APPENDIX E – UNIFORM FEDERAL POLICY-QUALITY ASSURANCE PROJECT PLAN

FINAL

UNIFORM FEDERAL POLICY QUALITY ASSURANCE PROJECT PLAN

REMEDIAL INVESTIGATION FOR THE RICOCHET AREA MUNITIONS RESPONSE SITE IN STATE GAME LANDS 211, PENNSYLVANIA

CONTRACT NO.: W9133L-09-F-0304

Prepared for:

NATIONAL GUARD BUREAU
1411 JEFFERSON DAVIS HIGHWAY
ARLINGTON, VA 22202-3231
and
PENNSYLVANIA ARMY NATIONAL GUARD
DEPARTMENT OF MILITARY AND VETERANS AFFAIRS
FORT INDIANTOWN GAP MILITARY RESERVATION
ANNVILLE, PA 17003

Prepared by

WESTON SOLUTIONS, INC.
1400 WESTON WAY
WEST CHESTER, PA 19380
WESTON Project No.: 12767.099.001.0042

MARCH 2010

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TABLE OF CONTENTS

Title **Page**

LIST OF ATTACHMENTS

Attachment A—Analytical Standard Operating Procedures (electronic)

Attachment B—Laboratory Quality Assurance Manual (electronic)

LIST OF FIGURES

Figure No. **Page**

LIST OF ACRONYMS

%R	percent recovery
%D	percent difference
BS	blank spike
CA	Corrective Action
CAR	Corrective Action Report
CCB	continuing calibration blank
CCV	continuing calibration verification
CLP	Contract Laboratory Program
COR	Contracting Officer Representative
CPR	cardiopulmonary resuscitation
CSM	Conceptual Site Model
CVAA	Cold Vapor Atomic Absorption
DMM	discarded military munitions
DoD	Department of Defense
DOT	Department of Transportation
DQI	Data Quality Indicator
DQO	Data Quality Objective
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
ERIS	Environmental Restoration Information System
GIS	Geographic Information System
HPLC	High Performance Liquid Chromatography
HRR	Historical Records Review
IATA	International Air Transport Association
ICAL	initial calibration
ICB	initial calibration blank
ICP	inductively coupled plasma
ICV	initial calibration verification
IDW	investigation derived waste
ISO	Industry Standard Object
IVS	Instrument Verification Strip
LCS	laboratory control sample
LIMS	Laboratory Information Management System
MB	method blank
MC	munitions constituent
MCGI	Meridian Consultant Group, Inc.

LIST OF ACRONYMS (CONTINUED)

MD	matrix duplicate
MDL	method detection limit
MEC	munitions and explosives of concern
MPC	Measurement Performance Criteria
MRS	Munitions Response Site
MS	matrix spike
MSD	matrix spike duplicate
N/A	not applicable
NGB	National Guard Bureau
OSHA	Occupational Safety and Health Administration
PAARNG	Pennsylvania Army National Guard
PADEP	Pennsylvania Department of Environmental Protection
PGC	Pennsylvania Game Commission
PQO	Project Quality Objective
QA	quality assurance
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QC	quality control
QSM	Quality System Manual
R	rejected
RI	Remedial Investigation
RL	reporting limit
RPD	relative percent difference
SI	Site Investigation
SDR	Sample Discrepancy Report
SOP	Standard Operating Procedure
TAL	target analyte list
TPP	Technical Project Planning
TSA	Technical System Audit
USACE	U.S. Army Corps of Engineers
UFP	Uniform Federal Policy
UXO	unexploded ordnance
WESTON®	Weston Solutions, Inc.

Introduction

This Quality Assurance Project Plan (QAPP) has been developed to support the Remedial Investigation (RI) at the Ricochet Area Munitions Response Site (MRS), located within State Game Lands 211, Pennsylvania. The QAPP provides information on five areas: (1) Project Management and Objectives, (2) Measurement and Data Acquisition, (3) Field Sampling Rationale, (4) Assessment and Oversight, and (5) Data Review. This document meets the requirements and elements set forth in the Department of Defense (DoD) Quality System Manual Version 4 (QSM), and the Uniform Federal Policy-Quality Assurance Project Plan Manual (United States Environmental Protection Agency, EPA505-B-04-900A, Version 1, 2005). This QAPP provides a process for obtaining data of sufficient quality and quantity to satisfy project needs. It describes policy, organization, functional activities, and the data quality objectives, and measures necessary to obtain adequate data for a given purpose. Additionally, it clearly identifies the rationale for selection of the proposed sampling locations, analysis, and specific procedures for collecting data during the RI. The field work and data evaluation will be completed in accordance with this QAPP. As any new procedure is required, addendums to this document will be issued.

All staff participating in project/field efforts are required to read this plan and become familiar with the analytical procedures and the implementation of these procedures to ensure that analytical/sample goals are met consistently. In addition, key personnel are responsible to mentor assigned staff in aspects of this UFP-QAPP that would have a potential impact on the work assigned to them.

Worksheet 1 — Title and Approval Page

Document Title: Final UFP-QAPP. Remedial Investigation of the Ricochet Area Munitions Response Site (MRS)

Lead Organization: National Guard Bureau (NGB) and Pennsylvania Army National Guard (PAARNG)

Preparer's Name and Organizational Affiliation: Kelly Spittler
Weston Solutions, Inc.

Preparer's Address, Telephone Number, E-mail Address: 1400 Weston Way
West Chester, PA 19380
610-701-3953
k.spittler@westonsolutions.com

Preparation Date (Day/Month/Year): March 2010

Investigative Organization's Senior Chemist: Kelly Spittler/Weston Solutions, Inc.

Investigative Organization's Project Manager: Gregory Daloisio/Weston Solutions, Inc.

Investigative Organization's QA Manager: Bob McGlade/Weston Solutions, Inc.

Approval Signatures:

Lead Organization's Program Manager: Kim Harriz/National Guard Bureau

Regulatory Organization: John F. Krueger/Pennsylvania Department of Environmental Protection

Worksheet 2 — QAPP Identifying Information

Site Name/Project Name: Ricochet Area MRS/MMRP RI
Site Location: State Game Lands 211, Pennsylvania
Site Number/Code: Not Applicable (N/A)
Operable Unit: N/A
Contractor Name: Weston Solutions, Inc.
Contract Number: W9133L-09-F-0304
Contract Title: Remedial Investigation (RI) at Ricochet Area at FTIG-003-R-01
Work Assignment Number: 12767.099.001

1. Identify guidance used to prepare QAPP: Uniform Federal Policy for Quality Assurance Project Plans: Part 1 UFP-QAPP Manual (March 2005).

2. Identify regulatory program: Military Munitions Response Program

3. Identify approval entities: NGB, PAARNG, and Pennsylvania Department of Environmental Protection (PADEP)

4. The QAPP is: Project-Specific

5. List dates scoping sessions that were held:
   a) RI Client/Contractor Kick-off Meeting – October 13, 2009
   b) Technical Project Planning (TPP)-1 – November 19, 2009
   c) TPP-2 – January 14, 2010

6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Received Date
Site Inspection Work Plan	September 2007

7. List organizational partners (stakeholders) and connection with lead organization: PADEP, Pennsylvania Game Commission (PGC), and Fort Indiantown Gap tenants.

8. List data users: NGB, PAARNG, PADEP, U.S. Environmental Protection Agency (EPA), U.S. Army Corps of Engineers (USACE) and WESTON

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below: All QAPP worksheets are applicable.

Worksheet 2 — QAPP Identifying Information (Continued)

Required QAPP Element(s) and Corresponding QAPP Worksheet(s)	Crosswalk to Required Documents	Optional Worksheet in QAPP Workbook	Required Information
Project Management and Objectives			
2.1 Title and Approval Page	RI WP Signature Page	1	- Title and Approval Page
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	RI WP Table of Contents	2	- Table of Contents - QAPP Identifying Information
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	RI WP Cover Letter APP/SSHP Signature page	3 4	- Distribution List - Project Personnel Sign-Off Sheet
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	RI WP Section 4 APP/SSHP Section 5	5 6 7 8	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	RI WP Sections 1, 2	9 10	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	RI WP Section 2.7	11 12	- Site-Specific Project Quality Objectives (PQOs) - Measurement Performance Criteria Table
2.7 Secondary Data Evaluation		13	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	RI WP Sections 2.5, 3, Appendix K	14 15 16	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table

Worksheet 2 — QAPP Identifying Information (Continued)

Required QAPP Element(s) and Corresponding QAPP Worksheet(s)	Crosswalk to Required Documents	Optional Worksheet in QAPP Workbook	Required Information
Measurement/Data Acquisition			
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	RI WP Section 3.10	17 18 19 20 21 22	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/Standard Operating Procedure (SOP) Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control Sample Summary Table - Sampling SOPs - Project Sampling SOP Reference Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	QAPP Attachment A	23 24 25	- Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	RI WP Section 3.10	26 27	- Sample Handling System - Sample Collection, Documentation Handling, Tracking, and Custody SOPs - Sample Custody Requirements Table - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal

Worksheet 2 — QAPP Identifying Information (Continued)

Required QAPP Element(s) and Corresponding QAPP Worksheet(s)	Crosswalk to Required Documents	Optional Worksheet in QAPP Workbook	Required Information
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	RI WP Section 3.10.8	28	- Quality Control (QC) Samples Table - Screening/Confirmatory Analysis Decision Tree
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	RI WP Section 3.10.9	29 30	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs
Assessment/Oversight			
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	RI WP Section 5	31 32	- Planned Project Assessments Table - Assessments and Response Actions - Audit Checklists - Assessment Findings and Corrective Action Responses Table
4.2 Quality Assurance (QA) Management Reports	RI WP Section 5	33	- QA Management Reports Table
4.3 Final Project Report			- All information obtained during RI Field work
Data Review			
5.1 Overview			
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	RI WP Section 3.10.11 RI WP Section 3.12	34 35 36 37	- Sampling and Analysis Verification (Step I) Process Table - Sampling and Analysis Validation (Steps IIa and IIb) Process Table - Sampling and Analysis Validation (Steps IIa and IIb) Summary Table - Data Usability Assessment

Worksheet 2 — QAPP Identifying Information (Continued)

Required QAPP Element(s) and Corresponding QAPP Worksheet(s)	Crosswalk to Required Documents	Optional Worksheet in QAPP Workbook	Required Information
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining			

Worksheet 3 — Distribution List

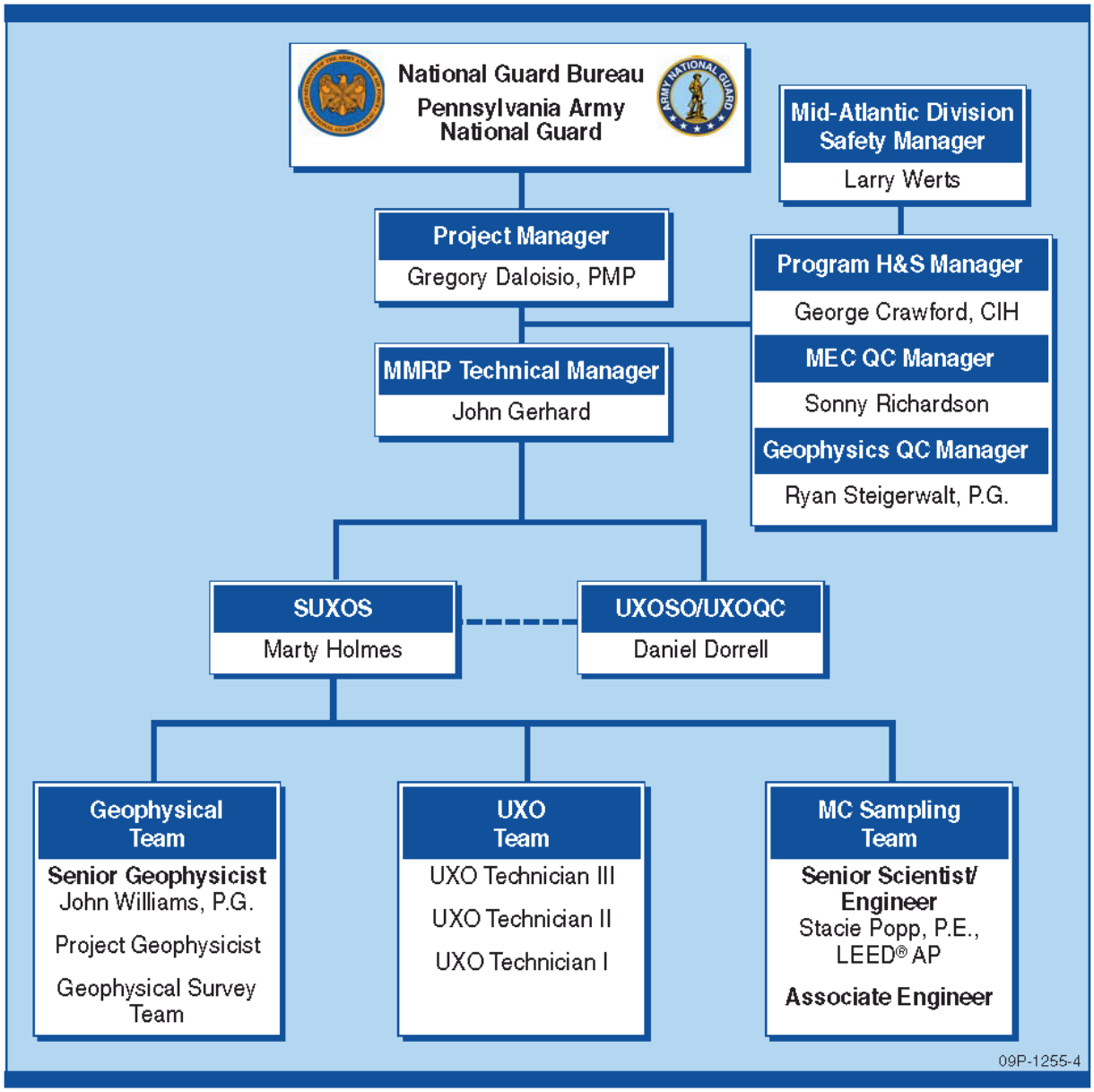
QAPP Recipients	Title	Organization	Number of Copies	Telephone Number	E-Mail Address
Kimberly Harriz	Contracting Officer Representative (COR) and Cleanup Program Manager	NGB	1	703-607-7991	Kim.Harriz@us.army.mil
Joan Anderson	Environmental Compliance	PAARNG	1	717-861-9414	joaanderso@state.pa.us
Lieutenant Colonel Chris Cleaver	Public Affairs Officer	PAARNG	1	717-861-8468	c-ccleaver@state.pa.us
Kenneth Beard	Environmental Group Manager	PADEP	1	717-783-9475	kbeard@state.pa.us
Cliff Opdyke	Risk Assessor	USACE	1	410-962-6765	Clifford.A.Opdyke@usace.army.mil
Gregory Daloisio	Project Manager	WESTON	1	610-701-3786	G.Daloisio@westonsolutions.com
John Gerhard	Deputy Project Manager/MMRP Technical Manager	WESTON	1	610-701-3793	J.Gerhard@westonsolutions.com
Bob McGlade	QA Manager	WESTON	1	610-701-3133	R.McGlade@westonsolutions.com
Stacie Popp-Young	WESTON	Project Engineer	1	610-701-3637	Stacie.Popp.Young@westonsolutions.com
Kelly Spittler	Chemist	WESTON	1	610-701-3953	K.Spittler@westonsolutions.com
Sherif Mina	Data Validator	MCGI	1	301-803-9207	S.Mina@meridiancgi.com
Elaine Walker	Project Manager	TestAmerica	1	303-736-0105	Elaine.Walker@testamericainc.com

A hard copy of the WP will also be made available to the field team during RI activities.

Worksheet 4 — Project Personnel Sign-Off Sheet

Project Personnel	Organization	Title	Signature	Date QAPP Read E-Mail Receipt
Kimberly Harriz	NGB	Contracting Officer Representative (COR) and Cleanup Program Manager		
Joan Anderson	PAARNG	Environmental Compliance		
Kenneth Beard	PADEP	Environmental Group Manager		
Cliff Opdyke	USACE	Risk Assessor		
Gregory Daloisio	WESTON	Project Manager		
John Gerhard	WESTON	MMRP Technical Manager		
Kelly Spittler	WESTON	Project Chemist		
Stacie Popp-Young	WESTON	Project Engineer		
Bob McGlade	WESTON	QA Manager		
Sherif Mina	MCGI	Data Validator		
Elaine Walker	TestAmerica	Project Manager		
TBD	WESTON	Field Personnel		
TBD	WESTON	Field Personnel		

Worksheet 5 — Project Organizational Chart



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Worksheet 6 — Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Point of Contact with NGB	WESTON Project Manager	Gregory Daloisio	610-701-3786	Reporting of project information to the NGB Project Managers through work plans, monthly progress reports, E-mail updates, teleconference calls, and meetings.
Manage All Project Phases	WESTON Project Manager WESTON Deputy Project Manager/MMRP Technical Manager	Gregory Daloisio John Gerhard	610-701-3786 610-701-3793	Primary modes of communication are telephone, E-mail, letter, document submittal; timing dependent on nature of communication and predefined schedules, as applicable and as requested by agencies.
QAPP Changes in the Field, Daily Field Progress Reports, Field Corrective Action	WESTON MMRP Technical Manager	John Gerhard	610-701-3793	Notify WESTON Project Manager and Project Chemist of changes to QAPP in the field and rationale for changes. Document changes in field daily progress reports and memoranda to WESTON, and NGB Project Managers. Field Engineer will complete daily field progress reports and forward to WESTON. Need for field corrective action will be determined by the Technical Manager and Project Manager and will be documented in the daily field progress reports and memoranda to WESTON and NGB Project Managers.
Reporting Laboratory Data Quality Issues	TestAmerica Laboratory Project Manager	Elaine Walker	303-736-0105	All QA/QC issues with project field samples will be reported by the laboratory to the Project Chemist and Contractor QA Officer.

Worksheet 6 — Communication Pathways (Continued)

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Laboratory Analytical Corrective Actions	Project Chemist Laboratory Project Manager	Kelly Spittler Elaine Walker	610-701-3953 303-736-0105	Need for laboratory corrective actions will be determined by the Project Chemist and/or laboratory Project Manager or QA Manager and will be documented in memoranda to WESTON and NGB Project Managers.
Data Tracking and Management, Release of Analytical Data, QAPP Amendments	Project Chemist	Kelly Spittler	610-701-3953	Project Chemist or her delegated representative will track data from collection of samples through login at laboratory to delivery by technical report/sample data group and electronic data delivery into database. Final analytical data cannot be released until validation is complete and Project Chemist has approved release. Changes to the QAPP will be approved by the WESTON and NGB Project Managers.

Worksheet 7 — Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Gregory Daloisio, PMP	Project Manager	WESTON	Provides overall management of the contract including cost, schedule and technical quality. Manages project staffing, day-to-day project operations and activities, deliverable completion, field investigations, quality control, and health and safety. Acts as the single point of contact for the contract. Maintains communication and coordination with NGB for the duration of the project, including progress and detailed cost reporting. Oversees the management and coordination between Contractor staff, subcontractors, and NGB.	B.S., Mechanical Engineering, 26 years of environmental experience, more than 20 years of Project Management experience
John Gerhard	MMRP Technical Manager	WESTON	Responsible for assisting Project Manager and providing senior technical support on MMRP/CERCLA process documents, sampling program design and implementation, and project team coordination. Initiates field corrective action if deemed necessary.	B.S., Environmental Resource Management, 12 years of environmental experience
Bob McGlade	QA/QC Manager	WESTON	Responsible for program quality management, including training and programmatic quality processes and controls. Provides senior technical support on CERCLA process documents and sampling program design and implementation.	B.S., Environmental Biology; 18 years of CERCLA hazardous waste site investigation and cleanup experience.

Worksheet 7 — Personnel Responsibilities and Qualifications Table (Continued)

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Kelly Spittler	Project Chemist	WESTON	Manages analytical and data validation subcontractors. Negotiates project specifications and coordinates the sample collection activity with laboratory capacity. Tracks all samples from collection through analysis, data validation, and report generation. Serves as the primary chemist for all analytical issues. Initiates corrective actions as deemed appropriate. Supervises the electronic loading for all analytical data to ensure compliance with contract requirements.	B.S., Chemistry, Data Validation Certifications; 22 years of project chemistry and data validation experience
Robert Hanisch	Laboratory Manager	TestAmerica	Supervises all laboratory personnel and provides guidance and direction as needed. Responsible for ensuring compliance and integration of facility operation with corporate and regulatory policies and procedures.	M.A., Chemistry, 35 years of environmental science and laboratory management experience

Worksheet 7 — Personnel Responsibilities and Qualifications Table (Continued)

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Karen Kuoppala	Laboratory Quality Assurance Manager	TestAmerica	Overall responsibility for the development, implementation and maintenance of the laboratory's quality system and QC activities. Performs internal system and technical audits. Creates corrective action plans and recommends projects for process improvement. Troubleshoots and resolves problems and provides technical and administrative guidance to laboratory staff. Obtains and maintains laboratory license and permits and oversees laboratory accreditation and certification programs. Serves as the liaison between the laboratory and regulatory offices.	B.A., Chemistry and Geology, 25 years of project, operations and quality assurance management
Elaine Walker	Laboratory Project Manager	TestAmerica	Serves as the laboratory's primary contact for the project. Utilizes a variety of project management tools for forecasting, and production status tracking. Assures laboratory compliance with project needs in both QC and project deliverables.	B.S., Geology, 19 years experience in client service and project management
Sherif Mina	Data Validator	Meridian Consulting Group, Inc.	Responsible for operations management and technical support. Attends data validation training refreshers in EPA Regions I, II and III. Performs data validation for analytical analyses under this contract, per the EPA Region III guidelines.	B.S., Chemistry, M.S., Applied Chemistry; 24 years experience in environmental laboratory operations; 17 years of data validation experience.

Worksheet 8 — Special Personnel Training Requirements Table

Project Function	Specialized Training by Title or Description of Course	Training Provider	Training Date[1]	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records/Certificates[2]
Field Sampling Team Lead	40-Hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Site Worker Training; 8-Hour OSHA Refresher Training; First Aid Cardiopulmonary Resuscitation (CPR)	Registered Training Organization – Various[1]	Varies	All	Various	Certificates available upon request and maintained at project office.
Field Technicians, Geologists, Environmental Scientists, Engineers	40-Hour OSHA Hazardous Waste Site Worker Training; 8-Hour OSHA Refresher Training; First Aid CPR	Registered Training Organization – Various[1]	Varies	All	All team personnel assisting in the performance of this contract.	Certificates available upon request and maintained at project office.

[1] Training Provider and date of training will vary from person to person due to individual scheduling of training.

[2] Training records and/or certificates are on file at the Weston Solutions, Inc., West Chester, Pennsylvania office.

Worksheet 9 — Project Scoping Session Participants Sheet

Project Name: Ricochet Area RI
Projected Dates of Sampling: March-May 2010
Project Manager: Gregory Daloisio, WESTON

Site Name: Ricochet Area MRS
Site Location: State Game Lands 211, Pennsylvania

Date of Session: November 19, 2009 and January 14, 2010
Scoping Session Purpose: Technical Project Planning

See Appendix D of the Work Plan for meeting minutes from the TPP meetings.

Comments/Decisions:

Scoping sessions will be an ongoing feature of the project as activities progress. Bi-weekly project status meetings between WESTON, NGB, PAARNG, and USACE and project personnel are conducted to discuss the following:

- Summary of progress for the project
- Key milestones / deliverables
- Upcoming site activities
- Issues
- Status of action items

Action Items:

See Appendix D of the Work Plan for meeting minutes from the TPP meetings.

Consensus Decisions:

See Appendix D of the Work Plan for meeting minutes from the TPP meetings.

Worksheet 10 — Problem Definition

Existing information on the nature and extent of potential munitions and explosives of concern (MEC) and munitions constituents (MC) contamination is insufficient to evaluate and recommend remedial alternatives.

Worksheet 11 — Project Quality Objectives/Systematic Planning Process Statements

The geophysical was developed based on applicable guidance criteria (i.e., EM 1110-1-4009) and other pertinent documents, along with a combination of geophysical mapping tools (including analog and digital instruments), survey patterns (transects and grids), and statistical tools (i.e., GIS spatial analyses and USACE UXO-Estimator calculator).

The Data Quality Objectives (DQOs) for MEC/MC characterization are presented in Section 2.7 of the Work Plan.

Worksheet 12 — Measurement Performance Criteria Tables

Worksheet 12.1 — Measurement Performance Criteria Table – Explosives Method SW-846 8330B

Matrix	Soil, Sediment
Analytical Group	Explosive Compounds
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Samples and/or Activity to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S and A)
Explosives in Soil/Sediment Sample placed in a Teflon-lined glass jar. (SOPs SS-3, G-9)	SW-846 8330B (SOPs A-1)	Field Precision	1 per 20 samples RPD< 50% (soil) RPD < 30% (water)	Field Duplicate	S and A
		Field Representativeness/ Accuracy/Bias	1 per 20 samples/matrix <½ PQL	Equipment Rinsate	S and A
		Accuracy/ Precision	Per Field Team submission See Table 15.6	Matrix Spike and Matrix Spike Duplicate	S and A
		Accuracy/Precision	One Every 3 months All analytes within ±15% of expected value	High Calibration Standard	A
		Accuracy/Bias	Each sample for each analyte RRT of the analyte within ±0.06 RRT units of the RRT	Retention Time Window	A

Worksheet 12.1 — Measurement Performance Criteria Table – Explosives Method SW-846 8330B (Continued)

Matrix	Soil, Sediment
Analytical Group	Explosive Compounds
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Samples and/or Activity to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S and A)
		Accuracy/Precision	Five-point calibration for all analytes prior to sample analysis Linear mean RSD for all analytes ±20%, with no individual analyte RSD >30%	Initial Calibration	A
		Accuracy/Bias	Daily before sample analysis Within ±15% of expected value	Initial Calibration Verification	A
		Precision	After every 10 samples and at end of sequence All analytes within ±15% of expected value	Continuing Calibration Verification	A
		Laboratory Representativeness/ Accuracy/Bias	Prior to sample analysis and after every 10 samples and at end of sequence <½ PQL	Instrument Blank Solution	A
		Accuracy/Bias	Every sample See Table 15.6	Surrogate	A
		Laboratory Representativeness/ Accuracy/Bias	1 per batch per matrix or 1 per 20 samples, whichever is more frequent <½ PQL	Method Blank	A
		Laboratory Accuracy/Sensitivity	1 per batch per matrix or 1 per 20 samples, whichever is more frequent See Table 15.6	Laboratory Control Sample	A

Worksheet 12.2 — Measurement Performance Criteria Table – Metal Analytes Method SW-846 6010B

Matrix	Soil, Sediment
Analytical Group	Metal Analytes
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Samples and/or Activity to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S and A)
Metals in Soil/Sediment Sample placed in a Teflon-lined glass jar. (SOPs SS-3, G-9) .	SW-846 6010B (SOP A-2)	Field Precision	1 per 20 samples RPD of ±20%, if concentration is ≥ 5x RL; or ± the RL if the concentration id < 5x RL	Field Duplicate	S and A
		Field Representativeness/ Accuracy/Bias	1 per 20 samples/matrix <½ PQL	Equipment Rinsate	S and A
		Accuracy/Bias	Per Field Team submission See Table 15.8	Matrix Spike	A
		Laboratory Precision	1 per 20 samples per matrix See Table 15.8	Laboratory Duplicate (Replicate)	A
		Accuracy/Precision	Daily prior to sample analysis (minimum 1 standard and a blank)	Initial Calibration	A
		Accuracy/Bias	Daily after initial calibration All analytes within ±10% of expected value	Initial Calibration Verification	A

Worksheet 12.2 — Measurement Performance Criteria Table – Metal Analytes Method SW-846 6010B (Continued)

Matrix	Soil, Sediment
Analytical Group	Metal Analytes
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Samples and/or Activity to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S and A)
		Accuracy/Bias	After every calibration/verification No analytes detected ≥½ reporting limit (RL)	Calibration Blank Initial Calibration Blank/Continuing Calibration Blank (ICB/CCB)	A
		Precision/Accuracy	After every 10 samples at the end of the analysis sequence All analytes within ±10% of expected value and RSD of replicate integrations <5%	Calibration Verification (Instrument Check Standard)	A
		Precision	At beginning of analytical run Must bet within ± 2x the RL of the analyte's true value or ± 20% of the analyte's true value, whichever is greater	Interference Check Solution	A
		Precision/Accuracy	Must agree within 10% of the original sample; only applicable if the analyte concentration is > a factor of 50 above the method detection limit (MDL) (ICP) or > a factor of 25 above the MDL (GFAA and CVAA)	Serial Dilution	A
		Laboratory Representativeness/ Accuracy/Bias	1 per batch per matrix <½ PQL	Method Blank	A
		Laboratory Accuracy/ Sensitivity	1 per batch per matrix or 1 per 20 samples, whichever is more frequent See Table 15.8	Laboratory Control Sample	A

Worksheet 12.3 — Measurement Performance Criteria Table – Mercury Methods SW-846 7470A/7471A

Matrix	Soil, Sediment
Analytical Group	Mercury
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Samples and/or Activity to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S and A)
Mercury in Soil/Sediment Sample placed in a Teflon-lined glass jar. (SOPs SS-3, G-9) .	SW-846 7470A/7471A (SOPs A-3, A-4)	Field Precision	1 per 20 samples RPD< 50% (soil) RPD < 30% (water)	Field Duplicate	S and A
		Field Representativeness/ Accuracy/Bias	1 per 20 samples/matrix <½ PQL	Equipment Rinsate	S and A
		Accuracy/Bias	Per Field Team submission See Table 15.2	Matrix Spike	A
		Laboratory Precision	1 per 20 samples per matrix RPD<20%	Laboratory Duplicate (Replicate)	A
		Accuracy/Precision	Daily prior to analysis Correlation coefficient ±0.995 for linear regression	Initial Calibration	A

Worksheet 12.3 — Measurement Performance Criteria Table – Mercury Methods SW-846 7470A/7471A (Continued)

Matrix	Soil, Sediment
Analytical Group	Mercury
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Samples and/or Activity to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S and A)
		Accuracy/Bias	Once per initial daily multipoint calibration Analyte within ±10% of expected value	Second Source Calibration Check Standard	A
		Laboratory Representativeness/ Accuracy/Bias	One per initial daily multipoint calibration No analyte detected ≥PQL	Calibration Blank	A
		Precision	After every 10 samples and at end of the analysis sequence Analyte within ±20% of expected value	Calibration Verification	A
		Laboratory Representativeness/ Accuracy/Bias	1 per batch per matrix <½ PQL	Method Blank	A
		Laboratory Accuracy/Sensitivity	1 per batch per matrix or 1 per 20 samples, whichever is more frequent See Table 15.2	Laboratory Control Sample	A

Worksheet 12.4 — Measurement Performance Criteria Table – DGM Geophysical Surveys

Quality Control Parameter	Instrument	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	Activity Used to Assess Measurement Performance
Background Noise	G-858	Accuracy	Vertical Gradient Standard Deviation <2.5 nanoTeslas (nT).	Run statistics on all data below a reasonable level (between +/-10nT).
Mean Acquisition Speed	G-858	Accuracy	<3 mph. 95% within max project design speed or demonstrated speed.	Run statistics on velocity between points in each file (create "velocity channel").
Along-Track Measurements	G-858	Accuracy	98% <= cm along line.	Run statistics on distance between points in each file.
Cross-Track Measurements	G-858	Accuracy	The across-track line spacing will not exceed 3 ft. on 95% of the data. 5% of the data may lie between 2.5 and 3 ft. This will allow for variation in spacing reporting caused by rough terrain.	Run statistics on distance between data lines in each file and a manual review based on gridded data between lines.
Coverage (Grids)	G-858	Accuracy	>90% coverage at project design spacing.	Coverage maps will be created per grid or data set.
Dynamic Detection Repeatability	G-858	Precision	Grids – Test item anomaly characteristics (peak response and size) repeatable with allowable variation +/- 25%.	Perform survey over a minimum (1) ISO QC test item per grid.
			QC Industry Standard Object (ISO) test item anomaly characteristics (peak and size) repeatable to +/-25%, with allowable variation.	
GPS Accuracy	G-858	Accuracy	Kinematic positional error at known monuments will not exceed +/- 20 cm.	Perform QC audit of positioning system error test records.
Instrument Latency	G-858	Accuracy	No zig-zag or chevron effects.	Perform latency tests for transect data. Correction will be based on specific correction factors at beginning and end of each day for tests.
Dynamic Positioning Repeatability	G-858	Accuracy	Transects – Demonstrate Instrument Verification Strip (IVS) reacquisition (reacquisition amplitude. ~ original and offset <=1m).	Perform repeat of the IVS transect twice daily.
			Grids – Position offset of test item target <= 35-cm +1/2 line spacing; (<=50cm +1/2 line spacing for fiducially positioned data).	Perform repeat of the IVS and QC seed program data.
Standard Response	G-858	Precision	Response above background to standard object will not vary more than +/-20%.	Perform standardization tests: QC audit of response test records.

Worksheet 12.4 — Measurement Performance Criteria Table – DGM Geophysical Surveys (Continued)

Quality Control Parameter	Instrument	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	Activity Used to Assess Measurement Performance
Magnetic Heading	G-858	Accuracy	No "striping" visible in vertical gradient data above a 0.2 nT per foot level between lines and no "striping" visible in total field data above a 0.4nT/ft level between lines.	Not Applicable
Target Selection	G-858	Accuracy	All dig list targets are selected according to project design/selection criteria and classification scheme.	By grid or dataset. Visual and manual review by QC Geophysicist
Anomaly Resolution	G-858	Accuracy	Resolved is defined as: (1) there is no geophysical signal remaining at the flagged/selected location, or (2) a signal remains but it is too low or too small to be associated with unexploded ordnance/ discarded military munitions (UXO/DMM), or (3) a signal remains but is associated with surface material which when moved results in low, or no signal at the interpreted location, or 4) a signal remains and a complete rationale for its presence exists.	Per Anomaly

Worksheet 12.5 – Measurement Performance Criteria Table – QC Tests for DGM Geophysical Surveys

Quality Control Parameter	Frequency	Instrument	Data Quality Indicators (DQIs)	Measurement Performance Criteria	Activity Used to Assess Measurement Performance
Six-line Test	1st Day of Project	G-858	Accuracy/Precision	The positions of the anomalies from the six passes will be evaluated to ensure the data are being located accurately.	Six passes over a known point. Passes 1 and 2 will have no spike object present. Passes 3 through 6 will have a spike object. Pass five will be walked slowly, and the sixth pass will be walked quickly.
Static Test	Start and End of Day	G-858	Precision	Pre- and post-survey responses should be within 20% of one another.	The test will record background responses for 3 minutes at the "QC stand," followed by a 3-minute static spike test over a standard QC item.
Personnel Test/Cable Shake	Start of Day	G-858	Accuracy	Readings should not exceed 3 nT/ft. The cable shake should not exhibit spikes in the data.	The operator will shake cables to ensure that cables and connectors are in good working order.
Latency Test	Start and End of Day	G-858/GPS	Accuracy	Apply correction value based on the lags or time differences observed in anomaly peak positions for the spike objects.	Traverse over a spike object at the end of the IVS bi-directionally.
Height Optimization Test	1st Day of Project	G-858	Accuracy/Precision	The signal to noise ratios for each test will be compared to determine the maximum signal-to-noise (S/N) ratio that reliably detects the smallest target object.	The operator will carry the instrument over the seed items with the sensors at different heights above the ground surface.
Octant Test	1st Day of Project	G-858	Accuracy/Precision	Document heading error for post-processing correction.	The operator will keep the sensors stationary and rotate around them through 360 degrees to determine if dropouts are more likely in a particular direction based on the sensor's positions relative to the earth's magnetic field.

Worksheet 12.5 – Measurement Performance Criteria Table – QC Tests for DGM Geophysical Surveys (Continued)

Quality Control Parameter	Frequency	Instrument	Data Quality Indicators (DQIs)	Measurement Performance Criteria	Activity Used to Assess Measurement Performance
DGM Repeatability	Daily part of IVS	G-858	Precision	Data are repeatable +/-20% of response amplitude, +/-20 cm for positional accuracy.	The operator will survey the IVS at minimum of twice daily.
False Positives	Duration of Project	Field Operations / G-858	Accuracy/Precision	The project goal is to achieve a false positive rate below 15%.	False positives will be documented in the database so that the 15% false positive metric can be monitored.

Worksheet 13 — Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Site Inspection (SI)	URS, *Final Site Inspection,* September 2008.	Background information on nature and distribution of MEC Geophysical and visual surveys Munitions constituent (MC) sampling and analysis (2007)	Revision of Conceptual Site Model (CSM), if needed. Development of RI approach. Guide MC sampling approach	Data gaps exist. Insufficient information about DMM or propellant disposal at former Cold Spring Firing Point. MEC surveys limited MC sampling data limited
Historical Records Review (HRR)	URS, *Final Historical Records Review,* June 1007	Background information on Army activities	Background information supports MEC/MC results or lack thereof	Incomplete information of past ranges and range activities

Worksheet 14 — Summary of Project Tasks

This worksheet provides the laboratory project tasks following MC sample collection and analysis. Section 3.10 of the Work Plan provides details of MC sampling project tasks (e.g., sampling, analysis, data management, document and record, and assessment tasks).

Data Reduction

Data reduction is the process for collecting and transforming measurements, through mathematical and/or statistical formulas, into final reportable measurements. The calculations may be performed manually or electronically. This worksheet describes the quality assurance processes that will be applied during data reduction to ensure that data collected at the site and data generated at the laboratory are valid.

Laboratory Data Reduction

Data reduction is performed by the analyst and consists of calculating concentrations in samples from the raw data. 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. Copies of all raw data and the calculations used to generate the final results, such as bound laboratory notebooks, strip-charts, chromatograms, spreadsheets, and computer record files, are retained on file, as specified in this QAPP.

Calculations and data reduction steps for various methods are summarized in the respective laboratory SOPs (see Attachment A) or program requirements.

Data Review

Data review is performed to assess whether the quality control requirements are met. Data review will be performed on 100% of the data deliverables.

Laboratory Data Review

The individual analyst continually reviews the quality of data through calibration checks, quality control sample results, and performance evaluation samples. The analyst initiates data review during, immediately following, and after the completed analysis. The Laboratory Supervisor, analyst, or data specialist performs a secondary review of the data. The peer reviewer is trained by the QA Worksheet, Worksheet Manager, or Unit Leader to perform the data review.

Documentation and Records

Laboratory Documentation

Analytical reports comprise final results (uncorrected for blanks and recoveries, unless specified), methods of analysis, levels of reporting, surrogate recovery data, and method blank data. In addition, special analytical problems will be noted in the case narratives. The number of significant figures reported will be consistent with the limits of uncertainty inherent in the analytical method. Consequently, most analytical results will be reported to no more than two or three significant figures. Data are normally reported in units commonly used for the analyses performed.

Concentrations in liquids are expressed in terms of weight or activity per unit volume (e.g., micrograms per liter [µg/L], or milligrams per liter [mg/L]). Concentrations in solid or semisolid matrices are expressed in terms of weight or activity per unit weight of sample (e.g., micrograms per kilogram [µg/kg], or milligrams per kilogram [mg/kg]). Solid and semisolid matrices will also be reported on a dry weight basis. Reporting limits take into account all appropriate concentration, dilution, and/or extraction factors.

If any analytical anomalies are encountered during the analyses (e.g., an out-of-control matrix duplicate), it is documented in a case narrative and copies of the Sample Discrepancy Reports (SDRs) or Corrective Action Reports (CARs) must be included in the laboratory data packages. Contract Laboratory Program (CLP)-type data packages will be submitted for this program. Samples will be submitted to the laboratory on a 21-day turnaround (quick turnaround time (TAT) may be requested, as needed). Both the full documentation package and electronic data will be provided on the actual due date.

Laboratory Record Keeping

At a minimum, subcontracted laboratories will retain all data related to sample preparation, analysis, and general observations in appropriate hardbound laboratory notebooks or files. Laboratory notebook pages must be reviewed, signed, and dated by the author and receive an independent secondary review by a peer or supervisor who signs/initials and dates the data pages.

Corrections to notebook entries are made by drawing a single line through the erroneous entry and writing the correct entry next to the one that is crossed out. All corrections are initialed and dated by the individual performing the correction.

After delivering acceptable hard copy and/or electronic data deliverables, the laboratory will store the original project data for at least 5 years unless otherwise specified in the subcontract agreement.

Assessment and Audit Tasks

A subcontractor laboratory Technical System Audit (TSA) audit may be performed at any time during this program. In the event that laboratory performance does not meet QAPP

requirements and/or significant data quality issues arise, WESTON reserves the right to perform additional system/project audits at any time throughout the program.

Checklists are to be used to ensure that all salient points are addressed and documented. The checklists are filled out legibly and reproducibly, in ink, by the auditor, and are signed and dated by the auditor when completed. The audit checklist is based on EPA laboratory evaluation criteria, the DoD QSM Version 4, the provisions of the Laboratory Quality Assurance Manual, and the laboratory SOPs.

Each system audit is immediately followed by a debriefing in which the auditor discusses his/her findings with the laboratory representatives. The debriefing serves a two-fold purpose: (1) laboratory management is afforded an early summary of findings, which allows them to begin formulating corrective strategies; and (2) the auditor has a chance to test preliminary conclusions and to correct any misconceptions before drafting his/her report.

The records from these assessments will be included in the project file. An abbreviated summary of the audits, including the name of the laboratory, the project for which the audit was performed, and the overall rating of the laboratory (acceptable or unacceptable), will be submitted to procurement for tracking. If a laboratory is assessed unacceptable, corrective actions will be implemented.

Data Verification Tasks

Data quality assessment is performed by evaluating the results of data verification, data evaluation, and/or data validation to determine the usability of the data for the original project objectives. Data verification, data evaluation, and data validation are each separate levels of review that can be performed by themselves or in conjunction with each other. Each of these levels of review is defined in the subworksheets below with the requirements for this project. While it is possible to apply these levels of review to field data, they are almost always associated only with analytical data from laboratories for field analyses.

