Tables 2-4). Stock standard solutions must be replaced after one year.

7.2.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampule is opened, if the standard is

supplied in a sealed ampule. If a vendor-supplied date has an earlier expiration than the date from preparation, the earlier date is used.

7.2.2. Neat Standard Materials - Neat materials expire following a period of 3 years from the date of receipt, or sooner if problems such as degradation occur.

7.2.3. Working Standard Solutions - Working standards expire following a period of 1 year from the preparation date or the expiration date of the stock solution, whichever is earlier. The solution may be replaced sooner if evidence of degradation is observed.

7.3. Preparation of Stock Solutions

7.3.1. Internal Standards - Prepare or purchase a stock solution in $MeCl_2$ of the five internal standards listed in Table 3 at concentrations of 2000 ug/mL.

7.3.2. Store stock standard solutions in Teflon-sealed screw-cap bottles at 0-6°C and protect from light. Stock standard solutions must be checked frequently for signs of degradation and evaporation, especially just before using them to prepare calibration standard solutions or spiking solutions.

7.3.2.1. Replace stock standard solutions every 12 months or more frequently if comparison with quality control check samples indicates a problem.

7.3.3. Calibration Standards

Prepare calibration working standard solutions by combining appropriate volumes of individual or mixed calibration standards with extraction surrogate stocks, internal standard stocks and diluting to volume with methylene chloride to obtain the solution concentrations given in Table 4. The suggested range is 50 ng/mL to 5000 ng/mL and 25 ng/mL to 1000 ng/mL for LVI . Calibration solutions must be replaced after one year from the date of preparation, or sooner if empirical data indicates the standard has undergone concentration or degradation. If one of the parent solutions has an earlier expiration date, that date must be used.

7.3.3.1. Prepare an Initial Calibration Verification (ICV) or Second Source Calibration Standard (SSCS), when available, to confirm the calibration curve. This standard will be analyzed after the initial calibration.

7.3.3.1.1. All standards must be stored at 0-6°C and must be freshly prepared if the calibration verification, internal standard, or native analyte standard indicates a

problem.

7.3.4. Internal Standard Spiking Solution

Use an appropriate volume of stock solution to prepare an internal standard spiking solution in methylene chloride with the concentrations shown in Table 3. Store at 6°C or less. Spike 10 µL of the internal standard spiking solution to the final sample extract just prior to analysis to achieve a final extraction of 0.5 µg/mL.

7.3.5. Calibration Verification Standard

7.3.5.1. The calibration verification standard shall be used for column performance checks to verify peak separation, and for daily calibration checks. Solution #3 or #4 from Table 4 shall be the calibration verification standard.

7.4. GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL o-decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 50µg/mL. This solution is valid for one year.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Refer to SOP WS-OP-0001 for sample preservation and storage.

8.2. Extracts are stored at < -10°C until at least 30 days following invoicing.

8.3. Extracts must be analyzed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Control Limits

The laboratory will establish historically derived recovery limits for laboratory control samples (LCS), matrix spikes (MS), and surrogates. QAPP or project specific limits may supersede, if mutually agreed upon.

9.1.1. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into TALS (when available) or other database so that accurate historical control charts can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.1.2. Refer to the QC Program document (WS-PQA-003) for further details of control limits.

9.2. Internal Standards

Internal standards are components similar in nature to the analytes of interest. These are added to every sample (including QC aliquots) and standard analyzed. The purpose is to enable calculations based on internal standard methodology. Internal standard recoveries are monitored to verify that instrument performance is acceptable. Criteria for standards are delineated in Section 10. If the internal standard data indicate instrument failure, the samples may require reanalysis. If not, the impact on sample data is evaluated and the data is flagged appropriately.

9.2.1. The internal standard (IS) responses in all QC and field samples are compared to the mid-point standard of the initial calibration. Alternatively, the IS response in the samples may be compared to the daily CCV, on a per batch basis, to meet client or program requirements.

9.2.2. Any samples that do not meet the same IS criteria set forth for the CCV (Section 10.4) must be evaluated for validity. If the change in internal standard response is a matrix effect confined to an individual sample, reanalysis may not be necessary, but should be verified by the department manager or client. If the change in internal standard response is due to instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

9.2.3. Any IS responses outside the criteria should be noted in the case narrative.

9.3. Extraction Surrogate Spike

9.3.1. Recoveries for each of the extraction surrogates must be within the historically derived, or QAPP specific recovery limit. If the extracts are diluted > 5x the surrogates will be diluted out, and not reportable. The data flag of NC-SRD will be used to indicate this.

9.3.2. If the extraction surrogate standard recoveries are outside of the acceptable limits, the cause of the failure should be investigated. Otherwise the sample is re-extracted if sufficient sample volume is available; subject to client approval, re-extraction of a smaller sample aliquot may be appropriate to mitigate low surrogate recovery caused by matrix interferences.

9.3.3. Recommended Corrective Actions for surrogate failures in MB, LCS, or LCSD (batch QC)

9.3.3.1. Check all calculations for errors.

9.3.3.2. Verify satisfactory instrument performance.

9.3.3.3. Recalculate the data and/or reanalyze the extracts if either of the

above checks reveals a problem.

9.3.3.4. If the problem persists the QC and associated samples may require re-extraction.

9.3.3.5. Review the analytical procedures with the performing laboratory personnel.

9.3.3.6. Re-extract the samples and associated QC if necessary (Example- low surrogate recovery in MB)

9.3.3.7. Document the failure in an anomaly, and any corrective action in the narrative.

9.3.4. Corrective actions for surrogate failures in field samples or MS/MSD:

9.3.4.1. Check all calculations for error.

9.3.4.2. Ensure that instrument performance is acceptable.

9.3.4.3. Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.

9.3.4.4. Evaluate objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interferences demonstrated by prior analyses)

9.3.4.5. Re-extract if necessary.

9.3.4.6. Document the failure and note it on the final report.

9.4. Method Blanks

One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water, and for solid samples, the method blank is an aliquot of sodium sulfate. For air samples, the method blank may be an aliquot of XAD resin, PUF, filter, or other matrix that is representative of the sample matrix. The QC aliquots are processed in the same manner and at the same time as the associated samples. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher. The laboratory will attempt to run all samples from a preparation batch within the same sequence on the same instrument. If this is not possible, samples may be run on different sequences or different instruments, and will be accompanied by an instrument blank.

9.4.1. Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.4.2. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.4.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.4.4. Refer to the QC Program document (WS-PQA-003) for further details of the corrective actions.

9.5. Laboratory Control Samples (LCS)

A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Table 5 unless otherwise specified by a client or agency.

9.5.1. If any analyte in the LCS is outside the method control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

9.5.2. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the LCS recovery is high, the samples are ND, and sample surrogate recoveries are good).

9.5.3. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.4. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples for aqueous and solid matrices, where sufficient sample volume is available. The MS/MSD is spiked with the same set of analytes as the LCS (See Table 5). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

9.6.1. If any individual recovery or RPD falls outside the acceptable range, corrective action must occur (WS-PQA-003, Section 7.6.5). The initial

corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

9.6.2. If the recovery for any component is outside QC limits for both the MS/MSD and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.

9.6.3. If an MS/MSD is not possible due to limited sample, then a LCS duplicate may be analyzed if required by the program or client. RPD of the LCS and LCSD are compared to the matrix spike limits.

9.6.4. The MS/MSD must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.9. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (WS-PQA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.

10.2. Instrument Tuning

At the beginning of every twelve-hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 10) are achieved for DFTPP (decafluorotriphenylphosphine).

10.2.1. Inject 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS

system. Obtain a background-corrected mass spectrum of DFTPP and confirm that all the key m/z criteria in Table 10 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.2.1.1. Evaluate the mass spectrum for DFTPP either at the apex of the peak, or ± 1 scan from the apex. Alternatively, average the mass spectra from 1 scan before the apex to 1 scan following the apex.

10.2.2. The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system.

10.2.2.1. DDT must be included in the tuning standard, and its breakdown must be < 20%. Refer to Section 12 for the appropriate calculations.

10.3. Initial Calibration

The initial calibration is required before any samples are analyzed, and then intermittently throughout sample analyses as dictated by results of the continuing calibration procedures described in this section, and after any major maintenance. The GC/MS system must be properly calibrated and the performance documented during the initial calibration.

10.3.1. GC/MS Tuning Criteria

Use a compound such as perfluorotributylamine (PFTBA) to verify that the intensity of the peaks is acceptable. If PFTBA is used, mass spectral peak profiles for m/z 69, 219, and 264 must be recorded, plotted and reported. The scan should display a minimum of ± two amu (i.e. m/z 67-71 for the m/z 69 profile) and the masses be within 0.50 amu of the target masses. The tune is verified at the beginning of every continuous sequence.

Note: *The requirement to analyze the instrument performance check solution is optional when analysis of Polyaromatic Hydrocarbons (PAHs)/pentachlorophenol is to be performed by the Selected Ion Monitoring (SIM) technique. (USEPA CLP SOW1.2)*

Note: *The EPA CLP Functional Guidelines for semivolatile organic analysis, Section II (Gas Chromatograph/Mass Spectrometer (GC/MS) Instrument Performance Check), Item B (Objective), states "GC/MS instrument performance checks are performed to ensure adequate mass resolution, identification, and to some degree, sensitivity. These criteria are not sample-specific. Conformance is determined using standard materials, therefore, these criteria should be met in all circumstances.*

Note: *This requirement does not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.*

10.3.2. GC Operating Conditions

The GC column performance must be documented during every analytical sequence. Table 8 summarizes GC operating conditions known to produce acceptable results with the column listed. The GC conditions must be established for the particular instrument by injecting aliquots of the calibration check standard. The valley height between benzo(b)fluoranthene and benzo(k)fluoranthene must be no more than ½ the height of the taller peak. It may be necessary to adjust the operating conditions slightly based on observations from analysis of these solutions. Thereafter, the calibration check standard must be analyzed daily to verify the performance of the system.

10.4. Calibration Procedure for Internal Standard Method

10.4.1. The internal standards used in this method are listed in Table 3. Use the base peak m/z as the primary m/z for quantitation of the standards, and the secondary m/z for confirmation purposes only. If interferences are noted one of the next two most intense masses may be used for quantitation.

10.4.2. Compounds are assigned to the internal standards as indicated in Table 6.

10.4.3. Using stock standards, prepare at least five calibration standard solutions, using the same solvent that is used in the final sample extract. Keep the internal standards at fixed concentrations. Table 4 lists the recommended calibration standard concentrations.

10.4.4. Calibrate the mass spectrometer response using a 2-5 µL aliquot of each calibration solution. Each solution must be analyzed once. Calculate the response factors (RFs) for each analyte:

10.4.4.1. Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations 1, 2, and 3 and verify that the criteria in Table 7 are met. **No sample analysis may be performed unless these criteria are met.**

10.4.4.2. Mean Response Factor - Use Equation 2 to calculate the mean RF for each compound (calibration standards and surrogate standards).

This is the average of the five RFs calculated for each compound (one RF calculated for each calibration solution).

10.4.5. If the %RSD for any analyte exceeds 30% then a linear calibration should be tried for all analytes with %RSD > 30%. Linear or quadratic curve fits may be used. Use of 1/Concentration2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990. The "Y" intercept (as printed on the Chrom ICAL plot) must be less than ± ½ RL. If the intercept exceeds this criterion, false positives or negatives could result. If the RL is elevated for client or project requirements, use the default RL or MDL.

10.4.6. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.7. The %RSD should be $\leq$15% for DOD projects and $\leq$25% for Method 8270D criteria.

10.4.8. Initial calibration verification standard (ICV) - When available, a second source standard should be used for ICV and analyzed with the initial calibration. Each compound must be within +/- 30% of its expected value. Under the DoD QSM, the criteria are +/- 20%D for all analytes.

Corrective actions for a failed ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards
- Narrate all affected data.

10.4.9. If time remains in the 12-hour period initiated by initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.10. Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

10.5. Continuing Calibration (Calibration Check)

The calibration check standard must be analyzed at the beginning of each analysis period, or at the beginning of every 12-hour shift if the laboratory operates during consecutive 12-hour shifts.

10.5.1. Open the perfluorotributylamine (PFTBA) to verify that the intensity of the peaks is acceptable. The mass spectral peak profiles for m/z 69, 219, and 264 must be recorded, plotted and reported. The scan should display a minimum of ± two amu (i.e. m/z 67-71 for the m/z 69 profile). The analysis must produce masses within 0.50 amu of the target masses to show acceptable spectral profiles. Use the same data acquisition parameters as those used during the initial calibration. This verification is performed at the beginning of every consecutive sequence and must be performed at least once a day

10.5.2. Inject a 2-5ul aliquot of the calibration check solution into the GCMS. Use the same data acquisition parameters as those used during the initial calibration.

10.5.3. Update the retention time windows for each of the compounds based on the daily standard. The retention time for the calibration standard must not change by more than 30 seconds from the most recent calibration check if no maintenance has been performed since the last check. If the retention time shifts by more than 30 seconds and no maintenance has been performed, inspect the chromatographic system for malfunctions and make the necessary corrections. Document acceptable performance with a new initial calibration curve.

10.5.4. Calculate the response factors. Refer to Section 12 for the appropriate equations.

10.5.5. Calculate the delta RF (ΔRF) which is the Relative Percent Difference (RPD) between the daily RF and the initial calibration mean RF. The measured RFs of all analytes (native and surrogate) must be within the criteria noted in Table 7, of the mean values established during the initial calibration. If this criterion is not satisfied, assess the potential impact on the data, if any possible impact is suspected appropriate corrective action must be performed. The corrective action may include re-injection of the continuing calibration standard, instrument maintenance, or injection of a new initial calibration curve, before sample extracts can be analyzed.

10.5.6. Check the EICP areas of the internal standards for the Continuing Calibration Verification Standard (CCV) versus the mid-point standard level of the most recent initial calibration sequence. If the area changes by a factor of greater than two (-50% to +100%), the analytical system must be inspected to determine the cause of the change and corrections must be made, as appropriate. If corrections are made, the CCV must be analyzed again to

verify the system is operating properly. If a system malfunction is found, the samples analyzed while the system was malfunctioning must be evaluated for potential impacts, and reanalyzed as necessary.

10.5.7. Recommended corrective actions for CCV failure:

- Re-analysis CCV.
- Instrument maintenance.
- A new initial calibration curve.

10.5.8. Once the above criteria have been met, sample analysis may begin.

11. PROCEDURE

11.1. Sample Extraction

Procedures for separatory funnel liquid/liquid extraction, sonication extraction, K-D concentration and N-Evap Concentration are described in SOP WS-OP-0001. Procedures for the extraction of air samples are described in SOP WS-OP-0006.

11.2. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.3. GC/MS Analysis

The laboratory may proceed with the analysis of QC and field samples only after demonstrating acceptable performance as specified in Section 10.

11.3.1. Calibrate the instrument as described in Section 10.

11.3.2. All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.

11.3.3. Approximately 1 hour before analysis, remove the sample extracts from the freezer and the internal standard solution from the refrigerator and allow them to warm to room temperature.

11.3.4. Add 10 µL of the internal standard solution to all QC and filed samples to

achieve a final concentration of 500 ng/mL. If the sample volume must be changed to achieve a desired detection limit, the internal standard solution concentration must be adjusted accordingly to achieve the target concentrations of Table 4.

11.3.5. Inject the sample extract into the instrument using the same injection technique as used for the standards.

11.3.6. The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.3.7. Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.3.8. Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

11.3.9. The presence of a given PAH is qualitatively confirmed if the criteria of Section 12.1 are satisfied. The response for any quantitation ion in the sample extract should not exceed the response of the highest concentration calibration standard. If the response exceeds the highest concentration calibration standard, the sample extract should be diluted to get the analyte response within the linear range of the calibration curve.

11.4. GC/MS Analysis - Large Volume Injection (LVI)

11.4.1. For samples being analyzed by LVI, add 5 µL of the internal standard solution to all samples and QC to achieve a final concentration of 500 ng/mL. (Final extract volume for LVI samples is 0.5 mL). If the sample volume must be changed to achieve a desired detection limit, the recovery spike solution concentration must be adjusted accordingly to achieve the target concentrations of Table 5

11.4.2. Inject a 5 µL aliquot of the sample extract on the instrument. Recommended GC/MS operating conditions are described in Table 8.

11.5. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the reporting limit and the matrix allows for analysis at a lesser dilution, the

sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.5.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a less dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.5.2. Reporting Dilutions

11.5.2.1. In general, the most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.5.2.2. Under DOD QSM, all valid dilutions for a sample must be reported.

11.5.3. Each time the sample extract is diluted, internal standard solution must be added to maintain a final concentration of 500 ng/mL for each internal standard component.

11.6. Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at < -10^{o}C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.7. Retention time criteria for samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.7.1. If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.8. Troubleshooting Guide

11.8.1. Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the Sacrament QAM, the following daily maintenance may be performed.

- Clip column front or back as necessary.

- Replace injection port liner as necessary.
- Replace gold seal as necessary.
- Replace septum as necessary.
- Perform mass calibration as necessary

11.8.2. Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12. CALCULATIONS/DATA REDUCTION

12.1. Qualitative Analysis

Identification Criteria: The presence of a given PAH is qualitatively confirmed if the criteria below are satisfied.

12.1.1. The elution of the sample component must compare to within +/- 0.2 minutes of the GC retention time of the standard component in the daily calibration check.

12.1.2. The characteristic ions of the component in the sample must match standard component characteristic ions.

12.1.3. A primary and secondary ion will be monitored for all labeled and unlabeled analytes, which have a secondary ion >10% of the primary. The secondary ion will be used for qualitative identification. The presence of the secondary ion will be dependent upon the analyte concentration in the sample. The secondary ion may not be present for analytes detected at < 5x the reporting limit. Samples with concentrations of the target analyte 5-10x the reporting should have the secondary ions present at a detectable level.

12.1.4. The relative intensities of the ions should agree within +/- 30% between the standard and sample spectra.

12.1.5. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification with the appropriate footnote and proceed with quantitation.

12.1.6. If in the judgment of the analyst the separation of benzo(b)fluoranthene and benzo(k)fluoranthene is insufficient for quantitation, the pair may be reported as a total peak, based on the average response factor of the two.

12.1.7. The internal standard areas in the samples will be monitored against the mid-

point initial calibration standard to evaluate possible instrument drift, but specific criteria or corrective actions would typically be defined in project specific QAPPs.

12.2. Calculations

12.2.1. Refer to policy QA-004-SAC for rules regarding significant digits in calculations. Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation.

12.2.2. Response Factors (RF) for native PAH and Surrogate Standards –Use the data obtained during initial calibration (10.2.) or continuing calibration (10.3).

Equation 1: Error! Objects cannot be created from editing field codes.

Where:

Ax = Area of the characteristic ion for the compound being measured

Ais = Area of the characteristic ion for the specific internal standard

Cx = Concentration of the compound being measured (µg/L)

Cis = Concentration of the specific internal standard (µg/L)

12.2.3. Mean Response Factors (RFavg) — Calculate the mean RF for each target PAH and surrogate standard, internal standard and alternate standard using Equation 2 and the RFs calculated according to Equation 1.

Equation 2:
$$RRF_{avg} = \frac{1}{n}\sum_{i=1}^{n}(RRF_i)$$

Where:

RRF = RRF calculated for calibration solution "i"

n = The number of data points derived from the calibration. The minimum requirement is a five-point calibration

12.2.4. Percent Relative Standard Deviation for Initial Calibration

Equation 3:
$$\%RSD = \frac{SD}{RF_{avg}} \times 100$$

Where:

RFavg = Mean of RFs from initial Calibration for a compound.

SD = Standard deviation of RFs from initial calibration for a compound.

$$= \sqrt{\sum_{i-1}^{n} \frac{(RFi - RFavg)^2}{n = 1}}$$

RFi = RF for calibration level i

n= number of RF values used.

12.2.5. Continuing Calibration ΔRF - ΔRF is the relative percent difference (RPD) between the daily RF and the mean RF calculated during initial calibration.

Equation 4:

$$\Delta RF = \frac{RF_c - RF_{avg}}{RF_{avg}} \times 100\%$$

Where:

RFavg = Mean response factor of a given analyte

RFc = The RF of a given analyte obtained from the continuing calibration

12.2.6. Total Mass of Target PAH or Surrogate Standard in Sample – (Ms)

Equation 5:

$$C_{ex} = \frac{R_x C_{is}}{R_{is}\overline{RF}}$$

Where:

C_{ex}= Concentration in extract, µg/mL

R_x = Response for analyte

R_{is}= Response for internal standard

C_{is} = Concentration of internal standard

12.2.7. Analyte Concentration in Sample, Aqueous Samples

Equation 6a:

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL)

V_o = Volume of water extracted (mL)

12.2.8. Analyte Concentration in Sample, Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis):

Equation 6b:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

Where:

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.2.9. MS/MSD percent recovery calculation.

Equation 7: Error! Objects cannot be created from editing field codes.

Where:

S_{SR} = Spike sample result

S_R = Sample result

S_A = Spike added

12.2.10. Relative % Difference calculation for the MS/MSD

Equation 8:

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit (MDL)

The laboratory must generate a MDL for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out.

15.1. 2 milliliter autovials contaminated with methylene chloride, after extracts are analyzed. After analysis, the vials are returned to long-term storage. When released for disposal, they are moved into vial waste collection carboys. When the carboy is full, or after no more than one year, it is transferred to the waste disposal area, where the vials are run through the vial eater for disposal.

15.2. Waste methylene chloride from instrument needle cleaning process. Waste methylene chloride is collected in vials as part of the instrument operation. When full, the contents of the vial are poured into collection carboys. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel methylene chloride solvent drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment

16. REFERENCES/CROSS REFERENCES

16.1. “Handbook of Analytical Quality Control in Water and Wastewater Laboratories”, USEPA EMSL, EPA-600/4-79-019 (3/78)

16.2. CARB Method 429. Determination of Polycyclic Aromatic Hydrocarbons (PAH) Emissions from Stationary Sources. July, 1997.

16.3. Carcinogens - "Working with Carcinogens", Department of Health, Education and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, Aug., 1977.

16.4. "OSHA Safety and Health Standards, General Industry," (29CFR1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).

16.5. "Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

16.6. USEPA CLP SOW for Organics Analysis SOM1.2 (6/2007) Section 9.2.1

16.7. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.8. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update IV, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270D, Revision 4, February 2007

17. METHOD MODIFICATIONS

17.1. There are no deviations from the method.

18. ATTACHMENTS

18.1. Table 1- PAH Target Analytes and Standard Reporting Limits

18.2. Table 2- Extraction Surrogate Spiking Solution Concentration

18.3. Table 3- Internal Standard Spiking Solution Concentration

18.4. Table 4- Concentrations of the PAH's in Working GC/MS Calibration Standards

18.5. Table 4A - Concentrations of the PAH's in Working GC/MS Calibration Standards using Large Volume Analysis.

18.6. Table 5-Concentrations of Compounds in Laboratory Control Spike Samples (LCS)

18.7. Table 6- Assignment of Internal Standards for Calculating RFs and Quantiting Target PAH and Surrogate Standards

18.8. Table 7- Minimum Requirements for Response Factors for Initial and Continuing Calibrations

18.9. Table 8- Recommended GC Operating Conditions for PAH analysis

18.10. Table 9- Recommended MS Operating Conditions

18.11. Table 10- DFTPP Key Ions and Ion Abundance Criteria for Method 8270

19. REVISION HISTORY

19.1. WS-MS-0008, Revision 2.8, Effective 06/21/2018

19.1.1. Added Section 7.4, "GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL o-decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the

Tuning Standard at 50µg/mL. This solution is valid for one year."

19.1.2. Added Section 10.2, "Instrument Tuning" and its associated subsections.

19.1.3. Added 2-Methylnaphhalene-d10 and Fluoranthene-d10 to Tables 2, 4, 4A, and 7.

19.1.4. Table 4, added 10ng/mL and 50ng/mL as additional concentrations.

19.1.5. Updated Table 9 with current Operating Conditions.

19.1.6. Added Table 10, "DFTPP Key Ions and Ion Abundance Criteria for Method 8270".

19.1.7. Removed revision history prior to 2015, it can be found in previous versions of this SOP.

19.1.8. Editorial changes.

19.2. WS-MS-0008, Revision 2.7, Effective 06/28/2016

19.2.1. Section 1.4 – Changed to "When undertaking projects for Department of Defense (DOD) and or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.

19.2.2. Table 4 – Changed column 1 from 50ng/mL to 25 ng/mL; removed 300 ng/mL level (column 7).

19.2.3. Editorial changes.

19.3. WS-MS-0008, Revision 2.6, Effective 03/20/2015

19.3.1. Inserted Section 6.3.1 – Acquisition Software: Chemstation Software Version D.

19.3.2. Inserted Section 6.3.2 – Data Processing Software: Chrom version 2.1

19.3.3. Changed Section 10.3.5 - The "Y" intercept (listed as "b" on the ICAL summary pages) must be less than ± ½ RL

19.3.4. Editorial changes

Table 1	
PAH Target Analytes	
Naphthalene	Pyrene
1-Methylnapthalene	Benzo(b)fluoranthene
2-Methylnapthalene	Benzo(k)fluoranthene
Acenaphthylene	Benzo(a)pyrene
Acenaphthene	Benzo(ghi)perylene
Fluorene	Indeno(1,2,3-cd)pyrene
Phenanthrene	Dibenz(ah)anthracene
Anthracene	Benzo(a)anthracene
Fluoranthene	Chrysene

Table 2	
Extraction Surrogate Spiking Solution Concentration	
Analytes	**ug/mL**
Nitrobenzene-d5	0.5
2-Fluorobiphenyl	0.5
Terphenyl-d14	0.5
2-Methylnapthalene-d10	0.5
Fluoranthene-d10	0.5

Table 3	
Internal Standards Spiking Solution Concentration	
Analytes	**ug/mL**
Naphthalene-d8	50
Acenaphthene-d10	50
Phenanthrene-d10	50
Chrysene-d12	50
Perylene-d12	50

Table 4 Concentrations of the PAH's in Working GC/MS Calibration Standards									
Components	Solution Concentrations (ng/mL)								
	1	2	3	4	5	6	7	8	9
Calibration Standard									
Naphthalene	10	25	50	100	250	500	1000	2000	5000
1-Methylnaphthalene	10	25	50	100	250	500	1000	2000	5000
2-Methylnaphthalene	10	25	50	100	250	500	1000	2000	5000
Acenaphthene	10	25	50	100	250	500	1000	2000	5000
Acenanapthalene	10	25	50	100	250	500	1000	2000	5000
Fluorene	10	25	50	100	250	500	1000	2000	5000
Phenanthrene	10	25	50	100	250	500	1000	2000	5000
Anthracene	10	25	50	100	250	500	1000	2000	5000
Fluoranthene	10	25	50	100	250	500	1000	2000	5000
Pyrene	10	25	50	100	250	500	1000	2000	5000
Benzo(a)anthracene	10	25	50	100	250	500	1000	2000	5000
Chrysene	10	25	50	100	250	500	1000	2000	5000
Benzo(b)fluoranthene	10	25	50	100	250	500	1000	2000	5000
Benzo(k)fluoranthene	10	25	50	100	250	500	1000	2000	5000
Benzo(a)pyrene	10	25	50	100	250	500	1000	2000	5000
Benzo(ghi)perylene	10	25	50	100	250	500	1000	2000	5000
Indeno(123-cd)pyrene	10	25	50	100	250	500	1000	2000	5000
Dibenz(ah)anthracene	10	25	50	100	250	500	1000	2000	5000
Extraction Surrogate Standards									
Nitrobenzene-d5	10	25	50	100	250	500	1000	2000	5000
Fluorobiphenyl	10	25	50	100	250	500	1000	2000	5000
Terphenyl-d14	10	25	50	100	250	500	1000	2000	5000
2-Methylnaphthalene-d10	10	25	50	100	250	500	1000	2000	5000
Fluoranthene-d10	10	25	50	100	250	500	1000	2000	5000
Internal Standard									
Naphthalene-d8	500	500	500	500	500	500	500	500	500
Acenaphthene-d10	500	500	500	500	500	500	500	500	500
Phenanthrene-d10	500	500	500	500	500	500	500	500	500
Chrysene-d12	500	500	500	500	500	500	500	500	500
Perylene-d12	500	500	500	500	500	500	500	500	500

Table 4A Large Volume Analysis (5uL) Concentrations of the PAH's in Working GC/MS Calibration Standards								
Components	Solution Concentrations (ng/mL)							
	1	2	3	4	5	6	7	8
Calibration Standard								
Naphthalene	25	50	100	200	400	600	800	1000
1-Methylnaphthalene	25	50	100	200	400	600	800	1000
2-Methylnaphthalene	25	50	100	200	400	600	800	1000
Acenaphthene	25	50	100	200	400	600	800	1000
Acenanapthalene	25	50	100	200	400	600	800	1000
Fluorene	25	50	100	200	400	600	800	1000
Phenanthrene	25	50	100	200	400	600	800	1000
Anthracene	25	50	100	200	400	600	800	1000
Fluoranthene	25	50	100	200	400	600	800	1000
Pyrene	25	50	100	200	400	600	800	1000
Benzo(a)anthracene	25	50	100	200	400	600	800	1000
Chrysene	25	50	100	200	400	600	800	1000
Benzo(b)fluoranthene	25	50	100	200	400	600	800	1000
Benzo(k)fluoranthene	25	50	100	200	400	600	800	1000
Benzo(a)pyrene	25	50	100	200	400	600	800	1000
Benzo(ghi)perylene	25	50	100	200	400	600	800	1000
Indeno(123-cd)pyrene	25	50	100	200	400	600	800	1000
Dibenz(ah)anthracene	25	50	100	200	400	600	800	1000
Extraction Surrogate Standards								
Nitrobenzene-d5	25	50	100	200	400	600	800	1000
Fluorobiphenyl	25	50	100	200	400	600	800	1000
Terphenyl-d14	25	50	100	200	400	600	800	1000
2-Methylnaphthalene-d10	25	50	100	200	400	600	800	1000
Fluoranthene-d10	25	50	100	200	400	600	800	1000
Internal Standard								
Naphthalene-d8	500	500	500	500	500	500	500	500
Acenaphthene-d10	500	500	500	500	500	500	500	500
Phenanthrene-d10	500	500	500	500	500	500	500	500
Chrysene-d12	500	500	500	500	500	500	500	500
Perylene-d12	500	500	500	500	500	500	500	500

Table 5
Concentrations of Compounds in Laboratory Control Spike Samples (LCS)

Compound	Aqueous (ng/L)	Solid (ng/g)
Naphthalene	500	25
1-Methylnapthalene	500	25
2-Methylnapthalene	500	25
Acenaphthylene	500	25
Acenaphthene	500	25
Fluorene	500	25
Phenanthrene	500	25
Anthracene	500	25
Fluoranthene	500	25
Pyrene	500	25
Benzo(a)anthracene	500	25
Chrysene	500	25
Benzo(b)fluoranthene	500	25
Benzo(k)fluoranthene	500	25
Benzo(ghi)perylene	500	25
Indeno(123-cd)pyrene	500	25
Dibenz(ah)anthracene	500	25

Table 6 Assignment of Internal Standards for Calculating RFs and Quantitating Target PAHs and Surrogate Standards	
Analyte	**Internal Standards**
Compound	
Naphthalene	Naphthalene-d8
1-Methylnaphthalene	Naphthalene-d8
2-Methylnaphthalene	Naphthalene-d8
Nitrobenzene-d5 (surr)	Naphthalene-d8
Acenaphthylene	Acenaphthene-d10
Acenaphthene	Acenaphthene-d10
Fluorene	Acenaphthene-d10
2-Fluorobiphenyl (surr)	Acenaphthene-d10
Phenanthrene	Phenanthrene-d10
Anthracene	Phenanthrene-d10
Fluoranthene	Phenanthrene-d10
Pyrene	Phenanthrene-d10
Terphenyl-d14 (surr)	Chrysene-d12
Benzo(a)anthracene	Chrysene-d12
Chrysene	Chrysene-d12
Benzo(b)fluoranthene	Perylene-d12
Benzo(k)fluoranthene	Perylene-d12
Benzo(a)pyrene	Perylene-d12
Indeno(1,2,3-c,d)pyrene	Perylene-d12
Dibenz(a,h)anthracene	Perylene-d12
Benzo(ghi)perylene	Perylene-d12

Table 7						
Minimum Requirements for Response Factors for Initial and Continuing Calibrations						
	8270C			8270D		
Analyte	Initial Calibration +/-% RSD	Second Source Criteria ΔRF (%)	Continuing Calibration Criteria ΔRF (%)	Initial Calibration ≤% RSD	Second Source Criteria ≤% RSD	Continuing Calibration Criteria ≤% RSD
Naphthalene	30	30	30	20	30	20
1-Methylnaphthalene	30	30	30	20	30	20
2-Methylnaphthalene	30	30	30	20	30	20
Acenaphthene	30	30	30	20	30	20
Acenaphthylene	30	30	30	20	30	20
Fluorene	30	30	30	20	30	20
Phenanthrene	30	30	30	20	30	20
Anthracene	30	30	30	20	30	20
Fluoranthene	30	30	30	20	30	20
Pyrene	30	30	30	20	30	20
Benzo(a)anthracene	30	30	30	20	30	20
Chrysene	30	30	30	20	30	20
Benzo(b)fluoranthene	30	30	30	20	30	20
Benzo(k)fluoranthene	30	30	30	20	30	20
Benzo(a)pyrene	30	30	30	20	30	20
Benzo(g,h,i)perylene	30	30	30	20	30	20
Indeno(1,2,3-c,d)pyrene	30	30	30	20	30	20
Dibenz(a,h)anthracene	30	30	30	20	30	20
Nitrobenzene-d_5	30	30	30	20	30	20
2-Fluorobiphenyl	30	30	30	20	30	20
d_{14}-Terphenyl	30	30	30	20	30	20
2-Methylnaphthalene-d10	30	30	30	20	30	20
Fluoanthene-d10	30	30	30	20	30	20

Table 8 Recommended GC Operating Conditions for PAH analysis	
Parameter	Setting
Column	HP-5MS (or equivalent) 30m x 0.25mm id x 0.25µm film
Carrier Gas/Linear Velocity	He at 40 cm/sec
Injection Mode	Splitless
Temperature Program	60°C, hold 1.0 min, 18°C /min to 150 °C, 9°C /min. to 300°C, hold until after Benzo(g,h,i)perylene elutes (approximately 1.5 minutes)
Injector Temperature	250°C

Table 9

Recommended MS Operating Conditions

Parameter	Setting	Ch	Mass	Dwell (ms)	Component
Program 1					
Type	SIM	1	54	20	Nitrobenzene-d_5*
High Mass	172	2	64	20	
Low Mass	54.0	3	68	20	Naphthalene-d_8*
Resolution	1 amu	4	71	20	
Ionization Mode	EI+	5	82	20	Nitrobenzene-d_5
Number of Channels	21	6	99	20	
Cycles/Second	1.00	7	102	20	Naphthalene*
		8	112	20	
		9	127	20	Naphthalene*
		10	128	20	Naphthalene Nitrobenzene-d_5*
		11	132	20	Perylene-d_{12}*
		12	134	20	Naphthalene-d_8*
		13	136	20	Naphthalene-d_8
		14	141	20	2-Methylnaphthalene*, 1-Methylnaphthalene*
		15	142	20	2-Methylnaphthalene, 1-Methylnaphthalene
		16	151	20	Acenaphthylene*
		17	152	20	2-Methylnaphthalene-d_{10}, Acenaphthylene, Acenaphthene*
		18	153	20	Acenaphthylene*, Acenaphthene
		19	154	20	Acenaphthene*
		20	158	20	
		21	160	20	Acenaphthene-d_{10}*
		22	162	20	Acenaphthene-d_{10}*
		23	164	20	Acenaphthene-d_{10}
		24	170	20	2-Fluorobiphenyl*
		25	171	20	2-Fluorobiphenyl*
		26	172	20	2-Fluorobiphenyl
Program 2					
Type	SIM	1	57	20	
High Mass	188	2	71	20	
Low Mass	80	3	80	20	Phenanthrene-d_{10}*

Table 9

Recommended MS Operating Conditions

Parameter	Setting	Ch	Mass	Dwell (ms)	Component
Resolution	1 amu	4	85	20	
Ionization Mode	EI+	5	94	20	Phenanthrene-d_{10}
Number of Channels	10	6	141	20	1-Methylnaphthalene*, 2-Methylnaphthalene*
		7	165	20	Fluorene*
		8	166	20	Fluorene
		9	174	20	
		10	176	20	Phenanthrene*, Anthracene*
		11	178	20	Phenanthrene, Anthracene
		12	184	20	
		13	188	20	Phenanthrene-d_{10}
		14	264	20	Perylene-d_{10}
		15	266	20	
		16	268	20	
		17	330	20	
		18	332	20	
Program 3					
Type	SIM	1	57	20	
High Mass	244	2	71	20	
Low Mass	100	3	85	20	
Resolution	1 amu	4	101	20	Fluoranthene*, Pyrene*
Ionization Mode	EI+	5	106	20	Fluoranthene-d_{10}*
Number of Channels	16	6	114	20	Benzo(a)anthracene*, Chrysene*
Cycles/Second		7	120	20	Chrysene-d_{12}*
		8	122	20	Terphenyl-d_{14}*, 2-Methylnaphthalene-d_{10}*
		9	200	20	Fluoranthene*, Pyrene*
		10	202	20	Fluoranthene, Pyrene
		11	208	20	Fluoranthene-d_{10}*
		12	212	20	Fluoranthene-d_{10}, Terphenyl-d_{14}
		13	226	20	Benzo(a)anthracene*, Chrysene*
		14	228	20	Benzo(a)anthracene, Chrysene
		15	236	20	Chrysene-d_{12}*
		16	240	20	Chrysene-d_{12}
		17	244	20	Terphenyl-d_{14}
Program 4					

Table 9

Recommended MS Operating Conditions

Parameter	Setting	Ch	Mass	Dwell (ms)	Component
Type	SIM	1	57	20	
High Mass	279	2	71	20	
Low Mass	125	3	85	20	
Resolution	1 amu	4	126	20	Benzo(b)fluoranthene*, Benzo(k)fluoranthene*, Benzo(a)pyrene*
Ionization Mode	EI+	5	132	20	Perylene-d_{12}*
Number of Channels	16	6	138	20	Indeno(1,2,3-cd)pyrene*, Benzo(g,h,i)perylene*
Cycles/Second		7	139	20	Dibenz(a,h)anthracene*
		8	146	20	
		9	252	20	Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(a)pyrene
		10	253	20	Benzo(b)fluoranthene*, Benzo(k)fluoranthene*, Benzo(a)pyrene*
		11	260	20	Perylene-d_{12}*
		12	264	20	Perylene-d_{12}
		13	276	20	Indeno(1,2,3-cd)pyrene, Benzo(g,h,i)perylene
		14	277	20	Indeno(1,2,3-cd)pyrene*, Benzo(g,h,i)perylene*
		15	278	20	Dibenz(a,h)anthracene
		16	279	20	Dibenz(a,h)anthracene*
		17	284	20	
		18	288	20	
		19	292	20	

** Ion used to monitor hydrocarbon interference*

Table 10
DFTPP Key Ions and Ion Abundance Criteria for Method 8270

Mass	Ion Abundance Criteria
51	30 – 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 – 60% of mass 198
197	<1% of mass 198 *** (see below for TO-13A)**
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 – 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198 *** (see below for TO-13A)**
443	17 – 23% of mass 442

Note: (* For Method TO-13A mass 197 must be less than 2% of mass 198 and mass 442 must be 40 - 110% of mass 198)

Title: Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry [Methods 200.8, 6020 and 6020A]

Approvals (Signature/Date):

Robert Hrabak, Technical Manager — 11/14/17 Date	Joe Schairer, Health & Safety Manager / Coordinator — for: 11/14/2017 Date
Lisa Stafford, Quality Assurance Manager — 11/14/2017 Date	Crystal Pollock, Laboratory Director — 11.14.17 Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA Method 200.8, 6020 and 6020A.

1.2. Table 1 lists the analytes contained in the standard analyte list for ICPMS. These are the analytes for which the laboratory maintains certification in the form of PT samples, MDLs, IDLs, DOCs and linear range analyses. Elements not in this list may be analyzed with the client's knowledge that we do not maintain certification for analytes, only standards, outside the scope of our standard product.

1.3. Methods 200.8, 6020 and 6020A are applicable to the determination of total recoverable analyses in ground water, surface waters, and TCLP, STLC, and SPLP leachates/extracts. These methods are also applicable to the total analysis of soils, sludges, sediments and wastes. All matrices require digestion prior to analysis with the exception of STLC extracts. At a minimum, dissolved samples and STLCs are matrix matched under the guidelines of WS-IP-0008. STLCs are to be matrix matched at a 10x dilution. Silver concentrations must be below 0.1 mg/L in aqueous samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data

1.4. Reporting Limits

1.4.1. The standard reporting limits for metals analyzed by ICP-MS are listed in Table 1.

1.4.2. Reporting limits may differ from those listed if prescribed in a reviewed and finalized client specific QAPP.

1.4.3. Reporting limits for samples analyzed under QSM criteria may differ from those shown in Table 1 and in many cases are specific to the client.

1.5. When undertaking projects for Department of Defense (DOD) and/or the Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, "Federal Program Requirements", must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Aqueous samples, digestates or leachates are introduced via a cross flow nebulizer into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a radio-frequency plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber

that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Isobaric interferences: Isobaric interferences associated with naturally occurring isotopes are automatically corrected by the instrument software.

4.2. Isobaric molecular interferences: Corrections for molecular interferences will be applied where appropriate based on known or suspected interferences.

4.3. See Tables 8 and 9 for a list of common interferences and the isotopes affected.

Note: Not all-possible interferences are listed.

4.3.1. Matrix interferences: Internal standards will be used to correct for some matrix interferences.

4.3.2. A representative sample(s) of the project or projects being analyzed will be screened to avoid using an analyte as an internal standard that is present in the samples at a concentration greater than 10% of the internal standard concentration.

4.3.3. Internal standards should be added at a level to give approximately 100,000 - 2,000,000 counts of raw signal intensity. The mass of the internal standard used should ideally be within ±50 amu of the mass of the affected analyte.

4.3.4. Severe matrix effects are monitored by comparing the internal standard intensities of the sample to the internal standard intensities of the initial calibration blank. If any internal standard intensity is less than 30% or greater than 150% of the internal standard intensity of the calibration blank, a fivefold dilution will be performed on the sample to correct for severe matrix effects, and the sample reanalyzed.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S

Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.1.2. This instrument produces RF energy. People with pacemakers should not go near the instrument while it is in operation.

5.1.3. Some extractions and digestions use hydrofluoric acid, which means that some samples to be analyzed are in an HF solution. The labels for all such extracts and digestates will be highlighted in yellow or marked with yellow tape. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S staff and the local supplement to the Corporate Safety Manual for guidance. Anyone working with HF must receive special training before starting work. Staff members who work around HF should also receive this training. HF acid solutions may not be used for any purposes except as prescribed in this or other TestAmerica Sacramento SOPs. Processes involving HF acid are classified as high-risk activities. Personnel involved must wear a face shield in addition to safety glasses or goggles.

WARNING: The TestAmerica Sacramento Emergency Response Team must be activated for any suspected exposure to HF liquid or fumes. After local emergency response, the victim will be transported to the UCD Medical Center Emergency Room. Appendix 1 is a detailed first aid plan for working with HF.

5.1.3.1. 2.5% calcium gluconate gel should be applied copiously and gently massaged into burn sites after rinsing under cold running water for one minute to remove HF acid from the surface of the skin. The person massaging should wear appropriate protective gloves. If the victim is conscious, they should consume small quantities of calcium or magnesium containing liquids, such as milk, Maalox, or Mylanta. If the burn site is too large to massage with calcium gluconate gel, cover it with gauze soaked in an Epsom salt solution (1 cup to 1 quart of cold water). Keep the wraps saturated with the Epsom salt solution.

5.1.3.2. Some metals react with HF to produce flammable hydrogen gas.

5.1.3.3. Glass reacts with HF to produce toxic silicon tetraflouride.

5.1.3.4. HF is classified as a poison, and must be stored in a locked cabinet when not in use.

5.1.3.5. Whenever HF is in use in a laboratory, a decontamination solution will be prepared before starting work. Take a small bucket (2-3 gallons) and fill it about 2/3-3/4 full with an Epsom salts solution (1/2 cup of Epsom salts per quart of water). Before removing gloves or handling anything else that HF on the gloves might be transferred to, carefully dip one hand at a time into the Epsom salts solution for a few seconds. Do not put the hand any deeper than necessary to bring the solution level to within 1-2 inches of the glove cuff. The Epsom salts solution will be prepared each day prior to beginning work with HF, and disposed at the end of the day down the laboratory sink.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide satisfactory protection.

5.1.5. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.6. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

5.2.1. Although no hazardous materials are used during this process, the following is a list of the materials which might be found in the digestates, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

5.2.2. A complete list of materials used in the method can be found in the reagents

and materials section of the extraction/digestion SOP related to the samples in question.

5.2.3. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid (1)	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrofluoric Acid (1)	Poison Corrosive Dehydrator	3 PPM - TWA	Corrosive to the skin and eyes. Contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. Severely corrosive to the respiratory tract. Permanent eye damage may occur. THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

Preventive maintenance schedules for instrumentation may be found below.

INDUCTIVELY COUPLED ARGON PLASMA/MASS SPECTROMETRY (ICAP/MS)	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics. Measure quartz torch for proper alignment when removed and cleaned. Clean spray chamber and nebulizer. Clean all filters and fans. Check chiller coolant level. Check and drain oil mist eliminator on roughing pumps.	As Needed
	Check sample waste container level. Check quartz torch condition. Check RF coil. Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing. Check condition of sampler and skimmer cones. Check oil level of roughing pumps.	Daily(2)
	Replace oil in roughing pumps.	Every 2-3 Months

6.1. ICP-MS Instrument, either:

6.1.1. Agilent 7700x (G3281A) ICP-MS, equipped with:

6.1.1.1. Autosampler (ASX-5000 Series)

6.1.1.2. Integrated Sample Introduction System (ISIS)

6.1.1.3. MassHunter Software G7201B version B.01.03, Build 393.8

6.2. Argon gas: High-purity grade (99.99%).

6.3. Helium gas: High-purity grade (99.99%) for use with Agilent Instrument.

6.4. Hydrogen gas: High-purity grade (99.99%) for use with Agilent Instrument

6.5. Water re-circulator / chiller.

6.6. Adjustable air displacement pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes, 13 x 100 mm polystyrene or polypropylene.

7. REAGENTS AND STANDARDS

Use reagent grade chemicals in all tests. “Certificates of Analysis” should be supplied with all chemicals purchased. If not supplied, contact the vendor. When received, label the certificate and the reagent container with the receipt date. Reagent containers also need to be labeled with the opened and expiration dates.

7.1. Reagent water is produced by a Millipore Nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.3. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.4. Instrument rinse: Add 2000 mL of reagent water to the 4000 mL rinse container. Add 80 mL of concentrated HNO_3, 80 mL of concentrated HCl, then dilute to volume with reagent water.

7.5. Stock standards are purchased as custom multi-element mixes and as single-element solutions. These must be plasma grade standards.

7.5.1. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles.

7.5.2. Upon making a standard, the process must be documented in the standards log book and the standard given a specific ID according to laboratory procedure.

7.5.3. All intermediate and working standards must be labeled with the name of the preparer, the date prepared, the expiration date, acid concentrations, and the standard specific ID.

7.5.4. Standards containing silver must be protected from light. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer.

7.5.5. If no expiration date is provided, the stock solutions may be used for up to one year from opening and must be replaced sooner if verification from an independent source indicates a problem.

7.6. Calibration Standard/Continuing Calibration Standard: see Table 2 for instructions on making this standard.

7.7. Initial Calibration Verification (ICV) standard: see Table 3 for instructions on making this standard.

7.8. Low Level Check Intermediate Standard: see Table 4 for instructions on making this standard.

7.9. ICSA standard: see Table 5 for instructions on making this standard.

7.10. ICSAB standard: see Table 6 for instructions on making this standard.

7.11. Internal Standard A Intermediate: See Table 7 for instructions on making this standard.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.2. All soils must be refrigerated to <6^{o}C.

8.3. The analytical holding time for metals by ICP-MS is 6 months from date of collection.

9. QUALITY CONTROL

9.1. Batch - a quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards.

9.1.1. A batch must be prepared within the same time frame.

9.1.2. A method blank (MB) and a laboratory control sample (LCS) must be prepared as a part of every batch.

9.1.3. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/MSD).

9.1.4. An analytical batch must include all QC samples, however they do not contribute to the maximum of 20 samples. See policy WS-PQA-003 of the Quality Control Program for more details.

9.2. One method blank (MB) must be prepared for every 20 samples.

9.2.1. A method blank consists of reagent water processed through all of the steps, and at the same time as the associated samples.

9.2.2. If a method blank exceeds ± the reporting limit/limit of quantitation for a given analyte then the samples associated with that batch must be re-prepared.

9.2.2.1. The exception is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo.

9.2.3. For samples requiring laboratory filtration and preservation, the method blank must also be filtered and preserved. See policy WS-PQA-003 of the Quality Control Program for further details.

9.2.4. DOD/DOE projects: the results of the method blank must be less than ½ the

reporting limit for a given analyte. The same exceptions and corrective actions apply as stated in section 9.2.2.1

9.3. A laboratory control sample (LCS) must be prepared for every 20 samples.

9.3.1. A LCS for water samples consists of reagent water spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples. The LCS for solid samples consists of PTFE boiling chips spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples.

9.3.2. The LCS recoveries are evaluated against in-house generated control limits, which are generated per policy WS-PQA-003 and found in the laboratory's LIM system.

9.3.3. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-prepared for the out of control analyte/s except when a LCS exhibits high recovery.

9.3.3.1. For such a case, those samples with analyte concentrations less than the reporting limit can be reported.

9.3.3.2. All corrective actions must be documented on a Non-conformance memo. See policy WS-PQA-003 of the Quality Control Program for further details.

9.3.4. For samples analyzed under the DOD/DOE QSM, there is no exception for reporting "ND" samples for a high LCS.

9.3.5. Random marginal exceedance: For analyte lists of 11-30 elements, 1 LCS random marginal exceedance is allowed. One out of control analyte may be accepted if the result is within 4 standard deviations of the mean. The QA department must validate all marginal exceedance incidents. A NCM must be filed when reporting a marginal exceedance.

9.4. The lower limit of quantitation (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on an as needed basis to demonstrate the desired detection capability. Ideally, this check sample and the low-level calibration verification standard will be prepared at the same concentrations with the only difference being the LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within ± 30% of their true value. This check should be used to both establish and confirm the lowest quantitation limit. The LLQC going through preparation procedure will be called LLCS for TALs identification.

9.5. A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples.

For method 200.8 analysis, an MS is prepared and analyzed for every 10 field samples, i.e., for a batch of 10 samples or fewer, one MS is required, for batches of 11 -20 samples, 2 MS are required. Compare the percent recovery to the method limits of 70-130%.

9.5.1. MS/MSD pairs are aliquots of a selected field sample spiked with all of the analytes of interest at known concentrations The MS/MSD pair must be processed in the same manner and at the same time as the associated samples.

9.5.2. Compare the percent recovery and relative percent recoveries to that in the historically generated control limits. These control limits can be found in the laboratory's LIM system and are generated per policy WS-PQA-003.

9.5.2.1. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS.

9.5.2.2. Re-extraction of the blank, LCS, selected field samples, and the MS/MSD may be required after evaluation and review of the MS/MSD results.

9.5.3. A duplicate control sample (LCS/LCSD) may be substituted when insufficient volume is provided to process a MS/MSD pair, and batch precision is required by the client or program.

9.5.3.1. The LCS and LCSD are evaluated independently for acceptance. See policy WS-PQA-003 of the Quality Control Program for further details.

9.5.4. Samples identified as field blanks, equipment blanks, or trip blanks should not be used for MS/MSD analysis.

9.5.5. If the amount of an analyte found in the un-spiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply.

9.6. Initial Calibration Verification (ICV/ICB) – Calibration accuracy is verified by analyzing a second source standard (ICV) immediately upon completion of instrument calibration.

9.6.1. This standard must be at a concentration different from that used to calibrate the instrument and different from the CCV standard.

9.6.2. The ICV must fall within +/- 10% of the true value of the standard solution. Table 3 shows the concentration of each standard target analyte in the ICV.

9.6.2.1. An ICB (initial calibration blank) prepared the same as the calibration blank must be analyzed immediately following the ICV to monitor low level accuracy and system cleanliness.

9.6.2.2. The ICB result must fall within +/- the reporting limit from zero.

9.6.3. If either the ICV or ICB fail to meet acceptance criteria the analyst should determine the cause and proceed with corrective action if warranted. This may require re-running the ICV or ICB, repouring and rerunning either the CCV or ICB, or remaking and reanalyzing the ICV or ICB. Alternatively, analysis may continue for those analytes which meet the acceptance criteria in the ICV and ICB. The analyst may also recalibrate and rerun the ICV and ICB to verify the calibration.

9.6.4. DOD/DOE QSM: The ICB and CCB results for all analytes must be less than less than ½ the reporting limit (LOQ) for a given analyte.

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard, the continuing calibration standard, after every 10 samples and at the end of an analytical run.

9.7.1. Ten samples include any analysis that registers with a result, even if not used.

9.7.2. For method 6020 analysis, the calibration standard is also used as the CCV. The CCV result must fall within +/-10% of the true value for that solution.

9.7.3. A CCB (continuing calibration blank) is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero.

9.7.4. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples.

9.7.5. Results for given analytes may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs for the analytes.

9.7.6. If a mid-run CCV or CCB fails for any number of analytes, the possible cause of failure should be investigated, and all samples analyzed since the last acceptable CCV and or CCB must be reanalyzed for the unacceptable analytes.

9.7.7. If a mid-run CCV or CCB fails, the CCV or CCB may be reanalyzed once and accepted if there is a reason for the initial out-of-control event such as carryover from a high concentration sample.

9.7.8. During the course of an analytical run, the instrument may be “resloped” or recalibrated on a CCV/CCB pair to correct for instrument drift. A

recalibration must then be followed immediately by a new analysis of a CCV/CCB pair before any further samples may be analyzed. Any samples bracketed (including prior to the reslope) by a failed CCV/CCB must be reanalyzed for the unacceptable analytes.

9.8. Low Level Check Standard (LLSTD, LLICV, or LLCCV): Following analysis of the ICV/ICB pair the low level check standard/s is analyzed spiked with concentrations of each analyte of interest at or below the reporting limit. For method 6020A, it is also recommended to analyze the standard after samples (refer also to Section 10.9 for details and criteria).

9.8.1. The percent recovery of the low level standard results must be +/-50% of the true concentration for all standards analyzed that meet or exceed the reporting limit.

9.8.2. DOD/DOE QSM: Low level check standard recovery must be ± 20%.

9.8.3. For Method 6020A the LLICV results must be ± 30% of the true concentration for all standards analyzed that meet or exceed the reporting limit.

9.8.4. If the recovery is out of control, the analyst should determine the cause and proceed with corrective action. Appropriate corrective actions include rerunning the LLSTD, repouring and rerunning the LLSTD, or remaking and reanalyzing the LLSTD. Alternatively, analysis may continue for those analytes which meet the acceptance criteria in the LLSTD. The analyst may also recalibrate and rerun the ICV/ICB and other QC necessary for the run.

9.9. For all analyses run under DOD/DOE criteria, linear range standards (LRSTD) are analyzed after the completion of calibration.

9.9.1. The linear range standards contain the analytes of interest at high levels.

9.9.2. The acceptance criterion for linear range standards is ± 10% of the true value.

9.9.2.1. If this criterion is not met for a given analyte/s, a lower concentration standard should be analyzed such that the criterion is met.

9.9.3. Once linear range has been established through the analysis of the linear range standards, no value can be reported that exceeds the concentration of the standards.