Initially, data are received at WESTON in both pdf (laboratory data package) and electronic data deliverable (EDD) formats, as discussed previously. Upon receipt of the laboratory deliverables, a data management staff member will verify that:

- Results were received for each requested analysis for each sample. If a result is missing, the staff member will determine whether the laboratory submitted a deficiency report that accounts for the missing data.

- The data deliverable will be inspected for completeness based on the requirements specified in this plan. Inspection will verify only that the report Worksheets are present, not that the data within the report Worksheets are complete.

- WESTON will perform data verification on every report submitted by a laboratory. Field results will be reviewed for completeness. In addition, once the EDD is verified, it will be loaded into the project's electronic database management program as "unvalidated"

for user access on the network. These analytical results will be considered preliminary until data validation is complete.

Electronic Data Verification

WESTON's standardized Electronic Data Management Program, developed by Geotech Computer Systems of Englewood, Colorado, is EnviroData. The EnviroData EDD is in an ASCII text file format, which can interface with Geographic Information System (GIS), allowing exportation of electronic deliverables in order to meet agency standard formats. All analytical results are required to be submitted in the WESTON format. See EDD specification in Figure 14-1. Additionally, as required by the National Guard Bureau (NGB), an Environmental Restoration Information System (ERIS) EDD format will be uploaded on a quarterly basis.

The EDDs will be compared to the pdf version of the laboratory data package by the WESTON Data Management Coordinator. WESTON will perform a cursory review of the electronic data results. If a discrepancy is identified, the laboratory will be requested to correct the error, or WESTON will use the result reported in the hard copy data by the laboratory.

Table 14-1 EDD Specification

Field Name Description:

Field Name	Data Type	Record Size	Description	Table
Site Name	Text	50	Site Name	Sites
Station Name	Text	25	Station identifier or name	Stations
Sample Date_D	Date/Time	8	Date sample was taken	Samples
Sample Matrix	Text	15	Sample matrix	Samples
Sample Top	Number(Sg)		Sample top	Samples
Sample Bottom	Number(Sg)		Sample Bottom	Samples
Depth Units	Text	15	Units for sample top and sample bottom	Samples
Duplicate Sample	Number(Int)		Duplicate samples	Samples
Field Sample ID	Text	20	Client assigned field sample ID	Samples
Lab Sample ID	Text	20	Lab sample ID	Samples
Alt Sample ID	Text	20	Alternate sample identification	Samples
Cooler ID	Text	20	Cooler ID number - for QA/QC	Samples
Sampler	Text	50	Name of person taking sample	Samples
Description	Text	50	Sample description	Samples
COC Number	Text	20	Chain-of-custody number	Samples
Delivery Group	Text	10	Sample delivery group	Samples
Filtered Sample	Text	20	Filter size	Samples
QC Sequence ID	Text	15	QC sequence identifier	Samples
QC Sample Code	Text	3	QC code for this sample	Samples
Task Number	Text	20	Task number under which sampling is done	Samples
Primary Sample	Text	20	Primary sample to which QC sample is tied	Samples
Sample Result	Text	255	Result of attempted sampling	Samples
Parameter Name	Text	60	Name of material analyzed for	Analyses
CAS Number	Text	20	CAS number of material analyzed for	Analyses
Alt Parameter Number	Text	20	Alternative number for parameter	Analyses
Superseded	Number(Int)		Analysis superseded by re-analysis?	Analyses
Analytic Method	Text	25	Method for performing analysis	Analyses

Sample Date_D – The date on which the sample was taken. Required.

Sample Matrix – The material that the sample is primarily composed of. Provide the full Sample Matrix name, such as "Soil". Required.

Sample Top and *Sample Bottom* – Soil sample depths or elevations, as instructed by the client. The fields should contain only numeric values. If these fields are not applicable (i.e., water samples) or are unknown to the laboratory, then they should be populated with zeros, for compatibility with ODBC databases. Required.

Table 14-1 EDD Specification (Continued)

Depth Units – Units for sample top and sample bottom. This is a coded field that is linked to the Reporting Units lookup table. If this information is unavailable to the lab, "Unknown" should be reported. These units can be entered into the import file by a Data Administrator. Required.

Duplicate Sample – This field is discussed previously. It should be a zero unless this is a duplicate sample. All analyses must have an entry for this field, with multiple QC samples entered as values incremented from one. Required.

Field Sample ID – The client-assigned field ID number for each sample. Optional.

Lab Sample ID – The sample identification number used internally by the laboratory. Optional.

Alt Sample ID – Another sample identification number if needed. Optional.

Cooler ID – Number to identify cooler in which primary samples and QC samples were shipped. Optional.

Sampler – Person taking the sample. Optional.

Description – Description of the sample, such as its condition. Optional.

COC Number – Chain-of-custody tracking number. Optional.

Delivery Group – Sample delivery group. This field is provided for use as a lab tracking field. It could be used to define a group of parameters. Optional.

Filtered Sample – Filter information at the sample level. Was the sample filtered, and if so, what size filter was used? It could also be used to identify whether the filtering occurred in the field or the lab. Entries are compared to the Filtered look-up table in the database. The lab can supply either the code or the Filter description, whichever is most consistent with their system (i.e., TOT vs. total), but must coordinate this with the client. Required.

QC Sequence ID – QC sequence identifier. This field is another lab tracking field, used to relate field samples to lab samples. Optional.

QC Sample Code – Code to identify QC samples. It ties to the **QC Codes** table, which contains codes for both the sample and analysis levels. The lab should supply the code if available, e.g., DUP for duplicate sample, or O for original sample. If this information is not available to the lab, enter "z" for Unknown. Required.

Task Number – The administrative task number under which sampling is done. Optional.

Primary Sample – Stores the Field Sample ID of the primary sample to which the QC sample is tied. This field is blank for original samples, may be blank for field QC samples that have been submitted blind to the lab. This number can be entered into the temporary import table by a Data Administrator. The import routine converts this to the sample number of the primary sample before storing it in the database. Optional.

Sample Result – The result of the sampling process, such as "successful", "dry", "no access". Its primary use is to indicate that obtaining a sample was attempted unsuccessfully. If not available from the lab, this field can be entered into the temporary import table by a Data Administrator. Optional. If a sample was attempted unsuccessfully, the sample fields should be filled in; however, all fields associated with analyses, including parameter name, CAS Number, and Alt Parameter Number, should be left blank. The system will then only attempt to import the sample information.

Parameter Name, CAS Number, Alt Parameter Number – Various combinations of these fields are used to identify the Parameter Name. Parameter Name should be always be provided. The system compares the Parameter name to the entries in the Parameters and Parameter Alias lookup tables. CAS Number and Alt Parameter Number are not required, but should be provided if possible to help ensure the correct parameter name assignment. If the Parameter Name does not match a lookup entry, the system compares either the CAS Number, or the Alt Parameter Number (frequently used for Storet codes), to Parameter table entries. Care should be taken that consistent numbers be provided. If Parameter Name is left blank, but a CAS Number or Alt Parameter Number is provided, the system assigns a parameter name from the lookup tables based on a number match. Using only numbers to designate the parameter is not recommended, since the program does not request confirmation of the parameter name that is assigned.

Superseded – This field is discussed above. It should be a zero unless the analysis is superseded by a later value in the same file, in which case the entry should be 1. This field is used in conjunction with the Value Code field, discussed later in this Worksheet. All analyses should have an entry. Required.

Analytic Method – Method used to perform the analysis. Optional

Table 14-1 EDD Specification (Continued)

Field Name	Data Type	Record Size6	Description	Table[9]
Value	Number(Sg)		Value measured during analysis	Analyses
Reporting Units	Text	15	Units of the analysis	Analyses
Flag Code	Text	4	Data qualifier	Analyses
Problem Code	Text	4	Problems encountered during analysis	Analyses
Validation Code	Text	4	Code from data validation	Analyses
Detected Result	Text	1	Was analyte detected	Analyses
Detect	Number(Sg)		Detection limit	Analyses
Limit Type	Text	4	Detection limit type	Analyses
Detect2	Number(Sg)		2nd detection limit	Analyses
LimitType2	Text	4	2nd detection limit type	Analyses
Error	Number(Sg)		Error range for this analysis	Analyses
Dilution Factor	Number(Sg)		Dilution factor	Analyses
Basis	Text	1	Analyzed wet or dry	Analyses
Filtered Analysis	Text	20	Filter/measure basis at analytical level	Analyses
Leach Method	Text	20	Leaching method	Analyses
Prep Method	Text	20	Lab preparation method	Analyses
Reportable Result	Text	1	Designates analysis as reportable result	Analyses
Anal Date_D	Date/Time	8	Date the analysis was performed	Analyses
Extract Date_D	Date/Time	8	Date the extraction was performed	Analyses
Lab Report Date_D	Date/Time	8	Lab analysis reporting date	Analyses
Lab	Text	10	Name of lab conducting analysis	Analyses
Lab Comments	Text	50	Lab comments about this analysis	Analyses
Analysis Lab ID	Text	20	Lab identification number for analysis	Analyses
Analytical Batch	Text	40	Lab batch ID number	Analyses
Value Code	Text	6	Differentiates between different results	Analyses
Run Code	Text	5	Run code for GC analyses	Analyses
QC Analysis Code	Text	3	QC code for this analysis	Analyses

Field Name Description:

Value – Measured result of the analysis. Optional, but should almost always be provided. For laboratory control spike and matrix spike samples, the results should be reported in percent recovery, with the units in %. Moisture content should be reported as a separate analytical record, with the units in %. They should be entered on a "by weight" basis, based on total weight.

Reporting Units – Units of the analysis. The entry provided should be the full abbreviation, such as "mg/L". Entries must match an entry in the Reporting Units lookup table in the database. Detection limits and radiologic error must be reported in the same units as the value. Required.

Table 14-1 EDD Specification (Continued)

Flag Code – One to four coded entries for the analytical flag describing the analysis. Each character in the field must match an entry in the Analytic Flags lookup table in the database. More than one flag can be entered. For example, if "b" (detected in blank) and "j" (estimated value) are both entered in the lookup table, then "bj" can be entered as an analytic flag (estimated value, detected in blank). If the analysis is considered a usable value, and would not otherwise have a flag, this field should contain the code for Detected Value (usually a "v"). If the flag is unknown, the field should contain a "z". Required.

Problem Code – Analytic problems are usually described in the narrative, and not included in the electronic format. If this field data is not provided, the field should contain a "z" for unknown. If the laboratory chooses to supply problems in the electronic file, then the codes must match entries in the Analytic Problems table. As with the Flag Code field, the entry can consist of from one to four approved codes. Required.

Validation Code – One to four flags associated with validation of analyses. The data validation organization usually provides this field, which can contain from one to four of these codes. Others should place a "z" for Unknown in this field. Required.

Detected Result – Supplied by the lab, this field should contain either "y" for yes, the analyte was detected, or "n" for no, the analyte was not detected. This field overlaps slightly with Flag Code. The purpose of this field is to separate the non-detect flag from other lab qualifiers, such as "j" or "b", for statistical, evaluation and validation purposes. Optional.

Detect – Detection limit for the analysis. Detection limits must be reported in the same units as the value. Optional.

Limit Type – Type of limit contained in the Detect field, such as "MDL", "PQL", "RL", etc. Optional.

Detect2 – A second detection limit. Standards should be set for which type of limit should be entered in each field for a given site, for example: IDL or MDL in the first column, CRDL or PQL in the second. Optional.

LimitType2 – Limit type for second detection limit. Optional.

Error – Standard error for radioactivity measurements. Optional.

Dilution Factor – Amount that the sample was diluted prior to analysis. Optional.

Basis – Analyzed wet or dry. Should be "w" for wet or "d" for dry. Can also report "n" for not applicable, or "z" for unknown. Required.

Filtered Analysis –Filter or measure basis information at the analysis level. Entries are compared to the Filtered look-up table in the database. As with the Filtered Sample field, the lab can supply either the code or the description for this field. Required.

Leach Method – Method used to leach sample. Entries are compared to the Leach Method lookup table to maintain consistency. Lab should supply the full name of the method. If the analysis was not leached, "None" should be reported. Required.

Prep Method – Method used to prepare sample separate from leaching. Optional.

Reportable Results – Flag for whether the result is to be used in reports. Report "Y" for yes, or "N" for no. Reported by labs or selected by Project Managers for multiple analyses from a selected sample, such as analyses at multiple dilutions. Optional.

Anal Date_D – Date on which the analysis was performed. Optional.

Extract Date_D – Date on which the material was extracted for analysis. Optional.

Lab Report Date_D – Date on which the lab reported the analysis. Optional.

Lab – Name of the laboratory performing the analysis. Optional.

Lab Comments – Lab comments about this analysis. Optional.

Analysis Lab ID – Lab identification number at the analysis level. LabSampleID tracks lab analyses at the sample level. This field is for identification numbers at the analysis level. Optional.

Analytical Batch – Lab batch identification number. Optional.

Value Code – Parameter value classification. This field identifies the analytical trial, and supplies the reason for a superseded analysis. It is a coded entry enforced by a lookup table. The lab should report the code, such as "RE" for re-extracted, "DL" for dilution, etc., or "O" for original analysis. Required.

Run Code – Confirmation run identification. This is a coded entry enforced by a lookup table. The lab should supply the code, such as "PR" for primary run, "n" for not applicable, or "z" for Unknown. Required.

QC Analysis Code – QC code at the analysis level. It ties to the **QC Codes** table, which contains codes for both the sample and analysis levels. The lab should supply the code for this field, such as "TIC" for tentatively identified compound, or "O" for original analysis. Required.

Data Evaluation

Data evaluation is performed to assess whether the quality control requirements for field duplicates, laboratory duplicates, field blanks, trip blanks, surrogates, matrix spikes, percent solids, laboratory blanks, and laboratory control samples were met.

Data evaluation will be performed on 100% of the laboratory deliverables generated during this program. In addition, some technical review will be performed by WESTON's Project Chemist.

Data Validation

Data validation is a systematic process to ensure that all chemical analytical information meet uniform requirements and to determine that the usability and defensibility of the data are adequate for their intended use. Analytical results will be independently evaluated by a third party; according to the appropriate agency data validation guidelines applicable for the site location (see Worksheet 36). In conjunction with the data validation guidelines, the project chemist will examine the project-specific Work Plan, the method-specific criteria, and the laboratory SOPs to determine the overall usability of the analytical results. All applicable analytical data packages will be validated to ensure compliance with specified analytical, QA/QC requirements, data reduction procedures, data reporting requirements and required accuracy, precision, and completeness criteria.

Data validation will be performed on 100% of the CLP-type data deliverables.

The CLP-type data packages will be validated at Manual Level M3 for organic compounds and Manual Level IM2 for inorganic compounds, following the most recently promulgated versions of the EPA Region III *Modifications to the National Functional Guidelines* for organic and inorganic data review, and the EPA Region III *Innovative Approaches to Data Validation.* Methods for which no data validation guidelines exist will be validated following the *National Functional Guidelines* deemed most appropriate by the data validator.

Upon completion, the data validator will provide a data validation report that is compliant with the guidelines established in the previously referenced documents. In addition, the validator will provided an annotated EDD that contains all data result qualifiers. These data qualifiers will then be uploaded into the project database, which will then be made accessible to the WESTON project team and will be available for upload to ERIS.

Worksheet 15 — Reference Limits and Evaluation Tables

Worksheet 15.1 — Reference Limits and Evaluation Table – Explosives Method SW-846 8330B (Soil/Sed)

Analyte	CAS Number	Achievable Laboratory Limits		Precision and Accuracy Method Performance Criteria[3]	
		Project RL[4]	MDL	LCS/MS/MSD Recovery Limits	LCS/MS/MSD Precision
		Soil[2]	Soil	Soil	
		(mg/kg)	(mg/kg)	%	RPD <
HMX	2691-41-0	0.10	0.0227	75-125	30
RDX	121-82-4	0.20	0.0430	70-135	30
1,3,5-Trinitrobenzene	99-35-4	0.10	0.0138	75-125	30
1,3-Dinitrobenzene	99-65-0	0.10	0.0166	80-125	30
Nitrobenzene	98-95-3	2.0	0.085	75-125	30
Tetryl	479-45-8	0.20	0.0439	10-150	30
2,4,6-Trinitrotoluene	118-96-7	0.10	0.0307	55-140	30
4-Amino-2,6-dinitrotoluene	1946-51-0	0.10	0.0299	80-125	30
2-Amino-4,6-dinitrotoluene	35572-78-2	0.10	0.0329	80-125	30
2,6-Dinitrotoluene	606-20-2	0.10	0.0191	80-120	30
2,4-Dinitrotoluene	121-14-2	0.10	0.0147	80-125	30
2-Nitrotoluene	88-72-2	0.20	0.0472	80-125	30
4-Nitrotoluene	99-99-0	0.20	0.0365	75-125	30
3-Nitrotoluene	99-08-1	0.20	0.0640	75-120	30
1, 2-Dinitrobenzene (surrogate)	100-25-4	NA	NA	83-122[1]	NA

[1] *Surrogate Control Limits.*
[2] *If % solids is <30%, additional sample needs to be analyzed to ensure the detection limits are met.*
[3] *The QA/QC criteria presented in this table reflect the most recently promulgated values as reported by the laboratory; therefore, they may differ from those values presented in the associated SOP found in Attachment A.*
[4] *MIS sampling will not be performed for this project; therefore the MIS special sample preparation will not be required at the laboratory.*

Worksheet 15.2 — Reference Limits and Evaluation Table – Metals Method SW-846 6010B (Soil/Sed)

		Achievable Laboratory Limits		Precision and Accuracy Method Performance Criteria[2]	
		Project RL	MDL	LCS/MS/MSD Recovery Limits	LCS/MS/MSD Precision
Analyte	CAS Number	Soil[1]	Soil	Soil	Soil
		(mg/kg)	(mg/kg)	%	RPD <
Aluminum	7440-36-0	50	1.55	80-120	20
Antimony	7440-36-0	2.0	0.38	80-120	20
Arsenic	7440-38-2	2.5	0.66	80-120	20
Barium	7440-39-3	2.0	0.076	80-120	20
Beryllium	7440-41-7	0.50	0.033	80-120	20
Cadmium	7440-43-9	0.50	0.041	80-120	20
Calcium	7440-70-2	100	14.1	80-120	20
Chromium	7440-47-3	3.5	0.058	80-120	20
Cobalt	7440-48-4	1.0	0.10	80-120	20
Copper	7440-50-8	5	0.217	80-120	20
Iron	7439-89-6	80	3.8	80-120	20
Lead	7439-92-1	0.9	0.27	80-120	20
Magnesium	7439-95-4	30	3.7	80-120	20
Manganese	7439-96-5	4.5	0.10	80-120	20
Mercury	7439-97-6	17	5.53	87-111	20
Nickel	7440-02-0	4.0	0.123	80-120	20
Potassium	7723-14-0	300	41	80-120	20
Selenium	7782-49-2	3.0	0.86	80-120	20
Silver	7440-21-3	1.5	0.16	75-120	20
Sodium	7440-22-4	500	59	80-120	20
Thallium	7440-28-0	3.0	0.65	80-120	20
Vanadium	7440-62-2	2.0	0.094	80-120	20
Zinc	7440-66-6	8.0	0.398	80-120	20

[1] *If % solids is <30%, additional sample needs to be analyzed to ensure the detection limits are met.*
[2] *The QA/QC criteria presented in this table reflect the most recently promulgated values as reported by the laboratory; therefore, they may differ from those values presented in the associated SOP found in Attachment A.*

Worksheet 16 — Project Schedule/Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Dates(s) of Initiation	Anticipated Date of Completion		
Draft RI WP	WESTON	09/28/09	10/23/09	Draft RI WP with Appendices	10/23/09
TPP Meeting 1 (Kickoff and CSM)	WESTON, USACE, EPA, NBG, PAARNG	11/19/09	11/19/09	Written Meeting Agenda Written Meeting Minutes	11/19/09
Army Review / Comments – WP	NGB, PAARNG	10/26/09	11/30/09	Written Review Comments	11/30/09
Draft Final RI WP	WESTON	12/23/09	12/31/09	Draft Final RI WP with Appendices	12/31/09
Stakeholder Review / Comment – WP	NGB, PAARNG, EPA	01/04/10	02/01/10	Written Review Comments	02/01/10
Final RI WP	WESTON	02/09/10	02/16/10	Final RI WP with Appendices	02/16/10
RI Fieldwork (MEC)	WESTON	03/25/10	05/10/10	Safety and field logs and forms, Photographic log, GPR data table, Daily reports, and Daily DQCP – to be included in the appendices to the RI Report	05/10/10
TPP Meeting 2 (WP and FW Approach)	WESTON, USACE, EPA, NBG, PAARNG	01/14/10	01/14/10	Written Meeting Agenda Written Meeting Minutes	01/14/10
Draft QAPP	WESTON	09/28/09	10/23/09	Draft QAPP with Appendices	10/23/09
Army Review of QAPP	NGB,	10/26/09	11/30/09	Written Review Comments	11/30/09

Worksheet 16 — Project Schedule/Timeline Table (Continued)

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Dates(s) of Initiation	Anticipated Date of Completion		
	PAARNG				
Response to Comments for QAPP	WESTON	12/01/09	12/22/09	Response to Comments	12/22/09
Draft Final QAPP	WESTON	12/23/09	12/31/09	Draft Final QAPP with Appendices	12/31/09
Stakeholder Review / Comment – QAPP	NGB, PAARNG, EPA	01/04/10	01/28/10	Review with Written Comments	01/28/10
Final QAPP	WESTON	03/09/10	03/15/10	Final QAPP with Appendices	03/15/10
RI Fieldwork (MC)	WESTON	04/22/10	05/07/10	MC Sampling Logs, Data Analysis to be included in RI Report	05/07/10
Draft RI Report	WESTON	05/27/10	08/19/10	Draft RI Report with Appendices	08/19/10
Army Review / Comments – RI Report	NGB, PAARNG	08/20/10	10/01/10	Written Review Comments	10/01/10
Draft Final RI Report	WESTON	10/19/10	11/01/10	Draft Final RI Report with Appendices	11/01/10
Stakeholder Review / Comment –RI Report	NGB, PAARNG, EPA	11/02/10	02/10/11	Written Review Comments	02/10/11
MRSPP	WESTON	09/28/09	11/13/10	MRSPP as Appendix to RI Report	11/13/10
Final RI Report	WESTON	02/18/11	03/04/11	Final RI Report with Appendices	03/04/11

Worksheet 17 — Sampling Design and Rationale

Soil/sediment sampling will be conducted to assess if MC has been released to soil in the vicinity of MEC. Discrete samples will be collected biased to the location beneath the item or to the closest possible location. Discrete sampling was selected rather than composite sampling because the most likely release mechanism for constituents was assessed to be leaking from cracked UXO/DMM or degradation of the metal casings directly to the ground surface rather than aerial dispersion and deposition of constituents from detonation. Discrete samples will be analyzed for explosives and target analyte list (TAL) metals. Composite sampling will be conducted for locations where munitions have been blown-in-place (BIP) in accordance with the 7-Wheel Sampling approach outlined in the Cold Regions Research Engineering Laboratory (CRREL) Special Report (SR) 96-15 (CRREL, 1996). Composite samples will be analyzed only for explosives.

To satisfy regulatory interest and support the screening of MC and the evaluation of risk, background sampling for metals will also be conducted. Thirteen discrete surface samples will be collected randomly distributed in areas not potentially affected by MEC/MC and analyzed for TAL metals.

Worksheet 18 — Sampling Locations and Methods/SOP Requirements Table

Sampling locations will coincide with the identification of MEC and munitions debris, as appropriate. Table 18-1 lists the number of environmental and background samples anticipated.

Table 18-1 Samples Expected

Sampling Location / ID Number	Matrix	Depth	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
FIGR01-SS01-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS02-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS03-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS04-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS05-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS06-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS07-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS08-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS09-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS10-00 FIGR01-SS10-02	Soil	0-6	TAL Metals	2	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS11-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS12-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS13-00	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	VSP (background)
FIGR01-SS14-01	Soil	0-6	TAL Metals	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS15-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS16-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS17-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS18-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS19-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)

Worksheet 18 — Sampling Locations and Methods/SOP Requirements Table (Continued)

Sampling Location / ID Number	Matrix	Depth	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
FIGR01-SS21-01 FIGR01-SS21-02	Soil	0-6	TAL Metals, Explosives	2	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS22-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS23-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS24-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS25-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS26-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS27-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS28-04	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS29-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS30-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS31-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS32-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS33-01	Soil	0-6	TAL Metals, Explosives	1	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS34-01 FIGR01-SS34-02	Soil	0-6	TAL Metals, Explosives	2	Worksheet 21, Table 21-1	Discrete (point source)
FIGR01-SS35-01	Soil	0-6	Explosives	TBD	Worksheet 21, Table 21-1	Composite[a] (point source)

[a] All composite samples will be obtained utilizing the 7-Sample Wheel Approach (see SOP SS-4).

Worksheet 19 — Analytical SOP Requirements Table

Matrix	**Analytical Group**	**Concentration Level**	**Analytical and Preparation Method / SOP Reference**	**Sample Size**	**Containers** (number, size, and type)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time** (preparation / analysis)
Soil / Sediment[a]	Explosives	Low	SW8330B (SOP A-1)	2 grams	250-mL amber glass	Cool 4±2°C	14 days to extract/40 days to analysis
Soil / Sediment[a]	Metals	Low	SW6010B (SOP A-2)	1.2 grams	500-mL glass	Cool 4±2°C	6 months
Soil / Sediment[a]	Mercury	Low	SW7470A/7471A (SOP A-4)	2 grams	Analyze from metals jar	Cool 4±2°C	28 days

[a] All sediment samples should have % solids ≥30%. If the % is <30%, additional sample needs to be collected and analyzed to ensure that detection limits are met.

Worksheet 20 — Field Quality Control Sample Summary Table

Matrix	Analytical Group	Analytical and Preparation Method/ SOP Reference	No. of Sampling Locations	No. of Field Duplicate Pairs	No. of MS	No. of Field Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Laboratory
Soil / Sediment	Explosives	SW8330B (SOP A-1)	Unknown	1 per 20 samples	1 per 20 samples	1 per day	1 per day	0	Unknown
Soil / Sediment	Metals	SW6010B (SOP A-2)	Unknown	1 per 20 samples	1 per 20 samples	1 per day	1 per day	0	Unknown
Soil / Sediment	Mercury	SW7470A/7470A (SOP A-4)	Unknown	1 per 20 samples	Per Field Team Submission	1 per day	1 per day	0	Unknown

Worksheet 21 — Project Sampling SOP References Table

The field sampling is being performed in accordance with WESTON SOPs provided in Appendix F of the Work Plan. Table 21-1 provides a list of applicable SOPs.

Table 21-1 List of Applicable SOPs

SOP NO.	TASK
GENERAL SOPs	
G-1	Field Documentation
G-3	Field Sample Numbering
G-4	Quality Assurance/Quality Control Sampling
G-6	Decontamination
G-7	Management of Investigation Derived Waste (IDW)
G-8	Sample Chain-of-Custody
G-9	Sample Packing and Shipping
G-10	Surveying
G-11	MEC Anomaly Avoidance
MEDIA-SPECIFIC SOPs	
Soil and Sediment	
SS-2	Sediment Sampling
SS-3	Soil Sampling
SS-4	Post-BIP Sampling

Worksheet 22 — Field Sampling Equipment Calibration, Maintenance, Testing, and Inspection Table

This worksheet is not applicable.

Worksheet 23 — Analytical SOP References Table

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
A-1	Nitroaromatc and Nitroamine Explosive Compounds by High Performance Liquid Chromatography (HPLC) {SW-846 8330A & 8330B]	Definitive	Explosives	HPLC	TestAmerica-Denver	N
A-2	ICP Analysis for Trace Elements by SW-846 Method 6010B	Definitive	Metals	ICP	TestAmerica-Denver	N
A-3	Mercury in Water by Cold Vapor Atomic Absorption (CVAA) [SW7470A]	Definitive	Mercury	Cold Vapor	TestAmerica-Denver	No
A-4	Mercury in Solids by Cold Vapor Atomic Absorption [SW7471A]	Definitive	Mercury	Cold Vapor	TestAmerica-Denver	No
A-5	QA/QC Requirements for Federal Programs	Definitive	Various	NA	TestAmerica-Denver	N

Worksheet 24 — Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
HPLC	SW-846 8330B	As needed	≤ 20% RSD Correlation Coefficient R ≥ 0.995	Instrument maintenance, standard, inspection, recalibration	Laboratory Analyst	SOP A-1
ICP	SW-846 6010B	As needed	≤ 20% RSD Correlation Coefficient R ≥ 0.995	Instrument maintenance, standard, inspection, recalibration	Laboratory Analyst	SOP A-2
Cold Vapor	SW-846 7470A/7471A	As needed	≤ 20% RSD Correlation Coefficient R ≥ 0.995	Instrument maintenance, standard, inspection, recalibration	Laboratory Analyst	SOPs A-3, A-4

Worksheet 25 — Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
HPLC	Replace septa, clean injection port, clip replace column	SW-846 8330B	Leak test, column and injection port inspection	Daily or as needed	Passing CCV	Perform maintenance, check standards, recalibrate	Laboratory Analyst	SOP A-1
ICP	Torch, nebulizer, spray chamber, auto sampler, pump tubing	SW-846 6010B	Check connections, flush lines, clean nebulizer	Daily or as needed	Passing calibration	Perform maintenance, check standards, recalibrate	Laboratory Analyst	SOP A-2
Cold Vapor	Pump tubing, absorption cell and lens cleaning	SW-846 7470A/7471A	Check connections, flush sample lines	Daily or as needed	Passing calibration	Perform maintenance, check standards, recalibrate	Laboratory Analyst	SOPs A-3, A-4

Worksheet 26 — Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): WESTON, West Chester, Pennsylvania
Sample Packaging (Personnel/Organization): WESTON, West Chester, Pennsylvania
Coordination of Shipment (Personnel/Organization): WESTON, West Chester, Pennsylvania
Type of Shipment/Carrier: Laboratory Courier/Federal Express –Priority Overnight
SAMPLE RECEIPT AND ANALYSIS, See Laboratory Quality Assurance Manual (QAM), Appendix A
Sample Receipt (Personnel/Organization): Receiving Supervisor, TestAmerica, Denver, CO
Sample Custody and Storage (Personnel/Organization): Sample Custodian, TestAmerica, Denver, CO
Sample Preparation (Personnel/Organization): Organic and/or Inorganic Prep Supervisor, TestAmerica, Denver, CO
Sample Determinative Analysis (Personnel/Organization): Organic and/or Inorganic Laboratory Analyst, TestAmerica, Denver, CO
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample analysis): 60 days
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 356 days
SAMPLE DISPOSAL
Personnel/Organization: TestAmerica, Denver, CO
Number of Days from Analysis: >60 days

Worksheet 27 – Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to the laboratory):

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a chain-of-custody record (Figure 27-1) will be completed for each sample shipment by the field team. The chain-of-custody, which may be more than one page long, will list each sample in a shipping container (cooler). The chain-of-custody will be placed in a resealable plastic bag and taped to the inside lid of the container. Each time the samples are transferred, the signatures of the persons relinquishing and receiving the samples, as well as the date and time of transfer, will be documented. The transfer from the field team to the shipper and from the shipper to the laboratory will be documented by the airbill instead of the chain-of-custody. The laboratory is required to maintain a copy of the chain-of-custody and airbill as part of the laboratory's project records.

Chain-of-custody seals (see Figure 27-2) are used to determine whether any tampering has occurred during transport of samples. These signed and dated seals will be fastened to the right and left sides of each shipping cooler by the person responsible for packaging for both on-site and off-site sample analyses. If the coolers are opened before receipt at the laboratory, the seals will not be intact.

WESTON expects to ship samples on the same day the samples are collected. When it is not possible to ship the samples on the day of collection, the field team will store the samples in refrigerators designated for sample storage at the site or in coolers. If the samples are stored in coolers and the sample preservation requirements include refrigeration, ice or the equivalent will be used to keep the samples cold. The coolers or refrigerators will be secured in either a locked room or compartment or otherwise sealed to prevent tampering until the samples are transferred to the shipping service. Specific details for field sample storage are discussed in Subsection 3.10.9.4 of the Work Plan.

Unless previous screening results, site knowledge, or other information indicate the samples are hazardous, all samples collected and shipped for analysis will be treated as environmental samples. Samples, whether classified as hazardous or as environmental samples, will be shipped in compliance with the applicable regulations. The United States Department of Transportation (DOT) and the International Air Transport Association (IATA) has established specific regulations governing the packaging of hazardous and environmental samples for shipment. These regulations include specifications for packing materials, shipping containers, and shipping labels. All samples will be shipped in accordance with these regulations based on the best available knowledge of the samples being collected. See Subsection 3.10.10 of the Work Plan.

Electronic Sample Tracking

The electronic sample tracking process is initiated with the receipt of the hard copy chain-of-custody and the associated sample attribute forms. The field sample coordinator is responsible for emailing these documents to WESTON's Project Chemist at the end of each sampling day. The receipt date is stamped on these documents and an analytical batch file is created for storage of all hard copy documentation related to the specific batch. WESTON's data management sample coordinator compares the chain-of-custody and the laboratory confirmation for discrepancies; any issues are documented and reconciled.

Sample Identification Procedures:

Samples collected at the site must be uniquely labeled. All samples will be identified with a label attached directly to the container (see Figure 27-3). Sample label information will be completed using waterproof black marker. The labels will contain the following information:

- Sample ID.
- Time and date of collection.
- Project Name.
- Analysis Requested.
- Preservative (if any).
- Sample source/location.
- Sampler's initials.

From a data management perspective, the key requirement for the field sample identifier is that it is a unique name. In addition, for sample tracking purposes, the identifier has implicit coding of sample information, including site, location ID, sample type, sample depth or date collected.

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal):

The designated sample custodian(s) and staff are responsible for samples received at the laboratory. In addition to receiving samples, the sample receipt staff is also responsible for documentation of sample receipt and storage before and after sample analysis. Summaries of the minimal laboratory receipt procedures are:

- Upon receipt, sign, date, and document the time of sample receipt on the airbills or other shipping manifests received from the couriers.

- Sign the chain-of-custody assuming custody of the samples. If a chain-of-custody is not received with a set of samples, the laboratory will immediately notify the WESTON Project Manager.

- Inspect the sample cooler for integrity and then document the following information:

  - Type of courier and whether the samples were shipped or hand delivered (copies of the airbills are maintained).

  - Availability and condition of custody information.
  - Sample temperature ambient or chilled.
  - Actual temperature of the temperature blank.
  - Presence of leaking or broken containers and indication of sample preservation.

- Verify the holding time is not exceeded. If a sample has exceeded holding time, then the WESTON Project Chemist will be notified.
- Match the sample container information (e.g., sample tag/label), chain-of-custody records, and all pertinent information associated with the sample. The sample custodian then verifies sample identity to ensure that all information is correct. Any inconsistencies are resolved with WESTON through the Laboratory Project Manager and corrective action measures are documented before sample analysis proceeds.

  Samples submitted to off-site laboratories will be stored at 4 to 6°C for a minimum of 60 days following the completion of analyses and/or issue of final reports. Sample extracts and metals digestates will be stored for a period of 1 year following submittal of final reports. Laboratories are also responsible for the proper management and disposal of all sample residuals and extracts, following all applicable federal, state, and local laws; rules; and regulations.

Figure 27-1 Example Chain-of-Custody Record

Chain of Custody Record

Restoring Resource Efficiency

Client _____ Contact Name _____
Site Name _____ Contact Phone No. _____
W.O. _____ Turn-around-Time _____
Laboratory _____ Sampler _____

Analysis Requested by Group by Container
(number listed for total containers per analysis group)

Preservative

Lab Batch Number:

Lab ID	Sample ID	Matrix QC			Total Num of Containers	Matrix	Date Collected	Sample Time	App.IX VOA	Total PCBs by Aroclors	Homologs	Congeners		Herbicide	Dioxin/Furan	Appx. IX Metals	CN	Sulfide	TOC	Grain Size	Method TO-4 (Air)				
		MS	MSD	DUP																					

Field Remarks/Comments

Lab Use Only

Temp of Cooler when Received, C°

1	2	3	4

COC Tape was present on outer package	Y	N
COC Tape was unbroken on outer package	Y	N
COC Tape was present on sample	Y	N
COC Tape was unbroken on sample	Y	N
Received in good condition	Y	N
Labels Indicate Properly Preserved	Y	N
Received within Holding Time	Y	N

Relinquished by	Received by	Date	Time	Relinquished by	Received by	Date	Time

Page 1 of 1

Figure 27-2
Chain-of-Custody Seal

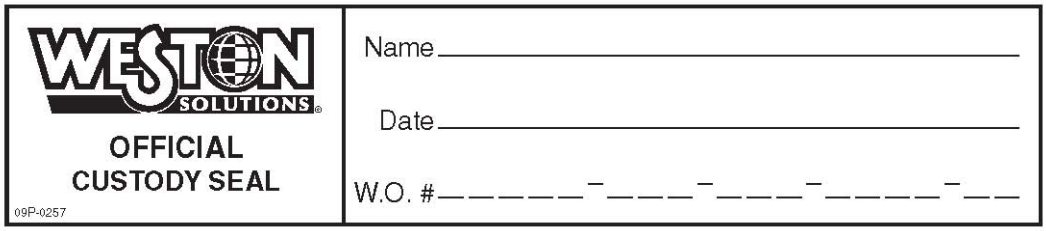

Figure 27-3
Jar/Bottle Label

PROJECT NAME	
SAMPLE ID	**SAMPLE DATE**
SAMPLED BY	**SAMPLE TIME**
PRESERVATIVE	___ **GRAB** ___ **COMPOSITE**
ANALYSIS REQUESTED	

Worksheet 28 – QC Samples Tables

Worksheet 28.1 – QC Samples Table – Explosives in Soil/Sediment

Matrix	Soil, Sediment
Analytical Group	Explosives
Concentration Level	Low
Sampling SOP	G-4
Analytical Method / SOP Reference	SW-846 8330B (SOP A-1)
Sampler's Name	WESTON – TBD
Field Sampling Organization	WESTON
Analytical Organization	TestAmerica, Denver, CO
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	As needed (see CCV passing criteria below and SW-846 8000 method)	%RSD <20%, or Correlation coefficient R≥0.99	If the acceptance criteria were not met, re-calibration is performed before any samples may be analyzed.	Analyst	Laboratory Accuracy	%RSD <20%, or Correlation coefficient R≥0.995
Initial calibration verification (ICV)	1 per ICAL, analyzed after ICAL, before field samples	%D ≤15%	If the acceptance criteria were not met, re-calibration is performed before any samples may be analyzed.	Analyst	Laboratory Accuracy	%D ≤15%
Continuing Calibration Verification (CCV)	Opening CCV, then every 10 samples, with closing CCV	%D ≤15%	If the criterion has not achieved corrective action, re-calibration is performed before any samples may be analyzed. Corrective action may include reanalysis of the samples.	Analyst	Laboratory Accuracy	%D ≤15%

Worksheet 28.1 – QC Samples Table – Explosives in Soil/Sediment (Continued)

Method Blank (MB)	1 per extraction batch	<1/2 RL	The source of the contamination is investigated and eliminated before proceeding with further analysis. Corrective actions are: 1. Samples ND – report without qualification 2. Samples >10X contamination level – report with qualification 3. Samples <10x contamination – re-extract and reanalyze. Insufficient sample -qualify and footnote	Analyst/Prep analyst	Absence of interference/ contamination	<1/2 RL
Laboratory Control Sample (LCS)	1 per extraction batch	%Recovery = (Calculated Value/True Value) *100%[1]	Source of poor recovery is investigated and eliminated before proceeding with further analysis, corrective actions are: 1. Biased high, samples ND – report without qualifications. 2. Biased low – re-extract and reanalyze.	Analyst/Prep analyst	Laboratory Accuracy/Method bias in ideal matrix	%Recovery = (Calculated Value/True Value) *100%
Field Duplicate	1 per 20 field samples	All Target Compounds RPD ≤50% (soil/sediment)	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL).	Field Personnel/ WESTON Chemist	Sampling Precision	All Target Compounds RPD ≤50% (soil/sediment)

Worksheet 28.1 – QC Samples Table – Explosives in Soil/Sediment (Continued)

Matrix Spike (MS)	1 per 20 samples or one for each extraction batch	%Recovery = (Calculated Value - Sample Value/True Value) *100%[1]	If the recoveries indicate that the problem is procedure related, re-extraction and reanalysis is required. If the recoveries indicate that the failures are matrix-related, refer to Blank Spike as measure of method performance in clean matrix. The WESTON Project Chemist will be contacted and a decision will be made to either report the data as is with a notation in the analytical narrative or if the samples should be re-extracted and reanalyzed.	Analyst/Prep analyst	Precision and Accuracy in field samples	%Recovery = (Calculated Value - Sample Value/True Value) *100%
Matrix Spike Duplicates (MSD)	1 per 20 samples or one for each extraction batch	%Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2[1]	See above	Analyst/Prep analyst	Precision and Accuracy in field samples	%Recovery = (Calculated Value – Sample Value/True Value) *100%RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2

Worksheet 28.1 – QC Samples Table – Explosives in Soil/Sediment (Continued)

Surrogate Spikes	Every sample	%Recovery = (Calculated Value/True Value) *100%[1]	Reason for poor recoveries is investigated and eliminated before further analytical activities. Corrective actions are: 1. High bias, samples ND – report without qualification. 2. Low bias – re-extract and reanalyze. Insufficient volume – qualify and footnote	Analyst/Prep analyst	Individual sample preparation efficiency control	%Recovery = (Calculated Value/True Value) *100%
Cooler Temperature Blank	One per cooler	4±2°C	Notify WESTON Project Chemist. WESTON will evaluate effect on samples and indicate to laboratory whether to proceed with analysis. Resampling may be required.	Sample Custodian/WESTON Project Chemist	Accuracy in field samples	4±2°C

[1] *Acceptance criteria for surrogates, LCSs, MSs, and MSDs are included under the appropriate method in Worksheet 15.*

Worksheet 28.2 – QC Samples Table – Metals in Soil/Sediment

Matrix	Soil, Sediment,
Analytical Group	Metals
Concentration Level	Low
Sampling SOP	G-4
Analytical Method / SOP Reference	SW-846 6010B (SOPs A-2)
Sampler's Name	WESTON – TBD
Field Sampling Organization	WESTON
Analytical Organization	TestAmerica, Denver, CO
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL	If the criterion is not met for the blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Data qualifiers will be applied as appropriate according to EPA Region III guidelines.	Field Personnel/ WESTON Chemist	Field Accuracy/Bias Contamination and Representativeness	All Target Compounds <1/2 RL

Worksheet 28.2 – QC Samples Table – Metals in Soil/Sediment (Continued)

Equipment Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL	If the criterion is not met for the blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Data qualifiers will be applied as appropriate according to EPA Region III guidelines.	Field Personnel/ WESTON Chemist	Field Accuracy/Bias Contamination and Representativeness	All Target Compounds <1/2 RL
Field Duplicate	1 per 20 field samples	All Target Compounds RPD ≤50% (soil/sediment)	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is nondetect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL).	Field Personnel/ WESTON Chemist	Sampling Precision	All Target Compounds RPD ≤50% (soil/sediment)
Initial Calibration (ICAL)	As needed (see CCV passing criteria below)	%RSD <5%, or Correlation coefficient R>0.995	If the acceptance criteria were not met, re-calibration is performed before any samples may be analyzed.	Analyst	Laboratory Accuracy	%RSD <5%, or Correlation coefficient R>0.995
Initial calibration verification (ICV)	1 per ICAL, analyzed after ICAL, before field samples	%D ≤10%	If the acceptance criteria were not met, re-calibration is performed before any samples may be analyzed.	Analyst	Laboratory Accuracy	%D <10%

Worksheet 28.2 – QC Samples Table – Metals in Soil/Sediment (Continued)

Continuing Calibration Verification (CCV)	Opening CCV, then every 10 samples, with closing CCV	%D ≤10%	If the criterion has not achieved corrective action, re-calibration is performed before any samples may be analyzed. Corrective action may include reanalysis of the samples.	Analyst	Laboratory Accuracy	%D <10%
Method Blank (MB)	1 per extraction batch	<1/2 RL	The source of the contamination is investigated and eliminated before proceeding with further analysis. Corrective actions are: 1. Samples ND – report without qualification 2. Samples >10X contamination level – report with qualification 3. Samples <10x contamination – re-extract and reanalyze. Insufficient sample -qualify and footnote	Analyst/Prep analyst	Absence of interference/ contamination	<1/2 RL
Laboratory Control Sample (LCS)	1 per extraction batch	%Recovery = (Calculated Value/True Value) *100%; 80%<%Recovery≤120%[1]	Source of poor recovery is investigated and eliminated before proceeding with further analysis, corrective actions are: 1. Biased high, samples ND – report without qualifications. 2. Biased low – re-extract and reanalyze. Insufficient volume – qualify and footnote	Analyst/Prep analyst	Laboratory Accuracy/Method bias in ideal matrix	%Recovery = (Calculated Value/True Value) *100%; 80%<%Recovery≤120%

Worksheet 28.2 – QC Samples Table – Metals in Soil/Sediment (Continued)

Matrix Spike (MS)	1 per 20 samples or one for each extraction batch	%Recovery = (Calculated Value - Sample Value/True Value) *100%: 80%≤%Recovery≤120%[1]	If the recoveries indicate that the problem is procedure related, re-extraction and reanalysis is required. If the recoveries indicate that the failures are matrix-related, refer to Blank Spike as measure of method performance in clean matrix. The WESTON Project Chemist will be contacted and a decision will be made to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and reanalyzed.	Analyst/Prep analyst	Precision and Accuracy in field samples	%Recovery = (Calculated Value -Sample Value/True Value) *100%: 80%≤%Recovery≤120%
Matrix Duplicates (MD)	1 per 20 samples or one for each extraction batch	All Target Compounds RPD <20.	If the criterion is not met for the lab dup, the sample set should be reanalyzed. The analytical QC results should be evaluated and entire batch re-digested if necessary.	Analyst/Prep analyst	Precision in field samples	All Target Compounds RPD <20.
Cooler Temperature Blank	One per cooler	4±2°C	Notify WESTON Project Chemist. WESTON will evaluate effect on samples and indicate to laboratory whether to proceed with analysis. Resampling may be required.	Sample Custodian/ WESTON Project Chemist	Accuracy in field samples	4±2°C

[1] *Acceptance criteria for LCSs, MSs, and MSDs are included under the appropriate method in Worksheet 15.*

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Worksheet 28.3 – QC Samples Table – Mercury in Soil/Sediment

Matrix	Soil, Sediment,
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	G-4
Analytical Method / SOP Reference	SW-846 7470, 7471A (SOPs A-13, A-24, A-25)
Sampler's Name	WESTON – TBD
Field Sampling Organization	WESTON
Analytical Organization	TestAmerica, Denver, CO
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	As needed (see CCV passing criteria)	Correlation coefficient R≥0.995	If the acceptance criteria were not met, re-calibration is performed before any samples may be analyzed.	Analyst	Laboratory Accuracy	Correlation coefficient R≥0.995
Initial calibration verification (ICV)	1 per ICAL, analyzed after ICAL, before field samples	%D ≤10%	If the acceptance criteria were not met, re-calibration is performed before any samples may be analyzed.	Analyst	Laboratory Accuracy	%D <≤10%
Continuing Calibration Verification (CCV)	Opening CCV, then every 10 samples, with closing CCV	%D ≤20%	If the criterion has not achieved corrective action, re-calibration is performed before any samples may be analyzed. Corrective action may include reanalysis of the samples.	Analyst	Laboratory Accuracy	%D ≤20%
Method Blank (MB)	1 per extraction batch	<1/2 RL	The source of the contamination is investigated and eliminated before proceeding with further analysis. Corrective actions are:	Analyst/Prep analyst	Absence of interference/ contamination	<1/2 RL

Worksheet 28.3 – QC Samples Table – Mercury in Soil/Sediment (Continued)

			1. Samples ND – report without qualification 2. Samples >10X contamination level – report with qualification 3. Samples <10x contamination – re-extract and reanalyze. Insufficient sample -qualify and footnote			
Laboratory Control Sample (LCS)	1 per extraction batch	%Recovery = (Calculated Value/True Value) *100%; 80%≤%Recovery≤120 %[1]	Source of poor recovery is investigated and eliminated before proceeding with further analysis, corrective actions are: 1. Biased high, samples ND – report without qualifications. 2. Biased low – re-extract and reanalyze. Insufficient volume – qualify and footnote	Analyst/Prep analyst	Laboratory Accuracy/Method bias in ideal matrix	%Recovery = (Calculated Value/True Value) *100%; 80%≤%Recovery≤120%
Field Duplicate	1 per 20 field samples	All Target Compounds RPD ≤50% (soil/sediment)	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and usefulness of the data. If one of the duplicate pair is detected above the RL and the remaining pair is nondetect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL).	Field Personnel/ WESTON Chemist	Sampling Precision	All Target Compounds RPD ≤50% (soil/sediment)

Worksheet 28.3 – QC Samples Table – Mercury in Soil/Sediment (Continued)

Matrix Spike (MS)	1 per 20 samples or one for each extraction batch	%Recovery = (Calculated Value - Sample Value/True Value) *100%: 80%<%Recovery<120 %[1]	If the recoveries indicate that the problem is procedure related, re-extraction and reanalysis is required. If the recoveries indicate that the failures are matrix-related, refer to Blank Spike as measure of method performance in clean matrix. The WESTON Project Chemist will be contacted and a decision will be made to either report the data as is with notation in the analytical narrative or if the samples should be re-extract and reanalyzed.	Analyst/Prep analyst	Precision and Accuracy in field samples	%Recovery = (Calculated Value – Sample Value/True Value) *100%: 80%<%Recovery<120%
Matrix Spike Duplicates (MSD)	1 per 20 samples or one for each extraction batch	%Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2: %RPD ≤ 20%[1]	See above	Analyst/Prep analyst	Precision and Accuracy in field samples	%Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is average value of concentrations in MS and MSD, (XA + XB)/2: %RPD ≤ 20%
Cooler Temperature Blank	One per cooler	4±2°C	Notify WESTON Project Chemist. WESTON will evaluate effect on samples and indicate to laboratory whether to proceed with analysis. Resampling may be required.	Sample Custodian/ WESTON Project Chemist	Accuracy in field samples	4±2°C

[1] *Acceptance criteria for LCSs, MSs, and MSDs are included under the appropriate method in Worksheet 15.*

Worksheet 29 — Project Documents and Records Table

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records
▪ Field Notebooks ▪ DQCR ▪ Site Maps ▪ Chain-of-Custody Records ▪ Custody Seals ▪ Air Bills	▪ Daily observations and notes, personnel on site, samples collected, date, time, communications, tailgate safety meeting items, unusual incidents/events, etc. ▪ Documenting sample points, notations of true site conditions ▪ Soil lithology, sample depth, sample numbers, nos. of containers, requested analyses, preservation. ▪ Field surveys	▪ Chain-of-Custody Forms ▪ Sample Receipt, Sample Condition, Custody, and Internal Tracking Records ▪ Laboratory Information Management System (LIMS) login ▪ Run logs – sample chronology ▪ Standard traceability logs ▪ Calibration logs ▪ Non-conformance records ▪ Communications logbooks ▪ QC Sample identification (blanks, replicates, duplicates, LCS, MS/MSD) ▪ Laboratory data qualifiers ▪ Instrument calibration logs ▪ Instrument maintenance logs ▪ Electronic data deliverables ▪ Case narrative ▪ Laboratory sample identification ▪ Reporting forms ▪ Quality assurance/quality control forms ▪ MDL/RL Studies ▪ Laboratory Accreditation Certificates ▪ Quality Assurance Manual ▪ Analytical SOPs ▪ Sample disposal records	▪ Quality Assurance Manual ▪ Laboratory Accreditation Certificates ▪ Communication logbooks ▪ EDDs with site-specific goals evaluation ▪ PDF of Final Laboratory Technical Report ▪ Weekly health and safety communications ▪ Safety audit checklists ▪ Validation reports on applicable samples

Worksheet 30 — Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ ID Number	Analytical SOP+	Data Package Turnaround Time	Laboratory/Organization (name and address, contact person and telephone number)	Backup Laboratory/ Organization (name and address, contact person and telephone number)
Soil, Sediment	Explosives 8330B	Low	TBD	SW8330B SOP A-2	Level IV 21 calendar days	TestAmerica Laboratories, Inc. 301 Alpha Drive RIDC Park Denver, CO 15238 (office) 412-963-7058 (fax) 412-963-2468	TestAmerica National Network
	ICP Metals 6010B	Low		SW6010B SOP A-4			
	Mercury 7471A, 7470A	Low		SW7470A/7471A SOPs A-3, A-4			

+ See Worksheet 19 for complete list of applicable methods for preparation, cleanup and analysis.

Worksheet 31 — Planned Project Assessments Table

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Review of QAPP, SOPs and DCQR with Field Staff	1/prior to sampling start up	Internal	WESTON	John Gerhard Technical Manager WESTON	John Gerhard Technical Manager WESTON	John Gerhard Technical Manager WESTON	John Gerhard Technical Manager WESTON
Daily Logbook and Field Forms	Daily	Internal	WESTON	Chris Hikel Project Engineer WESTON	John Gerhard Technical Manager WESTON	John Gerhard Technical Manager WESTON	John Gerhard Technical Manager WESTON
Laboratory Assessment for appropriate Certifications, Capacity and QAPP Review with Staff	1/prior to sampling start up	Internal	WESTON	Kelly Spittler Project Chemist WESTON	Robert Hanisch Laboratory Director TestAmerica Elaine Walker Project Manager TestAmerica	Robert Hanisch Laboratory Director TestAmerica Elaine Walker Project Manager TestAmerica	Kelly Spittler Project Chemist
Daily Tailgate Safety Meeting	Daily	Internal	WESTON	Chris Hikel Project Engineer, or Field Geologist WESTON	John Gerhard Technical Manager WESTON	John Gerhard Technical Manager WESTON	John Gerhard Technical Manager WESTON
Field Sampling and COC Review Against QAPP Requirements	Daily	Internal	WESTON	Kelly Spittler Project Chemist WESTON	John Gerhard Technical Manager & Field Geologist/ Environmental Scientist WESTON	John Gerhard Technical Manager & Field Geologist/ Environmental Scientist	Kelly Spittler Project Chemist WESTON
Laboratory Report Deliverables and Analytical Results Against QAPP Requirements	Per Sample Delivery Group	Internal	WESTON	Kelly Spittler Project Chemist WESTON	Robert Hanisch Laboratory Director TestAmerica Elaine Walker Project Manager TestAmerica	Robert Hanisch Laboratory Director TestAmerica Elaine Walker Project Manager TestAmerica	Kelly Spittler Project Chemist WESTON
Validation	Per Sample Delivery Group	Internal	MCGI	Sherif Mina Data Validator MCGI	Karen Kuoppala QA Manager TestAmerica	Karen Kuoppala QA Manager TestAmerica	Sherif Mina Data Validator MCGI

Worksheet 32 — Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Review of QAPP with Field Staff	Contained with written report Daily QC Report for that day.	Gregory Daloisio Project Manager, WESTON John Gerhard Technical Manager, WESTON	Immediately not to exceed with 24 hours.	Daily QC Report would be amended with corrective action.	Kelly Spittler Project Chemist WESTON	Immediate within 24 hours.
Laboratory Assessment for appropriate Certifications, Capacity and QAPP Review with Staff	Receipt of copies of certifications. Email traffic concerning lab capacity prior to sampling start-up. QAPP Sign-off sheet received from laboratory.	Gregory Daloisio Project Manager, WESTON	Immediate.	Response to email.	Gregory Daloisio Program Manager, WESTON John Gerhard Technical Manager, WESTON	48 hours after notification.
Daily Safety Meeting	Verbal debriefing and daily sign off log. If a safety violation occurs, a Supervisor Injury Employee Report is completed.	Gregory Daloisio Project Manager, WESTON	Immediately not to exceed 24 hours.	Included as part of the process of the Supervisor Injury Employee Report.	Gregory Daloisio Project Manager, WESTON	Immediate within 24 hours.
Daily Field Reporting and Field Forms	Contained with written report.	John Gerhard Technical Manager, WESTON	Immediately not to exceed 24 hours.	Daily QC Report would be amended with corrective action.	John Gerhard Technical Manager, WESTON	Immediate within 24 hours.
Field Sampling and COC Review Against QAPP Requirements	Communication may be in the form of email traffic	Gregory Daloisio Project Manager, WESTON John Gerhard Technical Manager, WESTON	24 hours after sampling.	Response to email.	Gregory Daloisio Project Manager, WESTON John Gerhard Technical Manager, WESTON	48 hours after notification.

Worksheet 32 — Assessment Findings and Corrective Action Responses (Continued)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Laboratory Report Deliverables and Analytical Results Against QAPP Requirements	Communication may be in the form of email traffic	Gregory Daloisio Project Manager, WESTON John Gerhard Technical Manager, WESTON	24 hours after completion of analytical.	If required laboratory reports will be amended and corrections noted in the analytical narrative.	Gregory Daloisio Project Manager, WESTON John Gerhard Technical Manager, WESTON	72 hours after notification.
Data Verification	Communication may be in the form of email traffic requesting additional laboratory forms, back up data that may be missing and or clarification of the analytical report.	Karen Kuoppala QA Manager, TestAmerica	24 hours after finding deficiency.	If required laboratory reports will be amended and corrections noted in the analytical narrative and contained with the validation report.	Kelly Spittler Project Chemist, WESTON	Up to 7 days.
Validation	Communication may be in the form of Email traffic requesting additional laboratory forms, back up data that may be missing and or clarification of the analytical report.	Elaine Walker Project Manager, TestAmerica	24 hours after finding deficiency.	If required laboratory reports will be amended and corrections noted in the analytical narrative and contained with the validation report.	Sherif Mina Data Validator, MCGI	Up to 7 days.

Worksheet 33 — QA Management Reports Table

Type of Report	**Frequency** (daily, weekly monthly, quarterly, annually, etc.)	**Projected Delivery Date(s)**	**Person(s) Responsible for Report Preparation** (title and organizational affiliation)	**Report Recipient(s)** (title and organizational affiliation)
Progress Reports	Monthly Progress Reports	Monthly after project start up	John Gerhard Technical Manager WESTON	Elaine Walker Project Manager, TestAmerica
Validation Report	For each round of soil/sediment or other media sampling	30 days after completion of analytical data	Sherif Mina – Data Validator, MCGI	Kelly Spittler Project Chemist WESTON
Final Report	Completed as Draft, Draft Final, and Final RI Report		Gregory Daloisio Project Manager, WESTON John Gerhard Technical Manager, WESTON	Elaine Walker Project Manager, TestAmerica

Worksheet 34 – Sampling and Analysis Verification (Step I) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Field Staff Training	Personnel assigned to the project, including field personnel and subcontractors, will be qualified to perform the tasks to which they are assigned. Field personnel will have basic field investigation knowledge for multimedia sampling. This includes but is not limited to basic sampling techniques, field testing methodology, monitoring wells installation, task-specific sampling methods, decontamination of field sampling equipment, maintenance of environmental paperwork, and how to avoid cross contamination. In addition to education and experience, specific training may be required to qualify individuals to perform certain activities. Training will be documented appropriately and the forms placed in the project file as a record. Project personnel will receive an orientation to the Work Plan and the Accident Prevention Plan (APP) as appropriate to their responsibilities before participation in project activities. Training of field personnel will be provided by the Site Supervisor, the QA Officer, or by a qualified designee.	Internal	Gregory Daloisio, WESTON John Gerhard, WESTON Kelly Spittler, WESTON
QAPP	A copy of the reviewed and approved version of the QAPP will be distributed to the laboratory and be available for review for all WESTON/personnel involved in this project. It is the responsibility of the WESTON Project Chemist to ensure delivery of a copy of QAPP to the laboratory. The laboratory QA manager is responsible for review of QAPP with laboratory staff. The WESTON project manager and Technical Manager will be responsible for ensuring that all staff has reviewed the final QAPP.	Internal / External	Gregory Daloisio, WESTON John Gerhard, WESTON Kelly Spittler, WESTON Elaine Walker, TestAmerica, Denver, CO Karen Kuoppala, TestAmerica, Denver, CO
Laboratory Quality Assurance Manual	TestAmerica has a detailed Quality Manual, Rev. 1, dated 06/19/2009, that is designed to meet the quality program requirements of NELAC and ISO Guide 25. This Quality Manual is included in Attachment B. Columbia Analytical and TestAmerica are both NELAC certified (ELAP or Navy Certifications will be applicable as of October 2009).	Internal / External	Kelly Spittler, WESTON Robert Hanisch, TestAmerica, Denver, CO Elaine Walker, TestAmerica, Denver, CO

Worksheet 34 – Sampling and Analysis Verification (Step I) Process Table (Continued)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Laboratory Staff Training	Laboratory senior management staff retains oversight responsibility for the data integrity program and retains the ultimate responsibility for execution of the data integrity program elements. Senior laboratory management staff is responsible for providing the resources required to conduct SOPs, ethics training, and operate data integrity evaluation procedures. Laboratory employees receive technical ethics training during new employee orientation. All employees are required to attend ethics refresher training and to sign an ethical conduct agreement annually, which verifies their understanding of the laboratories ethics policy and the analyst's ethical responsibilities. Training on data integrity procedures and SOPs are conducted by the individual departments' group leaders within the laboratory. All records of training are retained at the laboratory in the individual staff training folders and are maintained by the laboratory quality assurance officer. All information related to staff qualifications, experience, external training courses, and education are placed into the individuals training file. Verification documentation for laboratory orientation, health and safety, and quality assurance training is also maintained with the training file. Additional training documentation is added to the files as it occurs. This includes data for initial and continuing demonstrations of proficiency, performance evaluations, study data and notes, and attendance lists from individual and group training sessions.	Internal	Organic and Inorganic Worksheet Managers* Kenneth Grzybowski, TestAmerica, Denver, CO
Laboratory Certifications	TestAmerica, Columbia and TestAmerica have current National Environmental Laboratories Accreditation Conference NELAC (ELAP or Navy Certifications will be applicable as of October 2009).	Internal / External	Robert Hanisch, TestAmerica, Denver, CO Kelly Spittler, WESTON
Field Logbooks	The sample number will be traceable to the site, location, and depth (where applicable). The sample identification and description will be recorded by the Task Order Manager or representative in the sample collection logs. Task Order Manager will perform daily reviews of field log books each day of sampling.	Internal	John Gerhard, WESTON
Sample Location Verification	The Task Order Manager will verify that the sample technicians have collected the samples from the proper locations and depths as described in Worksheet 18.	Internal	John Gerhard, WESTON

Worksheet 34 – Sampling and Analysis Verification (Step I) Process Table (Continued)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody – Field Level	WESTON sample coordinator will generate COCs forms prior to field sampling in accordance to the sample matrices and analytical tests required as described in Worksheet 19. Upon, completion of the COCs forms by the field technicians and prior to placement in the cooler the Task Order Manager will review the COCs against the field logbooks, Worksheet 18 and Worksheet 19 to insure that the samples, sample volumes, and sample nomenclature match the COC forms and the required analytical tests have been notated. A review of the COC form for completeness will also be conducted.	Internal	John Gerhard, WESTON
Chain-of-Custody – WESTON Project Chemist	Upon, completion of the COC the field technician will either fax or email the completed COC form to the WESTON Project Chemist. A review of the COC form against Worksheet 18 and Worksheet 19 will be conducted to ensure proper analytical test	Internal	Kelly Spittler, WESTON
Chain-of-Custody – Analytical Laboratory	All samples to be analyzed by the fixed-base laboratory will be shipped via overnight courier service. Upon receipt, a representative of the laboratory shall check the integrity of the custody seals, then locate, sign, and date the COC. The laboratory is responsible for verifying that the COC and containers are in agreement. The COC, a Cooler Receipt Form, and information regarding any discrepancies between the COC and bottle labels will be faxed to the Project Chemist prior to preparation for analysis. The Laboratory Information Management System will provide evidence of sample custody from receipt by the laboratory until appropriate disposal.	Internal	TestAmerica Sample Management Technicians*
LIMs Login – Analytical Laboratory	A review of the COC form against the laboratory LIMs login and the project analytical requirement as contained within Worksheet 19 will be conducted to ensure proper analytical tests have been assigned and a review of the login for correctness will be conducted.	Internal	Elaine Walker, TestAmerica, Denver, CO
LIMs Login –WESTON Project Chemist	A secondary review of the COC form against the laboratory LIMs login and the project analytical requirement as contained within Worksheet 19 will be conducted to ensure proper analytical tests have been assigned and a review of the login for correctness will be conducted.	External	Kelly Spittler, WESTON
Sample Receipt Form – WESTON Project Chemist	TestAmerica will provide within 48 hours of receipt of samples a copy of the sample receipt form, any discrepancies between the COC and the sample containers will be noted and contained as part of the analytical record.	External	Kelly Spittler, WESTON

Worksheet 34 – Sampling and Analysis Verification (Step I) Process Table (Continued)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Laboratory Corrective Action and Report Procedure	Routine corrective action is defined as procedures used to return out of control analytical systems back to control. This level of corrective action applies to all analytical quality control parameters and analytical system specification as defined in the laboratory SOPs. Bench analysts have full responsibility and authority for performing routine corrective action. Routine corrective actions are documented as part of the analytical record. Defective processes, holding time violations, systematic errors and quality defects that occur are to be reported by the bench chemist immediately to the Worksheet supervisor and a non-conformance record initiated. The Worksheet supervisor will notify the designated Laboratory Project Manager (Elaine Walker) who will then notify the WESTON Project Chemist (Kelly Spittler). All notifications must be made in a timely manner. The non-conformance record should become part of the analytical record.	Internal / External	Elaine Walker, TestAmerica, Denver, CO Kelly Spittler, WESTON
Analytical Data Package – Laboratory	All data produced by the laboratory will be required to undergo several levels of review, which will include two levels of management review at the laboratory. The laboratory will review the data packages internally for completeness and verify that all of the required forms and raw data are included for each data package type. Random data packages may be chosen by the TestAmerica, QA Officer for additional audits.	Internal	Elaine Walker, TestAmerica, Denver, CO Robert Hanisch, TestAmerica, Denver, CO
Analytical Data Package / Laboratory Quality Control – WESTON Project Chemist	The WESTON Project Chemist will verify that data has been received for all samples that have been sent to the laboratory. An evaluation of this data will be performed to determine whether the laboratory met the QC requirements for the analytical as stated in the analytical methods and laboratory SOPs. Refer to Worksheets 19 and 28.	External	Kelly Spittler, WESTON
Laboratory Electronic Data Deliverables	The laboratory will provide an electronic data deliverables in ASCII text format that has been generated by the laboratory LIMs system. The WESTON Project Chemist will review these files for correctness and completeness. Project specific action goals as defined in Worksheet 15 will be added and evaluated. Any quality control issues that may impact the data use will be evaluated. The project manager and site manager will be notified immediately of any samples that exceed the project action goals.	External	Kelly Spittler, WESTON

* Refer to the Laboratory QAM in Attachment B.

Worksheet 35 — Sampling and Analysis Validation (Steps IIa and IIb) Process Table

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field Sampling	Ensure that all sampling protocols were followed according to the SOPs attached.	John Gerhard, WESTON
IIa	Analytical SOPs	Ensure that all laboratory analytical SOPs were followed.	Robert Hanisch, TestAmerica, Denver, CO
IIa	Documentation of Method QC Results	Establish that all method quality control were analyzed for and in control as listed in the analytical SOPS. If method QA was not in control, the laboratory will have contacted WESTON of non-conformant situation prior to report generation for guidance.	Kelly Spittler, WESTON
IIa/IIb	Documentation of QAPP QC Samples Results	Establish that all QAPP required QC samples were collected. Establish that the collected QC samples met the required limits as established in the QAPP.	John Gerhard, WESTON Kelly Spittler, WESTON
IIa/IIb	Documentation of Analytical Reports for Completeness	Ensure that from the Chain-of-Custody generated in the field to the delivery of the analytical data that the appropriate analytical samples have been collected, appropriate site identifications have been used, and the correct analytical methods have been applied. Review the analytical reports to establish that all required forms, case narratives, samples, Chains-of-Custody, logbooks, and raw data have been included.	Kelly Spittler, WESTON
IIb	Project Quantitation Limits	Review laboratory analytical met the project quantitation limits specified in QAPP worksheet 15.	Kelly Spittler, WESTON
IIa/IIb	Project Action Limits	Review and add project action limits to the laboratory electronic data deliverable. Flag samples and notify project manager of samples that exceed the project action limits.	Kelly Spittler, WESTON
IIa/IIb	Data Verification	Data Verification will be performed on all samples. Data verification that sample analysis was performed as stated in the QAPP and per the laboratory SOPs.	Kelly Spittler, WESTON
IIa/IIb	Data Validation	Validation will be performed on all samples. Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited EPA SW-846 methodology. Validation Qualifiers applied as Manual Level M3 for organic compounds and Manual Level IM2 for inorganic compounds following the most recent version of the EPA Region III *Modifications to the National Functional Guidelines* for organic and inorganic data review, and the EPA Region III *Innovative Approaches to Data.* Methods for which no data validation guidelines exist will be validated following the *National Functional Guidelines* deemed most appropriate by the data validator. The data validator will receive all laboratory packages and analytical results electronically. Additionally, the validator will be required to submit final validation reports via pdf format and must provide an annotated laboratory analytical result EDD with applicable data validation qualifiers and/or result value modifications.	Sherif Mina, MCGI, Data Validator**

Worksheet 36 — Sampling and Analysis Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Validation Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Soil Sediment	Explosives SW8330B ICP Metals SW6010B	Tier III	Project Validation Criteria as per QAPP worksheets 12, 15, 19, 28, 37 and cited EPA SW-846 methodology. Validation Qualifiers applied as per Manual Level M3 for organic compounds and Manual Level IM2 for inorganic compounds following the most recent version of the EPA Region III *Modifications to the National Functional Guidelines* for organic and inorganic data review, and the EPA Region III *Innovative Approaches to Data Validation*. Methods for which no data validation guidelines exist will be validated following the *National Functional Guidelines* deemed most appropriate by the data validator.	Sherif Mina, MCGI Validator*

*Meridian Consultant Group, Inc. (MCGI), 1997 Annapolis Exchange Parkway, Suite 300, Annapolis, MD 21401.

Worksheet 37 — Data Usability Assessment

Based on the current oversight responsibilities and limited analytical scope, this data usability assessment worksheet outlines the approach that will be taken as the analytical scope expands during the contract period of performance.

Data quality indicators (DQI), such as precision, accuracy, completeness, representativeness, and comparability measurements, aid in the evaluation process and are discussed below.

Precision

The most commonly used estimates of precision are the relative percent difference (RPD) for cases in which only two measurements are available, and the percent relative standard deviation (%RSD) when three or more measurements are available. This is especially useful in normalizing environmental measurements to determine acceptability ranges for precision because it effectively corrects for the wide variability in sample analyte concentration indigenous to samples.

Precision is represented as the RPD between measurement of an analyte in duplicate samples or in duplicate spikes. RPD is defined as follows:

$$RPD = \frac{|C_1 - C_2|}{\frac{C_1 + C_2}{2}} \times 100$$

Where:
C_1 = First measurement value
C_2 = Second measurement value

The % RSD is calculated by the standard deviation of the analytical results of the replicate determinations relative to the average of those results for a given analyte. This method of precision measurement can be expressed by the formula:

$$\%RSD = \frac{\sqrt{\sum_{I-1}^{N}\left(\frac{RF_i - RF}{N-1}\right)}}{RF} \times 100$$

Where:
RF = Response factor
N = Number of measurements

Precision control limits for evaluation of sample results are established by the analysis of control samples. The control samples can be method blanks fortified with surrogates (e.g., for organics), or laboratory control samples (LCS) purchased commercially or prepared at the laboratory. The LCS is typically identified as blank spikes (BS) for organic analyses.

For multi-analyte methods, the LCS or BS may contain only a representative number of target analytes rather than the full list.

The RPD for duplicate investigative sample analysis provides a tool for evaluating how well the method performed for the respective matrix.

Accuracy/Bias

Accuracy control limits are established by the analysis of control samples, which are water and/or solid/waste matrices.
For organic analyses, the LCS may be a surrogate compound in the blank or a select number of target analytes in the blank spike. The LCS is subjected to all sample preparation steps. When available, a solid LCS may be analyzed to demonstrate control of the analysis for soil. The amount of each analyte recovered in an LCS analysis is recorded and entered into a database to generate statistical control limits. These empirical data are compared with available method reference criteria and available databases to establish control criteria.

The percent recovery (% R) for spiked investigative sample analysis (e.g., matrix spike) provides a tool for evaluating how well the method worked for the respective matrix. These values are used by the client to assess a reported result within the context of the project data quality objectives. For results that are outside control limits provided as requirements in the QAPP, corrective action appropriate to the project will be taken and the deviation will be noted in the case narrative accompanying the sample results. Percent recovery is defined as follows:

$$\%\,\text{Recovery} = \frac{(A_T - A_0)}{A_F} \times 100$$

Where:

A_T = Total amount recovered in fortified sample
A_0 = Amount recovered in unfortified sample
A_F = Amount added to sample

Accuracy for some procedures is evaluated as the degree of agreement between a new set of results and a historical database or a table of acceptable criteria for a given parameter. This is measured as percent difference (%D) from the reference value, and is primarily used by the laboratory as a means for documenting acceptability of continuing calibration.

The %D is calculated by expressing, as a percentage, the difference between the original value and new value relative to the original value. This method for precision measurement can be expressed by the formula:

$$\%\,D = \frac{C_1 - C_2}{C_1} \times 100$$

Where:

C_1 = Concentration of analyte in the initial aliquot of the sample.
C_2 = Concentration of analyte in replicate.

Completeness

Project-specific completeness goals account for all aspects of sample handling, from collection through data reporting. The level of completeness can be affected by loss or breakage of samples during transport, as well as external problems that prohibit collection of the sample. The following calculation is used for determining the percent complete:

$$\text{Completeness} = \frac{A}{B} \text{ x } 100$$

Where:
A = Number of usable data points.
B = Total number of data points collected.

The formula for sampling completeness is:

$$\text{Sampling Completeness} = \frac{\text{Number of locations sampled}}{\text{Number of planned sample locations}} \text{ x } 100$$

An example formula for analytical completeness is:

$$\text{VOC Analytical Completeness} = \frac{\text{Number of Usable Data Points}}{\text{Expected Number of Usable Data Points}} \text{ x } 100$$

The ability to meet or exceed completeness objectives is dependent on the nature of samples submitted for analysis.

The following table lists the completeness goals for this program. If the completeness goal is not met because of controllable circumstances, then the samples will be recollected and reanalyzed, as necessary, to meet the completeness objective. If the completeness goal is not met because of uncontrollable circumstances, such as inaccessible sample points, matrix interferences, etc., then the deficiency will be evaluated.

Project Completeness Goals

Task	Subtask	Completeness Goal
Sampling	Sample Collection	95%
Analytical Measurements	All Laboratory Analyses	95% of collected analytes
		80% of each target analyte

Representativeness

Data representativeness for this project is accomplished by implementing approved sampling procedures and analytical methods that are appropriate for the intended data uses, and which are established within this QAPP.

Comparability

Comparability of data sets generated for this project will be obtained through the implementation of standard sampling and analysis procedures, by the use of traceable reference materials for laboratory standards, and by expressing the results in comparable concentration units.

Sensitivity/Selectivity

Sensitivity is the ability of the method or acceptable sensitivity instrument to detect the contaminant of concern and other target compounds at the level of interest. Quantitative measurement performance criteria need to be determined for acceptable sensitivity to ensure that the quantitation limits can be routinely achieved for each matrix, analytical parameter, and concentration level.

Quantitative measurement performance criteria need to be determined for acceptable sensitivity to ensure that the quantitation limits can be routinely achieved for each matrix, analytical parameter, and concentration level. The use of standards and instrument calibration will enable the instrument to identify and differentiate between various compounds/analytes of interest and interferences.

Assessment of Data Usability

Assessment of the data usability is an important component and will be performed as a preliminary step of the data interpretation phase.

In addition, data assessment is considered the final step in the data evaluation process and can be performed only on data of known and documented quality. As described in Worksheet 36, data generated for this project will undergo a formalized evaluation/validation process, following EPA Region 3 protocol. For this project, all data will be assessed for usability, regardless of the data evaluation/validation process implemented. As mentioned previously, data usability goes beyond validation in that it

evaluates the achievement of the DQOs based on the comparison of the project DQIs and individual study-specific work plans, with the obtained results. The results of the data usability assessment, and particularly any changes to the DQOs necessitated by the data not meeting usability criteria, will be included in each final report.

Primarily, the assessment of the usability will follow procedures described in appropriate EPA guidance documents, particularly *Guidance for Data Useability in Risk Assessment* (Publication No. 9285.7-05FS, September 1992), and will be conducted according to the process outlined below.

Sampling and Analysis Activities Evaluation

The first step of the data usability evaluation will include a review of the sampling and analysis activities in comparison to project-specific DQIs and study-specific workplans. Specific limitations to the data, i.e., results that are qualified as estimated (J/UJ), or rejected (R), will be determined and documented in the database. The data acquisition and evaluation process consists of a series of procedures that were designed to maximize final data quality as outlined in the following Figure.

Achievement of DQIs

The second part of data usability pertains to the achievement of the program-specific DQIs. Each investigator will compare the performance achieved for each data quality criterion against the expected and planned performance. In general, this comparison will follow from the DQIs used to define each DQO. This comparison is the most critical component of the assessment process. Any deviation from planned performance will be documented and evaluated to determine whether corrective action is advisable. Potential corrective actions will range from resampling and/or reanalysis of data, to qualification or exclusion of the data for use in the data interpretation. In the event that corrective action is not possible, the limitations, if any, of the data with regard to achieving the DQOs will be noted.

In conjunction with the DQI achievement review, the investigators will need to make decisions for the use of qualified values, which are a consequence of the formalized evaluation/validation process. Data qualifiers will be applied to individual data results. Data usability decisions will be made based on the assessment of the usability of each of these results for the intended purpose. Evaluation will describe the uncertainty (bias, imprecision, etc.) of the qualified results. Cumulative QC exceedances from the DQIs may require technical judgment to determine the overall effect on the usability of the data. Decisions about usability of qualified data for use in risk assessment will be based on the EPA document mentioned, which allows for the use of estimated values. Finally, data users may choose to determine final data usability qualifiers as a result of this overall examination and decision process.

Achievement of DQOs

The third step in the data usability process concerns achievement of the DQOs. Once the data set has been assessed to be of known quality, data limitations have been documented, and overall result applicability/usability for its intended purpose has been determined, the final data assessment can be initiated by considering the answers to the following questions:

- Are the data adequate to determine the extent to which hazardous substances have migrated or to what extent they were expected to migrate from potential hazardous substance source areas?
- Do the data collected adequately characterize the nature and extent of potential hazardous substance source areas at the site?
- Are the data statistically adequate to evaluate on a per chemical and per media basis?
- Do the data collected allow assessment of hydrogeologic factors, which may influence contaminant migration/distribution?
- Is the sample set sufficient to develop site-specific removal and disposal treatment methodologies?
- Have sufficient data been collected to evaluate how factors including physical characteristics of the site and climate and water table fluctuations affect contaminant fate and transport?
- Have sufficient data been collected to determine the toxicity, environmental fate, and other significant characteristics of each hazardous substance present?
- Is the data set sufficient to evaluate the potential extent and risk of future releases of hazardous substances, which may remain as residual contamination at the source facility?

The study principal investigators, in conjunction with the project team, will need to formulate solutions if data gaps are found as a result of problems, biases, trends, etc., in the analytical data, or if conditions exist that were not anticipated in the development of the DQOs. It is particularly important that each data usability evaluation specifically address any limitations on the use of the data that may result from a failure to achieve the stipulated DQO.

If the project scope changes, the DQOs will be expanded. The DQOs will address the specific action limits and measurable performance criteria, in order to make appropriate decisions on the analytical data.

ATTACHMENT A

ANALYTICAL STANDARD OPERATING PROCEDURES

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TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Nitroaromatic and Nitroamine Explosive Compounds by High Performance Liquid Chromatography (HPLC) [SW-846 8330A & 8330B]

Approvals (Signature/Date):

Susan Decker — Date 4/6/09
Technical Specialist

Dennis Jonsrud — Date 4-10-09
Technical Manager

Adam Alban — Date 10 Apr 09
Health & Safety Manager / Coordinator

Karen Kuoppala — Date 4-14-09
Quality Assurance Manager

Robert C. Hanisch — Date 4/10/09
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the determination of nitroaromatic and nitroamine explosive residues by high performance liquid chromatography (HPLC) using dual columns and dual UV wavelengths. This includes analysis in water, soil, and sediment matrices. The instrumental analysis is based on Methods 8330A and 8330B.

1.2 This SOP does not include the extraction procedures. For those details, please refer to DV-OP-0017 *Solid Phase Extraction of Nitroaromatic and Nitroamine Explosive Compounds and Picric Acid from water samples by SW-846 3535A* and DV-OP-0018 *The Extraction of Nitroaromatic and Nitroamine Explosive Compounds and Picric Acid from Soil Samples by SW-846 8330A and 8330B.*

1.3 On occasion clients may request modifications to this SOP. Requests for modifications must be received in writing and will be communicated to the laboratory through client requirements in the LIMS.

1.4 Application of 8330A versus 8330B

1.4.1 This procedure is for analysis by either Method 8330A or 8330B. The most important differences in the two source methods are the more rigorous sample collection and preparation measures in 8330B, which are designed to produce more representative results. The more rigorous 8330B process is specifically intended to complement the multi-incremental field sampling process described in Appendix A of 8330B. If multi-incremental or equivalent systematic sampling processes are not employed in the field, then the extra laboratory homogenization and subsampling effort 8330B requires (see details in DV-OP-0018) may add little or no improvement in the overall precision of results.

1.4.2 A larger sample size is used for 8330B (10 g) than is used for 8330A (2 g).

1.4.3 8330A only describes the cyano (CN) column for confirmation. 8330B gives the option of either CN or pheny-hexyl (Luna) columns for confirmation. Because it provides better sensitivity and resolution, TestAmerica Denver routinely uses the Luna column for both methods.

1.4.4 In addition, 8330B added compounds to the potential analyte list. TestAmerica Denver offers any or all of the compounds shown in Appendix 1 of this SOP by both methods.

1.5 Analytes, Matrix(s), and Reporting Limits

The list of analytes and their CAS numbers can be found in Appendix 1.

The working ranges of this method are as follows:

ANALYTES	SOILS	WATERS
Standard Analytes	0.20 µg/g – 50 µg/g	0.25 µg/L - 25 µg/L
Nitroglycerin and PETN	2 µg/g – 5000 µg/g	2.5 µg/L - 250 µg/L
Picric Acid	0.20 µg/g – 50 µg/g	0.25 µg/L - 25 µg/L
3,5-Dinitroaniline	0.20 µg/g – 50 µg/g	0.25 µg/L – 25 µg/L

2.0 Summary of Method

2.1 Instrument calibration is performed by external standardization using a minimum of five concentration levels.

2.2 A water/methanol gradient program is used for HPLC separation (see details in Appendix 4). Compounds are tentatively identified based on retention time and detection by the UV detector using the primary Phenomenex ODS column. Confirmation is performed by the UV detector using the phenyl-hexyl (Luna) column (see Appendix 4 for instrument conditions).

3.0 Definitions

3.1 Explosives: As used in this SOP, the term "explosives" refers specifically to the analytes listed in Appendix 1. These include compounds that can be readily detonated with heat, shock, or ignition, such as nitroglycerin, RDX, and TNT. It also includes production by-products and degradation products of true explosives.

3.2 Definition of terms used in this SOP may be found in the Glossary section of the TestAmerica Denver Quality Assurance Manual (QAM).

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks.

4.2 Contamination by carryover can occur when a low-concentration sample is analyzed immediately following a high-concentration sample.

4.3 Co-elution of target analytes with non-target analytes can occur, resulting in false positives or biased high results.

4.4 The inclusion of vegetation is not recommended given the nature of the detector and different uses the data will potentially support (USACE comment – Issue #306 Audit Database).

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual (RSM) 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 nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; non-disposable gloves must be cleaned immediately.

WARNING: Soil samples with explosive concentrations greater than 2% cannot be accepted by the laboratory unless they have a moisture content of 25% or greater. Under no circumstances shall a soil sample with an explosive concentration greater than 10% be accepted by the laboratory.