9.9.3.1. If sample concentrations exceed that of the linear range standards for any given analyte, the sample requires dilution at a level where all analytes fall below the level of the linear range standards.

9.10. Interference Check Analysis (ICSA/ICSAB): Interference check standards ICSA and ICSAB are analyzed directly after analysis of the low level check standard.

9.10.1. The ICSA/ICSAB are essentially remnants of method 6010 where the analytes spiked in the standard at high levels are considered interferents. For method 6020 these are not normally interferents, but represent analytes that might be seen at higher levels in real world samples.

9.10.2. ICSA (interference check standard A) contains only the high level analytes (interferents) without any spiking of the analytes more notably determined at lower relative concentrations. See Table 5 for the composition of the ICSA.

9.10.3. ICSA is also spiked with 10 ppm bromide to evaluate the correction required for bromide interference on selenium 82. Once the ICSA has been analyzed, the correction factor should be adjusted such that the result for selenium 82 is less than +/- the reporting limit.

9.10.4. Method 6020 does not place any acceptance criteria on ICSA, but only requires that it be analyzed.

9.10.4.1. For standard work, the ICSA concentration of non-spiked analytes should be less than two times the reporting limit to continue analysis. If it exceeds this limit, an NCM should be filed to explain the reason. Acceptable narrations include:

- All associated samples have high levels of the analyte (greater than 10x the level found in the ICSA)
- All associated samples are non-detect for the analyte.

Additional corrective actions include rerunning, repouring, and remaking and rerunning the ICSA.

9.10.4.2. DOD/DOE QSM 5.1 requires that the absolute value of the concentration of non-spiked analytes be less than ½ the reporting limit/LOQ for a given analyte. Refer to Section 9.10.4.1 for corrective action.

9.10.5. The acceptance criteria for all of the methods does not apply when trace impurities are validated by the Certificate of Analysis for the ICSA solution that cause the non-spiked analytes to show positive concentrations outside of acceptance criteria. In such a case, a NCM is required that identifies the impurities and their certified concentrations in the standard.

9.10.6. ICSAB (interference check sample AB) is the same as ICSA, but all of the non-interfering analytes are spiked into the ICSA at the concentrations shown in Table 6.

9.10.7. The ICSAB results for the low level (non-interfering) analytes must fall within 80 – 120% (79.5 – 120.49 with rounding) of the true value. If the ICSAB does not meet these criteria for any analyte, the analyst should determine the cause and proceed with corrective action. Acceptable corrective actions include rerunning the ICSAB, repouring and rerunning the ICSAB, or remaking and reanalyzing the ICSAB. Alternatively, analysis may continue for those analytes which meet the acceptance criteria in the ICSAB. The analyst may also recalibrate and rerun the ICV/ICB and other QC necessary for the run.

9.10.8. For Method 6020A verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the ICSA and ICSAB.

9.11. Dilution test (Serial Dilution) – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix.

9.11.1. One sample per preparation batch must be processed as a dilution test.

9.11.2. The test is performed by running a sample at a 5X (1+4) dilution.

9.11.3. Samples identified as field blanks cannot be used for dilution tests.

9.11.4. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 100X the MDL.

9.11.4.1. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.12. Post-digestion spikes: One sample in every batch may be spiked with all of the elements of interest at the instrument following sample preparation. The matrix spiking solution is generally used as the post-digestion spike resulting in the same concentration spike level as the matrix spikes.

9.13. Internal standards are added continuously to all analyses in an analytical run. The intensities must be monitored for every analysis.

9.13.1. For method 6020, the internal standard (IS) intensities of the ICV/CCVs and ICB/CCBs should be within 80 – 120% recovery relative to the internal standard intensities of the calibration blank. If the internal standards for the

ICV/CCV/CCB exceed the criteria, terminate the analysis, correct the problem, and reanalyze affected samples.

IS intensities in samples must be within 30 to 120% of the IS intensities for the initial calibration blank, although suppression influencing sample results could be expected at levels less than 60-70% recovery. If the 30 – 120% criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. Narrate any findings.

9.13.2. For method 6020A, the internal standard (IS) intensities in field and QC (including ICV/CCV and ICB/CCB) samples must be greater than 30% and less than 150% of the IS intensities for the initial calibration standard. If the intensity in the sample is less than 30% of the intensity in the initial calibration standard, ensure that the instrument has not drifted by checking the intensities in the nearest calibration blank. If low intensities are also present in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, and reanalyze the affected samples. If drift has not occurred, remove matrix effects by diluting the sample. Dilute at least 5x and reanalyze. Repeat if the first dilution is not sufficient to ameliorate the matrix effects. Narrate any findings.

9.13.3. For Method 200.8 the internal standard (IS) intensities in field and QC samples must be within 60-125 % of the IS intensities for the initial calibration blank. If internal standards for the ICV/CCV/CCB exceed the 60-125% criteria, terminate the analysis, correct the problem, recalibrate, and reanalyze the affected samples, Otherwise, if the 60 – 125% criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. Narrate any findings.

9.13.4. For DoD/DOE QSM work the internal standard (IS) intensities in field and QC (including ICV/CCV and ICV/CCB) samples must be greater than 30% and less than 150% of the IS intensities for the initial calibration standard. If the recoveries are acceptable for the QC samples but not the field samples, the field samples may be considered to suffer from a matrix effect. Remove matrix effects by diluting the sample at least 5x and reanalyze. Repeat if the first dilution is not sufficient to ameliorate the matrix effects. Narrate any findings.

9.13.5. Table 7 shows the composition of the internal standard A solution. This standard is diluted further at 2X or 3X prior to analysis.

9.13.6. The internal standard raw intensity counts must be printed on the raw data. The percent recovery of each internal standard relative to the calibration blank or the blank associated with a previous re-slope is printed on the raw data.

9.14. High Level Continuing Calibration Verification (High Level CCV) – This standard is used to verify the instrument's dual detector calibration.

9.14.1. The High Level CCV is analyzed at the beginning of the run to demonstrate whether the instrument is accurately measuring concentration at concentrations near the upper limit of the linear range. If the values are greater than ± 20%D a new dual detector calibration may be required.

9.14.2. Should values in the High Level CCV exceed the ± 20%D criteria, only values less than or equal to the nominal values of the mid-level continuing calibration may be reported.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to Policy CA-Q-P-003, "Calibration Curves and Selection of Calibration Points."

10.2. Instrument start-up

10.2.1. Follow the instrument start-up procedure outlined in the Perkin Elmer Elan 6000 Operator's Manual and Agilent 7700X Operator's Manual.

10.3. Instrument Tuning

10.3.1. Agilent 7700X

10.3.1.1. A US EPA Tune Check Sample must be performed in the Helium [He] mode and Hydrogen [H2] mode. The US EPA Tune Check Sample contains Ba, Be, Ce, Co, In, Li, Mg, Pb, Rh, Tl, U and Y at 10 ppb. In the [He] mode the RSD% for mass 24, 59, 115, and 208 must be below 5%. In the [H2] mode mass 9, 24, 59, 115, and 208 must be below 5%.

10.3.1.2. Resolution must be < 0.9 amu full width at 10% peak height. The Agilent 7700X resolution is maintained at approximately 0.75 amu.

10.3.1.3. Mass calibration must be within ± 0.1 amu from the true value.

10.4. Initial calibration must be performed daily and each time the instrument is set up.

10.4.1. Calibration consists of a blank and a single calibration standard. Table 2 shows the concentration of all analytes in the calibration standard. Use the average of three integrations for both calibration and sample analyses. Initial calibration acceptance is based upon the ICV falling within +/- 10% recovery from the true value.

10.5. Following calibration, an ICV/ICB pair is analyzed to confirm the accuracy of the calibration (section 9.5).

10.6. Low level check standards are analyzed to confirm the sensitivity of the analytical run at the reporting limit for the samples (section 9.7).

10.7. For DOD/DOE, linear range standards are analyzed after the completion of the calibration as per section 9.8.

10.8. Continuing calibration verifications, CCV/CCB pair, are analyzed every ten (10) samples and at the end of the analytical sequence as per section 9.6.

10.9. For Method 6020A, the low level continuing calibration verification (LLCCV) standard should also be analyzed at the end of each analysis batch. A more frequent LLCCV analysis, i.e., every 10 samples may be necessary if low-level sample concentrations are anticipated and the system stability at low end of the calibration is questionable. The acceptance criteria for the LLCCV standard should be ± 30% of its true value.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. All samples require a preparation prior to analysis depending on the matrix being evaluated. Preparation of each batch requires a method blank, LCS, and matrix spikes and/or matrix spike duplicates. The concentration of analytes in the LCSs and matrix spikes are listed in the individual preparation methods.

11.2.1. For method 6020 and 6020A soils analysis, EPA method 3050B is used to

prepare all samples (SOP WS-IP-0002).

11.2.2. For method 200.8, 6020, 6020A aqueous samples, and TCLP leachate samples are prepared using the total recoverable digestion procedure, method 3005A (WS-IP-0001).

11.2.3. For method 200.8, 6020, 6020A dissolved samples, and STLC are prepared using the matrix matching procedure (SOP WS-IP-0008).

11.3. Before setting up an analytical run, read the QAS for all of the lots being analyzed. The QAS is created by the project managers and defines any special criteria required for client specific analyses.

11.4. An instrument backlog is generated from the TALs data system. This backlog lists the Job numbers that were assigned to each batch of samples. The backlog also shows project due date, number of samples and sample matrix. The instrument backlog aids the analyst in deciding the lot IDs to analyze on a given day.

11.5. An analytical run will consist of all customer samples and quality control samples analyzed under a daily initial calibration. Each new initial calibration will begin a new analytical run.

11.6. Using utility software, the analyst can generate a sample run log directly from the instrument software.

11.7. In order to use the ICP-MS data upload program into TALs, the following naming conventions must be followed:

11.7.1. Samples are identified by the 5 character work order number.

11.7.2. Quality control parameters have a prefix or suffix before or after their sample ID.

Parameter	Prefix/Suffix
Method Blanks	MB
LCS	LCS
LCS Duplicate	LCSD
Matrix Spike	MS
Matrix Spike Duplicate	MSD
Sample Duplicate	DU
Serial Dilution	SD
Post-digestion Spike	PDS

11.8. Set-up

11.8.1. Open a new dataset using the date and instrument in the title. For instance the

first run (A) on instrument 2 on JAN 1, 2013 would be 070113A2.

11.8.2. Open the appropriate method if one already exists or create a new one for the analytes to be quantitated in the run. A method with all of the analytes has been generated and can be modified to the specific analytes required throughout any given analytical run.

11.8.3. All masses which could affect data quality should be monitored to determine potential interferences. Secondary masses are evaluated per SW 846 protocols.

11.8.4. Internal standards are added to all standards and samples by the instrument peristaltic pump and a mixing tube prior to analysis.

11.8.5. Use of an existing autosampler table is suggested. A read delay of 45 to 60 seconds is used between all analyses.

11.9. The order of analysis for the initial QC samples and calibration should be:

1. Rinse
2. CAL Blank
3. STD1 (Calibration Standard)
4. ICV (Second source, must be ± 10% of true value)
5. ICB
6. LLSTD1
7. LLSTD2, etc
8. ICSA (Interference check solution with Bromine, for Selenium. ± RL for Se.)
9. ICSAB (Interference check solution, ± 20% of true value)
10. LRSTD1
11. LRSTD2, etc
12. Rinse (up to 3 rinses)
13. CCV1
14. CCB1
15. CCV2
16. CCB2

11.10. To continue the analytical run, add an additional 10 runs followed by CCV/CCB, and repeat for up to 24 hours.

11.11. For standard jobs, samples must be diluted and reanalyzed for any elements which are present in concentrations exceeding 90% of the upper linear range limit as determined in quarterly linear range studies. For DOD/DOE jobs, samples must be diluted and

reanalyzed for any elements which are present at concentrations exceeding DOD/DOE linear range (high level CCV).

11.12. If the sample (B) analyzed immediately after a sample (A)with detections greater than the instrument linear range has detections for the same elements, the following sample (B) must be reanalyzed to demonstrate whether the detections are the result of carryover or actually present.

12. CALCULATIONS/DATA REDUCTION

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left(\frac{Found(ICV)}{True(ICV)} \right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left(\frac{Found(CCV)}{True(CCV)} \right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \times \left(\frac{SSR - SR}{SA} \right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100 \left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2} \right)} \right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V1 = Final volume in liters after sample preparation

V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg\ /\ Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V = Final volume in liters after sample preparation

W = Weight in Kg of wet sample digested

S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100 \times \left(\frac{Found(LCS)}{True(LCS)} \right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%\ \text{Difference} = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)

S =Dilution test result (Instrument reading $\times$ 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported in accordance with the TestAmerica Sacramento significant figure policy (WS-PQA-004).

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits (Method 6020) or the static recovery limits of 85-115% (Method 200.8).

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste

disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Acid waste consisting of unused sample, analyzed sample solution and rinse solution. This is collected in four-gallon plastic carboys. When full or after no more than one year, the waste is dumped into an acid waste drum in the H-3 closet. When full to no less than one inch and no more than four inches of the top, or after no more than 75 days, the drum will be transferred to the waste collection area for disposal.

15.2. Miscellaneous disposable glassware, plastic vials with snap top caps, autosampler tubes and similar solid waste. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, September, 1994.

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update IV, Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, February 2007.

16.3. Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111), Method 200.8, Determination of Trace Elements in Waters by Inductively Coupled Plasma – Mass Spectrometry, Revision 5.4, 1994

16.4. EPA Method 200.8 EMSL office of Research & Development, Cincinnati, OH (Draft

16.5. Agilent Series 7700 ICP-MS Users Manual

16.6. WS-PQA-021, “Federal Program Requirements”

16.7. Recommended ICP-MS References

16.7.1. K. E. Jarvis, A. L. Gray, and R. S. Honk, Handbook of Inductively Coupled Plasma Mass Spectrometry; Chapman & Hall, New York 1992.

16.7.2. A. R. Date and A. L. Gray, Applications of Inductively Coupled Mass Spectrometry, Chapman & Hall, New York 1989. ISBN # 0-41201721-0

17. METHOD MODIFICATONS

Deviations from Source Method and Rationale

17.1. As a conservation of space, the raw data will only contain the mean and standard deviation results on the standard instrument summary report. As long as the standard deviation data is shown, the analyst can make decisions on replicate quality based on the instrument summary report.

17.2. This SOP may be used to analyze for elements not included in Method 6020, as long as appropriate QC samples spiked with the non-routine analytes are run with acceptable results. Clients should be notified that we do not carry method performance indicators for these analytes.

17.3. For reporting consistency, an ICB/CCB is acceptable if the result is <RL. Method 6020 states that the results of the calibration blank (CCB) are to be less than 3x the IDL. If not, terminate the analytical sequence, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica Sacramento has adopted an absolute control limit of +/- RL from zero for calibration blank criteria for standard operations. Exceptions are defined in this SOP.

17.4. The ICSA and ICSAB are being made as needed with an expiration date of 5 months or less depending on stock solutions expiration dates. Method 6020 states that the ICSA and ICSAB need to be prepared fresh weekly from ultra pure reagents. TestAmerica Sacramento has adopted to make these standards as needed due to the stability of the purchased stock solutions (manufacturer's expiration date or <1 year).

18. ATTACHMENTS

Tables referenced in the body of the SOP

18.1. Table 1 -Standard Target Analyte List

18.2. Table 2 -Calibration and Calibration Check Standard Preparation

18.3. Table 3 -Initial Calibration Verification Standard

18.4. Table 4 -Low Level Check Intermediate Standard Preparation

18.5. Table 5 -Interference Check Solution A

18.6. Table 6 -Composition of the ICSAB Standard

18.7. Table 7 -Internal Standard A Stock

18.8. Table 8 -Common Molecular Ion Interferences in ICPMS

18.9. Table 9 -Recommended Analytical Isotopes and Additional Masses (Elements of Interest)

18.10. Table 10 -Recommended Analytical Isotopes and Additional Masses (Rare Earth Elements and other Elements)

18.11. Table 11 - Elemental Equations used in Calculations

19. REVISION HISTORY

19.1. WS-MT-0001, Revision 4.3, Effective 11/21/2017

19.1.1. Added to Section 11.11, "For DOD/DOE jobs, samples must be diluted and reanalyzed for any elements which are present at concentrations exceeding DOD/DOE linear range (high level CCV).

19.1.2. Added Section 11.12, "If the sample (B) analyzed immediately after a sample (A)with detections greater than the instrument linear range has detections for the same elements, the following sample (B) must be reanalyzed to demonstrate whether the detections are the result of carryover or actually present."

19.1.3. Editorial changes.

19.2. WS-MT-0001, Revision 4.2, Effective 10/19/2017

19.2.1. Removed Section 6.1.1, "Perkin Elmer/Sciex ELAN 6000 ICP-MS or equivalent, utilizing Elan software, version 2.3.2 or equivalent."

19.2.2. Revised Section 9.10.4.2 to "DOD/DOE QSM 5.1 requires that the absolute value of the concentration of non-spiked analytes be less than ½ the reporting limit (LOQ) for a given analyte. Refer to Section 9.10.4.1 for corrective action."

19.2.3. Added Section 9.13.4, "For DoD/DOE QSM work the internal standard (IS) intensities in field and QC (including ICV/CCV and ICV/CCB) samples must be greater than 30% and less than 120% of the IS intensities for the initial calibration standard. If the recoveries are acceptable for the QC samples but not the field samples, the field samples may be considered to suffer from a matrix effect. Remove matrix effects by diluting the sample at least 5x and reanalyze. Repeat if the first dilution is not sufficient to ameliorate the matrix effects. Narrate any findings."

19.2.4. Removed all references to the Perkin Elmer/Sciex Elan model 6000.

19.2.5. Editorial changes.

19.3. WS-MT-0001, Revision 4.1, Effective 08/18/2017

19.3.1. Section 9.2.4, removed “including QSM version 4.2 and AFCEE version 4.0.”

19.3.2. Section 9.6.4, revised to read “DOD/DOE QSM: The ICB and CCB results for all analytes must be less than less than ½ the reporting limit for a given analyte.”

19.3.3. Section 9.9, deleted “(QSM, AFCEE)”.

19.3.4. Deleted Section 16.7, “Department of Defense Quality Systems Manual for Environmental Laboratories (QSM), Version 4.2, October 2010.

19.3.5. Added Section 16.7, “WS-PQA-021, ‘Federal Program Requirements’”.

19.3.6. Deleted Section 16.8, “AFCEE QAPP, Version 4.0, February 2005.

19.3.7. Added Section 16.8, “Recommended ICP-MS References”.

19.3.8. Revised Section 17.1 to read, “As a conservation of space, the raw data will only contain the mean and standard deviation results on the standard instrument summary report. As long as the standard deviation data is shown, the analyst can make decisions on replicate quality based on the instrument summary report.”

19.3.9. Editorial changes.

19.4. WS-MT-0001, Revision 4.0, Effective 11/04/2016

19.4.1. Section 7.4, changed “…add 40 mL of concentrated HCl …” to “…add 80 nil of concentrated HCl....”

19.4.2. Removed Section 9.6.4 as references to AFCEE are no longer relevant.

19.4.3. Editorial changes.

19.5. WS-MT-0001, Revision 3.9, Effective 09/09/2015

19.5.1. Section 9.13.1, changed to include ICV/CCV and ICB/CCB criteria for internal standards (formerly section 9.13.4).

19.5.2. Section 9.13.2, changed the first two sentences to read, “For method 6020A, the internal standard (IS) intensities in samples must be greater than 30% and less than 150% of the IS intensities for the initial calibration standard. If the intensity in the sample is less than 30% of the intensity in the initial calibration standard, ensure that the instrument has not drifted by checking the intensities in the nearest calibration blank.”

19.5.3. Section 9.13.3, changed the first two sentences to read, “For Method 200.8 the internal standard (IS) intensities in field and QC samples must be within 60-125 % of the IS intensities for the initial calibration blank. If internal standards for the ICV/CCV/CCB exceed the 60-125% criteria, terminate the analysis, correct the problem, recalibrate, and reanalyze the affected samples,”

19.5.4. Section 9.13.4 and 9.13.4.1 – removed. Incorporated into 9.13.1. These revisions are to be congruent with current TestAmerica interpretation of IS requirements for ICPMS methods.

19.5.5. Added references to DOD/DOE in place of DOD where applicable.

19.6. WS-MT-0001, Revision 3.8, Effective 12/12/2014

19.6.1. Added EPA Method 200.8 to Scope and Application Sections 1,1 and 1,3.

19.6.2. Section 6.4 – Changed Argon gas to Hydrogen gas.

19.6.3. Added Section 9.13.3 – “For Method 200.8 the internal standard (IS) intensities in the samples must be within 60-125 % of the IS intensities for the initial calibration blank. If the 60 – 125% criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. Narrate any findings.

19.6.4. Added Section 10.3.2 for Agilent 7700X instrument parameters.

19.6.5. Added the following paragraph following Section 9.5 -“For method 200.8 analysis, an MS is prepared and analyzed for every 10 field samples, i.e., for a batch of 10 samples or fewer, one MS is required, for batches of 11 -20 samples, 2 MS are required. Compare the percent recovery to the method limits of 70-130%.

19.6.6. Added the following to the end of Section 13.3.2 – “or the static recovery limits of 85-115% (Method 200.8)“

19.6.7. Editorial changes..

19.7. WS-MT-0001, Revision 3.7 Effective 08/29/2014

19.7.1. Added Section 9.10.8, “For Method 6020A verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the ICSA and ICSAB.)

19.7.2. Editorial changes.

19.8. WS-MT-0001, Revision 3.6, Effective 09/30/2013

19.8.1. Revised Section 9.5.3, 9.7.3 (number changed from 9.7.2) and 9.9.4 to reflect how analytical runs and decision making proceeds.

19.8.2. Removed Section 10.4 (Screening for Internal Standards)

19.8.3. Added Section 11.11 (regarding dilutions for analytes > linear range study)

19.8.4. Added Section 9.14 (High Level CCV) to reflect current practice to improve data quality.

19.8.5. Added references to method 6020A in Sections 1.3, 11.2, and 16.2

19.8.6. Added reference to preventive maintenance schedules in Section 6, and added Table 12 (Preventative Maintenance).

19.8.7. Deleted references to AFCEE 3.1 in Sections 9.3

19.8.8. Inserted Section 9.4 (low level QC Check sample)

19.8.9. Inserted Section 9.8.3 regarding the low-level standard frequency and criteria for method 6020A.

19.8.10. Sections 9.6.3, 9.8.4, 9.10.4.1, and 9.10.7 Clarified the corrective action procedures.

19.8.11. Section 10.3, removed elements not required in the 6020/6020A tuning solution.

19.8.12. Added Section 10.8, low-level calibration standard at the end of the run.

19.8.13. Section 11.4 through 11.7, minor edits to reflect procedure using TALS vs. the older LIMs.

19.8.14. Editorial changes.

19.9. WS-MT-0001, Revision 3.5, Effective 07/05/2013

19.9.1. Modified Section 10.3.3 to follow what is in the method regarding tuning.

19.9.2. Editorial changes.

19.10. WS-MT-0001, Revision 3.4, Effective 09/14/2012

19.10.1. Removed requirement for making ICSA and ICSAB standards weekly from

Section(s) 7.9 and 7.10.

19.10.2. Removed references to AFCEE 3.1.

19.10.3. Editorial changes.

19.11. WS-MT-0001, Revision 3.3, Effective 06/10/2011

19.11.1. Inserted Cd (mass 114) in Table 1.

19.11.2. Deleted reference to 6020A in Method heading and reference (Section 19.2).

19.11.3. Removed Section 6.5: Class A volumetric pipettes.

19.11.4. Section 9.11: replaced “must be spiked” with “may be spiked”.

19.11.5. Section 10.4.1: replaced “representative samples must be screened” with “it is recommended that soil samples be screened”.

TABLE 1			
Method 6020 Standard Target Analyte List			
Element	**Symbol**	**Aqueous RL mg/L**	**Soil RL mg/Kg**
Aluminum	Al	0.05	5.0
Antimony	Sb	0.002	0.2
Arsenic	As	0.002	0.2
Barium	Ba	0.001	0.1
Beryllium	Be	0.001	0.1
Boron	B	0.05	5.0
Cadmium	Cd (mass 111)	0.001	0.1
Cadmium	Cd (mass 114)	0.001	0.1
Calcium	Ca	0.05	30
Chromium	Cr	0.002	0.2
Cobalt	Co	0.001	0.1
Copper	Cu	0.002	0.2
Iron	Fe	0.05	5.0
Lead	Pb	0.001	0.1
Lithium	Li	0.01	1.0
Magnesium	Mg	0.05	5.0
Manganese	Mn	0.001	0.2
Molybdenum	Mo	0.002	0.2
Nickel	Ni	0.002	0.2
Phosphorous	P	0.05	20
Potassium	K	0.05	5.0
Selenium	Se (mass 82)	0.002	0.2
Selenium	Se (mass 78)	0.003	0.3
Silver	Ag	0.001	0.1
Sodium	Na	0.05	50
Strontium	Sr	0.005	0.5
Thallium	Tl	0.001	0.1
Tin	Sn	0.01	2.0
Titanium	Ti	0.005	0.5
Vanadium	V	0.01	1.0
Zinc	Zn	0.005	1.0

Table 2				
Calibration Standard and Continuing Calibration Verification Standard				**Final Volume = 500 mL**
Element	**Stock Standard ID**	**Stock Standard Concentration (mg/L)**	**Spike Volume (mL)**	**Final Concentration mg/L**
Aluminum	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Antimony	CPI-ICPMS-1B	5.0	5.0	0.050
Arsenic	CPI-ICPMS-1B	10	5.0	0.10
Barium	CPI-ICPMS-1B	10	5.0	0.10
Beryllium	CPI-ICPMS-1B	10	5.0	0.10
Boron	CPI-ICPMS-1B	50	5.0	0.10
Cadmium	CPI-ICPMS-1B	10	5.0	0.10
Calcium	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Chromium	CPI-ICPMS-1B	10	5.0	0.10
Cobalt	CPI-ICPMS-1B	10	5.0	0.10
Copper	CPI-ICPMS-1B	10	5.0	0.10
Iron	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Lead	CPI-ICPMS-1B	10	5.0	0.10
Lithium	CPI-ICPMS-1B	10	5.0	0.10
Magnesium	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Manganese	CPI-ICPMS-1B	10	5.0	0.10
Molybdenum	CPI-ICPMS-1B Spex ZCAL-34	10 20	5.0 2.5	0.20
Nickel	CPI-ICPMS-1B	10	5.0	0.10
Phosphorous	Spex ZCAL-34	1000	2.5	5.0
Potassium	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Selenium	CPI-ICPMS-1B	10	5.0	0.10
Silver	CPI-ICPMS-1B	5.0	5.0	0.050
Sodium	CPI-ICPMS-1B Spex ZCAL-34	10 1000	5.0 2.5	5.1
Strontium	CPI-ICPMS-1B	10	5.0	0.10
Thallium	CPI-ICPMS-1B	5.0	5.0	0.050
Tin	CPI-ICPMS-1B	10	5.0	0.10
Titanium	CPI-ICPMS-1B Spex ZCAL-34	10 20	5.0 2.5	0.20
Vanadium	CPI-ICPMS-1B	10	5.0	0.10
Zinc	CPI-ICPMS-1B	10	5.0	0.10
Sulfur	Spex ZCAL-34	1000	2.5	5.0
Carbon	Spex ZCAL-34	2000	2.5	10
Chloride	Spex ZCAL-34	7200	2.5	36

TABLE 3

Method 6020 Initial Calibration Verification Standard
Stock: CPI CALLAB-3 Spike Volume: 4.0 mL
Final Volume: 500 mL

Element	Concentration mg/L	Element	Concentration mg/L
Aluminum	0.8	Manganese	0.08
Antimony	0.04	Molybdenum	0.08
Arsenic	0.08	Nickel	0.08
Barium	0.08	Phosphorous	0.8
Beryllium	0.08	Potassium	0.8
Boron	0.8	Selenium	0.08
Cadmium	0.08	Silver	0.04
Calcium	0.8	Sodium	0.8
Chromium	0.08	Strontium	0.08
Cobalt	0.08	Thallium	0.04
Copper	0.08	Tin	0.08
Iron	0.8	Titanium	0.08
Lead	0.08	Vanadium	0.08
Lithium	0.08	Zinc	0.08
Magnesium	0.8		

Table 4				
Low Level Check Intermediate Standard - Method 6020			**Final Volume = 500 mL**	
Element	**Stock Standard ID**	**Stock Standard Conc'n (mg/L)**	**Spike Volume (mL)**	**Final Conc'n (mg/L)**
Aluminum	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Antimony	CALLAB-3	5.0	0.5	0.005
Arsenic	CALLAB-3	10	0.5	0.01
Barium	CALLAB-3	10	0.5	0.01
Beryllium	CALLAB-3	10	0.5	0.01
Boron	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Cadmium	CALLAB-3	10	0.5	0.01
Calcium	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Chromium	CALLAB-3	10	0.5	0.01
Cobalt	CALLAB-3	10	0.5	0.01
Copper	CALLAB-3	10	0.5	0.01
Iron	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Lead	CALLAB-3	10	0.5	0.01
Lithium	CALLAB-3 Single	100 1000	0.5 0.02	0.05
Magnesium	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Manganese	CALLAB-3	10	0.5	0.01
Molybdenum	CALLAB-3	10	0.5	0.01
Nickel	CALLAB-3	10	0.5	0.01
Phosphorous	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Potassium	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Selenium	CALLAB-3	10	0.5	0.01
Silver	CALLAB-3	5.0	0.5	0.005
Sodium	CALLAB-3 Single	100 1000	0.5 0.2	0.5
Strontium	CALLAB-3	10	0.5	0.01
Thallium	CALLAB-3	5.0	0.5	0.005
Tin	CALLAB-3 Single	100 1000	0.5 0.045	0.1
Titanium	CALLAB-3 Single	100 1000	0.5 0.02	0.05
Vanadium	CALLAB-3 Single	100 1000	0.5 0.045	0.1
Zinc	CALLAB-3 Single	100 1000	0.5 0.02	0.05

TABLE 5			
*** Method 6020 Interference Check Solution A** **Stock: Spex ZCAL-34 Spike volume: 10 mL** **Bromide 1000 mg/L stock Spike volume: 1.0 mL** **Final Volume: 100 mL**			
Element	**Concentration mg/L**	**Element**	**Concentration mg/L**
Br^-	100	P	100
Al	100	S	100
Ca	100	C	200
Fe	100	Cl^-	720
K	100	Mo	2.0
Mg	100	Ti	2.0
Na	100		

*** Make new standard weekly**

TABLE 6 Composition of the ICSAB Standard			
Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.05	Na	100.1
Al	100.1	Ni	0.10
As	0.10	Pb	0.10
B	0.10	Sb	0.05
Ba	0.10	Se	0.10
Be	0.10	Si	0.10
Ca	100.1	Sn	0.10
Cd	0.10	Sr	0.10
Co	0.10	Ti	2.0
Cr	0.10	Tl	0.05
Cu	0.10	U	0.10
Fe	100.1	V	0.10
K	100.1	Zn	0.10
Li	0.1	P	100
Mg	100.0	S	100
Mn	0.10	C	200
Mo	2.10	Cl-	720

Table 7			
Internal Standard A Stock - Method 6020			**Final Volume = 4000 mL**
Element	**Stock Standard Concentration (mg/L)**	**Spike Volume (mL)**	**Final Concentration (mg/L)**
Lithium	500	80	10
Scandium	1000	8.0	2.0
Germanium	10000	3.0	7.5
Indium	1000	3.0	0.75
Thullium	1000	2.0	0.5

TABLE 8[1]

COMMON MOLECULAR ION INTERFERENCES IN ICP-MS

Molecular Ion	Mass	Element Interferences[2]	Molecular Ion	Mass	Element Interferences[2]
BACKGROUND MOLECULAR IONS					
NH^+	15		$^{38}ArH^+$	39	
OH^+	17		$^{40}ArH^+$	41	
OH_2^+	18		CO_2^+	44	
C_2^+	24		CO_2H^+	45	Sc
CN^+	26		ArC^+, ArO^+	52	Cr
CO^+	28		ArN^+	54	Cr
N_2^+	28		$ArNH^+$	55	Mn
N_2H^+	29		ArO^+	56	
NO^+	30		$ArOH^+$	57	
NOH^+	31		$^{40}Ar^{36}Ar^+$	76	Se
O_2^+	32		$^{40}Ar^{38}Ar^+$	78	Se
O_2H_+	33		$^{40}Ar_2^+$	80	Se
$^{36}ArH^+$	37				
MATRIX MOLECULAR IONS – Chloride					
$^{35}Cl0^+$	51	V	$^{37}Cl0H^+$	54	Cr
$^{35}Cl0H^+$	52	Cr	$^{35}Cl0^+$	51	V
$^{37}Cl0^+$	53	Cr	$^{35}Cl0H^+$	52	Cr
$Ar^{35}Cl^+$	75	As	$Ar^{37}Cl^+$	77	Se
MATRIX MOLECULAR IONS – Sulfate					
$^{32}SO^+$	48		$^{34}SOH^+$	51	V
$^{32}SOH^+$	49		SO_2^+, S_2^+	64	Zn
$^{34}SO^+$	50	V, Cr			
$Ar^{32}S^+$	72		$Ar^{34}S^+$	74	
MATRIX MOLECULAR IONS – Phosphate					
PO^+	47		PO_2^+	63	Cu
POH^+	48				
ArP^+	71				
MATRIX MOLECULAR IONS – Group I, II Metals					
$ArNa^+$	63	Cu	$ArCa^+$	80	
ArK^+	79				
MATRIX OXIDES[3]					
TiO	62-66	Ni, Cu, Zn	MoO	108-116	Cd
ZrO	106-112	Ag, Cd			

[1] *Table reference from Method 200.8, Section 13.2.6*

[2] *Method elements or internal standards affected by the molecular ions.*

[3] *Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes be monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.*

TABLE 9 RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED [1]			
Isotope	Element of Interest	Isotope	Element of Interest
27	Aluminum [2]	80,**78**,**82**,**76**,**77**,74	Selenium
121,123	Antimony [2]	**107**,**109**	Silver [2]
75	Arsenic [2]	**23**	Sodium [2]
138,137,136,**135**,134,132,130	Barium [2]	203, **205**	Thallium [2]
9	Beryllium [2]	**51**,**50**	Vanadium [2]
114,112,**111**,110,113,116,106, **108**	Cadmium [2]	**66**, **67**, **68**	Zinc [2]
42,**43**,**44**,46,48	Calcium [2]	83	Krypton
52,**53**,**50**,54	Chromium [2]	72	Germanium
59	Cobalt [2]	139	Lanthanum
63,**65**	Copper [2]	140	Cerium
56,**54**,**57**,58	Iron [2]	129	Xenon
206,**207**,**208**	Lead [2]	**118**	Tin
24,**25**,**26**	Magnesium [2]	105	Palladium
55	Manganese [2]	47,**49**	Titanium
202,**200**,199,201	Mercury	125	Tellurium
98,96,92,**97**,94	Molybdenum	69	Gallium
58,**60**,62,**61**,64	Nickel [2]	35,37	Chlorine
39	Potassium [2]		

[1] *From Method 6020 CLP-M, Table 9*

[2] *Element approved for ICP-MS determination by SW846 Method 6020 CLP-M*

NOTE: Isotopes recommended for analytical determination are **bold**.

TABLE 10			
RECOMMENDED ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED			
Rare Earth Elements	**ICPMS Preferred Mass**	**Elemental Equations**	**Additional Masses**
Lanthanum	138.906		
Cerium	139.905		
Praseodymium	140.907		
Neodymium	141.908	-0.125266 * ^{140}Ce	142.910, 144.912
Samarium	151.920	-0.012780 * ^{157}Gd	144.912
Europium	152.929		
Gadolinium	157.924	-0.004016 * ^{163}Dy	156.934
Terbium	158.925		
Dysprosium	163.929	-0.047917 * ^{166}Er	
Holmium	164.930		
Erbium	165.930		
Thulium	168.934		
Ytterbium	173.939	-0.005935 * ^{178}Hf	171.937
Lutetium	174.941		
Other Elements			
Boron	11.009		
Calcium	43.956		
Cesium	132.905		
Gallium	68.926		
Germanium	71.922		
Gold	196.967		
Hafnium	177.944		176.944
Holmium	164.930		
Iridium	192.963		
Lithium	7.016		
Tungsten	183.951	-0001242* ^{189}Os	
Uranium	238.050		
Yttrium	88.905		
Zirconium	238.050		
Niobium	92.906		
Palladium	104.905		
Phosphorus	30.994		
Platinum	194.965		
Rhenium	186.965	-0.099379 * ^{189}Os	
Rhodium	102.905		
Rubidium	84.912		
Ruthenium	101.904	-0.045678 * ^{105}Pd	
Scandium	44.956		
Strontium	87.906		
Tantalum	180.948		
Tellurium	127.905	-0.072348 * ^{129}Xe	
Thorium	232.03		

TABLE 11
ELEMENTAL EQUATIONS USED TO CALCULATE RESULTS

Element	Elemental Equation	Note
Al	(1.000) (^{27}C)	
Sb	(1.000) (^{121}C)	
As	(1.000) (^{75}C) - (3.1278)[^{77}C) - (1.0177)(^{78}C)]	Correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank.
Ba	(1.000) (^{137}C)	
Be	(1.000) (^{9}C)	
Cd	(1.000) (^{111}C) - (1.073) [(^{108}C) - (0.712) (^{106}C)]	Correction of MoO interference. An additional isobaric elemental correction should be made if palladium is present.
Cr	(1.000) (^{52}C)	In 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank.
Co	(1.000) (^{59}C)	
Cu	(1.000) (^{63}C)	
Pb	(1.000) (^{206}C) + (1.000) (^{207}C) + (1.000) (^{208}C)	Allowance for isotopic variability of lead isotopes.
Mn	(1.000) (^{55}C)	
Mo	(1.000) (^{98}C) - (0.146) (^{99}C)	Isobaric elemental correction for ruthenium.
Ni	(1.000) (^{60}C)	
Se	(1.000) (^{82}C)	Some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction.
Ag	(1.000) (^{107}C)	
Tl	(1.000) (^{205}C)	
Th	(1.000) (^{232}C)	
U	(1.000) (^{238}C)	
V	(1.000) (^{51}C) - (3.127) [(^{53}C) - (0.113) (^{52}C)]	Correction of chloride inference with adjustment for Cr53. Cl0 51/53 ratio may be determined from the reagent blank.
Zn	(1.000) (^{66}C)	

* Method elements or internal standards affected by the molecular ions.

Title: Inductively Couple Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW-846 Methods 6010B & 6010C and EPA Method 200.7 [Method 6010B, 6010C and 200.7]

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of trace metals by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) using EPA Method 6010B and 6010C and EPA Method 200.7.

1.2. Table 2 lists elements currently approved under NELAC standards for analysis by methods 6010B, 6010C and 200.7. Additional elements may be analyzed under these methods provided that the method performance criteria presented in Section 13 are met.

1.3. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. See Table 2 for Reporting Limits (RLs).

1.4. Methods 6010B and 6010C are applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, tissues, biological materials, and TCLP, STLC, and SPLP leachates/extracts. All matrices require digestion prior to analysis with the exception of STLC extracts. All samples (with the exception of STLC extracts) are digested by method 3005A/3010A to accommodate matching of matrix in the calibration standards. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data. In addition, digested samples that contain tin at concentrations greater than 1% should be prepared again using <1 g of sample weight.

1.5. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water and waste water. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although not required, all samples will be digested by method 3005A to accommodate matching of the matrix in the calibration standards. Silver concentrations must be below 0.1 mg/L in aqueous samples and 50 mg/Kg in solid matrix samples.

1.6. This SOP is not applicable for the analysis of drinking waters.

1.7. When undertaking projects for Department of Defense (DoD) or the Department of Energy, the relevant criteria in QA Policy WS-PQA-021 “Federal Program Requirements” must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. This method describes multi-elemental determinations by ICP-AES using a simultaneous optical system with axial and radial viewing of the plasma. The

instrument measures characteristic emission spectra by optical spectrometry. Samples are introduced via a nebulizer and the aerosol that is produced is transported to the plasma torch where excitation occurs. Emission specific spectra are produced by radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are measured by a charge-coupled device (CCD) detector(s). The photocurrents from the CCDs are processed and controlled by a computer system.

2.2. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

2.3. The possibility of additional interferences should also be recognized and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

2.4. Refer to the appropriate SOPs for details on sample preparation methods.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP.

4.2. Spectral interferences are caused by several causes.

4.2.1. Overlap of a spectral line from another element.

4.2.2. Unresolved overlap of molecular band spectra.

4.2.3. Background contribution from continuous or recombination phenomena.

4.2.4. Stray light from the line emission of high concentration elements.

4.3. These interferences in Section 4.2 should be measured and evaluated after scans of analyte wavelengths and their adjacent sides have been performed.

4.4. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.5. Inter-element correction factors (IECs) or other instrument correction routines are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element correction factors must be applied to the analyte to remove the effects of these unwanted emissions.

4.6. Physical interferences are generally considered to be effects associated with sample transport and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high total dissolved solids (TDS) or high acid concentrations. If physical interferences are present, use of an argon humidifier, peristaltic pump, mass flow controller, internal standardization, and/or a high solids nebulizer can reduce the effect.

4.7. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique, but if observed can be minimized by buffering the sample, matrix matching, or standard addition procedures.

4.8. Memory interferences result when analytes from a previous sample carryover into a new sample. Theses interferences occur from sample buildup onto and release from sample introduction components such as the autosampler sipper, nebulizer, sample capillaries, spray chamber, plasma injector, and torch. The memory effect is element and matrix dependent. It may result from the analysis of high analyte concentrations or the uptake and action of non-target matrix compounds. Analysts should recognize memory effects and re-analyze samples when necessary.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide satisfactory protection. Additional PPE (personal protective equipment) such as goggles or a face shield and apron are required when transferring instrument waste containers.

5.1.2. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.3. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.4. This instrument produces RF energy. People with pacemakers should not go near the instrument while it is in operation.

5.1.5. The ICP plasma emits strong UV light and is harmful to vision. Anyone around the instrument must avoid looking directly at the unshielded plasma.

5.1.6. Some extractions and digestions use hydrofluoric acid, which means that some samples to be analyzed are in an HF solution. The labels for all such extracts and digestates will be highlighted in yellow. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S staff and the local supplement to the Corporate Safety Manual for guidance. Anyone

working with HF must receive special training before starting work. Staff members who work around HF should also receive this training. HF acid solutions may not be used for any purposes except as prescribed in this or other SOPs for this laboratory. Processes involving HF acid are classified as high-risk activities. Personnel involved must wear a face shield in addition to safety glasses or goggles.

WARNING: The laboratory's Emergency Response Team must be activated for any suspected exposure to HF liquid or fumes. After local emergency response, the victim will be transported to the UCD Medical Center Emergency Room.

Appendix 4 is a detailed first aid plan for working with HF.

5.1.6.1. 2.5% calcium gluconate gel should be applied copiously and gently massaged into burn sites after rinsing under cold running water for one minute to remove HF acid from the surface of the skin. The person massaging should wear appropriate protective gloves. If the victim is conscious, they should consume small quantities of calcium or magnesium containing liquids, such as milk, Maalox, or Mylanta. If the burn site is too large to massage with calcium gluconate gel, cover it with gauze soaked in an Epsom salt solution (1 cup to 1 quart of cold water). Keep the wraps saturated with the Epsom salt solution.

5.1.6.2. Some metals react with HF to produce flammable hydrogen gas.

5.1.6.3. Glass reacts with HF to produce toxic silicon tetraflouride.

5.1.6.4. HF is classified as a poison, and must be stored in a locked cabinet when not in use.

5.1.6.5. Whenever HF is in use in a laboratory, a decontamination solution will be prepared before starting work. Take a small bucket (2-3 gallons) and fill it about 2/3-3/4 full with an Epsom salts solution. Before removing gloves or handling anything else that HF on the gloves might be transferred to, carefully dip one hand at a time into the Epsom salts solution for a few seconds. Do not put the hand any deeper than necessary to bring the solution level to within 1-2 inches of the glove cuff. The Epsom salts solution will be prepared each day prior to beginning work with HF, and disposed at the end of the day down the laboratory sink.

5.2. Primary Material Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review

the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid (1)	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrofluoric Acid (1)	Poison Corrosive Dehydrator	3 PPM - TWA	Corrosive to the skin and eyes. Contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. Severely corrosive to the respiratory tract. Permanent eye damage may occur. **THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.**
Triton X-100 (alkyl aryl polyether alcohol)	Irritant	1 PPM – TWA 5 PPM – STEL	Irritates the respiratory tract, causes coughing and shortness of breath. Can cause severe eye irritation with inflammation, swelling and chemical burns to cornea.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. A Thermo 6500 Duo ICP utilizing iTEVA version 2.4.0.8 software, with radio frequency generator, and computer controlled pneumatic mass flow controllers and multi-channel peristaltic pump. A concentric nebulizer and cyclonic spray chamber are included in the sample introduction system.

6.2. Argon gas supply, high purity grade (99.99%).

6.3. Nitrogen gas.

6.4. Compressed air.

6.5. Water re-circulator / chiller.

6.6. Adjustable air displacement pipettes.

6.7. Class A glass volumetric pipettes.

6.8. Class A volumetric flasks.

6.9. Autosampler tubes, 13 x 100 mm polystyrene or polypropylene.

7. REAGENTS AND STANDARDS

7.1. Use reagent grade chemicals in all tests. "Certificates of Analysis" should be supplied with all chemicals purchased. If not supplied, contact the vendor. When received, label the certificate and the reagent container with the receipt date. Reagent containers also need to be labeled with the opened and expiration dates.

7.2. Reagent water is produced by a Millipore nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.3. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.4. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.5. Instrument rinse: Add 2000 mL of reagent water to the 4000 mL rinse container. Add 80 mL of concentrated HNO_3 and 200 mL of concentrated HCl and dilute to volume with reagent water.

7.6. Dilution Solution: Add 800 mL of reagent water to the 1000 mL dilution container. Add 20 mL of concentrated HNO_3 and 50 mL of HCl and dilute to volume with reagent water.

7.7. Stock standards are purchased as custom multi-element mixes and as single-element solutions. These must be plasma grade standards.

7.7.1. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles.

7.7.2. Standards containing silver must be protected from light. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer.

7.7.3. If no expiration date is provided, the stock solutions may be used for up to one year from opening and must be replaced sooner if verification from an independent source indicates a problem.

7.8. The LLICV intermediate standard is made completely from 1000 mg/L or 10,000 mg/L single source stock standards. Add 50 mL reagent water and 2.0 mL HNO_3 to a 100

mL volumetric flask. Table 7 shows the spiking levels of each stock used to make the LLICV intermediate standard. Each is spiked with the appropriate sized adjustable air displacement pipette. Dilute to volume with reagent water. The intermediate standard expires 6 months after it is made.

7.9. Linear Range Standard (LRS): Add 50 ml of reagent water to a 100 mL volumetric flask. Add 5.0 mL of concentrated HCl and 2.0 mL of concentrated HNO_3. Add mixed and single source standards as defined by Table 11. Dilute to volume with reagent water. The LRS expires 6 months after it is made.

7.10. Triton X-100 (alkyl aryl polyether alcohol).

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation but must be stored at 4 ± 2°C until the time of preparation.

9. QUALITY CONTROL

9.1. Batch - A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards. A batch must be prepared within the same time frame. A method blank (MB) and a laboratory control sample (LCS) or duplicate control sample (LCS/LCSD) must be prepared as a part of every batch. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/MSD), or in some instances a sample/sample duplicate. For method 200.7, an MS/MSD must be prepared for every ten samples within a batch. An analytical batch must include all QC samples, however they do not contribute to the maximum of 20 samples. Refer to the Quality Program document (WS-PQA-0003) for more details.

9.2. One method blank (MB) must be prepared for every 20 samples. A method blank consists of reagent water processed through all of the steps, and at the same time as the associated samples. If a method blank exceeds ± the reporting limit for a given analyte then the samples associated with that batch must be re-prepared. The exception is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo. For

samples requiring laboratory filtration and preservation, the method blank must also be filtered and preserved. Refer to the Quality Program document (WS-PQA-0003) for more details.

9.2.1. For samples analyzed under the DoD QSM, the results of the method blank must be ≤ ½ the RL for a given analyte. The same exceptions apply as stated above.

9.3. A laboratory control sample (LCS) must be prepared for every 20 samples. A LCS consists of reagent water spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-prepared except when a LCS exhibits high recovery. For such a case, those samples with analyte concentrations less than the reporting limit can be reported. All corrective actions must be documented on a Non-conformance memo. Refer to the Quality Program document (WS-PQA-0003) for more details.

9.3.1. For samples analyzed under QSM, there is no exception for reporting "ND" samples for a high LCS.

9.3.2. Random marginal exceedances: For analyte lists of 11-30 elements, 1 LCS random marginal exceedance is allowed. In this case the LCS must be within 4 standard deviations of the mean, and control charts must be evaluated to assure that the marginal exceedence is random, as defined in Policy WS-PQA-0003.

9.3.3. The laboratory analyzes a limit of quantitation or LOQ sample quarterly as part of DOD/DOE requirements. For method 6010C the LOQ sample is also used as the digested low level quantitation check standard (LLCS) and must be within ± 30% of the true values.

9.4. A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair must be prepared with every process batch of similar matrix, not to exceed twenty (20) samples. MS/MSD pairs are aliquots of a selected field sample spiked with all of the analytes of interest at known concentrations. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Re-extraction of the blank, LCS, selected field samples, and the MS/MSD may be required after evaluation and review of the MS/MSD results.

9.5. A duplicate control sample (LCS/LCSD) may be substituted when insufficient volume is provided to process a sample/sample duplicate or MS/MSD pair, and batch precision is required by the client or program. The LCS and LCSD are evaluated independently for acceptance. Refer to the Quality Program document (WS-PQA-0003) for more details.

9.5.1. Samples identified as field blanks, equipment blanks, or trip blanks should not be used for MS/MSD analysis.

9.6. Initial Calibration Verification (ICV/ICB) – Calibration accuracy is verified by analyzing a second source standard (initial calibration verification standard) immediately upon completion of instrument calibration. This standard must be at a concentration different from that used to calibrate the instrument and different from the CCV standard. The ICV must fall within ± 10% of the true value of the standard solution for method 6010B/6010C and ± 5% for Method 200.7. An ICB (initial calibration blank) prepared the same as the calibration blank must be analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within ± the reporting limit from zero. If either the ICV or ICB fail to meet acceptance criteria the analysis must be terminated, the problem corrected, and the instrument re-calibrated.

9.6.1. For all ICVs, the RSD between the two replicates run for each sample must be less than 5%. If this fails for any analyte, rerun the QC and all the samples affected by the out of control event.

9.6.2. For QSM, the ICB and CCB results for all analytes must be less than 1/2 the LOQ.

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard (continuing calibration standard) after every 10 samples. Ten samples include any analysis that registers with a result, even if not used. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within ±10% of the true value for that solution for methods 6010B, 6010C, and 200.7. A CCB (continuing calibration blank) is analyzed immediately following each CCV. The CCB result must fall within ± RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the possible cause of failure should be investigated, and all samples analyzed since the last acceptable CCV and or CCB must be reanalyzed.

9.8. Lower limit of quantitation check sample (LLICV): Following analysis of the ICV/ICB pair the LLICV is analyzed spiked with concentrations of each analyte of interest at or sometimes below the reporting limit. The percent recovery of the LLICV results must be ± 50% of the true concentration. If the recovery is out of control, rerun the LLICV once. If still outside of control limits resolve the problem and if necessary re-calibrate the instrument. If some elements fail to meet criteria for this sample the run can be continued and sample results used for elements that do pass criteria.

9.8.1. Since reporting limits may vary for some programs requiring a LLICV sample, the analyst may need to make up different concentrations.

9.8.2. For QSM, the LLICV sample criterion is ± 20%.

9.8.3. For method 6010C the acceptance limits are ± 30% of the true values. The LLQC does not need to be made from a different source than the calibration standards. It must also be run at the end of the analytical run where samples are reported for method 6010C. For this reason it may be advantageous to run this with each CCV/CCB pair although this is optional for the analyst.

9.9. Dilution test (Serial Dilution) – A dilution test may be performed if the MS/MSD results are out of compliance to determine whether significant physical or chemical interferences exist due to the sample matrix. The test is performed by running a sample at a 5X (1+4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50X the MDL. If the results are not within 10%, the possibility of chemical or physical interference exists. For standard work the dilution test is not required but is optional. For QSM and for some QAPPs the dilution test is required if the MS/MSD are out of compliance. The sample chosen for MS/MSD is often used for the dilution test and post spike, in accordance with QSM and QAPP requirements.

9.9.1. For QSM samples that fail the serial dilution must then be evaluated using a post-digestion spike.

9.9.2. For Method 200.7, no MSAs (Method of Standard Addition) are required as long as an internal standard is being used for all analytes.

9.10. Post-digestion spikes: If the MS/MSD results are out of compliance a post digestion spike may be performed at the time of analysis. The matrix spiking solution is generally used as the post-digestion spike at the same concentrations as the matrix spikes. For standard work the post digestion spike is optional. For QSM/DOD work it is required if the MS/MSD fail.

9.10.1. If the recovery of the post-digestion spike for an element is outside of 75-125%, a matrix effect should be suspected when the spike level is greater than 25% the concentration of the analyte in the sample. <u>If the serial dilution and the post digestion spike fail, it is recommended to do a higher dilution to dilute out the matrix interference that could cause them to fail. Further dilution is not required by QSM, but it may be required on a project basis..</u>

9.10.2. For method 6010C the post spike recovery limits are 80-120% to trigger corrective actions such as flagging data or doing further dilutions and diluted post spikes.

9.10.3. For method 200.7, if the recovery of the post-digestion spike for an element is

outside of 85-115%, interference should be suspected when the spike level is greater than 20% the concentration of the analyte in the sample.

9.10.4. For method 200.7, and 6010B/6010C, no MSAs are required as long as an internal standard is being used for all analyses.

9.11. Interference Check Analysis (ICSA/ICSAB) – The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. All analytes should be spiked into the ICSAB solution; therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom mix. If the ICP will display over-correction as a negative number then the non-routine elements can be controlled from the ICSA as described below. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.11.1. The ICSAB results for the interferents must fall within 80 – 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.11.2. ICSA results for the non-interfering elements must fall within ± 2X the reporting limit from zero. If the ICSA results for the non-interfering elements do not fall within ± 2X RL from zero the field sample data must be evaluated. For QSM the ICSA result can not be greater than ½ LOQ unless an analyte is confirmed as being a contaminant in the spiking solution.

9.11.2.1. If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.

9.11.2.2. If the affected element was not required then the sample data can be accepted.

9.11.2.3. If the interfering elements are not present in the field sample at a concentration that would result in a false positive or negative result greater than ± 2x RL from zero, then the field sample data can be accepted.

9.11.2.4. If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA. The interference must be within the linear range, or the sample must be diluted.

9.11.2.5. If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.12. For QSM, a linear range standard (LR) is analyzed after the completion of calibration. The linear range standard contains analytes at high levels such that no sample concentration exceeds these levels. If sample concentrations exceed that of the linear range standard for any given analyte, the sample requires dilution at a concentration less than that in the linear range standard. This standard differs from the linear dynamic range standard defined in Section 10.4.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.

10.2. Background Correction Points – To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Alternatively, users may choose multivariate calibration methods. In this case, background correction

points are superfluous since whole spectral regions are processed. MSF is not used for method 6010 analyses at this laboratory.

10.3. Inter-element Corrections (IECs) – ICP interelement correction factors must be determined prior to the analysis of samples and every six months thereafter.

10.3.1. If the instrument is adjusted in any way that may affect the IECs, the IECs must be re-determined.

10.3.2. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by confirming results against a certificate of analysis (COA), using a standard from a second source, or by an alternate analytical method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs.

10.3.3. Refer to the instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference resulting in a false analyte signal greater than ± the RL (Table 2).