5.1.2 If a sample is expected to have an explosive concentration ≥2% (but less than 10%), the EH&S Coordinator and Group Leader shall be notified before any work is performed. Additional safety precautions may be implemented as required due to high concentrations of explosives.

5.1.3 Soil samples with high concentrations (between 2 and 10%) of explosives should not be ground using a mortar and pestle. Visual observation of a soil samples is important prior to grinding samples. Any samples containing metal fragments, powders, waxy appearing pieces, or other suspicious material should be brought to the attention of the Group Leader and the EH&S Coordinator before proceeding with the procedure. Bypassing the grinding step and proceeding to solvent dilution is an alternative for samples that are determined to be unsafe to grind.

5.2 Primary Materials Used

The following is a list of materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not contain all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagent and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

MATERIAL [1]	HAZARDS	EXPOSURE LIMIT [2]	SIGNS AND SYMPTOMS OF EXPOSURE
ACETONITRILE	Flammable Poison	40 PPM – TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
METHANOL	FLAMMABLE Poison IRRITANT	200 PPM - TWA	A slight irritant to the mucous membranes. Toxic effects are exerted upon the nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause the skin to be become dry and cracked. Skin absorption can occur, symptoms may parallel inhalation exposure. Irritant to the eyes.
PHOSPHORIC ACID	CORROSIVE	1 PPM - TWA	Ingestion can cause severe burns to the throat, mouth, and stomach, abdominal pain and nausea. Severe exposures by ingestion can lead to shock, circulatory collapse, and death. Inhalation is not an expected hazard unless misted. Corrosive, contact with skin or eyes can cause redness, pain, severe burns, blurred vision, and permanent eye damage.
SODIUM HYDROXIDE	CORROSIVE POISON	2 MG/M^3	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, runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and can cause burns that may result in permanent impairment of vision, even blindness with greater exposures.
(1) ALWAYS ADD ACID TO WATER TO PREVENT VIOLENT REACTIONS. (2) EXPOSURE LIMIT REFERS TO THE OSHA REGULATORY EXPOSURE LIMIT.			

6.0 Equipment and Supplies

6.1 Instrumentation

- HPLC System
- HPLC, equipped with a pump capable of achieving 4000 psi, a 250 µL loop injector, and a Diode Array Detector, Hewlett Packard Model 1090, or equivalent.
- Primary Column: Reverse phase HPLC column, 15 cm x 4.6 mm (5 µm), Phenomenex ODS (20) or equivalent.
- Confirmation Column: Luna Phenyl Hexyl 10 reverse phase HPLC column, 15 cm x 4.6 mm (3 µm) or equivalent.
- Hewlett Packard HPLC Chem Station for instrument control.
- Autosampler (optional).

6.2 Supplies

- Glass vials, various sizes.
  - Amber glass, 8.0 mL, with Teflon-lined screw caps, for the storage of final extracts.
  - Amber crimp-top vial with caps for analysis, 1.8 mL.
- Disposable pipettes, used for non-quantitative transfers only.
- Re-pipettor, 500 µL – used to dilute the acetonitrile soil extracts 2X with calcium chloride solution.

7.0 Reagents and Standards

7.1 Stock Standards

7.1.1 Stock standards are purchased as certified solutions or prepared from 100%, neat solutions. Stock standard solutions are stored at -10 °C to -20 °C, or per vender instructions. All stock standards must be protected from light and should be brought to room temperature before using.

7.1.2 Stock standard solutions must be replaced after 1 year or sooner if comparison with check standards prepared from an independent source indicates a problem. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule.

7.2 High-Level Standard Mixes Prepared from Stock Standards

7.2.1 Volume Measurements for all Standards Preparation

The volume of stock and intermediate standard solutions used in subsequent dilutions is measured using Hamilton syringes appropriate for the volume being measured and accurate to 2%. Standards are prepared either by using a syringe to measure the standard solution and making to volume with the appropriate solvent in a Class A volumetric flask, or by measuring the volumes of both the standard solution and the solvent using a calibrated syringe or Class A pipette and combining them in a vial.

7.2.2 High-Level Calibration Mix (CSA)

A solution is prepared to contain all standard analytes at a concentration of 100 µg/mL each in acetonitrile (see details in standards database instructions). Nitroglycerin and PETN are prepared at 1000 µg/mL in acetonitrile.

7.2.3 High-Level Spike Mix

All analytes except the surrogate compound, 1,2-dinitrobenzene (1,2-DNB), are spiked into acetonitrile contained in an amber screw-top vial. The final concentration for all standard analytes is 100 µg/mL, except for nitroglycerin and PETN, which are prepared at 1,000 µg/mL.

7.2.4 High-Level Surrogate Mix

1,2-DNB is spiked into acetonitrile in a screw-top amber vial to achieve a l00 µg/mL final concentration.

7.2.5 These high level standards must be replaced every 6 months or sooner if comparison with check standards prepared from independent sources indicates a problem.

7.3 Intermediate-Level Calibration Standards (CSB)

7.3.1 Prepare a 20 µg/mL solution from the high-level (CSA) solutions as follows (nitroglycerin and PETN are prepared in the same mix at 200 µg/mL):

7.3.2 Partially fill a 5-mL volumetric flask with approximately 3.0 mL of acetonitrile.

7.3.3 Using a 1 mL Hamilton syringe, add 1.0 ± 0.02 mL of CSA and 1.0 mL of picric acid to the volumetric flask.

7.3.4 Bring to volume with acetonitrile and transfer to a labeled, screw-top amber vial.

7.3.5 Record all calculations in the standards database.

7.3.6 The shelf life of this material is 30 days.

7.4 Working-Level Standards for Five-Point Calibration Curve

7.4.1 Prepare calibration standards by diluting the intermediate standard solutions as shown in the table below using the 75%:25% (v/v) acidic water:ACN solution (described Section 7.8.7). These standards must be prepared fresh on the day of calibration and refrigerated if not used immediately. All volumes are measured using the appropriately sized Hamilton syringe.

Calibration Levels

Calibration Level	Final Concentration (µg/mL)			Vol. of CSB to Add (µL)	Vol. of Acidic Water/ACN Solution to Add (µL)
	Standard Analytes	Nitroglycerin & PETN	Picric Acid		
8	2.5	25.0	2.5	125 ± 1	875 ± 9
7	1.0	10.0	1.0	50 ± 0.5	950 ± 10
6	0.7	7.0	0.7	35 ± 0.4	965 ± 10
5	0.4	4.0	0.4	20 ± 0.2	980 ± 10
4*	0.25	2.5	0.25	12.5 ± 0.1	988 ± 10
3	0.1	1.0	0.1	5 ± 0.05	995 ± 10
2	0.05	0.5	0.05	2.5 ± 0.02	998 ± 10
1	0.01	0.1	0.01	10 ± 0.1 µL of Level 7	990 ± 10
* Level 4 concentration is used for the daily and continuing calibrations.					

7.5 Working-Level Spike Solution

7.5.1 8330 Soil LCS - Prepare the 8330 Soil LCS solution in acetonitrile at the concentrations listed in Appendix 3. This standard is stored in a freezer at –30°C to –25°C and given a six-month expiration date. The standard is allowed to come to room temperature before use and returned to the freezer as soon as possible.

7.5.2 8330 Water LCS – Prepare the 8330 Water LCS solution in acetonitrile at the concentrations listed in Appendix 3. This standard is stored in a freezer at -30°C to -25°C. This standard is stored in 4mL vials in order to protect the integrity of the compounds at these low concentrations as the smaller container size allows only the portion of the standard that is to be used that day come to room temperature. This standard is given a six-month expiration date.

7.5.3 3,5-Dinitroaniline LCS Standard – Prepare the 3,5-DNA LCS standard in acetonitrile at the concentrations listed in Appendix 3. This standard is stored in a freezer at –30°C to –25°C and given a six-month expiration date. The standard is allowed to come to room temperature before use and returned to the freezer as soon as possible. This standard is used only for method 8330B and is added to separate LCS and MS/MSD samples as this compound cannot be completely resolved from tetryl and nitrobenzene on the primary column.

7.6 Working-Level Surrogate Solution

7.6.1 8330 Soil Surrogate – Prepare the 8330 Soil Surrogate solution in acetonitrile at the concentration listed in Appendix 3. This standard is stored in a freezer at –30°C to –25°C and given a six-month expiration

date. The standard is allowed to come to room temperature before use and returned to the freezer as soon as possible.

7.6.2 8330 Water Surrogate - Prepare the 8330 Water Surrogate solution in acetonitrile at the concentration listed in Appendix 3. This standard is stored in a freezer at –30°C to –25°C and given a six-month expiration date. The standard is allowed to come to room temperature before use and returned to the freezer as soon as possible

7.7 Second Source Initial Calibration Verification Solution

The second source standard must be obtained from a different source than the standards used for initial calibration. This standard is used to verify the accuracy of the calibration standards.

7.7.1 High-Level Second Source Mix

A total of 9 separated mixes are purchased. A solution is purchased containing 2,4-diamino-6-nitrotoluene at 100 µg/mL. A solution is purchased containing 2,6-diamino-4-nitrotoluene at 100 µg/mL. A solution is purchased with PETN at 1000 µg/mL. A solution is purchased with nitroglycerin at 1000 µg/mL. A solution is purchased with picric acid at 1000 µg/mL. A solution is purchased with 3,5-dinitroaniline at 1000 µg/mL. Two additional solutions are purchased that contain the remaining compounds at 1000 µg/mL. A solution containing the surrogate 1,2-dinitrobenzene at 1000 µg/mL.

7.7.2 Intermediate-Level Second Source Mix

Two solutions are prepared from the standards in section 7.7.1. One solution contains the surrogate and all compounds except 3,5-dinitroaniline at a concentration of 20µg/mL, except PETN and nitroglycerin which are at a concentration of 200µg/mL. A separate intermediate solution is prepared for 3,5-Dinitroaniline at 20 µg/mL.

7.7.3 Working-Level Second Source Mix

Prepare a solution containing all compounds except 3,5-dinitroaniline at a concentration of 0.40 µg/mL, except PETN and nitroglycerin, which are at a concentration of 4.0 µg/mL. A separate solution containing 3,5-dinitroaniline is prepared at a concentration of 0.40 µg/mL. These solutions are prepared using the 75%:25% (v/v) acidic water:ACN solution (described Section 7.8.5). These standards must be prepared fresh on the day of calibration. All volumes are measured using the appropriately sized Hamilton syringe.

7.8 Reagents

7.8.1 Reagent Water – For method blanks and laboratory control samples. TestAmerica Denver has two ELGA water purification systems. The water coming from the ELGA system should be 17-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026.

7.8.2 HPLC Grade Water

7.8.3 Acetonitrile, CH3CN - HPLC grade (ACN).

7.8.4 Methanol - HPLC grade, distilled in glass.

7.8.5 Glacial Acetic Acid – Reagent Grade

7.8.6 Phosphoric acid, H3PO4 – Reagent Grade.

7.8.7 Acidic Water:ACN Solution, 75%:25%

Add 3 drops phosphoric acid to a mixture of 300 mL of reagent water plus 100 mL of acetonitrile (ACN). Volumes are measured using a Class A graduated cylinder.

7.8.8 Calcium Chloride, CaCl2 Solution, 5 g/L

Place 5 ± 0.05 g of reagent grade CaCl2 into a one-liter volumetric flask containing approximately 500 mL of deionized water. Swirl the solution until the CaCl2 is dissolved. Add approximately 1 mL of 85% H3PO4 to acidify the solution and make to volume with deionized water.

7.8.9 Eluents for Standard Analytes

- Primary Column: Place methanol and reagent water in individual solvent containers on the HPLC. The HPLC is programmed to mix the solvents.
- Confirmation Column: Place methanol, and reagent water in individual solvent containers on the HPLC. The HPLC is programmed to mix the solvents.

7.8.10 Eluents for Picric Acid

- Primary Stock: Slowly add 115.29 grams of 85% Phosphoric acid (molecular weight = 97.9924 g) to approximately 500 mL of water in a 1L beaker. Place a stir bar in the beaker and the beaker in an ice bath on top of a magnetic stirrer. Slowly add 150 mL of 10M (10M = 10N) sodium hydroxide allowing time for the mixture to cool down between additions. Tranfer to a 1L volumetric flask and bring to volume with DI water. The final pH of this solution should be 7.2.

7.8.11 Working Eluent Primary Column: Using approximately 1-L of water, 1 mL of primary stock solution, and 20 uL of Glacial Acetic acid to adjust the pH of the buffer to approximately 7.0. Make fresh before each run.

7.8.12 Working Eluent Confirmation Column: Using approximately 1-L of water, 2 mLs of primary stock solution, and 65 µL of Glacial Acetic acid to adjust the pH of the buffer to approximately 7.0. Make fresh before each run.

7.8.13 The pH of the working eluent may be modified by the analyst to improve compound resolution on the confirmation column.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples should be collected in duplicate one-liter amber glass bottles with Teflon-lined caps. For method 8330A, soil samples should be collected in eight-ounce wide mouth jars with Teflon-lined caps. When sampling for DoD projects that must comply with DoD QSM, version 3 requirements for drying and grinding the entire contents of a soil sample container, a separate container should be used to collect a soil sample for this analysis. For method 8330B, it is not uncommon to receive samples of 1kg or more. Samples may be shipped in wide mouth jars or clean plastic bags.

8.2 Samples and sample extracts must be stored in amber glass containers at 4 ± 2 °C from the time of collection through analysis, except during drying.

8.3 Soil and sediment samples should be air dried at ambient temperature until dry enough to sieve. See DV-OP-0018 for details. Once the sample is air dried, the sample can be stored at room temperature.

8.4 All water samples must be extracted within 7 days of collection and analyzed within 40 days after extraction begins. All soil and sediment samples must be extracted within 14 days of collection and analyzed within 40 days after extraction begins.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Amber glass	1 Liter	Cool 4 ± 2°C	7 Days	SW846 8330A
Soils	Glass/ plastic	4 grams (8330A)/up to 1 Kg (8330B)	Cool 4 ± 2°C	14 Days	SW846 8330A/B

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.2 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Control Program.

9.3 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.4 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.5 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.6 Initial Method Demonstrations

An initial demonstration of capability (IDOC), retention time study, and a method detection limit study must be performed by an analyst before that analyst can analyze samples. Current MDLs are found in the laboratory LIMS system. See Section 12 of this SOP for further details.

9.7 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. 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. The method blank must be run on each instrument. See QC Policy DV-QA-003P for further details.

9.8 Method Blank (MB)

A method blank (MB) must be prepared and analyzed with each batch of samples. The MB consists of reagent water for aqueous samples, and Ottawa sand for soil samples, with surrogates added. The MB is created at the time of extraction after the samples have been dried sieved, and ground and is then carried through all extraction and analysis steps. 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.

Acceptance Criteria: The MB must not contain any analyte of interest at or above one-half the RL or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

Corrective Action: When the MB fails acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize, or eliminate the problem. All samples in the QC batch that have concentrations of target analytes greater than the RL, but less than 10 times the concentration in the MB must be re-prepared and reanalyzed.

If there are no target analytes greater than the RL, then the data may be reported with qualifiers if this is acceptable to the client.

If the MB acceptance criteria are not met and re-preparation and reanalysis are not possible, then the

sample data associated with the unacceptable MB must be qualified. This nonconformance must be addressed in the project or case narrative and the client must be notified.

9.9 Laboratory Control Sample (LCS)

One LCS must be analyzed with each batch of samples. The LCS must contain specified analytes of interest and must be carried through the entire analytical procedure. For water samples, the LCS is prepared by spiking the analytes of interest into reagent water. For soil samples, the LCS is prepared by spiking the analytes of interest into Ottawa sand. The LCS is created at the time of sample extraction after the samples have been dried sieved and ground. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

Acceptance Criteria: The LCS recovery for each spiked analyte must be within established control limits. Laboratory default control limits are calculated as ± 3 standard deviations around the mean of historical data, as described in Policy QA-003. Control limits are maintained in the LIMS. In accordance with the NELAC 2003 Standard (Quality Systems, Appendix D), a marginal exceedance within ± 4 standard deviations is allowed for one of the analytes. This is based on the number of analytes typically spiked for this method, which is between 11 and 30. These acceptance criteria may be superseded by project-specific limits, as applicable.

Corrective Action: If recoveries for all spiked analytes are not within the acceptance limits, including the one allowed marginal exceedance, the analytical system is out of control and corrective action must occur. Generally this requires re-extraction and reanalysis of all associated samples. If the LCS is biased high and all associated samples are ND, not detected, it may be possible to report results with an NCM (see requirements for individual programs).

9.10 Matrix Spike Sample (MS) and Matrix Spike Duplicate (MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. The soil matrix spikes are created at the time of extraction. Spikes and surrogate compounds are added after the sample has been dried, sieved, and ground. One MS/MSD pair must be processed for each preparation batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks, equipment blanks, or rinse blanks cannot be used for MS/MSD analysis.

NOTE: The MS/MSD must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out, or will exceed the linear range of the calibration standards.

Acceptance Criteria: The spike recoveries must fall within established control limits. The relative percent difference (RPD) between the MS and MSD must be less than or equal to the established RPD limit. Control limits are calculated as ± 3 standard deviations around the mean of historical data, as described in Policy DV-QA-003P. Control limits are maintained in the LIMS. Some federal programs require LCS limits to be used for MS/MSD evaluation (see SOP DV-QA-024P).

Corrective Action: If analyte recovery in the MS and/or MSD or the RPD between the MS and MSD fail acceptance criteria, but the recovery of the LCS is in control, then the method is considered to be in control and the samples in the batch do not require re-preparation and reanalysis, unless the results indicate that a spiking error may have occurred. The reasons for accepting the batch must be documented in the case narrative. If the recovery of the LCS also fails acceptance criteria, then the samples in the batch must be re-prepared and reanalyzed.

9.11 Sample Duplicate

Although not typically required for organic analyses, a duplicate sample may be required for project-specific quality control. In this case, a sample duplicate is a second aliquot of one of the samples in the batch. Field blanks cannot be used for duplicate testing. The results for duplicates are reported separately, and cannot be averaged when reporting results. Sample duplicate results are used to evaluate the precision of the method. As such, results should be greater than or equal to the RL for a valid statistical comparison.

Acceptance Criteria: The RPD between the sample and the sample duplicate results must be less than the established limit.

Corrective Action: Results for samples that do not meet acceptance limits, particularly if due to difficulties in subsampling, shall be discussed in the final report case narrative, after client notification and agreement.

9.12 Sample Replicates

Replicate analyses are not part of the laboratories standard quality control samples. In the event that replicate analyses are required for a given project, the lab will extract triplicate aliquots after grinding on the client designated sample. The lab will determine the %RSD as defined below. Results for the %RSD as well as the individual replicate results will be reported to the client. The method suggests that the %RSD for the subsampling error is acceptable if it is <10%.

The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{s}{\overline{C}} \times 100\%$$

Where s is the standard deviation of the average concentration and is calculated as follows:

$$s = \sqrt{\frac{\sum_{i=1}^{n} (C_i - \overline{C})^2}{n-1}}$$

In the event that the laboratory is requested to perform the evaluation of field replicate precision, three field replicates designated by the client will be processed through the entire homogenization and extraction steps. The %RSD for these replicates will be calculated as indicated above and reported to the client.

9.13 Grinding Blank

Refer to SOP DV-OP-0018 for details on how the grinding blanks for soils by method 8330B are prepared.

Acceptance Criteria: The grinding blank must not contain any analyte of interest at or above one-half of the or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

Corrective Action: If the composite grinding blank results are greater than the acceptance limits, then the individual grinding blanks will be extracted and analyzed to determine when the contamination occurred and exactly which samples were affected. Samples associated with a contaminated grinding blank producing positive results for the same contaminant must be reprocessed and reanalyzed. If unground sample is not available, then the potential carry-over between samples must be described in a non-conformance memo and discussed in the final report case narrative.

9.14 Sample QC Frequency

The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency
Method Blank (MB)	1 in 20 or fewer samples
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples
Matrix Spike (MS)[2]	1 in 20 or fewer samples
MS Duplicate (MSD)[2]	1 in 20 or fewer samples
Surrogates	every sample[3]

[1] LCS Duplicate (LCSD) is performed if insufficient sample is available for the MS/MSD and it is requested by the client/project/contract.

[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client....predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)

9.15 Instrument QC

9.15.1 External calibration is used for this analysis. Prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in Appendix 2.

9.15.2 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 and any changes in instrument operating parameters (including solvent flows, oven temperatures, etc.).

9.15.3 With the exception noted in Section 9.15.4 below, it is NOT acceptable to remove points from the 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.

9.15.4 A level may be removed from the calibration if the reason can be clearly documented (i.e., a broken vial or an injection error). A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration.

9.16 HPLC Startup

All electronic equipment should be allowed to warm up for 30 minutes. During this period, at least 15 void volumes of mobile phase are passed through the column and continued until the diode array detector's (DAD) baseline has stabilized.

9.17 External Standard Calibration:

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 HPLC 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.

9.18 Initial Calibration:

9.18.1 Average response factors (RF) or least-squares linear regression may be used to fit the data. The percent relative standard deviation (%RSD) or correlation coefficients (r) are used to evaluate the linearity of the initial calibration data. Calculations for both approaches are shown in Section 11.3 and 11.4.

9.18.2 If average response factors are used, %RSD must be ≤ 20.

9.18.3 If a linear regression function is used, r must be ≥ 0.995, which is equivalent to $r2 \geq 0.990$.

- The following requirements must be met for any calibration to be used:
- Response must increase with increasing concentration.
- Calibration curves will not be forced through the origin.
- The intercept of the curve at zero response should ideally be less than the MDL for the analyte. At a minimum the intercept must be less than ½ the reporting limit.

9.19 Calibration Verification

9.19.1 Initial Calibration Verification (ICV)

A second-source verification standard must be analyzed with each initial calibration. The calculated concentration of the analytes in this standard may not be greater than 20% different from the calibration standard.

9.19.2 Continuing Calibration Verification (CCV)

9.19.2.1 The working calibration curve or RF must be verified by the analysis of a mid-point continuing calibration standard at the beginning of the analysis sequence, after every 10 samples (laboratory QC samples included), and at the end of the analysis sequence. Results are acceptable for any individual compound if the %D (percent difference between the standard and measured values of the CCV standard) is ≤ 20%. According to SW-846 Method 8000B, the calibration verification is also acceptable if the average of the %D values for all the analytes is ≤ 15%. This average is calculated by summing all the absolute %D results in the calibration (including surrogates) and dividing by the number of analytes. However, TestAmerica Denver also requires that no single analyte result exceeds 30 %D.

NOTE: In order to comply with the DOE QSM version 3, results are acceptable for any individual compound if the %D is ≤ 20%. The use of the grand mean is not acceptable. (Refer to Policy DV-QA-024P.)

9.19.2.2 In addition, the retention times are updated with each continuing calibration standard.

9.19.2.3 For any analyte detected above the method detection limit, the result for that analyte in the preceding CCV must have a %D of ≤ 20% on the column used for quantitation (program specific requirements may be more stringent see SOP DV-QA-024P).

9.19.2.4 Second column confirmation is performed using the Luna Phenyl Hexyl column.

9.19.2.5 It is not necessary to run a CCV standard at the beginning of the sequence if the first 10 samples are analyzed immediately after the completion of the initial calibration.

9.19.2.6 The last sample in the sequence must be followed by an ending CCV standard. The ending CCV serves the analyst in judging the validity of the sequence.

9.19.3 Corrective Actions for Continuing Calibration:
If the percent difference for any analyte falls outside of ± 20%, corrective action must be taken. This may include back flushing the column, changing the frit on the front end of the column, or other minor instrument adjustments, followed by reanalyzing the standard. If the response for any analyte still varies by more than 20%, a new calibration curve must be prepared and analyzed.

9.19.4 Corrective Action for Samples
Reported sample results must be bracketed by successful CCVs. When a CCV fails, all samples run since the last successful calibration verification must be reanalyzed.

10.0 Procedure

- One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision 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 is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.
- Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.1 Extraction of Water Samples - *Please reference DV-OP-0017 for details.*

10.1.1 Samples are extracted using a 500 mL initial volume and are concentrated to a 5 mL final volume.

10.2 Extraction of Soil Samples – *Please reference DV-OP-0018 for details.*

10.2.1 Samples for method 8330A are extracted using a 2 g initial weight and a 10 mL final volume. Samples for method 8330B are extracted using a 10 g initial weight and a 20 mL final volume.

10.2.2 Prior to analysis, dilute the extract, or a portion of the extract, exactly 1:1 with the calcium chloride solution that is described in section 7.8.8. This is normally done by the instrument analyst.

10.3 HPLC Analysis

10.3.1 Analyze the samples using the chromatographic conditions given in Appendix 4. All positive measurements above the method detection limit observed on the primary Phenomenex ODS column are confirmed by injection onto the confirmation Luna Phenyl Hexyl column.

10.3.2 Analytes are introduced by direct injection of the extract. Samples, standards, and QC samples must be introduced using the same procedure.

10.3.3 Analytical Sequence

10.3.3.1 The analytical sequence starts with an initial calibration or a Continuing Calibration Verification (CCV).

10.3.3.2 The CCV includes analyzing standards that contain all analytes, and updating the retention time windows. See Section 9.19.2 for details.

10.3.3.3 If there is a break in the analytical sequence greater than 12 hours, a new CCV standard must be analyzed before proceeding with the sequence.

10.3.4 Retention Time Windows

10.3.4.1 Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day for a three-day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). The width of the retention time window for each analyte is defined as ± three times the standard deviation.

10.3.4.2 The chromatograms in Appendixes 5 and 6 summarizes the estimated retention times on both the Phenomenex and Luna columns for many of the compounds analyzed using this method.

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

10.3.4.4 If the retention time window, as calculated above, is less than ± 0.07 minutes, use ± 0.07 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix. The absolute retention time windows are closely monitored and adjusted based on each calibration verification standard. This minimizes the possibility of false positive and false negative qualitative identifications.

10.3.4.5 The laboratory must calculate new retention time windows each time a new column is used. 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, but, updated with the retention times from the new initial calibration.

10.3.5 Corrective Action for Retention Times

10.3.5.1 The retention time window for all compounds must be updated from each CCV. All samples analyzed after the last compliant standard must be reanalyzed unless the following

conditions are met for any compound that elutes outside the retention time window:

10.3.5.2 The retention time of that compound in the standard must be within the retention time range equal to twice the original window.

10.3.5.3 No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

10.3.6 Daily Retention Time Windows

The center of the retention time window is adjusted to the retention time of each analyte, as determined in the each CCV.

10.3.7 Calibration Range and Dilutions

10.3.7.1 If the concentration of any analyte exceeds 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. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for the analyte(s) that were found to be over the calibration range in the high sample, they must be reanalyzed to rule out carryover. It may also be necessary to dilute samples because of matrix interferences.

10.3.7.2 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.

10.3.8 Guidance for Dilutions Due to Matrix Interference:

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

10.3.9 Reporting Dilutions

Some programs (e.g., South Carolina and AFCEE) and some projects require reporting of multiple dilutions (check special requirements in LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported. Record the resulting peak sizes in peak area units. Calculation of concentration is covered in Section 11.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

11.1.1 Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the method detection limit. The required quantitation level is defined as the LIMS test code reporting limit for standard reports, adjusted for initial weight and volume and any dilutions. A UV detector wavelength of 254 nm is used to quantify and report all analytes except PETN and Nitroglycerin that are quantified and reported using a UV detector wavelength of 215 nm.

11.1.2 Identification is confirmed if a peak is also detected within the retention time window on a dissimilar column.

11.2 Second-Column Confirmation

11.2.1 Detection of compounds on the primary column is confirmed using a second column. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, and this column is calibrated using the same calibration levels as the primary. As an option, and as specified in Method 8000, mass spectrometric confirmation may be used. The RPD between confirmed results should agree within 40%.

11.2.2 Regardless of the relative percent difference (RPD) between the responses on the two columns, the higher of the two results is reported unless there is obvious interference documented on the chromatogram. If the RPD exceeds the 40% criteria, the reported result is qualified.

11.2.3 If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

11.2.4 If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

The RPD between two results is calculated using the following equation:

$$\%RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\%$$

Where R1 is the result for the first column and R2 is the result for the second column.

11.3 Average Response Factor

11.3.1 Calibration can be based on the average response factor for an analyte, if the relative standard deviation for the average response factor is ≤ 20%. Average response factor is calculated as follows:

$$\text{Average response factor} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where:

n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

11.3.2 The percent relative standard deviation (%RSD) is calculated as follows:

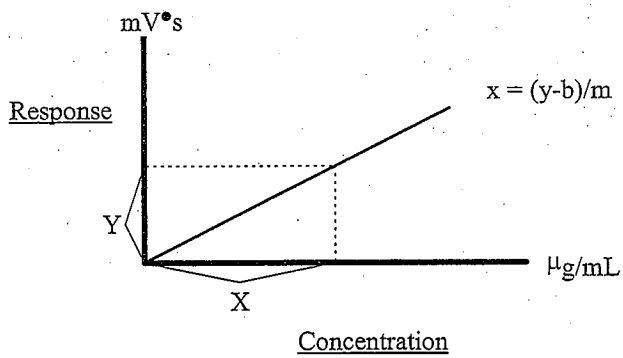
$$\%RSD = \frac{s}{\overline{RF}} \times 100\%$$

11.3.3 Where s is the standard deviation of the average response factor and is calculated as follows:

$$s = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}}$$

11.4 Linear Regression

The linear calibration fit uses the following function (External Standard):



To establish the calibration curve, standards of known concentration are analyzed and the response for each standard is plotted as a function of the concentration of the analyte of interest. Linear regression is used to establish the linear function relating response to concentration in the standard, i.e., y = mx + b, where y is the HPLC response, x is the concentration of the analyte in the standard (µg/mL), m is the slope of the calibration curve and b is the intercept. To determine the concentration of an unknown sample extract, the equation is solved for x, as follows:

$$x = \frac{y - b}{m}$$

Where:

b = The intercept of the calibration curve.
m = The slope of the calibration curve.
y = The HPLC response for the analyte in the sample extract.
x = The concentration of the analyte (µg/mL) in the sample extract injected into the HPLC.

11.5 % Difference Calculation for CCV Evaluation

The percent difference for the analysis of a CCV standard is calculated as follows:

$$\%\ \text{Difference} = \left(\frac{\text{Expected Value} - \text{Measured Value}}{\text{Expected Value}} \right) \times 100\%$$

11.6 Concentration in Aqueous Samples

The concentration of analyte in the original aqueous sample is calculated as follows:

$$C_s = C_i \times \frac{V_t}{V_s} \times DF$$

Where:

C_s = Concentration of analyte in sample (µg/L)
Ci = Concentration of analyte in the extract injected into the HPLC (µg/mL)
Vt = Volume of total extract (mL)
Vs = Volume of sample extracted (mL)
DF = Dilution factor

11.7 Concentration in Non-aqueous Samples

The concentration of analyte in the original non-aqueous sample is calculated as follows:

$$C_s = C_i \times \frac{V_T}{W} \times S \times DF$$

Where:

Cs = Concentration of analyte in sample (µg/g)
Ci = Concentration of analyte in the extract injected into the HPLC (µg/mL)
VT = Total volume of original extract in mL. For method 8330A this is normally 10 mL. For method 8330B this is normally 20 mL.
W = Weight (mass) of sample extracted in g (normally this is 2 g).
S = Factor (usually 2) to account for the 1:1 dilution of the final extract as described in Section 10.2.2.
DF = Dilution factor, as appropriate.

12.0 Method Performance

12.1 Initial Demonstration of Capability

12.1.1 An initial demonstration of capability for each method must be performed prior to analyzing samples.

12.1.2 For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study (described in Section 12.2 below).

12.1.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples.

12.1.4 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 successful analysis of an extracted standard at the reporting limit and a single point calibration.

12.2 Method Detection Limit (MDL)

A valid method detection limit (MDL) study for each analyte of interest must be performed prior to analyzing samples for the first time and annually thereafter. Separate soil MDL studies are performed for 8330A using 2 g and 8330B using 10 g of Ottawa Sand. The procedure for determining detection limits is defined in Policy DV-QA-005P.

12.3 Analyst Training and Qualification

12.3.1 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 experience. See SOP DV-QA-0024 for details of training requirements.

12.3.2 Each analyst performing the method must complete an initial demonstration of capability (IDOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

13.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. When reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Flammable solvent waste – Waste Stream C

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.2 Method 8330, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 0, September 1994.

15.3 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.4 Method 8330A, Nitroaromatics and Nitramines by High Performance Liquid Chromatography, Revision 1, January 1998.

15.5 Method 8330B, Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography, Revision 2, October 2006.

16.0 Method Modifications:

16.1 Deviations from Method Source and Rationale

Method 8330 prescribes the shelf life for standards as follows:

Standards	Concentration	Shelf Life
Stock standards	1,000,000 µg/L (1,000 ppm)	One year
Intermediate standards	2.5 to 1,000 µg/L	Thirty days
Working standards	1 to 500 µg/L	Daily

16.2 This SOP describes the use of 100,000 µg/L high-level standards, which are assigned a six-month shelf life based on TestAmerica's experience with these materials. Further, a 20,000 µg/L standard mix is characterized as an intermediate-level, and assigned a thirty-day shelf life.

16.3 Acidic water (pH < 3) is added to the concentrated extract, dilutions, and all calibration and check standards in place of reagent water. This is to preserve any Tetryl present in the extract.

17.0 Attachments

Appendix 1. Analyte List
Appendix 2. Suggested Calibration Levels (µg/mL)
Appendix 3. Spike Levels
Appendix 4. Suggested Instrument Conditions
Appendix 5. Example Chromatogram from Primary Column
Appendix 6. Example Chromatogram from Confirmation Column

18.0 Revision History

- Revision 11, dated 10 April 2009
  - The details on the extraction procedures have been removed from this SOP. These details can be found in DV-OP-0017 and DV-OP-0018
  - The table in Section 1.4 was revised to include 3,5-dinitroaniline
  - Section 6.2 was revised to include a 500 µL re-pipettor. The pipettor is used to perform the 2X dilution of the soil acetonitrile extracts with the calcium chloride solution.
  - Section 7.8.9 Eluents for Standard Analytes was revised to correct the confirmation column eluent. The eluent is made from methanol and reagent water. Acetonitrile is not used in this reagent.
  - Section 7.8.10 Eluents for Picric Acid was revised to describe more clearly exactly how this reagent is brought up to final volume.
  - Section 7.8.12 Working Eluent Confirmation Column was revised to correct the volume of primary stock solution and glacial acetic acid used in this reagent.
  - Section 9.8 *Method Blank* and Section 9.13 *Grinding Blank* were revised to reflect the laboratory's current practice of controlling method blanks to ½ the reporting limit, or 1/10 of the concentration found in the samples.
  - Section 9.18.3 was revised to state the intercept should be less than the MDL, but at least less than ½ the RL.
  - Section 10.3.1 was revised to state that all detections on the primary column above the method detection limit are confirmed.
  - Section 10.3.3 was revised to clarify that a sequence can either start with an ICAL or a CCV.
  - Section 12.1 *Initial Demonstration of Capability* was revised to eliminate the need for a QC check sample that contains all analytes to be processed through the drying and grinding procedure. The laboratory has performed PT samples that have gone through the grinding process described in method 8330B, but per the PT instructions the sample was not dried prior to grinding.
  - Appendix 1 was revised to remove the approximate retention times from the table. The retention times are updated from each CCV. See Sections 10.3.4 through 10.3.6 for details. This Appendix was also revised to indicate that 3,5-dinitroaniline is only spiked when specifically requested.
  - Appendix 4 was revised to correct the length of the primary column and the column parameters for both primary and confirmation column.
  - Appendix 5 *Organic Extractions Checklist* was removed and replaced with *Example Chromatogram from Primary Column*
  - Appendix 6 *Explosive Soil Extraction Checklist* was removed and replaced with

Example Chromatogram from Confirmation Column

- Revision 10, dated 01 October 2008
  - Re-formatted SOP
  - Updated reference section to reflect the SW-846 update IV method 3535A.
  - Updated all reference to method 3535 to method 3535A.
  - Added 3,5-dinitroaniline as a spike compound for method 8330B
  - Revised the extraction of water samples so that the SPE cartridge is eluted directly into the extract storage vial.
  - Added clarification and requirements for the storage of the working level LCS and surrogate standards.
  - Revised section 9.13.1 to have the grinding blanks associated with the sample that is ground after the blank instead of before the blank in order to ensure that any contamination could be more easily traced.
- Changes from Previous Version
  - The type of SPE cartridge was changed in order to achieve better recoveries of picric acid.

Appendix 1. Analyte List

Compound	CAS #	Symbol
Octahydro-1,3,5,7-tetranitro-1,3,5,7,-tetrazocine	2691-41-0	HMX
Hexahydro-1,3,5-trinitro-1,3,5-triazine	121-82-4	RDX
1,3,5-Trinitrobenzene	99-35-4	1,3,5-TNB
1,3-Dinitrobenzene	99-65-0	1,3-DNB
Methyl-2,4,6-trinitrophenyl nitramine	479-45-8	Tetryl
Nitrobenzene	98-95-3	NB
2,4,6-Trinitrobenzene	118-96-7	2,4,6-TNT
4-Amino-2,6-dinitrotoluene	19406-51-0	4-Am-DNT
2-Amino-4,6-dinitrotoluene	35572-78-2	2-Am-DNT
2,6-Dinitrotoluene	606-20-2	2,6-DNT
2,4-Dinitrotoluene	121-14-2	2,4-DNT
2-Nitrotoluene	88-72-2	2-NT
4-Nitrotoluene	99-99-0	4-NT
3-Nitrotoluene	99-08-1	3-NT
Nitroglycerin	55-63-0	NG
PETN	78-11-5	PETN
2,4-Diamino-6-nitrotoluene**	6629-29-4	--
2,6-Diamino-4-nitrotoluene**	59229-75-3	--
Picric Acid	88-89-1	PA
3,5-Dinitroaniline**	618-87-1	3,5-DNA
1,2-Dinitrobenzene (surrogate)	528-29-0	1,2-DNB

**Non-standard spike analytes. These three compounds are only spiked when specifically requested.

Appendix 2. Suggested Calibration Levels (μg/mL)

Compound	Level 1	Level 2	Level 3	Level 4*	Level 5	Level 6	Level 7	Level 8
HMX	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
RDX	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
1,3,5-Trinitrobenzene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
1,3-Dinitrobenzene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
Tetryl	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
Nitrobenzene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
2,4,6-Trinitrobenzene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
4-Amino-2,6-dinitrotoluene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
2-Amino-4,6-dinitrotoluene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
2,4-Dinitrotoluene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
2,6-Dinitrotoluene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
2-Nitrotoluene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
3-Nitrotoluene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
4-Nitrotoluene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
Nitroglycerin	0.1	0.5	1.0	2.5	5	7	10.	25
PETN	0.1	0.5	1.0	2.5	5	7	10.	25
2,4-Diamino-6-ditrotoluene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
2,6-Diamino-4-nitrotoluene	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
Picric Acid	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5
1,2-Dinitrobenzene (surrogate)	0.01	0.05	0.10	0.25	0.5	0.7	1.0	2.5

* This level is used for the daily and continuing calibration standards.