10.3.4. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

10.3.5. Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the trace ICP as reflected by the ICSA response. Single element solutions of aluminum, calcium, iron, magnesium, and other interferents may be analyzed routinely to verify IEC factors on target analytes such as arsenic, lead, selenium, and thallium.

10.3.6. Spectral overlap may be avoided by using an alternative wavelength when available. Refer to the ICP instrument manual for specific procedures to use multivariate calibrations.

10.4. Linear Dynamic Range Verification (LDR) – The linear dynamic range must be determined every six months for each analyte wavelength used on each instrument. The linear dynamic range is the concentration above which results cannot be reported without dilution of the sample. The standards used to define the linear dynamic range limit must be analyzed during a routine analytical run. For the initial determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the

signal responses from a minimum of three to five different concentration standards across the estimated range. One standard should be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LDRs, new dynamic ranges must be determined. The LDR data must be documented and kept on file.

10.5. Rinse Time Determination – Rinse times must be determined annually. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see Section 10.4) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). A rinse period of at least 60 seconds between samples and standards is recommended. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

10.6. Calibration must be performed daily and each time the instrument is set up. The calibration curve must consist of a minimum of a blank and a standard. Calibration must be in accordance with the instrument manufacturer's procedure. Flush the system with the calibration blank between each standard or as the manufacturer recommends. Refer to the specific ICP instrument manual for detailed set up and operation protocols.

10.6.1. Tables 2-5 show the concentration of each calibration standard. All standards are made in 5% HCl and 2% HNO_3 solution. Most of the elements are calibrated using a blank and a single standard. A multipoint calibration of 2 or 3 points may be required on some elements to improve linearity at the lower and upper limits of the range. If multiple levels are used, the r-value must be ≥ 0.995. Elements may be separated into different calibration standards due to compatibility issues of the various vendor mixtures.

10.6.2. Working calibration and calibration verification solutions may be used for up to 6 months and must be replaced sooner if verification from an independent source indicates a problem.

10.7. Analysis of the initial calibration verification standards (ICV) immediately follows calibration of the instrument unless a linear range standard is being analyzed per QSM criteria. Stock second source standards for preparation of the initial calibration verification standards are a combination of custom multi-element mixes and single element solutions. Each must be from a different vendor than those standards supplied

for the calibration standards. If from the same vendor, they must be supplied with a separate Lot ID. Tables 7 and 8 show the concentrations of stock solutions used to make the ICV working standard. All standards are made in 5% HCl / 2% HNO_3.

10.7.1. The ICV standard may be used for up to 6 months and must be replaced sooner if verification from an independent source indicates a problem.

10.8. LLICV Sample: A standard made at the concentration at, or in some cases below, the reporting limit for all analytes in the method must be analyzed after the ICV/ICB. For the working LLQC, add 50 mL of reagent water, 10 mL of HCl, and 2 mL of HNO_3 to a 100 mL volumetric flask. Spike this with 1.0 mL of the intermediate LLQC standard and dilute to volume with reagent water. This solution may be used for up to 6 months and must be replaced sooner if verification from an independent source indicates a problem.

10.8.1. For QSM work, as separate QSM LLICV sample is made at slightly higher reporting limits.

10.9. Analysis of the ICSA follows the LLICV sample. To make the ICSA standard, add 300 mL of reagent water to a 500 mL volumetric flask. Add 25 mL of concentrated HCl and 5 mL of concentrated HNO_3. Add 50 mL of Inorganic Ventures mixed standard CLPP-ICS-A and dilute to volume. See Table 10A for ICSA standard concentrations.

10.10. ICSAB analysis directly follows that of ICSA. ICSAB is made by adding 300 mL of reagent water to a 500 mL flask. Add 25 mL of concentrated HCl and 10 mL of concentrated HNO_3. Add 50 mL of Inorganic Ventures mixed standard CLPP-ICS-A and 5.0 mL of Inorganic Ventures CLPP-ICS-B4. The final concentrations are those defined by Tables 10A and 10B.

10.10.1. ICSA and ICSAB may be made every six months and must be replaced sooner if verification from an independent source indicates a problem.

10.10.2. Some regulatory criteria may require that ICSA and ICSAB be analyzed routinely throughout and analytical run or at the beginning and end of the run.

10.11. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity,

chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. All samples require a preparation prior to analysis depending on the matrix being evaluated. Preparation of each batch requires a method blank, LCS, and matrix spikes and/or matrix spike duplicates. The concentration of analytes in the LCSs and matrix spikes are listed in the individual preparation methods.

11.2.1. Method 200.7 specifies exceptions where samples do not need to be digested, but for proper matrix matching all method 200.7 samples are to be prepare using method 3005A (total recoverable metals.)

11.2.2. For method 6010B and 6010C soils analysis, EPA method 3050B is used to prepare all samples.

11.2.3. For method 6010B and 6010C aqueous samples, samples are prepared using either the total digestion procedure method 3010A, although some clients request total recoverable metals using method 3005A..

11.2.4. For many method 6010B and 6010C dissolved samples, we use a matrix matching procedures defined under SOP WS-IP-0008.

11.3. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30-60 minutes of warm-up is suggested).

11.4. Profile and/or align the instrument in accordance to the instrument manufacturer's recommended procedures.

11.5. Rinse the instrument with the calibration blank to flush the system. Verify internal standard(s) will be added to standards and samples by the instrument.

11.6. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported.

11.6.1. To support some client-specific programs, it may be necessary to evaluate the relative standard deviation of exposures for field samples. If this is necessary, the criteria will be ≤ 20% or [±RL] if the RL for that element is 5 µg/L or less and the detected concentration is less than twice the RL.

11.7. Before setting up an analytical run, read the QAS for all of the lots being analyzed. The QAS is created by the project managers and defines any special criteria required for client specific analyses.

11.8. Calibrate the instrument according to the guidelines in Section 10.6.

11.9. Analyze a LR (linear range) standard for DOD QSM. Target analytes must be within ± 10% of the true value to be acceptable. The concentration of each analyte in the linear range standard is defined in Table 12.

11.10. Analyze ICVs/ICB after the calibration sequence. Section 9.6 defines the acceptance criteria for ICVs/ICB analysis for all method referenced in this document. The concentrations of the ICVs are defined in Tables 7 and 8.

11.11. Analyze a low-level standard(s) at or below the RL (LLICV sample). Follow QAPP acceptance criteria. Standard work acceptance limits are ± 50% of true value. For QSM, the acceptance criterion is ± 20% of true value, but the reporting limits differ and therefore the concentrations of analytes in the QSMLLICV differ. Work performed under method 6010C uses acceptance limits of ± 30%. Table 8 defines the concentration of analytes in the LLICV analyzed at laboratory target analyte list reporting limits.

11.12. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in Section 10.5 it can be demonstrated that a shorter rinse time may be used. Triton-X is added to the rinse solution to facilitate the rinse process.

11.13. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.14. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run.

11.15. For unattended overnight auto-runs it is strongly recommended that the frequency of ICSA/ICSAB analysis be increased to every 4 hours when they are required by client specific QC requirements.

11.16. Internal standards are added to all of the QC and samples analyzed. The concentration of analytes in the internal standard is defined in Table 11.

11.16.1. Typically used internal standards are: yttrium or scandium. Indium may be used as an alternative. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.16.2. The internal standard (IS) must be added to every sample and standard at the

same concentration. The internal standard is added to each analytical sample automatically through use of a third pump channel and mixing coil.

11.16.3. The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.16.4. The internal standard raw intensity counts must be printed on the raw data.

11.16.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

11.16.5.1. If the internal standard counts fall within ± 20% of the counts observed in the ICB then the data is acceptable.

11.16.5.2. If the internal standard counts in the field samples are more than ± 20% higher than the expected level, the field samples must then be diluted and reanalyzed, the IS concentrations must be raised, or a different internal standard must be used.

11.17. Full method required QC must be available for each wavelength used in determining reported analyte results.

11.18. Guidelines are provided in Appendix 1 for minimizing contamination of samples and standards, in Appendix 2 for preventive maintenance, and in Table 1 for troubleshooting.

11.19. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed 90% of the linear range or use an alternate wavelength for which QC data are established. If an interelement correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an over range analyte may not be required to be reported for a sample, if that analyte is a possible interferent for any requested analyte in that sample, the sample must be diluted, and affected analytes reported from the dilution. Acid strength must be maintained in the dilution of samples.

11.20. For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

11.20.1. Recovery of the analyte in the matrix spike is not at least 50%,

11.20.2. The concentration of the analyte does not exceed the regulatory level, and,

11.20.3. The concentration of the analyte is within 20% of the regulatory level.

11.20.4. The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table 13.

11.20.5. Appendix 1 provides guidance on performing MSA analyses.

12. CALCULATIONS/DATA REDUCTION

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left(\frac{\text{Found(ICV)}}{\text{True(ICV)}} \right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left(\frac{\text{Found(CCV)}}{\text{True(CCV)}} \right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \times \left(\frac{SSR - SR}{SA} \right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates is calculated according to the following equations:

$$RPD = 100 \left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2} \right)} \right]$$

Where:

MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$\text{mg / Kg, dry weight} = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100 \times \left(\frac{Found(LCS)}{True(LCS)} \right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%\ \text{Difference} = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)
S =Dilution test result (Instrument reading $\times$ 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported in accordance with this laboratory's significant figure policy (WS-PQA-004).

13. METHOD PERFORMANCE

Prior to analysis of any analyte using either Method 200.7 or Method 6010B/6010C, the following requirements must be met:

13.1. Prior to use, the following data must be generated and kept on file for any newly installed instrument: Analytical dynamic range (Section 10.4) determination and verification of interelement correction/spectral interference correction (Section 10.3), and method and instrument detection limits (discussed below). Method 200.7 also requires use of a "Quality Control Standard" which is equivalent to the ICV discussed in Section 10.7.

13.2. Instrument Detection Limit (IDL) – The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined annually. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be re-determined.

13.2.1. IDLs shall be determined by multiplying by three the average of the standard deviations obtained on three non-consecutive days from the analysis of a blank solution with seven consecutive measurements per day.

13.2.2. Each measurement must be performed as though it were a separate analytical sample.

13.2.3. Each measurement must be followed by a rinse and/or any other procedure normally performed between the analyses of separate samples.

13.2.4. The IDL measurement must consist of the same number of replicates used for analytical samples with the average result used for reporting.

13.3. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.4. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.4.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.4.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4.3. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.5. Training Qualification:

The group/team leader or the supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Acid waste consisting of unused sample, analyzed sample solution and rinse solution. This is collected in four-gallon plastic carboys. When full or after no more than one year, the waste is dumped into an acid waste drum in the H-3 closet. When full to no less than one inch and no more than four inches of the top, or after no more than 75 days, the drum will be transferred to the waste collection area for disposal.

15.2. Miscellaneous disposable glassware, plastic vials with snap top caps, autosampler tubes and similar solid waste. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the applicable steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996, Method 6010B.

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Revision 3, February 2007, Method 6010C.

16.3. Determination of Metals and Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, 1994, Method 200.7, Revision 4.4.

16.4. Department of Defense Quality Systems Manual for Environmental Laboratories (QSM), Version 5.1, 2017.

16.5. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

16.6. WS-IP-0008: Matrix Matching

16.7. WS-PQA-003: Laboratory Wide Quality Control Program

16.8. WS-QA-0006: MDLs and IDLs

17. METHOD MODIFICATIONS

17.1. Modifications/interpretations from Method 6010B.

17.1.1. This laboratory uses mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as noted in the methods.

17.1.2. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. In determining IECs, because of lack of definition clarification for “concentration range around the calibration blank,” this laboratory has adopted the procedure in EPA CLP ILMO4.0.

17.1.3. Section 8.5 of Method 6010B recommends that whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because this laboratory receives no prior information from clients regarding when to expect a new or unusual matrix, the laboratory may select to perform a dilution test on one sample in each analytical batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At this facility, matrix interference is determined by evaluating data for the LCS and MS/MSD. The laboratory requires documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.4. IDL’s are analyzed annually, or when there is a change to the instrument, instead of quarterly.

17.2. Modifications from Method 6010B

17.2.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.2.2. Method 6010B states that the results of the calibration blank are to agree within 3X the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within three standard deviation of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. This laboratory has adopted an absolute control limit of ± RL from zero for calibration blank criteria.

17.3. Modifications from Method 200.7

17.3.1. Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration bank signal at the same wavelength. The laboratory utilizes the CLP IDL definition as defined in Section 13.2 of this SOP.

17.3.2. The calibration blank is prepared in an acid matrix of 2% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.3.3. Method 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm for a majority of analytes. For the remaining analyt4s, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV verification of calibrations standard accuracy is independent of the ICV concentration used.

17.3.4. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 states that results should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

17.3.5. Method 200.7 states that the CCB should be less than the IDL, but > the lower 3o-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. This laboratory

has adopted an absolute control limit of the RL from zero for calibration blank criteria. Section 9.7 provides the detailed corrective action criteria that must be followed.

18. ATTACHMENTS

18.1. Appendix 1 – Method of Standard Addition (MSA) Guidance

18.2. Appendix 2 – Contamination Control Guidelines

18.3. Appendix 3 – Preventative Maintenance

18.4. Appendix 4 – Hydrofluoric and Safety Guidelines

18.5. Table 1 – Troubleshooting Guide

18.6. Table 2 –Method 200.7 and6010B Target Analyte Lists

18.7. Table 3 – Calibration Standard 1 Concentrations: Final Volume = 1000 mL

18.8. Table 4 – Calibration Standard 2 Concentrations: Final Volume = 500 mL

18.9. Table 5 – Calibration Standard 3 Concentrations: Final Volume = 500 mL

18.10. Table 6 – Calibration Standard 4 Concentrations: Final Volume = 500 mL

18.11. Table 7 – ICV4 Concentrations: Final Volume = 500 mL

18.12. Table 8 – ICV2A Concentrations: Final Volume = 500 mL

18.13. Table 9 – PQL CRI Intermediate Standard Concentrations: Final Volume = 100 mL

18.14. Table 10A – ICSA Standard Concentrations: Final Volume = 500 mL

18.15. Table 10B – ICSAB Standard Concentrations: Final Volume = 500 mL

18.16. Table 11 – Internal Standard Concentrations: Final Volume = 1 L

18.17. Table 12 – Linear Range Standard (QSM Analyses Only): Final Volume = 100 mL

18.18. Table 13 – TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

18.19. Table 14 – Cross Reference of Terms Used in Method 6010B/6010C and by the Sacramento Laboratory

19. REVISION HISTORY

19.1. WS-MT-0003, Revision 5.9, Effective 03/14/2018

19.1.1. Throughout SOP removed references to AFCEE 4.0.

19.1.2. Section 1.4, removed “and samples for dissolved metals. Although digestion is not specifically required by the method.”

19.1.3. Section 4.5 removed, “The Perkin Elmer Optima instrument can use Multicomponent Spectral Fitting (MSF) which also corrects for spectral interference by measurement of both the interferents and the matrix background contribution.”

19.1.4. Section 9.6.1, removed “and CCVs.”

19.1.5. Section 9.6.2, changed “less than 2x MDL” to “less than ½ LOQ”.

19.1.6. Section 9.10.1, added “Further dilution is not required by QSM, but it may be required on a project basis.”

19.1.7. Section 9.11.2, added “For QSM the ICSA result can not be greater than ½ LOQ unless an analyte is confirmed as being a contaminant in the spiking solution.”

19.1.8. Section 10.8, changed “1 mL of HNO3” to “2 mL HNO3”.

19.1.9. Section 10.9, changed “5 mL of HCl” to “10 mL of HCl”.

19.1.10. Section 10.10, changed “5 mL of concentrated HNO3’ to “10 mL of concentrated HNO3”.

19.1.11. Updated references throughout SOP from “QSM Version 4.2” to “QSM”.

19.1.12. Removed revision history prior to 2016. It can be found in previous versions of this SOP.

19.1.13. Table 2, removed Zirconium and Lithium from analyte list and updated reporting limits.

19.1.14. Editorial changes.

19.2. WS-MT-0003, Revision 5.8, Effective 10/03/2017

19.2.1. Section 10.6.1, revised “5% HCl and 1% HNO_3 solution” to “5% HCl and 2% HNO_3 solution.”

19.2.2. Section 10.7, revised “5% HCl / 1% HNO_3” to “5% HCl / 2% HNO_3.”

19.2.3. Editorial changes.

19.3. WS-MT-0003, Revision 5.7, Effective 02/24/2017

19.3.1. Section 6.2 – Replaced “welding grade or equivalent” with “high purity grade (99.99%).

19.3.2. Editorial changes.

19.4. WS-MT-0003, Revision 5.6, Effective 10/28/2016

19.4.1. Section 7.5; change “…add 40 mL of HNO_3…” to “…add 80 mL of HNO_3....”

19.4.2. Section 7.6; change “…add 10 mL of concentrated HNO_3…” to “…add 20 mL of concentrated HNO_3....”

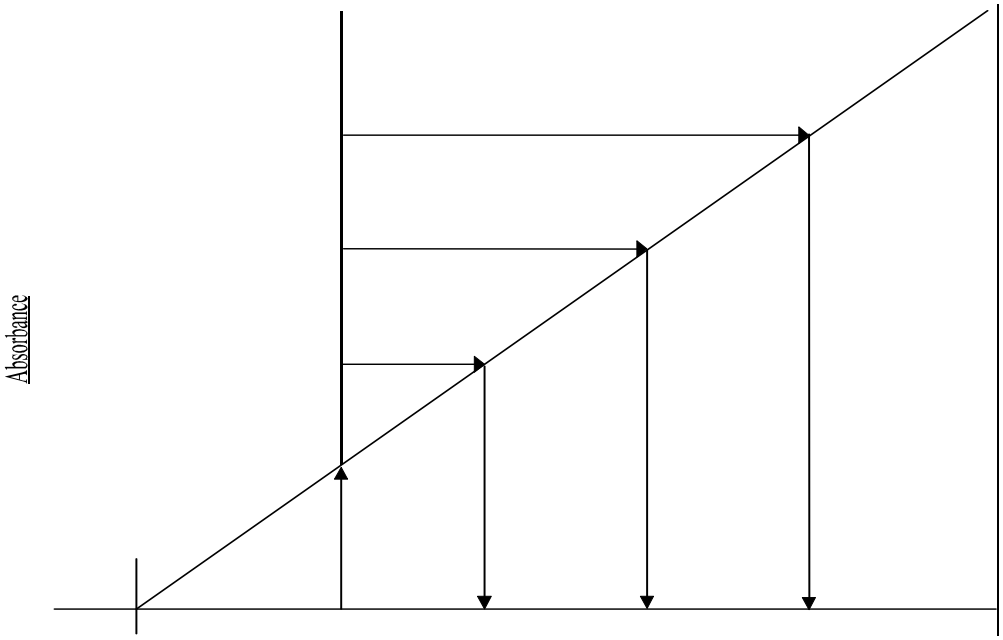
19.4.3. Section 7.9; change “…add 1 mL of HNO_3…” to “…add 2 mL of HNO_3....”

19.4.4. Section 17.3.2: change “ The calibration blank is prepared in an acid matrix of 1% HNO_3/5% HCl…” to “The calibration blank is prepared in an acid matrix of 2% HNO_3/5% HCl…”

19.4.5. Editorial changes.

19.4.6. Appendix 1 – MSA GUIDANCE (Method of Standard Addition)

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.



In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.

For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Appendix 2 – Contamination Control Guidelines

Procedures strongly recommended to prevent contamination:

- Clean all work areas used to prepare standards and spikes before and after each use.
- Wash all glassware with detergent and tap water and rinse with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Do not use powdered gloves of any variety, or latex gloves (powdered or powder-free) in the metals laboratory. Both latex and glove powder contain silica, zinc and other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.
- Check glassware for cracks and etches before each use and discard if found. Etched glassware can cause cross contamination of any metallic analytes.
- Cover autosampler trays to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.
- Check laboratory HVAC systems are working properly

Helpful hints in the identification of the source of contaminants:

- Yellow pipette tips and volumetric caps can sometimes contain cadmium.
- Some sample tubes have been found to contain lead and other analytes.
- The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.
- New glassware especially beakers can be a source of silica and boron.
- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Latex gloves can contain over 500 ppb of zinc.

Appendix 3 – Preventative Maintenance

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log. Document when the instrument has returned to control.

The following procedures are required to ensure that that the instrument is fully operational.

Preventive Maintenance Schedule	
Daily	
Check peristaltic pump tubing, sample capillary tubing, tubing joints, and autosampler sipper probe.	Check air, argon, and nitrogen gas pressure
Check rinse solution and fill if needed	Check waste container and empty if needed
Perform data backup from instrument PC	Check back pressure and droplet size to verify nebulizer is not clogged.
Clean plasma injector and torch to remove accumulated deposits	Check / Clean axial and radial purge windows
Check exhaust vent	Monitor manganese intensities during torch alignment
As Needed	
Replace peristaltic pump tubing, sample capillary tubing, tubing joints and autosampler sipper probe.	Replace axial and radial purge windows
Monthly	
Clean exterior of instrument and chiller Clean air filters on instrument and chiller unit to remove dust	Check coolant level in chiller
Biannually	
	Check / Clean coolant filter

Appendix 4 – Hydrofluoric and Safety Guidelines

Washington Occupational Health Associates

1120 19th Street, N. W.
Suite 410
Washington, D.C. 20036
Phone 202-436-6698
or 800-777-WOHA
Fax. 202-223-6525

To: Don Dihel, Corporate Director EH&S, QES
From: Samuel J. Scott, M.D., M.P.H.
Date: June 21, 1999
Re: Safety Measures with use of Hydrofluoric acid

In response to your request for recommendations of safety/emergency supplies and equipment for use in the event of exposure to hydrofluoric acid, we have compiled the following list:

Supplies and Equipment

1. Shower
2. Eye irrigation station
3. Calcium gluconate gel 2.5%
4. Epsom salts
5. Milk of Magnesia
6. Calcium gluconate tablets
7. Oxygen tank, tubing and oxygen mask capable of delivery of 100% oxygen
8. **Special First Aid Kit** containing injectable calcium gluconate solution. This kit should be carried with the employee to the hospital emergency room. It is quite possible the emergency room will not have the necessary supplies necessary to deal with this type of chemical exposure.

First Aid Measures

Skin Contact:

1. Flush area copiously with water for at least 15 minutes.
2. Remove contaminated clothing immediately.
3. After washing exposed skin, use gloves to rub a generous amount of calcium gluconate gel 2.5% into burn area.

4. For areas too large to apply the gel, use an Epsom salts solution in a concentration of ½ to 1 cup of Epsom salts in one quart of iced water. Immerse the limb into a bucket of solution or soak the solution into gauze and apply to the wound. This dressing should be replaced or re-soaked every two minutes.
5. If area affected is greater than 2 inches by 2 inches, give 6 tablets of calcium gluconate orally.
6. If area affected is greater than 4 inches by 4 inches, assume significant inhalation injury and treat accordingly.
7. Except for small burns, refer to the emergency room and continue applying calcium gluconate gel or Epsom salt soaks en route.

Caution: Hydrofluoric acid causes delayed burns over several hours so the immediate care is essential to prevent further harm.

Inhalation:

1. Administer oxygen 100%.
2. Resuscitate as necessary.
3. Transport to the nearest emergency room.

Eye Contact:

1. Immediately flush the eyes at the irrigation station.
2. Get medical assistance immediately.

Ingestion:

1. Do NOT induce vomiting.
2. Have the victim drink two large glasses of water or milk.
3. Give two ounces of milk of magnesia.
4. Give six tablets of calcium gluconate.

I hope this provides the information you requested. Should you have further questions or require additional information, please do not hesitate to contact one of the physicians at WOHA.

\\fileserver\P\QES\HFacidguidelines.dot

TABLE 1: Troubleshooting Guide

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Check spray chamber is draining Lower Torch
Instrument Drift	Check chiller (not cooling properly) Replace torch, spray chamber (cracked) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks
Erratic Readings, Flickering Torch or High RSD	Check for argon or air leaks Check nebulizer settings Check pump tubing (clogged, worn) Check spray chamber drainage Increase uptake time or flush time (too short) Check peristaltic pump tension adjustment Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Check integration time (too short) Check torch alignment Check sample matrix
Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Check argon and nitrogen gas Replace tubing (clogged, leaks, worn) Check torch alignment Clean, Replace purge windows Clean, Replace optics

TABLE 2: Method 200.7 and 6010B/6010C Target Analyte List

ELEMENT	Symbol	Standard Reporting Limit (ug/L) Water	Standard Reporting Limit (mg/kg) Soil	QSM Reporting Limit (ug/L) Water	QSM Reporting Limit (mg/kg) Soil
Aluminum	Al	200	20	200	21
Antimony	Sb	20	2.0	15	1.5
Arsenic	As	20	2.0	21	2.1
Barium	Ba	5.0	1.0	21	2.0
Beryllium	Be	2.0	0.2	3.0	0.3
Boron	B	100	10	200	20
Cadmium	Cd	2.0	0.2	3.0	0.3
Calcium	Ca	500	50	500	100
Chromium	Cr	8.0	0.5	12	1.2
Cobalt	Co	5.0	0.5	6.0	0.6
Copper	Cu	10	1.5	20	2.5
Iron	Fe	100	10	100	10
Lead	Pb	5.0	1.0	10	2.0
Magnesium	Mg	500	50	500	50
Manganese	Mn	5.0	0.5	12	—
Molybdenum	Mo	20	2.0	30	3.0
Nickel	Ni	5.0	1.0	10	1.0
Phosphorus	P	500	50	500	50
Potassium	K	1000	100	1000	100
Selenium	Se	20	2.0	25	2.0
Silicon	Si	500	**	—	—
Silver	Ag	5.0	0.5	5.0	0.5
Sodium	Na	1000	100	1000	500
Strontium	Sr	50	5.0	—	—
Sulfur	S	500	50	—	—
Thallium	Tl	20	2.0	15	2.0
Tin	Sn	20	10	—	—
Titanium	Ti	10	1.5	10	3.0
Vanadium	V	5.0	0.5	20	3.0
Zinc	Zn	10	2.0	20	3.0

TABLE 3: Calibration Standard 1 Concentrations: Final Volume = 1000 mL

Element	Stock Standard ID	Stock Std Concentration (mg/L)	Spike Volume (mL)	Final Concentration (mg/L)
Aluminum (1)	QCAL-1A	10000	5.0	50
Aluminum (2)	QCAL-1A	10000	5.0	50
Antimony	Single source	1000	1.0	1.0
Arsenic	QCAL-5	1000	1.0	1.0
Barium	QCAL-6D	1000	5.0	5.0
Beryllium	QCAL-6D	200	5.0	1.0
Boron	QCAL-6C	1000	2.0	2.0
Cadmium	QCAL-5	1000	1.0	1.0
Calcium	QCAL-2A	10000	5.0	50
Chromium	QCAL-6D	1000	5.0	5.0
Cobalt	QCAL-6D	1000	5.0	5.0
Copper	QCAL-6D	1000	5.0	5.0
Iron (1)	QCAL-1A	10000	5.0	50
Iron (2)	QCAL-1A	10000	5.0	50
Lead	QCAL-5	1000	1.0	1.0
Lithium	QCAL-7B	1000	5.0	5.0
Magnesium	QCAL-2A	10000	5.0	50
Manganese	QCAL-6D	1000	5.0	5.0
Molybdenum	QCAL-6C	1000	2.0	2.0
Nickel	QCAL-6D	1000	5.0	5.0
Phosphorus	QCAL-7B	1000	5.0	5.0
Potassium	See Calibration Std 3			
Selenium	QCAL-5	1000	1.0	1.0
Silicon	See Calibration Std 3			
Silver	Single Source	1000	1.0	1.0
Sodium (1)	QCAL-3A	10000	5.0	50
Sodium (2)	QCAL-3A	10000	5.0	50
Strontium	QCAL-7B	1000	5.0	5.0
Sulfur (1)	See Calibration Std 3			
Sulfur (2)	See Calibration Std 3			
Thallium	QCAL-5	1000	1.0	1.0
Tin	QCAL-7A	1000	5.0	5.0
Titanium	QCAL-6C	1000	2.0	2.0
Vanadium	QCAL-6D	1000	5.0	5.0
Zinc	QCAL-6D	1000	5.0	5.0

TABLE 4: Calibration Standard 2 Concentrations: Final Volume = 500 mL

Element	Stock Standard ID	Stock Std Concentration (mg/L)	Spiking Volume (mL)	Final Concentration (mg/L)
Aluminum (2)	QCAL-1A	10000	12.5	250
Calcium	QCAL-2A	10000	12.5	250
Iron (2)	QCAL-1A	10000	12.5	250
Magnesium	QCAL-2A	10000	12.5	250
Phosphorous	Single Source	10000	2.5	50
Sodium (1)	QCAL-3A	10000	12.5	250
Sodium (2)	QCAL-3A	10000	12.5	250

TABLE 5: Calibration Standard 3 Concentrations: Final Volume = 500 mL

Element	Stock Standard ID	Stock Std Concentration (mg/L)	Spiking Volume (mL)	Final Concentration (mg/L)
Potassium	Single Source	1000	2.5	5.0
Silicon	QCAL-7A	1000	2.5	5.0
Sulfur (1)	QCAL-7A	1000	2.5	5.0
Sulfur (2)	QCAL-7A	1000	2.5	5.0
Antimony	Single Source	1000	0.5	1.0

TABLE 6: Calibration Standard 4 Concentrations: Final Volume = 500 mL

Element	Stock Standard ID	Stock Std Concentration (mg/L)	Spiking Volume (mL)	Final Concentration (mg/L)
Potassium	Single Source	10000	5.0	100
Sulfur (1)	QCAL-7A	10000	5.0	100
Sulfur (2)	QCAL-7A	10000	5.0	100
Phosphorous	Single Source	10000	5.0	100

TABLE 7: ICV4 Concentrations: Final Volume = 500 mL
ICV4 is made from Spex XCAL-56-500
Spiking volume = 5.0 mL

Element	Stock Std Concentration (mg/L)	Final Concentration (mg/L)
Aluminum (1)	100	10
Aluminum (2)	100	10
Antimony	2.5	0.25
Arsenic	2.5	0.25
Barium	10	1.0
Beryllium	2.5	0.25
Boron	5.0	0.5
Cadmium	2.5	0.25
Calcium	100	10
Chromium	10	1.0
Cobalt	10	1.0
Copper	10	1.0
Iron (1)	100	10
Iron (2)	100	10
Lead	2.5	0.25
Lithium	10	5.0
Magnesium	100	10
Manganese	10	1.0
Molybdenum	5.0	0.5
Nickel	10	1.0
Phosphorus	10	1.0
Potassium	100	10
Selenium	2.5	0.25
Silicon	10	1.0
Silver	2.5	0.25
Sodium (1)	100	10
Sodium (2)	100	10
Strontium	10	1.0
Sulfur (1)	See ICV2A	
Sulfur (2)	See ICV2A	
Thallium	2.5	0.25
Tin	10	1.0
Titanium	5.0	0.5
Vanadium	10	1.0
Zinc	10	1.0

TABLE 8: ICV2A Concentrations: Final Volume = 500 mL

Element	Stock Standard ID	Stock Std Concentration (mg/L)	Spiking Volume (mL)	Final Concentration (mg/L)
Phosphorous	Single Source	1000	1.0	2.0
Sulfur	Single Source	1000	1.0	2.0
Silicon	Single Source	1000	0.5	1.0
Antimony	Single Source	1000	0.125	0.25

TABLE 9: LLICV Intermediate Standard Concentrations: Final Volume = 100 mL
Intermediate standard made from 1000 mg/L single element sources.
The final LLICV concentration is a 100X dilution of the intermediate standard.

Element	Stock Standard Spike Level (Ml)	Intermediate Concentration (mg/L)	Final LLICV Concentration (mg/L) = LL1	QSMLLICV Intermediate Concentration (mg/L) = LL2	Final QSMLLICV Concentration (mg/L) = LL2
Aluminum	1.0	20	0.2	20	0.2
Antimony	0.05	2.0	0.02	3.0	0.03
Arsenic	0.1	2.0	0.02	4.0	0.04
Barium	0.05	0.5	0.005	2.0	0.02
Beryllium	0.02	0.2	0.002	0.3	0.003
Boron	0.5	10	0.1	20	0.2
Cadmium	0.02	0.2	0.002	0.3	0.003
Calcium	1.0	50	0.5	50	0.5
Chromium	0.05	0.5	0.005	1.0	0.01
Cobalt	0.05	0.5	0.005	1.0	0.01
Copper	0.05	1.0	0.01	1.5	0.015
Iron	0.25	210	0.1	10	0.1
Lead	0.03	0.5	0.005	1.0	0.01
Lithium	0.1	1.0	0.01	2.0	0.02
Magnesium	1.0	50	0.5	50	0.5
Manganese	0.05	0.5	0.005	1.0	0.01
Molybdenum	0.1	2.0	0.02	2.0	0.02
Nickel	0.05	0.5	0.005	1.0	00.
Phosphorus	1.0	50	0.5	50	0.5
Potassium	2.5	100	1.0	100	1.0
Selenium	0.05	2.0	0.02	4.0	0.04
Silicon	0.5	50	0.5	50	0.5
Silver	0.05	0.5	0.005	0.5	0.005
Sodium	2.5	100	1.0	50	0.5
Strontium	0.25	5.0	0.05	5.0	0.05
Sulfur	0.5	50	0.5	50	0.5
Thallium	0.1	2.0	0.02	3.0	0.03
Tin	0.2	2.0	0.02	2.0	0.02
Titanium	0.05	1.0	0.01	1.0	0.01
Vanadium	0.05	0.5	0.005	2.0	0.02
Zinc	0.05	1.0	0.01	2.0	0.02

TABLE 10A: ICSA Standard Concentrations: Final Volume = 500 mL
ICSA is made from IV CLPP-ICS-A
Spiking volume = 50

Element	Stock Standard Concentration (mg/L)	Final ICSA Concentration (mg/L)
Aluminum	**5000**	**500**
Calcium	**5000**	**500**
Iron	**2000**	**200**
Magnesium	**5000**	**500**

TABLE 10B: ICSAB Standard Concentrations: Final Volume = 500 mL
ICSAB is made from IV CLPP-ICS-B4 + ICSA
Spiking volume = 5.0 mL

Element	Stock Std Concentration (mg/L)	Final Concentration (mg/L)
Antimony	**60**	**0.6**
Arsenic	**10**	**0.1**
Barium	**50**	**0.5**
Beryllium	**50**	**0.5**
Cadmium	**100**	**1.0**
Chromium	**50**	**0.5**
Cobalt	**50**	**0.5**
Copper	**50**	**0.5**
Lead	**5.0**	**0.05**
Manganese	**50**	**0.5**
Nickel	**100**	**1.0**
Selenium	**5.0**	**0.05**
Silver	**20**	**0.2**
Thallium	**10**	**0.1**
Vanadium	**50**	**0.5**
Zinc	**100**	**1.0**

TABLE 11: Internal Standard Concentrations: Final Volume = 1 L

Element	Stock Standard Concentration (mg/L)	Spiking Volume (mL)	Final Concentration (mg/L)
Yttrium	**1000**	**6.0**	**6.0**

TABLE 12: Linear Range Standard (QSM analyses only): Final Volume = 100 mL

Element	Stock Standard ID	Stock Std Concentration (mg/L)	Spike Volume (mL)	Final Concentration (mg/L)
Antimony	**Single source**	**1000**	**0.5**	**1.0**
Arsenic	**QCAL-5**	**1000**	**0.5**	**5.0**
Barium	**QCAL-6D**	**1000**	**0.5**	**10.0**
Beryllium	**QCAL-6D**	**200**	**0.5**	**2.0**
Boron	**QCAL-6C**	**1000**	**0.5**	**5.0**
Cadmium	**QCAL-5**	**1000**	**0.5**	**5.0**
Chromium	**QCAL-6D**	**1000**	**0.5**	**10.0**
Cobalt	**QCAL-6D**	**1000**	**0.5**	**10.0**
Copper	**QCAL-6D**	**1000**	**0.5**	**10.0**
Lead	**QCAL-5**	**1000**	**0.5**	**5.0**
Lithium	**QCAL-7B**	**1000**	**0.5**	**5.0**
Manganese	**QCAL-6D**	**1000**	**0.5**	**10.0**
Molybdenum	**QCAL-6C**	**1000**	**0.5**	**5.0**
Nickel	**QCAL-6D**	**1000**	**0.5**	**10.0**
Phosphorus	**QCAL-7B**	**1000**	**0.5**	**5.0**
Potassium	**QCAL-7A**	**10000**	**0.5**	**100**
Selenium	**QCAL-5**	**1000**	**0.5**	**5.0**
Silicon	**QCAL-7A**	**1000**	**0.5**	**5.0**
Silver	**Single Source**	**1000**	**0.1**	**1.0**
Sodium (1)	**QCAL-3A**	**10000**	**0.5**	**100**
Sodium (2)	**QCAL-3A**	**10000**	**0.5**	**100**
Strontium	**QCAL-7B**	**1000**	**0.5**	**5.0**
Sulfur	**QCAL-7A**	**1000**	**0.5**	**5.0**
Thallium	**QCAL-5**	**1000**	**0.5**	**5.0**
Tin	**QCAL-7A**	**1000**	**0.5**	**5.0**
Titanium	**QCAL-6C**	**1000**	**0.5**	**5.0**
Vanadium	**QCAL-6D**	**1000**	**0.5**	**10.0**
Zinc	**QCAL-6D**	**1000**	**0.5**	**10.0**

TABLE 13: TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	10000
Barium	10000	100000	10000
Cadmium	100	1000	250
Chromium	500	5000	1000
Lead	500	5000	2500
Selenium	250	1000	10000
Silver	500	5000	250

TABLE 14: Cross Reference of Terms Used in Methods 6010B/6010C and the Sacramento Laboratory

EPA 200.7	SW6010B/C	Sacramento
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument Check Standard	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	NA	NA
Laboratory fortified blank (LFB)	NA	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Reagent Blank	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Interference Check Sample	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

Title: Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A [Method SW846 7470A]

Approvals (Signature/Date):

Robert Hrabak — Date 7/26/18
Technical Manager

Joe Schairer — Date 7/30/18
Health & Safety Manager / Coordinator

Lisa Stafford — Date 7/26/2018
Quality Assurance Manager

Chris Williams — Date 8/1/18
Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A. Method 7470A is applicable to the preparation and analysis of mercury in ground water, wastewater, wastes, wipes, TCLP, SPLP, STLC leachates, and samples from stationary source sampling trains. All matrices require sample preparation prior to analysis. This also applies to digestates prepared by SOP WS-IP-0007.

1.2. When undertaking projects for Department of Defense (DOD) and/or the Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, "Federal Program Requirements", must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Samples are digested using a combination of acids and strong oxidizers, converting all forms of mercury to mercuric ions. The samples are treated with hydroxylamine sulfate solution to remove excess oxidizing reagents. The digestates are analyzed using Leeman Labs automated mercury analyzers. The mercuric ions are reduced to atomic mercury vapor with the addition of stannous chloride into a gas-liquid separator. The mercury vapor is purged into an absorption cell with argon gas. The relative absorbance of the mercury vapor is detected at a wavelength of 253.7- nm via a lamp/detector system.

2.2. Reporting limit: 0.2 µg/L.

2.3. Calibration Range: 0.2 µg/L to 10 µg/L.

2.4. This analytical method is restricted to use by, or under the supervision of an analyst experience in the operation of cold vapor analysis and the evaluation of the resulting data.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Potassium permanganate, which is used to breakdown organic mercury compounds also eliminates possible interference from sulfide. Concentrations as high as 20 mg/L

of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. High levels of free chlorine can cause a positive interference. Seawaters, brines and industrial effluents high in chlorides require additional permanganate (as much as 5.0 mL) since chloride is converted to free chlorine during oxidation. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

***Note**: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.*

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The front half air train samples extracted are in a 2% HF solution. The labels fro all such extracts and digestates should be highlighted in yellow. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S staff and the local supplement to the Corporate Safety Manual for guidance. Anyone working with HF must receive special training before starting work. Staff members who work around HF should also receive this training. HF and solutions may not be used for any purposes except as prescribed in this or other Sacramento SOPs. Processes involving HF acid are classified as high-risk activities. Personnel involved must wear a face shield in addition to safety glasses or goggles.

<u>WARNING</u> The Sacramento Emergency Response Team must be activated for any suspected exposure to HF liquid or fumes. After local emergency response,

the victim will be transported to the UCD Medical Center Emergency Room.

5.1.1.1. 2.5% calcium gluconate gel should be applied copiously and gently massaged into burn sites after rinsing under cold running water for one minute to remove HF acid from the surface of the skin. The person massaging should wear appropriate protective gloves. If the victim is conscious, they should consume small quantities of calcium or magnesium containing liquids, such as milk, Maalox, or Milanta. If the burn site is too large for massage with calcium gluconate gel, cover it with gauze soaked in an Epsom salt solution (1 cup to 1 quart of cold water). Keep the wraps saturated with Epsom salt solution.

5.1.1.2. Some metals react with HF to produce flammable hydrogen gas.

5.1.1.3. Glass reacts with HF to produce toxic silicon tetrafluoride.

5.1.1.4. HF is classified as a poison, and must be stored in a locked cabinet when not in use.

5.1.1.5. Whenever HF is in use in a laboratory, a decontamination solution will be prepared before starting work. Take a small bucket (2-3 gallons) and fill it about 2/3 – 3/4 full with an Epsom salts solution. (1/2 cup of Epsom salts per quart of water). Before removing gloves or handling anything else that HF on the gloves might be transferred to, carefully dip one hand at a time into the Epsom salts solution for a few seconds. Do not put the hand any deeper than necessary to bring the solution level to within 1-2 inches of the glove cuff. The Epsom salts solution will be prepared each day prior to beginning work with HF, and disposed at the end of the day down the laboratory sink.

5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled.

5.1.3. Latex, nitrile, and vinyl gloves provide adequate protection against the chemicals and reagents typically used during this process. However, if any organic solvents are used, or any sample matrix contains organic solvents, only nitrile gloves should be used.

5.1.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.5. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware

represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.6. Mercury is a highly toxic element that must be handled with care. Mercury vapor is toxic, so all work must be done in a functioning fume hood and vapors must be vented into a hood. If any volume of mercury reagent or liquid mercury is spilled, the Emergency Response Team must be activated, code yellow, for spill clean-up.

5.1.7. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.8. Do not look directly into the beam of the mercury lamp. The UV light from the lamp is harmful to the eyes.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Hydroxylamine Sulfate	Corrosive	None	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Corrosive to the eyes. Irritant and possible sensitizer. May cause burns to the skin.
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator Poison	1 mg/m^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid (1)	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Hydrofluoric Acid (1)	Poison Corrosive Dehydrator	3 PPM - TWA	Corrosive to the skin and eyes. Contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. Severely corrosive to the respiratory tract. Permanent eye damage may occur. **THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.**
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT

6.1. Preventative and routine maintenance is described in the “Schedule of Routine Maintenance” in the QAM Preventative Maintenance (Section 20.2) and Schedule of Routine Maintenance (Table 20.2).

6.2. An autoclave capable of obtaining conditions of 15 psi at 120°C for 15 minutes. The temperature of the autoclave is verified daily using an “LASCAR” , or equivalent,

temperature log device. In order to achieve a minimum 15 minutes at 120°C, the autoclave is set for a run duration of 25 minutes.

6.3. Automated mercury analyzer. Leeman Hydra AA (or equivalent) with autosampler and WinHg Runner 1.5 software.

6.4. Computer with a printer.

6.5. Top-loading balance capable of accurately weighing 0.01 g.

6.6. 14 mL polystyrene test tubes for the autosampler.

6.7. 50 mL centrifuge tubes.

6.8. Argon gas supply.

6.9. Pump windings – dimensions per instrument manufacturer requirements.

6.10. Bottle-top dispenser: re-pipetters.

6.11. Volumetric adjustable air displacement pipets.

6.12. Class A volumetric flasks.

6.13. pH indicator strips (pH range 0-14).

7. REAGENTS AND STANDARDS

7.1. Reagent water is produced by a Millipore nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. Reagent water is checked on a daily basis as per SOP WS-QA-0014, (Monitoring Reagent Grade Laboratory Water).

7.2. Nitric acid (HNO_3), concentrated, analytical reagent grade.

7.3. Sulfuric acid (H_2SO_4), concentrated, analytical reagent grade.

7.4. Potassium permanganate, 5% solution (w/v): Dissolve 5.0 g of potassium permanganate for each 100 mL of reagent water.

7.5. Potassium persulfate, 5% solution (w/v): Dissolve 5.0 g of potassium persulfate for each 100 mL of reagent water.

7.6. Stannous chloride solution: Add 25 g of stannous chloride and 15 mL concentrated HCl to 250 mL of reagent water. This mixture is a suspension and should appear cloudy. This solution must be made every 12 hours.

7.7. Sodium chloride-hydroxylamine sulfate solution: Add 12.0 g of sodium chloride and 12.0 g of hydroxylamine sulfate for each 100 mL of reagent water.

7.8. All standards must be stored in polyethylene or polypropylene bottles.

7.9. 10 ppm mercury standards: Two separate stock standards purchased from separate manufacturers or different lots from the same manufacturer. One standard is used to make the ICV standard and the other for instrument calibration, CCVs, LCSs, and MS/SDs. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year from opening, and must be replaced sooner if verification from an independent source indicates a problem.

***Note**: Upon receipt of any stock standard, the "Certificate of Analysis" should be immediately filed in the proper location labeled with the receipt date. The receipt date and date the container is opened must be labeled on the container. Also write the new expiration date on the bottle which is the manufacturer's expiration date or one year after opening the stock standard.*

7.10. 0.1 ppm working mercury standards (for each stock): add 0.5 mL of stock 10 ppm source standard and 1 mL HNO_3 to a 50 mL volumetric flask and dilute to volume with reagent water. These standards must be made daily.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of analysis.

8.2. Aqueous samples must be preserved with nitric acid to a pH of <2 at the time of sampling and may be stored in either plastic or glass. Refrigeration is not required. Preservation **must** be verified prior to analysis

9. QUALITY CONTROL

9.1. Batch - A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards. A batch must be analyzed within the same time frame. A method blank (MB), laboratory control sample (LCS) or laboratory control sample duplicate (LCSD) are distilled and analyzed as a part of every batch. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/SD), or in some instances a sample/sample duplicate. An analysis batch must include all QC samples, however they do not contribute to the maximum of 20 samples (see policy WS-PQA-003 (Quality Control Program) for more details).

9.2. One method blank must be processed and reported for every 20 samples. If a method blank exceeds +/- the reporting limit, then the samples must be re-extracted. The exception is samples that are less than the reporting limit and those that exceed 10X the concentration of the analyte in the method blank. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo (see policy WS-PQA-003(Quality Control Program) for further details).

9.3. A laboratory control sample (LCS) must be processed and reported for every 20 samples prepared and analyzed. An LCS/LCSD is required per prep batch for all air train samples. A LCS consists of reagent water spiked with the analyte of interest and processed through all of the steps, and at the same time as the associated samples. If a LCS is outside of percent recovery acceptance criteria, all of the samples associated with that LCS must be re-extracted. One exception is when a LCS exhibits a high recovery, those samples with analyte concentrations less than the reporting limit can be reported. All corrective actions must be documented on a Non-conformance memo (see policy WS-PQA-003 (Quality Control Program) for further details).

9.4. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. MS/MSD pairs are aliquots of a selected field sample spiked with a known concentration of the analyte of interest. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Re-extraction of the blank, LCS, selected field samples, and/or the MS/MSD may be required after evaluation and review.
***Note**: For air train samples the MS/MSD is typically performed on one Back Half (HNO_3/H_2O_2) fraction.*

9.4.1. Samples identified as field blanks, equipment blanks, or trip blanks should not be used for sample/sample duplicate nor MS/MSD analysis.

9.4.2. A laboratory control sample (LCS or LCS/LCSD) may be substituted when insufficient volume is provided to process a sample/sample duplicate or MS/MSD pair if required by program or client. The LCS and LCSD are evaluated independently for acceptance (see policy QA-003-SAC (Quality Control Program) for further details.

9.5. Initial Calibration Verification (ICV/ICB) – Calibration accuracy is verified by analyzing a second source standard immediately upon completion of instrument calibration. This standard must be at a concentration different from that used to calibrate the instrument and different from the CCV standard. The ICV must fall within +/- 10% of the true value of the standard solution. An ICB prepared the same as the calibration blank must be analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the

reporting limit from zero. If either the ICV or ICB fail to meet acceptance criteria the analysis must be terminated, the problem corrected, and the instrument re-calibrated.

9.6. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. Ten samples include any analysis that registers with a result, even if not used. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument re-calibrated, the calibration verified, and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action includes re-preparation of the associated samples.

***Note**: The absolute vale of analyte in MB/ICB/CCB must be <1/2 LOQ, or 1/10th the amount measured in any sample, or 1/10th the regulatory limit. The recovery of the Low-level calibration standard (less than or equal to the LOQ) must be within ±20% of the true value. CCV must be within ±10% of the true value. If CCV fails the criteria, immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, repeat CCV and all associated samples since last successful CCV.*

10. CALIBRATION

10.1. All air displacement pipettes must be calibrated over their range of use at least monthly. If the analyst suspects a spiking volume problem, calibration may be required more often. See SOP WS-QA-0004 for pipettor calibration verification procedures.

10.2. All volumetric digestion vessels must be calibrated according to the frequency and procedures outlined in SOP WS-QA-0004. Prior to removing digestion vessels from the vendors box, verify that the box has a “TestAmerica Passed QC” sticker affixed to the outside. If there is not a sticker affixed, contact QA and have the lot checked before use.

10.3. All balances must be calibrated daily before use according to the procedures outlined in SOP WS-QA-0041.

10.4. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-P-003, Calibration Curves and Selection of Calibration Points.

10.5. Instrument calibration must be performed daily (once every 24 hours) or each time the instrument is off or non-operational for more than two hours.

10.6. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.7. Calibration standards must be processed through the preparation procedure as described in Section 11.9. Calibration standards must be prepared daily.

10.8. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a calibration blank. One standard must be at the reporting limit. Analyze standards in ascending order beginning with the blank

10.8.1. Calibration standard concentrations and the volume of 0.1 ppm working standard required to make 30 mL of standard:

0.1 ppm Hg Volume (mL)	0.06	0.15	0.3	1.5	3.0
Final Concentration (µg/L)	0.2	0.5	1.0	5.0	10.0

10.8.2. 30 g of 2% HNO_3 is added to each digestion vessel. An adjustable volumetric pipette is used to remove a volume of 2% HNO_3 corresponding to the spiking volume for that standard. The standard is then spiked with the appropriate volume of 0.1 ppm Hg standard.

10.9. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and re-calibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

10.10. The concentration of the ICV (section 9.5) is 2.0 ppb made by diluting 0.6 mL of the 0.1 ppm alternate source intermediate to 30 mL total volume with 2% HNO_3. See Section 10.4.2 for the spiking procedure.

10.11. The 5.0 ppb CCV is the same as the calibration standard.

10.12. The autoclave is monitored daily to assure samples are digested for the proper time interval at the appropriate temperature. An "ERTCO" or equivalent temperature logging device is put in the autoclave and undergoes the same program as the samples. This device can be connected to a computer and a program generates a graph of time vs temperature. These are submitted with the raw data.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. All preparation procedures must be carried out in a properly functioning hood.

11.3. All samples are to be checked out and back into sample control with the chain of custody documentation filled out completely. Samples are to be returned to the sample control area once all of the digestions have been initiated.

11.4. Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be accurate and legible. Always set the samples up on the sample cart in the order with which they are to be dispensed. Double-check the sample bottle IDs, once prior to pouring them into the digestion vessels and again before they are returned to the sample cart.

11.5. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludge and tissues (animal/plant) are usually logged in with solid test codes. When initiating preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge-like, organic liquid, lots of sediment etc.) contact the lab supervisor or project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.6. **Always** read the QAS for every project prior to establishing batch associations. This is meant to assure that all client requirements are satisfied and it also aids in creating more efficient batches. Setting up batches to minimize QC and meet all client requirements is a skill that must be developed by all new personnel with the aid of skilled sample preparation and analytical staff.

11.7. The following procedure must be followed for all aqueous sample preparations:

11.7.1. Use a small aliquot of each sample and measure the pH with pH indicator strips. If the bottles have an “X” on the tops, they have already been tested for other tests and do not require this step.

11.7.1.1. For samples with pH<2 put an "X" on top of the bottle indicating the samples were properly preserved. For these samples, record pH<2 on the preparation log.

11.7.1.2. For samples that test pH>2, notify the project manager immediately so they can contact the client. If the client requests the sample be preserved by the laboratory, add HNO_3 in 1.0 mL aliquots until the sample remains at pH<2 for at least 10 minutes. Let the sample stand for 24 hours and re-test the pH prior to preparation. As long as the pH<2, the sample can then be digested. A non-conformance memo must be created with an accounting of the anomalous event and the corrective action. A "Sample Preservation Log" must also be completed and filed with the raw data. Record "AF" for "Anomaly Filed" on the digestion log. Put an "X" on top of the bottle indicating the samples were properly preserved.

11.8. Sample Preparation:

11.8.1. All calibration and QC standards must be prepared in the same manner as the samples and be processed through all steps of the preparation procedure.

11.8.2. Transfer 30 mL of **well-mixed sample** to a 50 mL graduated centrifuge tube. The MS/SD sample should be shaken between pouring the separate aliquots.

11.8.2.1. For Air Train samples see SOP WS-IP-0007 for sample preparation procedures. For EPA Method 29, EPA Method 0060, or CARB 436 preparations on Air Trains:

11.8.2.1.1. Transfer 30mL of the $KMnO_4$ filtrate to a 50mL graduated centrifuge tube.

11.8.2.1.2. Transfer 30mL of the HCl filtrate into a 50mL graduated centrifuge tube.

11.8.2.1.3. Pipette 3mL of the Back Half Nitric Peroxide filtrate into a 50mL graducated centrifuge tube.

11.8.2.1.4. Pipette 3mL of the condensate into a 50mL graduatted centrifuge tube.

11.8.2.1.5. Transfer 30 mL of the Front Half digeste to a 50 mL graduated centrifuge tube. (**Warning**: Front Half contains hydrofluoric acid)

11.8.3. TCLP extracts are diluted 5X and STLC extracts diluted 10X prior to digestion. LCS and MS/SD spiking levels are 1.0 ppb times the dilution factor.

11.8.4. For each method blank and LCS required, weigh out 30 g of water. For the LCS, remove 0.3 mL with an adjustable volumetric pipette. Add 0.3 mL of the 0.1 ppm Hg intermediate used to make the calibration standards. The LCS mercury concentration is 1.0 ppb.

***Note**: For EPA Method 29, 0060 or CARB 36 samples add 27 mL of deionized nanopure H_2O to the Back Half (HNO_3/H_2O_2) and condensate.*

11.8.5. For each MS/SD, spike the samples directly with 0.3 mL of the 0.1 ppm Hg intermediate used to make the calibration standards. The MS/SD mercury concentration is 1.0 ppb.

11.8.5.1. EPA Method 29 and EPA Method 0060: Add 27mL of DI Nanopure water to the Back Half Nitric Peroxide and Condensate.

11.8.6. Add 2.0 mL of concentrated H_2SO_4 and 1.0 mL of concentrated HNO_3, to all samples and standards, mixing after each addition.

11.8.7. Add 5.0 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If additional Potassium Permanganate needs to be added to one of the samples, it also need to be added to all other samples in the batch and the QC samples (MB, LCS, MS, MSD and the curve). If after the addition of up to 5 mL additional permanganate the color does not persist, sample dilution may be required.

***Note**: When reporting analyses, the addition of excess reagent must be addressed through mathematical correction of the results to account for the resultant dilution effect.*

11.8.8. Add 2.5 mL of potassium persulfate solution.

11.8.9. Autoclave for 25 minutes at 120 °C and 15 lbs (to maintain temperature for 15 minutes).

11.8.9.1. Allow the autoclave to cool to less than 80°C where there is no psi of pressure remaining.

<u>Warning:</u> Caution must be used when opening the door of the autoclave as superheated steam may be present and can cause significant burns.

11.8.10. Allow all of the samples to cool to room temperature. Check that the volume is 40.5 mL (45.5 mL if additional permanganate added). If volume is <30 mL, add reagent H_2O to bring volume to 30 mL. If volume is >30 mL, record the volume.

11.9. Sample Analysis:

11.9.1. When ready to begin analysis, add 2mL of sodium chloride-hydroxylamine sulfate solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains). Mercury analysis is run using a Leeman PS200II or Hydra AA automated mercury analyzer. Results are reported as ug/L Hg.

11.9.1.1. Make certain the lamp is on and the pump windings are working properly. Replace any pump windings that do not exhibit a smooth flow of liquid or look stretched or flattened.

<u>Warning:</u> Do not look directly into the beam of the mercury lamp. The UV light from the lamp is harmful to the eyes.

11.9.1.2. Load the samples into the automated sampler racks according to the sample lists entered into the computer. Do a final check once all samples have been loaded on to the racks to assure proper sequencing.

11.9.1.3. Stannous chloride is automatically added to the samples by the instrument via the peristaltic pump and mixed with the sample in a liquid-gas separator. This must be freshly prepared every 12 hours.

11.9.2. Dilute and reanalyze all samples that exceed the 10 ppb linear range of the calibration.

11.9.3. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.9.4. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.9.5. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.9.6. The instrument may have a memory effect after a high concentration sample is analyzed. If sample A has a concentration greater than 20 ppb and sample B is immediately analyzed after a sample A, then sample B needs to be reanalyzed.

11.10. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data is reviewed periodically throughout the run.

12. CALCULATIONS/DATA REDUCTION

12.1. ICV/CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found_{(ICV\ /\ CCV)}}{True_{(ICV\ /\ CCV)}}\right)$$

12.2. Matrix spike recoveries are calculated according to the following equation:

$$\%\ R\ =\ 100\left(\frac{SSR\ -\ SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.3. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.4. The final concentration for an aqueous sample is calculated as follows:

$$\mu g/L = \text{ C x D}$$

Where:

C = Concentration ($\mu g/L$) from instrument readout
D = Instrument dilution factor

12.5. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found_{(LCS)}}{True_{(LCS)}}\right)$$

12.6. Appropriate factors must be applied to sample values if dilutions are performed.

12.7. Sample results should be reported with up to three significant figures in accordance with the TestAmerica Sacramento significant figure policy (see QA-004-SAC, Rounding and Significant Figures).

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.2. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-006 and policy S-Q-003.

13.3. Initial Demonstration

Each analyst must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration standard.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the historical acceptance criteria.

13.3.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.4. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

15.1. The following waste streams are produced when this method is performed.

15.1.1. Unused acidic digestate from the preparation process and remaining in the plastic tubes on the instrument. This acidic liquid is consolidated into a plastic LLE drum. When the drum is full or after no more than 75 days, move this drum to the main waste area for shipment

15.1.2. Aqueous acidic waste from the auto-analyzer. This is collected in a 1-gallon carboy. When the carboy is full, or after no more than one year, consolidate it into a plastic LLE drum for shipment.

15.1.3. Contaminated plastic vials from digestion and analysis. Pour any excess/unused sample from the vial into the aqueous acidic waste drum. Put the plastic vial into the contaminated lab trash. Collect all of the contaminated vials in a plastic bag, and move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.1.

16.3. WS-PQA-003, Quality Control Program.

16.4. WS-PQA-004, Rounding and Significant Figures.

16.5. WS-QA-006, Method Detection Limits and Instrument Detection Limits.

16.6. WS-QA-023, Nonconformance and Corrective Action System.

16.7. WS-IP-0007, Determination of Metals Emissions From Stationary Sources

17. METHOD MODIFICATIONS

17.1. Modification from Method 7470A.

17.1.1. The method has been modified for use with Leeman automated analyzers. The samples are prepared to a total volume of 30 mL versus the 100 mL volume specified in both methods. The addition of all reagents is modified relative to this decrease in volume. Stannous chloride in HCl is used in place of Stannous sulfate in H_2SO_4 (per instrument instructions). The analytical

method is automated, whereas the reference method is written for manual CVAA analysis.

17.1.2. STLC leachate analysis has been added to the methods.

17.1.3. The five point curve used by TestAmerica Sacramento has a 0.2 ppb Hg standard to accommodate analysis of one standard at our 0.2 ppm reporting limit. The reference method starts with a 0.5 ppb Hg standard and include a 2.0 ppb Hg standard. We do not run the 2.0 ppb Hg standard.

17.1.4. This method has been modified for use with the autoclave as an alternate digestion procedure (section 10.8.9).

18. ATTACHMENTS

18.1. Appendix I - MSA Guidance

18.2. Appendix II – Autoclave Usage Letter from RCRA National Inorganic Program

18.3. Appendix III - Contamination Control Guidelines

19. REVISION HISTORY

19.1. WS-MT-0005, Revision 5.7, Effective 08/06/2018

19.1.1. Section 2.1 changed, “hydrochloride” to “sulfate”.

19.1.2. Section 2.1 changed, “nitrogen gas” to “argon gas”

19.1.3. Section 6.3 changed to, “Automated mercury analyzer. Leeman Hydra AA (or equivalent) with autosampler and WinHg Runner 1.5 software”

19.1.4. Section 6.8 changed, “Nitrogen gas supply” to “Argon gas supply”.

19.1.5. Section 9.6 added note, “The absolute vale of analyte in MB/ICB/CCB must be <1/2 LOQ, or 1/10th the amount measured in any sample, or 1/10th the regulatory limit. The recovery of the Low-level calibration standard (less than or equal to the LOQ) must be within ±20% of the true value. CCV must be within ±10% of the true value. If CCV fails the criteria, immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, repeat CCV and all associated samples since last successful CCV.”

19.1.6. Section 10.4 updated reference, “CA-Q-P-003, Calibration Curves and Selection of Calibration Points.”

19.1.7. Section 10.12 added, “or equivalent” to “An “ERTCO” or equivalent temperature logging device is put in the autoclave and undergoes the same program as the sample.”

19.1.8. Section 11.8.2.1.1 removed, “Warning: Front Half contains hydrofluoric acid).”

19.1.9. Added Section 11.8.2.1.5, “Transfer 30 mL of the Front Half digeste to a 50 mL graduated centrifuge tube. (Warning: Front Half contains hydrofluoric acid).”

19.1.10. Section 11.8.10 changed, “30 mL” to “40.5 mL (45.5 mL if additional permanganate added)”.

19.1.11. Added Section 11.9.6, “The instrument may have a memory effect after a high concentration sample is analyzed. If sample A has a concentration greater than 20 ppb and sample B is immediately analyzed after a sample A, then sample B needs to be reanalyzed.”

19.1.12. Removed revision history prior to 2015, it can be found in previous versions of this SOP.

19.1.13. Editorial changes.

19.2. WS-MT-0005, Revision 5.6, Effective 07/31/2015

19.2.1. Updated Copyright Information statement on cover pager.

19.2.2. Section 8.3 – Added, “An LCS/LCSD is required per prep batch for all air train samples.”

19.2.3. Editorial changes.

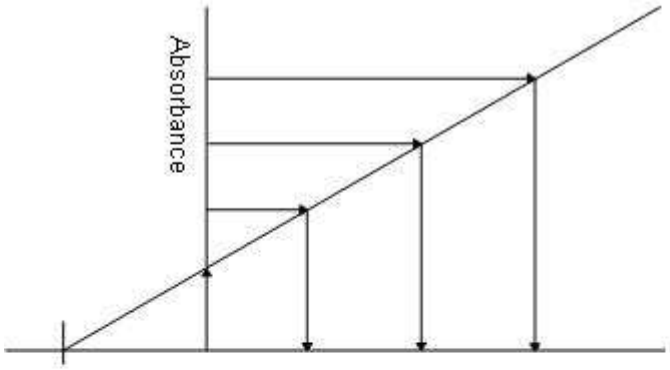
APPENDIX I. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX II
AUTOCLAVE USAGE LETTER

Ms. Debra K. White
Principal Inorganic Scientist
Quanterra Incorporated
4955 Yarrow Street
Arvada, CO 80002

Dear Deb,

Thank you for your letters of July 10, 1995 and September 25, 1995, seeking clarification on several issues regarding RCRA testing and analysis. As the Office of Solid Waste updates SW-846 methods, we will take every opportunity to incorporate your comments and suggestions. Our goal is to remove overly restrictive language from SW-846 methods, which does not effect method performance and to continue to move toward truly performance based methods.

Your first request is for a clarification regarding the acceptance of the autoclave to prepare aqueous samples for mercury analysis under EPA SW-846 Method 7470A. The heating device in Section 4.10 of the Method 7470A specifies "Hot plate or equivalent." An autoclave would classify as an equivalent heating device and should give equivalent results to the hot plate/water bath protocol called for in Sections 7.1 and 7.2 of Method 7470A. Method 245.1 CLP-M is analogous to Method 7470A and allows the autoclave option for sample preparation.

Your second request is for a clarification regarding scaling of sample size for RCRA analysis. In particular, you request processing 50 mL aliquots for aqueous metal digestion rather than the 100 mL sample size specified in the methods. This should not present any problems for pure aqueous samples (no solids) because of their homogeneous nature. As a "representative sample" can be assured, scaling causes no loss of precision or accuracy in the analysis. Solid samples on the other hand are frequently heterogeneous in nature and changing sample size presents a big problem in obtaining a "representative sample" and should not be allowed without proper sample preparation (ie. crushing, grinding, mixing, and splitting).

I hope that this information is helpful to your analytical program. If you have any questions, please feel free to call me at (202) 260-4778.

Sincerely,

Oliver M. Fordham, Jr.

National Inorganic Program
Manager for RCRA

APPENDIX III. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause-cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Company Confidential & Proprietary

Title: Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption [Methods 7471A & 7471B]

Approvals (Signature/Date):

Robert Hrabak — Date 7/2/18
Technical Manager

Joe Schairer — Date 7/2/18
Health & Safety Manager / Coordinator

Lisa Stafford — Date 6/29/2018
Quality Assurance Manager

Crystal Pollock — Date 7.2.18
Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A and SW-846 Method 7471B. These methods are applicable to the preparation and analysis for total mercury in soils, sediments, bottom deposits, and sludge materials. All matrices require sample preparation prior to analysis. When undertaking projects for Department of Defense (DoD) and/or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021 must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Samples are digested using a combination of acids and strong oxidizers, converting all forms of mercury to elemental mercury. The samples are treated with hydroxylamine hydrochloride solution to remove excess oxidizing reagents. The digestates are analyzed using an automated mercury analyzer. The mercuric ions are reduced to atomic mercury vapor with the addition of stannous chloride into a gas-liquid separator. The mercury vapor is purged into an absorption cell with Argon gas, and the relative intensity detected via a lamp/detector system. The concentration of mercury is calculated using linear regression via a computer and Leeman Labs software.

2.2. Reporting limit: 0.04 mg/Kg.

2.3. Calibration Range: 0.017 mg/Kg to 0.83 mg/Kg.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Potassium permanganate, which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. High levels of chloride can result in a positive interference from the release of free chlorine.

***Note**: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.*

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Mercury is a highly toxic element that must be handled with care. Mercury vapor is toxic, so all work must be done in a functioning fume hood and vapors must be vented into a hood. If any volume of mercury reagent or liquid mercury is spilled, the Emergency Response Team must be activated, code yellow, for spill clean-up.

5.1.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.3. Do not look directly into the beam of the mercury lamp. The UV light that these lamps generate is harmful to the eyes.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled.

5.1.5. Latex, nitrile and vinyl gloves provide adequate protection against the chemicals and reagents typically used during this process. However, if any organic solvents are used, or any sample matrix contains organic solvents, only nitrile gloves should be used.

5.1.6. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.7. Laboratory procedures such as repetitive use of pipettes, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid (3-0-1)	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydroxylamine Hydrochloride (2-0-1)	Corrosive Poison	None	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. Corrosive to the eyes. Irritant and possible sensitizer. May cause burns to the skin.
Mercury (10mg/L in stock standard, 0.1 mg/L in working standard) (3-0-0)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid (1) (3-0-2)	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

Nitric Acid (1) (4-0-1)	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate (3-0-0-0X)	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate (2-0-1-0X)	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Preventative and routine maintenance is described in the "Schedule of Routine Maintenance" in the QAM Preventative Maintenance (Section 20.2) and is listed below.

On an as needed basis:
Change pump tubing
Check/change Hg lamp
Clean optical cell
Change drying tube
Grease pump

On a daily basis:
Check sample tip for clogs
Check drying tube
Check pump tubing/drain tubing
Check gas pressure
Check liquid/gas separator
Check tubing
NOTE: Daily checks and verifications are performed prior to Instrument startup and are not documented in maintenance logs Unless problems are noted.

6.2. An autoclave capable of obtaining conditions of 15 psi at 121 ± 3°C for 15 minutes. The temperature of the autoclave is verified daily using an "LASCAR" temperature log device. In order to achieve a minimum of 15 minutes at 120 °C, the autoclave is set for a run duration of 25 minutes.

6.3. Automated mercury analyzers: HydraAA (or equivalent) with autosamplers and WinHg Runner 1.5 software, version CT Rev. 0.286 (or equivalent).

6.4. Computer and printer.

6.5. Top-loading balance capable of weighing 0.01 g

6.6. 40 mL plastic test tubes for the autosampler standards.

6.7. 14 mL test tubes for autosampler sample analysis.

6.8. 500 mL polypropylene containers.

6.9. 50 mL centrifuge tubes.

6.10. Argon gas supply.

6.11. Calibrated bottle-top dispenser.

6.12. Calibrated (daily) adjustable air displacement pipettes.

6.13. Class A volumetric flasks.

6.14. Teflon® boiling chips.

7. REAGENTS AND STANDARDS

7.1. Use reagent grade chemicals in all tests. "Certificates of Analysis" should be supplied with all chemicals purchased. If not supplied, contact the vendor. When received, label the certificate and the reagent container with the receipt date. Scan the "Certificate of Analysis" into the public drive- N:\Metals\Certificates of Analysis\Hg_COA. Login the reagent in TALS and attach the PDF document. Reagent containers also need to be labeled with the opened and expiration dates.

7.2. Reagent water is produced by a Millipore Nanopure system. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.3. All standards must be stored in polyethylene or polypropylene bottles.

7.4. 10 mg/L stock mercury calibration solution: Obtained from a commercial vendor. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year from opening, and must be replaced sooner if verification from an independent source indicates a problem.

7.5. 10 mg/L stock alternative source (ICV) mercury solution: Obtained from a different commercial vendor or with a different Lot ID from that of the calibration stock mercury solution. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year from opening, and must be replaced sooner if verification from an independent source indicates a problem.

7.6. 0.1 mg/L intermediate calibration standard: add 0.5 mL of the 10 mg/L stock calibration standard (Section 7.4) and 1 ml of concentrated HNO_3 to a 50 mL volumetric flask and dilute to volume with reagent water. This intermediate mercury standard must be made daily.

7.7. 0.1 mg/L intermediate ICV standard: add 0.5 mL of the 10 mg/L stock ICV standard (7.5) and 1 ml of concentrated HNO_3 to a 50 mL volumetric flask and dilute to volume with reagent water. This intermediate mercury standard must be made daily.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade.

7.9. Sulfuric acid (H_2SO_4), concentrated, trace metal grade.

7.10. Hydrochloric acid (HCl), concentrated, trace metal grade.

7.11. 10% HCl rinse solution: Add 250 mL of concentrated HCl to the rinse bottle and dilute to a final volume of 2.5L with reagent water.

7.12. Stannous chloride solution: Add 50 g of stannous chloride and 30 mL concentrated HCl and dilute to 500 mL with reagent water. This mixture is a suspension and should appear cloudy. This solution must be made daily.

7.13. Sodium chloride-hydroxylamine hydrochloride solution: Dissolve 240 g of sodium chloride and 240 g of hydroxylamine hydrochloride into 2000 mL of reagent water.

7.14. Potassium permanganate, 5% solution (w/v): Dissolve 100 g of potassium permanganate into 2000 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of analysis.

8.2. Solid samples should stored at 4°C ± 2°C until the time of analysis.

9. QUALITY CONTROL

9.1. Batch- A quality control batch is a set of no more than 20 field samples that consist of the same matrix and are processed using the same procedures, reagents and standards. A batch must be prepared within the same time frame. A method blank (MB) and a laboratory control sample (LCS) or duplicate control sample (LCS/LCSD) must be prepared as a part of every batch. Each batch must also be processed with a matrix spike/matrix spike duplicate (MS/SD), or in some instances a sample/sample duplicate. A 5X Serial Dilution is performed for all DOD programs. An analysis batch must include all QC samples, however they do not contribute to the maximum of 20 samples. See policy WS-PQA-003 of the Quality Control Program for more details.

9.2. One MB must be prepared for every 20 samples. A MB consists of boiling chips processed through all of the steps, and at the same time as the associated samples. If a MB exceeds ± the reporting limit, the MB should be re-run once. If the reanalysis MB still unacceptable, then all samples associated with MB must be re-prepared and reanalyzed along with other batch QC samples. The exception is that samples are less than the reporting limit and those that exceed 10X the concentration of the analyte in the MB. In such cases, the data can be reported and all corrective actions documented on a Non-Conformance memo. See policy WS-PQA-003 of the Quality Control Program for further details.

9.2.1. For samples analyzed under DoD/DOE QSM, the results of the MB must be within ± ½ the LOQ for a given analyte.

9.3. A laboratory control sample (LCS) must be prepared for every 20 samples. An LCS consists of boiling chips spiked with the analytes of interest and processed through all of the steps, and at the same time as the associated samples. If an LCS is outside of percent recovery acceptance criteria, the LCS should be re-run once. If the reanalysis is still unacceptable, all of the samples associated with that LCS must be re-prepared and reanalyzed along with other batch QC samples. The exception is when an LCS exhibits high recovery. For those samples with analyte concentrations less than the reporting limit can be reported. All corrective actions must be documented on a Non-conformance memo. See policy WS-PQA-003 of the Quality Control Program for further details.

9.4. A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair must be prepared with every process batch of similar matrix, not to exceed twenty (20) samples. MS/MSD pairs are aliquots of a selected field sample spiked with all of the analytes of interest at known concentrations. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits in the LCS. Re-extraction of the

blank, LCS, selected field samples, and the MS/MSD may be required after evaluation and review of the MS/MSD results.

***Note**: Samples identified as field blanks, equipment blanks, or trip blanks should not be used for MS/MSD analysis.*

9.5. A duplicate control sample (LCS/LCSD) may be substituted when insufficient volume is provided to process a MS/MSD pair, and batch precision is required by client or program. The LCS and LCSD are evaluated independently for acceptance. See policy WS-PQA-003 of the Quality Control Program for further details.

9.6. Quality Assurance Summaries: Certain clients may require specific project or program QA information that supersedes the SOP requirements. Quality Assurance Summaries (QAS) should be developed by the PMs to address these requirements. Sample preparation analysts are required to read and print each QAS for every project they prepare. These are transferred to the instrument analysts with the sample digestions.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution. An ICB prepared the same as a MB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. If either the ICV or ICB fail to meet criteria, the analysis must be terminated, the problem corrected, and the instrument recalibrated. If the cause of the ICV or ICB failure is not directly instrument related, the corrective action will include repreparation of the associated samples.

9.7.1. For samples analyzed under DoD/DOE QSM, the absolute value of the analyte in ICB must be less than ½ LOQ.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. Ten samples include any analysis that registers with a result, even if not used. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the possible cause of failure should be investigated, and all samples analyzed since the last acceptable CCV/CCB must be reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.8.1. For DoD/DOE QSM, the CCB results for all analytes must be less than the LOQ.

9.8.2. For samples analyzed under DoD/DoE QSM, a Low-Level Calibration check standard (LLCCV) must be analyzed before sample analysis. If the concentration of the lowest calibration standard is less than LOQ, the lowest standard may be re-quantified against the calibration curve as LLCCV. The recovery of LLCCV must within ±20% of the true value.

9.9. Dilution test (Serial Dilution)- A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5X dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50X the RL. If the results are not within 10%, the possibility of chemical or physical interference exists.

10. CALIBRATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation procedures.

10.2. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.3. Calibration standards must be processed through the preparation procedure as described in Section 11.

10.4. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards. One standard must be at the reporting limit. Analyze standards in ascending order beginning with the blank. Calibration standards must be prepared fresh daily from the 0.1 mg/L intermediate standard (Section 7.6). The standards are made by addition of the intermediate standard to 5 mL of reagent water into 50 mL centrifuge tubes. The calibration standard spike volumes and concentrations are as follows:

Calibration Standard Recipe					
Spike Volume (mL)	0.1	0.25	0.5	2.5	5.0
Concentration (ug/L)	0.2	0.5	1.0	5.0	10

10.5. The calibration curve must have a correlation coefficient of ≥0.995 and the intercept on the linear calibration must be < 0.1 ppb or the instrument shall be stopped and

recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient or an intercept on the linear calibration that is above 0.1 ppb.

10.6. The initial calibration curve is immediately followed by analysis of the initial calibration verification standard (Section 9.7). The 2.0 ug/L ICV is made by adding 5 mL reagent water to a 50 mL centrifuge tube, and adding 1.0 mL of 0.1 mg/L intermediate second source ICV standard.

10.7. The calibration is verified every 10 samples with a 5.0 ug/L CCV (Section 9.8) standard made the same way as the 5.0 ug/L calibration standard.

10.8. The autoclave is monitored daily to assure samples are digested for the proper time interval at the appropriate temperature. A "High Temperature Data Logger" temperature logging device is put in the autoclave and undergoes the same program as the samples. This device can be connected to a computer and a program generates a graph of time vs temperature. These are submitted with the raw data.

10.9. For details of the calculations used to generate the linear regression, and how to use the factors generated by the linear regression, refer to SOP CA-Q-P-003 "Calibration Curves (General)".

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. All preparation procedures must be carried out in a properly functioning hood.

11.3. All samples are to be checked out and back into sample control with the chain of custody documentation filled out completely. Samples are to be returned to the sample control area once all of the digestions have been initiated.

11.4. The proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be accurate and legible. Always set the samples up on the sample cart in the order with which they are to be dispensed.

Double-check the sample bottle IDs, once prior to weighing them into the centrifuge tubes and before they are returned to the sample cart.

11.5. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/plant), air-filters are usually logged in with solid test codes. When initiating preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as a solid but appears more like a waste (biphasic, organic liquid, etc.) or is aqueous in nature contact the lab supervisor or project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.6. Always read the QAS for every project prior to establishing batch associations. This is meant to assure that all client requirements are satisfied and it also aids in creating more efficient batches. Setting up batches to minimize QC and meet all client requirements is a skill that must be developed by all new personnel with the aid of skilled sample preparation and analytical staff.

11.7. Sample Preparation:

11.7.1. All standards are processed through the same sample preparation procedure as field samples including the initial calibration, ICV, and CCVs.

11.7.2. For solid samples, transfer approximately 0.6 (0.55~0.65) g of **homogenized** sample to a 50 mL centrifuge tube. Refer to SOP WS-QA-0018 for the proper sub-sampling technique. To assure maximum representation of the matrix, take three aliquots of approximately 0.2 g from different areas of the solid material. Attempting to measure exact weights may cause bias relative to matrix distribution therefore all of the weights are approximate. Record the exact weights on the preparation log.

11.7.3. For air-filter samples, cut 0.0833 part of the filter with a pre-measured template to achieve a RL of 0.12 ug/filter.

11.7.4. Weigh approximately 0.6g of Teflon® boiling chips into 50 mL centrifuge tubes for the MB and LCS.

11.7.5. Spike the LCS and MS/MSD with 1.0 mL of the 0.1 mg/L calibration intermediate (Section 7.6). This is a level of 2.0 ug/L in the digestate, and equivalent to 0.167 mg/kg

11.7.6. Add 5 mL of reagent water to all of the samples, and QC samples.

11.7.7. Add 2.0 mL of concentrated HNO_3 and 5.0 mL of concentrated H_2SO_4 to all of the samples and standards.

11.7.8. Add 5.0 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If after the addition of 5.0 mL additional permanganate the color does not persist, sample dilution may be required.

11.7.9. Autoclave all standards and samples for 15 minutes at 121 ± 3°C and 15 psi. Make sure the "High Temperature Data Logger" temperature-logging device is programmed and accompanies all digestions.

11.7.10. Allow the autoclave to cool to less than 80°C where the psi reading is down to zero, and add 20 mL of reagent water.

11.7.11. For samples under Incremental Sampling Method (ISM): Transfer 10 g of pre-weighed ISM sample into a 500 mL autoclavable container, spike 10 mL of 0.1 mg/L intermediate standard to LCS/MS/MSD, add 50 mL of reagent water to all samples and QCs, slowly add 50 mL concentrated sulfuric acid, 20 mL concentrated nitric acid, 50 mL of potassium permanganate solution, follow 11.7.9, add 200 mL of reagent water in 11.7.10, add 60 mL sodium chloride-hydroxylamine in 11.8 and finalized all volumes at 500 mL in 11.8.1.

WARNING: Caution must be used when opening the door of the autoclave as superheated steam may be present and can cause significant burns.

11.7.12. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.8. Sample Analysis:

11.8.1. When ready to begin analysis, add 6.0 mL of sodium chloride-hydroxylamine hydrochloride solution to all samples and standards to reduce the excess permanganate (the permanganate has been reduced when no purple color remains). Finalized all volumes (standards and samples) at 50 mL with reagent water. Mercury analysis is run using either a Hydra AA automated mercury analyzer. Results are reported mercury as ug/kg for soil, or ug/filter for air-filter.

11.8.1.1. Make certain the lamp is on and has been warmed up for a minimum of ½ hour.

11.8.1.2. Insert the line going into the injector rinse reservoir into the 10% HCl container. Insert the line going to the liquid-gas separator into the stannous chloride solution.

11.8.1.3. Adjust the stannous chloride, HCL rinse, and sample pump windings such that uptake is smooth and continuous with no pulsing. Make sure the waste pump windings are adjusted so there is no build up of liquid in the liquid-gas separator.

11.8.1.4. Load the samples into the automated sampler racks according to the sample lists entered into the computer. Do a final check once all samples have been loaded on to the racks to assure proper sequencing.

11.8.1.5. Begin the analysis.

11.8.2. Any samples with a concentration exceeding the highest calibration standard must be diluted with prepared blank solution such that the final result is within the range of the calibration curve.

11.8.3. If a sample result is negative and the absolute value of the negative result is greater than the reporting limit an interference should be suspected. Dilute the sample until it is less than the reciprocal of the reporting limit.

11.8.4. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB. Resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.9. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data is reviewed periodically throughout the run.

12. CALCULATIONS/DATA REDUCTION

12.1. ICV/CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found_{(ICV\ /\ CCV)}}{True_{(ICV\ /\ CCV)}}\right)$$

12.2. Matrix spike recoveries are calculated according to the following equation:

$$\%\ R\ =\ 100\left(\frac{SSR\ -\ SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.3. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.4. The final concentration for a soil sample is calculated as follows:

$$\mu g / Kg = C \times D \times \frac{0.05L}{W}$$

Where:

C = Concentration ($\mu g/L$) from instrument readout
D = Instrument dilution factor
W = Sample weight in Kg (0.006 Kg nominal)

12.5. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found_{(LCS)}}{True_{(LCS)}}\right)$$

12.6. Appropriate factors must be applied to sample values if dilutions are performed.

12.7. Sample results should be reported with up to three significant figures in accordance with the TestAmerica Sacramento significant figure policy (see WS-PQA-004, Rounding and Significant Figures).

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Acidic waste generated by the digestion and unused acidic digestate containing nitric and hydrochloric acid. This acidic liquid is consolidated into a plastic LLE drum in the H3 closet. When the drum is full, or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Contaminated disposable materials such as plastic vials, pipettes, and filters used during sample preparation and digestion are dumped into a yellow solid waste contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the landfill steel collection drum in the H3 closet. When the drum is full, or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Expired standards and samples containing high levels of mercury are transferred to the waste collection area for proper disposal.

16. REFERENCES/CROSS REFERENCES.

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision I, September 1994, Method 7471A (Mercury).

16.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Revision 2, February 2007, Method 7471B (Mercury).

16.3. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, 1974, Method 245.5, "Mercury in Sediment".

16.4. WS-PQA-003, Quality Control Program.

16.5. WS-PQA-004, Rounding and Significant Figures.

16.6. WS-QA-0006, Method Detection Limits and Instrument Detection Limits.

16.7. WS-QA-0023, Nonconformance and Corrective Action System.

16.8. WS-QA-0018, Sub-sampling

16.9. CA-Q-S-005, Calibration Curves (General)

17. METHOD MODIFICATIONS

17.1. Modifications from Method 7471A.

17.1.1. This method utilizes the alternative digestion procedure described in Section 11.2 of Method 7471B (Revision 2, February 2007) This procedure utilizes an autoclave rather than a hot plate (7471A), and uses sulfuric and nitric acids rather than aqua regia (a mixture of hydrochloric and nitric acids).

17.2. Modification from Method 7471A and 7471B

17.2.1. The method has been modified for use with the Leeman automated analyzer. The samples are prepared to a total volume of 50 mL with reagents, versus the 100 mL volume specified in both methods. The addition of all reagents is modified relative to this decrease in volume. Stannous chloride in HCl is used in place of stannous sulfate in H_2SO_4 (per instrument instructions). The analytical method is automated, whereas both methods are written for manual CVAA analysis.

17.2.2. The five point curve used by TestAmerica Sacramento has a 17 µg /Kg (0.2 µg/L for water equivalent) standard to accommodate analysis of one standard at our 17 µg/Kg reporting limit. Both reference methods start with an 83 µg /Kg (0.5 µg/L for water equivalent) standard.

17.2.3. Both reference methods include a 167 µg/Kg (2 µg/L for water equivalent) mercury standard. We do not run a point at this level.

18. ATTACHMENTS

18.1. Appendix 1, Contamination Control Guidelines

18.2. Appendix 2, Table of Reporting Limits for Air-filters.

19. REVISION HISTORY

19.1. WS-MT-0007, Revision 5.7, Effective 07/03/2018

19.1.1. Section 5.2, updated table with current hazard information.

19.1.2. Section 6.3 removed, “Leeman PS200II and”.

19.1.3. Removed Section 7.15, “Potassium persulfate, 5% solution (w/v): Dissolve 100 g of potassium persulfate into 2000 mL of reagent water.”

19.1.4. Section 9.2.1 changed, “RL” to “LOQ”.

19.1.5. Section 9.7 removed, “The ICB result must fall within ± the reporting limit (RL) from zero for DoD QSM”.

19.1.6. Added Section 9.7.1, “For samples analyzed under DoD/DOE QSM, the absolute value of the analyte in ICB must be less than ½ LOQ.”

19.1.7. Section 9.8.1 revised to, “For samples analyzed under DoD/DoE QSM, the absolute value of the analyte in CCB must be less than ½ LOQ.”

19.1.8. Added Section 9.8.2, “For samples analyzed under DoD/DoE QSM 5.1, a Low-Level Calibration check standard (LLCCV) must be analyzed before sample analysis. If the concentration of the lowest calibration standard is less than LOQ, the lowest standard may be re-quantified against the calibration curve as LLCCV. The recovery of LLCCV must within ±20% of the true value.”

19.1.9. Section 10.4 changed, “24 mL” to “5 mL”.

19.1.10. Section 10.4 removed, “Remove the spike volume from the 24 mL of reagent water before the addition of the intermediate standard.”

19.1.11. Section 10.4 removed note after Table, “Note: After addition of all reagents the final volume is 50 mL.”

19.1.12. Section 10.6 changed, “24 mL” to “5 mL”.

19.1.13. Section 10.6 removed, “withdrawing 1.0 mL reagent water”.

19.1.14. Section 11.7.2 revised to, “For solid samples, transfer approximately 0.6 (0.55~0.65) g of **homogenized** sample to a 50 mL centrifuge tube.”

19.1.15. Section 11.7.6 revised to, “Add 5 mL of reagent water to all of the samples, and QC samples.”

19.1.16. Section 11.7.8 removed note, “When reporting analyses, the addition of excess reagent must be addressed through mathematical correction of the results to account for the resultant dilution effect.”

19.1.17. Removed Section 11.7.9, “Add 8.0 mL potassium persulfate solution.”

19.1.18. Section 11.7.10 added, “and add 20 mL of reagent water.”

19.1.19. Added Section 11.7.11, “For samples under Incremental Sampling Method (ISM): Transfer 10 g of pre-weighed ISM sample into a 500 mL autoclavable container, spike 10 mL of 0.1 mg/L intermediate standard to LCS/MS/MSD, add 50 mL of reagent water to all samples and QCs, slowly add 50 mL concentrated sulfuric acid, 20 mL concentrated nitric acid, 50 mL of potassium permanganate solution, follow 11.7.9, add 200 mL of reagent water in 11.7.10, add 60 mL sodium chloride-hydroxylamine in 11.8 and finalized all volumes at 500 mL in 11.8.1.”

19.1.20. Section 11.8.1 added, “Finalized all volumes (standards and samples) at 50 mL with reagent water.”

19.1.21. Section 15.1 revised “extraction” to “digestion”.

19.1.22. Editorial changes.

19.2. WS-MT-0007, Revision 5.6, Effective 02/14/2017

19.2.1. Section 9.2 – inserted "…the MB should be re-run once. If the reanalysis MB still unacceptable, then all samples associated with MB must be re-prepared and reanalyzed along with other batch QC samples"

19.2.2. Section 9.3 – inserted, "…recovery acceptance criteria, the LCS should be re-run once. If the reanalysis is still unacceptable, all of the samples associated with that LCS must be re-prepared and reanalyzed along with other batch QC samples."

19.2.3. Inserted Section 11.7.3 – "For air-filter samples, cut 0.0833 part of the filter with a pre-measured template to achieve a RL of 0.12 ug/filter. "

19.2.4. Editorial changes.

19.3. WS-MT-0007, Revision 5.5, Effective 11/13/2016

19.3.1. Section 10.5 – Changed the last word of the last sentence from 1 ppb to 0.1 ppb.

19.3.2. Editorial changes.

19.4. WS-MT-0007, Revision 5.4, Effective 08/21/2015

19.4.1. Updated copyright statement on cover page.

19.5. Section 10.5 – Added to line 1, "and the intercept on the linear calibration must be less than 0.1 ppb…", and added to line 2, "…or an intercept on the linear calibration that is above 1ppb."

19.5.1. Editorial changes.…

19.6. WS-MT-0007, Revision 5.3, Effective 01/07/2014

19.6.1. Revision history prior to 2010 has been removed and is available in prior versions of this SOP.

19.6.2. Title changed to reference methods 7471A and 7471B.

19.6.3. Section 1.1 – Inserted reference to Method 7471B.

19.6.4. Added Section 11.10 – Routine and Preventative Maintenance for the Mercury Analyzer.

19.6.5. Section 17.1 revised to read, “This method utilizes the alternative digestion procedure described in Section 11.2 of Method 7471B (Revision 2, February 2007) This procedure utilizes an autoclave rather than a hot plate (7471A), and uses sulfuric and nitric acids rather than aqua regia (a mixture of hydrochloric and nitric acids).”

19.6.6. Section 17.2 inserted (“Modifications from Methods 7471A and 7471B”), and Section 17.1.2 through 17.1..4 grouped under Section 17.2.

19.6.7. Reference to EPA 7471B has been added.

19.7. WS-MT-0007, Revision 5.2, Effective 01/20/2012

19.7.1. Section 6.14: added “Teflon® boiling chips”.

19.7.2. Section 11.7.3: added “Weigh approximately 0.6g of Teflon® boiling chips into 50 mL centrifuge tubes for the MB and LCS.”

APPENDIX 1
CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross-contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX 2

Table 1, Reporting limits (RLs) for air-filters

Sample portion	RL
0.0833 part of filter (3/4" cut)	0.12 ug/filter (or sample)
0.125 part of filter(1" cut for special project only)	0.08 ug/filter (or sample)
1 cassette	0.01 ug/filter
1 cassette (sample volume in L provided by client) MB/LCS using 1000 L for calculation purpose	0.00001 ug/L (MB/LCS)

Title: Incremental Sampling Methodology of Soils and Sediments

Approvals (Signature/Date):

Koroush Vaziri Department Manager	2/9/2018 Date
Joe Schairer Health & Safety Manager / Coordinator	2/9/18 Date
Lisa Stafford Quality Assurance Manager	2/8/18 Date
Crystal Pollock Laboratory Director	2.9.18 Date

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1. SCOPE AND APPLICATION

1.1. The purpose of this procedure is to obtain sub-samples from client provided samples which represent the concentration of material in the entire parent sample.

1.2. This SOP describes the procedures for laboratory staff to follow during the preparation of samples for the incremental sampling methodology (ISM) procedure. These are guidelines for the preparation and sub sampling of soil/solid/sediment samples to be analyzed for routine organic and inorganic analyses.

1.3. The incremental sampling methodology sub sampling procedures are not applicable to volatile soil samples collected on Encore® samples for Method 5035. These are discrete samples and the entire sample is used for analysis.

2. SUMMARY OF METHOD

2.1. Samples received from the field may require processing including drying, removal of extraneous material, and sieving to be performed for different analyses so that a representative concentration can be determined. An entire client sample is first processed and the sample is then sub sampled using an incremental sampling methodology approach.

2.2. Care should be taken to ensure that these subsamples are representative of the component samples and are properly prepared and stored in accordance with the appropriate method of analysis.

2.3. The basic formula to use when working with clients to select the optimal approach for other methods or other precision objectives is given in Appendix 1 to this SOP. The Appendix defines the trade off between subsample size, particle size, and the desired level of precision.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. ISM – Incremental Sampling Methodology

3.4. Dry – sample(s) require drying.

3.5. Disaggregate – The act of breaking the soil clumps into individual small particles, but keeping the small pebbles and hard crystalline particles intact for sample(s) that require grinding with mortar and pestle or ball mill.

3.6. Sieve – sample(s) require sieving (default #10 2mm sieve)

3.7. Puck Mill – sample(s) require puck mill procedure

3.8. Ball Mill – sample(s) require ball mill procedure

3.9. Split – dividing a sample in half or smaller segments, but typically larger than analytical subsamples. There are several process options such as riffle splitter, rotary sectorial splitter and most commonly 1D slabcake. For Sacramento splitting has the same procedure as subsampling, therefore treat splitting as subsampling.

3.10. Subsample (also referred to as 2D slabcake) – sample(s) require subsampling to procure sample weights using the ISM.

3.11. As Received – sample(s) do not require drying

4. INTERFERENCES

4.1. If the samples are to be analyzed for metals testing, check the login/method/sample comments and with the PM to find out if the brass sieves (Teflon® screens are an alternative) and the aluminum foil (butcher paper is an alternative) used to spread samples out for drying are going to be problematic to the client.

4.2. Some clients require the use of Teflon® coated spatulas instead of the regular sterile sample scoops (polystyrene) due to the possibility of contamination. These Teflon® spatulas should be washed and decontaminated as per method requirements of the tests in question.

4.3. Interferences can occur when using scoops or spatulas. All scoops or spatulas shall be used for only one sample and then disposed, or thoroughly cleaned between samples. Material that may be acceptable for one analysis may cause contamination for another analysis. All plastic should be avoided if organic parameters are requested.

4.4. On rare occasions a client may ask for ISM processing for volatile samples. Please confirm with project manager that the request is correct. If so, then follow these guidelines. Volatile analytes may be lost during subsampling from non-Encore containers. Subsampling for volatile analyses should be done from a previously unopened container to the end of tube (where possible), and subsampling should be done as quickly as possible to avoid analyte loss.

4.5. Volatile and light semi-volatile analytes may be lost during the sample drying and grinding procedure. Consult the appropriate analytical SOP and QAS for guidance on the required drying and grinding procedure for the samples.

4.6. When the procedure is used to process samples for PFC samples, the following precautions must me used to prevent contamination or inferences. No Teflon® or Teflon® coated materials may be used. Samples should be dried on non-coated paper

and sieved using brass sieves. Subsampling must be performed using scoops or spatulas made of Teflon®- free materials (metal, HDPE, polystyrene or wood). Store subsamples in plastic jars that do not have Teflon lined lids.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Extensive homogenization, subsampling, and/or compositing of soil/solid/waste or liquid samples presents an extreme risk of repetitive motion injuries for the individual performing the operation. No single employee will homogenize, subsample, or composite these types of samples for longer than one hour continuously without taking a five-minute break away from this type of work and stretching his/her hands, wrists and arms. If the manager/supervisor and the employee involved identify at the start of the process that the work will take longer than one hour, the employee should take mini-breaks of 2-3 minutes every 25-30 minutes. If there is extensive homogenization, subsampling, and/or compositing that must be performed, or if it is extremely time sensitive, managers/supervisors must assign additional personnel to the effort, or rotate different staff members through the job in order to prevent injury to any employee.

5.1.2. The use of the Pulverizing Mill (ring and puck) or the Ball Mill during this process presents an extreme risk of damage to the ears/hearing loss. Ear muffs must be worn by all persons in the room when either the puck mill or the ball mill is in operation, and the hallway door must be closed when either the puck mill or the ball mill is in operation. It is recommended that persons in the room wear disposable ear plugs under the ear muffs.

5.1.3. The heavy weight of the puck and bowl (ring) used in the process presents an extreme risk of injury due to the number of times they must be moved back and forth between the mill, the hood and the sink, and body position and exertion while cleaning them in the sink. Ensure that associates assigned this task use proper lifting procedures, and take sufficient breaks during the process to allow back, neck and shoulder/arm muscles to recover. If the work being done will take longer than one hour, a 2-3 minute stretch break shall be taken every 25-30 minutes. Managers/supervisors will strongly consider

rotating this task between multiple associate to dissipate the impact on any single associate. This is especially significant if work may extend over multiple days.

5.1.4. The opening and closing process for the paint cans used during Ball Mill operations present serious risk for repetitive motion injuries, impact injuries and lacerations. Following the instructions in paragraph 5.1.1 will reduce the risk of repetitive motion injuries. When adding soil/solid sample material to the paint can, ensure that no sample material falls into the groove in the top of the can that the lid will be fitted into. This will ensure that sample material does not fly out of the groove when the lid is seated, and also ensures that the lid seats easily. Use only the rubber mallet to seat the lid into the groove, not your hand or any type of metal hammer, and ensure that your hand is well away from the impact point of the mallet. When opening paint cans after the ball mill operation is complete, do not use screwdrivers, spatulas, knives or any tools other than the commercial paint can openers that have been provided. Do not keep the hand that is not holding the paint can opener over or right next to the opener while in use in case it slips.

5.1.5. If sediment/soil samples have been frozen in glass jars, the freezing process may have cracked the jars when the sample expanded while freezing. After the samples have thawed, wear cut protective gloves while handling the jars until it can be confirmed that they have not cracked.

5.1.6. Any alternative procedures requested by a client must be reviewed by EH&S before they are put into practice.

5.1.7. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide sufficient protection when handling closed sample containers and most typical samples. Unusual or heavily contaminated samples must be evaluated to determine if there are any hazards for which a particular type of glove will not be appropriate.

5.1.8. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred, sub sampled, and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.2. Primary Materials Used

There are no materials used in this method, which would have a serious or significant hazard rating.

6. EQUIPMENT AND SUPPLIES

6.1. Shallow metal trays, approximately 18” wide by 30” long.

6.2. Inert tray lining – depending on the analytes requested, either of these two materials may be used to line the trays in 6.1:

6.2.1. Aluminum foil (not used if tests for metals are requested).

Note: As food-grade aluminum foil is often coated to alleviate sticking, it is not recommended when perfluorinated compounds are requested.

6.2.2. Unbleached Kraft (butcher) paper

To line the trays, create a “weigh boat” of either foil or paper, approximately 18 inches by 30 inches with a small lip around the perimeter. Place this on the tray prior to spreading sample material on it.

6.3. Sieves, various sizes, including 2mm (#10 sieve).

6.3.1. Sieves may be brass or patches of Teflon screening.

When perfluorinated compounds are requested, use the brass sieves only.

6.4. Teflon® coated spatulas – do not use if perfluorinated compounds are requested.

6.5. Sterile sample spoons (that are not rounded on the sampling side), made of polystyrene.

6.6. Tongue depressors

6.7. Analytical balance.

6.8. Mortar and pestle (manual and automated).

6.9. Sample containers, various sizes, glass and poly.

6.10. Fume hood.

6.11. Rubber mallets.

6.12. Commercial paint can opener.

Note: *Some samples such as biological tissues and pulp and paper products may require pre-preparation before subsampling or compositing. See the appropriate SOP for matrix specific procedures.*

6.13. Pulverizing Mill (ring and puck), ESSA model LM2-P or equivalent - for the grinding of soils per method 8330B

6.13.1. The grinding bowl and puck are cleaned after each use by washing with soap and water, rinsing with hot tap water, rinsing with DI water, and then rinsing with acetonitrile. A final wipe down of the bowl and puck while still wet with acetonitrile is done with a Kimwipe (TNT in particular is reported to be prone

to adhering to steel surface). In addition, sand blanks are used to monitor potential carry-over for each batch of samples.

6.13.2. After a grinding blank, the first step may be omitted: washing with soap and water.

6.14. Ball Mill (US Stoneware or equivalent) – for the disaggregation of solid samples.

6.14.1. 1 gallon and ½ gallon new paint cans are used (Manufactured by CL Smith). ½ gallon part number 12-2350ALL, ½ gallon lid 12-2400ALL.01. 1 gallon part number 12-1427STD.01 (includes lid). Paint cans are to be used one time then discarded.

6.14.2. The ball mill grinding material is manufactured by E.R. Advance Ceramics, part number BRUN125-90 (90% Burundum Grinding Media 1-1/4" x 1-1/4"). Grinding material can be reused after the cleaning procedure described in section 11.7.1.

7. REAGENTS AND STANDARDS

Reagents used for rinsing equipment are indicated in method SOPs.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

All component, subsamples, and composites will be stored in compliance with the analytical method under which they will be analyzed.

9. QUALITY CONTROL

Samples used for Matrix Spike and Matrix Spike Duplicates (MS/MSDs) and sample duplicate and sample triplicates should be homogenized and sub sampled using the same procedure incremental sampling methodology as the parent sample(s).

10. CALIBRATION

10.1. Balances used for subsampling or compositing for analysis and preparation should be calibrated as per SOP WS-QA-0041.

10.2. Balances used for non-analytical subsampling, i.e., for trans-shipment, do not require calibration as the weight is a rough value only.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor

and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. This method is dependent on the client project provided Data Quality Objectives. Depending on the nature of the project, samples may need to be dried, extraneous material may need to be removed and replicates may need to be run per sample or per batch of samples. It is important that the analyst confirm with the Project Manager prior to performing this procedure. Any project specific changes or modifications to the procedure should be noted in the form of a client specific amendment to the SOP or in the Quality Assurance Summary (QAS). The procedures documented below incorporate the commonly performed procedure. There are two primary procedures to consider, sample prep and subsampling.

11.3. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

Note: *Drying, sieving or subsampling for volatile analyses may lead to loss of analytes or contamination from common laboratory solvents. Any incremental sampling methodology procedures for volatiles should be performed in a solvent-free area and the mixing should be minimized to reduce the loss of volatile analytes. The potential for loss of volatile analytes should be discussed with the client before initiation of the program.*

11.4. Determine the required Incremental Sampling Methodology using the method code (see definitions above). The order in which the steps are stated are the order in which the steps need to be performed. For samples that have multiple requested methods, perform the method with the less rigorous steps first followed by the ISM with the more rigorous steps without repeating steps that were already performed. For example, if ISM_DD_SI_SS and ISM_DD_SI_PM_SS are both requested on a sample, first dry, then disaggregate, sieve, split/subsample, then puck mill the remainder and split/subsample. For another example, ISM_R_SS (wet) is done before ISM_DD_SI_SS (dry). Anytime wet ISM is requested before drying, make sure that % moisture is also subsampled.

11.4.1. ISM_DD_SI_SS – Dry, Disaggregate, Sieve, 2D slabcake, Subsample

11.4.2. ISM_DD_SI_PM_SS – Dry, Disaggregate, Sieve, Puck Mill, 2D slabcake, Subsample

11.4.3. ISM_DI_SI_SS – As received, Disaggregate, Sieve, 2D slabcake, Subsample

11.4.4. ISM_DI_SI_PM_SS – As received, Disaggregate, Sieve, Puck Mill, 2D slabcake, Subsample

11.4.5. ISM_DD_SP – Dry, Disaggregate, Split

11.4.6. ISM_DR_PM_SP – Dry, Puck Mill, Split

11.4.7. ISM_DI_SP – As Received, Disaggregate, Split

11.4.8. ISM_PM_SP = As Received, Puck Mill, Split

11.4.9. ISM_CUSTOM and ISM_CUST_WS – Custom ISM procedure. Consult the PM. Default to ISM_DD_SI_SS if there are no method notes.

11.4.10. ISM_R_SS – As Received, 2D slabcake, subsample

Before the first ISM step, weigh the entire sample. If the sample weighs more than 1.1kg, notify the project manager of the weights.

Note: *For QSM samples, record the initial weight of the sample on the "ISM Drying and Additional Information" form, QA-526B (Appendix 6).*

11.5. For samples that require Custom ISM

11.5.1. For Custom ISM samples, consult the comment section of the backlog, project documents, and if needed discuss with the project manager for specific instructions.

11.5.2. For Custom ISM- Homogenization

11.5.2.1. Do Not Dry Samples

11.5.2.2. Homogenize/Disaggregate the entire container using ball mill or mortar and pestle.

Sample aliquots are taken from the homogenized sample volume for each analysis. 2D slabcake ISM is not required for this procedure.

11.6. For samples that require drying

11.6.1. Spread the entire sample evenly on a tray lined with aluminum foil or butcher paper that is inert material and is free from any analytes of interest or interferences.

11.6.1.1. For samples that require 6010/6020/7471 aluminum foil cannot be used. For all analysis, butcher paper can be used.

11.6.1.2. For samples that require VOAs and % moisture, ensure the % moisture for VOA is done 'As Received' since VOA samples are preserved in MeOH.

11.6.1.3. Samples that are extremely wet or have considerable mass may required drying on multiple lined trays and re-combined later.

WARNING: If sediment/soil samples have been frozen in glass jars, the freezing process may have cracked the jars when the sample expanded while freezing.

Wear cut protective gloves while handling the jars until it can be confirmed that they have not cracked.

11.6.2. Samples should be placed on bakers racks or another location which allows for proper ventilation, and allowed to air-dry at room temperature. For very wet samples, it may be necessary to place the sample in a fume hood with the sash lowered to improve airflow over the sample. The sample should be dried until visibly dry and if uncertain a % moisture aliquot can be taken using the ISM to ensure dryness. Ensure to record the start and end date, time, and analyst initial in the QA-562 document (Appendix 4).

11.6.2.1. For QSM samples, the samples need to be dried till they are a consistent weight at room temperature (record the room temperature on form QA562-B). Consistent weight is determined by weighing an approximately 10g aliquot of the sample twice with 2 hours between measurements. If the weight difference is ≤5%, the sample is considered dry. If the difference is greater than 5%, the sample is dried for another two hours and this is repeated till the sample weight is consistent. The consistent weight measure is documented on form QA-562-B.

11.7. For samples that require disaggregating and sieving, splitting/subsampling.

11.7.1. Adequately decontaminate necessary equipment (sieves, pestles, mortars, grinding material) by brushing and washing with soap and water and rinsing with the appropriate solvent or reagent as outlined in the analytical procedure. Acetonitrile is an appropriate solvent for all analysis.

11.7.2. For samples requiring disaggregation/sieving

11.7.2.1. Break up any soil aggregate material with a clean object such as a mortar and pestle or ball mill (Section 11.8). Employ de-aggregation techniques if needed. Occasionally using a sterile scoop or tongue depressor may be necessary if the sample is sticky and a mortar and pestle is not effective. Ensure to disaggregate the entire sample. This may require repeating this step once with the materials that would not pass through a sieve.

11.7.2.2. Carefully sieve the sample using a 2mm sieve (#10), or the appropriate size as designated in the method method/login/sample comments. In some cases a smaller mesh size sieve may need to be used following 2mm sieve. Clients may ask to weigh the individual portions for particle size analysis.

Note: *For samples that require 6010/6020/7471 brass sieves may* ***not*** *be used. For samples that require PFC analysis (perfluorinated compounds) Teflon® sieves may* ***not*** *be used.*

11.7.2.3. Transfer the materials with particle size greater than the designated particle size (sieve mesh) into a second container, or dispose of as outlined in Section 15 if the client does not want to save the material. If the unsieveable material is collected in a secondary container, ensure it gets properly labeled by sample control and returned to a storage location for disposal at a further date.

11.7.2.4. For QSM samples, record the total weight of the dried and sieved material and the unsieved waste material and document it on form QA-562B.

11.7.3. For samples requiring subsampling/splitting

11.7.3.1. Subsample in duplicate only if the aliquot is to be sent out to a sister lab, dried after wet subsampling or Puck Mill will later be required. Ensure there is adequate volume left after subsampling for methods that will follow. If not, notify the project manager. Omission of the duplicate may be required.

11.7.3.2. Evenly distribute the sample into a weigh boat made out of butcher paper or aluminum foil (do not use aluminum foil if 6010/6020/7471 or perfluorinated compounds are requested). The sample layer should have a depth of approximately one-half inch or less to allow for sampling throughout the entire depth of the sample layer. If the depth is more the approximately one-half inch, split the sample evenly across a minimum amount of weigh boats.

11.7.3.3. Perform the subsampling by forming an imaginary 6x5 grid to visualize 30 squares in the sampling tray. Take one scoop using a sterile sample spoon (client may request use of Teflon® coated spatulas) from each square ensuring the aliquot is from the top to the bottom of the soil. (An unaltered tongue depressor may not be used because the rounded tip will not collect evenly from the top to the bottom of the sample). Each aliquot should contain approximately $1/30^{th}$ of the desired target mass. Collect the aliquot fractions in the appropriate containers. See Summary Sheet (Appendix 5) for targeted mass, appropriate containers for various analyses, and additional notes for each analysis. Ensure to include a MS/MSD for each preparation batch for each test, a sample duplicate and sample triplicate for 8330B, and a minimum of one MS/MSD pair per 10 samples for 6020 and 6010. For QSM samples, a duplicate and triplicate needs to be subsampled for every 20 samples.

11.7.3.4. In some cases with a smaller sample size, a 6x5 grid is not possible. Create a similar grid and subsample 30 times as evenly as possible.

11.7.3.5. If the final amount is less than the targeted amount, subsample from

random locations until the targeted amount is obtained.

11.7.3.6. If the final amount is greater than the targeted amount, pour the sample back and re-sample.

11.7.3.7. Ensure to record the initial, balance ID, and mass in the QA 562 (Appendix 4) document. For 8330B after puckmill, record the same data in 8330B Compositing/Incremental Sampling Methodology **(QA-702)** (Appendix 2).

11.7.3.8. Store the sub samples in the proper location for the test being conducted until they are ready for further preparation.

11.8. For samples that require ball mill (disaggregation)

11.8.1. Start with a dried sample, a new paint can (labeled with sample ID sticker and permanent marker), and clean grinding material.

11.8.1.1. Paint cans are ready to use from the manufacturer and are used only one time then discarded.

WARNING: When adding soil/solid sample material to the paint can, ensure that no sample material falls into the groove in the top of the can that the lid will be fitted into. Use only the rubber mallet to seat the lid into the groove, and ensure that your hand is well away from the impact point of the mallet. When opening paint cans after the ball mill operation is complete, only use the the commercial paint can openers that have been provided.

WARNING: The use of the Ball Mill during this process presents an extreme risk of damage to the ears/hearing loss. Ear muffs must be worn be all persons in the roof when the Ball Mill is in operation, and the hallway door must be closed while the mill is in operation. It is recommended that persons in the room wear disposable ear plugs under the ear muffs.