Appendix 3. Spike Levels

LCS/MS/MSD Spike Levels				
Method and Matrix	**Working Solution**		**Spike Amount**	**Final Concentrations**
	Standard Analytes	**Nitroglycerin & PETN**		**Standard Analytes / Nitroglycerin & PETN**
Water Method 8330A and Method 8330B	1 µg/mL	10 µg/mL	1 mL	2 µg/L / 20 µg/L
Soil Method 8330A and 833B	10 µg/mL	100 µg/mL	0.5 mL for 8330A 1.0 mL for 8330B	2.5 mg/Kg / 25 mg/Kg for 8330A 1.0 mg/Kg / 10 mg/Kg for 8330B
3,5-Dinitroaniline LCS Standard	10 µg/mL		1.0mL for 8330B Soils 0.1mL for 8330B Water	1.0 mg/Kg for 8330B Soils 2 µg/L for 8330B Waters

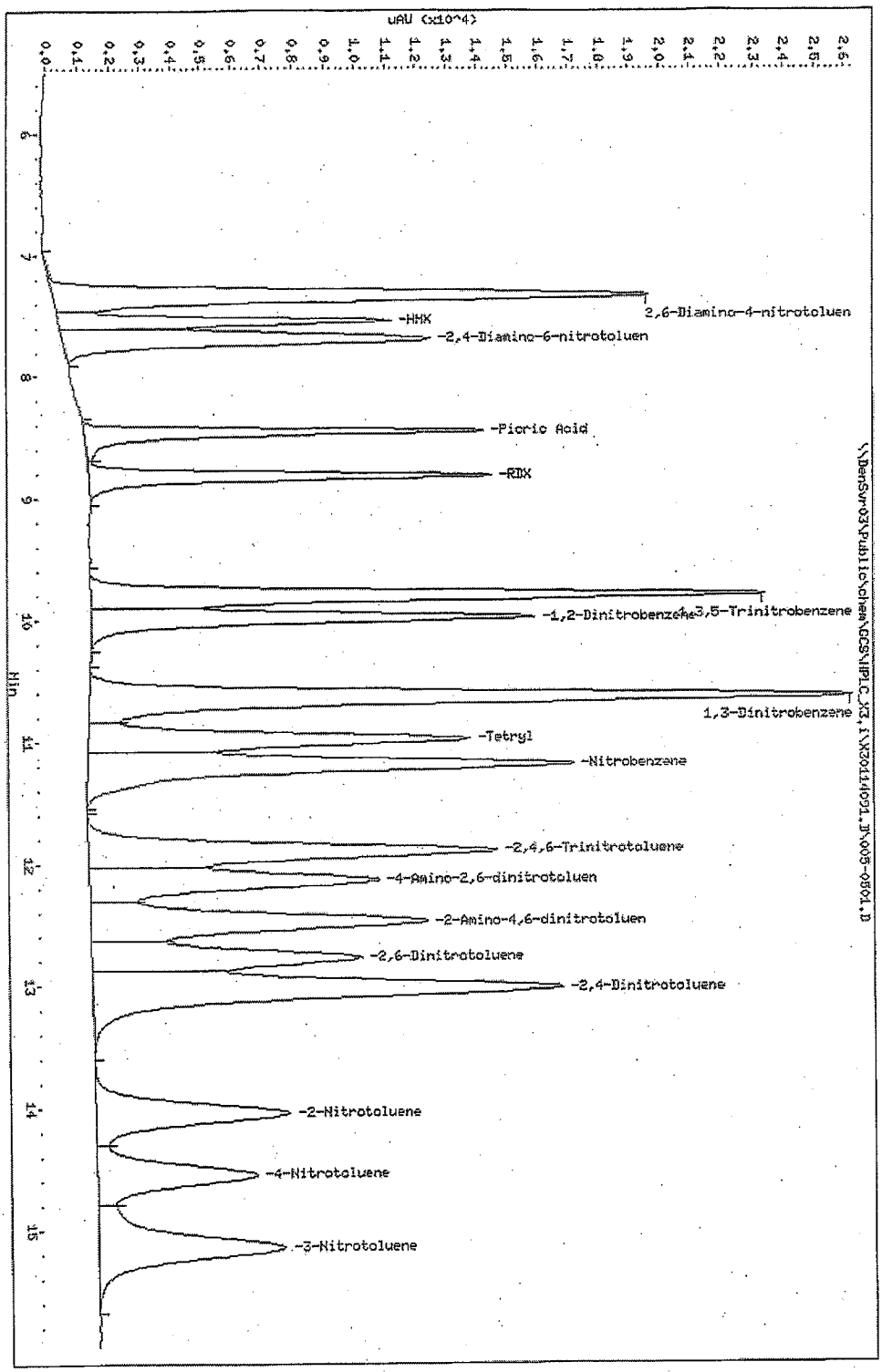
Surrogate Spike Levels			
Method and Matrix	**Working Solution 1,2-DNB**	**Spike Amount**	**Final Concentration**
Water Method 8330A and Method 8330B	1 µg/mL	1 mL	2 µg/L
Soil Method 8330A and 8330B	10 µg/mL	0.5 mL for 8330A 1.0 mL for 8330B	2.5 mg/Kg for 8330A 1.0 mg/Kg for 8330B

Appendix 4. Suggested Instrument Conditions

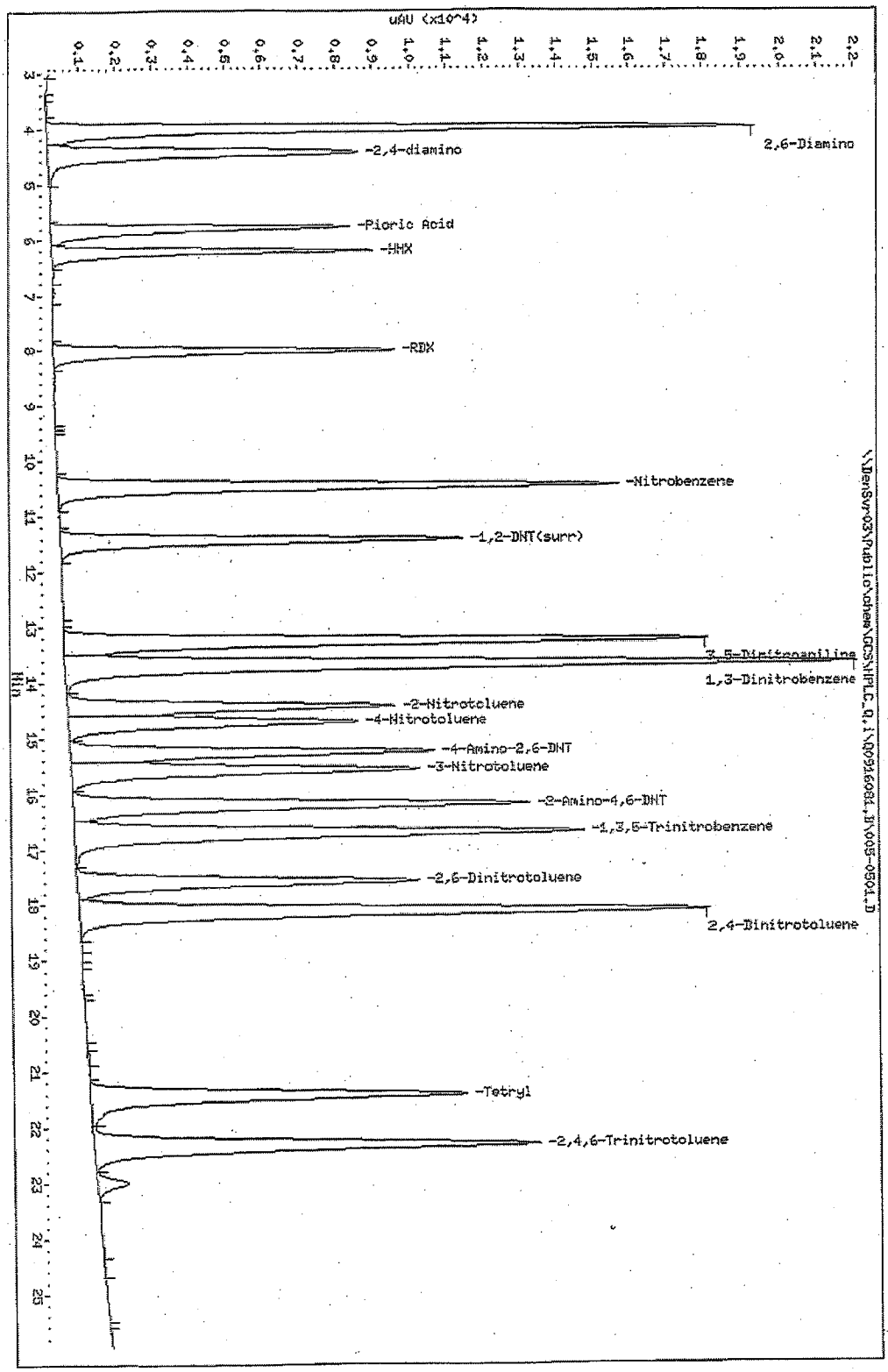
Instrument Conditions		
Column Types	**PRIMARY COLUMN Phenomenex ODS (20) 15 cm x 4.6 mm i.d.**	**CONFIRMATION COLUMN Luna Phenyl Hexyl 15 cm x 4.6 mm i.d.**
Detector - 1st Channel	uv 254 nm, 40 R 550 nm	
Detector - 2nd Channel	uv 215 nm, 40 R 450 nm	
General Parameters	Injection Volume: 100 µL	Temperature: 24.3°C Ambient

Suggested Column Parameters						
	Stop Time (min.)	**Post Time (min.)**	**Flow Rate (mL/min.)**	**Time (minutes)**	**% H_2O**	**% Methanol**
Gradient Column 1	16.0	3.0	1.0	0.0, 2.0, 5.76, 14.4, 15.0	90, 90, 40, 40, 90	10, 10, 60, 60, 10
Gradient Column 2	26.0	4.0	0.8	0.0, 26.0, 30.0	50, 25, 50	50, 75, 50

Appendix 5. Example Chromatogram from Primary Column



Appendix 6. Example Chromatogram from Confirmation Column



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TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

ICP Analysis for Trace Elements by SW-846 Method 6010B

Approvals (Signature/Date):

Doug Gomer 6/22/09
Doug Gomer Date
Metals Supervisor

Adam W. Alban 22 June 09
Adam Alban Date
Health & Safety Manager / Coordinator

Karen Kuoppala 6-23-09
Karen Kuoppala Date
Quality Assurance Manager

Robert C. Hanisch 6/22/09
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICPAES). This procedure references Method 6010B for hazardous waste (RCRA) testing.

1.2 The elements that can be determined by this procedure are listed in Attachment 1, together with the routine reporting limits. Additional elements may be analyzed under Method 6010 provided that the method performance criteria presented in Section 13.0 are met.

1.3 The laboratory digests water samples according to SOP DV-IP-0010. The methods require digestion of waters, with the following exceptions, i.e.:

- The sample is visibly transparent with a turbidity measurement of 1 NTU or less.
- The sample consists of one liquid phase and is free of particulate or suspended matter following acidification.

1.4 Silver concentrations must be below 1.0 mg/L in aqueous sample digestates and 100 mg/kg in solid matrix sample digestates. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data. Samples with silver concentrations exceeding these levels must be re-prepared and reanalyzed using a smaller sample amount.

1.5 The digestion procedure for soil samples is described in SOP DV-IP-0015.

1.6 State-specific requirements may take precedence over this SOP for drinking water sample analyses. Review special instructions for each project before starting work.

2.0 Summary of Method

2.1 The laboratory uses simultaneous ICPAES instruments, with both axial and radial viewing configurations. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs.

2.2 Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photo-multiplier tubes or a charge injection device (CID). The photo-currents from the photo-multiplier tubes or a charge injection device (CID) are processed and controlled by a computer system.

2.3 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.

2.4 Refer to the appropriate SOPs for details on sample preparation methods: DV-IP-0010 for aqueous samples, and DV-IP-0013 for soil samples.

3.0 Definitions

3.1 Trace ICP – in this SOP, an ICP with the viewing angle along the long axis of the torch (axial ICP) is referred to as a Trace ICP.

3.2 Dual View ICP – an ICP equipped with both radial and axial viewing capabilities.

3.3 Dissolved Metals - Those elements which pass through a 0.45-μm membrane. (The sample is acidified after filtration).

3.4 Potentially Dissolved Metals - Potentially dissolved metals is the concentration of metals in solution after acidifying the sample with nitric acid to pH <2, holding at room temperature for 8 to 96 hours, and then filtering through a 0.45-μm membrane filter. This definition is based on the Colorado surface water regulations.

3.5 Suspended Metals - Those elements which are retained by a 0.45-μm membrane.

3.6 Total Metals - The concentration determined on an unfiltered sample following vigorous digestion.

3.7 Total Recoverable Metals - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

3.8 Reporting Limit (RL) - The lowest concentration to which results are reported without qualification. Details concerning RLs are presented in Policy QA-009.

4.0 Interferences

4.1 Spectral, physical, and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by the following:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.2 A background correction technique is used 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.

4.3 Spectral Interferences

4.3.1 Inter-element correction factors (IECs) 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 signal. 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 corrections must be applied to the analyte to compensate for the effects of these unwanted emissions.

4.4 Physical Interferences

4.4.1 An internal standard (IS), yttrium or other suitable element, is added to all solutions to correct and monitor physical interferences. Use of a peristaltic pump and the mass flow controller also help to overcome physical interferences. Physical interferences are generally considered to be effects associated with sample transport, nebulization, 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 dissolved solids or high acid concentrations. If internal standard recoveries are not acceptable (see Section 0), then dilution of the sample may be necessary to overcome the interferences. Where the use of an internal standard might actually degrade the accuracy of the analytical result, sample results may be reported without IS correction.

4.5 Chemical Interferences

4.5.1 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.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual 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, nitrile or latex 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 nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
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.
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.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Thermo Jarrell Ash ICP 61E Trace Analyzer and Thermo Fischer ICP 6500E Trace Analyzer are currently used. Instruments with demonstrated equivalent performance can also be used

6.1.2 Radio Frequency Generator.

6.1.3 Argon gas supply, welding grade or equivalent.

6.1.4 Coolflow or appropriate water-cooling device.

6.1.5 Peristaltic Pump.

6.1.6 Autosampler.

6.2 Supplies

6.2.1 Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.2.2 Class A volumetric flasks.

6.2.3 Autosampler tubes.

7.0 Reagents and Standards

7.1 Shelf-Life

7.1.1 Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, then a one-year expiration will be assigned by the laboratory.

7.1.2 The expiration date of intermediate concentration standards or working standards cannot be later than the date assigned to any of the stock standards used to prepare the intermediate solution.

7.1.3 If visible deterioration is noted for any standard, it must be re-verified against a second-source. Any standard that does not verify must be replaced immediately.

7.2 Standards

7.2.1 Standards used for calibration and quality control purposes must be NIST traceable, where available. Multi-component custom blend standards must be verified against a second-source standard before they are put into use (the only exception is standards purchased directly from NIST), as described in SOP DV-QA-0015.

7.2.2 Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon,

polyethylene, or polypropylene bottles. Silver standards must be protected from light. The preparation frequency is governed by the parent standard with the earliest expiration date unless specified otherwise in this SOP. Detailed instructions regarding the preparation of standards and reagents are given in this section. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the Standards Log database.

7.2.3 Calibration and QC standards are prepared in water with hydrochloric and nitric acids in order to approximate the acidic matrix of the various digests analyzed. This is an important point. Even with the use of yttrium as an internal standard, deviations from these concentrations can cause physical effects, as discussed in Section 4.4 of this procedure.

7.3 Reagent Blank / Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

7.3.1 Fill a 20-liter carboy with about 18 liters of reagent water. Slowly add the appropriate amount of concentrated HNO_3 and concentrated HCl. Mix carefully.

7.4 Stock ICSA and ICSAB Standards

The following standards are purchased from commercial sources:

Stock ICSA & ICSAB Standard	Elements	Concentration (mg/L)
ICSA Std	Fe Al, Ca, Mg	2,000 5,000
ICSAB Std	Ba, Be, Co, Cr, Cu, Mn, V Ag, Cd, Ni, Pb, Zn	50 100
ICSAB 1	Li, Mo, Sb, Sr As, B, P Se K, Na	100 200 500 5000
ICSAB 1B	Tl	1,000
ICSAB 2	Ti Sn	100 1,000
10,000 Si	Si	10,000
Th	Th	1,000
U	U	1,000
Zr	Zr	1,000
S	S	1,000
Bi	Bi	1,000

7.5 ICSA Working Standard

A combined working ICSA standard is made in a 250-mL volumetric flask using the following volumes of the Stock ICSA and ICSAB Standards:

Stock Standard	Volume of Stock Added (mL)
ICSA Std	25
1,000 mg/L U Standard	0.5

Adjust to volume (250 mL) using the reagent blank solution. This produces the final ICSA standard concentrations shown in Attachment 5.

7.6 ICSAB Working Standard

A combined working ICSAB standard is made in a 250-mL volumetric flask using the following volumes of the Stock ICSAB Standards:

Combined Working ICSAB Standard

Stock Standard	Volume of Stock Added (mL)
ICSA Std	25
ICSAB Std	2.5
ICSAB 1	2.5
ICSAB 1B	2.5
ICSAB 2	2.5
10,000 mg/L Si	0.25
1,000 mg/L S	0.25
1,000 mg/L Th	0.5
1,000 mg/L U	0.5
1,000 mg/L Zr	0.25
1,000 mg/L Bi	0.25

Adjust to volume (250 mL) using the reagent blank solution. This produces the final ICSAB standard concentrations shown in Attachment 5.

7.7 High Calibration Check Standard

The high concentration check standard is the same as the Working ICAL Standards.

7.8 Laboratory Control Sample (LCS) Stock Standards

The LCS stock standards are purchased from commercial sources. The stocks are custom-made standards purchased at ready-to-use concentrations as follows:

LCS STOCK STANDARD	ELEMENTS	CONCENTRATION (MG/L)
ICP Prep Spike #3A	Ca, K, Mg, Na	5,000
	P	1,000
	Al, As, Ba, Se, Th, Tl, U, Bi	200
	Fe, Sr,	100
	Co, Mn, Ni, Pb, V, Zn	50
	Cu	25

	Cr Ag, Be, Cd	20 5
ICP Prep Spike #2	Sb, Zr B, Mo, Ti Sn Si (SiO_2)	50 100 200 1000 (2140)
Sulfur	S	200

Soil Batches – LCS spikes for soil batches are prepared by adding 1.0 mL of the LCS to a digestion tube containing 5 mL of reagent water. The AFCEE program requires the addition of 1 g of glass beads to the digestion tube.

Water Batches – LCS spikes for water batches are prepared by adding 0.5 mL of LCS Stock Standard to a digestion tube containing 50 mL of reagent water.

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

The same LCS stock standards described in the previous section, 7.8, are also used to prepare matrix spikes. The same media and spike concentrations are used as well.

7.10 Post Digestion Spike (PDS) Standards (Analyte Addition Spike Standards)

The custom standards tabulated below are purchased from a commercial source. Add 0.1 mL of each to 10 mL (100X) of digestate or dilution of digestate.

PDS Stock	Elements	Conc. (mg/L)
PDS 1	Ag, Be, Cd, Co, Cr, Cu, Mn, Ni, Sr, V Ba, Pb, Li As, Se, Tl, Zn, Th U Al, Fe P Ca, Mg, Na, K	5.0 10 20 50 100 200 2,000
PDS 2	Mo, Ti, Zr B, Sb, Sn Si	5.0 10 500

7.11 Initial Calibration (ICAL) Standards for the Axial ICP Instruments

7.11.1 Stock Calibration Standards

The following seven stock mixes are purchased from commercial sources.

Stock Calibration Standard	Elements	Conc (mg/L)
Cal STD 1	B, Mo, Sb, Ti Sn	100 1,000
Cal STD 2	Ag, Ba, Be, Cd, Co, Cr, Mn, Ni, Pb, Sr, V, Zn, Cu As	100 200
Cal STD 3	Al, Fe, Li, P, Se, Tl	1,000
Cal STD 4A	Ca, Mg K Na	1,000 2,500 10,000
Cal STD 4B	Si	10,000
U	U	1,000
Th	Th	1,000
S	S	1,000

7.11.2 Working ICAL Standard for Axial ICP

A combined working ICAL standard is prepared in a 500-mL volumetric flask using the following volumes of the Stock Calibration Standards. The preparation frequency is governed by the parent standard with the earliest expiration date.

Working ICAL Standard for Axial ICP

Stock Standard	Vol. of Stock Added (mL)
Cal STD1	5
Cal STD 2	5
Cal STD 3	5
Cal STD 4A	20
Cal STD 4B	5
1,000 mg/L S	5
1,000 mg/L Th	1
1,000 mg/L U	1

Adjust to volume (500 mL) using the reagent blank solution. This produces the final ICAL standard concentrations shown in Attachment 4.

7.12 Initial Calibration Verification (ICV) Stock Standards for the Axial ICP

The ICV stock standards are purchased from a different vendor than is used to supply the ICAL standards. The following five stock mixtures are purchased from commercial sources:

CCV Stock Standard	Elements	Conc (mg/L)
ICV 1A	B, Mo, Sb, Ti, Sn Si	100 10,000
ICV 2A	Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Sr, V, Zn P Al, Fe, Li, Se, Tl	100 400 1,000
ICV 3A	Ca, Mg K Na	1,000 2,500 10,000
U	U	1,000
Th	Th	1,000
S	S	10,000

7.12.1 ICV Working Standard for Axial ICP

A combined working ICV standard is made in a 200mL volumetric flask using the following volumes of the Stock ICV Standards. The preparation frequency is governed by the parent standard with the earliest expiration date.

Stock ICV Standard	Vol. of Stock Added (mL)
ICV 1A	0.5
ICV 2A	0.5
ICV 3A	2.0
10,000 mg/L S	0.04
1,000 mg/L Th	0.6
1,000 mg/L U	0.6

Adjust to volume (200 mL) using the reagent blank solution. This produces the final ICV standard concentrations shown in Attachment 4.

7.12.2 CCV Standard for Axial ICP

A combined working CCV standard is made in a 500-mL volumetric flask using the following volumes of the Stock Calibration Standards. The preparation frequency is governed by the parent standard with the earliest expiration date.

Stock Standard	Vol. of Stock Added (mL)
Cal STD1	2.5
Cal STD 2	2.5
Cal STD 3	2.5
Cal STD 4A	10
Cal STD 4B	2.5
1,000mg/L Th	0.5
1,000mg/L U	0.5
1,000 mg/L S	2.5

Adjust to volume (500 mL) using the reagent blank solution. This produces the final CCV standard concentrations shown in Attachment 4.

7.13 Reporting Limit (RL) Verification Stock Standard for the Axial ICP

The reporting limit verification stock standards are custom-made commercial standards as follows:

RL Stock Standard	Elements	Conc. (mg/L)
RL STD 1	Li, Ni, Se, Sr, Tl, V, Ag, As, Cr, Cu	10
	Zn	20
	Al, Fe	100
	Ca, Mg	200
	K, Na	1,000
	P	3,000
RL STD 2	Mo	15
	Sn	50
	Si	500
	B	100
	Sb, Ti	10
Solution 2	Pb	30
	Be	40
	Ba, Cd, Co, Mn	50

7.13.1 RL Verification Standards, Intermediate Concentrations

The intermediate level RL standard solutions are prepared weekly as follows:

Weekly RL Solution 2

Standard	Vol. of Stock Added (mL)	Final Volume (mL)
Solution 2	1	10

Th / U / S Intermediate Standards

Standard	Vol. of Stock Added (mL)	Final Volume (mL)
1,000 mg/L Th	1.0	100
1,000 mg/L U	6.0	100
1,000 mg/L S	10	100

Adjust to volume using the reagent blank solution.

7.13.2 RL Verification Standard, Daily Working Standards

The working level RL standards are prepared daily in a 100-mL volumetric flask as follows. See Attachment 1 for concentrations.

RL Standard	Vol. of Stock Added (mL)
RL Stock Standard #1	0.1
Weekly RL Standard #2	0.1
10 mg/L Th Standard	0.1
60 mg/L U Standard	0.1
100 mg/L S Standard	0.1
RL Standard #2	0.1

7.14 Initial Calibration (ICAL) Standards for the Dual View ICP

7.14.1 Stock Calibration Standards

The following stock solutions are purchased from commercial sources.

Stock Standard	Elements	Conc. (mg/L)
STLDEN-STD-2	Mo, Ti, Zr Sn Si	100 200 1,000
STLDEN-STD-3B	Ag, Al, B, Ba, Cd, Co, Cr, Cu, Be, Mn, Ni, Sr, V, Zn Li, P Ca, Na Mg K Fe	100 200 1,000 4,000 10,000 500

Stock Standard	Elements	Conc. (mg/L)
Al, Ca, Fe, Na, S Stocks	Al, Ca, Fe, Na, S	10,000
As, Pb, Sb, Se, Tl, Th, U, Bi Stocks	As, Pb, Sb, Se, Tl, Th, U, Bi	1,000

7.14.2 Working Initial Calibration Standard (ICAL1) for Dual View ICP

Add 5.0 mL each of STLDEN-STD-2 and STLDEN-STD-3B to a 500-mL volumetric flask partially filled with reagent blank solution. Add 1 mL of the As, Pb, Sb, Se, Tl, stock. Dilute to the mark with reagent blank.

7.14.3 Working Initial Calibration Standard (ICAL2a) for Dual View ICP

Add 10 mL of each of the Al, Fe, and 50 mL Na 10,000 mg/L stock solutions; 10 mL each of the Th and 20 mL of the U 1,000 mg/L stock solution; 2ml of 1000mg/l Bi; 1 mL of 10,000 mg/L Sulfur to a 1,000-mL volumetric flask partially filled with reagent blank and dilute to the mark with reagent blank.

7.15 Initial Calibration Verification (ICV) for Dual View ICP

7.15.1 ICV Stock Standards

The following stock solutions are purchased from commercial sources:

Stock Standard	Elements	Conc. (mg/L)
ICVL SOL A	Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Li, Mn, Ni, Pb, Sr, V, Zn Se, Tl Ca, Na Mg K	25 25 50 200 1,000 2,000
ICVL SOL B	Ag, Mo, Sb, Ti, Zr Sn Si P	25 50 200 200
ICVH Stock	Al, Ca, Fe,Na U, Th	4,000 500
Sulfur	S	1000
Bismuth	Bi	1000

7.15.2 Working High Initial Calibration Verification (ICVH)

Add 2.0 mL of the ICVH Stock, 0.1ml Bi and 0.8 mL of the Sulfur to a 200 mL volumetric flask partially filled with reagent blank solution and dilute to the mark.

7.15.3 Working Low Initial Calibration Verification (ICVL)

Add 2.0 mL of each of the ICVL SOL A and ICVL SOL B stock solutions to a 200-mL volumetric flask partially filled with reagent blank solution and dilute to the mark.

7.16 Reporting Limit Standard (RLSTD) for the Dual View ICP

7.16.1 RL Stock Standard

The following stock solutions are purchased from commercial sources:

Standard	Elements	Conc. (mg/L)
STLDEN-RL-1A	As, Sb, Se, Tl Pb	10 3.0
STLDEN-RL-2	Si Sn Mo, Ti, Zr	500 20 10
STLDEN-RL-3	Ag, Cr, Cu, Ni, Th, V, Zn, Li Al, B Ba, Cd, Co, Sr Be Ca, Mg Fe K, Na, P Mn U	10 100 5.0 1.0 200 30 1,000 3.0 60
Sulfur	S	100
Bismuth	Bi	100

7.16.2 Daily Working Reporting Limit Standard (RLSTD2 or RLSTD3)

Add 100 µL of each of STLDEN-RL-1A (optional for RLSTD2), STLDEN-RL-2, STLDEN-RL-3, Bismuth 100mg/L and Sulfur 100 mg/L to a 100-mL volumetric flask partially filled with reagent blank and dilute to the mark. Working RL standards must be prepared fresh each day.

7.17 Working High Continuing Calibration Verification (CCVH1) for Dual View ICP

Dilute 500 mL of the working ICAL2 solution (Section 7.14.3) to 1,000 mL with reagent blank solution.

7.18 Working Low Continuing Calibration Verification (CCVL1) for Dual View ICP

Dilute 500 mL of the working ICAL1 solution (Section 7.14.2) to 1,000 mL with reagent blank solution.

7.19 Reagents

7.19.1 Concentrated nitric acid (HNO_3), trace metals grade or better.

7.19.2 Concentrated hydrochloric acid (HCl), trace metals grade or better.

7.19.3 Reagent water must be produced by a Millipore DI system or equivalent, with a minimum resistivity of 1.0 Mohm/cm at 25°C.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50 mLs	HNO_3, pH < 2; Cool 4 ± 2°C	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool 4 ± 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

The exception is the analysis of dissolved silica by Method 200.7, which must be analyzed within 28 days from the date of collection.

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 are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. Initial and periodic performance studies (IDOCs, detection limits, linear range studies, IECs, background correction points, and rinse time determinations) are described in Section 13. The process of establishing control limits, and the use of control charts are described more completely in QA-003, Quality Control Program. Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents.

9.2 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See Policy DV-QA-003P for further details.

9.4 Method Blank

The blank is de-ionized water taken through the procedure as if it were a sample. A method blank is required with every batch of 20 or less samples.

Acceptance Criteria: The method blank must not contain any analytes of interest above the reporting limit or above one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL). Note that the DoD QSM and AFCEE 4.0 QAPP require that the blank be less than one half of the RL.

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be redigested and reanalyzed. A possible exception is the situation in which the analyte is not detected in any of the associated samples, but this can only be done with client approval and it must be addressed in the final report case narrative.

9.5 Laboratory Control Sample (LCS)

The LCS is prepared as described in Section 7.8. One LCS is required with each analytical batch.

Acceptance Criteria: The recovery of the LCS must be within historical control limits. Historical control limits are based on three standard deviations of past results, and must be 80-120% or tighter. In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the report narrative. The process of establishing control limits is described in more detail in the Policy DV-QA-003P. The control limits are stored in the lab's LIMS system.

Corrective Action: If the LCS recovery falls outside of the established limits, all associated samples must be redigested and reanalyzed

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

MS/MSDs are prepared as described in Section 7.8. One MS/MSD pair is required with each analytical batch. Note that some programs (e.g., North Carolina and South Carolina) require the MS/MSDs to be run at a 10% frequency. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. 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, these results may have immediate bearing on only the specific sample spiked.

Samples identified as field blanks cannot be used for MS/MSD analysis. Note that if client instructions on the chain of custody form tell the lab to use a field blank for the MS/MSD, this should be double-checked with the laboratory PM.

Acceptance Criteria: The recoveries for the MS and MSD must be within the historical control limits or the project-required control limits, whichever are appropriate. Historical control limits are based on three standard deviations of past results, and should be within the established project-specific method control limits, if they exist. The process of establishing control limits is described in more detail in Policy QA-003. The control limits are stored in the laboratory's LIMS system. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are flagged "MSB"

Corrective Action: If MSD/MSD recoveries fall outside of the established limits and the LCS is in control, the data will be flagged as outside of control limits. Document the results, which are then used by the lab PM to prepare the case narrative to warn the client that the sample result is suspect.

Acceptance Criteria: The relative percent difference (RPD) between the MS and MSD must be less than or equal to the historical RPD control limit. Historical control limits are based on three standard deviations of past results, and must be no greater than 20%.

Corrective Action: If the RPD fails to meet precision limit and the recoveries pass, the control limits should be checked as this would be a very rare occurrence if the limits are set properly. If the LCS is in control, it indicates long-term precision, and precision failures within the batch may be due to sample non-homogeneity. MS/MSD results which fall established control limits must be addressed in the narrative. Document the result, which is then used by the lab PM to prepare the case narrative.

9.7 Serial Dilution Test

A dilution test is performed for each batch of samples. The purpose of this test is to ensure that neither positive or negative interferences are biasing the analytical results. The serial dilution test should be performed on the same sample used to perform the MS/MSD.

Acceptance Criteria: If the analyte concentration is sufficiently high (minimally, a factor of 50 times the MDL), an analysis of a 1+4 dilution (e.g., 1 mL of sample diluted to 5 mL with reagent blank solution) must agree within ± 10% of the original determination.

Corrective Action: If the two results do not agree within ± 10%, then a chemical or physical interference is suspected. A qualifier flag is assigned to the data and an NCM prepared, which is then used by the lab PM to prepare the case narrative to warn the client the sample result is suspect.

9.8 Post Digestion Spike (PDS)

Whenever a new or unusual sample matrix is encountered, a PDS spike must be performed. The PDS spike is prepared as described in Section 7.10. Some programs, e.g., AFCEE, require a PDS analysis whenever the serial dilution test fails. Other programs, e.g., DoDQSM, require a PDS to be included in every batch. Check project requirements. For these programs, the same sample that was used for the serial dilution test should be used for the PDS.

Acceptance Criteria: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75-125% for Method 6010. The spike addition should produce a minimum level of 10 times to a maximum of 100 times the instrumental detection limit. Some analytes with unusually low IDLs or that are routinely found at elevated concentrations are spiked at levels greater than the suggested limit.

Corrective Action: If the spike is not recovered within the specified limits, a matrix effect is confirmed. The series of tests (MS/MSD, serial dilution, and PDS) should be described in NCMs so that they can be included in the report case narrative.

9.9 Method of Standard Additions (MSA)

This technique involves constructing a calibration curve in the sample matrix itself to compensate for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.

Attachment 11 provides more guidance on performing MSA analyses.

9.10 Interference Check Analysis (ICSA / ICSAB)

The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Sections 7.4, 7.5, and 7.6 for the preparation of the ICSA and ICSAB solutions. Attachment 5 lists the final concentrations. All analytes are spiked into the ICSAB solution. The ICSA and ICSAB solutions are analyzed at the beginning of the run.

Acceptance Criteria: The ICSAB results for the all analytes must fall within 80-120% of the true value. If any ICSAB analyte result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the samples rerun.

The absolute value of ICSA results for the non-interfering elements with reporting limits $\leq$ 10 µg/L must be $\leq$ 2 x RL. The absolute value of ICSA results for the non-interfering elements with RLs > 10 µg/L must be $\leq$ RL.

Corrective action: If the ICSA results for the non-interfering elements do not meet these limits, the field sample data must be evaluated as follows:

- 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.

- If the affected element was not required, then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration which would result in an absolute value > 2 x RL, then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal > 2.x RL, 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.
- If the data do 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.

9.11 Monitoring Internal Standard Results

Yttrium is automatically added as an internal standard (IS) to every solution tested through use of a third pump channel and mixing coil. 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).

Acceptance Criteria: If the internal standard counts fall within ±30% of the counts observed in the ICAL blank (STD1-Blank), then the data are acceptable.

Corrective Action: If the internal standard counts in the field samples are outside of the control limits, the following apply:

- The field samples must be diluted and reanalyzed;
- The IS concentrations must be raised; or
- A different internal standard must be used.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any unauthorized deviations from this procedure must also be documents as a

nonconformance, with a cause and corrective action described.

10.3 Sample Preparation

Solid and aqueous samples must be digested prior to analysis by the appropriate method (see SOPs DV-*IP-0010 and DV-IP-0015*).

10.4 Calibration

10.4.1 Instrument Start Up

Set up the instrument with the operating parameters recommended by the manufacturer. Complete any required preventative maintenance as indicated on the ICPAES Preventative Maintenance Log (see example in Attachment 14).

Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.4.2 Initial Calibration (ICAL)

The calibration curve is established on each day of operation using a blank and one standard. The preparation of the ICAL standards is described in Section 7. The final concentrations of the ICAL standards are presented in Attachments 3 and 4.

The validity of the calibration curve is confirmed by analysis of the ICV, CCV, ICB, and RL Check standards, which are run immediately after the ICAL. Some programs require a high-level verification check as well.

10.4.2 Initial Calibration Verification (ICV)

Calibration accuracy is verified using a second-source standard (ICV) that is at or below a concentration near the mid-point of the working range. The ICV is analyzed immediately after the ICAL. The preparation of this standard is described in Section 7. The concentrations of the ICV standard are presented in Attachments 3 and 4.

Acceptance Criteria: For Method 6010, the ICV result must fall within 10% of the true value for that solution. The standard deviation must be <5% (the laboratory is using at least two exposures for all ICP analyses).

Corrective Action: If the ICV fails to meet acceptance limits, the standard may be reanalyzed without modification to the instrument operating conditions. *Two consecutive, acceptable analyses are required before the analytical run may continue.* Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.4 Continuing Calibration Verification (CCV)

The preparation of the CCV solutions are described in Section 7. The final concentrations of the CCVs are presented in Attachments 3 and 4. Note that the CCV is made at a different concentration than the ICV to meet NELAC requirements. CCVs are analyzed after the ICV, after every ten samples, and at the end of the analytical run.

Acceptance Criteria: The CCV must be within 10% of the expected value to meet Method 6010 requirements. The relative standard deviation must be <5%.

Corrective Action: If the CCV fails to meet any of these criteria, the standard may be reanalyzed without modification to the instrument operating conditions. *Two consecutive, acceptable analyses are required before the analytical run may continue.* Otherwise, the instrument must be recalibrated and the samples reanalyzed since the last successful CCV must be reanalyzed.

10.4.5 Initial Calibration Blank (ICB)

System cleanliness is verified by analyzing an ICB after the first CCV. The preparation of the ICB is described in Section 7.

Acceptance Criteria: The absolute value of the ICB result must be < RL. Note that some programs (e.g., DoDQSM, v3.0) require the blank to be ≤ 2 x MDL.

Corrective Action: If the ICB fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.6 RL Calibration Check Standard (RLSTD)

Calibration accuracy at the RL is verified by analyzing a standard prepared at a concentration at or below the laboratory's standard reporting limit. The preparation of this standard is described in Section 7. The concentrations for the standard are listed in Attachments 3 and 4. Alternate RLSTD concentrations may be used as necessary to meet client requirements as long as an accurate description of the standard used is entered into the Standards Log database.

Acceptance Criteria: For routine work and for programs that allow the RL to be as low as 2 x MDL (e.g., AFCEE), the acceptance limits are ± 50% of the expected value. For some programs (e.g., DoDQSM), the RLSTD needs to be 5 x MDL, and the acceptance limit is then ± 20%.

Corrective Action: If the RL Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.4.7 High-Level Calibration Check Standard

The method 6010 defines the linear working range used for daily analysis based on the LDR studies performed every six months, in which case this standard is not required. However, some programs require verification of the high end of the linear range at different frequencies. For example, the AFCEE QAPP, version 4.0, requires evaluation of a high check standard every three months.

Acceptance Criteria: The result for this standard must be within 10% of the expected value.

Corrective Action: If the High-Level Calibration Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified. Alternately, results that do not exceed the level of the highest calibration standard may be accepted and reported.

10.4.8 Continuing Calibration Blank (CCB)

CCBs, prepared as in Section 7.3, are analyzed after each CCV.

Acceptance Criteria: The absolute value of the CCB must be less than the RL or less than 1/10 the concentration found in associated samples. Note that some programs (e.g., DoDQSM, v3.0) require the blank to be ≤ 2 times the MDL.

Corrective Action: If the CCB is greater than these limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, instrument maintenance should be considered, the calibration re-verified, and all samples analyzed since the last successful CCB must be reanalyzed.

10.5 Sample Analysis

10.5.1 Replicate Readings

The laboratory averages the results from two exposures for Axial and Dual View ICP for each standard, field sample, and QC sample due to sample

volume limitations of the autosampler tube. For Axial ICP analyses, the results of the sum channels for Se, Sb and Pb must be used for reporting.

10.5.2 Rinse Time Between Samples

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 0 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

10.5.3 The following analytical sequence is used:

Instrument Calibration
High Standard Verification
ICV
CCV
ICB
RL Verification Standard
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence with 10 samples between CCV/CCB pairs
CCV
CCB

10.5.4 Full method-required QC must be available for each wavelength used in determining reported analyte results. Guidelines are provided in the appendices for minimizing contamination of samples and standards (Attachment 13), performing preventive maintenance (Attachment 14), and troubleshooting (Attachment 12).

10.5.5 Dilutions for High Levels of Elements of Interest

For 6010, results must fall within the linear range. 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. Dilutions must be prepared using the reagent blank solution to maintain the correct acid strength.

10.5.6 Dilutions for High Levels of Interfering Elements

Dilutions are also required for an element that is included in an IEC calculation if it exceeds the linear range. If a dilution is not performed, 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 interferent for any

requested analyte in that sample, the sample must be diluted to a level at or below the working range.

11.0 Calculations / Data Reduction

ICV percent recoveries are calculated according to the following equation:

$$\%R = \left(\frac{\text{ICV Found Value}}{\text{ICV True Value}} \right) \times 100\%$$

CCV percent recoveries are calculated according to the following equation:

$$\%R = \left(\frac{\text{CCV Found Value}}{\text{CCV True Value}} \right) \times 100\%$$

Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \left(\frac{SSR - SR}{SA} \right) \times 100\%$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equation:

$$RPD = \left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2} \right)} \right] \times 100$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

The final concentration for a digested aqueous sample is calculated as follows:

$$\text{Final Concentration (mg/L)} = \frac{C \times V1 \times D}{V2}$$

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

The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$\text{Final Concentration (mg/kg), dry weight} = \frac{C \times V \times D}{W \times S}$$

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.

The LCS percent recovery is calculated according to the following equation:

$$\%R = \left(\frac{\text{LCS Found Value}}{\text{LCS True Value}} \right) \times 100\%$$

The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

Appropriate factors must be applied to sample values if dilutions are performed.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial MDL study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

Prepare seven standards at three to five times the estimated MDL concentration.

The MDL standards are processed through the entire analytical process, including the digestion.

Calculate the mean concentration found (X) in µg/L, and the standard deviation of the mean concentration in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.

MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy QA-005 for further details concerning MDL studies.

12.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard prepared at 1-3 times the calculated MDL concentration. An MDLV standard is analyzed immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

Acceptance Criteria: The calculated MDL is verified if the MDLV standard is detected and the result is significantly different than the blank.

Corrective Actions: If the first MDLV is not detected, the MDLV standard will be re-prepared and analyzed at twice the original concentration. The lowest concentration that produces a detectable signal will then be reported as the MDL.

12.3 Instrument Detection Limit Study

Instrument detection limit (IDL) studies are conducted quarterly for each instrument and each wavelength used for analysis.

Digest seven blanks and run them on three non-consecutive days.

Calculate the standard deviation for each day. The final IDL concentration is the average of the three daily standard deviation values.

See Policy DV-QA-014p for a discussion of IDL studies and evaluation of IDL results.

12.4 Linear Dynamic Range (LDR)

The LDR must be determined initially (i.e., at initial setup) and then every three months for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample.

The LDR must be determined from a linear calibration prepared in the normal manner using the normal operating procedures described in Sections 10 and 11.

The LDR is determined by analyzing successively higher standard concentrations of the analyte. A minimum of three standards is required for the initial and on-going studies, and one of the levels must be close to the upper end of the range. The highest concentration must be within 10% of the stated concentration.

The highest standard that meets this criterion defines the maximum concentration that can be reported for sample analysis without dilutions.

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.

12.5 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 of interest and record the apparent emission intensity from all other method analytes. 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. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

12.6 Interelement Corrections (IECs)

ICP interelement correction (IEC) factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be re-determined.

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 using a standard from a second source or an alternate 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.

Refer to the facility-specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which produces a false analytical result with an absolute value greater than the RLs shown in Attachment 1. Note that the USACE program requires a control limit of 2|MDL|, which is feasible when verified MDLs are used.

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."

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 and CID detector instruments as reflected by the ICSA response.

12.7 Rinse Time Determination

Rinse times must be determined annually.

To determine the appropriate rinse time for a particular ICP system, a standard containing the highest concentration level that would be reported for samples is 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).

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.

12.8 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

Four aliquots of the ICV are analyzed using the same instrumental conditions and procedures used to analyze samples. The analyst must employ ICV's from four distinct analytical sequences. Using these four ICV's demonstrates the analyst's ability to optimize and calibrate the instrument and to prepare analytical silutions. Calculate the mean recovery and standard deviation of the mean recovery for each analyte of interest.

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.

Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

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. Further details concerning the training program are described in SOP DV-QA-0024.

12.9 Training Requirements

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. Further details concerning the training program are described in SOP DV-QA-0024.

13.0 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).

Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

Acid solutions from ICP drain - Waste Stream J

Metals waste potentially contaminated with Cat 1 radioactive materials – Waste Stream RJ

Note: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996, Method 6010B.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 6010	This procedure uses mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.
2	EPA 6010	The alternate run sequence presented in Section 10.5.3 is consistent with method requirements. Additional QC (i.e., ICSA) analyses were added to accommodate the CLP protocol requirements.
3	EPA 6010	Method 6010 states 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." Because of the lack of definition for "concentration range around the calibration blank," the laboratory has adopted the procedure in EPA CLP ILMO4.0 for determining IECs,

Item	Method	Modification
4	EPA 6010	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 the laboratory sometimes does not receive prior information from clients regarding new or unusual matrices, the analyst may select to perform a dilution test on one sample in each preparation batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. In this procedure, matrix interference is determined by evaluating data for the LCS, MS/MSD, and serial dilutions. The laboratory must request 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.
5	EPA 6010	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.
6	EPA 6010	Method 6010B section 8.6.1.3 states that the results of the calibration blank are to agree within 3 times the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within three standard deviations 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. TA has adopted an absolute control limit of ± RL from zero for calibration blank criteria. See SOP Sections SOP Sections 10.4.8 for a detailed description of the required corrective action procedures.