11.8.2. Load the sample in the paint can. Half gallon cans can not be more than 2/3rd full and 1 gallon cans can not be more than 3/4th full. If needed, use two or more cans for one sample.

11.8.3. Place 3-4 clean grinding material stones in the half gallon cans and 4-5 in the 1 gallon can. Seal the lid with the rubber mallet and ensure that it is fully closed.

11.8.4. Load the cans onto the ball mill and start the rotation. Document the start time on the ISM sheet (QA-562).

11.8.5. Rotate the sample for a minimum of 2 hours. If the samples are not rotating

properly on the ball mill, they can be taped together with duct tape to ensure proper rotation. Make sure to tape the cans with the lids facing inward.

11.8.6. After removing the sample from the ball mill, record the time off in the ISM sheet (QA-562). Open the paint can in a hood and visually inspect the sample to ensure that it has been adequately disaggregated. If the sample is not adequately disaggregated after two hours, the sample can continue to rotate for up to 8 hours.

11.8.7. Transfer the material to an appropriate container or proceed with sieving and subsampling the sample (section 11.7.2 to 11.7.3).

11.9. For samples that require puck mill

11.9.1. Decontamination should occur prior to and after grinding of samples.

WARNING: The heavy weight of the puck and bowl (ring) used in the process presents an extreme risk of injury due to the number of times they must be moved back and forth between the mill, the hood and the sink, and body position and exertion while cleaning them in the sink. Ensure that associates assigned this task use proper lifting procedures, and take sufficient breaks during the process to allow back, neck and shoulder/arm muscles to recover. If the work being done will take longer than one hour, a 2-3 minute stretch break shall be taken every 25-30 minutes. Managers/supervisors will rotate this task between multiple associates to dissipate the impact on any single associate. This is especially significant if work may extend over multiple days.

11.9.2. Wash bowl (ring) and puck with laboratory-grade detergent (such as Liquinox) and water using steel wool pad on large surfaces and a wire brush in grooves, corners, etc. Rinse bowl and puck well with hot water and then rinse with DI water. Next, rinse with Acetonitrile and wipe down with a large Kim Wipe or paper towel. If sample residue is visible on Kim Wipe after wiping bowl and puck, further washing may be needed (there may be a reddish residue from rust or a grayish residue from the metal objects which does not require further washing). After a grinding blank, the first step of washing the bowl and puck with detergent and water with steel wool may be omitted.

***Note**: The minimum and maximum allowed sample size for this method (and grinder capacity) is 200-500g.*

11.9.3. Prior to grinding samples, a grinding blank must be created using 200g of Ottawa Sand. A grinding blank must also be created in between each sample.

WARNING: The use of the Pulverizing Mill (ring and puck) during the process presents an extreme risk of damage to the ears/hearing loss. Ear muffs must be worn by all persons in the room when the puck mill is in operation, and the

hallway door must be closed while the mill is in operation. It is recommended that persons in the room wear disposable ear plugs under the ear muffs.

11.9.4. To grind, add sample to the bowl and pulse six times in one minute intervals, with two minutes between pulses. To begin pulsing, close lid, open compressed air valve to 80psi, and start the mill. Some samples may need to be ground in separate fractions if the entire sample does not fit in the bowl.

11.9.4.1. Ensure to record the date, initial, puck, and start times on the Puck Mill (Appendix 3) sheet (QA-703).

11.9.5. Place the ground sample into appropriate container, decontaminate ring and puck, and create another grinding blank before moving on to the next sample. The ground sample is ready for the subsample/split (see 11.6.3).

11.9.6. Subsample approximately 10g of each grinding blank for each puck for all samples created in a preparation batch ensuring to include the grinding blank from before the first sample using the subsampling procedure described in WS-QA-0018. Using the ISM described in 11.6.5.2 and 11.6.5.3, subsample the composite grinding blank to be included within each preparation batch.

11.9.6.1. To name a grinding blank, increment in number for each grinding blank and addend the end with the puck mill letter. For example, if Puck A had the first grinding blank, then another grinding blank, then Puck B had a grinding blank then the first grinding blank from puck A will be named 1A, the second grinding blank from puck A will be named 2A, the first grinding blank from puck B will be named 3B etc.

11.9.6.2. When compositing the grinding blanks, ensure to document each grinding blanks name, grinding blank start date, and grinding blank start time subsampled in the document 8330B Compositing/Incremental Sampling Methodology (Appendix 2) sheet. Name the grinding blank composite "composite" followed by incrementing or each grinding blank composite. For example, the first grinding blank subsampled from puck 1A and 2A will be called "composite 1", the second grinding blank subsampled from 3B and 4B will be called "composite 2" etc.

11.9.6.3. For QSM samples, a grinding LCS is required for each method. Please consult with the project manager or client if a Standard Reference Material (SRM) is not commercially available for the method requested.

11.10. Batch all samples

11.10.1. Using the method codes above, batch all samples into the appropriate method. For samples with puck mill, ensure to include the grinding blank composite

sample(s) and grinding LCS. Use 'GB' as the sample ID for TALs and in the sample comments include which composite it is. For the grinding LCS, use "LCS" as the sample ID. No raw data needs to be entered. Ensure the batch start and end time is from the time the sample originally was started to the final ISM and ensure the samples are set to the batch time.

11.10.2. Scan all documents associated with each batch into the extractions folder under PM group. Rename file "zbatchnumber" and attach to the document tab in the batch in TALs.

11.10.3. Print out labels or the analytical batch paperwork. Ensure to make copies of the documents containing the weights for each analysis. 1st and 2nd level review the batch and deliver the paperwork and samples to each department with the requested analysis.

11.11. All jars created with ISM must be labeled with a TALS label prior to being placed in the refrigerator. For sendouts, use the color code system found in CA-T-WI-011 (ISM Workshare Communication).

12. CALCULATIONS/DATA REDUCTION

This section is not applicable to this procedure.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Contaminated disposable materials such as plastic vials, pipettes, empty sample containers, unused/excess sample matrix, and disposable spatulas. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When

the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. STL White Paper "Representative Sub-sampling Techniques for Inorganic Analytes", February 23, 2004.

16.2. "Guidance for Obtaining Representative Laboratory Analytical Sub samples from Particulate Laboratory Samples," USEPA, November 2003.

16.3. "Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities" ASTM D 6323-98 (Reapproved 2003)

16.4. EPA SW-846 Method 8330B. Nitroaromatics, Nitramines, and Nitrate esters By High performance Liquid Chromatography (HPLC). Revision 2, October 2006.

16.5. "Incremental Sampling Methodology [ISM] Program Minimum Standards", 05/9/2012 Revision 2

17. METHOD MODIFICATIONS

17.1. There are no deviations from the method unless otherwise specified by the QAS and checked with the group/team lead.

18. ATTACHMENTS

18.1. Appendix 1 – Consideration of Fundamental Error in Selecting ISM Options

18.2. Appendix 2 – 8330B Compositing/Incremental Sampling Methodology

18.3. Appendix 3 – Puck Mill

18.4. Attachment 4 – QA-562

18.5. Attachment 5 – Summary

18.6. Appendix 6 – ISM Drying and Additional Info

19. REVISION HISTORY

19.1. WS-QA-0028, Revision 4.6, Effective 02/09//2018

19.1.1. Added Section 3.8, "Ball Mill – sample(s) require ball mill procedure".

19.1.2. Added Section 5.1.4, "The opening and closing process for the paint cans used during Ball Mill operations present serious risk for repetitive motion injuries, impact injuries and lacerations. Following the instructions in paragraph 5.1.1 will reduce the risk of repetitive motion injuries. When adding soil/solid sample material to the paint can, ensure that no sample material falls into the

groove in the top of the can that the lid will be fitted into. This will ensure that sample material does not fly out of the groove when the lid is seated, and also ensures that the lid seats easily. Use only the rubber mallet to seat the lid into the groove, not your hand or any type of metal hammer, and ensure that your hand is well away from the impact point of the mallet. When opening paint cans after the ball mill operation is complete, do not use screwdrivers, spatulas, knives or any tools other than the commercial paint can openers that have been provided. Do not keep the hand that is not holding the paint can opener over or right next to the opener while in use in case it slips."

19.1.3. Added Section 6.12, "Ball Mill (US Stoneware or equivalent) – for the disaggregation of solid samples."

19.1.4. Added Section 6.12.1, "1 gallon and ½ gallon new paint cans are used (Manufactured by CL Smith). ½ gallon part number 12-2350ALL, ½ gallon lid 12-2400ALL.01. 1 gallon part number 12-1427STD.01 (includes lid). Paint cans are to be used one time then discarded."

19.1.5. Added Section 6.12.2, "The ball mill grinding material is manufactured by E.R. Advance Ceramics, part number BRUN125-90 (90% Burundum Grinding Media 1-1/4" x 1-1/4"). Grinding material can be reused after the cleaning procedure described in section 11.7.1."

19.1.6. Added Note after Section 11.4.10, "For QSM samples, record the initial weight of the sample on the "ISM Drying and Additional Information" form, QA-562B (Appendix 6)."

19.1.7. Added Section 11.6.2.1, "For QSM samples, the samples need to be dried till they are a consistent weight at room temperature (record the room temperature on form QA562-B). Consistent weight is determined by weighing an approximately 10g aliquot of the sample twice with 2 hours between measurements. If the weight difference is ≤5%, the sample is considered dry. If the difference is greater than 5%, the sample is dried for another two hours and this is repeated till the sample weight is consistent. The consistent weight measure is documented on form QA-562-B."

19.1.8. Added Section 11.7.2.4, "For QSM samples, record the total weight of the dryied and sieved material and the unsieved waste material and document it on form QA-562B."

19.1.9. Added to Section 11.7.3.3, "For QSM samples, a duplicate and triplicate needs to be subsampled for every 20 samples."

19.1.10. Added Section 11.8, "For samples that require ball mill (disaggregation)", and subsections 11.8.1 through 11.8.7, which describe the current procedure for samples requiring ball mill disaggregation.

19.1.11. After Section 11.8.1 added warning, "**WARNING: When adding soil/solid sample material to the paint can, ensure that no sample material falls into the groove in the top of the can that the lid will be fitted into. Use only the rubber mallet to seat the lid into the groove, and ensure that your hand is well away from the impact point of the mallet. When opening paint cans after the ball mill operation is complete, only use the the commercial paint can openers that have been provided.**"

19.1.12. After Section 11.8.1 added warning, "**WARNING: The use of the Ball Mill during this process presents an extreme risk of damage to the ears/hearing loss. Ear muffs must be worn be all persons in the roof when the Ball Mill is in operation, and the hallway door must be closed while the mill is in operation. It is recommended that persons in the room wear disposable ear plugs under the ear muffs.**"

19.1.13. Added Section 11.9.6.3, "For QSM samples, a grinding LCS is required for each method. Please consult with the project manager or client if a Standard Reference Material (SRM) is not commercially available for the method requested."

19.1.14. Revision history prior to 2015 has been removed, it is available in previous revisions of this SOP.

19.1.15. Removed 537/537 modified, 1311 (TCLP), 1312 (TCLP), 8081 (3546), and 8082 (3546) from Appendix 5 table.

19.1.16. Added Appendix 6, "ISM Drying and Additional Information for QSM Samples".

19.1.17. Editorial changes.

19.2. WS-QA-0028, Revision 4.5, Effective 03/02/2017

19.2.1. Section 5.1.2, added the following: "The heavy weight of the puck and bowl (ring) used in the process presents an extreme risk of injury due to the number of times they must be moved back and forth between the mill, the hood and the sink, and body position and exertion while cleaning them in the sink. Ensure that associates assigned this task use proper lifting procedures, and take sufficient breaks during the process to allow back, neck and shoulder/arm muscles to recover. If the work being done will take longer than one hour, a 2-3 minute stretch break shall be taken every 25-30 minutes. Managers/supervisors will strongly consider rotating this task between multiple associate to dissipate the impact on any single associate. This is especially significant if work may extend over multiple days."

19.2.2. Section 11.4, added the following: "For another example, ISM_R_SS (wet) is done before ISM_DD_SI_SS (dry). Anytime wet ISM is requested before

drying, make sure that % moisture is also subsampled."

19.2.3. Added Section 11.4.10 to read "ISM_R_SS = As Received, 2D slabcake, subsample."

19.2.4. Added Section 11.5, titled "For samples that require Custom ISM," including sections 11.5.1 through 11.5.2.

19.2.5. Section 11.7.3.1, amended to read: "Subsample in duplicate only if the aliquot is to be sent out to a sister lab, dried after wet subsampling or Puck Mill will later be required."

19.2.6. Section 11.7.3.1, added the following: "Ensure there is adequate volume left after subsampling for methods that will follow. If not, notify the project manager. Omission of the duplicate may be required."

19.2.7. Section 11.8.1, added the following after the section: "**WARNING: The heavy weight of the puck and bowl (ring) used in the process presents an extreme risk of injury due to the number of times they must be moved back and forth between the mill, the hood and the sink, and body position and exertion while cleaning them in the sink. Ensure that associates assigned this task use proper lifting procedures, and take sufficient breaks during the process to allow back, neck and shoulder/arm muscles to recover. If the work being done will take longer than one hour, a 2-3 minute stretch break shall be taken every 25-30 minutes. Managers/supervisors will strongly consider rotating this task between multiple associates to dissipate the impact on any single associate. This is especially significant if work may extend over multiple days."**

19.2.8. Section 11.9.2, removed the following: "For level 3 and level 4 projects attach the document to the raw or prep data for an analysis requested. Attach the batch to TALS and the deliverable." Added the following: "Rename file 'zbatchnumber' and attach to the document tab in the batch in TALS."

19.2.9. Section 11.10, added the following: "For sendouts, use the color code system found in CA-T-WI-011 (ISM Workshare Communication)."

19.2.10. Appendix 2, uploaded version 1.

19.2.11. Appendix 4, uploaded version 4.

19.2.12. Appendix 5, for test 8081 and 8082 added "(3550)"; for 8081, 8082, 8270, and TPH, AK102/AK103, and 8270 changed "French Square" to "Clear tall (8 oz.)."

19.2.13. Appendix 5, added 8081 (3546) and 8082 (3546).

19.2.14. Editorial changes.

19.3. WS-QA-0028, Revision 4.4, Effective 07/15/2016

19.3.1. Appendix 5 – Added 1311 (TCLP) and 1312 (SPLP) methods, container types, and amounts to Table.

19.3.2. Editorial changes.

19.4. WS-QA-0028, Revision 4.3, Effective 07/06/2016

19.4.1. Amended Appendix 5 to include Method 537 (PFCs) with note to avoid use of Teflon.

19.4.2. Editorial changes.

19.5. WS-QA-0028, Revision 4.2, Effective 06/22/2016

19.5.1. Added Section 4.6 to read: "When the procedure is used to process samples for PFC samples, the following precautions must me used to prevent contamination or inferences. No Teflon® or Teflon® coated materials may be used. Samples should be dried on non-coated paper and sieved using brass sieves. Subsampling must be performed using scoops or spatulas made of Teflon®- free materials (metal, HDPE, or wood). Store subsamples in plastic jars that do not have Teflon lined lids."

19.5.2. Following Section 6.2.1, added note to read, "Note: As food-grade aluminum foil is often coated to alleviate sticking, it is not recommended when perfluorinated compounds are requested."

Section 6.3.1, appended the following: "When perfluorinated compounds are requested, use the brass sieves only."

19.5.3. Section 6.4, appended the following, "– do not use if perfluorinated compounds are requested."

19.5.4. Note following 11.6.2.2, changed to read, "Note: For samples that require 6010/6020/7471 brass sieves may **not** be used. For samples that require PFC analysis (perfluorinated compounds) Teflon® sieves may **not** be used."

19.5.5. Section 11.6.3.2, first sentence changed to read, "Evenly distribute the sample into a weigh boat made out of butcher paper or aluminum foil (do not use aluminum foil if 6010/6020/7471 or perfluorinated compounds are requested)."

19.5.6. Editorial Changes.

19.6. WS-QA-0028, Revision 4.1, Effective 03/18/2016

19.6.1. Updated copyright paragraph on cover page.

19.6.2. Section 11.8.3 - added to the end of Section - "Attach the batch to TALS and

the deliverable."

19.6.3. Added Section 11.9 - "All jars created with ISM must be labeled with a TALS label prior to being place in the refrigerator."

19.6.4. Editorial changes.

19.7. WS-QA-0028, Revision 4.0, Effective 3/02/2015

19.7.1. Added Section 2.3 - The basic formula to use when working with clients to select the optimal approach for other methods or other precision objectives is given in Appendix 1 to this SOP. The Appendix defines the trade off between subsample size, particle size, and the desired level of precision.

19.7.2. Added Section(s) 3.3 – 3.10 to Definitions Section

19.7.3. Edited Sections 6.1 and 6.2 to include more detail.

19.7.4. Modified Section 11.4 – Determining Incremental Sampling Methodology.

19.7.5. Modified Section 11.5 – For samples that require drying.

19.7.6. Modified Section 11.6 – For samples that require disaggregating and sieving, splitting/subsampling

19.7.7. Modified Section 11.7 – For samples that require puck mill.

19.7.8. Added Section 11.8 – Batching all samples

19.7.9. Added Appendixes 2 – 5

19.7.10. Editorial changes.

Appendix 1

Consideration of Fundamental Error in Selecting ISM Options

The following formula given in ASTM D-6323 was used to produce the table that follows.

$$S^2 = 18 * f * e * d^3 / M_s$$

where,

S^2 = the relative variance of the contaminant concentration due to the fundamental error

f = shape factor, a dimensionless number, a value of 0.5 can be taken as typical (Pierre Gy, 1982)

e = the population's average density (g/cm^3). For this table a typical soil density of 2.5 g/cm^3 was used.

d = the diameter of the largest particle in centimeters, and

M_s = the mass of the sample in grams

Sample Mass and Maximum Particle Size to Achieve a Desired RSD

Subsample Mass (g)	Sieve Size (US Standard Mesh)	At 5% RSD Max Size (cm)	At 10% RSD Max Size (cm)	At 15% RSD Max Size (cm)
0.1	35	0.02	0.04	0.05
1	18	0.05	0.08	0.10
2	13	0.06	0.10	0.13
5	12	0.08	0.13	0.17
10	10	0.10	0.16	0.22
30	7	0.15	0.24	0.31
50	6	0.18	0.28	0.37
100	5	0.22	0.35	0.46

Appendix 2 (QA-702)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Page __ of __ Batch _____

Sacramento
8330B Compositing/Incremental Sampling Methodology

CRM Lot # _____

Sample ID		Date of ISM / Weighing (Initials)	Balance ID	8330B	Sample ID		Date of ISM / Weighing (Initials)	Balance ID	8330B	Composite #	GB #	GB Start Date	GB Start Time	Composite #	GB #	GB Start Date	GB Start Time
Login #	Smp #			10±0.1	Login #	Smp #			10±0.1								

Comments: Samples weighed using the Incremental Sampling Methodology (ISM) from puck mill samples.

_____

_____

QA-702 r1
RKE 11/08/2016

Appendix 3 (QA-703)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento
Puck Mill Record

Page __ of __Batch_____

Sample ID		Puck (circle one)	Date of Puck Mill & Initials	Pulse 1 Start time	Pulse 2 Start time	Pulse 3 Start time	Pulse 4 Start time	Pulse 5 Start time	Pulse 6 start time	Pulse 6 end time
Login #	Smp/ GB #									
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								
		A / B								

Comments:

Appendix 4 (QA-562)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Page __ of __Batch(es)_____

Sacramento
Incremental Sampling Methodology

Sample ID		Date/Time/ Initials Sample Drying Started	Date/Time/ Initials Sample Drying Ended	Date/ Initial of ISM/ Weighing	Balance ID	Sieve Size	Project Specific Data (Method and Required Sample Mass in Header) Record Final Mass of Each Requested Test. Primary on top left, backup on bottom right.									
							TPH	8081	8082	8290	6010	6020	7471			
Login #	Smp #						30 ±1g	30 ±1g	30 ±1g	10 ±0.5g	10 ±0.2g	10 ±0.2g	10 ±0.2g			

Comments: Subsample(s) created using the Incremental Sampling Methodology (ISM)_____

Particle Size: C = Coarse = 10 mesh (2mm)
F= Fine = 30 mesh (0.6 mm)

QA-562 r4
ERS 01/13/2016

Appendix 5
Sacramento Incremental Methodology Summary

Test	Container	Weight	Notes
8081 (3550)	Clear Tall 8oz	30±1g	When both 8081/8082 are requested, ISM only one 30±1g aliquot to be used for both tests. Perform a MS/MSD pair for both 8081 and 8082. Also called pesticides. For prep method 3546, use 15±1g.
8082 (3550)	Clear Tall 8oz	30±1g	When both 8081/8082 are requested, ISM only one 30±1g aliquot to be used for both tests. Perform a MS/MSD pair for both 8081 and 8082. Also called PCBs. For prep method 3546, use 15±1g.
8270-SIM	Clear Tall 8oz	10±1g	Also called PAH-SIM
TPH	Clear Tall 8oz	30±1g	Also called TEPH/TPH/8015
%Moisture	Snapcap	11±6g	Ensure to do a sample duplicate but no MS/MSD. The weight amount can be altered if insufficient sample size as long as it is representative.
6850	Centrifuge Tube	3±0.1g	Use a VOA vial for the backup container
8330A	VOA vial	2±0.1g	NA
8330B	VOA vial	10±0.1g	Should have a MS/MSD/Sample Duplicate/Sample Triplicate/Grinding LCS/GBs, check with PM or DM if the method is not ISM_DD_SI_PM_SS
6010	Snapcap	10±0.2g	Ensure a minimum of one MS/MSD pair per 12 samples
6020	Snapcap	10±0.2g	Ensure a minimum of one MS/MSD pair per 12 samples
7471	Centrifuge Tube	10±0.2g	Ensure a minimum of one MS/MSD pair per 12 samples
8290	Snapcap	10±0.5g	When both 8290/1668 are requested, ISM only one 20±1g aliquot to be used for both tests. A MS/MSD is required only if the samples are QSM work. The MS/MSD can be shared between both tests as well.
1668	Snapcap	10±0.5g	When both 8290/1668 are requested, ISM only one 20±1g aliquot to be used for both tests. A MS/MSD is required only if the samples are QSM work. The MS/MSD can be shared between both tests as well.
AK102/AK103	Clear Tall 8oz	30±1g	Check with DM to determine whether one MS/MSD pair or two MS/MSD pairs are needed
8270	Clear Tall 8oz	30±1g	NA
8280	Snapcap	10±0.2g	A MS/MSD is required only if the samples are QSM work.
8151	125 AGJ	50±1g	Extraction not performed locally. Ensure to do in duplicate with a MS/MSD pair. Label containers with labels from sample control. Try and get closer to 51g.

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Appendix 6 (QA-562B)
ISM Drying and Additional Information for QSM Samples

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento
ISM Drying and Additional Information

Page ____ of ____ Batch(es)

Drying Room Temperature °C:	Obs	Thermometer ID:
	High	
	Lo	

Sample ID		Consistent Weight Measurements (taken from a ~10g subsample)				
Login #	Sample #	Initial Weight/Time taken	Weight after 2hr/ Time taken	Weight after 4hr if needed/ Time taken	Balance ID	Initials and Date

Q: Document-Management/Forms/QA-562B r0 ISM Drying and Additional Info

QA-562B r1
RE 11/22/2017

Quality Assurance Manual Cover Page

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Title Page:

Quality Assurance Manual

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REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-I-P-002	Electronic Reporting and Signature Policy
CA-I-S-006	Software Testing, Validation and Verification
CW-I-P-006	Computer Systems Account and Naming Policy
CW-I-P-007	Computer Systems Password Policy
CA-L-P-002	Contract Compliance Policy
CA-Q-M-002	Corporate Quality Management Plan
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-006	Detection Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Corporate Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CW-F-S-007	Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation s
CW-L-S-004	Subcontracting
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recalls
CA-C-S-001	Work Sharing Process

REFERENCED LABORATORY SOPs

SOP Reference	Title
WS-PQA-013	Procedures to Address Customer Complaints
WS-QA-0050	Management of Change
WS-QA-0009	Document Archiving
WS-QA-0022	Employee Orientation and Training
WS-QA-0021	Preparation and Management of Standard Operating Procedures
WS-QA-0006	Method Detection Limits (MDL) and Instrument Detection Limits (IDL)
WS-PQA-0011	Manual Integration Documentation Procedures
WS-PQA-003	Quality Control Program
WS-QA-0018	Subsampling and Compositing of Samples
WS-QA-0003	Sample Receipt and Procedures
WS-QA-0035	Statistical Process Control / Control Chart
WS-EHS-0001	Waste Disposal

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Sacramento's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*
- *U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.*
- *U.S. Department of Denfense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013.*
- *U.S. Department of Denfense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.1, 2017.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Statement of Work for Inorganics & Organics Analysis, SOM, ISM, DLF and CBC, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th, 21st, and on-line Editions.
- *U.S. Department of Energy Order 414.1B, Quality Assurance, Approved April 29, 2004.*
- *U.S. Department of Energy Order 414.1C, Quality Assurance, June 17, 2005.*

- *U.S. Department of Energy Order 414.1D, Quality Assurance, April 25, 2011*
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge, tissue and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & Updating procedures (refer to SOP No. WS-QA-0021).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Sacramento is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., Chief Executive Officer, Executive VP Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Sacramento is presented in Figure 4-1.

4.2 Roles and Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Sacramento laboratory.

4.2.2 President and Chief Executive Officer (CEO)

The President and CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. The President and CEO establishes the overall quality standard and data integrity program for the Analytical Business, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.3 Senior Vice President of Operations (SVPO)

The COO reports directly to the President and CEO of TestAmerica. The SVPO~~VPO~~ oversees the operations of all TestAmerica. The VP's of Operations report directly to the SVPO.

4.2.4 Vice President of Operations

Each VP of Operations reports directly to the Senior VP of Operations and is a part of the Executive Committee. Each VP of Operations is responsible for the overall administrative and operational management of their respective laboratories. The VP's responsibilities include allocation of personnel and resources, long-term planning, goal setting, and achieving the financial, business, and quality objectives of TestAmerica. The VP's ensure timely compliance with Corporate Management directives, policies, and management systems reviews. The VP's are also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

4.2.5 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to the President and CEO. With the aid of the Executive Committee, Laboratory Directors, Quality Directors, Safety Manager, EH&S Coordinators and QA Managers, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and EH&S Programs within TestAmerica. Additional responsibilities include:

- Review of QA/QC and EHS aspects of Corporate SOPs & Policies, national projects and expansions or changes in services.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the analytical laboratories and a summary of any quality related initiatives and issues.
- Preparation of a monthly report that includes EH&S metrics across the analytical laboratories and a summary of any EH&S related initiatives and issues.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

4.2.6 Vice President of Client Service

The VP of Client Services leads the Client Service Organization (CSO) and is responsible for client satisfaction, driving operational excellence and improving client responsiveness. The VP provides direction to the Client Service Directors, Programs Managers and Project Managers.

4.2.7 Quality Assessment Director

The Quality Assessment Director reports to the VP-QA/EHS. The Quality Assessment Director has QA oversight of laboratories; responsible for the internal audit system, schedule and procedure; monitors laboratory internal audit findings; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Compliance Director, the Quality Systems Director, and the VP-QA/EHS, the Quality Assessment Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.8 Quality Compliance Director

The Quality Compliance Director reports to the VP-QA/EHS. The Quality Compliance Director has QA oversight of laboratories; monitors and communicates DoD / DoE requirements; develops corporate tools for ensuring and improving compliance; develops corporate assessment tools; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Systems Director and the VP-QA/EHS, the Quality Compliance Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.9 Quality Systems Director

The Quality Systems Director reports to the VP-QA/EHS. The Quality Systems Director has QA oversight of laboratories; develops quality policies, procedures and management tools; monitors and communicates regulatory and certification requirements; identifies common laboratory weaknesses; and monitors corrective action closures. Together with the Quality Assessment Director, Quality Compliance Director and the VP-QA/EHS, the Quality Systems Director has the responsibility for the establishment, general overview and maintenance of the Analytical Quality Assurance Program within TestAmerica.

4.2.10 Quality Information Manager

The Quality Information Manager is responsible for managing all company official documents (e.g., Policies, Procedures, Work Instructions), the company's accreditation database, intranet websites, external laboratory subcontracting, regulatory limits for clients on the company's TotalAccess website; internal and external client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions. The Quality Information Manager reports to the VP-QA/EHS; and works alongside the Quality Assessment, Quality Compliance and Quality System Directors and EHS Managers to support both the Analytical Quality Assurance and EHS Programs within TestAmerica.

4.2.11 Technical Services Director

The Technical Services Director is responsible for establishing, implementing and communicating TestAmerica's Analytical Business's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

4.2.12 Ethics and Compliance Officers (ECOs)

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – Corporate Counsel & VP of Human Resources and the VP-QA/EHS. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President and CEO, VPOs, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.13 Chief Information Officer (CIO)

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

4.2.14 Environmental Health and Safety Managers (Corporate)

The EHS Managers report directly to the VP-QA/EHS. The EHS Managers are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.15 Laboratory Director

TestAmerica Sacramento's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the laboratory and reports to their respective VP of Operations. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Providing one or more technical managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence

exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.

- Ensuring that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensuring that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensuring TestAmerica's human resource policies are adhered to and maintained.
- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensuring that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits.
- Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviewing and approving all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursuing and maintaining appropriate laboratory certification and contract approvals.
- Supporting ISO 17025 requirements.
- Supporting DoD/DOE ELAP requirements.
- Supporting The NELAC Institute (TNI) Standard requirements
- Ensuring client specific reporting and quality control requirements are met.
- Directing the management team, consisting of the QA Manager, the Operations Manager, the EH&S Coordinator and the Office Manager as direct reports.

4.2.16 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system. The QA Manager reports directly to the Laboratory Director and their Corporate Quality Director. This person is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. This person has documented training and/or experience in QA/QC procedures and the laboratory's Quality System. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Serving as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.

- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintaining records of all ethics-related training, including the type and proof of attendance.
- Maintaining, improving, and evaluating the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Reviewing external audit reports and data validation requests.
- Following-up with audits to ensure client QAPP requirements are met.
- Establishing reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Developing suggestions and recommendations to improve quality systems.
- Researching current state and federal requirements and guidelines.
- Directing the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring communication with laboratory staff and monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluating of the thoroughness and effectiveness of training.
- Assuring compliance with ISO 17025.
- Assuring compliance with DoD/DOE ELAP.
- Assuring compliance with The NELAC Institute (TNI) Standard.

4.2.17 Technical Manager (Manager of Operations) or Designee

The Technical Manager(s) (noted as Manager of Operations on the organizational chart) report(s) directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i.e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design versus demonstrated versus first run yield) utilization.

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.

- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.

- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.

- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.

- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.

- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.

- Directing department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.

- Complying with ISO 17025, The NELAC Institute (TNI) Standard, DoD/DOE ELAP and the various QC programs implemented at the Sacramento laboratory.

4.2.18 Client Services Manager

The CSM reports directly to the Client Service Director (Western Region) and indirectly to the Laboratory Director. The CSM serves as the interface between the laboratory's Project Management team, technical departments, and clients. The CSM shall:

- Oversee training and growth of the Project Management team.
- Act as technical liaison for the Project Management team.
- Provide human resource management support to the Project Management team.
- Assist PMs with responses to client inquiries or with resolutions to problems or complaints.
- Ensure that client specifications, when known, are met by communicating project and QA requirements to the laboratory.
- Notify Department Managers or supervisors of incoming projects and sample delivery schedules.
- Discuss with client any project-related problems, resolve service issues, and coordinate technical details with the laboratory staff.
- Monitor the status of projects in-house to ensure timely and accurate delivery of reports.
- Prepare price quotes or project bids.

4.2.19 Manager of Project Managers

The Manager of Project Management reports to the Regional Client Services Director and serves as the interface between the laboratory's technical departments and the laboratory's clients. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the duties of this position are outlined below:

- Managing technical training and growth of the Project Management team
- Serving as technical liaison for the Project Management team
- Providing human resource management of the Project Management team
- Ensuring that clients receive the proper sampling supplies
- Overseeing response to client inquiries concerning sample status
- Assisting clients regarding the resolution of problems concerning COC
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory
- Notifying the supervisors of incoming projects and sample delivery schedules
- Being accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates

- Discussing with clients any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff
- Providing information to staff with respect to specific quotes, sample log-in review, and final report completeness
- Monitoring the status of all data package projects in-house to ensure timely and accurate delivery of reports
- Informing clients of data package-related problems and resolve service issues
- Coordinating requests for sample containers and other services (data packages)

4.2.20 Project Manager

Project Managers are a liaison between the laboratory's clients and the analytical staff. They report directly to the Manager of Program Management. The Project Managers have signature authority for final reports, and review project data packages for completeness and compliance with client needs and quality requirements.
The Project Manager's responsibilities include:

- Ensuring client specifications are met by communicating project and quality assurance requirements to the laboratory
- Notifying laboratory personnel of incoming projects and sample delivery schedules
- Monitoring the status of all projects in-house to ensure timely delivery of reports
- Informing clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff
- Coordinating client requests for sample containers and other services
- Scheduling sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples
- Coordinating subcontract work
- Assisting clients in procuring the proper sampling supplies
- Responding to client inquiries concerning sample status
- Assisting clients with resolution of problems concerning Chains-of-Custody
- Invoicing completed data packages
- Generating credit or debit invoices to ensure proper payment

4.2.21 Project Administrator

The Project Administrator reports to the Manager of Project Management and designated Project Manager. The Project Administrator assists the Project Manager in servicing the client's needs and communicating those needs to the laboratory. The Project Administrator's responsibilities include:

- Collating data reports, expanded deliverables, and electronic data deliverables (EDDs) for delivery to clients.
- Writing case narratives accompanying data packages to communicate anomalies to clients
- Coordinating client requests for sample containers and other services
- Assisting clients in procuring the proper sampling supplies
- Assisting Project Managers in changing compound lists, TAT, and other LIMS set up tasks.
- Monitoring report due dates for timely delivery
- Invoicing completed data packages
- Generating credit or debit invoices to ensure proper payment

4.2.22 Department Manager, Team Leader, or Supervisor

Department Managers report directly to the Operations Manager. They supervise the daily activities of analysis within a given laboratory area, and either oversee the review and approval, or perform the review and approval of all analytical data within that area.

Specific responsibilities include, but are not limited to:

- Exercising day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results.
- Ensuring that analysts in their department adhere to applicable SOPs and the QA Manual.
- Coordinating the writing and reviewing of documentation for all test methods, i.e., SOPs, with regard to quality, integrity, regulatory requirements and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity includes insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances, improved LIMS utilization, capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Coordinating audit responses with the QA Manager.

- Complying with ISO 17025, The NELAC Institute (TNI) Standard, DoD/DOE QSM and the various QC programs implemented at the Sacramento laboratory.
- Participating in the selection, training (familiarization with SOP, QC, Safety and computer systems), developing performance objectives and standards of performance, appraising (measurement of objectives), scheduling, counseling, disciplining, and motivating analysts and documenting these activities in accordance with systems developed by the QA and Human Resources Departments.
- Evaluating staffing sufficiency and overtime needs.
- Encouraging the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Providing guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and corrective actions, the timely and accurate completion of performance evaluation samples and MDLs, for his/her department.
- Ensuring all logbooks are maintained, current, reviewed, and properly labeled or archived.
- Reporting all non-conformance conditions to the QA Manager, Operations Manager, and/or Laboratory Director.
- Ensuring that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/She has responsibility for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintaining adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieving optimum turnaround time on analyses and compliance with holding times.
- Conducting efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long term needs for budgetary planning.

4.2.23 Analyst

Analysts report to their respective Department Managers. They perform sample analyses and generate analytical data in accordance with documented procedures.

The responsibilities of the analysts are listed below:

- Collecting and preparing materials and supplies for the laboratory
- Retrieving samples from Sample Control for analysis

- Performing sample preparation by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Documenting standard and sample preparation, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Reporting all non-conformance situations, sample preparation problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Performing 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggesting method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Providing ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Working cohesively as a team member in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.24 Sample Custodian

The Sample Custodian ensures the implementation of proper sample receipt procedures, including maintaining chain-of-custody. The Sample Custodian logs samples into the LIMS and ensures that all samples are stored appropriately. Duties for the Sample Custodian include the following:

- Receiving and unloading samples or consignments in accordance with DOT regulations
- Verifying samples against the Chain of Custody (COC)
- Logging samples into the LIMS to assign a lot number for tracking purposes, and notifying Project Managers of any irregularities with the sample shipment.
- Labeling samples with lot number assigned and deliver the samples to the appropriate labs for analysis daily
- Monitoring freezer and cooler temperatures daily to confirm that the readings are within SOP guidelines
- Shipping all subcontracted samples to designated lab in accordance with DOT regulations as needed.

4.2.25 Quality Assurance Staff

The Quality Assurance staff members report to the QA manager. They have responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025, through involvement in the following activities:

- Assisting the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- Facilitating external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.
- Assisting the QA Manager in the preparation of new SOPs and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- Managing the performance testing (PT) studies, coordinating follow-up studies for failed analytes, and working with QA Manager and Laboratory Staff to complete needed corrective action reports.
- Serving as a project manager for proficiency testing samples and other QC samples.
- Reviewing and maintaining personnel training records.
- Assisting the QA Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- Managing certifications and accreditations.
- Monitoring for compliance with the following QA Metrics: Temperature Monitoring of refrigeration units; thermometer verifications and calibrations; balance verifications and callibrations; and Eppendorf/pipette calibrations.
- Periodically checking the proper use and review of logbooks.
- Assisting in the technical review of data packages which require QA review.
- Assisting the QA Manager in maintaining the laboratory's reference data to keep it current and accurate.
- Preparing certification applications for states as directed by QA Manager.
- Reviewing and maintaining personnel training records.
- Performing document control maintenance.
- Assisting departments in generating MDL spreadsheets and calculations, reviewing MDL studies submitted to QA.
- Assisting in control limit generation.
- Ensuring maintenance of records archives.

- Maintaining historical indices for all technical records including SOPs, QC records, laboratory data, etc.
- Assisting the QA Manager in meeting the responsibilities of the QA Department as described in laboratory policies and SOPs.

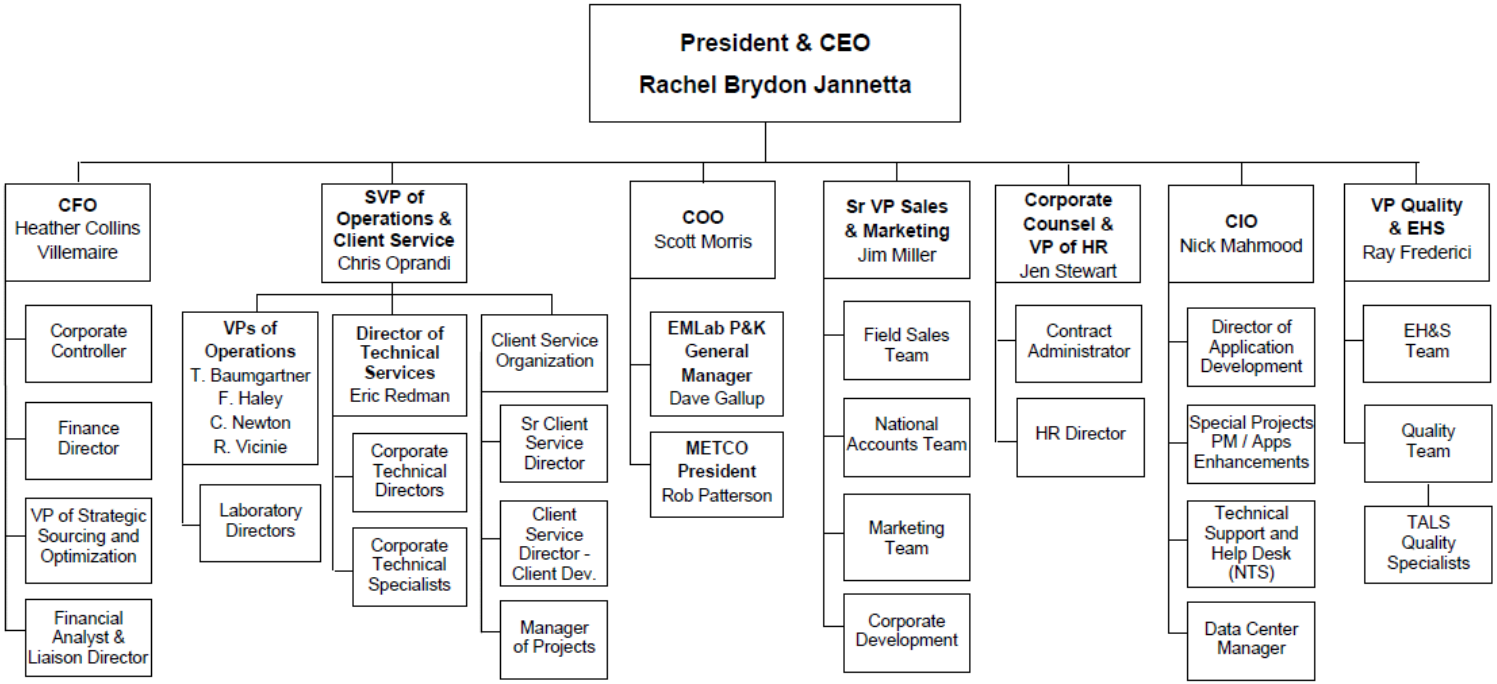
4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Crystal Pollock - Laboratory Director	Robert Hrabak - Technical Director, Manager of Dioxins, LCMS & Inorganics
Lisa Stafford - Quality Assurance Manager	Russell Evans - Quality Assurance Staff Crystal Pollock - Laboratory Director
Robert Hrabak - Technical Director, Manager of Dioxins, LCMS & Inorganics	Crystal Pollock - Laboratory Director
Victoria Nihart – Manager of Semivolatiles, & Organic Prep	Robert Hrabak - Technical Director, Manager of Dioxins, LCMS & Inorganics Crystal Pollock - Laboratory Director
Jill Kellmann - Manager of Project Management	David Herbert - Client Relations Manager (Corporate)
Joe Schairer - EHS Coordinator	Crystal Pollock – Lab Director

Figure 4-1. Corporate and Laboratory Organization Charts

All organizational charts are current as of the date noted. Contact the laboratory for the most recent organizational chart.



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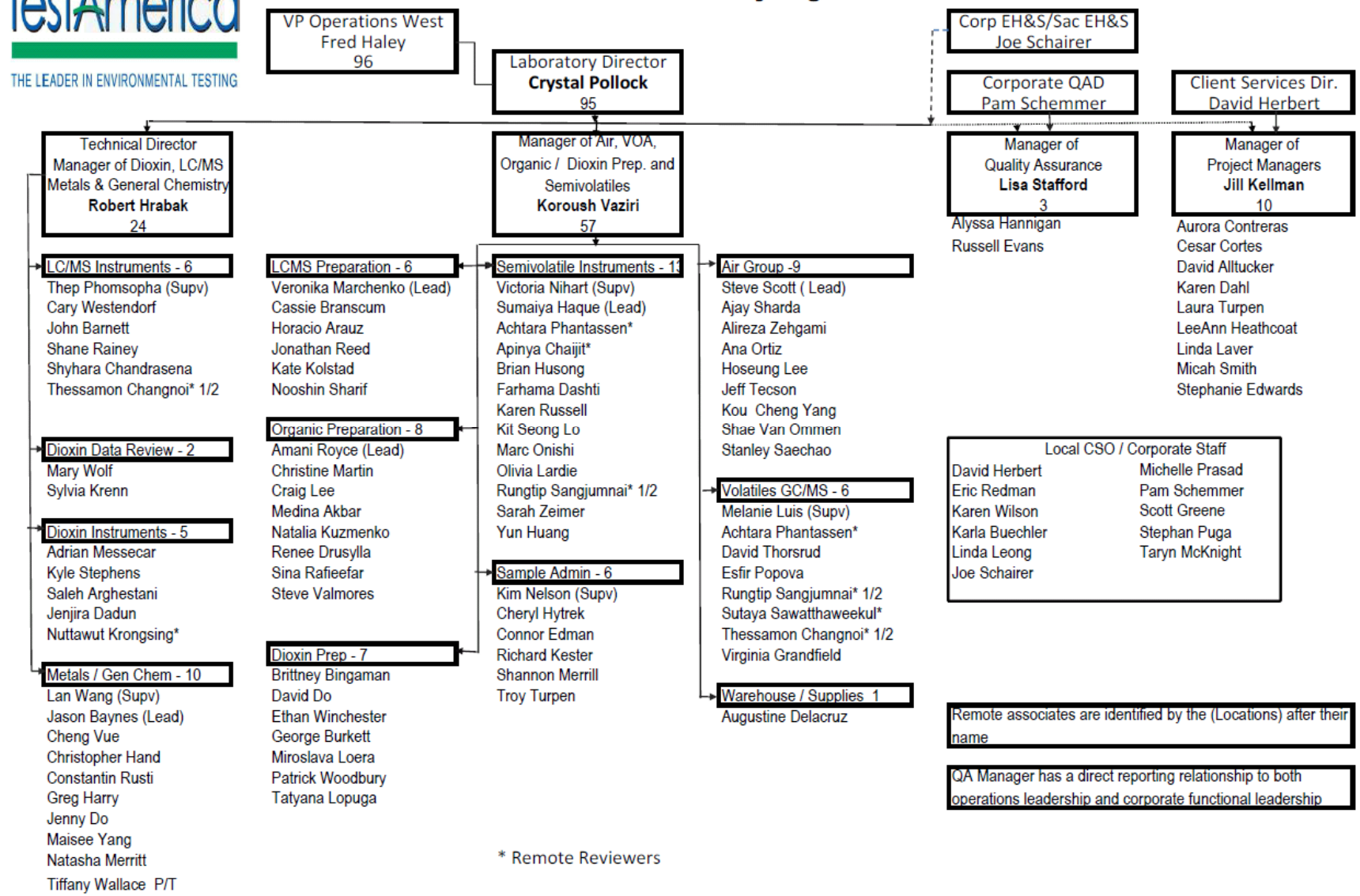
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento Laboratory Organization



SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- ❖ Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- ❖ Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- ❖ Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- ❖ Provide clients with the highest level of professionalism and the best service practices in the industry.
- ❖ Comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 Ethics and Data Integrity

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct (Corporate SOP No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-L-S-002).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.

- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterizes the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), inter-element corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 Criteria for Quality Indicators

The laboratory maintains Reference Data in the LIMS that summarizes the precision and accuracy acceptability limits for performed analyses. This data includes an effective date, is updated each time new limits are generated and is managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. The criteria for development of control limits is contained in SOP WS-QA-0035, "Statistical Process Control / Control Chart" and Section 24.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Manager and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in SOP WS-QA-0035, "Statistical Process Control / Control Chart" and Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. Control charts are generated according to laboratory SOP No. SOP WS-QA-0035, "Statistical Process Control / Control Chart"

5.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

6.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled

documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years (annually for documents applicable to drinking water and DoD/DOE programs), and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the QA share on the local server for the applicable revision, and are accessible using the laboratory's Intranet.

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP and SOP No. WS-QA-0021, Preparation and Management of Standard Operating Procedures". The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by department in the QA office. There is a table of contents. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP No. WS-QA-0021, "Preparation and Management of Standard Operating Procedures".

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. WS-QA-0021, Preparation and Management of Standard Operating Procedures.

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. The PM will also get approval by the Laboratory Director to commit to delivery schedules that are shorter than the published standard turnaround times (TATs). The Laboratory Director updates these TATs on a routine basis, and it is the responsibility of CSMs and PMs to review them prior to making commitments for the laboratory.

It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Contract Administrator
- VP of Operations
- Client Relations Manager
- Laboratory Project Manager
- Laboratory and/or Corporate Technical Managers / Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Account Executives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contracts Administrator, Account Executive, or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Contracts Department maintains copies of all signed contracts. TestAmerica Sacramento's Customer Service Organization maintains copies of all signed contracts on the computer network for reference locally.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. These records are archived by client and project in a restricted network folder accessible to laboratory department managers, project managers, and senior managers.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. Each Laboratory Project Manager keeps a phone log of conversations with the client. In addition, all conversations involving notification of important information, or actions directed by the client are documented with a follow up e-mail and archived in the contracts folder or the SDG documentation and case narrative. Instances include change in scope, alterations to the requests listed on a chain of custody, directions to proceed in the event of a non-conformance, and any other conversation that changes the direction of a COC or contract.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, a PM is assigned to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements. Quality Assurance Project Plans, if submitted by the client, will be evaluated per policy WS-PQA-0018.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are updated to the Quality Assurance Summary (QAS) and introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO/IEC 17025 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Allow the client access to supplemental information that pertains to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 Client Communication

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Any member of the laboratory's senior staff or any of the laboratory's identified technical experts is available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOPs on Subcontracting Procedures (CW-L-S-004).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

Project Managers (PMs), Client Service Managers (CSM), or Account Executives (AE) for the Export Lab (TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder. Standard TestAmerica Terms and Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., DoD and DOE projects) may require notification prior to placing such work. Documentation of approval is stored electronically in the quote folder within SACSALES share on a local laboratory server.

8.2 Qualifying and Monitoring Subcontractors

Whenever a PM or Client Services Manager becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.

- Subcontractors reviewed by TestAmerica – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the TestAmerica intranet site.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

8.2.1 When the potential sub-contract laboratory has not been previously approved, PMs or CSMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM) or Laboratory Director. The CRM or Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CW-L-S-004, Subcontracting.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. After the Corporate QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site and the finance group is concurrently notified for JD Edwards.

The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractors on our approved list can only be recommended to the extent that we would use them.

8.3 Oversight and Reporting

8.3.1 The status and performance of qualified subcontractors will be monitored by the Corporate Quality department. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance, Legal and Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. CSO personnel will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any

laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all CSO Personnel, Laboratory Directors, QA Managers and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is stored electronically in the quote folder within the SACSALES share on a local laboratory server.

8.3.2 For continued use of a subcontractor, verification of certification is placed upon the subcontractor for the defined project. Samples are subcontracted under Chain of Custody with the program defined as 'Accreditation Required' and the following statement for verification upon sample receipt:

Note: Since laboratory accreditations are subject to change, TestAmerica Laboratories, Inc. places the ownership of method, analyte & accreditation compliance upon our subcontract laboratories. This sample shipment is forwarded under Chain of Custody. If the laboratory does not currently maintain accreditation in the State of Origin listed above for analytes/tests/matrix being analyzed, the samples must be shipped back to the TestAmerica laboratory or other instructions will be provided. Any changes to accreditation status should be brought to TestAmerica Laboratories, Inc. attention immediately. If all requested accreditations are current to date, return the signed Chain of Custody attesting to said compliance to TestAmerica Laboratories, Inc.

For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

8.3.3 All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for all samples workshared within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time.

The use of any emergency subcontractor will require the PM to complete a JDE New Vendor Add Form in order to process payment to the vendor and add them to TALS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Controlled Purchase Requests and Fixed Asset Capitalization Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001. Approval information for the solvents and acids tested under SOP CA-Q-S-001 is stored on the TestAmerica Sharepoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. Many items used routinely are pre-qualified and placed into the on-site consignment system.

For items not available from the consignment system or items that are not used routinely, an order is placed in the JDE ordering system. Only personnel trained in the ordering program JDE may place orders using the program. All relevant information, including quantity, must be entered. Only approved vendors may be used. A vendor must be approved by corporate to be on the approved vendor list in JDE. The Laboratory Director or designee approves all orders placed in JDE.

9.3.2 Receiving

It is the responsibility of the purchasing manager to receive the shipment. For items received for the on-site consignment system, the purchasing manager verifies that the material received meets the quality level specified. This is documented by stamping the packing slip with "Received" and the date. For materials that are outside of the on-site consignment systems, it is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The purchasing manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared "public" folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of the grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals should not be used past the manufacturer's or SOP's expiration date unless 'verified' (refer to bullet 3 below). See laboratory SOP No. WS-QA-0017, "Standards and Reagent Preparation and Quality Control Check Procedures", for standard verification procedures.)

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained on the shared public folder on the computer network.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure would be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. For the automated “tank farm” in use through most of the laboratory, the minimum total pressure at which the system switches to the next bank of tanks is 250 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water’s specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified “clean” by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard. See laboratory SOP No. WS-QA-0017, “Standards and Reagent Preparation and Quality Control Check Procedures”, for standard QC procedures.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Each laboratory section maintains records of manufacturer’s certification and traceability statements on the network. These records include date of receipt, lot number (when

applicable), and expiration date (when applicable). Furthermore, certificates of analysis for standards are scanned and attached to the preparation record in the LIMS. Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager or QA Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench and inventoried in the master document list.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP WS-QA-0041, Calibration and Calibration Check of Balances. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed, and documentation of the review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc, are obtained from vendors with current and valid ISO 17025 accreditation for calibration of

the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory.

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following laboratory policy WS-PQA-013, Procedure to Address Customer Complaints.

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to laboratory policy WS-PQA-013, Procedure to Address Customer Complaints.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Manager and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

11.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the

Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Exec. Director of Quality & EHS and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, Executive VP of Operations, VP of Operations, and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.
When the laboratory discovers that erroneous or biased data may have been reported to clients or regulatory agencies, the procedures described in the corporate SOP CW-Q-S-005, Data Recalls, must be followed.
During investigation and correction of situations involving alleged incidents of misconduct or violation of the company's ethics policy, the procedures described in the corporate SOP CW-L-S-002, Internal Investigations, must be followed.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

11.4 Prevention of NonConforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically, on a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that

suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate VP of Operations and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc…). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Manager/Director, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memos (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints

- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports
- Health and Safety violations

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Laboratory SOP No. WS-QA-0023, Nonconformance and Corrective Action System, provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or CAR is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with the problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and CAR is entered into a database for tracking purposes and these are periodically reviewed to ensure that the corrective actions have taken effect.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. (Previously, a local database served this purpose.) An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. iCAT allows associated document upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis. Refer to Figure 12-1.
- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.

- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The SOP also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 12-1.
Example - Corrective Action Report

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Sacramento
Corrective Action Report

Title: <Enter Title Here>

Reference: <tracking information>

Initiated by: **Date:**

Responsible Party: **Date:** <date report submitted>

Description of Problem:
[enter text to briefly explain how the problem was discovered, who discovered it and when, and what work, if any, is affected]

Investigation Planned or Completed:
[enter text to briefly what was examined to determine the extent of the problem, when the investigation was conducted, what was the proximate cause(s), and what were the root causes. The key is to demonstrate that the investigation is comprehensive]

Root Cause Analysis
[True root cause analysis should involve multiple layers of questioning]
Examples:
- Why did this problem occur?
- What weaknesses are indicated by this problem?
- What Quality Systems mechanisms are in place that should have prevented this problem from occurring?
- Is this issue acute or chronic?
- Are changes needed to existing SOPs to correct this problem and prevent its recurrence?
- Are other departments affected by this issue?

Corrective Action Plan
[Based on the Root Cause Analysis outlined above, what action items need to be completed to correct this deficiency, and prevent its recurrence?]
Examples:
- Identify impacted lots
- Revise results/reports
- Initiate formal Data Recall
- Revise SOP
- Re-train staff

QA Monitoring of Corrective Action Status
[If an anomalous or isolated event, and no further action required, this section may be omitted. Otherwise, note the need for a routine follow-up assessment and the associated details (responsible party, due date, documentation necessary), or the need to add to the internal audit checklist for reassessment at a later date.

Closed by: _____ _____
<Name, title> Date

Table 12-1. Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	- Instrument response <MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards *(Analyst, Technical Manager(s))*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Technical Manager(s))*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in the LIMS or Project QAPP.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in the LIMS or Project QAPP.	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples *(QA Manager, Technical Manager(s))*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Technical Manager(s), Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002.
Client Complaints *(Project Managers, Lab Director/Manager, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director/Manager, *Technical Manager(s)*)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director/Manager, *Technical Manager(s)*)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:
1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants as defined in policy WS-PQA-003 **provided** they appear at similar levels in the reagent blank and samples. The ubiquitous contaminants include: methylene chloride, toluene, acetone, 2-butanone, phthalates and octachlorodibenzodioxin. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by the laboratory management, Corporate QA and TestAmerica's Executive Committee. These metrics are used ~~to~~ in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lesson Learned, Technical Services audit report, Technical Best Practices, or as Corporate or management initiatives.
The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/Process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any Preventive Actions/Process Improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in WS-QA-0050, Management of Change Procedures.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. More detailed information on retention of specific records is provided in CW-L-P-001, Records Retention Policy and CW-L-WI-001, TestAmerica Records Retention/Storage Schedule. Quality records are maintained by the QA department in a database or in specific folders on the local QA share on a corporate server, which is backed up as part of the regular backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by Department Managers.

Table 14-1. Record Index[1]

	Record Types [1]:	Retention Time:
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)

	Record Types [1]:	Retention Time:
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Business Operations	Refer to CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	Disposal Records (Add Permits?)	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual. All HR documents have different retention times.
	Administrative Policies	Indefinitely
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to CW-L-WI-001
	IT Records	Refer to CW-L-WI-001
	Corporate Governance Records	Refer to CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Logs are maintained in each storage box to note removal and return of records. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 14-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (lab reports and raw data) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
Ohio VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement
OSHA	30 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 and WS-PQA-017 for more information.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by the LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set.) Refer to SOP WS-QA-0009, Document Archiving. Instrument data is stored by project, except for inorganics and calibration data. Inorganics and calibration data is stored sequentially by instrument as appropriate. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

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

- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP WS-QA-0009.

- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'**.**

14.2 Technical and Analytical Records

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code**;**
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook, on a benchsheet or in the LIMS.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents**;**
- test results**;**
- standard and reagent origin, receipt, preparation, and use**;**
- calibration criteria, frequency and acceptance criteria**;**
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions**;**
- quality control protocols and assessment**;**
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats**.**

14.2.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a documented supervisory or peer review on a monthly basis.

14.3 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)**;**

- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value**;**
- copies of final reports**;**
- archived SOPs**;**
- correspondence relating to laboratory activities for a specific project**;**
- all corrective action reports, audits and audit responses**;**
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and

are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in a logbook or in the LIMS. Records are considered archived when noted as such in the records management system (a.k.a., document control.)

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and**,** when requested**,** to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CW-Q-S-003)	Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint responsibility: a) QA Manager or designee c) Technical Manager or Designee (Refer to CW-Q-S-003)	SOP Compliance Review Frequency • Minimum of every two years. • Annually for all methods and administrative SOPs relating to DoD/DOE programs.
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The

audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., MintMiner and Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. (Annually for methods and administrative SOPs related to DoD/DOE programs.) It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Soil, Water Supply, Water Pollution, Air, and round-robin studies for sediments and biological materials. When available for parameters tested by the laboratory, the laboratory will also participate in the DOE administered MAPEP program.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found within the 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been

affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Managers, their Quality Director as well as the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, VP of Operations, or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technical Managers, and QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel can be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources.
    - Adequacy of policies and procedures.
    - Future plans for resources and testing capability and capacity.

- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations and Quality Directors receive a monthly report from the Exec Director of Quality & EHS summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific labs.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, Conductivity, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Managers – **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Managers – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP (WS-QA-0022, Employee Orientation and Training).

17.4 Data Integrity and Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 66,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. In the event of a power outage, the laboratory can be equipped with a back up power supply for sample storage, as detailed in SOP No. WS-QA-0005, Temperature Monitoring and Corrective Action for Refrigerators and Freezers.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys and alarm codes are distributed to employees as necessary.

Employees wear photographic identification name cards while on the premises.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP WS-QA-0021 (Preparation and Management of Standard Operating Procedures).
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD/DOE SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Analysis and Sampling Procedures; 40 CFR Part 136 as amended by Method Update Rule; May 18, 2012.*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics & Organics Analysis, SOM, DLM, CBC, and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*

- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015..*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *Underground Storage Tanks Procedures Manual, State of Alaska Department of Environmental Conservation, Division of Spill Prevention and Response Contaminated Sites Program, November 7, 2002*
- *Tri-Regional Board Staff Recommendations for Preliminary Investigation and Evaluation of Underground Tank Sites, North Coast Regional Water Quality Control Board, San Francisco Bay Regional Water Quality Control Board and Central Valley Regional Water Quality Control Board, August 10, 1990*
- *Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997*
- *Compendium of Methods for the Determination of Air Pollutants in Indoor Air, (EPA 600/4-90-10, April 1990)*
- *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air, (EPA 625/R-96/010a, June 1999*
- *Methods for Determining Emissions of Toxic Air Contaminants from Stationary Sources, Stationary Source Test Methods, Volume 3, California Air Resources Board*
- *Leaking Underground Fuel Tank Guidance Manual, September 2012, California State Water Resources Control Board*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC, Lab SOP # WS-QA-0022) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst hasn't performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

19.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP. If the concentration is unspecified, the routine LCS spike level may be used.

19.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general 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 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

In accordance with Arizona Administrative Code R9-14-616.5f, documentation of each analyst's performance of proficiency testing, as applicable, will be maintained in the training record.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the

analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP. An SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Refer to the Corporate SOP No. CW-Q-S-006 or the laboratory's SOP No. WS-QA-0006 for details on the laboratory's MDL process.

19.8 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

19.9 Verification of Detection and Reporting Limits

Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at no more than 3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and no more than 4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. The analytes must be qualitatively identified or see SOP No. WS-QA-0006 for other options. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

For DoD ELAP certified methods, and methods utilized in support of DOE programs: Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD/DOE QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD/DOE QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation or utilized in support of DOE programs. Refer to the laboratory SOP WS-QA-0006, Method Detection Limits (MDL) and Instrument Detection Limits (IDL) for further details.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit and annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

For DoD ELAP certified methods and methods utilized in support of DOE programs: The laboratory quantitation limit is equivalent to the DoD/DOE Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD/DOE QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with the three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD/DOE projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

19.11 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.12 Estimation of Uncertainty of Measurement

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly,

and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy. If a problem is uncovered then the re-analysis will be repeated correctly. If no problem is uncovered then the laboratory will consult with the client to decide on actions needed.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Manager or Laboratory Director if unsure.

19.14 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOP Nos. CW-I-P-006, "Computer Systems Account and Naming Policy", CW-I-P-007, "Computer Systems Password Policy and CA-I-S-006, "Software Testing, Validation and Verification." The laboratory is currently running the TestAmerica Laboratory Information Management System ("TALS") which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Sequel Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 **Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protections, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.14.1.2 **Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 **Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The

spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and WS-PQA-011, Manual Integration Documentation Procedures.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/L) or micrograms per liter (µg/L) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1**%.** Units are defined in each lab SOP.

19.14.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.14.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst may print a copy of what has been entered to check for errors. This printout and the instrument's data file of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained within Chrom or the LIMS, based on the type of data.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample

ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z'd" out, signed and dated.
- Worksheets are created with the approval of the Technical Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are outlined in several SOPs (WS-PQA-003, "Quality Control Program"***,*** WS-PQA-012, "Technical Data Review Requirements***",*** WS-PM-0004, "Final Report Assembly and Third Level Data Review") to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (WS-PQA-0011, "Manual Integration Documentation and Practices"). The general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.14.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.14.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.14.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.14.4.5 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.14.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that the COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The project manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.14.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.4.8 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the

guideline for our internal SOP No. WS-PQA-0011, entitled “Manual Integration Documentation and Practices”.

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices. Instrument operators must assure that all manual integration documentation identifies the analyst, the date and the reason for the integration.

Figure 19-1. Example - Demonstration of Capability Documentation

Analyst Demonstration of Capability

TestAmerica Sacramento

Victoria Nihart

4/24/2017

Preparation Method(s):	3535
Analytical Method(s):	GCMSMS_NDMA
Matrix:	Water
Method Description:	Nitrosamines by Isotope Dilution and GC/CI/MS/MS
Preparation SOP No:	WS-IDP-0020 Rev. 3.3
Analytical SOP No:	WS-MS-0012 Rev. 2.1

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of test method(s) and laboratory SOPs are available for all personnel on-site.
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

Technical Director	**Signature**	**Date** 5/3/17
Quality Assurance Officer	**Signature**	**Date** 5/3/17

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Analyst Demonstration of Capability

ANALYST DEMONSTRATION OF CAPABILITY

Method GCMSMS_NDMA **Laboratory:** TestAmerica Sacramento
Method Desc: Nitrosamines by Isotope Dilution and GC/CI/MS/MS
Analyst: Victoria Nihart **Limit Group:** MSS - NDMA - Water - QC

Current Limits — Recovery / Precision
Demonstration of Capability — Recovery / Precision

N-Nitrosodimethylamine
All values within Control limits
Analysis Dates: 6/1/2016 to 6/3/2016

LCL	UCL	Std Dev	Units
		20	%

Mean	Std Dev	Units		Amount	Amount/RL
90.09	8.53983	%	Pass	2.0	1

Laboratory ID	Anal Date	Batch	Smp	Analyst	Prep Analyst	Result	Units	Spike Amount	RL	% Rec		In Rec Limits?
LLCS 320-110927/3-A	06/01/2016	112005	17	Nihart, Victoria M	Mantri, Anil	1.618	ng/L	2.0	2.0	81		Pass
LLCSD 320-110927/4-A	06/01/2016	112005	18	Nihart, Victoria M	Mantri, Anil	1.962	ng/L	2.0	2.0	98	19	Pass
LLCS 320-111960/3-A	06/03/2016	112484	4	Nihart, Victoria M	Kuzmenko, Natalia	1.931	ng/L	2.0	2.0	97		Pass
LLCSD 320-111960/4-A	06/03/2016	112484	5	Nihart, Victoria M	Kuzmenko, Natalia	1.695	ng/L	2.0	2.0	85	13	Pass

N-Nitrosodimethylamine-d6
All values within Control limits
Analysis Dates: 6/1/2016 to 6/3/2016

LCL	UCL	Std Dev	Units
25	150		%

Mean	Std Dev	Units		Amount
75.77	7.50265	%	Pass	100.0

Laboratory ID	Anal Date	Batch	Smp	Analyst	Prep Analyst	Result	Units	Spike Amount	% Rec		In Rec Limits?
LLCS 320-110927/3-A	06/01/2016	112005	17	Nihart, Victoria M	Mantri, Anil	80.88	ng/L	100.0	81		Pass
LLCSD 320-110927/4-A	06/01/2016	112005	18	Nihart, Victoria M	Mantri, Anil	81.20	ng/L	100.0	81	0.4	Pass
LLCS 320-111960/3-A	06/03/2016	112484	4	Nihart, Victoria M	Kuzmenko, Natalia	65.13	ng/L	100.0	65		Pass
LLCSD 320-111960/4-A	06/03/2016	112484	5	Nihart, Victoria M	Kuzmenko, Natalia	75.88	ng/L	100.0	76	15	Pass

Precision = standard deviation of percent recoveries of spiked control samples.

4/24/2017

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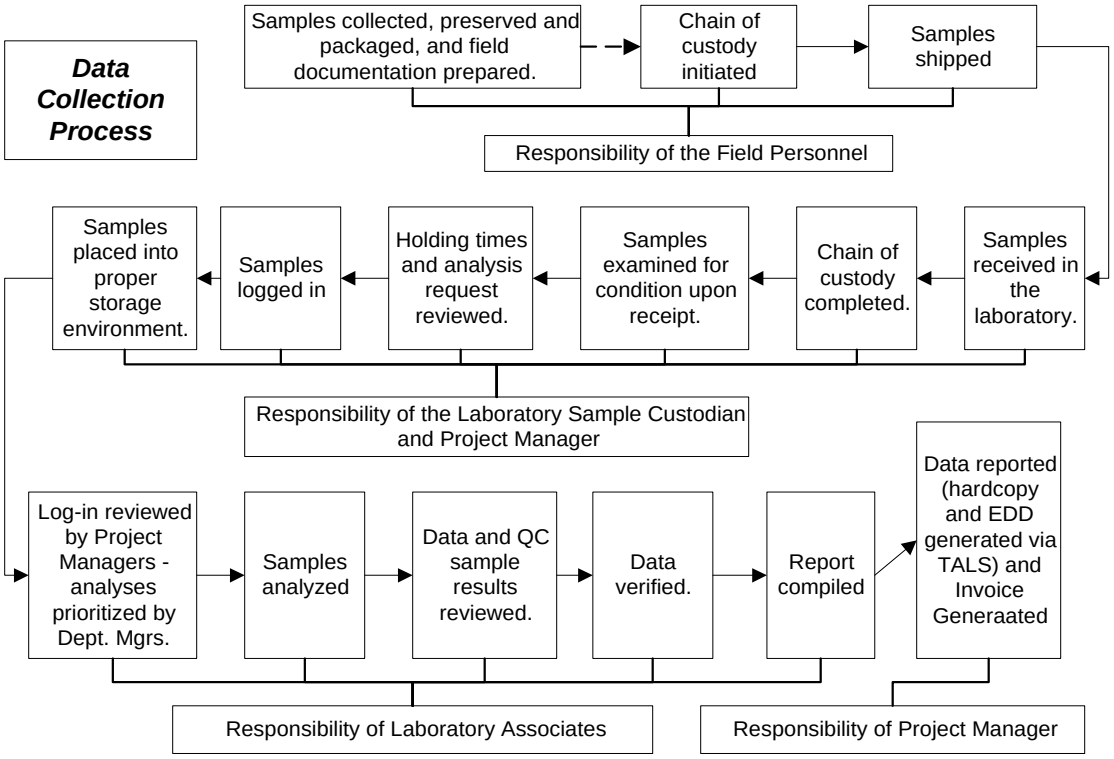
Figure 19-2. Example: Work Flow



SECTION 20. EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may be / are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or

instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLv) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by an ISO 17025 qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. See SOP No. WS-QA-0041, "Calibration and Calibration Check of Balances" for more details.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in their logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer at temperatures bracketing the range of use.

- If the temperature measuring device is used over a range of 10℃ or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10℃, then the verification must bracket the range of use.

IR thermometers, digital probes and thermocouples are calibrated quarterly**.** IR Thermometers should be calibrated over the full range of use, including ambient, iced (4 degrees) and frozen (0 to -5 degrees), per the Drinking Water Manual.

The digital NIST thermometer is recalibrated every five years by an approved outside service and the provided certificate of traceability is kept on file. Alternately a new NIST thermometer with certificate of traceability from the manufacturer may be purchased. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the SOP No. WS-QA-0016, "Thermometer Calibration."

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample storage are monitored 7 days a week; and each working day for units used for standard storage.

Ovens and water baths are monitored on days of use. Drying oven temperature must be recorded before and at the end of use. For example, an oven used for moisture determination must have its temperature recorded at the start and end of the drying process. Temperature must be ± 5% of set temperature for DoD/DOEwork.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept at > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, and water baths can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements. See SOP WS-QA-0004, “Maintenance and Calibration Check of Fixed and Adjustable Volume Autopipettors, Autodispensers and Volumetric Containers”.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy. The laboratory also assigns a unique ID# to each syringe. The delivery volume of each syringe is verified gravimetrically before initial use.

20.3.6 Autoclaves

Autoclaves used for sample digestion are capable of maintaining conditions of 15 psi at 120ºC for 15 minutes. The temperature of the autoclave is verified quarterly.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually however, the annual requirement does not apply to Isotope Dilution methods.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least each daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. (Exception: Some QC programs, such as the DoD/DOE QSM Version 5, require bracketing standards with internal standard calibration). The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements - see specific SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with unacceptable calibration verification may be fully useable under the following special conditions and reported based upon discussion and approval of the client:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision

level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Guidelines for evaluating and reporting TICs are in the specific laboratory SOPs.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS Tuning

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Example: Instrumentation List

Instrument Type	Number in Use
Autoanalyzer	1
Autotitrator	1
Cold-Vapor Analyzers	1
GC/HRMS	6
GC/MS - Semivolatiles	9
GC/MS - Volatiles	8
GC/MS – Volatile Air	12
GC/MS/MS	1
GC-ECD/ECD	6
GC-FID/FID	2
GC-FID	1
GC-FPD	1
GC-TCD/TCD	1
HPLC	5
HPLC/MS/MS	5
ICP	1
ICP/MS	1
Ion Chromatograph	3
Spectrometer	1

Table 20-2. Example: Schedule of Routine Maintenance

INSTRUMENT	MAINTENANCE	FREQUENCY
APCI/ESI LC/MS/MS	Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes.	As Needed
	Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that corona and multiplier are functioning.	Daily(2)
	Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable.	Semi-Annually
	Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.	Annually
HIGH PRESSURE LIQUID CHROMATOGRAPH(1)	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements. Rinse flow cell with 1N nitric acid if dirty flow cell. Change pump seals when flow becomes inconsistent. Backflush column if applicable. Change in-line filters for solvents.	As Needed
	Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse delivery lines to prevent contamination of the new solvent. Check gas supply if applicable. Flush with an appropriate solvent to remove all bubbles. Pre-filter all samples.	Daily(2)
	Change pump seals.	Every 6-9 Months

INSTRUMENT	MAINTENANCE	FREQUENCY
GAS CHROMATOGRAPH(1)	Replace septum. Clean injector port Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Change glass wool plug in injection port and/or replace injection port liner when front portion of capillary column is removed. Replace or repair flow controller if constant gas flow cannot be maintained. Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace igniter. ECD: follow manufacturers suggested maintenance schedule PID: Clean lamp window or replace. Replace seals. Replace fuse. Reactivate external carrier gas dryers. HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents. Check inlets, septa.	As Needed
	Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. Check temperatures of injectors and detectors. Verify temperature programs. Check baseline level. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Daily(2)
	Oxidation and Reduction Catalysts: Perform leak checks. Replace/condition when poor response is observed.	Quarterly
	ECD: perform wipe test.	Semi-Annually
PURGE AND TRAP SYSTEMS	Change trap. Check purge flow. Flush lines (after foaming sample). Periodic leak checks (when replace traps/spargers) Replace/condition traps and/or spargers (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), and clean or replace glassware/spargers. Bake trap as needed to correct for high background. Change trap whenever loss of sensitivity, or erratic response or failing resolution is observed. Purge & trap autosamplers: leak check system, clean sample lines, valves.	As Needed
	Bake out trap & analyze primers (as needed) prior to commencing analysis.	Daily(2)
GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROMETER(1)	Replace septum. Clean injector port. Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Replace injection port liner when front portion of capillary column is removed. Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed.	As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels. Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination. Replace filaments when both filaments burn out or performance indicates need for replacement. Check mass calibration (PFTBA or FC-43). Check ion source and analyzer (clean, replace parts as needed). Check vacuum, relays, gas pressures and flows. Change oil in the mechanical rough pump. Relubricate the turbomolecular pump-bearing wick. HP 7673 Autosampler: Replace syringe.	
	Check for sufficient gas supply. Check for correct column flow and/or inlet pressure. Check temperatures of injector, detector. Verify temperature programs. Check inlets, septa. Check baseline level. Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks. Autosampler: fill wash bottle, dispose of waste bottle contents. Air Autosampler: Check for proper operation. Leak check system.	Daily(2)
	Replace the exhaust filters on the mechanical rough pump every 1-2 years.	Annually
GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETER(1)	Full Bake-Out. Change oil in rotary pump. Change oil in diffusion pump. Replace o-rings. Solvent rinse the flight tube. Clean the first field free region. Check detector voltages. Clean and dust connectors, etc on the outside of the instrument. Check the vacuum: ~5 x. 10^{-7} MBAR on both analyzer ion gauges, and ~5 x 10^{-6} MBAR on the source, with no helium flowing. Check isolation valve for leaks, correct if needed. Check for thermal trip by taking the magnet to maximum current, and verify that the coolant flow is acceptable. Replace septum. Clean injector port. Cut off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required. Replace injection port liner when front portion of capillary column is removed. Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms	As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination. Replace filaments when performance indicates need for replacement.	
	Check resolution sensitivity. Check stability. Check for sufficient gas supply. Check for correct column flow and/or inlet pressure. Check temperatures of injector, detector. Verify temperature programs. Check inlets, septa. Check baseline level. Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds. Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Daily(2)
COLD VAPOR ATOMIC ABSORPTION (LEEMAN PS 200) (1)	Change pump tubing. Check/change Hg lamp. Clean optical cell. Change drying tube. Grease pump.	As Needed
	Check sample tip for clogs. Check drying tube. Check pump tubing/drain tubing. Check gas pressure. Check liquid/gas separator. Check tubing.	Daily(2)
INDUCTIVELY COUPLED ARGON PLASMA/MASS SPECTROMETRY (ICAP/MS)(1)	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics. Measure quartz torch for proper alignment when removed and cleaned. Clean spray chamber and nebulizer. Clean all filters and fans. Check chiller coolant level. Check and drain oil mist eliminator on roughing pumps.	As Needed
	Check sample waste container level. Check quartz torch condition. Check RF coil. Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing. Check condition of sampler and skimmer cones. Check oil level of roughing pumps.	Daily(2)
	Replace oil in roughing pumps.	Every 2-3 Months
ICP(1)	Check that argon feed pressure is 80-120 psi. Check that chiller coolant pressure is 45-80 psig, no leaks. Check purge and shear gasses. Nitrogen purge gas pressure 40-120 psig, compressed air shear gas pressure 80-120 psig. Check radial purge and axial windows for deposits. Check that nebulizer is not clogged. Check that capillary tubing is clean and in good condition. Check that peristaltic pump windings are secure. Check that exhaust vent is operational Check that torch, glassware, aerosol injector tube are clean.	Daily(2)

INSTRUMENT	MAINTENANCE	FREQUENCY
	Clean plasma torch assembly to remove accumulated deposits. Check RF coil. Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance. Clean filters on back of power unit to remove dust. Replace when needed: peristaltic pump tubing. sample capillary tubing. autosampler sipper probe. Check performance with manganese. Check O-rings. Clean/lubricate pump rollers	Monthly or As Needed
	Check chiller coolant filter. (may require more or less frequently)	Semi-Annually
	Notify manufacturer service engineer for scheduled preventive maintenance service.	Annually
ION CHROMATOGRAPH(1)	Clean micromembrane suppressor when decreases in sensitivity are observed. Check fuses when power problems occur. Change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated. De-gas pump head when flow is erratic. Check all air and liquid lines for discoloration and crimping, if indicated. Check/change bed supports guard and analytical columns, if indicated.	As Needed
	Check plumbing/leaks. Check eluent level. Check gases. Check pump pressure. Check conductivity meter.	Daily(2)
	Check pump heads for leaks. Check filter (inlet).	Weekly
	Change pump seals. Change injection valve. Clean conductivity cell. Check conductivity cell for calibration.	Annually
ALPKEM COLORIMETRIC AUTO ANALYZER(1)	Prepare fresh reagents. Replace tubing. (About every 100 hours of use)	As Needed
	Check detector. Make sure there are no trapped bubbles in detector cell. Check Valves Check peristaltic tubing. Check sampler.	Daily(2)
	Clean pump, and XYZ Sampler.	Weekly
	Lubricate pump roller.	Monthly
	Clean pump rollers with steel wool and lubricate.	Semi-Annually
CHEMICAL OXYGEN DEMAND (COD) REACTOR(1)	Electronics serviced.	As Needed
	Check temperature with NIST reference thermometer.	Annually
AUTO TITRATOR(1)	Electronics serviced.	As Needed
	Calibrate with check standards.	Daily(2) (When Used)

INSTRUMENT	MAINTENANCE	FREQUENCY
	Inspect electrodes daily, clean as needed. Inspect electrode proper levels of filling solutions daily, fill as needed. Clean probe, each use. Prime buret Check rinse water reservoir.	
CONDUCTANCE METER(1)	Electronics serviced. Replace batteries	As Needed
SPECTROPHOTOMETER(1)	Replace lamp. Replace fuse.	As Needed
	Check instrument manual. Perform wavelength calibration. Replace lamp annually or when erratic response is observed.	Annually
PH METER(1)	Clean electrode. Refill reference electrode.	As Needed
	Inspect electrode. Verify electrodes are properly connected and filled. Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer.	Daily(2)
TURBIDIMETER(1)	Electronics serviced.	As Needed
	Clean instrument housing.	Monthly
DIGESTION BLOCK	Check temperature with NIST thermometer.	Annually
SONICATOR (1)	Replace probe tip. Disassemble and clean sonicator probe tips. Tune sonicator assembly (if recommended by manufacturer)	As Needed
	Inspect probe tips for inconsistencies (etching/pitting).	Daily(2) (When Used)
ANALYTICAL/TOP LOADING BALANCES(1)	Check using ASTM Class 3 weights once daily or before use. Clean pan and weighing compartment.	Daily(2)
REFRIGERATORS/WALK-IN COOLERS(1)	Manufacturer cleaning and calibration.	Annually
	Refrigerant system and electronics serviced.	As Needed
	Temperatures checked and logged.	Daily(2)
OVENS(1)	Electronics serviced.	As Needed
	Temperatures checked and logged.	Daily(2)
ZYMARK PE WORKSTATION	Change O-rings whenever there are visible leaks or poor sealing on the SPE columns. Sample lines are clean after samples have been extracted by SPE with a program “Clean Sample Lines” with methanol followed by water. Occasionally for a more rigorous cleaning, or after a highly contaminated sample, a mixture of methanol/DCM at 50:50 may be used in place of methanol, follow by methanol, then water (never use acetone). Syringe pump may be primed using a program “Prime Solvent Lines” whenever air bubbles are suspected in the lines from running out of solvents and whenever solvents are changed. Syringe pump in good condition – replace if showing signs of wear or suspected of poor performance. Sample pumps may be re-calibrated whenever major	As Needed

INSTRUMENT	MAINTENANCE	FREQUENCY
	repairs are performed, or whenever the pumps are suspected to be out of calibration. Follow manufacturer's procedure for re-calibrating the sample pumps. For method 8330, the pump loads 1050 mL of sample on the SPE. It should used up the whole sample bottle (quart bottles and 1-L bottles).	
SONICATION WATER BATH(1)	If the water bath is dirty, empty and refill with tap water. A couple drops of anti-bacterial solution may be added to inhibit the growth of bacteria in the water. The water level in the sonication batch should be about 1.2 to 1 inch from the top while in operation. Do not allow sonication batch to operate with water bath at lower levels. If the level is low, add more water, if the levels is too high, remove water to the proper level.	As Needed

Footnotes to Preventive Maintenance Tables

(1) Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.

(2) Daily checks and verifications are performed prior to instrument startup and are not documented in maintenance logs unless problems are noted.

(3) Where there are differences between this table and the tables present in method SOPs, the table in the method SOP should be followed.

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program) or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International al Laboratory Accreditation Cooperation) or APLC (Asia-Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

The calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty.

The uncertainty calculations of the calibration laboratory are supported by uncertainty budgets and are represented by expanded uncertainties typically using a coverage factor of k=2 to approximate the 95% confidence level. This explanation accompanies the measurement result and the associated uncertainty.

The tolerance uncertainty ratio (TUR) is calculated using the expanded uncertainty of the measurement, not the collective uncertainty of the measurement standards. A statement to this effect accompanies the TUR along with the coverage factor and confidence level.

The calibration report or certificate submitted to TestAmerica Sacramento contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis

upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

The calibration laboratory supports in-house calibration systems: documented procedures for in-house calibrations, evidence by a report, certificate, or sticker, for an appropriate amount of time; training records of calibration personnel; certificates from accreditation services demonstrating traceability to national or international standards of measurement; procedures for evaluating measurement uncertainty; timely and documented recalibration of reference standards. When subcontracting to a calibration laboratory, TestAmerica Sacramento does not use a firm who subcontracts the work.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

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If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the ‘true’ value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a

second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. [Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.]

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are scanned and retained on the local server. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and SOP No. WS-QA-0017, "Standards and Reagents and Quality Control Check Procedures".

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material (for 1613B dioxin/furan analyses the purity must be 98% or corrections must be made). Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database or standards logbook.

- Standard ID
- Description of Standard

- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically or in logbooks for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from the preparation logbook)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the SDS section of OASIS.

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions.
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificate may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for analysis include any necessary reanalysis. However, there are some programs and regulators, which determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative.

As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located SOP Nos. WS-QA-0018, “Subsampling and Compositing of Samples (Method ASTM D 6323-98**)**” and WS-QA-0028, “Incremental Sampling Methodology of Soils and Sediments”.

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her

view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the COC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, legal COCs will be generated per the Manual for Certification of Laboratories Analyzing Drinking Water, Fifth Edition, January 2005, Appendix A, and SOP No. WS-QA-0003, "Sample Receipt and Procedures".

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections and in SOP No. WS-QA-0003, "Sample Receipt and Procedures".

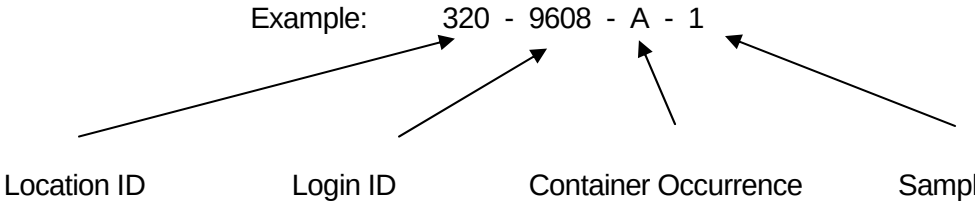
23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on the lot receipt checklist and within the non-conformance program and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record. Laboratory receipt procedures are described in more detail in SOP No. WS-QA-0003.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Sacramento Laboratory (Location 320) is the receiving laboratory. Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 320 - 9608 - A - 1 - **A** ←——**Secondary Container Occurrence**

Example: 320-9608-A-1-A would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. WS-QA-0003.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples are returned to the secure sample control area. Empty sample containers are marked as Destroyed in the LIMS sample checkin-checkout module and are disposed by the analytical staff. All samples are kept in the refrigerators for 30 days past invoicing, unless other arrangements have been made with the client.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 Hazardous Samples and Foreign Soils

Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

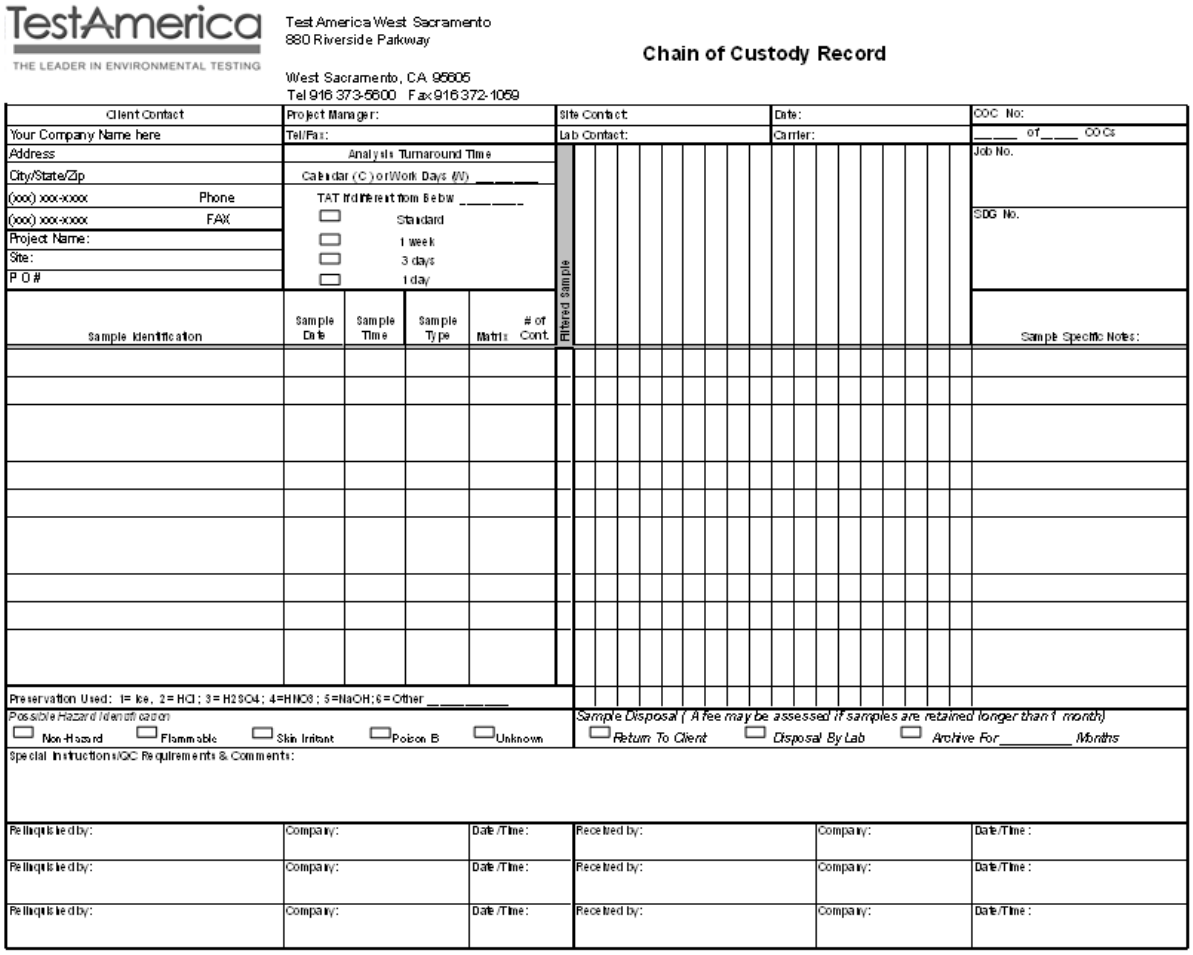
Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. An exception is samples contained in laboratory-owned air sample canisters. These are held for a minimum of 24 hours after the project report is sent, prior to evacuating the canister and returning it to the equipment pool. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: WS-EHS-001, "Waste Disposal"). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Example: Chain of Custody (COC)



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West Sacramento, CA 95605
Tel 916 373-5600 Fax 916 372-1059

Chain of Custody Record

Client Contact	Project Manager:	Site Contact:	Date:	COC No:
Your Company Name here	Tel/Fax:	Lab Contact:	Carrier:	____ of ____ COCs
Address	Analysis Turnaround Time			Job No.
City/State/Zip	Calendar (C) or Work Days (W) _____			
(xxx) xxx-xxxx Phone	TAT if different from Below _____			SDG No.
(xxx) xxx-xxxx FAX	☐ Standard			
Project Name:	☐ 1 week			
Site:	☐ 3 days			
P O #	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type	# of Matrix Cont.	Filtered Sample	Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)
☐ *Return To Client* ☐ *Disposal By Lab* ☐ *Archive For _____ Months*

Special Instructions/QC Requirements & Comments:

Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:

Figure 23-2. Example: Sample Acceptance Policy

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SACRAMENTO LABORATORY
SAMPLE ACCEPTANCE POLICY
(Effective 02/02/2015)

Page 1 of 2

The TNI Standard and TestAmerica Sacramento have specific requirements under which all samples will be received by the laboratory for analysis. TestAmerica Sacramento will review your sample shipment against those requirements as listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

VOA vials should be stored in controlled conditions. Exposure of trip blanks to temperature fluctuations is likely to cause development of bubbles in the trip blanks.

When completing the chain of custody form, please note that you must sign your name in the "relinquished by" box.

Requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples, shall be provided.
- Samples must be accompanied by written disclosure of the known or suspected presence of any hazardous substances, as defined by applicable federal or state law.
- Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).
- Each sample shall be collected in the appropriate sample container and labeled with unique, durable and indelible identification.
- Drinking water samples for Method 1613B that may have residual chlorine must be checked and treated in the field, or collected in sodium thiosulfate preserved containers.
- Containers of water meant for perchlorate analysis should have adequate headspace to prevent anaerobic microbial degradation. A void approximately 1/3 of the container volume is sufficient.
- The samples shall arrive at the laboratory with adequate remaining holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.
- Received samples must not exhibit obvious signs of damage, contamination or inadequate preservation.
- Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C and above freezing (0°C). For methods with other temperature criteria (e.g. some bacteriological methods require ≤ 10 °C), the samples must arrive within ± 2° C of the required temperature or within the method specified range.

    1. Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements above. In these cases, the samples shall be considered acceptable if the samples were received on ice.
    2. If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.
    3. Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

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TestAmerica
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SACRAMENTO LABORATORY
SAMPLE ACCEPTANCE POLICY

(Effective 02/02/2015)

- For samples undergoing chemical warfare degradate analysis, the sample must be screened for agent prior to shipment in accordance with appendix 10 of our Sample Receipt Procedure (WS-QA-0003).
- Samples containing mammalian tissue will not be accepted without prior coordination with a project manager. Additional conditions for receipt and handling of tissue are outlined in appendix 11 of our Sample Receipt Procedure (WS-QA-0003).
- Air canisters (SUMMA® and other brands) have additional requirements:
    - Never write or affix a label directly on a canister. A special tag is attached to each canister for this purpose.
    - Complete the Canister Field Data Record with the initial and final vacuum/pressure reading for each canister during sampling.
    - Close all valves completely prior to shipping or transporting.
    - Return canisters, filters, flow controllers, vacuum flow regulators, and any other supplied equipment must be returned even if they were not used. Pack equipment carefully to minimize in-transit damage Sampling equipment that is damaged, lost or not returned will be invoiced to the client at the replacement cost. Delayed return of equipment to the laboratory may result in additional rental charges.
    - Do not attempt to adjust or alter any equipment, as it may result in loss of sample integrity as well as equipment damage that may be invoiced to the client.

The laboratory will notify the client/Project Manager upon sample receipt if the samples fail to meet any of the above requirements.

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Figure 23-3. Example: Cooler Receipt Form

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Sacramento
Sample Receiving Notes

Job:_____

Tracking #_____ SO / PO / FO / UPS / Other _____

Use this form to record Sample Custody Seal, Cooler Custody Seal, Temperature & corrected Temperature & other observations. File in the job folder with the COC.

Notes:_____

Therm. ID: **AK-2 / AK-3 / AK-5 / AK-6 / HACCP** / Other_____

Ice_____ Wet_____ Gel_____ Other _____

Cooler Custody Seal: _____

Sample Custody Seal: _____

Cooler ID:_____

Temp: Observed_____

From: Temp Blank ☐ Sample ☐

NCM Filed: Yes ☐ No ☐

	Yes	No	NA
Perchlorate has headspace?	☐	☐	☐
CoC is complete w/o discrepancies?	☐	☐	☐
Samples received within holding time?	☐	☐	☐
Sample preservatives verified?	☐	☐	☐
Cooler compromised/tampered with?	☐	☐	☐
Samples compromised/tampered with?	☐	☐	☐
Samples w/o discrepancies?	☐	☐	☐
Sample containers have legible labels?	☐	☐	☐
Containers are not broken or leaking?	☐	☐	☐
Sample date/times are provided.	☐	☐	☐
Appropriate containers are used?	☐	☐	☐
Sample bottles are completely filled?	☐	☐	☐
Zero headspace?*	☐	☐	☐
Multiphasic samples are not present?	☐	☐	☐
Sample temp OK?	☐	☐	☐
Sample out of temp?	☐	☐	☐

Initials:_____Date:_____Time_____

*Containers requiring zero headspace have no headspace, or bubble < 6 mm (1/4")

Q:\DOCUMENT-MANAGEMENT\FORMS\QA-812 REV. 1.6. SAMPLE RECEIVING NOTES 2018-05-18.DOC QA-812 r1.6 05/18/2018

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Bottle Lot Inventory

Lot ID:_____

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
VOA*																				
VOAh*																				
AGB																				
AGBs																				
250AGB																				
250AGBs																				
250AGBn																				
500AGB																				
____AGJ																				
500AGJ																				
250AGJ																				
125AGJ																				
____CGJ																				
500CGJ																				
250CGJ																				
125CGJ																				
PJ																				
PJn																				
500PJ																				
500PJn																				
500PJna																				
500PJzn/na																				
250PJ																				
250PJn																				
250PJna																				
250PJzn/na																				
Acetate Tube																				
_____"CT																				
Encore																				
Folder/filter																				
PUF																				
Petri/Filter																				
XAD Trap																				
Ziploc																				
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20

h = hydrochloric acid s = sulfuric acid na = sodium hydroxide n = nitric acid zn = zinc acetate

Number of VOAs with air bubbles present / total number of VOA's

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SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, reflux, evaporation, and drying. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

Table 24-1. Example – Negative Controls

Control Type	Details
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous

volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 30 data points (more points are preferred, however, fewer (minimum of 20) may be used to establish tentative acceptance limits in select circumstances).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

- The maximum acceptable recovery limit will be 150%. Some specific methods or SOPs may allow for higher recoveries.

- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

- If either the high or low end of the control limit changes by ≤ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. See SOP WS-QA-0035 for further details.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.

- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Or, for TNI and DoD/DOE work, there are an allowable number of random Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed or prepared electronically on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report for Samples) with a "sample results" column header.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. work order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##, where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.
- In most cases, the applicable COC is an integral part of the report.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., Sampling information).

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.22 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.23 When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

25.2.24 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.25 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary report). A complete report must be sent once all of the work has been completed.

25.2.26 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.27 A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory's accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

25.2.28 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level II is a report with the features described in Section 25.2 above, plus summary information, including results for the method blank reported to the laboratory MDL if required, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. No raw data is provided unless it is necessary to provide the relevant calibration information.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in electronic deliverable form via e-mail, posting to an FTP site, or CD ROM. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services in addition to the test report as described in section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. TestAmerica Sacramento offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD, and a copy filed on the QA share of the local server.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CW-L-S-004).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationery and the report includes any accompanying documentation.

25.6 Client Confidentiality

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are to meet all requirements of this document and to include a cover letter.

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R". Every page will have the report generation date present, to prevent confusion between report versions.

When the report is re-issued, a notation of "Revision "with the revision number is placed on the cover/signature page of the report. The case narrative is updated *with* a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example*: *Report was revised on 11/3/11 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/11.*

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

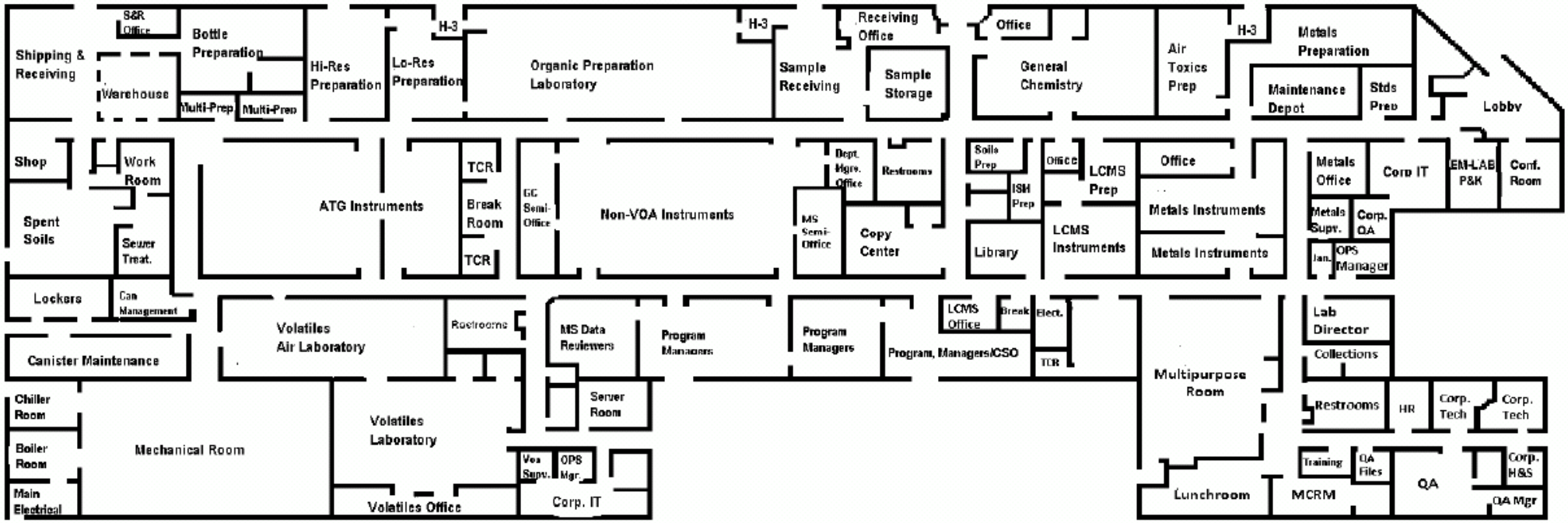
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. Laboratory Floor Plan



Facility Size	Square Feet
Total Area	66.000
Lab Area	44,725
Storage Area	5200
	Linear Feet
Bench Top	3036
Hoods	464

Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Air Sample Bag: A sampling container for air samples, commonly referred to as Flex-Film or Tedlar bag, in 1.0-L or 3.0-L volumes, that is constructed of proprietary material (E.G., SKC or ESS).

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

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

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 the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to 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.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. 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 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: The minimum concentration of a substance (an analyte) 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. (40 CFR Part 136, Appendix B)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Passivated Canister: A sampling container for air samples; commonly referred to as a SUMMA canister, SilcoCan or T.O.-Can in 1.0, 1.8 6, or 15 L volumes.

1) SUMMA canister: A spherical stainless steel canister, of which the interior has been specially treated by a process (SUMMA passivation) that renders all surfaces inert to VOCs.

2) SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most VOCs.

3) T.O.-Can: A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek using a proprietary electropolishing process and is extensively cleaned using an ultrasonic method that ensures a high-quality passivated surface that maintains the stability of VOCs during storage.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Quality System Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2^{nd} order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2^{nd} order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

A2LA – American Association for Laboratory Accreditation
ANSI – American National Standards Institute
ASQ – American Society for Quality
CAR – Corrective Action Report
CCB – Continuing Calibration Blank
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICB – Initial Calibration Blank
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SDS - Safety Data Sheet
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
TALS – TestAmerica LIMS system
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Sacramento maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

The certificates and accredited parameter lists are available, for each State/Program organization at www.testamericainc.com under Analytical Services Search – Certifications.

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TestAmerica Certifications

Laboratory	Program	Authority	Identification	Expiration Date
TestAmerica Sacramento	DoD ELAP	L-A-B	L2468	01/20/2018
TestAmerica Sacramento	Federal	US Fish & Wildlife	LE148388-0	10/31/2017
TestAmerica Sacramento	Federal	USDA	P330-11-00436	12/30/2017
TestAmerica Sacramento	Federal	USEPA UCMR	CA00044	11/06/2018
TestAmerica Sacramento	NELAP	Florida	E87570	06/30/2017
TestAmerica Sacramento	NELAP	Illinois	200060	03/17/2018
TestAmerica Sacramento	NELAP	Kansas	E-10375	10/31/2017
TestAmerica Sacramento	NELAP	Louisiana	30612	06/30/2017
TestAmerica Sacramento	NELAP	New Hampshire	2997	04/18/2018
TestAmerica Sacramento	NELAP	New Jersey	CA005	06/30/2017
TestAmerica Sacramento	NELAP	New York	11666	04/01/2018
TestAmerica Sacramento	NELAP	Oregon	4040	01/28/2018
TestAmerica Sacramento	NELAP	Pennsylvania	68-01272	03/31/2018
TestAmerica Sacramento	NELAP	Texas	T104704399	07/31/2017
TestAmerica Sacramento	NELAP	Utah	CA00044	02/28/2018
TestAmerica Sacramento	NELAP	Virginia	460278	03/14/2018
TestAmerica Sacramento	State Program	Alaska (UST)	UST-055	12/18/2017
TestAmerica Sacramento	State Program	Arizona	AZ0708	08/11/2017
TestAmerica Sacramento	State Program	Arkansas DEQ	88-0691	06/17/2018
TestAmerica Sacramento	State Program	California	2897	01/31/2018
TestAmerica Sacramento	State Program	Colorado	CA00044	08/31/2017
TestAmerica Sacramento	State Program	Connecticut	PH-0691	06/30/2017
TestAmerica Sacramento	State Program	Hawaii	N/A	01/29/2018
TestAmerica Sacramento	State Program	Maine	CA0004	04/18/2018
TestAmerica Sacramento	State Program	Michigan	9947	01/31/2018
TestAmerica Sacramento	State Program	Nevada	CA00044	07/31/2017
TestAmerica Sacramento	State Program	Washington	C581	05/05/2018
TestAmerica Sacramento	State Program	West Virginia (DW)	9930C	12/31/2017
TestAmerica Sacramento	State Program	Wyoming	8TMS-L	01/29/2017 *

Appendix 4: Listing of Methods Performed

Preparation Only Methods

Method	Aqueous	Solid	Waste	Biological	Air
Organics					
Calif. CAM-WET	X	X	X		
EPA 1311	X	X	X		
EPA 3510C	X				
EPA 3535	X				
EPA 3540B		X			
EPA 3542					X
EPA 3546		X			
EPA 3550B		X		X	
EPA 3580A			X		
EPA 3600C	X	X	X		
EPA 3620B	X	X	X		
EPA 3630C	X	X	X		
EPA 3640A	X	X		X	
EPA 5030B	X	X	X		
EPA 5035	X	X	X		
Inorganics					
Calif. CAM WET	X	X	X		
EPA 1311	X	X	X		
EPA 1312 (E/W)	X	X	X		
EPA 3005A	X				
EPA 3010A	X				
EPA 3050B		X	X	X	

Organics Methods Performed

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
Volatile Organics	SW846 8260B	X	X	X		
	SW846 8260C	X	X	X		
	EPA 624	X				
	TO-14A					X
	TO-15					X
Sulfur Containing Compounds	EPA 15/16					X
Fixed Gases	ASTM D1946					X
	EPA 3C					X
Base Neutrals and Acids (BNAs)	SW846 8270C	X	X	X	X	
	SW846 8270D	X	X	X	X	
	EPA 625	X				
	TO-13A					X
	IP-7					X
	EPA 23					X
Organochlorine Pesticides	SW846 8081A	X	X	X	X	
	SW846 8081B	X	X	X	X	
	EPA 608	X				
	TO-4A					X
	TO-10A					X
PCBs (Aroclors)	EPA 8082	X	X	X	X	
	EPA 8082A	X	X	X	X	
	EPA 608	X				
	TO-4A					X
	TO-10A					X
PCB Congeners	EPA 1668A	X	X	X	X	X
	EPA 1668C	X	X	X	X	X
Petroleum Hydrocarbons	EPA 8015B	X	X	X		
	EPA 8015D	X	X	X		
	CA LUFT	X	X	X		
	AK101	X	X	X		
	Ak102	X	X	X		
	AK103	X	X	X		
	GRO/DRO	X	X	X		
Nitroaromatics and Nitroamines	EPA 8330A	X	X	X		
	EPA 8330B	X	X	X		
	WS-LC-0010	X	X	X		
Nitrosamines	WS-MS-0012	X	X			

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
PAHs	EPA 8270C (SIM Isotope dilution)	X	X	X	X	X
	EPA 8270C (SIM)	X	X	X		
	CARB 429	X	X	X	X	X
	TO-13A					X
1,4-Dioxane	WS-MS-0010	X				
Alkyl Phenols	WS-MS-0013	X	X		X	
Perfluorinated Compounds	WS-LC-0025	X	X	X	X	
	ISO 25101	X				
(including PFOA/PFOS)	EPA 537	X				
Dioxins & Furans	EPA 1613B	X	X			
	EPA 8290	X	X	X	X	
	EPA 8290A	X	X	X	X	
	EPA 8280A	X	X	X	X	
	EPA 8280B	X	X	X	X	
	EPA 0023A					X
	EPA 23					X
	TO-9					X

Metals Methods Performed

Parameter	Methods	Aqueous	Solid	Waste	Biological	Air
Trace Metals	EPA 6010B	X	X	X	X	X
	EPA 6020	X	X	X	X	X
	EPA 0060					X
	EPA 200.7	X				
	EPA 200.8	X				
	EPA 12					X
	CARB 12					X
	EPA 29					X
	CARB 436					X
Hardness	SM 2340B	X				
Mercury	EPA 7470A	X				
	EPA 245.1	X				
	EPA 7471A		X	X	X	X
	EPA 101A					X
	ASTM D6784-02					X
	EPA 0060					X
	EPA 29					X
	CARB 436					X

Inorganics Methods Performed

Parameter	Method	Aqueous	Solid	Waste	Biological	Air
Alkalinity (Carbonate, Bicarbonate, Total)	SM 2320B	X				
Bromide, Chloride, and Fluoride	EPA 300.0	X				
	EPA 9056	X	X			
	EPA 9057					X
	EPA 26A					X
	CARB 421					X
Chromium, Hexavalent	EPA 7196A	X				
	EPA 0061					X
	EPA 306					X
	CARB 426					X
Conductivity	EPA 9050A	X				
	SM 2510 B	X				
Demand, Chemical Oxygen	EPA 410.4	X				
Moisture	ASTM 2216		X			
Nitrate	EPA 353.2	X				
	EPA 300.0	X				
	EPA 9056	X	X			
	CARB 421					X
Nitrate-Nitrite	EPA 353.2	X				
Nitrite	EPA 353.2	X				
	EPA 300.0	X				
	EPA 9056	X	X			
	CARB 421					X
Nitrocellulose	EPA 353.2	X	X			
	WS-WC-0050	X	X			
Orthophosphate	EPA 300.0	X				
	EPA 9056	X	X			
Particulates in Air	EPA 5					X
	40 CFR Part 50					X
Perchlorate	EPA 314.0	X				
	EPA 331.0	X				
	EPA 6850	X	X			
pH	SM 4500 H+ B	X				
	EPA 150.2	X				
	EPA 9040A	X				
	EPA 9041A	X				
	EPA 9045C		X	X		
Solids, Total	SM 2540 B	X				
Solids, Total Dissolved	SM 2540 C	X				

Solids, Total Suspended	SM 2540 D	X				
Sulfate	EPA 300.0	X				
	EPA 9056	X	X			

Appendix 5. Data Qualifiers

Qualifier Organic	Qualifier Inorganic	Footnote
U	U	Analyte analyzed for but was not detected.
J	B	Estimated result. Result is less than RL.
E	I	Estimated result. Result concentration exceeds the calibration range.
B	J	Method blank contamination. The associated method blank contains the target analyte at a reportable level.
P	*	Relative percent difference (RPD) is outside stated control limits.
a	N	Spiked analyte recovery is outside stated control limits.
*		Surrogate recovery is outside stated control limits.
PG		The percent difference between the original and confirmation analyses is greater than 40%.

Title: Determination of All Types of Residue in Water, Wastes, and Solid Samples [Standard Methods 2540B-1997, 2540C-1997, 2540D-1997, and 2540G-1997]

Approvals (Signature/Date):

Robert Hrabak, Technical Manager — Date 7/2/18	Joe Schairer, Health & Safety Manager / Coordinator — Date 7/2/18
Lisa Stafford, Quality Assurance Manager — Date 6/29/2018	Crystal Pollock, Laboratory Director — Date 7.2.18

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1. SCOPE AND APPLICATION

1.1. This SOP is applicable to the determination of settleable solids, total solids, total suspended solids, total dissolved solids, and suspended solids using gravimetric techniques. This SOP is based on the residue methods contained in *Methods for Chemical Analysis of Waters and Wastes* (MCAWW) and *Standard Methods for the Examination of Water and Wastewater* (SM).

1.2. This SOP is applicable to drinking, surface, and saline waters and domestic and industrial wastes.

1.3. This SOP is also applicable to solid and semisolid samples such as river and lake sediments, sludges separated from water and wastewater treatment processes, and sludges from vacuum filtration, centrifugation, or other sludge dewatering processes following Standard Method 2540G.

1.4. The Total Solids (TS) protocol is based on MCAWW Method 160.3 and Standard Methods 2540B and 2540G.

1.5. The Total Dissolved Solids (TDS) protocol is based on MCAWW Method 160.1 and Standard Method 2540C.

1.6. The Total Suspended Solids (TSS) protocol is based on MCAWW Method 160.2 and Standard Method 2540D.

1.7. Methods 160.1, 160.2, 160.3 and 160.5 were withdrawn by the Methods Update Rule (MUR) of March 12, 2007, and replaced by the appropriate Standard Methods for the Examination of Water and Wastewater.