17.0 Attachments

Attachment 1 Metals Analyzed by ICP and Reporting Limits
Attachment 2 Matrix Spike and Aqueous Laboratory Control Sample Levels
Attachment 3 Trace ICP Initial Calibration &Continuing Calibration Verification Standards
Attachment 4 Interference Check Sample Concentrations
Attachment 5 TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels
Attachment 6 Intrepid II Duo Initial Calibration &Continuing Calibration Verification Standards
Attachment 7 Summary of Quality Control Requirements
Attachment 8 ICP Data Review Checklist
Attachment 9 MSA Guidance
Attachment 10 Troubleshooting Guide
Attachment 11 Contamination Controls
Attachment 12 Preventative Maintenance

18.0 Revision History

- Revision 1, dated 03 June 2009
  - Sec 1.5, 10.3 Updated SOP number for soil digestions
  - Sec 9.6 Updated flagging to MSB from NC
  - Sec 12.2 updated MDLV standard to 1-3 times MDL
  - Sec 12.3 Updated IDL study for digested blanks
  - Added Bismuth to all the tables and standards lists
  - Updated attachment 7 to include DoD and AFCEE requirements

- Revision 0, dated 18 April 2008
  - Integration for TestAmerica and STL operations
  - Split the ICP Water SOP into 2 SOPs – 200.7 and 6010
  - Deleted all references of Method 200.7 from this SOP
  - Updated formatting
  - Updated references

Attachment 1

Metals Analyzed by ICP and Reporting Limits

ELEMENT	Symbol	CAS #	6010 Analyte	Reporting Limit (µg/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	100	10
Antimony[trace]	Sb	7440-36-0	X	10	1
Arsenic[trace]	As	7440-38-2	X	15	1
Barium	Ba	7440-39-3	X	10	1
Beryllium	Be	7440-41-7	X	1	0.1
Bismuth	Bi	7740-69-9		100	10
Boron	B	7440-42-8		100	10
Cadmium[trace]	Cd	7440-43-9	X	5	0.5
Calcium	Ca	7440-70-2	X	200	20
Chromium	Cr	7440-47-3	X	10	1
Cobalt	Co	7440-48-4	X	10	1
Copper	Cu	7440-50-8	X	15	2
Iron	Fe	7439-89-6	X	100	10
Lead[trace]	Pb	7439-92-1	X	9	0.8
Lithium	Li	7439-93-2	X	10	5
Magnesium	Mg	7439-95-4	X	200	20
Manganese	Mn	7439-96-5	X	10	1
Molybdenum	Mo	7439-98-7	X	20	2
Nickel	Ni	7440-02-0	X	40	4
Phosphorus	P	7723-14-0	X	3,000	300
Potassium	K	7440-09-7	X	3,000	300
Selenium[trace]	Se	7782-49-2	X	15	1.3
Silicon	Si	7631-86-9		500	50
Silver[trace]	Ag	7440-22-4	X	10	1
Sodium	Na	7440-23-5	X	1	100
Strontium	Sr	7440-24-6	X	10	1
Sulfur	S	7704-34-9	X	200	2
Thallium[trace]	Tl	7440-28-0	X	15	1.2
Thorium	Th	7440-29-1		15	15
Tin	Sn	7440-31-5		100	10
Titanium	Ti	7440-32-6		10	1
Uranium	U	7440-61-1		60	20
Vanadium	V	7440-62-2	X	10	2
Zinc	Zn	7440-66-6	X	20	2
Zirconium	Zr	7440-67-7	X	15	1

Attachment 2

Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (µg/L)	Matrix Spike Level (µg/L)
Aluminum	2,000	2,000
Antimony	500	500
Arsenic	2,000	2,000
Barium	2,000	2,000
Beryllium	50	50
Bismuth	2,000	2,000
Boron	1,000	1,000
Cadmium	50	50
Calcium	50,000	50,000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1,000	1,000
Lead	500	500
Lithium	1,000	1,000
Magnesium	50,000	50,000
Manganese	500	500
Molybdenum	1,000	1,000
Nickel	500	500
Phosphorous	10,000	10,000
Potassium	50,000	50,000
Selenium	2,000	2,000
Silicon	10,000	10,000
Si (as SiO_2)	21,400	21,400
Silver	50	50
Sodium	50,000	50,000
Strontium	1,000	1,000
Sulfur	2,000	2,000
Thallium	2,000	2,000
Thorium	2,000	2,000
Tin	2,000	2,000
Titanium	1,000	1,000
Uranium	2,000	2,000
Vanadium	500	500
Zinc	500	500
Zirconium	500	500

Attachment 3

Axial ICP Initial Calibration &Continuing Calibration Verification Standards

Element	Calibration Level	ICV (µg/L)	CCV (µg/L)
Aluminum	10,000	2,500	5,000
Antimony	1,000	250	500
Arsenic	2,000	250	1,000
Barium	1,000	250	500
Beryllium	1,000	250	500
Cadmium	1,000	250	500
Calcium	40,000	10,000	20,000
Chromium	1,000	250	500
Cobalt	1,000	250	500
Copper	1,000	250	500
Iron	10,000	2,500	5,000
Lead	1,000	250	500
Magnesium	40,000	10,000	20,000
Manganese	1,000	250	500
Molybdenum	1,000	250	500
Nickel	1,000	250	500
Potassium	100,000	25,000	50,000
Selenium	10,000	2,500	5,000
Silver	1,000	250	500
Sodium	400,000	100,000	200,000
Strontium	1,000	250	500
Sulfur	10	2	5
Thallium	10,000	2,500	5,000
Thorium	2,000	3,000	1,000
Tin	10,000	250	5,000
Vanadium	1,000	250	500
Uranium	2,000	3,000	1,000
Zinc	1,000	250	500

Attachment 4

Interference Check Sample Concentrations

Element	ICSA (µg/L)	ICSAB (µg/L)
Aluminum	500,000	500,000
Antimony	-	1,000
Arsenic	-	2,000
Barium	-	500
Beryllium	-	500
Bismuth	-	1,000
Cadmium	-	1,000
Calcium	500,000	500,000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200,000	200,000
Lead	-	1,000
Magnesium	500,000	500,000
Manganese	-	500
Molybdenum	-	1,000
Nickel	-	1,000
Potassium	-	50,000
Selenium	-	5,000
Silver	-	1,000
Sodium	-	50,000
Sulfur	-	1,000
Thallium	-	10,000
Vanadium	-	500
Zinc	-	1,000
Tin	-	10,000
Thorium	-	10,000
Uranium	2,000	2,000
Zirconium	-	1,000

Attachment 5

TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (µg/L)	Regulatory Limit (µg/L)	Spike Level (µg/L)
Arsenic	500	5000	4000
Barium	10000	100000	12000
Cadmium	100	1000	1100
Chromium	500	5000	5200
Lead	500	5000	5500
Selenium	250	1000	3000
Silver	500	5000	1050
Copper	100	N/A	2250
Zinc	200	N/A	2500

Attachment 6

6000 Dual View Calibration, ICV & CCV Standards

Element	Calibration Level	ICV (µg/L)	CCV (µg/L)
Aluminum Lo	1,000	250	500
Aluminum Hi	100,000	40,000	50,000
Antimony	2,000	250	1,000
Arsenic	2,000	250	1,000
Barium	1,000	250	500
Beryllium	1,000	250	500
Bismuth	2,000	500	1000
Cadmium	1,000	250	500
Calcium	10,000	2,000	5,000
Chromium	1,000	250	500
Cobalt	1,000	250	500
Copper	1,000	250	500
Iron Lo	5,000	250	2,500
Iron Hi	100,000	80,000	50,000
Lead	2,000	250	1000
Magnesium	40,000	10,000	20,000
Manganese	1,000	250	500
Molybdenum	1,000	250	500
Nickel	1,000	250	500
Phosphorous	2,000	2,000	1,000
Potassium	100,000	20,000	50,000
Selenium	2,000	500	1,000
Silver	1,000	250	500
Sodium Lo	10,000	2000	5,000
Sodium Hi	500,000	40,000	250,000
Strontium	1,000	250	500
Sulfur	10,000	4,000	5,000
Thallium	2,000	500	1,000
Thorium	10,000	3,000	5,000
Tin	2,000	500	1,000
Vanadium	1,000	250	500
Uranium	20,000	5,000	10,000
Zinc	1,000	250	500
Zirconium	1,000	250	500

Attachment 7

Summary Of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between multiple exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
CCV	After the ICV, after every 10 samples and at the end of the run.	90-110% recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
ICB	Beginning of every analytical run, immediately following the initial CCV.	The result must be within +/- RL from zero. <2x MDL for DoDQSM and AFCEE 4.0	Terminate analysis; Correct the problem; Recalibrate.
CCB	Immediately following each CCV (except for the CCV following the ICV).	The result must be within +/- RL from zero. <2x MDL for DoDQSM and AFCEE 4.0	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.10	See Section 9.10
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.10
Dilution Test	One per prep batch.	For samples > 50x MDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.

See Section 10.5.6 for run sequence to be followed.

Attachment 7

Summary of Quality Control Requirements (Continued)

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. <1/2 the reporting limit for DoDQSM and AFCEE 4.0 Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.4)	Re-run once in a clean tube. If >RL or ½ RL depending on program, re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.5)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike (MS)	One per sample preparation batch of up to 20 samples.	75 – 125% recovery or tighter in-house control limits.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate (MSD)	One per sample preparation batch of up to 20 samples. 10% frequency for some programs (see 0)	75 – 125 % recovery; RPD ≤ 20% or tighter in-house control limits.	See Corrective Action for Matrix Spike.

Attachment 8
ICP Data Review Checklist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Denver

ICP Data Review Checklist

Run/Project Information:		
Run Date:	**Analyst:**	**Instrument:**
Prep Batches Run:		
Methods Used: 6010B / 200.7		

Review Items	**Yes**	**No**	**N/A**	**2nd Level**
A. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits?				
2. Method blank done per prep batch and < RL or CRDL (CLP)?				
3. MS run at required frequency and within limits?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
5. Serial dilution done per prep batch (or per SDG for CLP)?				
6. Post digest spike analyzed if required (CLP & AFCEE only)?				
B. Calibration/Instrument Run QC				
1. ICV/CCV analyzed at appropriate frequency and within control limits ? (**6010B**: CLP = 90 - 110%; **200.7:** ICV = 95 - 105%, CCV = 95 - 105% for 40 CFR 136B, 90 - 110% for RCRA / SDWA) If not in control, was the CCV reanalyzed twice to show return to control as per NELAP?				
2. ICB/CCB analyzed at appropriate frequency and < RL or < CRDL (CLP) or < 2X MDL (AFCEE 4.0)?				
3. High Standard (HIGH) reanalyzed before samples and recovered within QC limits? (6010B/200.7: 95-105%)				
4. RL STD run and recovered within QC limits ? (± 50% for non-CLP, ± 20% for DoD / AFCEE 4.0 / USACE)				
5. ICSA/ICSAB run at required frequency and within SOP limits? (ICSA < 2X MDL AFCEE 4.0)				
C. Sample Results				
1. For 6010B, were samples with concentrations > the linear range for any parameter diluted and reanalyzed? For 200.7, were samples with concentrations within 90% of the linear range diluted and reanalyzed?				
2. Are all reported results bracketed by in control QC?				
D. Other				
1. Are all nonconformances documented appropriately?				
2. Calculations checked for errors?				
3. Transcriptions checked for errors? (Example: Are dilution factors that are entered into the sequence log correct?)				
4. All client/project specific requirements met?				
5. Date/time of analysis verified as correct?				

Analyst: _____ **Date:** _____

Comments: _____

_____

_____

2nd Level Reviewer: _____ **Date:** _____

Comments: _____

_____

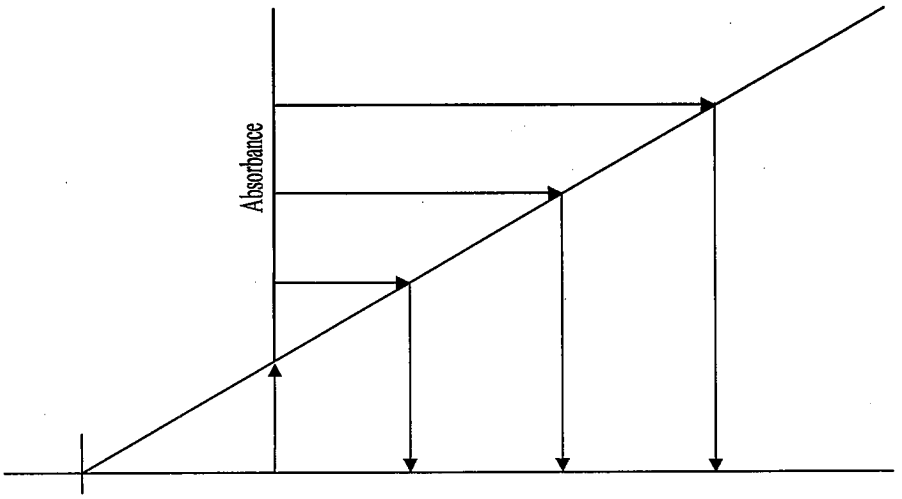
_____

Attachment 9
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 absolute value of the point of interception of the horizontal axis is the concentration of the unknown.



oncentratio

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cted

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 (r=0.995 or greater) 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.

Attachment 10

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 Clean or replace mixing chamber
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) 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 Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

Attachment 11

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 gloves should not be used in the metals laboratory because the powder contains silica and zinc as well as other metallic analytes.

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

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

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

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 contain over 500 ppb of zinc.

Attachment 12
Preventive Maintenance

TestAmerica
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ICP PREVENTIVE MAINTENANCE LOG

TestAmerica Denver

MAINTENANCE	MONTH:			YEAR:			
Daily > Fill rinse solution / IS solution. > Make RL check standard solution. > Check torch tip for residue. > Check nebulizer flow. > Check waste container & empty as needed. > Check expiration dates of all standards. **Weekly** > Replace pump tubing. **As Needed** > Clean nebulizer & drain chamber. > Replace torch; realign and optimize. > Replace rinse / standard bottles. > Replace capillary tubing / sipper probe. **Monthly** > Clean air filters on instrument. > Check internal fluid reservoir. **Yearly** > Change internal cooling fluid.	1	2	3	4	5	6	7
	8	9	10	11	12	13	14
	15	16	17	18	19	20	21
	22	23	24	25	26	27	28
	29	30	31				

L:\QA\Edit\Logbooks\Metals\ICP Preventive Mtce Log

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TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Mercury in Water by Cold Vapor Atomic Asorption (CVAA) [SW 7470A]

Approvals (Signature/Date):

Richard Clinkscales — Date
Inorganic Operations Manager

Adam Alban — Date
Health & Safety Manager / Coordinator

Karen Kuoppala — 08-07-09 — Date
Quality Assurance Manager

Robert C. Hanisch — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A.

1.2 Method 7470 is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, wipes, TCLP, EP and other leachates/extracts.

1.3 All matrices require sample preparation prior to analysis.

1.4 The reporting limit is 0.0002 mg/L (0.2 µg/L), except for TCLP leachates that have a 0.002 mg/L (2 ug/L) reporting limit.

2.0 Summary of Method

This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3.0 Definitions

3.1 Dissolved Metals: Those elements that pass through a 0.45-µm membrane. (Sample is acidified after filtration).

3.2 Total Metals: The concentration determined on an unfiltered sample following digestion.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 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.

4.3 Copper also 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.

4.4 Chlorides can cause a positive interference. Seawaters, brines, and industrial effluents high in chlorides will require dilution. During the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

4.5 Interference from certain volatile organic materials that absorb at the wavelength used for the method may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.6 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.7 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual 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, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

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

5.1.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.3 Potassium permanganate is a strong oxidizing agent. It is incompatible and must be stored separately from hydroxylamine hydrochloride and stannous chloride, the reducing agents used in this procedure, and from acids.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
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	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.
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.
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.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Temperature controlled, mechanically re-circulating water bath capable of maintaining a temperature of 90-95°C.
- Mercury Auto-analyzers; either of the following can be used, or equivalent:
  - CETAC Mercury Analyzer with Autosampler and Auto-Diluter
  - Perkin-Elmer FIMS Mercury Analyzer with Autosampler

6.2 Supplies

- Digestion Tubes – disposable glass, 18mm x 150mm, plastic cap
- Argon, 99.999% purity
- Calibrated automatic pipettes or Class A glass volumetric pipettes (see SOP DV-QA-0008 for details on calibrating mechanical pipettes).
- Class A volumetric flasks.
- Thermometer, non-mercury column, accurate to ±1°C at 95 °C (see SOP DV-QA-0001 for calibration details).
- Disposable cups or tubes.
- 0.45 µm syringe filters

7.0 Reagents and Standards

NOTE: The preparation of reagents and standards is recorded in Standards Log, a computerized database. The reagents and standards are listed on the bench sheet and the pipettes and/or balances used in the preparations are also listed on the bench sheet. A printout from Standards Log accompanies the bench sheet to provide preparation details for each prepared reagent and standard.

7.1 Reagent water: 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 Nitric acid (HNO3): concentrated, trace metal grade or better.

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

7.4 Hydrochloric Acid, 3%, carrier solution for the FIMS Analyzer:
Add about one liter of reagent water to a two-liter plastic bottle. Add 60 mL of concentrated hydrochloric acid and dilute to the mark with reagent water.

7.5 Sulfuric acid (H2SO4): concentrated, trace metal grade or better.

7.6 Calibration Blank, Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Method Blank (MB), 1% HNO3:
Add 0.5 L of concentrated HNO3 to a 50-liter carboy partially filled with reagent water. Dilute to 50 L with reagent water.

7.7 Stannous Chloride (SnCl2) Solution, reagent grade, 1.3% (w/v) per manufacturer's instructions. (PE FIMS Only)

7.7.1 Place approximately 100 mL of deionized water in a 2-L volumetric flask.

7.7.2 Slowly add 60 mL of concentrated HCl to the flask and swirl to mix.

7.7.3 Add 26.4 grams of SnCl2 to the flask.

7.7.4 Place a large stir bar in the flask and put the flask on a stir plate.

7.7.5 Stir the contents of the flask until the reagent is completely dissolved.

7.7.6 Remove the stir bar and bring to volume with deionized water.

7.8 Stannous Chloride Solution, reagent grade, 10% (w/v) per manufacturer's (CETAC) instructions

7.8.1 Place approximately 100 mL of deionized water in a 2-L volumetric flask

7.8.2 Slowly add 200 mL of concentrated HCl to the flask and swirl to mix.

7.8.3 Add 200 grams of SnCl2 to the flask.

7.8.4 Place a large stir bar in the flask and put the flask on a stir plate.

7.8.5 Stir the contents of the flask until the reagent is completely dissolved.

7.8.6 Remove the stir bar and bring to volume with deionized water.

7.9 Sodium chloride-hydroxylamine hydrochloride solution:
Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride (reagent grade) to every 100 mL of reagent water.

NOTE: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.10 Potassium permanganate (KMnO4), 5% solution (w/v):
Dissolve 5 g of potassium permanganate (reagent grade, "suitable for mercury determination") for every 100 mL of reagent water.

7.11 Potassium persulfate (K2S2O8), 5% solution (w/v):
Dissolve 5 g of potassium persulfate, reagent grade, for every 100 mL of reagent water.

7.12 Purchased Mercury Stock Solutions

7.12.1 Primary Mercury Calibration Standard Solution, 1,000 mg/L

7.12.2 Second-source Mercury Standard (different vendor than primary calibration standard), 100 mg/L.

7.13 Calibration Working Standard Solution, 10 mg/L.

7.13.1 Add approximately 90 mL of 1% HNO3 to a 100 mL Class A volumetric flask.

7.13.2 Pipet 1.00 mL of the 1000 mg/L primary mercury calibration standard solution (see Section 7.12.1) into the flask.

7.13.3 Dilute to the mark on the flask with 1% HNO3.

7.13.4 Stopper the flask and shake to mix.

7.13.5 Transfer the solution to a 125 mL Nalgene bottle.

7.13.6 Document the preparation of the solution in the Standards Log database.

7.13.7 Prepare this solution fresh monthly or more often if necessary.

7.14 Daily Calibration Working Solution, 100 µg/L

7.14.1 Add approximately 90 mL of 1% HNO3 to a 100 mL Class A volumetric flask.

7.14.2 Pipet 1.00 mL of the 10 mg/L Calibration Working Standard solution (see Section 7.13) into the flask.

7.14.3 Dilute to the mark on the flask with 1% HNO3 (final volume of 100.0 mL).

7.14.4 Stopper the flask and shake to mix.

7.14.5 Transfer the solution to a 125 mL Nalgene bottle.

7.14.6 Document the preparation of the solution in the Standards Log database.

7.15 Daily Initial Calibration (ICAL)Standards.

7.15.1 To each of six volumetric flasks, add approximately 80 mL of 1% HNO3.

7.15.2 For each calibration level, add the amount of Daily Calibration Working Solution to the flask as indicated in the following table and bring the solution to a final volume of 100.0 mL. The final concentration for each calibration level is listed in the following table:

Daily ICAL Standards

Calibration Level	Volume of Daily Calibration Working Solution (100 µg/L)	Final Hg Concentration (µg/L)
1	0.20	0.2
2	0.50	0.5
3	1.0	1.0
4	2.0	2.0
5	5.0	5.0
6	10.0	10.0

7.15.3 Stopper each flask and mix thoroughly.

7.15.4 Document the preparation of the solution in the Standards Log database.

7.15.5 Prepare the calibration solutions each day prior to calibration.

7.16 Continuing Calibration Verification Standard, 5.0 µg/L.

7.16.1 The CCV is prepared exactly as the 5.0 µg/L calibration standard, and from the same source. Refer to Section 7.15.

7.16.2 Prepare sufficient volume of the standard for analysis of a CCV after every 10 samples.

7.17 Second-Source Initial Calibration Verification (ICV) Intermediate Standard, 700 µg/L.
Add 0.7 mL of the 100 mg/L ICV stock standard (see Section 17.12) to a 100 mL volumetric flask partially filled with 1%HNO3 and dilute to the mark. Record this information in the Standards Log database.

7.18 Second-Source Initial Calibration Verification (ICV) Working Standard, 7.00 µg /L.
Add 1.0 mL of the 700 µg/L ICV intermediate standard (see Section 7.17) to a 100 mL volumetric flask partially filled with 1%HNO3 and dilute to the mark. Record this information in the Standards Log database.

7.19 Laboratory Control Sample (LCS), 5 µg/L
The LCS is prepared by adding 0.5 mL of the 100 µg/L Daily Calibration Working Standard to 10 mL of reagent blank in a digestion tube.

7.20 Matrix Spike and Matrix Spike Duplicate (MS/MSD), 5 µg/L

7.20.1 The MS is prepared by adding 0.5 mL of the 100 µg/L Daily Calibration Working Solution to a digestion tube containing a second10-mL aliquot of the selected sample.

7.20.2 The MSD is prepared in the same manner as the MS using a third aliquot of the selected sample.

7.21 Reporting Limit (RL) Check Standard, 0.2 µg/L
The 0.2 µg/L calibration standard is analyzed as a sample to verify the reporting limit. Denoted as RL or RLSTD in the run sequence.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50 mLs	HNO_3, pH < 2	28 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.1.1 Preparation Batch

A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.1.2 Method Blank (MB)

The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than the reporting limit or less than 10% of the mercury concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If mercury was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.1.3 Laboratory Control Sample (LCS)

The LCS is a blank to which a known concentration of the target analyte has been added. At least one aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure.

Acceptance Criteria: Maximum control limits for LCS recoveries for Method 7470A are 80-120%. In-house control limits based on three standard deviations of the mean of past results are used as long as they are at least as tight as the limits in the methods (see TestAmerica Denver Policy DV-QA-003P for further details on establishing control limits).

Corrective Action: If LCS recoveries are outside established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and mercury is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

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

A matrix spike (MS) is a second aliquot of a selected field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a third aliquot of the same sample (spiked exactly as the MS) prepared and analyzed along with the sample and matrix spike. One MS/MSD pair must be processed for each preparation batch. Some programs may require the use of sample duplicates in place of or in addition to MS/MSDs. In addition, some programs will allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Attachment 1).

Acceptance Criteria: Control limits are statistically determined based on three standard deviations of the mean of the laboratory's historical data. The recoveries for the MS and MSD must fall within 75-125%. The relative percent difference between the MS and MSD cannot exceed 20%.

Corrective Action: If analyte recoveries or the RPD between duplicates fall outside the acceptance range, then LCS recovery must be in control for the data to be reported. If there is no evidence of analytical problems and all other QC criteria are met, then qualified results may be reported and the situation must be described in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that

analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC, then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

9.1.5 Serial Dilution

Some programs (e.g., DoD programs) require that a fivefold (1+4) dilution must be included in each analytical batch for each sample matrix.

Acceptance Criteria: The results must be within 10% of the expected value, assuming that the sample concentration is at least 25x the MDL concentration.

Corrective Action: If the control limit is not met, all associated sample results must be qualified.

9.1.6 Post-Digestion Spikes

Some programs (e.g., Dupont, DOE) require the inclusion of a post-digestion spike in each analytical batch. The post-digestion spike is prepared by adding 0.25 mL of the 100 µg/L Daily Calibration Working Solution to 6.6 mL of filtered sample digestate. Post-digestion spikes are performed as an additional check for matrix interference.

Acceptance Criteria: The percent recovery limits for the post-digestion spike are 85 to 115%.

Corrective Action: If the acceptance criteria are not met, all associated sample results must be qualified.

9.1.7 Method of Standard Addition (MSA)

The method of standard additions is an option for the analysis of samples shown to have significant matrix effects, e.g., unacceptably low MS/MSD recoveries or under certain conditions for TCLP analysis (see Attachment 5)

9.2 Instrument QC

9.2.1 Initial Calibration (ICAL)

9.2.1.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)*.

9.2.1.2 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.

9.2.1.3 Calibrate using six standards and a blank. The concentration levels are listed in Attachment 1.

NOTE: It is generally not acceptable to reject calibration points for this method.

9.2.1.4 The calibration curve must have a correlation coefficient of ≥ 0.995 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.

9.2.2 Initial and Continuing Calibration Blanks

9.2.2.1 An initial calibration blank is tested immediately after the daily ICAL standards.

Acceptance Criteria: The absolute value of the blank result must be less than the reporting limit. Some programs require that blanks be less than 2x the MDL (refer to special project requirements).

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

9.2.2.2 Continuing calibration blanks are run after every 10 samples and at the end of the run.

Acceptance Criteria: The absolute value of the blank result must be less than the reporting limit. Some programs require that blanks be less than 2x the MDL (refer to special project requirements).

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

9.2.3 Initial Calibration Verification (ICV), 7 µg/L

The accuracy of the calibration standards is verified by testing a second source standard (ICV).

Acceptance Criteria: The ICV result must be within 10% of the true value.

Corrective Action: If the ICV acceptance limit is exceeded, the analysis should be terminated, the accuracy of the calibration standards checked, and the instrument recalibrated.

9.2.4 Reporting Limit Check Standard (RL), 0.2 µg/L

The accuracy of results at the reporting limit is verified by testing a standard in every analytical run that is prepared at the reporting limit concentration.

Acceptance Criteria: The results for this standard must be within 50% of the expected value (20% for USACE and DoD projects).

Corrective Action: If the RL check acceptance limit is exceeded, the analysis should be terminated, the instrument operation checked, and the instrument recalibrated.

9.2.5 Continuing Calibration Verification (CCV), 5.0 µg/L

Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the run. The CCV must be a mid-range standard at a concentration other than that of the ICV.

Acceptance Criteria: The CCV result must fall within 20% of the true value.

Correction Action: Sample results may be reported only when bracketed by valid CCV pairs. If a mid-run CCV fails, the CCV may be re-analyzed once without modification to the instrument's operating conditions. If the re-analyzed CCV is found to be in control, the CCV analysis must be repeated with successful results or the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV failure was not directly instrument related, the associated samples must be re-prepared and reanalyzed.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM shall be filed in the project file.

10.1 Sample Preparation

10.1.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB), as well as the field samples, are processed through the digestion procedure.

10.1.2 Transfer 10.0 mL of well mixed sample and 20.0 mL of each calibration and calibration verification standard to a clean sample digestion tube. The additional volume of calibration and calibration verification standards is necessary to ensure sufficient volume to complete the analytical sequence. Be sure to add twice the amount of reagents to the calibration and calibration standards. Additional CCV and CCB solution may have to be prepared for larger sample runs.

10.1.3 Prepare an MB, LCS, MS, and MSD for each batch.

10.1.3.1 The MB consists of 10.0 mL of 1% HNO3.

10.1.3.2 The LCS is prepared by adding 0.5 mL of the 100 µg/L Daily Calibration Working Solution to 10 mL of 1% HNO3 in a digestion tube.

10.1.3.3 The MS is prepared by adding 0.5 mL of the 100 µg/L Daily Calibration Working Solution to a digestion tube containing a second 10-mL aliquot of the selected sample.

10.1.3.4 The MSD is prepared in the same manner as the MS using a third aliquot of the selected sample.

10.1.4 Add 0.5 mL of concentrated H2SO4 and 0.25 mL of concentrated HNO3 to the samples in the digestion tubes, mixing after each addition.

10.1.5 Add 1.5 mL of 5% potassium permanganate solution to each sample. For samples high in organic materials or chlorides, dilute the sample until the purple color persists for at least 15 minutes.

10.1.6 Add 0.8 mL of potassium persulfate solution, cap the vial, and heat for two hours in a water bath at 90 - 95°C. Record the start and stop times and the temperature on the bench sheet. Verify that a purple color persists or a black precipitate is present after the two hours of heating. If this is not true, repeat the digestion using a smaller aliquot of sample.

10.1.7 Allow the samples and standards to cool at room temperature.

10.2 Calibration

10.2.1 All calibration standards are digested together with samples, as described in Section 10.1, prior to analysis.

10.2.2 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).

10.3 Sample Analysis

NOTE: Because of differences between various makes and models of CVAA instrumentation, detailed push-button operating instructions are not provided here. Refer to the specific instrument-operating manual for detailed autosampler setup and operation protocols.

NOTE: The injection of samples and the addition of stannous chloride are done automatically by the instrument. Refer to the specific instrument manual for details.

10.3.1 When ready to begin analysis, add 0.6 mL of sodium chloride-hydroxylamine hydrochloride solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains.

10.3.2 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

10.3.3 If the sample results are negative and the absolute value is greater than the reporting limit, the sample must be diluted and reanalyzed.

10.3.4 The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.3.5 If any samples have a visible precipitate, then filter those samples plus the method blank and LCS through 0.45 µm syringe filters.

NOTE: The method blank and LCS must be subject to all preparation and analysis steps in order to accurately represent the batch.

10.3.6 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB. Re-sloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

10.3.7 The following analytical sequence must be used for Method 7470A. Refer to Quality Control Section 9.0 and Attachment 2 for quality control criteria to apply to Method 7470A.

Instrument Calibration
ICB
ICV
RL
Maximum of 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete the run.
CCV
CCB

NOTE: Samples included in the count between CCVs include the method blank, LCS, MS, MSD, and field samples.

10.3.8 For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

- Recovery of the analyte in the matrix spike is not at least 50%;
- The concentration of the analyte does not exceed the regulatory level; and
- The concentration of the analyte is within 20% of the regulatory level.

The reporting and matrix spike levels for TCLP analyses are detailed in Attachment 1. Attachment 5 provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

10.3.9 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.

10.3.10 See Attachment 7 for guidelines for minimizing contamination of samples and standards. See Attachment 8 for guidance on troubleshooting and preventive maintenance.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 **Concentration** = Hg concentration $(\mu g/L) = C \times D$

Where:

C = Concentration (µg/L) from instrument readout
D = Instrument dilution factor

11.4 Appropriate factors must be applied to sample values if dilutions are performed.

11.5 Sample results should be reported with up to three significant figures in accordance with the TestAmerica significant figure policy (DV-QA-004P).

11.6 **Documentation and Record Management**
The following documentation comprises a complete CVAA raw data package:

- Sample preparation bench sheet(s), to include the batch number, list of samples, preparation analyst and date, instrument analysis analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes). See Attachment 3.
- Raw data (direct instrument printout).
- Run log printout from instrument software where this option is available or manually-generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Attachment 4.

- Standards Documentation to include source, lot, preparation date, and expiration date. A printout from Standards Log provides all required information except identification of measuring equipment used.
- Nonconformance summary (if applicable).

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

- Prepare seven samples at three to five times the estimated MDL concentration.
- Prepare and analyze the MDL standards as described in Section 10.
- Calculate the average concentration found (X) in µg/L, and the standard deviation of the concentration(s) in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level, as described in Policy # DV-QA-005P) for each analyte.
- MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy DV-QA-005P for further details concerning MDL studies.
- The current MDL value is maintained in the TestAmerica Denver LIMS.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- Four LCSs 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.
- If the recoveries do not meet the historical acceptance criteria, the test must be repeated. Repeated failures indicate the need for the laboratory to evaluate the analytical procedure and take corrective action.
- Further details concerning demonstrations of proficiency are described in SOP# DV-QA-0024.

12.3 Training Requirements

12.3.1 The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

12.3.2 Each analyst performing the method must complete a demonstration of capability (DOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or

other acceptable QC samples. The results of the DOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. DOCs are approved by the Quality Assurance Manager and the Technical Director. DOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually. Initial Demonstration of Capability

13.0 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).

14.0 Waste Management

14.1 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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produce when this method is carried out:

- Aqueous Acidic (Metals) - Corrosive - Waste Stream J
- Expired reagents and standards – Contact the Waste Coordinator.

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, March 2005.

15.3 U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 7470A	Chapter 1 of SW846 specifies the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
2	EPA 7470A	This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."
3	EPA 7470A	Methods 7470A and 7471A state that working mercury standards "should be prepared fresh daily." The laboratory frequently prepares up to three batches of mercury samples, including digested calibration standards, each day. The third batch is typically prepared and digested late in the day, and then is analyzed the morning of the next day. The laboratory has developed the following information demonstrating that analysis within 24 hours, but on the second calendar day from preparation produces reliable results and is acceptable to the EPA: • Successful proficiency testing PT results for samples that were prepared and analyzed within 24 hours, but on successive days (e.g., ERA WP-66); • Successful analysis of true NIST mercury standards within every analytical batch; and • A written comment from the EPA MICE Hotline stating that, with the supporting lab data, their opinion was that the laboratory's practice is "within the letter of the method as written."
4	EPA 7470A	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.

17.0 Attachments

Figure 1: Aqueous Sample Preparation Flow Chart
Figure 2: CVAA Mercury Analysis Flow Chart
Attachment 1: Mercury Reporting Limits, Calibration Levels, QC Standard and Spiking Levels
Attachment 2: Summary of Quality Control Requirements
Attachment 3a: Example Metals Preparation Benchsheet - FIMS
Attachment 3b: Example Metals Preparation Benchsheet -CETAC
Attachment 4: Example Raw Data Checklist
Attachment 5: MSA Guidance
Attachment 6: Troubleshooting Guide
Attachment 7: Contamination Control Guidelines
Attachment 8: Preventative Maintenance

18.0 Revision History

- Revision 0.2 dated 07 August 2009
  - Sections 7.17 and 7.18 were updated to use 1% HNO3 from reagent blank.
  - Sections 10.1.3.1 and 10.1.3.2 were updated to use 1% HNO3 from reagent blank.
  - Changed SOP name DV-QA-003P from QC Policy to Quality Assurance Program.

- Revision 0.1, dated 16 February 2008
  - Section 9.1.2: Changed control limit to 10% to match soil SOP
  - Section 9.2.2: Changed the stated control limits for special projects from ½ the RL to 2x the MDL
  - Deleted section 12.2 for IDL requirements
  - Section 12.3: Noted that LCSs will be used for verification

Figure 1.

Aqueous Sample Preparation Flow Chart

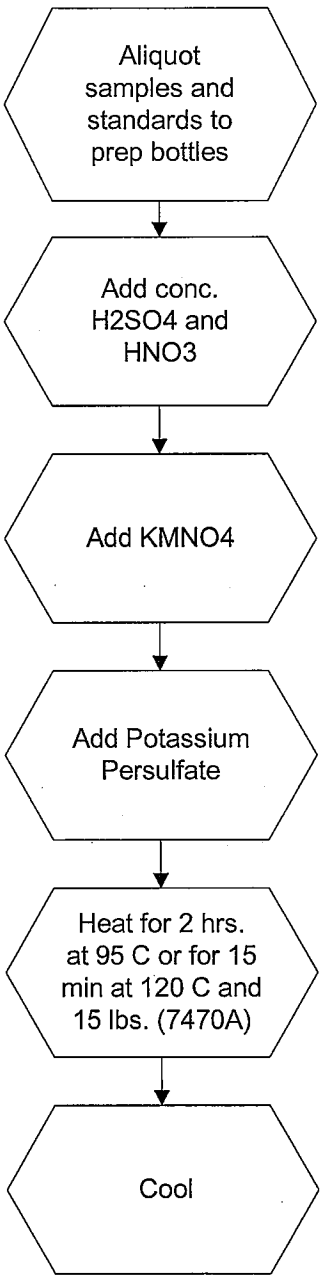
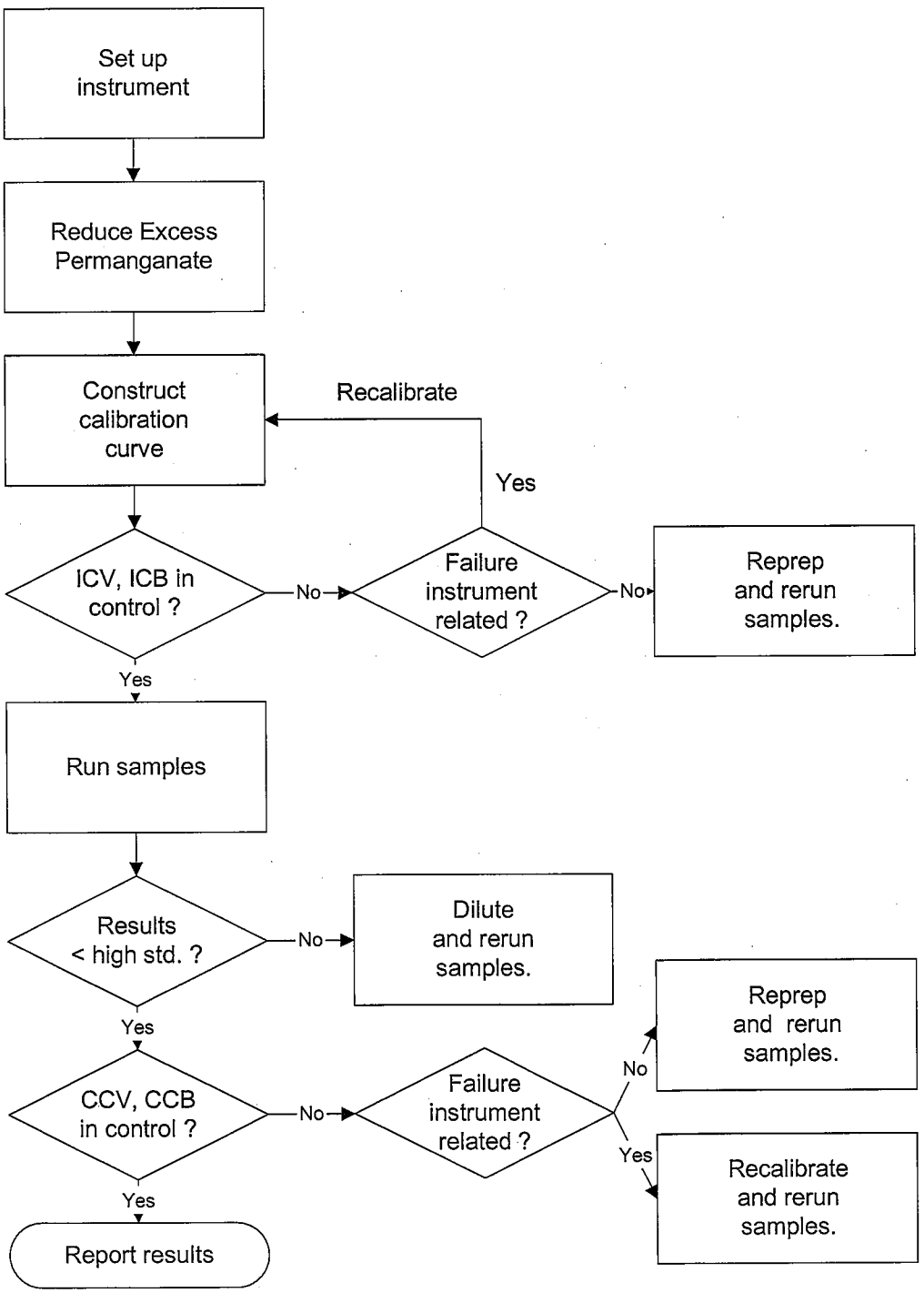


Figure 2.

CVAA Mercury Analysis Flow Chart



Attachment 1.

Mercury Reporting Limits, Calibration Levels, QC Standard and Spiking Levels (µg/L)

Standard Aqueous RL	0.2
TCLP RL	0.2
Std 0	0
Std 1	0.2
Std 2	0.5
Std 3	1.0
Std 4	2.0
Std 5	5.0
Std 6	10.0
ICV	7.0
LCS/CCV	5.0
Aqueous MS	5.0
TCLP MS	5.0

Attachment 2.

Summary of Quality Control Requirements

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
ICV	Beginning of every analytical run.	90 - 110% recovery	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.2.3).
ICB	Beginning of every analytical run, immediately following the ICAL.	Absolute value must be < RL, 2x the MDL for DoD	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.2.2).
CCV	Every 10 samples and at the end of the run.	80 - 120% recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.2.5).
CCB	Immediately following each CCV.	Absolute value must be < RL, 2x the MDL for DoD	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (see Section 9.2.2).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than the RL (< ½ RL for DoD) Sample results greater than 10x the blank concentration are acceptable.	Re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.1.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	In-house 3 standard deviation control limits, not to exceed 80-120% recovery.	Terminate analysis; Correct the problem; Re-digest and reanalyze all samples associated with the LCS (see Section 9.1.3).
Matrix Spike	One per 10 samples preparation batch of up to 20 samples.	In-house 3 standard deviation control limits, not to exceed 75-125% recovery	In the absence of client-specific requirements, flag the data (see Section 9.1.4).
Matrix Spike Duplicate	See Matrix Spike	In-house 3 standard deviation control limits, not to exceed 20% RPD	See Corrective Action for Matrix Spike.

Attachment 3a.

Example Metals Preparation Benchsheet – FIMS

SUPPLEMENTAL METALS PREP SHEET
(Used in conjunction with METALS PREP LOG/BATCH SUMMARY)

Hg PREP & ANALYSIS - WATERS

SOP: DEN-MT-0015 QC Batch #: _____

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
TestAmerica Denver

Prep Date: 1/14/08	Prep By: DAW	Analysis Date: 1/14/08	Analyst: DAW
Balance ID: H53865		***Thermometer ID:*** MT 4025	

Digestion Cycles	Start Time	Temp °C	End Time	Temp °C
	12:10	93	14:10	93

Purple color persists or black ppt present: [X] Yes [ ] No If "No", explain in Comments below.

Digestion Tube Lot # :

For dissolved mercury only, were samples filtered in the lab? [ ] Yes [ ] No
One or more samples were filtered prior to analysis at the instrument. [X] Yes [ ] No
If "yes", then the method blank and the LCS were also filtered in the same manner using the same type of filter.
Analyst(s) Initials:

Reagents Used

Reagent	Manufacturer	Lot #	Standards Log #	Vol (mL)
HNO_3	JT Baker	E27027		0.25
H_2SO_4	Fisher	E43040		0.5
HCl	JT Baker	E40030		used by instrument
1.3% $SnCl_2$	Fisher	E20623	STD-1033-08	added by instrument
NaCl / NH_2OH	Fisher	E13654	STD-1032-08	0.6
	Fisher	E10644		
$KMnO_4$	Fisher	C30618	STD-1020-08	1.5
$K_2S_2O_8$	Fisher	062641	STD-6324-08	0.8

Parent Calibration Stock Standard

	Lot #	Verification #	Exp. Date
Second Source	A2-HG-02019	STD-2112-07	06/01/08
Primary Calibration	E00414	STD-1008-08	04/03/08

Standards Preparation **Final digestate volume = 10 mls**

Standards	Final Conc	Parent Standard	Standards Log #	Vol (mL)	Pipette
Cal Working	10 mg/L	Primary Cal	See Attached Standards Log Printouts	1.00	20
Daily Cal Working	100 ug/L	Cal Working		1.00	20
ICAL 0.2	0.2 ug/L	Daily Cal Working		0.2	20
ICAL 0.5	0.5 ug/L	Daily Cal Working		0.5	20
ICAL 1	1.0 ug/L	Daily Cal Working		1.0	20
ICAL 2	2.0 ug/L	Daily Cal Working		2.0	20
ICAL 5	5.0 ug/L	Daily Cal Working		5.0	19
ICAL 10	10 ug/L	Daily Cal Working		10.0	19
CCV	5 ug/L	Daily Cal Working		5.0	20
ICV Intermed	700 ug/L	ICV Stock		0.70	20
ICV Daily Working	7.0 ug/L	ICV Intermed		1.00	20
LCS	5 ug/L	Daily Cal Working		0.5	20
MS/MSD	5 ug/L	Daily Cal Working		0.5	20
RL	0.2 ug/L	Daily Cal Working		0.2	20

Second Source ICV Intermediate Stock Standard Prep **Standards Log #:** STD-1017-08

NOTE: Details for each reagent & standard prep are documented in the attached Standards Preparation Logbook Record.

Comments

I certify that all information above is correct and complete.

Signature: ***Date:***

REVIEWED BY: _____ Date: _____

Attachment 3b.

Example Metals Preparation Benchsheet – CETAC

SUPPLEMENTAL METALS PREP SHEET
(Used in conjunction with METALS PREP LOG/BATCH SUMMARY)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
TestAmerica Denver

Hg PREP & ANALYSIS - WATERS

SOP: DEN-MT-0015 QC Batch #: _____

Prep Date: 1/15/08	Prep By: DAW	Analysis Date: 1/16/08	Analyst: DAW
Balance ID: H53865		***Thermometer ID:*** MT 4025	

Digestion Cycles	Start Time	Temp °C	End Time	Temp °C
	15:30	93	17:30	93

Purple color persists or black ppt present: [X] Yes [ ] No If "No", explain in Comments below.

Digestion Tube Lot # :

For dissolved mercury only, were samples filtered in the lab? [ ] Yes [ ] No

One or more samples were filtered prior to analysis at the instrument. [ ] Yes [ ] No

If "yes", then the method blank and the LCS were also filtered in the same manner using the same type of filter.

Analyst(s) Initials: [ ]

Reagents Used

Reagent	Manufacturer	Lot #	Standards Log #	Vol (mL)
HNO_3	JT Baker	E27027		0.25
H_2SO_4	Fisher	E43040		0.5
HCl	JT Baker	E40030		used by instrument
10% $SnCl_2$	Fisher	E20625	STD-1019-08	added by instrument
NaCl / NH_2OH	Fisher	E13654	STD-1032-08	0.6
	Fisher	E09641		
$KMnO_4$	Fisher	C30618	STD-1020-08	1.5
$K_2S_2O_8$	Fisher	062641	STD-6084-07	0.8

Parent Calibration Stock Standards

	Lot #	Verification #	Exp. Date
Second Source	AS-HG2049	STD-2102-07	06/01/08
Primary Calibration	500424	STD-1008-08	04/03/08

Standards Preparation **Final digestate volume = 10 mls**

Standards	Final Conc	Parent Standard	Standards Log #	Vol (mL)	Pipette
Cal Working	10 mg/L	Primary Cal	See Attached Standards Log Printouts	1.00	20
Daily Cal Working	100 ug/L	Cal Working		1.00	20
ICAL 0.2	0.2 ug/L	Daily Cal Working		0.2	20
ICAL 0.5	0.5 ug/L	Daily Cal Working		0.5	20
ICAL 1	1.0 ug/L	Daily Cal Working		1.0	20
ICAL 2	2.0 ug/L	Daily Cal Working		2.0	20
ICAL 5	5.0 ug/L	Daily Cal Working		5.0	19
ICAL 10	10 ug/L	Daily Cal Working		10.0	19
CCV	5 ug/L	Daily Cal Working		5.0	20
ICV Intermed	700 ug/L	ICV Stock		0.70	20
ICV Daily Working	7.0 ug/L	ICV Intermed		1.00	20
LCS	5 ug/L	Daily Cal Working		0.5	20
MS/MSD	5 ug/L	Daily Cal Working		0.5	20
RL	0.2 ug/L	Daily Cal Working		0.2	20

Second Source ICV Intermediate Stock Standard Prep **Standards Log #:** STD-1017-08

NOTE: Details for each reagent & standard prep are documented in the attached Standards Preparation Logbook Record.

Comments

I certify that all information above is correct and complete.

Signature: ***Date:***

REVIEWED BY: _____ **Date:** _____

Attachment 4.

Example Raw Data Checklist

TESTAMERICA-DENVER

Applicable QC Batches:_____

_____

_____

Industrial Mercury Analysis Raw Data Checklist

Revision 2 -March 5, 2008

Analyst's Checklist

	yes	no	n/a
1. Were the special instructions for prep and/or analysis followed?	☐	☐	☐
2. Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3. Is the blank less than the reporting limit or properly anomalized?	☐	☐	☐
4. Is the LCSs within limits or properly anomalized?	☐	☐	☐
5. Is the ICV and all CCVs within limits?	☐	☐	☐
6. Are all CCBs within ± one reporting limit from zero?	☐	☐	☐
7. Were the CCVs and CCBs run with up to 10 samples between each set?	☐	☐	☐
8. Are the reporting limits correct and reflect any dilutions?	☐	☐	☐
9. Are the number of significant figures correctly reported?	☐	☐	☐
10. Are the benchsheets complete (including calibration and standard verification #'s)?	☐	☐	☐
11. Are all comments, footnotes, and anomalies properly documented?	☐	☐	☐
12. Are holding time violation forms completed and attached?	☐	☐	☐
13. Has all sample data been entered into LIMS?	☐	☐	☐
14. Has all QC data been entered into LIMS?	☐	☐	☐
15. Has the data entered into LIMS been checked for errors?	☐	☐	☐
16. For TCLP results is the sample data within 20% of Regulatory Level (0.2 mg/L)?	☐	☐	☐

Analyst's Name:_____ Date:_____

Data Review's Checklist

	yes	no	n/a
1. Have the calculations been checked?	☐	☐	☐
2. Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3. Is all the QC data within the control limits and/or properly anomalized?	☐	☐	☐
4. Are all the significant figures and reporting limits correct?	☐	☐	☐
5. Have any comments, footnotes, and anomalies been properly documented?	☐	☐	☐
6. Have any data errors been documented and entered into LIMS?	☐	☐	☐
7. Is prep date correct in LIMS?	☐	☐	☐
8. Has the data package been copied and filed?	☐	☐	☐
9. If TCLP result within 20% of Reg. Level (0.2 mg/l) and MS < 50%, was MSA performed?	☐	☐	☐

Reviewed by:_____ Date:_____

Comments:_____

_____

_____

Anomalies:_____

_____

_____

Attachment 5.

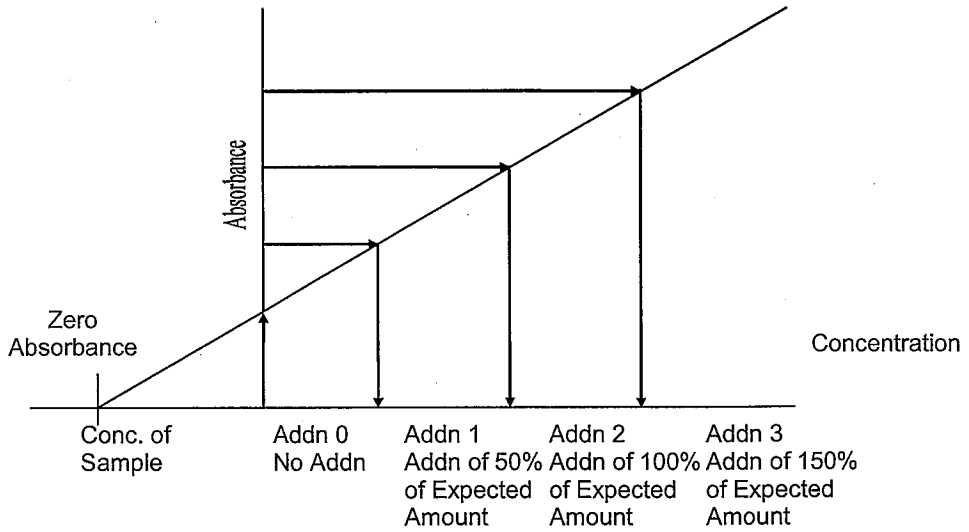
MSA Guidance

Method of Standard Addition (MSA)

Four equal volume aliquots of sample are measured and known amounts of standards are added to three of the 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 an analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. The absorbance (or response) is plotted on the vertical axis 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. The correlation coefficient (r) and the x-intercept (where y=0) of the curve are calculated. 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.

Attachment 6.

Troubleshooting Guide

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak
Erratic Readings	Source lamp not aligned properly Lamp not pre-warmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

Attachment 7.

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 gloves should not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes.
- 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.
- If an unusually high sample is analyzed, segregate the glassware and soak with sulfuric acid prior to routine cleaning.

Attachment 8.

Preventative Maintenance

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, record the date, time, and instrument number; describe the problem; and explain the corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational:

Cold Vapor Atomic Absorption (Leeman PS 200 and CETAC Analyzer)

Daily	Monthly	Annually
Change rinse solution.	Check Hg lamp intensity.	Change Hg lamp.
Optimize light path.		Check liquid/gas separator.
Check nitrogen flow.		
Check tubing. Replace as needed.		
Check drain.		
Check condition of dryer		

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TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

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Title: Mercury in Solids by Cold Vapor Atomic Absorption

[SW 7471A]

Approvals (Signature/Date):

Doug Gomer 8/6/09
Doug Gomer Date
Technical Specialist

Adam W. Alban 05 Aug 09
Adam Alban Date
Health & Safety Manager / Coordinator

Karen Kuoppala 08-07-09
Karen Kuoppala Date
Quality Assurance Manager

Robert C Hanisch 8/7/09
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A.

1.2 Method 7471A is applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, and sludge-type materials. All matrices require sample preparation prior to analysis. This is not an appropriate procedure for the digestion of tissues or other organic matrices, which require the use of EPA 245.6 instead.

1.3 The routine reporting limit for mercury in solid matrices is 17 µg/kg.

2.0 Summary of Method

A representative portion of the sample is digested in aqua regia in the first digestion cycle and potassium permanganate in the second cycle. Mercury is reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorption of light at 253.7nm is calibrated as a function of mercury concentration.

3.0 Definitions

3.1 **Total Mercury:** Inorganic forms of mercury are effectively dissolved by the acids used in the digestion. The potassium permanganate reagent breaks down organo-mercury compounds to inorganic forms that are detected by this procedure.

3.2 **Aqua Regia:** A 3:1 mixture of hydrochloric and nitric acids. This mixture is effective at dissolving metals in the solid form.

4.0 Interferences

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1 Potassium permanganate "suitable for mercury determination" is specified because of the potential for mercury contamination in the reagent. In addition, potassium permanganate crystals will absorb mercury vapors from the air. Reagent bottles must be kept tightly closed to avoid contamination.

4.2 Potassium permanganate, in addition to breaking down organic compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 ppm of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.3 Copper has also been reported to interfere; however, copper concentrations as high as 10 ppm had no effect on the recovery of mercury from spiked samples.

4.4 Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous

chloride is added. 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.

4.5 Interference from certain volatile organic materials that absorb at the wavelength used for the method may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.6 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be lessened by reducing the volume of original sample used. Alternatively, EPA Method 245.6 can be used.

4.7 The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual 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 nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Potassium permanganate is a strong oxidizing agent. It is incompatible and must be stored separately from hydroxylamine hydrochloride and stannous chloride, the reducing agents used in this procedure, and from acids.

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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury Nitrate Solutions	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.
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 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.
Hydroxylamine hydrochloride	Corrosive Poison	No OSHA PEL listed for this compound	Direct contact with skin or eyes causes irritation. May cause skin sensitization, an allergic reaction. Inhalation or ingestion may cause methemoglobinemia and resulting cyanosis (bluish discoloration of skin due to deficient oxygenation of the blood), and labored breathing.
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.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Temperature controlled, mechanically re-circulating water bath capable of maintaining a temperature of 90-95°C.

6.1.2 Mercury Autoanalyzers, either of the following can be used, or equivalent:

6.1.2.1 CETAC Mercury Analyzer with Autosampler and Auto-Diluter

6.1.2.2 Perkin-Elmer FIMS Mercury Analyzer with Autosampler

6.2 Supplies

- Digestion Tubes – disposable glass, 18mm x 150mm, plastic cap
- Argon, 99.999% purity
- Calibrated automatic pipettes or Class A glass volumetric pipettes (see SOP # DV-QA-0008 for details on calibrating mechanical pipettes)
- Class A volumetric flasks.
- Thermometer, non-mercury column, accurate to ±1°C at 95 °C (see SOP # DV-QA-0001 for calibration details).
- Disposable cups or tubes.
- Glass beads, <1 mm diameter, acid washed.
- 0.45 µm syringe filters

7.0 Reagents and Standards

The preparation of reagents and standards is recorded in Standards Log, a computerized database. The reagents and standards are listed on the bench sheet and the pipettes and/or balances used in the preparations are also listed on the bench sheet. A printout from Standards Log accompanies the bench sheet to provide preparation details for each prepared reagent and standard.

7.1 **Reagent water:** Reagent water 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 **Nitric acid (HNO3):** concentrated, trace metal grade or better.

7.3 **Hydrochloric acid (HCl):** concentrated, trace metal grade or better.

7.4 **Calibration Blank, Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Method Blank (MB), 1% HNO3:**

7.4.1 Add 0.5 L of concentrated HNO3 to a 50-L carboy partially filled with reagent water.

7.4.2 Dilute to 50 L with reagent water.

7.5 **Stannous Chloride (SnCl2) Solution, reagent grade, 1.3% (w/v) per manufacturer's instructions. (PE FIMS Only)**

7.5.1 Place approximately 100 mL of deionized water into a 2-L volumetric flask.

7.5.2 Slowly add 60 mL of concentrated HCl to the flask and swirl to mix.

7.5.3 Add 26.4 grams of SnCl2 to the flask.

7.5.4 Place a large stir bar in the flask and put the flask on a stir plate.

7.5.5 Stir the contents of the flask until the reagent is completely dissolved.

7.5.6 Remove the stir bar and make to volume with deionized water.

7.6 Stannous Chloride Solution (SnCl2), reagent grade, 10% (w/v) per manufacturer's instructions. (CETAC Only)

7.6.1 Place approximately 100 mL of deionized water into a 2-L volumetric flask

7.6.2 Slowly add 200 mL of concentrated HCl to the flask and swirl to mix.

7.6.3 Add 200 grams of SnCl2 to the flask.

7.6.4 Place a large stir bar in the flask and put the flask on a stir plate.

7.6.5 Stir the contents of the flask until the reagent is completely dissolved.

7.6.6 Remove the stir bar and make to volume with deionized water.

7.7 Sodium chloride-hydroxylamine hydrochloride solution:

Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride (reagent grade) to every 100 mL of reagent water.

NOTE: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.8 Potassium permanganate, 5% solution (w/v):

Dissolve 5 g of potassium permanganate (reagent grade, "suitable for mercury determination") for every 100 mL of reagent water.

7.9 Purchased Mercury Stock Solutions

Primary Mercury Calibration Standard Solution, 1,000 mg/L

7.10 Calibration Working Standard Solution, 10 mg/L

7.10.1 Add approximately 90 mL of 1% HNO3 to a 100 mL Class A volumetric flask.

7.10.2 Pipet 1.00 mL of the 1000 mg/L primary mercury calibration standard solution into the flask.

7.10.3 Dilute to the mark on the flask with 1% HNO3.

7.10.4 Stopper the flask and shake to mix.

7.10.5 Transfer the solution to a 125-mL Nalgene bottle.

7.10.6 Document the preparation of the solution in the Standards Log database.

7.10.7 Prepare this solution fresh monthly or more often if necessary.

7.11 Daily Calibration Working Solution (100 µg/L)

7.11.1 Add approximately 90 mL of 1% HNO3 to a 100-mL volumetric flask.

7.11.2 Add 1.00 mL of the 10 mg/L Calibration Working Standard (see section 7.10).

7.11.3 Bring the solution to a final volume of 100.0 mL.

7.11.4 Stopper and mix thoroughly.

7.11.5 Document the preparation in the Standards Log database.

7.11.6 Prepare this solution each day prior to calibration.

7.12 Initial Calibration (ICAL) Standards

The initial calibration standards are prepared directly in the digestion tubes as follow:

ICAL	Daily Cal Working Std (mL)	1% HNO3 (mL)	Final Conc (µg/L)
Blank	0.0	5.0	0.0
Std 1	0.1	4.9	0.20
Std 2	0.25	4.75	0.50
Std 3	0.5	4.5	1.0
Std 4	1.0	4.0	2.0
Std 5	2.5	2.5	5.0
Std 6	5.0	0.0	10.

7.13 Second-Source Initial Calibration Verification (ICV) Daily Intermediate Standard, approximately 700 µg/L.

Add 700uL of the 100 mg/L ICV Standard to a 100-mL volumetric flask partially filled with 1% HNO3 and dilute to the mark. Record this information in the Standards Log database.

7.14 Second-Source Initial Calibration Verification (ICV) Daily Working Standard, approximately 7.00 µg/L.

Add 0.5 mL of the 700 µg/L ICV Daily Intermediate Standard (see section 7.13) to a soil digestion tube and add 4.5 mL of 1% HNO3. Record this information in the Standards Log database.

7.15 Continuing Calibration Verification (CCV) Standards, 5 µg/L

7.15.1 The CCVs are prepared exactly as the 5.0 µg/L ICAL standard shown above (see section 7.12).

7.15.2 Prepare sufficient volume of the standard for analysis of a CCV after every 10 samples.

7.16 Laboratory Control Standard (LCS), 417 µg/kg

7.16.1 The LCS is prepared in an empty digestion tube for which 0.6 g of glass beads are used.

7.16.2 Add 2.5 mL of the 100 µg/L Daily Calibration Working Standard (see section 7.11) to a digestion tube. No additional reagent water is added at this time, per method, but is accounted for when digestate is brought to the final volume of 50 mL.

7.16.3 This is equivalent to a 5.0 µg/L ICAL standard, which is the concentration that appears on the raw data printout from the instruments.

7.17 Matrix Spike and Matrix Spike Duplicate (MS/MSD), 417 µg/kg

MS/MSD pairs are spiked in the same manner as the LCS (see section 7.16) and prepared in the same manner as the samples, using 0.6 g of sample.

7.18 Reporting Limit (RL) Check Standard, 17 µg/kg

7.18.1 Add 0.1 mL of 100 µg/L Daily Calibration Working Standard (see section 7.11) and 4.9 mL of reagent water to a digestion tube.

7.18.2 This is equivalent to a 0.2 µg/L ICAL standard, which is the concentration that appears on the raw data printout from the instruments.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils	Glass	3 grams	Cool 4 ± 2°C	28 Days	N/A

9.0 Quality Control

This section describes routine quality control practices, which are also summarized in Attachment 2. Preparation of QC materials is described in Section 7. Initial calibrations and calibration verifications are discussed in Section 10. Initial performance studies are described in Section 12. Current control limits are stored in the laboratory LIMS system.

9.1 The process of establishing control limits, and the use of control charts are described more completely in DV-QA-003P, Quality Control Program.

9.2 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP # DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents.

9.4 Preparation Batch

A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate pair (MS/MSD). As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.5 Method Blank (MB)

The MB consists of an empty vessel or <1-mm glass beads (for DoD and AFCEE projects) containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. At least one method blank (MB) must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than the reporting limit or less than 10% of the mercury concentration found in the associated samples, whichever is higher. Some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If mercury was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.6 Laboratory Control Sample (LCS), 417 µg/kg

The preparation of the LCS is described in Section 7.16. At least one aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure.

Acceptance Criteria: Maximum control limits for LCS recoveries are 80-120%. In-house control limits based on three standard deviations of the mean of historical results are used as long as they are at least as tight as 80-120% (see Policy QA-003 for further details on establishing control limits).

Corrective Action: If LCS recoveries are outside established control limits, the system is out of control and corrective action must occur. If recoveries are above control limits and mercury is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD), 417µg/kg

One MS/MSD pair must be processed for each preparation batch. Some programs may require the use of sample duplicates in place of or in addition to MS/MSDs. In addition, some programs will allow spikes to be reported only for project-related samples. Samples identified as field blanks cannot be used for MS/MSD analysis.

Acceptance Criteria: Control limits are statistically determined based on three standard deviations of the mean of the laboratory's historical data. The MS/MSD recovery must fall within 75-125%; the relative percent difference (RPD) between the MS and MSD cannot exceed 20%.

Corrective Action: If analyte recovery or RPD fails acceptance criteria, the LCS recovery must be in control for the data to be reported. If there is no evidence of analytical problems and all other QC criteria are met, then qualified results may be reported and the situation must be described in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC, then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

9.8 Serial Dilution

Some programs (e.g., DoD and AFCEE programs) require that a fivefold (1+4) dilution must be included in each analytical batch for each sample matrix.

Acceptance Criteria: The results must be within 10% of the expected value, assuming that the sample concentration is at least 25x the MDL concentration.

Corrective Action: If the result fails the acceptance criteria, all associated sample results must be qualified.

9.9 Post-Digestion Spikes

Some programs require the inclusion of a post-digestion spike in each analytical batch. The post-digestion spike is prepared by adding 0.5 mL of the 100 µg/L Daily Calibration Working Solution to 10 mL of filtered sample digestate. Post-digestion spikes are performed as an additional check for matrix interference.

Acceptance Criteria: The percent recovery limits for the post-digestion spike are 85 to 115%.

Corrective Action: If the result fails the acceptance criteria, all associated sample results must be qualified.

9.10 Method of Standard Addition (MSA)

The method of standard additions is an option for the analysis of samples shown to have significant matrix effects, e.g., unacceptably low MS/MSD recoveries or under certain conditions for TCLP analysis (see Attachment 4 for details).

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM shall be filed in the project file.

10.1 Sample Preparation

10.1.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB), as well as the field samples, are processed through the digestion procedure.

10.1.2 Incrementally select at least 3 small subsamples of a well mixed sample to obtain a total of 0.6 ± 0.12 g of sample and place the subsamples into a sample digestion tube. (See DV-QA-0023 for additional information on subsampling.)

10.1.3 Prepare an MB, LCS, MS, and MSD for each batch. The MB is either an empty digestion tube or is prepared by placing 0.6 g of glass beads in a digestion tube, depending on client requirements. The LCS is prepared by adding 2.5 mL of the 100 µg/L Daily Calibration Working Solution to a digestion tube. The MS is prepared by adding 2.5 mL of the 100 µg/L Daily Calibration Working Solution to a digestion tube containing a second aliquot of the chosen sample. The MSD is prepared in the same manner as the MS using a third aliquot of the chosen sample.

10.1.4 Add 5.0 mL of reagent water to all un-spiked field samples and the method blanks. Add 2.5 mL of reagent water to the LCS, MS and MSD.

10.1.5 Add 3.75 mL of concentrated HCl and 1.25 mL of concentrated HNO3 mixing after each addition.

10.1.6 Heat for 2 minutes in the water bath at 90 - 95 ° C. Record the start and stop times and the temperature on the bench sheet.

10.1.7 Allow the samples and standards to cool at room temperature.

10.1.8 Add 19 mL of reagent water.

10.1.9 Add 15 mL of 5% potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may need to 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 up to 25 mL additional permanganate, the color does not persist, sample dilution prior to reanalysis may be required.

NOTE: It is important that equal volumes of the potassium permanganate solution are added to all solutions in the batch. Unequal volumes used with the automated method will result in dilution errors. Unequal volumes used with the manual method will result in differing purging efficiencies.

10.1.10 Cap the samples and standards and heat for 30 minutes in a water bath at 90 - 95°C. Record the start and stop times and the temperature on the bench sheet. The analyst will verify that a purple color persists or a black precipitate is present after the thirty minutes of heating. If this is not true, the digestion must be repeated using more potassium permanganate or less sample.

10.1.11 Allow the samples and standards to cool at room temperature.

10.1.12 Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate.

10.2 Calibration

All calibration standards are digested together with samples, as described in Section 10.1, prior to analysis. Preparation of calibration materials is described in Section 7.

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).

10.2.1 Initial Calibration (ICAL)

10.2.1.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)*.

10.2.1.2 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.

10.2.1.3 Calibrate using six standards and a blank (see section 7.12).

NOTE: It is generally not acceptable to reject calibration points for this method.

10.2.1.4 The calibration curve must have a correlation coefficient of ≥ 0.995 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.

10.2.2 Initial and Continuing Calibration Blanks

10.2.2.1 An initial calibration blank (ICB) is tested immediately after the daily ICAL standards.

Acceptance Criteria: The absolute value of the blank result must be less than the reporting limit. As noted with the method blank, some programs require that

results for blanks must be less than 2X the MDL (refer to special project requirements).

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

10.2.2.2 Continuing calibration blanks (CCBs) are run after every 10 samples and at the end of the run.

Acceptance Criteria: The absolute value of the blank result must be less than the reporting limit. As just noted, some programs require that results for blanks must be less than 2X the MDL (refer to special project requirements).

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

10.2.3 Initial Calibration Verification (ICV), approximately 7.0 µg/L

The accuracy of the calibration standards is verified by testing a second source standard (ICV).

Acceptance Criteria: The ICV recovery must be within 90-110%.

Corrective Action: If the ICV acceptance limit is exceeded, the analysis should be terminated, the accuracy of the calibration standards checked, and the instrument recalibrated.

10.2.4 Reporting Limit Check Standard (RL), 0.2 µg/L

The accuracy of results at the reporting limit is verified by testing a standard in every analytical run that is prepared at the reporting limit concentration.

Acceptance Criteria: The results for this standard must be within 50% of the expected value (20% for some programs).

Corrective Action: If the RL check acceptance limit is exceeded, the analysis should be terminated, the instrument operation checked, the instrument recalibrated, and associated samples reanalyzed.

10.2.5 Continuing Calibration Verification (CCV), 5.0 µg/L

Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the run.

Acceptance Criteria: The CCV recovery must be within 80-120%.

Correction Action: Sample results may be reported only when bracketed by valid CCV pairs. If a mid-run CCV fails, the CCV may be re-analyzed once without modification to the instrument's operating conditions. If the re-analyzed CCV is found to be in control, the CCV analysis must be repeated with successful results or the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV failure was not directly instrument related, the associated samples must be re-prepared and reanalyzed.

10.3 Sample Analysis

NOTE: Because of differences between various makes and models of CVAA instrumentation, detailed push-button operating instructions are not provided here. Refer to the specific instrument operating manual for detailed autosampler setup and operation protocols.

NOTE: The injection of samples and the addition of stannous chloride are done automatically by the instrument. Refer to the specific instrument manual for details.

10.3.1 Set up the instrument and autosampler according to the manufacturer's instructions.

10.3.2 Allow the samples to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.3.3 Filter all samples and standards prior to analysis using a 0.45-µm syringe filter.

10.3.4 Analyze the standards and samples according to the manufacturer's instructions.

10.3.5 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for mercury concentrations that exceed the highest calibration standard.

10.3.6 If the sample results are negative and the absolute value is greater than the reporting limit, the sample must be reanalyzed.

10.3.7 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB. Re-sloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

10.3.8 The analytical sequence listed below must be followed. Refer to Quality Control Section 9.0 and Table I (Appendix A) for quality control limits.

Instrument Calibration

ICB

ICV

RL

Maximum of 10 samples

CCV

CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run

CCV

CCB

NOTE: Samples included in the count between CCVs include the method blank, LCS, MS, MSD, and field samples.

10.3.9 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Concentration

$$\text{Concentration} = \text{mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:

C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4 Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Sample preparation bench sheet(s), to include the batch number, list of samples, preparation analyst and date, instrument analysis analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes). See Attachment 3.
- Raw data (direct instrument printout).

- Run log printout from instrument software where this option is available or manually-generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Attachment 3
- Standards Documentation to include source, lot, preparation date, and expiration date. A printout from Standards Log provides all required information except identification of measuring equipment used.
- Nonconformance summary (if applicable).

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial MDL study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

12.1.1 Prepare seven standards at three to five times the estimated MDL or the currently established MDL concentration.

12.1.2 Digest and analyze the MDL standards as described in Section 10.

12.1.3 Calculate the mean concentration found (X) in µg/L, and the standard deviation of the mean concentration in µg/L, for each analyte. Then, calculate the MDL (single-tailed, 99% confidence level) for each analyte.

12.1.4 MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system.

12.1.5 See Policy DV-QA-005P for further details concerning MDL studies.

12.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard.

12.2.1 Prepare an MDLV standard at 1-2 times the calculated MDL concentration.

12.2.2 Analyze the MDLV standard immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

12.2.3 The calculated MDL is verified if the MDLV standard is detected, nominally signal to noise ratio > 3, under routine instrument conditions.

12.2.4 If the first MDLV is not detected, re-prepare the MDLV standard at twice the original concentration and analyze. The lowest concentration that produces a detectable signal will then be reported as the MDL.

12.2.5 For each day, the mean and standard deviation for the seven standards are calculated. The final IDL concentration is three times the average of the three daily standard deviation values.

12.2.6 See Policy DV-QA-014P for a discussion of IDL studies and evaluation of IDL results.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

12.3.1 Analyze four aliquots of the LCS 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.

12.3.2 Calculate the mean recovery and standard deviation of the mean recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria for the LCS, 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.

12.3.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The group/team leader has the responsibility for ensuring that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

13.0 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).

14.0 Waste Management

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 procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.1 The following waste streams are produce when this method is carried out:

- Aqueous Acidic (Metals) - Corrosive - (J)

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

- Expired reagents and standards – Contact the Waste Coordinator.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, March 2005.

15.3 U.S.EPA Statement of Work for Inorganic Analysis, ILMO3.0

16.0 Method Modifications:

Item	Method	Modification
1	7471A	An additional QC analysis, RL verification, is added
2	7471A	Methods 7470A and 7471A state that working standards "should be prepared fresh daily." The laboratory frequently prepares up to three batches of mercury samples, including digested calibration standards, each day. The third batch is typically prepared and digested late in the day, and then is analyzed the morning of the next day. The laboratory had developed the following information demonstrating that analysis within 24 hours, but on the second calendar day from preparation produces reliable results and is acceptable to the EPA: • Successful proficiency testing (PT) results for samples that were prepared and analyzed within 24 hours, but on successive days • Successful analysis of true NIST mercury standards within every analytical batch; and • A written comment from the EPA MICE Hotline stating that, with the supporting lab data, their opinion was that the laboratory's practice is "within the letter of the method as written."
3	7471A	Chapter 1 of SW-846 specifies the use of reagent water with a purity equivalent of ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
4	7471A	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.

17.0 Attachments

Attachment 1: Mercury Preparation & Analysis Flowchart
Attachment 2: Summary of Quality Control Requirements
Attachment 3: Example Supplemental Metals Prep Sheet for Hg Prep & Analysis - Solids
Attachment 4: Example Data Review Checklist
Attachment 5: MSA Guidance
Attachment 6: Instrument Maintenance

18.0 Revision History

- Revision 2.2, dated 07 August 2009
  - Removed Reagent Blank from Section 7.4
  - Changed table header name in section 7.12 to say 1%HNO3 from Reagent Water
  - Changed sections 7.13 and 7.14 to use 1% HNO3 from reagent blank

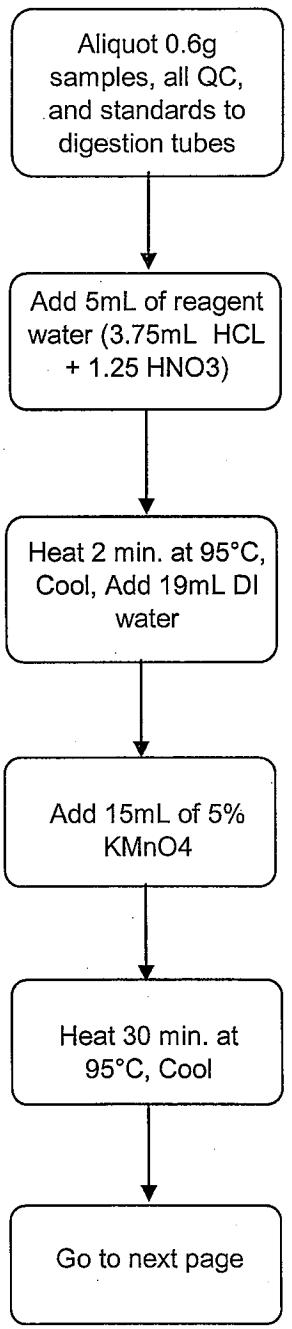
- Revision 2.1, dated 16 February 2009
  - Section 10.2.5: Update the corrective action to match the other Hg SOPs
  - Deleted Section 12.3 for IDL requirement

- Revision 2, dated 28 December 2007
  - Integration for TestAmerica and STL operations.
  - Changed aliquot size from 0.3g to 0.6g.
  - Made changes to concentration to reflect the aliquot change throughout the SOP.
  - Reformatted the SOP.

- Revision 1, dated 11 October 2005
  - The method summary has been updated to reflect the actual procedure performed.
  - The definition of aqua regia was corrected to indicate three parts HCl to one part HNO3.
  - Corrected section 10.1.5 to require the addition of 1.9 ml of HCl and 0.6 ml of HNO3
  - In section 10.2.3, changed the true value for the ICV to "approximately 2.0 µg/L," since the value can change slightly each time the standard is prepared.
  - Added the instruction to digest the IDL solutions in the same way as samples are digested to section **Error! Reference source not found.**, and clarified that the IDL is three times the average of the standard deviations for the three daily IDL studies.
  - Corrected section 10.1.10 to add verification that the permanganate color persists during the thirty minute digestion.
  - Corrected 10.1.12 to reflect the laboratory's preference for using hydroxylamine hydrochloride to reduce the excess permanganate.
  - Added recipe for 1.3 % SnCl2 for the PE FIMS analyzer to section 7.5.
  - Corrected sections 7.10 and 7.11 to reflect a volumetric preparation rather than gravimetric.
  - Added instructions to prepare reagent blank in section 7.4.
  - Added detailed preparation information for the ICV.
  - Added instructions for preparing the MB, LCS, MS, and MSD to section 10.1.3.
  - Added instructions add 2.5 ml of reagent water to the MB and field samples to compensate for the volume of the spikes added to the samples and the LCS in section 10.1.4.

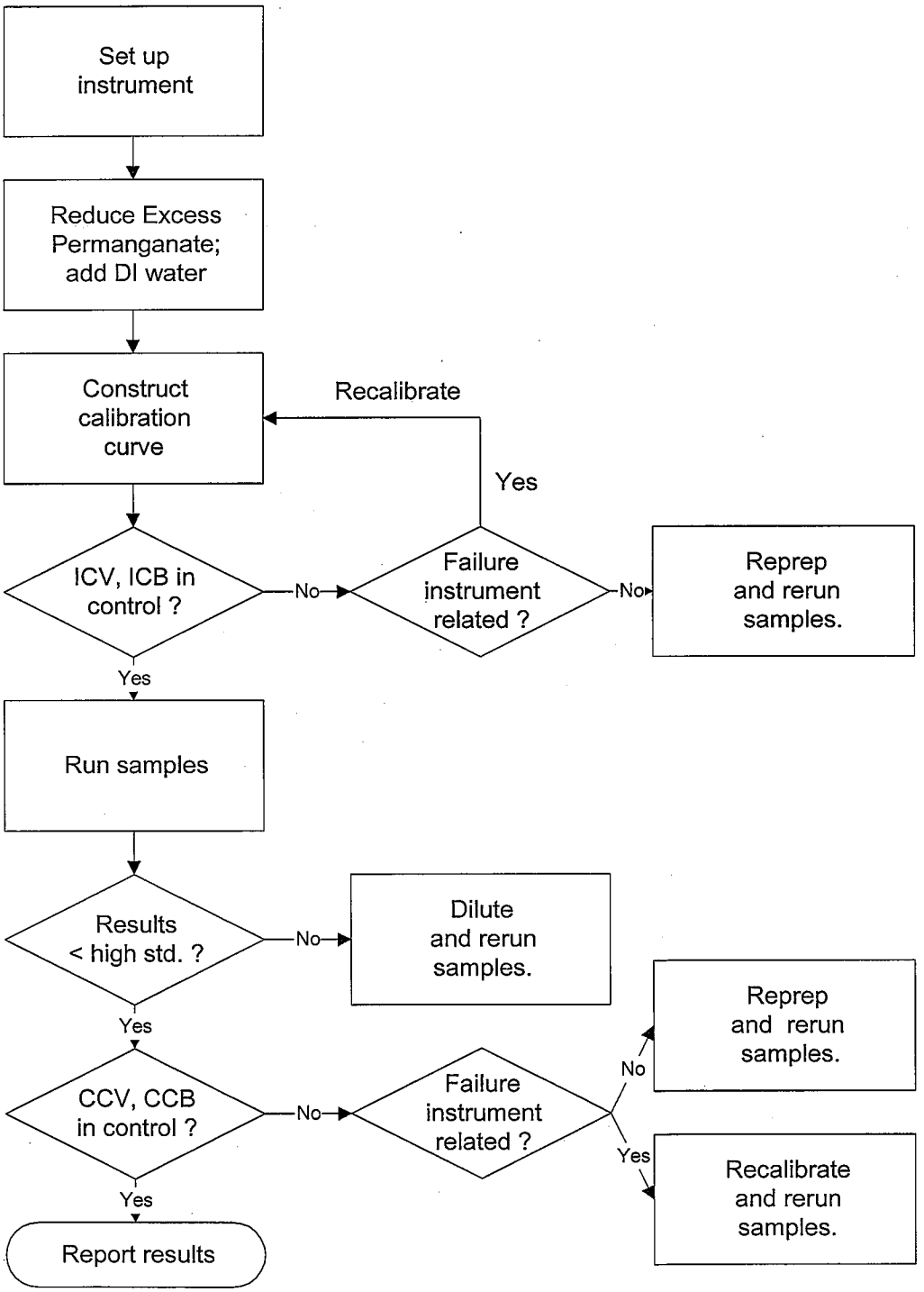
Attachment 1.

Mercury Preparation & Analysis Flowchart



Attachment 1.

Mercury Preparation & Analysis Flowchart (continued)



Attachment 2.

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICB	Immediately following ICAL	Absolute value < RL (2x MDL for DoD)	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
ICV	Following ICB	90- 110% recovery	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
RL Check Standard	Following the ICV	50-150% recovery (80-120% for DoD)	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
CCV	Every 10 samples and at the end of the run	80 - 120 % recovery.	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
CCB	Immediately following each CCV	Absolute value < RL (2x MDL for DoD)	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
Method Blank	One per sample preparation batch of up to 20 samples.	≤RL (≤1/2 the RL for DoD) Sample results greater than 10% the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion	Redigest and reanalyze samples. Note exceptions under criteria section.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 80 - 120%	Terminate analysis; correct the problem; redigest and reanalyze all samples associated with the failed LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 75-125%	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate	See Matrix Spike	Recovery within statistical control limits, not to exceed 75-125 % recovery or in-house control limits; RPD ≤ 20%	See Corrective Action for Matrix Spike.

Attachment 3.