1.8. The method covers a range of 10 mg/L to 20,000 mg/L for TS and TDS. The range is 5 mg/L - 20,000 mg/L for TSS. As a practical matter, the final residue weight of a field sample shall be limited to between 2.5 mg and about 200 mg. For TS on solid samples, the range is 0.1% to 100% with an aliquot size of 5-10 g.

1.9. When undertaking projects for Department of Defense (DoD) and/or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, “Federal Program Requirements”, must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Total Solids (TS): A well-mixed aliquot of the sample is quantitatively transferred to a pre-weighed evaporating dish and evaporated to dryness at 103-105°C. The increase in weight over that of the weight of the empty dish represents the total solids. For solid samples, the result is reported in % Total Solids.

2.2. Total Dissolved Solids (TDS): A well-mixed aliquot of the sample is filtered through a glass fiber filter. The filtrate is quantitatively transferred into a pre-weighed evaporating dish, evaporated in an oven set slightly below boiling (96-98°C), and dried to a constant weight at 180 ± 2°C. The increase in weight over that of the weight of the empty dish represents the total dissolved solids. The filter from this procedure may also be used for TSS determination.

2.3. Total Suspended Solids (TSS): A well-mixed aliquot of the sample is filtered through a pre-weighed glass fiber filter. The residue on the filter is dried to a constant weight at 103-105°C. The increase in weight over that of the pre-weighed filter represents the TSS content. The filtrate from this procedure may be used for TDS determination.

3. DEFINITIONS

3.1. Total Solids (TS): The term applied to the residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at 103-105°C. Total solids includes "total suspended solids," the portion of solids retained by a filter, and "total dissolved solids," the portion that passes through the filter.

3.2. Total Dissolved Solids (TDS): Those solids passing through a 2.0 µm nominal pore size (or smaller) glass fiber filter and dried to constant weight at 180 ±2°C. TDS is also referred to as filterable residue.

3.3. Total Suspended Solids (TSS): Those solids retained by a 2.0 µm nominal pore size (or smaller) glass fiber filter and dried to constant weight at 103-105°C. TSS is also referred to as non-filterable residue.

3.4. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.5. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. These materials must be routinely demonstrated to be free from interferences under the conditions of analysis by running method blanks.

4.2. Non-homogeneous samples may give erroneous results. Samples shall be mixed as thoroughly as possible before removing an aliquot for analysis.

4.3. Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter shall be excluded from the sample if it is determined that their inclusion is not desired

in the final result. The presence/removal of these artifacts shall be noted in the Laboratory Information Management System (LIMS) referred to as TALS.

4.4. Samples containing large amounts of solids may filter slowly. Prolonged filtration times resulting from filter clogging may produce high TSS results due to increased colloidal materials captured on the clogged filter. For samples with visible high TSS content, use a smaller volume.

4.5. Oil and grease in the samples may cause unreliable results due to difficulty in drying to constant weight. Floating oil and grease, if present, shall not be included in the sample. The Project Manager must be informed and an anomaly filed when this type of sample is analyzed. Oil and grease can be included for TS analysis if dispersed with a blender prior to pouring.

4.6. Filtration apparatus, filter material, pre-washing, post-washing, and drying temperatures are specified because these variables have been shown to affect the results.

4.7. The temperature at which the residue is dried has an important bearing on the results. Weight losses due to volatilization of organic matter, mechanically occluded water, water of crystallization, and gases from heat-induced chemical decomposition, as well as weight gains due to oxidation, depend on temperature and time of heating.

4.8. Pay close attention to desiccation after drying. Minimize opening the desiccator because moist air may enter. Some samples may be stronger desiccants than those used in the desiccator and may take on water.

4.9. Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation, and rapid weighing.

4.10. TDS samples containing high concentrations of bicarbonate may require careful and possibly prolonged drying to ensure that all the bicarbonate is converted to carbonate.

4.11. Total residue should be limited to about 200 mg as some samples dry with the formation of a crust that prevents water evaporation, requiring special handling to deal with this.

4.12. Some samples may have fine suspended solids, which pass through the glass fiber filter causing high TDS results.

4.13. Aluminum pans should not be used for TS or TDS analyses. Components in some samples may react to form aluminum compounds, causing unreliable results.

4.14. For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material prior to TSS determination.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1. Specific Safety Concerns or Requirements

5.1.1. The use of vacuum systems during sample filtering presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed, or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.2. Analysts must use tongs or heat protective gloves when handling hot materials being taken out of ovens.

5.1.3. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically-resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, PVC, and nitrile gloves all provide adequate levels of protection against the chemicals used in this SOP.

5.1.4. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, all samples must be opened, transferred, and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.5. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

There are no materials with a health rating of 3 or 4 used in this method that have a serious or significant hazard rating. A complete list of materials used in the method can be found in Section 7. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6. EQUIPMENT AND SUPPLIES

6.1. Analytical balance capable of weighing to 0.0001 g.

6.2. Vacuum filtration apparatus.

6.3. Vacuum pump equipped with moisture trap.

6.4. Glass fiber filter disks, 47 mm, without organic binder (eg. Whatman Filter Paper 934-AH) or equivalent, 2.0 µm nominal pore size (or smaller).

Note: *Commercially available pre-weighed filters may be used for TSS analysis.*

6.5. Desiccant containing a color indicator of moisture concentration or an instrumental indicator.

6.6. Drying ovens set at 103-105°C and 180 ± 2°C. Separate ovens should be maintained at appropriate temperatures if possible.

6.7. Thermometers, NIST traceable.

6.8. Conductivity meter and associated apparatus.

6.9. Glass beakers/evaporation dishes, 'to contain', assorted sizes.

6.10. Graduated cylinders, Class A, assorted sizes.

6.11. Volumetric flasks, Class A, assorted sizes.

6.12. Aluminum weighing dishes large enough to hold a 47 mm filter.

6.13. Forceps for handling filters.

6.14. Crucible tongs.

6.15. Zetex gloves or other gloves capable of providing protection at 180°C.

7. REAGENTS AND STANDARDS

7.1. Reagent water: Distilled or deionized water, free of analyte of interest. Water must contain particles no larger than 0.20 micron and have a resistance of at least 18 ohms.

7.2. LCS for TSS: Obtain a commercially prepared standard, such as ERA Hardness standard. True values vary depending on the specific lot obtained.

7.3. LCS for TDS and TS: Obtain a commercially prepared standard, such as ERA Minerals standard. True values vary depending on the specific lot obtained.

7.4. Spike solutions for matrix spikes: 5,000 mg/L NaCl. Weigh 5.0 g of reagent grade sodium chloride into a 1 L volumetric flask and dilute to volume with reagent water.

7.5. Commercially available reference materials may also be used.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Samples should be collected in either plastic or glass bottles.

8.2. Samples must be stored at 4 ± 2°C to minimize microbiological decomposition of solids. Bring samples to room temperature before analysis.

8.3. To achieve the reporting limits listed, the recommended minimum volumes are as follows:

Parameter	Volume (mL)	Reporting Limit
TS	100	10 mg/L
TDS	100	10 mg/L
TSS	100	5 mg/L

Smaller volumes can be used when high analyte levels are suspected. Reporting limits must be adjusted accordingly.

8.4. For TS on solid samples, the reporting limit is 0.1 % Total Solids, with an aliquot size of 5-10 g.

8.5. Low level TSS can be done with this method using 500 mL of sample. The reporting limit is 1 mg/L. The volume used for the prep blank must be the same as the sample. The LCS volume must be 100 mL to prevent filter overload. Low level TSS is done only if specifically requested by the client.

8.6. The holding times for TS, TDS, and TSS are 7 days.

8.7. The holding time for SS is 48 hours.

9. QUALITY CONTROL

The QC Program document (WS-PQA-003) provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

9.1. Table 1 at the end of this section provides a summary of quality control requirements including type, frequency, acceptance criteria, and corrective action.

9.2. Initial Demonstration of Capability (IDOC): This requires the analysis of four QC check samples. The QC check sample is a well-characterized, laboratory-generated sample containing the analyte(s) of interest that is used to monitor method

performance. The results of the initial demonstration study must be acceptable before the analyst performs the analysis of samples under this SOP. Further details are in Section 13.2.

9.3. Batch Definition: The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a MB, LCS, and a MS/MSD. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See policy WS-PQA-003 for further definition of the batch.

9.4. Method Blank (MB): One MB must be processed with each batch of 20 or fewer samples. The MB consists of reagent water that is carried through the entire analytical procedure, including filtration as applicable. The MB must be processed at the same time and in the same manner as the associated samples. The MB is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The volume of reagent water used must be the same as the volume listed in Section 8.3. Note: See 8.5 for low level TSS volume.

9.5. Laboratory Control Sample (LCS): One LCS must be processed with each batch of 20 or less samples. The LCS must be carried through the entire analytical procedure. The LCS must be processed at the same time and in the same manner as the associated samples. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Refer to Section 7 for instructions on how to obtain the LCS solutions. The LCS must be the same volume as the method blank, except for low level TSS. See Section 8.5.

9.6. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair must be processed with every process batch of similar matrix, not to exceed twenty (20) samples. The MS/MSD are aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside control limits must be within control limits for the LCS. Corrective actions must be documented in a Non-Conformance Memo (NCM), then implemented when recoveries of any spike analyte is outside control limits provided in LIMS or by the client. Reprocessing of the MB, LCS, the selected field sample(s), and/or the MS/MSD may be required after evaluation and review. Note: For TSS the MS/SD is not applicable. See Section 9.7 and 9.8.

9.7. A duplicate control sample (LCSD or DCS) may be substituted when insufficient sample volume is provided to process a MS/MSD pair if required by client or program. The LCSD is evaluated in the same manner as the LCS.

9.8. Sample Duplicate (DU): A duplicate sample is required for TSS analysis. Sample results should agree within 20 % if both the sample and sample duplicate results are >5X RL.

TABLE 1

Acceptance Criteria and Corrective Actions-Residue

QC Type	*Acceptance Criteria*	*Corrective Action*
Laboratory Control Sample (LCS)	TDS: 80%-120% Recovery TSS: 85%-115% Recovery TS: 80%-120% Recovery	Re-pour and reanalyze the batch unless the client agrees that another corrective action is acceptable.
Method Blank (MB)	±Reporting Limit	Re-pour and reanalyze all associated field samples with reportable levels of analyte. If all associated field sample results are ND, report the data and file an anomaly. Also, if contamination exceeds the RL in the method blank, but the contamination is less than 10x the amount of TDS in the samples, narrate and report.
Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) Spike Level- (500 ppm)	TDS: 85%-115% Recovery, 20% RPD TS: 80%-120% Recovery, 20% RPD	Re-pour and reanalyze to confirm if RPD fails.
Sample Duplicate (DU)	20% RPD; does not apply if sample and duplicate results are <5X the RL.	Re-pour and reanalyze for confirmation.
TDS/EC (electrical conductivity) ratio	0.47 ~ 0.93	See Below

Table 2	
Corrective Actions for Residue	
Step 1: Investigate the physical properties of the sample.	1. Floatables, sediment content, viscosity, color, reaction to air exposure, smell, hygroscopic crystals, difficulty drying, etc. are enough to cause a failed TDS/EC ratio. Results can be reported with a note on the benchsheet when these apply. 2. If the sample spilled or splattered, or too much sediment was present, the sample must be reanalyzed.
Step 2: Reanalyze for EC	1. If the rerun EC confirms the original, and the TDS/EC ratio still fails, rerun TDS. 2. If the rerun EC differs from the original, and the TDS/EC ratio passes, report the TDS result and record new EC result. 3. EXCEPTIONS 3.1. If the EC is < 50 umhos/cm and TDS is < 200mg/L, report result. Note on the benchsheet that the TDS/EC ratio does not apply to samples with EC < 50 uhmos/cm. 3.2. If the EC is > 20,000 umhos/cm, and TDS is > 18,000mg/L, report result. Note on the benchsheet that the TDS/EC ratio does not apply to samples with EC > 20,000 uhmos/cm.
Step 3: Reanalyze for TDS	If the rerun TDS confirms the original, and the TDS/EC ratio still fails, report the original with a note on the benchsheet. If the rerun TDS differs from the original, and the TDS/EC ratio passes, report the rerun. File an HTV, if needed.

10. CALIBRATION

10.1. Since this method is based on gravimetric techniques, there is no calibration in the usual sense. Proper balance operation will be verified daily, prior to sample analysis, by following the balance calibration SOP WS-QA-0041. Analytical balance calibration must be performed daily (every 24 hours).

10.1.1. Preventative Maintenance for Balances

On a daily basis, clean the pan and weighing compartment, to ensure that no dust or particles are interfering with the movement of the pan.

10.2. The oven temperature must be monitored using a calibrated thermometer on days the unit is in use and recorded in TALS and on the Oven Temperature Log, form QA-436, located in the temperature logbook for the oven. The Thermometer S/N, Oven ID #, and temperature range are recorded on the Oven Temperature Log form prior to use. Date/Time (MM/DD/YY HH:MM), Analyst initials, Sample Status, Observed/Corrected Temperature (°C), Test (TS/TSS/TDS), Sample Batch number, and Temp ok status (Y/N) are recorded on the Oven Temperature Log form when samples or sample remains are placed in the oven or taken out of the oven. If temperature readings fall outside of the acceptable range, notify the QA Department and file a NCM. If the unit was used on a day the temperature was not recorded,

contact the QA Department and the Department Manager. File a NCM for all affected samples.

10.2.1. Preventative Maintenance for Ovens

The electronics are serviced on an as-needed basis.

10.3. The conductivity of the water must be monitored and recorded daily in the Conductivity Logbook following the water monitoring SOP WS-QA-0014. The conductivity must be less than 1.0 umhos/cm (at 25°C). If the conductivity reading on the water system exceeds this level, do not use the water for these procedures and notify the supervisor immediately.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a NCM and must be approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified.

11.2. Any unauthorized deviation from this procedure must also be documented in a NCM with a cause and corrective action described.

11.3. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

11.4. Proper sample identification is extremely important in any analytical procedure. Labeling of evaporating dishes and filter holders must be done in a manner to ensure connection with the proper sample.

11.5. If possible, analyze all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

11.6. Note any sample abnormalities in TALS during pouring and after drying.

11.7. If there is limited sample volume or high solid content, smaller amounts of sample may need to be processed then detailed in the following sections. This occurrence must be noted in TALS and reporting limits must be adjusted appropriately.

11.8. Proceed to the appropriate section for the desired method as follows:

Total Solids (TS)	11.9
Total Dissolved Solids (TDS)	11.10
Total Suspended Solids (TSS)	11.11

11.9. Total Solids, 2540B and 2540G

11.9.1. Preparation of Evaporating Dish

11.9.1.1. If only total solids are to be measured, heat clean dish to 103-105°C for one hour. Transfer dishes to dessicator for at least 1.5 hours.

Note: *To maintain consistent conditions, the drying time and cooling time between weighings should be the same.*

11.9.1.2. Weigh immediately to the nearest 0.1 mg. Record the weight in TALS.

11.9.1.3. Store the dish in the desiccator until ready to use.

11.9.2. Sample Preparation

Prescreen the samples using a calibrated conductivity meter to determine the required sample volume or dilution. Record the sample conductivity in TALS. See Section 12.1 for formula, if needed.

11.9.3. Sample Analysis

11.9.3.1. Transfer a measured aliquot of well-mixed sample to the pre-weighed, labeled dish. Record the volume of the sample to the nearest mL in TALS.

11.9.3.2. Record the date and time the samples were poured. This will be used as the analysis time.

11.9.3.3. For soil and solid samples, weigh 5-10 grams of well-homogenized sample into the dish.

11.9.3.4. For the LCS, measure 100 mL of the LCS solution (Section 7.2) and pour into the dish.

11.9.3.5. For the MB, measure 100 mL of reagent water and pour into the dish.

11.9.3.6. For MS/MSD aliquots, add 10 mL of spike solution (Section 7.3) to the sample in the dish.

11.9.3.7. Evaporate the sample to dryness in a drying oven. The temperature should be lowered down to 98°C to prevent boiling and splattering of the sample.

11.9.3.8. Dry the evaporated sample for at least one hour at 103-105°C. Dry soil and solid samples for % TS for a minimum of 12 hours at 103-105°C.

11.9.3.9. **Using tongs and heat resistant gloves**, remove the weighing dish from the oven and place in a desiccator and cool to room temperature for 1.5 hr.

11.9.3.10. Weigh the dish to the nearest 0.1 mg. Record the weight in TALS.

11.9.3.11. Return the samples to the oven for another hour, then cool in a desiccator for 1.5 hours, and reweigh. If the weights are constant, analysis is complete. If the weights vary by more than 4% of the previous weight or 0.5 mg (whichever is less), repeat the drying, cooling, desiccating and weighing process.

Note: *For residue values > 12.5 mg, 0.5mg is an appropriate cut-off for determining constant weight.*

Note: *When weighing a dried sample, be alert to changes in weight due to air exposure and/or sample degradation. Minimize time spent in weighing or dish exposure to ambient air.*

11.9.3.12. If sample fails to reach a constant weight after three weighings, the lowest final weight is used and an NCM is filed.

11.9.3.13. See Sections 12.2 and 12.3 for equations, if needed.

11.10. Total Dissolved Solids, 2540C

11.10.1. Preparation of Evaporating Dishes

11.10.1.1. If only total dissolved solids are to be measured, heat clean dish to 180 ± 2°C for one hour. Transfer dishes to dessicator for at least 1.5 hours.

11.10.1.2. Weigh immediately to the nearest 0.1 mg. Record the weight in TALS.

11.10.1.3. Store in the desiccator until ready to use.

11.10.2. Sample Preparation

Prescreen the samples using a calibrated conductivity meter to determine the required sample volume or dilution. See Section 12.1 for formula, if needed.

Note: *TDS is typically 55%-90% of the conductance result. The exact relationship depends on the compounds present in the samples and may not hold for very high concentrations, for samples containing non-ionic species, or for samples with conductance greater than 10,000 umhos/cm or less than 10 umhos/cm. 2 mL is the smallest volume that can be used for analysis.*

Conductance Value (umhos/cm)	Sample Volume (mL)
<2000	100
2,000-5,000	50
5,000-7,500	25
7,500-10,000	10
10,000-20,000	5
>20,000	2

11.10.3. Sample Analysis

11.10.3.1. Thoroughly rinse the entire filtration apparatus with reagent water before filtering each sample.

11.10.3.2. Assemble the filtering apparatus, place a glass fiber filter in the apparatus, pre-wet the filter using reagent water, and begin suction.

Note: *If the sample also requires TSS, pre-weigh the prepared filter and refer to Section 11.11 for additional guidance.*

11.10.3.3. Shake the sample vigorously and rapidly transfer 100 mL (or a smaller portion which will yield between 10 and 200 mg dried residue) to the filter funnel by means of a graduated cylinder. If more than 10 minutes are required to complete filtration, decrease sample size.

Note: *For samples high in sediment that need to be analyzed for TDS only, obtain a smaller sample aliquot for the TDS analysis. Additionally, multiple filters may be used if performing only TDS analysis (and not TSS).*

11.10.3.4. For the MB, process 100 mL of reagent water.

11.10.3.5. For the LCS, process 100 mL of the LCS solution. Refer to Section 7.3 for instructions on how to obtain the LCS.

11.10.3.6. For MS/MSD aliquots, add 10 mL of the spiking solution (Section 7.3).

11.10.3.7. Rinse the graduated cylinder, funnel walls, and filter with three successive 10 mL portions of reagent water and allow for complete drainage between washings. Continue to apply vacuum after filtration is complete to remove as much water as possible.

11.10.3.8. Transfer the filtrate (including the washings) to a pre-weighed evaporating dish. Rinse the receiving flask with 10-25 mL of reagent water and transfer washings into the dish to ensure complete transfer of the sample.

Note: *Record the date and time the samples were poured. This will be used as the analysis time.*

11.10.3.9. Evaporate the samples to dryness in an oven set slightly below boiling (96-98°C). If filtrate volume exceeds dish capacity, add successive portions to the same dish after evaporation.

11.10.3.10. Dry the evaporated sample in an oven for at least one hour at 180 ± 2 °C.

11.10.3.11. **Using tongs and heat resistant gloves**, remove the weighing dish from the oven and place in a desiccator and cool to room temperature for 1.5 hr.

11.10.3.12. Immediately weigh the dish to the nearest 0.1 mg. Record the weight in TALS.

11.10.3.13. Return the samples to the oven for another hour, cool in a desiccator for 1.5 hours, and reweigh. If weights are constant, analysis is complete. If weights vary by more than 4% of the previous weight or 0.5 mg (whichever is less), repeat the drying, cooling, and desiccating process.

Note: *For residue values > 12.5 mg, 0.5mg is an appropriate cut-off for determining constant weight.*

11.10.3.14. If sample fails to reach a constant weight after three weighings, the lowest final weight is used and an NCM is filed.

11.10.3.15. See Sections 12.4 and 12.5 for equations, if needed.

11.11. Total Suspended Solids, 2540D

11.11.1. Preparation of Glass Fiber Filter Disc

Note: *As an alternative to the steps outlined below, prepared and pre-weighed filters may be purchased for use under this method.*

11.11.1.1. Place the glass fiber filter discs, one at a time, in the membrane filtration apparatus.

11.11.1.2. While the vacuum is applied, wash the disc with three successive 20 mL volumes of distilled water.

11.11.1.3. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings.

11.11.1.4. Remove the filter from the membrane filtration apparatus, place in a labeled aluminum weighing dish and dry in an oven at 103-

105°C for one hour.

11.11.1.5. **Using tongs and heat resistant gloves**, remove the weighing dish from the oven, place in a desiccator and cool to room temperature for one hour.

11.11.1.6. Weigh the cooled filter using an analytical balance to the nearest 0.1 mg. Record the weight in TALS. Handle the filters only with forceps.

11.11.2. Selection of Sample Volume

11.11.2.1. For a 47 mm diameter filter, filter 100 mL of sample or less depending on the visible particulates present in the sample. Limit the sample size to that yielding no more than 200 mg residue.

11.11.2.2. If during filtration of this initial volume the filtration rate drops rapidly, or if filtration time exceeds 5-10 minutes, a smaller volume of sample should be processed.

11.11.2.3. Record the sample volume used in TALS.

11.11.3. Sample Analysis

11.11.3.1. Assemble the filtering apparatus, place the pre-weighed glass fiber filter in the apparatus, pre-wet the filter using reagent water, and begin suction.

Note: *Handle the filters only with forceps.*

11.11.3.2. Shake the sample vigorously and quantitatively transfer 100 mL (or an appropriate aliquot) of the sample to the filter using a graduated cylinder or pipet.

Note: *If Total Dissolved Solids (TDS) is also required, the filtrate may be used. Refer to Section 11.10 for additional guidance.*

11.11.3.3. Remove all traces of water by continuing to apply vacuum after the sample has passed through.

11.11.3.4. With suction on, rinse the graduated cylinder, filter, suspended solids residue, and filter funnel wall with three 10 mL portions of reagent water allowing complete drainage between washings.

11.11.3.5. Remove all traces of water by continuing to apply vacuum after the sample has passed through.

11.11.3.6. Carefully remove the filter from the filter support and transfer to

a weighing dish or aluminum pan.

11.11.3.7. Record the date and time the samples were poured. This will be used as the analysis time.

11.11.3.8. Dry the filter for at least one hour at 103-105°C.

11.11.3.9. **Using tongs and heat resistant gloves**, remove the weighing dish from the oven and place in a desiccator and cool to room temperature for one hour.

11.11.3.10. Immediately weigh the filters to the nearest 0.1 mg. Record the weight in TALS.

11.11.3.11. Return the samples to the oven for another hour, cool in a desiccator for 1 hour, and reweigh. If weights are constant, analysis is complete. If weights vary by more than 4% of the previous weight or 0.5 mg (whichever is less), repeat the drying, cooling and desiccating process.

11.11.3.12. If sample fails to reach a constant weight after three weighings, the lowest final weight is used and an NCM is filed.

11.11.3.13. See Section 12.6 for equations, if needed.

12. CALCULATIONS/DATA REDUCTION

12.1. The following formula should be used to approximate the required sample volume:

(2000 x 100)/Conductivity = mL sample to use

Note: *To avoid "oddball" reporting limits, round the determined sample volume to one of the following volume increments: 2, 5, 10, 20, 25, 50 or 100 mL.*

12.2. Calculate **Total Solids, 2540B** as follows:

$$\text{Total Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg) (Use lowest final weight achieved.)
B = weight of dish (mg)
C = volume of sample (mL)

12.3. Calculate **% Total Solids, 2540G** as follows:

% Total Solids = ((A-B) x 1000)/(C-B)

Where: A = weight of dried residue + dish (mg) *(Use lowest final weight achieved.)*
B = weight of dish (mg)
C = weight of wet sample + dish (mg)

12.4. Calculate **Total Dissolved Solids, 2540C** as follows:

$$\text{Total Dissolved Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg) (Use lowest final weight achieved.)
B = weight of dish (mg)
C = volume of sample (mL)

12.5. Calculate the expected TDS range from conductance (as umhos/cm).

Expected TDS_{low} (mg/L) = (Conductance x 0.55)

Expected TDS_{high} (mg/L) = (Conductance x 0.9)

12.6. Calculate **Total Suspended Solids, 2540D** as follows:

$$\text{Total Suspended Solids, mg / L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of filter + residue (mg) (Use lowest final weight achieved.)
B = weight of filter (mg)
C = volume of sample filtered (mL)

12.7. The lowest final sample weight is used for calculating solids content.

12.8. If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility of ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.2.1. MDLs and MDL verifications for TS and TDS are conducted using the NaCl spike described in Section 7.4 diluted as needed.

13.2.2. MDLs and MDL verifications for TSS are conducted using the LCS solutions (Section 7.2). The appropriate volume to provide approximately 5mg/L in 100 mL is determined, and that volume is added to sufficient reagent water to make a total of 100 mL in the funnel.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. For TS and TDS, four aliquots are prepared. Each aliquot consists of 0.8 mL of the NaCl matrix spike solution (Section 7.4) and 100 mL reagent water. These four aliquots analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of each aliquot is 40 (39.6825) mg/L.

13.3.2. For TSS, four aliquots are prepared using the LCS solution (Section 7.2). These four aliquots analyzed using the same procedures used to analyze samples, including sample preparation.

13.3.3. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.5. The reporting limit is 10 mg/L for TS, and TDS.

13.6. The reporting limit is 5 mg/L for TSS.

13.7. The Initial Demonstration of Capability study as detailed in Section 9.2 must be acceptable before the analysis of field samples under this SOP may begin.

14. POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out.

15.1. Used filters, miscellaneous disposable glassware, and contaminated gloves are disposed of in the yellow hazardous lab trash buckets. When the bucket is full, or after no longer than one year, tie the plastic bag liner shut and put the lab trash into the

hazardous landfill steel collection drum in the H3 closet. When the drum is full, or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Liquid samples that have been filtered as part of the analytical process are collected in a satellite carboy. When full, or after no more than one year, this carboy is moved to the waste disposal area for consolidation and disposal.

16. REFERENCES/CROSS REFERENCES

16.1. Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1979: Methods 160.1, 160.2, 160.3, 160.4 and 160.5.

16.2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992: Methods 2540A-G and 2510A.

16.3. Standard Methods for the Examination of Water and Wastewater, 19th Edition, 1995: Methods 2540A-G and 2510A.

16.4. Standard Methods for the Examination of Water and Wastewater, Methods 2540B-1997, 2540C-1997, 2540D-1997 and 2510B-1997.

16.5. 40 CFR Parts 122 and 136.

16.6. Quality Control Program, WS-PQA-003, current revision.

16.7. Calibration and Calibration Check of Balances, WS-QA-0041, current revision.

16.8. Monitoring of Reagent-Grade Laboratory Water, WS-QA-0014, current revision.

17. METHOD MODIFICATIONS

17.1. Samples are not taken through the drying, cooling, and weighing cycle more than three times. If the sample fails to reach constant weight within three cycles the lowest final weight is used for the calculation, and an NCM is filed.

17.2. The filter cleaning procedure outlined in Method 160.1 and SM2540C is not applied. This SOP requires the use of method blanks to document system cleanliness.

17.3. All samples are not analyzed in duplicate. Instead, one sample duplicate or a matrix spike and matrix spike duplicate are analyzed per batch. This allows for precision monitoring.

17.4. Additional sample is not added to the dish until the residue is at least 25 mg. Instead, the volumes listed in Section 8.3 are used to obtain the established reporting limits. Dilutions can be made when high analyte levels are suspected.

17.5. Duplicates must agree within 20% of each other, instead of 5% of their average, as listed in reference method 2540.

17.6. Method 160.3 (TDS), Section 7.5 specifies that samples are evaporated to dryness on a steam bath. This SOP specifies (Section 11.10.3.9) that samples are evaporated to dryness in an oven set slightly below boiling . Both techniques accomplish the same goal, to reduce spattering and potential loss of sample during evaporation.

17.7. SM 2540D specifies transferring a stirring sample using a pipette. This SOP specifies (Section 11.11.3.2) that a vigorously shaken sample aliquot may be quantitatively transferred by graduated cylinder or pipette.

17.8. SM 2540D indicates that the washed and dried filter should go through more than one weighing cycle to establish a constant weight. This SOP specifies (Section 11.11.1) that the filter is weighed only once.

18. ATTACHMENTS

18.1. Figure 1 - Oven Temperature Log, QA-436.

19. REVISION HISTORY

19.1. WS-WC-0002, Revision 4.8, Effective 07/03/2018

19.1.1. Section 11.10.2 table revised, “<2,500” to “<2,000” and “2,500-5,000” to “2,000-5,000”.

19.1.2. Section 15.1 revised to, “Used filters, miscellaneous disposable glassware, and contaminated gloves are disposed of in the yellow hazardous lab trash buckets. When the bucket is full, or after no longer than one year, tie the plastic bag liner shut and put the lab trash into the hazardous landfill steel collection drum in the H3 closet. When the drum is full, or after no more than 75 days, move it to the waste collection area for shipment.”

19.1.3. Removed revision history prior to 2016. It can be found in previous versions of this SOP.

19.1.4. Editorial changes.

19.2. WS-WC-0002, Revision 4.7, Effective 07/24/2017

19.2.1. Section 11.10.3.9, changed “95 - 98°C” to “96 - 98°C”.

19.2.2. Section 11.10.3.13, changed “1 – 1.5 hours” to “1.5 hours”.

19.2.3. Editorial changes.

19.3. WS-WC-0002. Revision 4.6, Effective 07/03/2017

19.3.1. Section 9.8, Table 1, revised Acceptance Criteria for Sample Duplicate to read "20% RPD; does not apply if sample and duplicate results are <5X the RL" to clarify.

19.3.2. Section 9.8, Tables 1 and 2, changed all references of "EC/TDS ratio" to "TDS/EC ratio" to reflect the way the calculation is set up in TALS.

19.3.3. Editorial changes.

19.4. WS-WC-0002, Revision 4.5, Effective 10/11/2016

19.4.1. Table 1 - Method Blank, Corrective Action column: Added - "Also, if contamination exceeds the RL in the method blank, but the contamination is less than 10x the amount of TDS in the samples, narrate and report."

19.4.2. Editorial changes.

19.5. WS-WC-0002, Revision 4.4,, Effective 7/08/2016

19.5.1. Inserted Section 1.9 – "When undertaking projects for Department of Defense (DoD) and/or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, "Federal Program Requirements", must be checked and incorporated."

19.5.2. Section 10.2 – Updated to reflect use of thermocouple thermometers rather than digital oven display for monitoring purposes.

19.5.3. Updated NOTE following Section 11.10.3.3.

19.5.4. Inserted Figure 1 – Oven Temperature Log (QA-456).

19.5.5. Editorial changes.

Figure 1

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Sacramento
Oven Temperature Log
(In Use Monitoring Only)

Thermometer S/N: _____ Oven ID #: _____

Date/Time (MM/DD/YY HH:MM)	Initials	Sample Status (Check one) In	Remains In	Out	Temp (Centigrade) Obs	Corr.	Test (See Below)	TALS Batch(es)	Temp ok? Y/N

Temperatures: **Obs** = Observed Temperature. Always complete this column. **Corr** – Corrected Temperature. Write "NA" unless the thermometer has a correction factor. If a correction factor, write the corrected value (ex: Obs. value is 100, correction factor is 2, then corrected value is 100-2 = 98. Add the correction factor to the comments (below).
If temperature readings fall outside the appropriate range (listed below), inform the QA Department and file an anomaly. Temperature is only monitored on days the unit is in use. If the unit has been used on a day that the temperature was not taken, contact QA and department manager. File an anomaly for all affected samples.

Acceptance Criteria:

Test Method/Phase	Temperature
Total Solids (TS)	103 - 105
Total Suspended Solids (TSS)	103 - 105
Percent Moisture (%M)	105 - 120

Test Method	Test Phase	Temperature
Total Dissolved Solids (TDS)	Pre-Treatment (TDS-PRE)	180 ± 2 (178 – 182)
	Evaporation (TDS-EVAP)	96 - 98
	Post-Evaporation (TDS-POST)	180 ± 2 (178 – 182)

COMMENTS: _____

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Appendix B

Standard Operating Procedures

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Standard Operating Procedure No. 001
for
Sample Labels

Prepared by

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Revision 0
December 2014

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1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to delineate protocols for the use of sample labels. Every sample will have a sample label uniquely identifying the sampling point and analysis parameters. An example label is provided below. Other formats with similar levels of detail are acceptable.

PROJECT NAME _____ PROJECT NUM. _____
SAMPLE LOCATION/SITE ID _____
DATE: ____/____/____ TIME: _____:_____
ANALYTES: METALS VOC EXPLOSIVES ORGANICS OTHER
FILTERED: [NO] [YES]
PRESERVATIVE: [NONE] [HNO_3] [OTHER _____]
SAMPLER: _____

2. MATERIALS

The following materials may be required: sample label and indelible laboratory marker.

3. PROCEDURE

The following sections describe how to use the sample labeling system.

3.1 LABEL INFORMATION

As each sample is collected/selected, fill out a sample label. Enter the following information on each label:

- Project name
- Project number
- Location/site identification—Enter the media type (i.e., well number, surface water, soil, etc.) sampling number, and other pertinent information concerning where the sample was taken
- Date of sample collection

- Time of sample collection
- Analyses to be performed (NOTE: Due to number of analytes, details of analysis should be arranged with laboratory *prior to start of work)*
- Whether filtered or unfiltered (water samples only)
- Preservatives (water samples only)
- Number of containers for the sample (e.g., 1 of 2, 2 of 2).

3.2 ROUTINE CHECK

Double-check the label information to make sure it is correct. Detach the label, remove the backing, and apply the label to the sample container. Cover the label with clear tape, ensuring that the tape completely encircles the container.

3.3 RECORD INFORMATION

Record the sample number and designated sampling point in the field logbook, along with the following sample information:

- Time of sample collection (each logbook page should be dated)
- Location of the sample
- Organic vapor meter or photoionization meter readings for the sample (when appropriate)
- Any unusual or pertinent observations (oily sheen on groundwater sample, incidental odors, soil color, grain size, plasticity, etc.)
- Number of containers required for each sample
- Whether the sample is a quality assurance sample (split, duplicate, or blank).

3.3.1 Logbook Entry

A typical logbook entry might look like this:

- 7:35 a.m. Sample No. MW-3. PID = 35 ppm
- Petroleum odor present. Sample designated MW-3-001.

NOTE: Duplicate samples will be given a unique sample designation rather than the actual sample number with an added prefix or suffix. This will prevent any indication to the laboratory that this is a duplicate sample. This fictitious sample number will be listed in the logbook along with the actual location of the sample.

3.4 SHIPMENT

Place the sample upright in the designated sample cooler. Make sure there is plenty of ice in the cooler at all times.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

5.1 INCIDENTAL ODORS

Note that although incidental odors should be noted in the logbook, it is unwise from a safety and health standpoint to routinely "sniff test" samples for contaminants.

5.2 DUPLICATE SAMPLE

No indication of which samples are duplicates is to be provided to the laboratory.

6. REFERENCES

U.S. Environmental Protection Agency. 1980. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans. QAMS-005/80.

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Standard Operating Procedure No. 004
for
Sample Packing and Shipping

Prepared by

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Revision 1
September 2018

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PROJECT-SPECIFIC VARIANCE FORM

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

☐ **A. Variances required; cite section(s) of the SOP to which there is a variance**

☐ **B. No variances**

SOP No. 004	
SOP Section	**Variance**

Project Manager (Name)

Project Manager (Signature) Date

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DOCUMENT REVISION HISTORY

ORIGINAL (MASTER) DOCUMENT REVISION HISTORY				
Revision Number	**Revision Date**	**Revision Summary**	**Revised By**	**Reviewed By**
1	25 September 2018	Systematic update and review	Cristina Radu, Amanda Kohn	Matthew Bowman

1. SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for the packing and shipping of environmental samples to the laboratory for analysis. Additional requirements are applicable when shipping samples under the U.S. Environmental Protection Agency's Superfund Contract Laboratory Program.

NOTE: Samples collected from process wastewater streams, drums, bulk storage tanks, soil, sediment, or water samples from areas suspected of being highly contaminated could require shipment as dangerous goods; procedures for shipping of such samples are not covered in this SOP.

2. MATERIALS

The following materials may be required:

- Clear tape
- Custody seals
- Ice
- Packing material
- Plastic garbage bags
- Sample documentation
- Waterproof coolers (hard plastic)
- Zip-seal plastic bags.

3. PROCEDURE

Refer to SOP Numbers (Nos.) 001, 002, 016, and 039 as applicable.

Samples will be placed in clean, bubble-wrap lined sample coolers with double-bagged ice immediately after collection to ensure proper preservation. Most sample analyses require that the sample material is maintained at 2-6 degrees Celsius (°C). It is also important to ensure that sample containers are maintained at all times at the temperature required by the analytical method used to analyze the sample media; as such, samples should be retained in a chilled cooler during the inventory, quality control, and packaging process.

Check cap tightness and wipe down outside of each sample container. Verify that information on sample labels is correct and matches chain-of-custody forms. Ensure that both waterproof labels and indelible ink are used to label sample containers. Clear tape should be placed completely over the label. Wrap breakable sample containers in bubble wrap. Enclose each sample in a clear zip-seal plastic bag.

Prepare cooler for shipping. Empty any water that has accumulated in coolers from melting ice. Securely seal all valves and/or drain holes in the shipping container, both inside and out, with duct tape to prevent leakage in the event of sample container breakage or melting ice. Place several layers of bubble wrap on top of absorbent material and line the cooler sidewalls with bubble wrap. Line cooler with open garbage bag.

Prepare sample containers for shipping as follows:

- ***Glass Containers***—Wrap each glass sample container in bubble wrap or closed cell foam sheets. It is acceptable to package up to three 40-milliliter vials in one bubble wrap bag that is usually provided by the analytical laboratory. Enclose sample containers in a clear zip-seal plastic bag.

- ***Polyethylene Containers***—Place sample containers in clear zip-seal bags.

- ***Zip-Seal Bags***—Double-bag the samples to ensure that moisture will not reach the label.

Place all the sample containers upright inside garbage bag. Do not stack glass containers or lay them on their sides. Add additional bubble wrap between and around sample containers as needed to ensure containers do not shift during transport. If a second garbage bag was used, tie the (inner) garbage bag to isolate samples.

Double bag and seal loose, fresh ice to prevent melting ice from soaking the packing material. Fill gallon-size or larger zip-seal bags with fresh ice about two-thirds full and squeeze excess air out of the bags before sealing. Turn bag upside down and place in a second zip-seal bag, also removing excess air. Prepare sufficient bags to cover sample containers and ensure that the proper temperature (2-6° C) is maintained during transport.

Place ice on top of sample containers. Ensure that packing material does not insulate samples from ice. Do not use loose ice in sample coolers. Do not use bagged ice as packing material between or around sample bottles. Tie the garbage bag ensuring that the cooler lid will close securely.

Place a temperature blank into the cooler. The temperature blank consists of a plastic bottle containing either potable or deionized water. Temperature blanks are typically provided by the analytical laboratory. If temperature blanks are not provided, field staff must add a clean container filled with deionized water; ensure the cap is tight and container is labeled before placing in cooler.

If aqueous volatile organic analyte samples are being submitted, ensure a trip blank sample set is placed in each cooler containing volatile organic analyte samples. Trip blanks are used to check for contamination of volatile organic compound samples during handling, storage, and shipment from field to laboratory. The trip blanks consist of volatile organic analyte vials filled with deionized water and are typically provided by the analytical laboratory. Ensure that the trip blank samples and analyses are included on the chain-of-custody record.

Make copies of sample documentation (chain-of-custody forms or other field records) and retain in field files for record. Enclose the original field documentation forms in a waterproof plastic bag and tape the bag to the underside of the cooler lid. If more than one cooler is being used, each cooler will have its own documentation.

Seal coolers with signed and dated custody seals such that if the coolers were opened, the custody seals would be broken. Place clear tape over the custody seals to prevent damage to the seals.

Tape the cooler shut with packing tape over the hinges and custody seals. Tape should be wrapped around the cooler a minimum of five times. Ship all samples via overnight delivery on the same day they are collected if possible. Project-specific shipping requirements (e.g., Saturday delivery, communication with the receiving laboratory, etc.) should be discussed with the sample manager or project manager during project planning.

After samples are packaged within shipping containers, place shipping labels clearly on the outside of the container; clearly mark the number of containers in the shipment on the shipping label. Mark each cooler as “1 of 2,” “2 of 2,” etc.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

The project manager and field team leader are responsible for determining if samples collected during a specific field investigation meet the definitions for dangerous goods. If a sample meets or is suspected to meet the definition of “dangerous goods” per the Dangerous Goods Regulation of the International Air Transport Association, then that sample must be handled according to the instructions given for that material. Dangerous goods must be prepared for shipping only by personnel trained and certified by International Air Transport Association in dangerous goods shipment.

6. REFERENCES

Not applicable.

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Standard Operating Procedure No. 057
for
Multi-Incremental Sampling

Prepared by

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Revision 0
December 2014

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1. INTRODUCTION

1.1 SCOPE AND APPLICATION

Multi-incremental (MI) sampling (sometimes designated by the acronym MIS), originally utilized by the mining industry, was initially proposed for environmental sampling at explosives-related sites (U.S. Environmental Protection Agency Solid Waste 846 Method 8330B, Appendix A Collecting and Processing of Representative Samples for Energetic Residues in Solid Matrices from Military Training Ranges [2006]). MI sampling is particularly effective at such sites because explosives residue is found in surface soil as opposed to at depth, and the potentially impacted areas are relatively easy to delineate because the area of the firing ranges is, in most cases, well defined.

Although MI sampling was initially implemented for the assessment of impacts from explosives, there has been recent movement to extend the list of acceptable contaminants to include metals, semivolatile organic compounds, and even volatile organic compounds (State of Alaska Department of Environmental Conservation [2009] and State of Hawai'i Department of Health [2009]). However, the adequacy of MI sampling is evaluated on a case-by case basis at the time the planning documents are prepared to ensure that the resulting analytical data are appropriate to make the decisions required by the project. This evaluation process considers:

1. Planning elements based on the decisions to be made for each potentially complete pathway (based on the conceptual site model), including contaminants distribution, hot spot size, future land use scenarios, contaminant fate and transport, etc.

2. Sample preparation procedures to be employed by the analytical laboratory (limitations and impacts on the analytical data due to the various preparation methods that can be employed)

3. Data evaluation requirements (i.e., the data needing to meet a certain level of confidence). In addition to technical considerations, stakeholders' input is also folded into the planning stages. Consequently, specific field requirements may be outlined in the planning documents for the sampling program implementation to supplement this Standard Operating Procedure (SOP).

This SOP focuses on the actual collection of MI samples, not project planning or data evaluation to follow, and assumes that successful project planning and scoping have been performed, documented, and agreed to by all stakeholders. Because Sampling Units (SUs) are defined so that the mean concentration value obtained is relevant to an explicitly articulated end use of the data, it is imperative that any changes to the SUs or sampling strategy deemed necessary by actual field conditions unanticipated at the time the sampling plan was designed should be made by the project technical lead rather than by field personnel. This way, field deviations from the approved plan during sample collection will not negatively impact the adequacy of the data for the planned purpose.

1.2 GLOSSARY OF TERMS

1.2.1 Sampling Units

An SU (sometimes termed Decision Unit [DU]) is the area and depth of soil (the sampled population) to be characterized by the average concentration of the MI sample. A DU may contain several SUs that are sampled using MI techniques or may consist of just one SU. SUs are restricted to actual source zones and must incorporate only areas that are similar as far as impact (i.e., not to "dilute" contamination) as well as future use. SUs/DUs selected based on future land use scenarios may be called Exposure Units. SUs must be delineated so that the mean analyte concentrations obtained are directly relevant to well defined project objectives. They are the smallest volume of soil for which a concentration value will be obtained, and the basic unit about which a decision or conclusion based on an analytical result can be made.

1.2.2 Decision Units

A DU is a specific area (or volume of soil) about which a decision is to be made. In the ideal and most direct case, the DU and SU are the same volume of soil. As noted above, a DU may be composed of a single SU, or may include multiple SUs, if the DU is very large in size. The important thing is that the entire area of a DU is consistent as far as contamination distribution and future use/exposure scenario, just like an SU. Either all or a percent of the SUs composing the DU may be sampled in an MI fashion, the number of SUs sampled depending on the confidence of the data that are extended from the SUs to the DU.

1.2.3 Grid Cell

A grid cell is a sub-division of the SU. SUs are divided into uniform-size grid cells, and one increment is collected from each cell, from the same relative location within each grid cell. The shape of the cells is not specified—the only criterion for cell shape selection is that the cells should be of equal size (they can be triangular, square, rectangular, etc.) so the increments collected from each cell are equally weighted over the SU.

1.3 GENERAL CONCEPTS

The use of standard discrete samples to characterize soil contamination has two significant sources of error:

1. Field sampling error is at least 10 times greater than analytical (laboratory-associated) error.

2. A source of analytical error was found to be that in sample processing and sub-sampling (a single subsample from the 4- or 8-ounce soil jar is taken at the laboratory).

Depending on the areal and vertical contaminant distribution profile, MI sampling and processing are designed to minimize these sources of error, resulting in an average concentration that is a much more precise and accurate estimate for the SU.

It is also important to note that the horizon characterized by MI sampling is usually superficial, although MI can be implemented at greater depth, this resulting in much higher associated sampling costs.

The purpose of this SOP is to delineate protocols for the application of MI field sampling of surface soil. The procedure, which can be adapted to allow for MI sampling in other environments, i.e., in an excavation trench, has been adapted from U.S. Army Corps of Engineers sampling guidance (2009).

2. EQUIPMENT AND MATERIALS

The following equipment and materials may be required:

- Spray paint[1], pin flags, or rope to mark either grid corners or outline the sampling grid
- Incremental sampling tool (i.e., the MI tool developed by the Cold Regions Research and Engineering Laboratory or alternative[2] coring device); stainless steel spoons or scoops may be used but only in conjunction with scales, so that aliquots of equal mass are collected from each location
- Clean Zip-lock® bags, 5-gallon plastic containers, or other appropriate large container for placing the increments; the size of the container should be adequate to hold the sample volume, which is approximately 1-2 kilograms
- If MI sampling is used for volatile organic compound analysis, the increments of equal mass are collected with tools such as En Core® sampler and placed in a container obtained from the analytical laboratory that is partially filled with methanol
- Coolers and ice for cold storage of samples after collection
- Field logbook and pen with waterproof black ink for field documentation
- Global Positioning System instrument or other survey equipment to document locations of DU or SUs

[1] Avoid if spray paint is likely to affect MI sample quality.

[2] A source for the MI sampling tool shown in this SOP is Ike Loukos, LES Engineering, Inc. Telephone No. 301-471-3393, email i.loukos@att.net.

- Personal protective equipment should be worn during sample collection as required by the Health and Safety Plan for the project.

3. MULTI-INCREMENTAL SAMPLING PROCEDURE

Increments of soil will be collected within each cell of the SU. Increments should be approximately of the same weight. For surface soil sampling, a coring tool may be used to facilitate the rapid collection of uniform, representative increments from a consistent depth interval. This way, equal volumes are collected for each increment and equal mass is obtained under the assumption that the density of the sampled medium is uniform across the cell of the SU. The size of the coring tool will be selected based on the volume of the increments, which is in turn calculated based on number and depth of the increments and the fact that an adequate total sample mass is typically 1-2 kilograms dry weight (to overcome effects of compositional heterogeneity due to the inherent particulate nature of soil and sediment). It is not necessary to determine by the Global Positioning System location of every increment collected, as long as the SU has been properly identified and the relative position of the increment location within each cell is recorded.

The SU or DU will be demarcated in the field using pin flags, spray paint, or rope and fixed with a Global Positioning System. Increments will be selected as defined in the sampling plan.

Prior to MI sampling activities, the field team will don the personal protective equipment. The increments will be collected from the depth specified in the planning documents (usually up to 6 inches deep) using a coring tool or other method that ensures equal volume is collected for each increment. Unless specified in the sampling plan, the vegetative mat will be included in the sampled interval. Of note is that some plans may require only sampling native soil; the horizontal limits of sampling will be dependent on past disposal practices and the decision to be made. If used, the stainless-steel sampler will be pushed into the soil until the sampler is full and will not penetrate further. The sampler is then removed carefully, and the soil is pushed out of the sampler with the lever on the side of the instrument (see photos below).

Place the sample (increment or aliquot) directly into a large re-sealable bag, 5-gallon bucket, or alternative large container (note the above photos show placing the aliquot into a sampler's hand only for aliquot visualization purposes). Field experience has found that placing samples into a decontaminated 5-gallon bucket and then pouring the whole sample into a bag is a better process. The likelihood of spilling increases with the use of a bag alone because as the bag fills up it is harder to eject additional soil increments into the bag. The bucket is more stable and may prevent loss of fines. The holes left by sampling will be filled using surrounding soil or, if necessary, sand may be used to bring the subsurface sampling areas back to original grade. Soil samples should not include large rocks or pebbles unless they are part of the overall soil matrix. It is not necessary to decontaminate the sampling tool between the increments within a DU or SU.

If collecting an MI sample for volatile organic compound analysis, a wide-mouth glass container and methanol will be obtained from the analytical laboratory for sample aliquot preservation. The collection of the increments will be performed using EnCore™ or TerraCore™ sampling tools, meaning that a much smaller increment volume will be collected, resulting in a smaller total sample volume. The field team will place the 5- to 15-milligram increments into the glass container and care should be taken to follow the health and safety precautions associated with methanol handling. To prevent loss of methanol through volatilization, the sample container will be kept closed as much as feasible and only opened to place sample aliquots within the container.

Prior to the collection of replicate samples or MI samples from another SU or DU, the sampling tool will be decontaminated according to requirements set forth in EA SOP No. 005 – Field Decontamination. The replicate samples from the same SU/DU will be collected following a different path, as shown in Figure SOP No. 057-1. The specific relative location of the replicate increments within each SU cell will be established in a random manner to eliminate potential bias. To select the relative increment location for a replicate increment in a cell, the cell may be divided in turn into sub-grids and a sub-cell may be selected by randomly generating a number on a calculator. Another selection method is performed by rolling a dice for a 6 × 6 sub-grid in the SU cell; the first die would indicate the row and the second die the column of this sub-grid.

The large re-sealable bag containing the total sample volume will be labeled with indelible ink and then double-bagged. The samples will be bubble-wrapped and taped for shipping and placed into iced coolers for transport under chain-of-custody protocol to the analytical laboratory. The field procedures will follow the requirements set forth in EA SOP No. 002 – Chain-of-Custody Form and EA SOP No. 004 – Sample Packing and Shipping. Copies of the chain-of-custody forms and shipping documents will be retained in the project file. Field activities will be documented according to logbook procedures specified in EA SOP No. 016 – Surface Water, Groundwater, and Soil/Sediment Field Logbooks.

4. MAINTENANCE

Not applicable.

5. PRECAUTIONS

Safety precautions documented in the Site Health and Safety Plan will be followed. If sampling procedures are to occur in areas where unexploded ordnance is known or potentially exist, the area will not be entered until unexploded ordnance support is provided. If, at any time, an unsafe condition is identified, stop work immediately until the unsafe condition is mitigated. If sampling for volatile organic compound analysis, follow precautions associated with handling methanol. Also, because much larger quantities of methanol are employed for MI sampling, follow all requirements associated with transportation of these samples. In most cases, these samples are driven to the analytical laboratory rather than shipped via air, which constitutes a limitation in using this method at sites not located in close proximity of a laboratory.

6. REFERENCES

State of Alaska Department of Environmental Conservation. 2009. *Draft Guidance on Multi Increment Soil Sampling.* Division of Spill Prevention and Response Contaminated Sites Program. March.

State of Hawai'i Department of Health. 2009. *Technical Guidance Manual for the Implementation of the Hawai'i State Contingency Plan Interim Final.* Office of Hazard Evaluation and Emergency Response. June.

U.S. Army Corps of Engineers. 2009. Interim Guidance 09-02, *Implementation of Incremental Sampling of Soil for the Military Munitions Response Program.* 20 July.

U.S. Environmental Protection Agency. 2006. *SW-846 Method 8330B, Appendix A Collecting and Processing of Representative Samples for Energetic Residues in Solid Matrices from Military Training Ranges*.

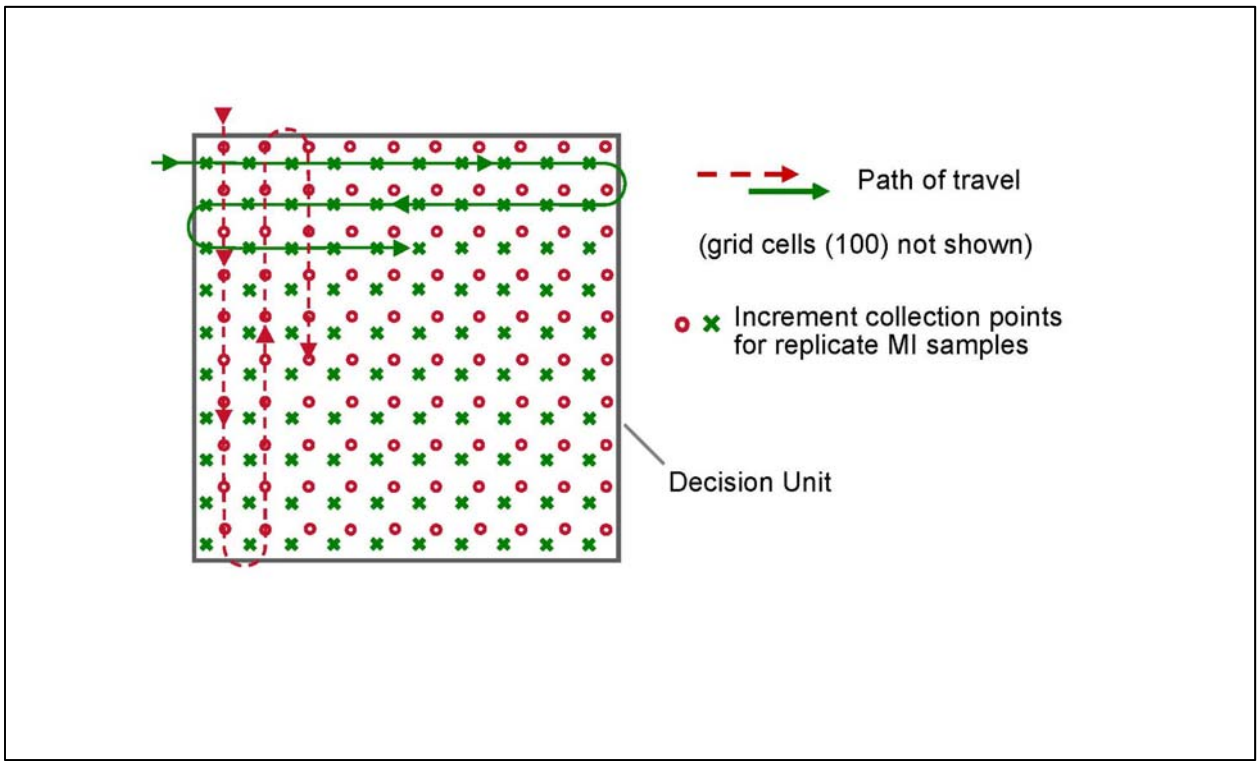

Figure SOP057-1. Example incremental sampling in a Decision Unit.

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Appendix C

Site Health and Safety Plan

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SITE HEALTH AND SAFETY PLAN

FOR

PHASE II ENVIRONMENTAL SITE ASSESSMENT

Old Goodwill

Tamuning, Guam

October 2018

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Attachment B	Safety Audit Checklist
Attachment C	Tailgate Health and Safety Form
Attachment D	Activity Hazard Analyses
Attachment E	Health and Safety SOPs
Attachment F	EA Occupational Health Program
Attachment G	Accident Loss and "Near Miss" Reports

FIGURE

Figure 1	Hospital Map

ACRONYMS AND ABBREVIATIONS

AHA	Activity Hazard Analysis
ANSI	American National Standards Institute
CFR	Code of Federal Regulations
CIH	Certified Industrial Hygienist
CSP	Certified Safety Professional
DISID	Department of Integrated Services for Individuals with Disabilities
EA	EA Engineering, Science, and Technology, Inc., PBC
EOD	explosive ordinance disposal
ESA	Environmental Site Assessment
°F	degrees Fahrenheit
HAZWOPER	Hazardous Waste Operations and Emergency Response
HR	heart rate
MEC	munitions and explosives of concern
OSHA	Occupational Safety and Health Administration
PPE	personal protective equipment
SHSO	Site Health and Safety Officer
SHSP	Site Health and Safety Plan
SOP	Standard Operating Procedure
UXO	unexploded ordinance

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1. INTRODUCTION

1.1 Purpose

The Site Health and Safety Plan (SHSP) specifies mandatory operating procedures, identifies physical, chemical, and biological hazards, establishes personal protection standards, and provides for response to emergency situations that may arise during the environmental investigation at the at the Old Goodwill site, which is now the proposed Rehabilitation Center for Guam Department of Integrated Services for Individuals with Disabilities (DISID) (herein referred to as the "subject site"), Tamuning, Guam. This plan currently addresses activities that will be conducted during the environmental investigations.

This SHSP has been prepared in accordance with the Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) Standard 29 Code of Federal Regulations (CFR) 1910.120, OSHA Hazard Communication Standard 29 CFR 1910.1200 (CFR, 2010), and all applicable health and safety procedures of EA Engineering, Science, and Technology, Inc., PBC (EA) (EA, 2017). Additional safety and health requirements are found in the Activity Hazard Analyses (AHAs), supplemental plans and EA's Health and Safety Standard Operating Procedures (SOPs) as identified below.

The procedures and standards in this SHSP are prepared for employees of EA. These policies are based on the available current information on potential hazards and contaminants, as identified in previous investigations. Personnel covered by this SHSP must consider newly acquired data or conditions when conducting their work, and must use appropriate, generally accepted practices to safeguard the health of onsite personnel.

1.2 Health and Safety Policy

The following basic policies apply to field operations involving hazardous wastes:

1. Personnel assigned to field operations involving hazardous wastes will participate in a medical surveillance program for hazardous waste operations.
2. Only personnel who have been certified and trained through the Federal OSHA HAZWOPER (29 CFR 1910.120) will be assigned to both supervise and do work involving hazardous waste or hazardous substances.

Compliance with this SHSP will be documented by the Site Health and Safety Officer (SHSO) in the master copy of the SHSP. All personnel will be required to signify that they have read and understand the contents of the SHSP (Attachment A).

Health and safety issues associated with specific activities, methods, and equipment must be provided by the SHSO to the rest of the team members. The forum for disseminating this

information includes pre-project health and safety briefings, daily tailgate meetings, and site inspections. An example of the site inspection form can be found in Attachment B.

All personnel shall follow the EA's Corporate Safety and Health Program Manual (EA, 2017).

1.3 Site History and Description

The subject site is located off Route 1, Marine Corps Drive, in Tamuning across from John F. Kennedy High School and is south of Kmart located within Tamuning, Guam. The subject site is 5.7 acres in size containing three different Lots, as shown in Figure 2. The subject site (Lot No. 5138-2-R3 NEW) was parceled out creating two more lots: Lot Nos. 5138-2-R3 NEW-1 & 5138-2-R3 NEW-2. During this time, a semi-concrete building was built creating the vocational rehabilitation center on Lot No. 5138-2-R3 NEW-1-R2. Two other buildings were then built on Lot No. 5138-2-R3 NEW-1-2 including a car wash shed on Lot No. 5138-2-R3 NEW-1-1. The eastern boundary of the subject site are within Lot 5138-2-R3 NEW-1 R/W. Lot 5138-2-R3 NEW-1-1, Lot 5138-2-R3 NEW-1-2 and Lot 5138-2-R3 NEW-1 R/W is a subdivision of Basic Lot 5138-2-R3 NEW-1.

The subject site was transferred to the Government of Guam and was established as a rehabilitation center in March 1988. In May 1993, a 20-year lease agreement was made between the Guam Rehabilitation and Workshop Center Inc. also known as Department of Vocational Rehabilitation and the Government of Guam. The facility was utilized as a Sheltered Workshop Training Center which included bookbinding, small engine repair, packaging of earphone sets for the former Continental Airlines, and a ceramic pottery shop. In August 2006, the subject site was administratively transferred over to DISID and is now under the jurisdiction of the Government of Guam.

In addition, as the subject property was historically utilized by the Guam Rehabilitation and Workshop Center in 1966 with the purpose of providing work activities for people with disabilities. Work activities included food services training, school furniture repair, book binding for the library, manufacturing ceramic pottery, cabinet making, picture framing, construction of shipping crates for the military, engine repair services, construction of shipping crates for the military, and manufacturing of local arts and crafts.

The subject site is currently unoccupied. There are fences surrounding the subject site, illegal dumping has occurred along the road side adjacent to the parcel.

1.4 Project Objective

Based on the recommendation from the Phase I Environmental Site Assessment (ESA) (EA, 2018), a Phase II ESA will be conducted. The results of the proposed investigation will be used to characterize the nature and extent of contamination at the subject site and make recommendations based on the findings.

1.5 Project Organization

The Chain of Command that will be observed during these investigations is discussed below.

Program Health and Safety Director

The EA Program Health and Safety Director is a Certified Industrial Hygienist (CIH) and a Certified Safety Professional (CSP) and will be responsible for establishing, implementing, monitoring, and administering and providing oversight to the health and safety program. The Program Health and Safety Director is responsible for ensuring that the company Health and Safety program is in compliance with Federal, State, and contract specific health and safety requirements and that this SHSP addresses the health and safety requirements for the environmental assessment of the subject site. He will approve all amendments to this plan. Mr. Peter Garger, CIH, CSP is the EA Program Health and Safety Director.

Project Manager

The Program Manager is responsible for directing all project related activities in a safe manner, coordinating the project team, auditing compliance with this SHSP, and for reviewing all technical reports and SHSP amendments prepared by the project team. Mr. Bob Shambach is the Project Manager.

Site Health and Safety Officer

The SHSO will provide daily tailgate health and safety briefings for all personnel prior to any onsite activities. A copy of the tailgate health and safety meeting form is located in Attachment C. The briefings will include such topics as onsite hazards and site-specific emergency procedures. The SHSO will maintain health and safety training records for all onsite personnel and will record in a daily logbook the site conditions, any site monitoring activities, personal protective equipment (PPE) used and any upgrading or downgrading of PPE levels, and other site specific or personnel related health and safety information.

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2. HAZARD ASSESSMENT

The following section outlines the AHAs, referenced SOPs, and Chemical Hazards associated with this project:

2.1 Field Activities

The following lists the site activities for the Phase II ESA at the subject site:

- Mobilization of supplies and heavy equipment
- Site Preparation
- Collection of surface soil samples
- Collection of petroleum product sample, if applicable.

A list of AHAs related to the field activities at the subject site are presented in Attachment D. The AHAs will be reviewed periodically and revised as applicable.

During daily safety tailgate meetings, applicable AHAs will be reviewed with the work crew prior to commencing work. All site visitors will be required to review the daily tailgate safety issues and sign the visitor log. Applicable SOPs are presented in Attachment E of this plan.

The AHAs should be revised for site-specific activities and reviewed with work crew before commencing any work.

Based on the Phase I ESA (EA, 2018), the following contaminants could be present on the site:

- Polychlorinated biphenyls
- Polycyclic aromatic hydrocarbons
- Metals.

2.2 Exploratory Excavation

Using an experienced excavation contractor, heavy equipment will be used to perform exploratory (excavation) activities to determine if the vent pipe previously identified on-site leads to an underground storage tank. These operations will be conducted in accordance with OSHA regulations.

The site is not considered to have Unexploded Ordinance (UXO) in the areas of investigation. Because Guam was a battleground during World War II it is possible that Munitions and Explosives of Concern **(**MEC) including UXO and discarded military munitions may be encountered. If materials believed to be MEC are discovered, work will stop, the area will be evacuated immediately, and field personnel will call 911. Emergency first responders will contact Navy Explosive Ordnance Disposal (EOD) personnel for removal/disposal of suspected MEC or identify unknown objects. Work will commence once EOD has cleared and declared the area safe.

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3. HEALTH AND HAZARD MONITORING

3.1 Special Medical Monitoring

The medical surveillance program documents that personnel are capable of performing their assigned activities and that the health of employees is not compromised by potential exposure to chemical or physical agents found at work sites. This program is designed to support and monitor the effectiveness of the primary health and safety goal of controlling worker exposure to hazardous materials. Medical surveillance is performed by or under the direct supervision of a licensed physician board certified in occupational medicine. All field personnel (and management personnel onsite) are required to have a current medical certification in accordance with OSHA standards (i.e., 1910.120, 1926.65) prior to entering regulated areas (Attachment F). Medical monitoring approvals shall be documented and maintained in each employee's personnel file.

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4. HEALTH AND SAFETY

4.1 Personal Protection Equipment Requirements

Unless otherwise approved by SHSO, all activities will include PPE. The AHA tables summarize the PPE anticipated during performance of field work based on previous investigations at the site.

4.1.1 Foot Protection

Safety-toe boots will be worn by onsite employees during investigation activities. All safety-toe boots will meet the requirements specified in American National Standards Institute (ANSI) Standard Z41.1-1967 as required by 29 CFR 1926.96 and 29 CFR 1910.136.

4.1.2 Hand Protection

Appropriate hand protection, as determined in the hazard assessment, will be used by EA employees when employees' hands are exposed to hazards such as those from skin absorption and harmful substances; severe cuts or lacerations, severe abrasions, punctures, chemical burns, thermal burns, and harmful temperature extremes.

4.1.3 Head Protection

Head protection (hard hats) will be worn by field personnel whenever possible danger of head injury exists from impact, or falling or flying objects. The hard hat will meet requirements in ANSI Standard Z89.1-1969 or Z89.2-1972 as required by 29 CFR 1926.100 and 29 CFR 1910.135. Hard hats may be labeled with EA logo stickers or adhesive name stickers but otherwise will not be painted, altered, or worn backwards unless the manufacturer had approved such use.

4.2 Chemical Hazards

OSHA requires that lists of hazardous chemicals known to be present be compiled for the workplace as a whole or for individual work areas.

The Project Manager will develop and maintain the hazardous chemical list for each project. The list may be included as part of the SHSP for HAZWOPER projects which will be available and accessible at a location made known to all affected parties during the conduct of field operations. Alternatively, or for non-hazardous waste operations and emergency response field operations, the list may be maintained with the project files but will be available and accessible at a location made known to all affected parties during the conduct of field operations.

The lists of hazardous chemicals will be kept current and updated, as necessary. The lists of hazardous chemicals will contain the location, the name of each chemical as referenced on the Safety Data Sheets, the type of compound (i.e., flammable, corrosive, poison, etc.), the date of the inventory, and the name of the person generating the list.

4.3 Slip/Trip/Fall Hazards

Work sites may be in remote locations, and some areas may have slippery locations, and other slip/trip/fall hazards. Team members are required to wear ANSI Z41 approved steel toed or approved composite footwear and are to take special care in obtaining sure footing and walking slowly, when necessary.

4.4 Heat Stress

As Guam has a warm and humid climate, heat stress is a concern during site activities. The stress of working in a hot environment can cause a variety of illnesses including heat exhaustion or heat stroke; the latter can be fatal. Wearing impermeable clothing can significantly increase heat stress. To reduce or prevent heat stress, frequent rest periods and controlled beverage consumption to replace body fluids and salts may be required. If necessary, a work rest regimen will be established based on the physiological monitoring guidance described below. Quantitative physiological monitoring for heat stress may be conducted when ambient temperatures exceed 78 degrees Fahrenheit (°F). Physiological monitoring for heat stress includes using the worker's heart rate (primary indicator) and oral temperature (secondary indicator) measured during work assignments. The frequency of monitoring depends on the ambient temperature, the PPE level, and the degree of acclimation the person has to the heat. To determine the initial monitoring frequency, after a work period of moderate exertion, use the following information:

Adjusted Temperature*	Level D	Level C
90°F or above	after 45 minutes	after 15 minutes
87.5 to 90°F	after 60 minutes	after 30 minutes
82.5 to 87.5°F	after 90 minutes	after 60 minutes
77.5 to 82.5°F	after 120 minutes	after 90 minutes
72.5 to 77.5°F	after 150 minutes	after 120 minutes

* Adjusted air temperature (°F) = observed temp + (13 x percent sunshine). Air temperature measured with bulb shielded from radiant heat. Percent sunshine is the time sun is not covered by clouds thick enough to produce a shadow (100 percent = no cloud cover and a sharp, distinct shadow; 0 percent = no shadows). From The Industrial Environment, its Evaluation and Control; U.S. Department of Health and Human Services, 1973.

The following procedures and action levels are to be used for the physiological monitoring of heat stress:

Heart Rate (HR): HR will be measured by the radial pulse for 30 seconds as early as possible in the resting period. The HR at the beginning of the rest period should not exceed 110 beats per minute. If the HR is higher, the next work period should be shortened by one third, while the length of the rest period stays the same. If the pulse rate exceeds 110 beats per minute at the beginning of the next rest period, the next work cycle should be shortened by another 33 percent.

Oral Temperature: Use a clinical thermometer to measure the oral temperature at the end of the work period (before drinking). If oral temperature exceeds 99.6°F, shorten the next work cycle by

one third without changing the rest period. If oral temperature exceeds 99.6°F at the beginning of the next rest period, shorten the following work cycle by on third. DO NOT allow a field team member to wear OSHA Level C protection when oral temperature exceeds 100.6°F.

Personal Monitoring Devices: The use of personal monitoring devices is encouraged during the initial work periods, if applicable. This will help each worker monitor his/her physiological response to heat, humidity, and the work activity. As the worker acclimates, the work rest regimen can be adjusted accordingly.

Even though physiological monitoring is not always necessary, it is essential that personnel understand the significance of heat stress and its recognition. Personnel will be trained to recognize the symptoms of heat stress and the appropriate action to take upon recognition.

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5. CONTAMINATION CONTROL, SANITATION, AND WASTE MANAGEMENT

The purpose of site control is to minimize chemical exposures, hazards, and other injuries to workers and the public during site activities.

5.1 Site Entry Requirements

In order to allow an individual into regulated areas of the site, he/she must meet the following requirements:

- Review of this SHSP and sign-off on the SHSP Acknowledgement form (Attachment A)
- Document completion of HAZWOPER
- Obtain authorization from the SHSO
- Don the appropriate PPE.

5.2 Waste Materials Management

The investigation-derived wastes produced at the site will consist of waste PPE. PPE will be segregated into separate waste streams. Visibly clean PPE will be combined with miscellaneous trash and disposed at the local municipal landfill. General trash will be hauled to the local municipal landfill for disposal.

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6. EMERGENCY RESPONSE PLAN

This SHSP has been established to allow site operations to be conducted in a manner that minimizes hazardous health impacts on both employees and the community. In addition, this Emergency Response Plan has been developed to cover extraordinary conditions that might occur at the site. Prior to the commencement of work, personnel will be familiar with emergency condition identification, notification, and response procedures in accordance with the EA's Corporate Health and Safety Plan (EA, 2017). Emergency response training shall be a part of the initial SHSP pre-work training required of all employees. Additional discussions or refreshers shall take place periodically throughout the project as part of the daily tailgate safety meetings. See the Tailgate Safety Meeting Form for contact numbers and emergency call information (Attachment C). In case of an emergency, the crew must be transported to the designated medical center.

6.1 Emergency Conditions

Fire, medical emergency, spill, severe weather, criminal/terrorist activity, incidents, accidents, injuries, and mishaps are the conditions that may lead to an emergency situation.

6.2 Evacuation and Rally Points

The SHSO will review procedures (including life-threatening or immobilizing injuries) during the first tailgate safety meeting. Topics to be covered include conditions leading to an emergency, location of rally point, contact numbers and head counts, if applicable.

The SHSO shall designate an assembly point in case of an emergency. Whenever an employee becomes aware of an emergency condition, the employee shall immediately proceed to the assembly point and notify the Emergency Coordinator. The SHSO will act as the Emergency Coordinator, but if the SHSO is unavailable or cannot be contacted, the Field Team Leader will act as the Emergency Coordinator.

6.3 Directions to Hospital

The emergency telephone numbers will be posted at the site. The Guam Memorial Hospital will be used for medical assistance (Figure 1).

The address is:

850 Gov. Carlos G. Camacho Road
Oka, Tamuning, Guam 96913-3128

The Guam Memorial Hospital emergency contact number is **647-2281 or 647-2489.**

The route to the Guam Memorial Hospital is as follows:

1. Turn right (heading north) onto Marine Corps Drive.
2. Turn left onto Route 14A.
3. Then turn left onto Route 14.
4. Hang a right after the Archbishop Felixberto Flores Circle unto Father Duenas Drive which leads directly to the hospital.

6.4 Reporting

6.4.1 Employee Exposure/Injury Incident Reporting

All incidents resulting in an exposure or injury to personnel on site (employee or otherwise) are to be recorded on the Accident/Loss Form found in Attachment G. This form is to be completed by the SHSO, and submitted to the Corporate Health and Safety Director, and the Project Manager within 24 hours of the incident. In the event of an accident EA will maintain the emergency contact information for all employees. This information is available by contacting the EA office (671) 646-5231.

6.4.2 "Near Miss" Incidents

A "near miss" is a situation that did not quite result in an accident or injury. Just as much can be learned about weaknesses in the job situation from events that almost happened as from things that actually occurred; therefore, a "near miss" should be investigated with the same diligence as an accident. "Near miss" reporting should be completed within a few hours of observation to ensure accurate documentation using the "Near Miss" Incident Report form found in Attachment G.

7. REFERENCES

American National Standards Institute. American National Standards Institute Standard Z358.1. Current edition

Code of Federal Regulation (CFR). Title 29, Part 1910. *Occupational Safety and Health Standards*. Current edition

EA Engineering, Science, and Technology, Inc, PBC (EA). 2017. *Corporate Safety and Health Program Manual,* February.

EA. 2018. Phase I Environmental Site Assessment Report, Old Goodwill, Tamuning, Guam. September.

U.S. Department of Health and Human Services, The *Industrial Environment, its Evaluation and Control.* Current edition

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Figure

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COORDINATE/REFERENCE: WGS_1984_UTM_Zone_55N

0.25 0.125 0 0.25 0.5 Miles

Legend

Hospital Route

EA Engineering, Science, and Technology, Inc., PBC
1001 Army Drive, Suite 103,
Barrigada, 96913-1402
Telephone: (671) 646-5231
Facsimile: (671) 646-5230

Project No/TO.:RFP No. 2015-001
Guam EPA Brownfields 2017
Phase II ESAs

Figure 1
Hospital Map

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Attachment A
Health and Safety Signature Form

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SITE HEALTH AND SAFETY PLAN SIGNATURE FORM

SITE NAME/NUMBER: _____

LOCATION: _____

I have read, understood, and agreed to comply with the provisions of the above-referenced Site Health and Safety Plan (HASP) for work activities on this site.

PRINTED NAME	SIGNATURE	COMPANY	DATE

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Attachment B
Safety Audit Checklist

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SAFETY AUDIT CHECKLIST

Site: _____ Prepared by: _____
Location: _____ Audit Date: _____
Project No.: _____ Project Manager: _____
Client: _____ Site Safety and Health Officer : _____

Rating	S	U	N/A	Comments
Site Health and Safety Plan (SHSP) General Requirements				
Was a pre-entry safety briefing conducted? If so, did it include the following: • Disclosure of potential hazards? • Procedures for clearances/entry to restricted areas? • Emergency response? • Vehicle rules/regulations? • Equipment to be used? • Material handling restrictions? • Transporting/storing hazardous materials? • Personal protective equipment (PPE)? • Applicable standard operating procedures? • Methods of decontamination? • Responsibilities for safety of personnel/property? • Location/use of Material Safety Data Sheets (MSDS)? • Safe work practices?				
Approved SHSP on site?				
SHSP compliance agreement form signed by onsite personnel, including subcontractors?				
New activities or hazards identified and incorporated into revised SHSP?				
Names of onsite personnel recorded in field logbook or daily log?				
Applicable MSDSs on site or available?				
Hazard labeling practices currently being used?				
Designated Site Health and Safety Officer (SHSO) present? • Designated alternate SHSO? • SHSO conducts daily inspections of site/work zones? • Records of daily inspections available for review?				
Daily tailgate safety meetings conducted and documented?				
Onsite personnel meet SHSP requirements for medical examinations, fit testing, and training (including subcontractors)?				
Documentation of training, medical examinations, and fit tests available from employer (as applicable)?				
Compliance with specified safe work practices?				
Exclusion (EZ), Contamination Reduction (CRZ), and Support Zones (SZ) delineated and enforced?				
Windsock, flag, or ribbons in place to indicate wind direction?				
SZ located upwind from EZ and CRZ, as practicable?				
Emergency Planning				
Emergency telephone numbers posted?				
Emergency telephone numbers up to date?				
Emergency route to hospital posted?				
Local emergency providers notified of site activities?				

Rating	S	U	N/A	Comments
Review weather emergency procedures?				
Adequate safety equipment inventory available?				
First aid provider and first aid supplies available?				
Eyewash station(s) functioning and in place?				
Communication equipment readily available for emergencies?				
Any reported accidents/incidents at this site? If so, are the accident/incident reports available for review?				
Air Monitoring				
Monitoring equipment specified in SHSP available and in working order (See Instrumentation list below)?				
Monitoring equipment calibrated and calibration records available?				
Personnel know how to operate monitoring equipment and equipment manuals available on site?				
Environmental and personnel monitoring performed as specified in SHSP?				
Heat stress monitoring being conducted and "cool-down" breaks implemented?				
Air monitoring instrumentation includes: • Combustible gas meter? • Oxygen meter? • Organic vapor analyzer? • Hydrogen sulfide monitor				
PPE				
Proper dermal protection worn when handling/ contacting hazardous chemicals or contaminated environmental media?				
Required PPE (hard hats, safety boots / shoes, eye protection with side shields) being worn?				
Reflective vests available? Worn when required?				
Hearing protection available? Worn when required?				
Heavy Equipment Operations				
Equipment operators experienced/properly trained?				
Dust control measures implemented in EZ, as necessary?				
Equipment regularly inspected and maintained?				
Utility lines located and marked prior to construction activities?				
Clearance/digging permits kept onsite and available for review?				
Drill rigs/elevated equipment maintaining minimum 10-ft distance from energized (50 kV) overhead power lines?				
Traffic control barricades in place (28-in traffic cones/flags/barricade tape)?				
Proper PPE, including hearing protection and reflective vests in use?				
When backing a vehicle up is a spotter used?				
Supplies				
Decontamination equipment and supplies on site?				
Fire extinguishers (functioning, inspected, and in field vehicles)?				
Spill cleanup supplies on site?				

Rating	S	U	N/A	Comments
Power Tools/Electrical Hazards				
Proper PPE used?				
Lock-out/Tag-out system in place and observed?				
Power tools and power cords inspected and maintained?				
Investigation-derived Waste (IDW)				
Wastes properly disposed of?				
Designated location for drummed IDW?				
IDW containers properly labeled?				

Additional Comments:

_____ _____

Auditor's Signature Site Safety and Health Officer's Signature

_____

Date

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Attachment C
Tailgate Health and Safety Form

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TAILGATE SAFETY MEETING FORM

EA Engineering Project Number: 15672.01

Client: Guam Environmental Protection Agency

Work Title: Phase II Environmental Site Assessment

Work Site: Old Goodwill, Route 1, Marine Corps Drive Tamuning, Guam

Site ID: _____

Scope of Work: Surface soil sampling and petroleum product sampling

Personal Protective Equipment: Level D, steel toe boots, hard hat, safety vest, safety glasses, and Nitrile gloves. **Note: No sunscreen or bug replant will be used, they may interfere with sampling efforts.**

Potential Chemical Hazard: PCBs (liquid product), Metals, and PAHs

Potential Physical Hazards: Slip, trip, falls, heat, biological. Be sure of stable footing while traversing the site. Monitor for heat stress.

Off-Site Emergency Contacts: Fire, Police, and Ambulance **911**. Guam Memorial Hospital at **646-4282**

EA On-Site Health and Safety Officer: Tim Chargualaf at **646-5231;** Alternate SHSO: Jaquay Soriano at **646-5231**

EA Corporate Project Safety Officer: Peter Garger (EA) at **401-790-6338**

Project Manager Bob Shambach (EA) at **646-5231,** cell **727-6094**

Daily Record

Date:
Time:
Tailgate Meeting Conducted By:

List of Attendees	List of Signatures
1.	
2.	
3.	
4.	
5.	
6.	
7.	
8.	
9.	
10.	

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Attachment D
Activity Hazard Analyses

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ACTIVITY HAZARD ANALYSIS

Principal Step	Potential Hazards	Recommended Controls
Mobilization of equipment and supplies/ Site Setup	• Heavy lifting	• Use proper lifting techniques, size up the load, use teamwork, never twist or turn when lifting. • Use mechanical lifting devices whenever feasible. • Steel-toed boots.
	• Slips, trips, and falls	• Tripping hazard will be identified and feasible engineering controls implemented. • Good housekeeping practices will be observed. • Maintain proper illumination in work areas.
	• Faulty/damaged equipment	• Continual inspection of work areas. • Equipment will be inspected upon arrival and at the beginning of each shift. • Routine thorough inspection of equipment by a competent mechanic or personnel trained and familiar with the equipment.
	• Vehicle Safety	• Follow manufacturer's recommended payload • Use seat belts/rollover protection system • For all-terrain vehicles, gloves and hardhats are required • Utilize only licensed and trained operators • Ensure equipment is not operated on excessive grades to prevent rollovers • Follow all traffic laws. Vehicles will not be driven at speeds greater than the posted speed limit, with due regard for weather, traffic, intersections, width and character of the roadway, type of motor vehicle, and any other existing condition. • Defensive driving principles will be practiced.
	• Noise	• Wear hearing protective devices (Ear muffs/plugs) when working, when using or near high noise producing equipment, or when directed by SHSO in response to noise monitoring. • Ensure adequate maintenance on equipment. • Conduct periodic sound level surveys.
	• Heat	• Wear appropriate clothing. • Have cool drinks (water and/or electrolytes) available and take small drinks frequently. • Take regular breaks in shaded, cool areas and monitor temperature as specified in Heat Stress Monitoring Program. • Use Buddy System and report signs of heat stress to the SHSO immediately
	• Severe weather	• Shut down operations during severe electrical storms, heavy rain, high wind, and evacuate site/take cover. • Where there are warnings or indications of impending severe weather, conditions will be monitored and appropriate action taken.

EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS

ACTIVITY HAZARD ANALYSIS

PPE • Long pants • Long sleeves (optional) • Hardhat • Safety boots (steel or composite toe) • Safety glasses (potential eye injury hazard areas) • Reflective vest • Hearing protection, as needed	• Vehicles • Equipment daily • Routine mechanical • Slings, chains, ropes.	• Vehicle operators will possess a valid permit for equipment being operated. • Lifting and general back awareness training.

ACTIVITY HAZARD ANALYSIS

Principal Step	Potential Hazards	Recommended Controls
Excavation Activities & Entering Excavation for Sampling	• Exposure to contaminants	• Wear protective equipment as required. Skin contact with potentially contaminated media by using nitrile gloves during sampling. • Use other appropriate PPE, as conditions change or action levels are met.
	• Exposure to Fugitive Dust	• Operator shall position bucket as close to the hopper as possible and limit free air space between bucket and hopper while dumping. • Dump bucket slowly. If dust is visible during operations, reduce speed, or utilize water spray to control fugitive dust emissions. • If possible, equipment operator and all support ground personnel shall be located upwind.
	• Lifting	• Get assistance with moving object that may be awkward or heavier than 50 pounds (loaded sample coolers)
	• Noise	• Wear hearing protective devices (Ear muffs/plugs) when working, when using or near high noise producing equipment, or when directed by SHSO in response to noise monitoring. • Ensure adequate maintenance on equipment. • Conduct periodic sound level surveys.
	• Heat	• Wear appropriate clothing. • Have cool drinks (water and/or electrolytes) available and take small drinks frequently. • Take regular breaks in shaded, cool areas and monitor temperature as specified in Heat Stress Monitoring Program. • Use Buddy System and report signs of heat stress to the SHSO immediately
	• Chemical exposure (sample preservatives)	• Be familiar with safety data sheets (SDS) for chemical products used to preserve samples. • Transport in appropriate packaging. (See Department of Transportation guidelines for appropriate equipment to transfer required aliquot to sample containers). Eyewash in accordance with 29 CFR 1910.151. • Wear proper PPE including hand and eye protection. • Review SDSs before use. • Properly label all chemicals. • Ensure proper storage. • Follow proper hand washing procedures. • Have eyewash readily available.

ACTIVITY HAZARD ANALYSIS

Principal Step	Potential Hazards	Recommended Controls
Excavation Activities & Entering Excavation for Sampling (continued)	• Manual Lifting	• Train/Utilize correct lift techniques. • Personnel will not lift more than 50 lb. • Use Buddy System. • Use mechanical lifting procedures whenever possible.
	• Load hazards	• Load trucks on even ground surface. • Distribute load on trucks evenly. • NEVER STAND UNDER A SUSPENDED LOAD.
	• Slips, Trips, and Falls	• Be aware of physical hazards - watch for uneven ground, rocks, dirt clods, etc. • Practice good housekeeping. • Use care when walking on the ground surface, especially when the ground surface is wet in the morning and/or during and after rain. • Clean all spills immediately • Proper PPE to include steel-toe boots which provide good footing. • Survey area and remove any trip hazards.
	• Biological	• Exercise caution in unknown areas. • Wear clothing in such a way as to restrict access by insects. • After working in infested areas, shower, inspect and properly remove insects.
	• Equipment Failure/Hazards	• Never leave equipment unattended while running. • When leave equipment, always leave safety lever in non-operating position • Inspect equipment prior to daily operation • Ensure all roll cages and guards are in place and back up alarms operate • Original equipment manufacturer's (OEM) equipment modifications only • Use machine guarding and enclosures • Utilize only trained and experienced operators for operation of equipment • Site specific training - Toolbox safety meetings, fall protection systems (if applicable)
	• Contact with Overhead and Underground Utilities	• Verify utility information prior to excavation. Intrusive soil activities conducted within a five foot “Buffer Zone” (horizontal or vertical, as measured from the outside edge of the utility) of any utility (electric, gas, high pressure chemical storage tanks, pipelines, sewers, etc.) may require the use of non-aggressive excavation methods such as hand excavation using non-conductive hand tools, use of an air spade, hydroexcavation, or similar means. • Spotter will assist the operator/workers to identify unknown conditions during excavation. • If a previously unknown utility line is identified, uncovered, or disturbed during excavation/trenching activities, the excavation activity shall stop immediately and project management notified. Excavation shall not recommence until the line has been evaluated, identified, traced, and proper precautions have been implemented. • Excavation spoil piles should not be placed atop surface features or ground markings identifying the locations of underground utilities.

ACTIVITY HAZARD ANALYSIS

Excavation Activities & Entering Excavation for Sampling (continued)	• Struck By/Against Heavy Equipment	• Maintain radio/verbal communication. • Make eye contact with operators and indicate your movements with hand signals before approaching equipment. • Equipment will not be approached on blind sides. • Personnel will understand and review hand signals. • All machines will be equipped with backup alarms and lighting. • Obey all posted speed limits. • Properly maintain/inspect equipment.
	• Severe weather	• Shut down operations during severe electrical storms, heavy rain, high wind, and evacuate site/take cover. • Where there are warnings or indications of impending severe weather, conditions will be monitored and appropriate action taken.

EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
• Level C & D PPE (Levels will be determined by SHSO) : o Level C: Tyvek® coveralls, APR respirators, hard hat, safety glasses, steel-toed boots, traffic vests, earplugs, gloves o Level D: hard hat, safety glasses, steel-toed boots, traffic vests, earplugs, gloves • Heavy Equipment • AM/FM RADIO • Eye wash	• Respirators (before each use, pre-project inspection and fit testing by Industrial Hygienist)	• Proper use of PPE and its limitations. • Proper use of eyewash • All field personnel will have Site Safety and Health Plan/Activity Hazard Analysis training; Hazard Communication training and Daily Tailgate Safety Meetings. Applicable Operator Certifications, HAZWOPER Certifications, Safe Tool Use, Fire Safety training, Emergency Response Training, First Aid/Cardiopulmonary Resuscitation training. • Only qualified operators permitted to operate. Qualifications and competency reviewed by Site Supervisor. • Excavation competent person for excavations greater than 4-feet in depth.

ACTIVITY HAZARD ANALYSIS

Principal Step	Potential Hazards	Recommended Controls
Handling Contaminated Soil	• Exposure to contaminants	• Wear protective equipment as required. Skin contact with potentially contaminated media by using nitrile gloves during sampling. • Use other appropriate PPE, as conditions change or action levels are met.
	• Exposure to Fugitive Dust	• Operator shall position bucket as close to the hopper as possible and limit free air space between bucket and hopper while dumping. • Dump bucket slowly. If dust is visible during operations, reduce speed, or utilize water spray to control fugitive dust emissions. • If possible, equipment operator and all support ground personnel shall be located upwind.
	• Lifting	• Get assistance with moving object that may be awkward or heavier than 50 pounds (loaded sample coolers)
	• Noise	• Wear hearing protective devices (Ear muffs/plugs) when working, when using or near high noise producing equipment, or when directed by SHSO in response to noise monitoring. • Ensure adequate maintenance on equipment. • Conduct periodic sound level surveys.
	• Heat	• Wear appropriate clothing. • Have cool drinks (water and/or electrolytes) available and take small drinks frequently. • Take regular breaks in shaded, cool areas and monitor temperature as specified in Heat Stress Monitoring Program. • Use Buddy System and report signs of heat stress to the SHSO immediately
	• Chemical exposure (sample preservatives)	• Be familiar with material safety data sheets for chemical products used to preserve samples. • Transport in appropriate packaging. (See Department of Transportation guidelines for appropriate equipment to transfer required aliquot to sample containers). Eyewash in accordance with 29 CFR 1910.151. • Wear proper PPE including hand and eye/face protection. • Review SDSs before use. • Properly label all chemicals. • Ensure proper storage. • Follow proper hand washing procedures. • Have eyewash readily available.

ACTIVITY HAZARD ANALYSIS

Principal Step	Potential Hazards	Recommended Controls
Handling Contaminated Soil (continued)	• Manual Lifting	• Train/Utilize correct lift techniques. • Personnel will not lift more than 50 lb. • Use Buddy System. • Use mechanical lifting procedures whenever possible.
	• Load hazards	• Load trucks on even ground surface. • Distribute load on trucks evenly. • NEVER STAND UNDER A SUSPENDED LOAD.
	• Slips, Trips, and Falls	• Be aware of physical hazards - watch for uneven ground, rocks, dirt clods, etc. • Practice good housekeeping. • Use care when walking on the ground surface, especially when the ground surface is wet in the morning and/or during and after rain. • Clean all spills immediately • Proper PPE to include steel-toe boots which provide good footing. • Survey area and remove any trip hazards.
	• Biological	• Exercise caution in unknown areas. • Wear clothing in such a way as to restrict access by insects. • After working in infested areas, shower, inspect and properly remove insects.
	• Equipment Failure/Hazards	• Never leave equipment unattended while running. • When leave equipment, always leave safety lever in non-operating position • Inspect equipment prior to daily operation • Ensure all roll cages and guards are in place and back up alarms operate • Original equipment manufacturer's (OEM) equipment modifications only • Use machine guarding and enclosures • Utilize only trained and experienced operators for operation of equipment • Site specific training - Toolbox safety meetings, fall protection systems (if applicable)
	• Contact with Overhead and Underground Utilities	• Verify utility information prior to excavation. Intrusive soil activities conducted within a five foot "Buffer Zone" (horizontal or vertical, as measured from the outside edge of the utility) of any utility (electric, gas, high pressure chemical storage tanks, pipelines, sewers, etc.) may require the use of non-aggressive excavation methods such as hand excavation using non-conductive hand tools, use of an air spade, hydroexcavation, or similar means. • Spotter will assist the operator/workers to identify unknown conditions during excavation. • If a previously unknown utility line is identified, uncovered, or disturbed during excavation/trenching activities, the excavation activity shall stop immediately and project management notified. Excavation shall not recommence until the line has been evaluated, identified, traced, and proper precautions have been implemented. • Excavation spoil piles should not be placed atop surface features or ground markings identifying the locations of underground utilities.
Principal Step	Potential Hazards	Recommended Controls

ACTIVITY HAZARD ANALYSIS

Handling Contaminated Soil (continued)	• Struck By/Against Heavy Equipment	• Maintain radio/verbal communication. • Make eye contact with operators and indicate your movements with hand signals before approaching equipment. • Equipment will not be approached on blind sides. • Personnel will understand and review hand signals. • All machines will be equipped with backup alarms and lighting. • Obey all posted speed limits. • Properly maintain/inspect equipment.
	• Severe weather	• Shut down operations during severe electrical storms, heavy rain, high wind, and evacuate site/take cover. • Where there are warnings or indications of impending severe weather, conditions will be monitored and appropriate action taken.

EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
• Level C & D PPE (Levels will be determined by SHSO) : o Level C: Tyvek® coveralls, APR respirators, hard hat, safety glasses, steel-toed boots, traffic vests, earplugs, gloves o Level D: hard hat, safety glasses, steel-toed boots, traffic vests, earplugs, gloves • Heavy Equipment • AM/FM RADIO • Eye wash	• Respirators (before each use, pre-project inspection and fit testing by Industrial Hygienist)	• Proper use of PPE and its limitations. • Proper use of eyewash • All field personnel will have Site Safety and Health Plan/Activity Hazard Analysis training; Hazard Communication training and Daily Tailgate Safety Meetings. Applicable Operator Certifications, HAZWOPER Certifications, Safe Tool Use, Fire Safety training, Emergency Response Training, First Aid/Cardiopulmonary Resuscitation training. • Only qualified operators permitted to operate. Qualifications and competency reviewed by Site Supervisor. • Excavation competent person for excavations greater than 4-feet in depth.

Attachment E
Health and Safety SOPs

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MOTOR VEHICLE SAFETY POLICY

The risk of injury to personnel and property damage is probably greater during operation of vehicles than at any other time while under the employment of EA. The purpose of this section is to provide a means to implement an effective maintenance program and appropriate training to minimize this risk. By far, the most important factor in reducing the risk of injury, damage, and loss is through employee adherence to safe driving practices.

AUTHORIZATION AND TRAINING

No employee will be authorized to operate an EA vehicle unless he or she has a valid driver's license.

No employee will be authorized to operate a vehicle with a trailer or a vehicle requiring the use of mirrors due to restricted vision until he or she has been instructed in their safe operation and use.

No employee will be authorized to operate a fork lift until he or she has received training in its safe operation.

OPERATION

Operators of company-owned, leased, and privately owned vehicles while on company business will:

- Drive vehicles in a safe and courteous manner
- Obey traffic regulations; traffic citations will be the responsibility of the operator
- Use safety belts (including passengers)
- Abstain from drinking alcoholic beverages or using drugs prior to or while driving (including passengers)

MAINTENANCE

Operations Managers will establish a maintenance schedule for EA vehicle, which is consistent with the manufacturer's servicing recommendations.

Employees using EA vehicles will make their own inspection prior to each use, including:

- Coolant level
- Oil level
- Fuel level
- Windshield wipers and washer fluid
- Headlights
- Turn signals/flashers
- Tail lights/brake lights
- Tire inflation and condition (tread)

Malfunctions and deficiencies will be reported to the Office Administrator for corrective action.

VEHICLE SAFETY EQUIPMENT

The following is a suggested list of equipment to be maintained in EA vehicles:

- First aid kit
- Fire extinguisher
- Safety flares or reflective triangles
- Spare tire and jack.

HEAT STRESS SYMPTOMS AND APPROPRIATE ACTIONS

	Description	How to Recognize	What To Do
Heat Rash			
	A skin irritation caused by excessive sweating during hot, humid weather. It can occur at any age but is most common in young children.	Looks like a red cluster of pimples or small blisters. It is more likely to occur on the neck and upper chest, in the groin, under the breasts, and in elbow creases.	Provide a cooler, less humid environment and keep the affected area dry. Dusting powder may be used to increase comfort, but avoid using ointments or creams, as they keep the skin warm and moist and may make the condition worse.
Heat Cramps			
	Usually affect people who sweat a lot during strenuous activity. This sweating depletes the body's salt and moisture. The low salt level in the muscles causes painful cramps. Heat cramps may also be a symptom of exhaustion.	Muscle pains or spasms -- usually in the abdomen, arms, or legs -- that may occur in association with strenuous activity. If you have heart problems or are on a low-sodium diet, get medical attention for heat cramps.	If medical attention is not necessary, take the following steps: - Stop all activity and sit quietly in cool place - Drink clear juice or a sports drink - Do not return to strenuous activity for a few hours after the cramps subside -- further exertion may lead to exhaustion or stroke Seek medical attention if the cramps do not subside in 1-hr.
Heat Exhaustion			
	A milder form of heat stress that can develop after several days of exposure to high temperatures and inadequate or unbalanced replacement of fluids. Those most prone to heat exhaustion are elderly people, people with high blood pressure, and people working or exercising in a hot environment.	- Heavy sweating - Paleness - Muscle cramps - Tiredness - Weakness - Dizziness - Headache - Nausea or vomiting - Fainting	Cooling measures that may be of help: - Cool, non-alcoholic beverages - Rest - Cool shower, bath, or sponge bath - An air-conditioned environment

HEAT STRESS SYMPTOMS AND APPROPRIATE ACTIONS

	Description	How to Recognize	What To Do
Heat Stroke			
	Occurs when the body is unable to regulate its temperature. The body's temperature rises rapidly, the sweating mechanisms fail, and the body is unable to cool down. Body temperature may rise to 106 degress F or higher within 10-15 minutes. Heat stroke can cause death or permanent disability if emergency treatment is not provided.	Warning signs of heat stroke: - Extremely high body temp. - Red, hot, and dry skin - Rapid, strong pulse - Throbbing headache - Dizziness - Nausea - Confusion - Unconsciousness	Have someone call for immediate medical assistance if you see these signs. While you are waiting for the assistance, begin cooling the victim by: - Get victim to shady place - Cool victim rapidly with whatever methods you can (e.g. cool cloth, cool shower or water splashes) - Monitor body temperature and continue cooling efforts Sometimes a victim's muscles will begin to twitch uncontrollably. If this happens, keep the victim from injuring himself/herself, but do not place any objects in the mouth and do not give fluids. If vomiting occurs, make sure airway is open by turning victim on his or her side.
Sunburn			
	Should be avoided due to skin damage. Although discomfort is usually minor and healing often occurs within a week, a more severe sunburn may require medical attention.	Symptoms are well known: skin becomes red, painful, and abnormally warm after sun exposure.	Consult a physician if these symptoms are present: - Fever - Fluid-filled blisters - Severe pain Also, remember these tips: - Avoid repeated sun exposure - Apply cold compress or water - Do not break blisters

Attachment F
EA Occupational Health Program

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EA OCCUPATIONAL HEALTH PROGRAM

During EA operations, personnel may be exposed to environmental factors and stresses that could affect their well-being. Recognition and evaluation of the importance of these environmental factors (e.g., exposure to airborne contaminants, heat stress, and noise) will determine the need for measures to minimize and/or control exposures. In addition, medical surveillance and periodic exposure monitoring will be provided to ensure that the stresses are not affecting the health of employees.

RESPONSIBILITIES

Director of Safety and Health

The Director of Safety and Health will:

1. Coordinate with All One Health, EA's Corporate Physician, to provide guidance and oversee the Medical Surveillance Program and make all medical determinations
2. Administer the Occupational Health Program, including coordination of EA's medical and first aid program.
3. Periodically conduct an industrial hygiene survey of EA operations involving potential employee exposure to chemicals, noise, and/or radiation. During the survey, areas requiring additional evaluation, e.g., air sampling, will be identified and scheduled for a follow-up investigation.
4. Develop an occupational health management information system. This system will be used to provide medical personnel with data regarding employees' exposures to potentially hazardous materials.
5. Make recommendations to management regarding the minimization or control of exposures to potentially hazardous materials.

Review projects or programs before their application to evaluate the potential impact on the health of employees.

Corporate Physician

The Corporate Physician will:

1. Consult with EA on the appropriate examination and tests for employees potentially exposed to hazardous materials.
2. Where and when warranted, oversee personnel medical evaluations including employees ability to wear respiratory protection

MEDICAL SURVEILLANCE

Employees whose jobs require them to work with chemicals at levels that may be potentially hazardous to their health will be provided physical examinations. The Director of Safety and Health and EA's Corporate Physician will identify employees to be included in the Medical

Surveillance Program. The purpose of the examination is to establish the baseline status of the employee's health and to determine the suitability of the employee for the job. Periodic surveillance may be necessary for individuals potentially exposed to hazardous materials regularly to ensure their work exposure is not impairing their well being.

The Corporate Physician will notify the employee, in writing, of the results of their physical examination and medical tests.

Participants in the Medical Surveillance Program will initially complete the enclosed General Medical Questionnaire and the Periodic Medical Questionnaire annually thereafter. The completed questionnaires will become part of each employee's permanent Safety and Health file as well as being used for the determination of their status in the Medical Surveillance Program.

FREQUENCY OF MEDICAL EXAMINATIONS

Personnel must pass a comprehensive medical examination prior to assignment to field sampling and exploration activities. The purpose of the examination is to establish the individual's baseline physiological data and to determine his/her ability to wear PPE, especially respirators.

The examination will be repeated annually for employees who are potentially exposed to hazardous materials for 30 days or more per year, employees who wear respirators for 30 days or more per year, and for employees who are members of hazardous materials teams. Whenever an employee has developed signs or symptoms indicating possible overexposure to hazardous substances or health hazards, or the employee has been exposed above the established exposure levels in an emergency situation, a medical examination will be performed. A medical examination will also be performed whenever an employee is terminated or reassigned to work that does not require his/her participation in the medical program. Employees who are potentially exposed to hazardous materials less than 30 days per year will receive biennial examinations.

Content of Medical Examinations

The protocol for medical examinations will be determined by the examining physician.

CERTIFICATION FORM TO EMPLOYER

The certification form provides a written opinion regarding each examined employee which includes the following:

1. The physician's written opinion as to whether the employee has detected medical conditions which would place the employee at increased risk of material impairment of the employee's health from work in hazardous waste operations or from the use of respirators.
2. The physician's recommended limitations upon the employee's work assignment.

3. A statement that the employee has been informed by the physician of the results of the medical examination and any medical conditions which require further examination or treatment.

The written opinion must not reveal specific findings or diagnoses unrelated to the occupational exposure.

Recordkeeping Requirements

EA's medical provider will maintain a secure medical and exposure records storage location. The following information must be retained for duration of employment plus 30 years for each employee:

1. The name and social security number of the employee.
2. Physician's written opinions as described above.
3. Employee medical complaints related to exposure to hazardous substances.
4. Medical results.

FIRST AID TREATMENT

First aid training will be provided as needed. Designation of the appropriate individuals to be trained will be made by the Site Safety and Health Officer or Regional Safety and Health Manager or the Operations Managers. Crews working in locations that require more than a 15-minute response time for medical assistance but are unlikely to encounter serious medical harm will be required to have first aid and CPR training. If operations are such that serious medical emergencies may be encountered, then first aid and CPR training will be provided whenever response times are four minutes or more.

Only trained individuals will be permitted to treat EA employees. First aid supplies will be maintained in their custody, and it will be their responsibility to ensure that an adequate supply of materials is on hand at all times. First aid kits will also be maintained in boats and vehicles.

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Attachment G
Accident/Loss and "Near Miss" Reports

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ACCIDENT/LOSS REPORT

THIS REPORT MUST BE COMPLETED BY THE INJURED EMPLOYEE OR SUPERVISOR AND FAXED TO EA CORPORATE HUMAN RESOURCES WITHIN 24 HOURS OF ANY ACCIDENT. THE FAX NUMBER IS **(410) 771-1780**.

NOTE WHENEVER AN EMPLOYEE IS SENT FOR MEDICAL TREATMENT FOR A WORK RELATED INJURY OR ILLNESS, PAGE 4 OF THIS REPORT MUST ACCOMPANY THAT INDIVIDUAL TO ENSURE THAT ALL INVOICES/BILLS/CORRESPONDENCE ARE SENT TO HUMAN RESOURCES FOR TIMELY RESPONSE.

A. DEMOGRAPHIC INFORMATION:

NAME OF INJURED EMPLOYEE: _____
HOME ADDRESS: _____
HOME PHONE: _____ DATE OF BIRTH: _____
AGE: _____ SEX: M F
MARITAL STATUS: _____ HOURLY RATE: _____
SOCIAL SECURITY NUMBER: _____ DATE OF HIRE: _____
NUMBER OF DEPENDENTS: _____
EMPLOYEES JOB TITLE: _____
DEPT. REGULARLY EMPLOYED: _____
WAS THE EMPLOYEE INJURED ON THE JOB: Y N
PRIMARY LANGUAGE OF THE EMPLOYEE: _____

B. ACCIDENT/INCIDENT INFORMATION:

DATE OF ACCIDENT: _____ TIME OF ACCIDENT: _____
REPORTED TO WHOM: _____ NAME OF SUPERVISOR_____
EXACT LOCATION WHERE ACCIDENT OCCURRED (**including street, city, state, and County**):
_____
_____

EXPLAIN WHAT HAPPENED (include what the employee was doing at the time of the accident and how the accident occurred): _____
_____
_____
_____

DESCRIBE THE INJURY AND THE SPECIFIC PART OF THE BODY AFFECTED (i.e. laceration, right hand, third finger):
_____
_____

OBJECT OR SUBSTANCE THAT DIRECTLY INJURED EMPLOYEE: _____
_____
NUMBER OF DAYS AND HOURS EMPLOYEE USUALLY WORKS PER WEEK: _____
IS THE EMPLOYEE EXPECTED TO LOSE AT LEAST ONE FULL DAY OF WORK? _____
DOES THE EMPLOYEE HAVE A PREVIOUS CLAIM? Y N if yes, STATUS: Open Closed
WAS THE EMPLOYEE ASSIGNED TO RESTRICTED DUTY? _____

C. ACCIDENT INVESTIGATION INFORMATION

WAS SAFETY EQUIPMENT PROVIDED? Y N If yes, was it used? Y N
WAS AN UNSAFE ACT BEING FORMED? Y N If yes, describe_____
WAS A MACHINE PART INVOLVED? Y N If yes, describe _____
WAS THE MACHINE PART DEFECTIVE? Y N If yes, in what way _____
WAS A 3RD PARTY RESPONSIBLE FOR THE ACCIDENT/INCIDENT? Y N
If yes, list Name, address, and phone number_____
WAS THE ACCIDENT/INCIDENT WITNESSED? Y N
If yes, list Name, address, and phone number: _____

D. PROVIDER INFORMATION

WAS FIRST AID GIVEN ON SITE? Y N
If yes, what type of medical treatment was given _____
PHYSICIAN INFORMATION (if medical attention was administered)
NAME: _____
ADDRESS (incl. city, state, and zip):_____
PHONE: _____
HOSPITAL ADDRESS (incl. Name, address, city, state, zip code, & phone)_____
_____
WAS THE EMPLOYEE HOSPITALIZED? Y N If yes, on what date_____
WAS THE EMPLOYEE TREATED AS AN OUTPATIENT, RECEIVE EMERGENCY TREATMENT OR AMBULANCE SERVICE? _____

PLEASE ATTACH THE PHYSICIAN'S WRITTEN RETURN TO WORK SLIP

***NOTE* A PHYSICIAN'S RETURN TO WORK SLIP IS REQUIRED PRIOR TO ALLOWING THE WORKER TO RETURN TO WORK**

E. AUTOMOBILE ACCIDENT INFORMATION (complete if applicable)

AUTHORITY CONTACTED AND REPORT #_____
EA EMPLOYEE VEHICLE YEAR, MAKE, AND MODEL_____
V.I.N._____ PLATE/TAG #_____

OWNER'S NAME AND ADDRESS: _____
_____
DRIVER'S NAME AND ADDRESS: _____
_____
RELATION TO INSURED: _____DRIVER'S LICENSE #_____
DESCRIBE DAMAGE TO YOUR PROPERTY: _____
_____
_____
DESCRIBE DAMAGE TO OTHER VEHICLE OR PROPERTY: _____
_____
_____
OTHER DRIVER'S NAME AND ADDRESS: _____
_____
OTHER DRIVER'S PHONE: _____
OTHER DRIVER'S INSURANCE COMPANY AND PHONE_____
_____
LOCATION OF OTHER VEHICLE: _____
NAME, ADDRESS, AND PHONE OF OTHER INJURED PARTIES:_____
_____
_____
WITNESSES NAME: _____ PHONE:_____
ADDRESS: _____
STATEMENT:_____
_____
SIGNATURE: _____
NAME: _____ PHONE: _____
ADDRESS:_____
STATEMENT:_____
_____
SIGNATURE: _____

F. ACKNOWLEDGEMENT

NAME OF SUPERVISOR: _____
DATE OF THIS REPORT: _____ REPORT PREPARED BY:_____

I have read this report and the contents as to how the accident/loss occurred is accurate to the best of my knowledge.

Signature: _____ Date: _____
Injured Employee

I am seeking medical treatment for a work related injury/illness.

Please forward all bills/invoices/correspondence to:

EA ENGINEERING, SCIENCE, AND TECHNOLOGY, INC.

11019 McCORMICK ROAD

HUNT VALLEY, MD 21031

ATTENTION: Michele Bailey
HUMAN RESOURCES

(410) 584-7000
(410) 771-1780 – FAX

"NEAR MISS" INCIDENT REPORT

A "near miss" is a potential hazard or incident that has not resulted in any personal injury or property damage. Unsafe working conditions, unsafe employee work habits, improper use of equipment, or use of malfunctioning equipment have the potential to cause work-related injuries. It is everyone's responsibility to report and correct these potential accidents/incidents immediately.

Please complete this form as a means to report these "near miss" situations.

Department/Location _____ Date: _____ Time: _____ a.m. / p.m.

Please check all appropriate conditions:

_____ Unsafe act _____ Unsafe equipment _____ Other
_____ Unsafe condition _____ Unsafe use of equipment

Description of incident or potential hazard _____

Employee Signature _____ Date _____

"NEAR MISS" INVESTIGATION

Description of the "Near Miss" Condition _____

Causes (primary and contributing) _____

Corrective Action Taken (i.e., remove the hazard; replace, repair, or retrain in the proper procedures for the task) _____

Signed _____ Date Completed _____

Form not completed within 4 hours of incident for the following reason(s) _____

_____ _____
Corporate Safety and Health Director Date

******Forward to Corporate Director of Safety and Health when completed.******

Revised July 2006

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