Example Supplemental Metals Prep Sheet for Hg Prep & Analysis - Solids

SUPPLEMENTAL METALS PREP SHEET
(Used in conjunction with METALS PREP LOG/BATCH SUMMARY)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
TestAmerica Denver

Hg PREP & ANALYSIS - SOLIDS

SOP: DEN-MT-0016 **QC Batch #:** _____

Prep Date:	Prep By:	Analysis Date:	Analyst:
Balance ID: H53865		***Thermometer ID:***	

Digestion Cycles	Start Time	Temp °C	End Time	Temp °C

Purple color persists or black ppt present: ☐ Yes ☐ No If "No" explain in Comments below.

Digestion Tube Lot # : **Syringe Filter Lot # :**

Reagents Used

Reagent	Manufacturer	Lot #	Standards Log #	Volume (mL)
HNO_3	Fisher	1105080		9
HCl	Fisher	4105090		0.5
$SnCl_2$	Fisher	050966		added by instrument
$NaCl / NH_2OH$	Fisher	051512		3
	Fisher	045294		
$KMnO_4$	Fisher	050469		as needed

Parent Calibration Stock Standards

	Lot #	Verification #	Exp. Date
Second Source	991304	STD-3261-04	06/14/06
Primary Calibration	E60400	STD-1981-03	04/11/06

Standards Preparation

Standards	Final Conc	Parent Standard	Standards Log #	Vol (mL)	Pipette
Cal Working	10 mg/L	Primary Cal		1.00	
Daily Cal Working	100 ug/L	Cal Working	See	1.00	
ICAL Blank	0 ug/L	Daily Cal Working	Attached	0.0	
ICAL 1	0.2 ug/L	Daily Cal Working	Standards Log	0.1	
ICAL 2	0.5 ug/L	Daily Cal Working	Printouts	0.25	
ICAL 3	1.0 ug/L	Daily Cal Working		0.5	
ICAL 4	2.0 ug/L	Daily Cal Working		1.0	
ICAL 5	5.0 ug/L	Daily Cal Working		2.5	
ICAL 6	10 ug/L	Daily Cal Working		5.0	
ICV Daily Intermediate	200 ug/L	ICV Intermediate		1.00	
ICV Daily Working	7 ug/L	ICV Daily Intermed.		0.5	
CCV	5 ug/L	Daily Cal Working		2.5	
LCS & MS	333.4 ug/kg	Daily Cal Working		2.5	
MD	333.3 ug/kg	Daily Cal Working		0.1	

Second Source ICV Intermediate Standard Prep **Standards Log #:**

Weight of Second Source Standard: grams **Conc:** mg/L

NOTE: Details for each reagent and standard preparation are documented in the attached Standards Preparation Logbook Record.

Comments:

Analyst: _____ **Date:** _____

Attachment 4.

Example Data Review Checklist

TESTAMERICA-DENVER

Applicable QC Batches:_____

_____

_____

Industrial Mercury Analysis Raw Data Checklist

Revision 3 -December 19, 2007

Analyst's Checklist

	yes	no	n/a
1. Were the special instructions for prep and/or analysis followed?	☐	☐	☐
2. Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3. Is the blank less than the reporting limit or properly anomalized?	☐	☐	☐
4. Is the LCSs within limits or properly anomalized?	☐	☐	☐
5. Is the ICV and all CCVs within limits?	☐	☐	☐
6. Are all CCBs within ± one reporting limit from zero?	☐	☐	☐
7. Were the CCVs and CCBs run with up to 10 samples between each set?	☐	☐	☐
8. Are the reporting limits correct and reflect any dilutions?	☐	☐	☐
9. Are the number of significant figures correctly reported?	☐	☐	☐
10. Are the benchsheets complete (including calibration and standard verification info)?	☐	☐	☐
11. Are all comments, footnotes, and anomalies properly documented?	☐	☐	☐
12. Are holding time violation forms completed and attached?	☐	☐	☐
13. Has all sample data been entered into LIMS?	☐	☐	☐
14. Has all QC data been entered into LIMS?	☐	☐	☐
15. Has the data entered into LIMS been checked for errors?	☐	☐	☐
16. For TCLP results, is the sample data within 20% of Regulatory Level (0.2 mg/L)?	☐	☐	☐

Analyst's Name:_____ Date:_____

Data Review's Checklist

	yes	no	n/a
1. Have the calculations been checked?	☐	☐	☐
2. Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3. Is all the QC data within the control limits and/or properly anomalized?	☐	☐	☐
4. Are all the significant figures and reporting limits correct?	☐	☐	☐
5. Have any comments, footnotes, and anomalies been properly documented?	☐	☐	☐
6. Have any data errors been documented and entered into LIMS?	☐	☐	☐
7. Is prep date correct in LIMS?	☐	☐	☐
8. Has the data package been copied and filed?	☐	☐	☐
9. If TCLP result within 20% of Reg. Level (0.2 mg/l) and MS < 50%, was MSA performed?	☐	☐	☐

Reviewed by:_____ Date:_____

Comments:_____

_____

_____

Anomalies:_____

_____

_____

Attachment 5.

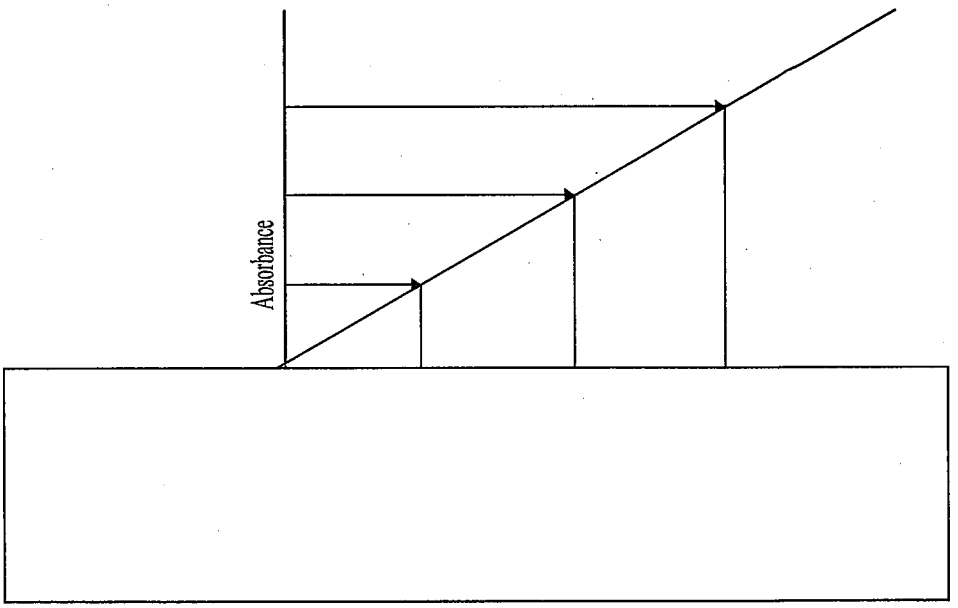
MSA Guidance

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.

Attachment 6.

Instrument Maintenance

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, record the date, time and instrument number, then identify the problem and corrective action in the maintenance log. When the instrument is returned to service, record the return to service, the date, and any tests performed to verify proper operation.

The following preventative maintenance procedures are <u>required</u> to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption

Daily	Monthly	Annually
Change rinse solution.	Check Hg lamp intensity.	Change Hg lamp.
Optimize light path.		Check liquid/gas separator.
Check argon flow.		
Check tubing. Replace as needed.		
Check drain.		
Check condition of dryer		

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TestAmerica Laboratories, Inc.
TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002

Phone: 303-736-0100
Fax: 303-431-7171

Title: QA/QC Requirements for Federal Programs

Approvals (Signature/Date):

Karen Kuoppala 10-06-09
Karen Kuoppala Date
Quality Assurance Manager

Robert C Hanisch 10/6/09
Robert C. Hanisch Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 PURPOSE

This policy describes TestAmerica Denver requirements for ensuring compliance with Federal Program quality assurance (QA) and quality control (QC) requirements for environmental analyses as dictated in the Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), DoD Perchlorate Handbook, Department of Energy Quality Systems for Analytical Services (DOE QSAS), and HQ Air Force Center for Environmental Excellence Guidance for Control Deliverables, Appendix C: Quality Assurance Project Plan (AFCEE QAPP), all of which prescribe requirements that are more stringent than or otherwise differ from requirements in the laboratory's standard procedures. Variance requests, if needed, will be provided to TestAmerica Denver clients on a project-specific basis.

2.0 SCOPE

2.1 This policy shall be enforced and followed throughout the laboratory.

2.2 This policy includes QA/QC requirements of the following Federal environmental analysis program requirements documents:

Federal Department	Document Title	Version
Department of Defense (DoD)	Quality Systems Manual for Environmental Laboratories (QSM)	Version 3 Final, January 2006
Department of Defense (DoD)	Quality Systems Manual for Environmental Laboratories (QSM)	Version 4.1 Final, April 2009
Department of Defense (DoD)	Perchlorate Handbook	August 2007
Department of Energy (DOE)	Quality Systems for Analytical Services	Revision 2.4, October 2008
HQ Air Force Center for Environmental Excellence (AFCEE)	Appendix C: Quality Assurance Project Plan (QAPP)	Version 4.0.02, May 2006

2.3 Any additional or contradictory instructions in approved project planning documents take precedence over those given in this policy.

2.4 General requirements for review of project objectives for any program prior to work acceptance are described in Section 7.0 of the TestAmerica Denver Quality Assurance Manual (QAM).

2.5 Further details concerning contract review, verification of certification requirements, determination of project-specific technical requirements (e.g., methods, analyte lists, QC requirements), TAT, deliverables, documentation of project requirements, and organizational responsibilities are given in Policy DV-QA-005P, "Project Information Requirements."

3.0 SAFETY

3.1 There are no specific safety hazards associated with this SOP.

3.2 During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 Detection Limit (DL): The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%.

4.2 Grey Boxes: Both the DoD QSM and DOE QSAS are based on the NELAC Quality Standards, Chapter 5 and use the same formatting and numbering. Both the DoD and DOE documents use boxes shaded in grey to add clarifications or additional requirements that are more explicit or go beyond those in the NELAC Standard.

4.3 Initial Calibration Verification (ICV): This term has a slightly different meaning in the DoD QSM than typical usage. In the DoD QSM, it refers to the first calibration verification standard run each day. TestAmerica Denver SOPs use this term to mean a second-source standard analyzed immediately following the initial calibration.

4.4 Limit of Detection (LOD): The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

4.5 Limit of Quantitation (LOQ): The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

4.6 Marginal Exceedances: Sporadic failures of LCS results outside of the 3-standard-deviation control limits, but less than 4 standard deviations. The NELAC Standard, DoD QSM, AFCEE QAPP, and DOE QSAS define the number of marginal exceedances allowed as a function of the number of analytes in the LCS.

4.7 Method Detection Limit (MDL): Detection limits as determined according to testAmerica Denver Policy DV-QA-005P. Also called "limit of detection" or LOD by NELAC. In almost all cases, MDLs or LODs are determined according to the instructions provided in 40 CFR Part 136B. If a different approach is used for a specific method, that approach is documented in the SOP.

4.8 Limit of Detection Verification (LODV): LOD verification is the process of conducting a test to prove that the calculated 40CFR-136-B detection limit is reliable. The verification of the LOD shall be confirmed by qualitative identification of the analytes in a QC sample in each quality system matrix at 2-3 times the LOD for single analyte tests or 3-4 times the LOD for multiple analyte methods. The LOD shall be verified quarterly.

4.9 Limit of Quantitation Verification (LOQV): LOQ verification is the process of conducting a test to demonstrate recovery of the analyte within the method acceptance criteria, or client data quality objectives for accuracy. The LOQ shall be confirmed by successful analysis of

a QC sample containing each analyte of concern in each quality system matrix at 1-2 times the claimed LOQ.

4.10 Reporting Limit (RL): Standard RLs are determined according to TestAmerica Denver Policy DV-QA-009P. Both the DoD and AFCEE specify requirements for RLs with respect to MDLs. These distinctions are included in this SOP.

4.11 NELAC: National Environmental Laboratory Accreditation Conference. A voluntary association of state and federal officials who foster the generation of laboratory data of known and documented quality through adoption of national standards.

4.12 QAPP: Quality Assurance Project Plan

4.13 USACE: United States Army Corps of Engineers, one of the DoD services.

5.0 POLICY

5.1 Work falling under the requirements of any of the Federal programs listed in Section 2.2 will be conducted in accordance with the practices given in this policy, unless changes are specifically discussed in advance with a client and approved in writing.

5.2 Any requirements in this policy that are more stringent or otherwise different than the laboratory standard operating procedures (SOPs) or the LQM take precedence and must be followed, unless superseded by approved project-specific instructions.

5.3 Although this policy presents the requirements of the most current version of each of the guidance documents listed in Section 2.2, the laboratory may still receive samples that are governed by a previous version, which may differ from the current version. Project Managers must include a notice to analysts in the Client Requirements section of quotes in the LIMS to inform analysts which version of the guidance document applies.

5.4 Methods and compounds beyond those given in the respective Federal guidance documents may require changes to some of the general QC requirements. For example, special consideration would be needed for analysis of the full Appendix IX list of compounds. All such changes will be discussed with the client in advance, and will not be used until approved.

6.0 RESPONSIBILITIES

6.1 The bench level analysts, group leaders, and data reviewers are responsible for following the requirements specified in this policy for DoD, AFCEE, and DOE projects.

6.2 The Project Manager is responsible for informing the laboratory via LIMS test codes, special instructions in LIMS, and QASs, the necessity of applying DoD, AFCEE, or DOE requirements, as appropriate. In the communication to the analysts, the Project Manager must differentiate work involving different versions of the guidance documents. The Project Manager is also responsible for a final review of the data package. The Project Manager reviews for completeness, accuracy of deliverables, and correct invoicing. In addition, the Project Manager must inform the QA Manager when the first data packages will be available for the required QA review.

6.3 The Customer Service Manager is responsible for communicating the laboratory's capabilities to clients, including making arrangements for review and approval of all required variances and clarifications, as specified in "TestAmerica Denver Technical Approach to the DoD QSM" and "TestAmerica Denver Technical Approach to AFCEE QAPP Version 4.0."

6.4 The Quality Assurance Manager (QAM) is responsible for ensuring that work performed by the laboratory complies with the DoD QSM, DoD Perchlorate Handbook, AFCEE QAPP, or DOE QSAS, as applicable. The QAM is responsible for setting up structured analysis codes (SACs) in the LIMS that incorporate the appropriate MDLs, RL, data flagging, and control limits as specified in the applicable requirements document, i.e., DoD QSM, AFCEE QAPP, DoD Perchlorate Handbook, or DOE QSAS. The QAM is also responsible for QA oversight, which includes review of non-conformances, review of control charts, and other performance indicators. These QA activities are explained in more detail in the Quality Assurance Manual (QAM).

7.0 REVISION HISTORY

Revision 5.1, dated 18 September 2009
- Clarified requirements for QA review of DoD data packages.
- Edited Attachments to reflect current practice.

Revision 5.0, dated 26 August 2009
- Clarified CCV criteria for dual column.
- Added requirements for DoD Version 4.1 of the QSM.

Revision 4.3, dated 08 June 2009
- Added requirements for DoD Version 4.1 of the QSM.

Revision 4.2, dated 13 June 2008
- Added tables for the associated surrogates/internal standards to the methods 8270C SIM, 8270C and 8260B.
- Revised marginal exceedence language.

Revision 4, dated 28 January 2008
- Integration for TestAmerica and STL operations.
- In Section 1.2, the source document versions were updated.
- The variance and clarification documents included in sections 13 and 14 have been specified as examples.

8.0 Attachments

8.1 Attachment 1:Flagging Protocols for LIMS

8.2 Attachment 2:Associated Surrogates and Internal Standards for 8270C SIM

8.3 Attachment 3:Associated Surrogates and Internal Standards for 8270C

8.4 Attachment 4:Associated Surrogates and Internal Standards for 8260B

8.5 Attachment 5 – Target Analyte Lists for DoD Version 4.1 Projects

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS

The following table summarizes DoD, AFCEE, and DOE requirements that are generally applicable to all areas of the laboratory.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
Detection Limits Limits of Detection (LOD) Method Detection Limit (MDL) Detection Limit (DL)	Establish MDLs for all matrix-method-analyte combinations. Determine MDLs according to 40 CFR 136B or an acceptable alternative method. If a method employs a cleanup step for only some samples, then determine an MDL for the method with and without the cleanup. Run an **MDLV** check sample at approximately **2X** the current MDL immediately following the MDL study. • The analyte must be reliably detected **and** identified using method-specific criteria. • All requirements for analyte detection must be met, e.g., ion abundance, second column confirmation, or pattern recognition. • If the method does not include confirmation of an analyte, then the MDLV check sample must produce a signal that is 3X the instrument's noise. 10X rule: The ratio of the ***mean recovered concentration*** (not the spiked concentration) must be between 1 - 5 for a water matrix, and between 1 - 10 for other matrices. An annual MDL study is **not** required as long as MDLV check samples are successfully measured **quarterly**. The MDL must be ≤ $^1/_3$ RL. **Version 4.1 DL** Establish DLs for all matrix-method-analyte combinations. Determine DLs according to 40 CFR 136B or an acceptable alternative method.	Establish MDLs for all matrix-method-analyte combinations at initial method setup. Determine MDLs according to 40 CFR 136B. Run an **MDLV** at approximately **2X** the current MDL immediately following an MDL study. • All analytes must be detected and identified by method-specific criteria. • Or the verification check must produce a response that is at least 3X the instrument noise level and greater than the response in the blanks associated with the MDL study. If MDLV fails, then run the MDLV at a higher level and set the MDL at that higher level or re-do the MDL study. 10 X rule: The ratio of the spike amount to the MDL must be <10, otherwise repeat the study using a smaller spike. Demonstrations of MDLs must be provided to AFCEE in their prescribed format before analyzing any of their samples. An annual MDL study is **not** required if MDLVs are run **quarterly**. MDLs must be ≤ ½ the RLs in AFCEE QAPP Section 7 tables.	The LOD is equivalent to MDL. The protocols for determining LODs shall be documented. Establish MDLs for all matrix-method-analyte combinations. Perform an initial MDL study according to 40 CFR 136B. Include **all** sample processing steps of the analytical method in the determination of LOD. The MDL must be updated or verified at a specified frequency (i.e., verified annually). A new MDL study is needed only if there is a significant change that affects the method's sensitivity. The MDL must be ≤ $^1/_3$ RL.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	Version 4.1: LOD Run an **LOD** (equivalent to the MDLV spike) check sample at approximately **2-3X** the current DL (1-4 times for multi-analyte methods) immediately following the DL study. The LOD must be analyzed on each instrument for a given method. This spike concentration establishes the LOD. • The analyte must be reliably detected **and** identified using method-specific criteria. • The LOD must be analyzed on each instrument. • All requirements for analyte detection must be met, e.g., ion abundance, second column confirmation, or pattern recognition. • If the method does not include confirmation of an analyte, then the MDLV check sample must produce a signal that is 3X the instrument's noise. • If the LOD fails, then the LOD must be performed at a higher concentration and pass two consecutive LOD verifications at the higher concentration. The LOD will be set at the higher concentration. An annual DL study is **not** required as long as LOD check samples are successfully measured **quarterly**.		
Reporting Limits (RLs) Limit of Quantitation (LOQ)	RLs are defined by the client and related to project-specific action levels. Must be at least 3 times the MDL. A small number of marginal exceptions may be allowed. Exceptions are allowed for multi-component	Use AFCEE QAPP Section 7 RLs. RL in Section 7 must be at least 2X MDL. Must verify the RL by including a standard ≤ RL as the lowest point on the calibration curve. All results shall be reported ≥ MDL, with an F-flag for results between the MDL and RL to	RL must be > 3X MDL. LOQ is equivalent to PQL. For analyte calibration curves of more than two points, the lowest point above the LOD (MDL) determines the LOQ (RL). The LOQ (RL) must be ≥ 3X LOD (MDL).

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	analytes such as PCBs, toxaphene, GRO, and DRO: The LOQ is not based solely on the LOD (MDL) of the various components, but on the concentration of the mixture at which the pattern becomes recognizable to the analyst. The RL must lie within the calibration range, at or above the LOQ. If a client requires an RL below the lowest standard of the calibration curve and below the LOQ, then method modification is required. For methods that require only one standard (i.e., lower limit of curve is the origin), the RL shall be no lower than the low-level check standard, designed to verify the integrity of the curve at the lower limits. The lowest standard of the calibration establishes the LOQ, which must be ≥ 3X LOD. The quantitation range is defined by the low and high calibration standards. Results reported to the client that are below the RL must be flagged appropriately: ND ≤ MDL: U-flag, report LOD adjusted by any dilution factors used. MDL - RL: J-flag, reported as estimation **Version 4.1 – LOQ** The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. • The LOQ must be set within the calibration range. • The LOQ must be verified quarterly. • The procedure for determining the LOQ must empirically demonstrate precision and accuracy. • If the method is modified, precision and bias at the new LOQ must be demonstrated and reported.	indicate an estimated value. Do not report results < MDL.	Verify the LOQ (RL) annually for each quality system matrix, method, and analyte. Measure a spiked sample with analytes at 1-2 X the LOQ (RL). This is <u>not</u> needed if the LOD is verified annually.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
Initial Calibration Criteria	Include <u>all</u> reported target analytes and surrogates in the initial calibration. Use a minimum of 5 contiguous calibration points for organics and 3 points for inorganics. The RL must lie within the calibration range, at or above the LOQ. Do not exclude any initial calibration points without technical justification. Acceptance Criteria: • Linear regression correlation coefficient ≥0.995 • Maximum %RSD of 20% for each analyte Deviations permitted only with DoD approval. When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted and reanalyzed to bring them within the calibration curve. When sample concentrations fall outside the calibration range, high or low, the resulting data shall be qualified as having estimated values and shall be flagged.	All analytes must be present in initial and continuing calibrations. The minimum number of calibration points is specified in Section 7 of the QAPP. All points used for the calibration must be contiguous. If more than the minimum number of points is used, all standards must be included. The only exception is a standard at either end of the calibration can dropped provided that the minimum number of standards is included. The lowest standard must be at or below the RL for each analyte in the method. Calibrations must meet method-specific criteria in Section 7 of QAPP. All calibration criteria shall satisfy SW-846 requirements at a minimum. For GC and GC/MS, when using RFs, use the average RF from the initial calibration. All results reported in analyzed samples must be within the calibration range.	Samples with concentrations that exceed the calibration range must be diluted to fall within the range, if there is enough sample and prep/analysis will fall within holding time criteria. Sample shall not be initially diluted unless all required project limits can be achieved. Otherwise samples shall be analyzed at a lower dilution. All exceptions must be discussed with the contract technical point of contact. The LOQ = PQL (practical quantitation limit).
Second-Source Standard (Initial Calibration Verification or ICV)	Measure a second-source standard after the ICAL to verify the ICAL (initial calibration verification or ICV). The concentration of the second-source standard shall be at or near the middle of the calibration range. Acceptance criteria must be at least as stringent as those established for the CCV. CCV must pass acceptance criteria before analyzing samples. Second source standards are available for all DoD methods/analytes.	The initial calibration must be verified by a second-source standard (initial calibration verification or ICV). A second-source standard is a standard purchased from a vendor different from that supplying the material used in the initial calibration. It can be used for the CCVs and/or LCSs. Only when there is not a separate vendor, then the use of different lot numbers from the same vendor will be acceptable.	*The DOE QSAS does not specify anything in addition to the NELAC standard.*
Continuing Calibration Verification (CCV)	Verify the initial calibration prior to sample analysis with an acceptable CCV in each analytical batch. As long as the CCV is	Perform CCV daily before sample analysis (unless an initial calibration and second-source standard verification is performed immediately	*The DOE QSAS does not specify anything in addition to the NELAC standard.*

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	acceptable, a new ICAL is not necessary. When reference methods specify a CCV frequency in terms of a per sample basis, this shall be field samples only. However, QC samples must be run with their associated batches. The grouping of QC samples from more than one batch is not acceptable. If the method does specify a frequency, each batch of 20 samples shall be bracketed by CCVs. CCV ≤ middle of the calibration range The CCV may be from the same source as the calibration standards. Compare CCVs to the ICAL. Criteria may include % difference from the mean response factor for the ICAL, minimum response factor checks, and confirmation that the RT is within an acceptable window. The % difference must be <15% of the ICAL for organic methods, and 10% for inorganic methods, unless otherwise specified by the reference method. If CCV fails: • Need project-specific permission to report data • Use Q flag and explain in the case narrative. If initial corrective action attempts fail and the CCV results are still outside established acceptance criteria, and the laboratory chooses to demonstrate the success of routine corrective action through the use of 2 consecutive CCVs, then the concentrations of the 2 CCVs must be at 2 different levels within the original calibration curve. At least one of the CCV standards shall fall below the middle of the initial calibration range. **NOTE:** For dual column methods the CCV **MUST** meet the aforementioned criteria for both columns.	before sample analysis). CCV frequency is specified in Section 7 of the QAPP. Check instrument calibration using ALL target analytes identified in project-specific requirements. If no project-specific analytes are specified, use default analytes listed in Section 7 of QAPP. CCV ≤ middle of the calibration curve. Do not use the continuing calibration verification to update the RFs from the initial calibration. CCVs cannot be used as the LCS, except for methods that do not involve sample preparation (e.g., VOAs).	

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
Control Limits	In order of priority: • Use project-specific limits, if specified. • Use control limits in the QSM (Appendix DoD-D v3 and G v4.1), unless other limits have been approved. • If no DoD limits, then use lab in-house limits. The laboratory limits must be at least as good as the DoD limits where they exist, or as good or better than published limits for similar methods. The lower LCS control limit must be ≥ 10%. Requirements for in-house limits: • Must be statistically derived. Limits at ±3 times the standard deviation of the recovery data are recommended. • Must meet limits specified in the method, if available • Must be updated annually and re-established after major changes in the analytical process (e.g., new instrument, new analyst). • Must be based on at least 30 data points generated under the same analytical process. • Must not exclude failed LCS recovery data and statistical outliers unless there is a documented and scientifically valid reason. • Using control charts to detect trends and prevent out-of-control conditions is recommended. **NOTE:** The TAL Denver QA Office maintains separate LIMS QC programs with verified MDLs and with the appropriate control limits, i.e., project-specific limits, QSM limits, or in-house control limits.	In order of priority: • Use project-specific control limits based on PQOs, if available. • Use the AFCEE LCS control limits in Section 7 of the QAPP. • Use lab in-house limits. • The lab may use in-house limits for the Section 7 methods, but they must be within the AFCEE limits. A number of sporadic marginal exceedances of the LCS control limits are allowed (see "Marginal Exceedances" below). TAL Denver maintains separate LIMS QC programs with verified MDLs and with either AFCEE QAPP limits or in-house control limits, as appropriate (e.g., the A4 QC Program). Other QC Programs may be created as needed. **NOTE:** The TAL Denver QA Office maintains separate LIMS QC programs with verified MDLs and with the appropriate control limits, i.e., project-specific limits, AFCEE QAPP limits or in-house control limits. The LIMS flags out-of-control data based on the limits entered for the QC program. Analysts are responsible for investigating out-of-control data and implementing corrective actions.	In order of priority: • Use acceptance criteria published in the mandated method. • Where there are no published criteria, determine initial criteria and document the method used to establish the limits or utilize client-specified assessment criteria. NELAC marginal exceedances may be used, but for organic analytes only. **NOTE:** The TAL Denver QA Office maintains separate LIMS QC programs with verified MDLs and with the appropriate control limits, i.e., method-specific limits, project-specific limits, or in-house control limits. The LIMS flags out-of-control data based on the limits entered for the QC Program. Analysts are responsible for investigating out-of-control data and implementing corrective actions.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	The LIMS flags out-of-control data based on the limits entered for the QC program. Analysts are responsible for investigating out-of-control data and implementing corrective actions.		
Marginal Exceedances (ME)	MEs apply to methods with more than 10 analytes. • Set the ME limit at 4 standard deviations around the mean. • MEs must be random. • Allowed 5 MEs if > 90 analytes; 4 MEs if 71–90 analytes; 3 MEs if 51–70 analytes; 2 MEs if 31–50 analytes; 1 ME if 11-30 analytes. The same analyte exceeding the LCS control limits 2 out of 3 consecutive LCSs indicates non-random behavior. DoD does not allow any project-specific analytes of concern to exceed its LCS control limits, even marginally. The use of marginal exceedences should be addressed with the client prior to the beginning of a project.	MEs apply to methods with more than 10 analytes. The number of MEs must not exceed 5% of the total number of analytes in the LCS. • Set the ME limit at 4 standard deviations around the mean. • Must be random. • Allowed 5 MEs if > 90 analytes; 4 MEs if 71–90 analytes; 3 MEs if 51–70 analytes; 2 MEs if 31–50 analytes; 1 ME if 11-30 analytes. The same analyte exceeding the LCS control limits 2 out of 3 consecutive LCSs indicates non-random behavior. Monitor the pattern of MEs to ensure random behavior. The use of marginal exceedences should be addressed with the client prior to the beginning of a project.	As required by NELAC, MEs apply to methods with more than 10 analytes. • Set the ME limit at between 3 and 4 standard deviations around the mean. • Must be random. • Allowed 5 MEs if > 90 analytes; 4 MEs if 71–90 analytes; 3 MEs if 51–70 analytes; 2 MEs if 31–50 analytes; 1 ME if 11-30 analytes. The same analyte exceeding the LCS control limits 2 out of 3 consecutive LCSs indicates non-random behavior. Monitor the pattern of MEs to ensure random behavior. The use of marginal exceedences should be addressed with the client prior to the beginning of a project.
Control Charts	The quality manager must ensure continuous improvement through the use of control charts and other method performance indicators. Use of control charts is strongly recommended. Control limits must be continually monitored for shifts in mean recovery, changes in standard deviation, and development of trends.	Use of control charts to track all analytes spiked into the LCS is recommended. Use the control charts to establish in-house control limits. Update charts as needed, e.g., when there is a significant change in analytical system. At a minimum, update charts annually. Review charts each time a point is added so that corrective action is timely.	QA officer must periodically review control charts at a specified frequency for out-of-control conditions and initiate appropriate corrective actions. QA Officer shall be empowered to stop unsatisfactory work, or prevent the reporting of results generated from an out-of-control measurement system.
Batch	When there is no separate preparation method used, e.g., volatiles in water, the batch is defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed	An AFCEE analytical batch (AAB) is a group of samples (not exceeding 20 environmental samples plus associated laboratory QC samples) that are similar in composition (matrix) that are extracted or digested at the same time and with the same lot of reagents and analyzed	A DOE batch is a prep batch or part of a prep batch that is analyzed with the same instrumentation, method sequence, and lots of reagents, and with the manipulations common to each samples within the same time period or in continuous sequential time periods. It is

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	the analysis of 20 environmental samples.	together as a group. The MS and MSD are treated as environmental samples.	preferable that the analytical batch be the prep batch. If equipment restrictions limit the number of samples in any particular step, the samples in the batch are processed continuously and consecutively until the entire batch is competed.
Laboratory Control Sample (LCS)	Include <u>all</u> target analytes in the LCS. For multi-component analytes, the LCS should be spiked with the same constituents as the calibration standard. Analyze 1 LCS per preparation batch. Use evaluation and acceptance criteria in QSM Appendixes DoD-B and DoD-D (version 3) and DoD-F and G (version 4.1). No project-specific analyte of concern can exceed its LCS control limits, even marginally. DoD-defined poor performers, listed below, should not be used to control the batch: 8270 Water 4-nitrophenol benzoic acid phenol phenol-d5 or d6 (surrogate) 8270 Solid 3,3'-dichlorobenzidine benzoic acid 4-chloroaniline 8151 Solid dinoseb 8330 Solid Tetryl	An LCS has failed if ... • A project-specific analyte of concern falls outside the limits. • An LCS has more than the allowed number of MEs. • A single analyte exceeds its ME limits. Once an LCS has failed, corrective action is required.	The LCS is a quality system matrix (see NELAC), known to be free of analytes of interest, spiked with known and verified concentrations of analytes. For soil samples for metals where a digestion is required to get the analyte in solution, use a solid certified reference material (CRM). If a CRM with all requested analytes is not available, use a CRM with a representative amount of analytes of concern. When the suggested solid CRM is not available for the LCS, a matrix spike and LCS pair may be used. Composition of the soil LCS shall be documented in the case narrative. Compare results to acceptance criteria in the reference method, if available, or to lab in-house limits. An LCS failure indicates that the analytical system is "out of control."
LCS Corrective Action	Samples analyzed along with an LCS that is determined to be "out of control" shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.	All exceedances of the LCS control limits, marginal or otherwise, for any analyte, are subject to corrective action: • Attempt to identify the error and find a solution. • Document all findings and corrective action.	Any affected samples associated with an out-of-control LCS shall be reprocessed for reanalysis or the results reported with appropriate data qualifying codes.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
		• Re-prep and reanalyze all AFCEE samples in the affected batch for the failed analytes on a system that is in control or rerun batch with new LCS. • If analyte fails a second time or not enough sample to reanalyze, then all results in the batch for that analyte must be flagged (Q flag). • Document in case narrative.	
Matrix Spike and Duplicate (MS and MSD)	DoD clients should provide a minimum of 3 MS/MSD samples for a new or unknown matrix. In this case, compare the % mean recoveries and standard deviations for each analyte recovered in the new matrix to the DoD LCS means and control limits generated for clean matrices. These should be at least as good as those published in Appendix DoD-D (v3) and G (v4.1). Analyze one MS and MSD using the same environmental matrix collected for the specific DoD project in each prep batch of DoD samples. If there is insufficient sample material, it must be noted in the case narrative. All target analytes must be spiked in the project-specific MS and MSD. The RPD must be calculated as a comparison of measured concentrations. Results for MS and MSD must be evaluated against DoD acceptance criteria for the LCS. Corrective actions for matrix spike failures can vary from project to project and depend on project-specific data quality objectives (DQOs). • Organic cleanup procedures should be considered, i.e., sulfur removal for 8081A and 8082, sulfuric acid washing for 8082, Florisil column cleanup for 8081A, and GPC for biota samples.	Only AFCEE samples shall be used for spiking. Spike each analyte at ≤ midpoint of calibration curve. Use AFCEE Table 7 acceptance limits. If MS or MSD recoveries fail, evaluate to determine whether due to matrix effect or analytical error. Qualify analytes in all related samples and flag per AFCEE QAPP Sections 7 and 8.	In the absence of specific regulatory method criteria for the calculation of the RPD, follow the requirements of SW-846 for the calculation of the RPD.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	• Dilution of the sample can reduce the level of interferences, but will result in higher reporting limits and so should be compared to DQOs. • Re-preparation and reanalysis to confirm matrix interference is not beneficial if the laboratory has objective evidence of matrix interferences. This should be discussed in advance. If reanalysis is required when documented evidence of interference is present, this should be specified in the project documents and communicated to the analysts accordingly.		
Surrogates	Compare surrogate spike recoveries to project-specific limits specified by the client or the QSM limits, if project-specific limits are not available. If DoD limits are not available, use in-house limits.	Add surrogates to all samples, controls, and blanks per the reference Method. No limits are specified in the AFCEE QAPP. When outside of limits, troubleshoot, resolve problems, re-prepare, and reanalyze samples on a system that is in control. Otherwise qualify sample results with a Q flag.	*The DOE QSAS does not specify anything in addition to the NELAC standard.*
Internal Standards	Add internal standards to samples, controls, and blanks per reference Method. No limits are specified in the QSM. Reanalyze any samples associated with out-of-control results.	Add internal standards to samples, controls, and blanks per reference Method. No limits are specified in the AFCEE QAPP. When outside of limits, troubleshoot, resolve problems, re-prepare and reanalyze affected samples on system that is in control. Otherwise qualify results with a Q flag.	*The DOE QSAS does not specify anything in addition to the NELAC standard.*
Method Blanks (MB)	Analyze one blank per batch. Acceptance criteria for MB: • < ½ of the reporting limit for each analyte, • or < $^1/_{10}$ of the regulatory limit, • or < $^1/_{10}$ of the sample results, whichever is greater. For common lab contaminants (defined in TAL Denver's variance requests), the concentration cannot exceed the reporting limit.	Include one blank in every AFCEE batch. The blank should be < MDL, otherwise investigate the source of contamination. MB must be < ½ RL, or < RL for common lab contaminants. If MB fails acceptance criteria: • Take action to correct, minimize, or eliminate the problem. • Reprocess samples if NOT ND and if MB	The method blank shall consist of a quality system matrix (see NELAC) that is similar to the associated samples and is known to be free of the analytes of interest. Analyze at least one blank per batch. Acceptance Criteria: • Any target analyte in MB < RL, or • Target analyte is < $^1/_{10}$ the amount measured in any sample, or

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	If the blank is contaminated: • Investigate and take measures to minimize or eliminate contamination. • Any sample associated with a failed blank must be reprocessed, unless the sample is ND. • If insufficient volume remains to reprocess the sample, then apply a B-flag to all results for the specific analyte(s) in all samples associated with the failed blank. • Document any corrective action. **NOTE:** TAL Denver's LIMS automatically applies a B flag to results when the method blank exceeds the MDL, rather than ½ RL (i.e., we are over-flagging). However, analysts take corrective action only if the blank result exceeds ½ RL.	> $^{1}/_{10}$ of any sample in the batch, or there is evidence the contamination affects the samples. • If no sample remains to reprocess, then apply a B-flag to all affected results. • If analyte is detected in the MB, but not in samples, no flag needed. • Also evaluate MB for any TICs found. • When MB fails, but corrective action is ineffective or not performed, then apply B-flag to data. • Document blank contamination in the case narrative. **NOTE:** TAL Denver's LIMS automatically applies a B flag to results when the method blank exceeds the MDL, rather than ½ RL (i.e., we are over-flagging). However, analysts take corrective action only if the blank result exceeds ½ RL.	• Blank contamination does not affect sample results as per the test method requirements or specific project DQOs. If the blank is contaminated: • Investigate the source of the contamination and take measures to minimize or eliminate the problem. • Reprocess all affected samples or appropriately qualify data if associated MB fails acceptance criteria. • In all cases, the corrective action must be documented in the case narrative. • Contact the client shall to discuss implementation of corrective action.
Sample Duplicates	"Matrix duplicates" are replicate aliquots of the same sample taken through the entire analytical procedure. The matrix duplicate provides a usable measure of precision only when target analytes are found in the selected sample. The frequency of duplicate analysis is determined by the project or as specified in the mandated method. If the known concentration of concern is >5 times the LOQ, a matrix duplicate may be analyzed in place of the MSD. A matrix spike is still required. Duplicate analyses should be performed at a minimum frequency of one per preparatory batch per matrix type. Results are used to assess analytical precision for a given matrix and are expressed as RPD, which must be calculated as a comparison of	One duplicate is required for every 20 field samples. Use AFCEE QAPP Section 7 MS/MSD RPD limits.	Duplicates are identical splits of individual samples that are analyzed by the lab to test for method reproducibility in a given matrix. DOE does not require sample duplicates for most environmental chemistry tests.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	measured concentrations. Acceptance criteria are either specified in the mandated method or established by the lab.		
Holding Time (HT)	Holding time is the time elapsed from the time of sampling to the time of extraction or analysis, as appropriate. Include both date and time of analysis as part of the laboratory report. If the time of the sample collection is not provided, assume the most conservative, i.e., the earliest, time of day. **NOTE:** TAL Denver routinely reports date and time of analysis. However, TAL Denver normally calculates holding time to the same units as expressed in the reference methods. For example, if the holding time is expressed in hours, then TAL Denver measures to the hour; and if expressed in days, then to the day. TAL Denver practice is consistent with the reference methods and the standard practice of EPA data validators. For most projects, this is not considered a variance, TAL Denver ensures that this practice is brought to the attention of project personnel.	Sample prep and analysis must be completed within the method-required HT. HT begins at the time of sample collection. HTs are determined on the basis of days, hours, and minutes. Preparation HT is calculated from the time of sample collection to the time of completion of the sample preparation process as described in the method, prior to any necessary extract cleanup and/or volume reduction. If no prep, analysis HT is calculated from sample collection to completion of all analytical runs, including dilutions, second column confirmations, and any required re-analyses. When sample prep is required prior to analysis, the analysis HT is calculated from the time of prep to completion of all analytical runs. If HT is exceeded, the result must be flagged and identified in the case narrative. Samples not preserved or analyzed in accordance with preservation and HT requirements, must be re-sampled and reanalyzed at the discretion of and at no additional cost to AFCEE.	"Holding time" is the duration between the date and time of sample collection and the dates and times of sample preparation (extraction/distillation) and/or analysis.
Client Approval Needed for Some Preferred Methods	Any modifications to existing method requirements require project-specific approval by DoD personnel. Soxhlet extraction (SW3540) is preferred by the USACE. Written approval is needed to use sonication (SW3550B) instead. The laboratory performs SW3540 only for Method 8141A/B, as required. Although solid-phase extraction (SPE SW3535) is preferred for explosives in water samples, the	Specific screening methods must be selected from AFCEE QAPP Table 6-1 unless a variance is requested. Screening QC are prescribed in Table 6.2-1. All screening data must be flagged with an "S" qualifier. Sample preparation methods are listed in Table 7.1-1. Method SW3535A, SPE extraction of aqueous samples, is recommended, especially for	Laboratory-selected or modified methods must be approved by the DOE client prior to use.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	EPA source method (SW8330) does not mention it. For that reason, written approval is required to use SPE instead of the older salting-out procedure. The USACE requires TAL Denver to advise its clients that the HPLC method for polynuclear aromatic hydrocarbons (SW8310) has a very weak confirmational ability, and that GC/MS selective ion monitoring (SIM) is preferred. TAL Denver performs only the methanol extraction option for 5035/8015B-GRO and 5035/8021B, and is not set up to perform the low-level option. For any DoD QSM projects involving volatile organics by GC, this must be made clear to clients at the project initiation phase and the project RL requirements carefully checked against the routine laboratory MDLs and RLs.	explosives. The EPA source method, SW8330, does not mention it. For that reason, written approval is required to use SPE instead of the older salting-out procedure. Determinative methods are listed in Table 7.2-1. Use listed analytes as defaults when project does not specify analytes. The following AFCEE Tests are not performed at TAL Denver: • Low-level 8021B in soil, we don't have an ARCON autosampler in the GC Department. We perform medium level GRO (methanolic dilution) and achieve the AFCEE RL. • Organophosphorus pesticides 8141A in water, we cannot meet control limits & use different surrogates. • Alkaline digestion 3060A for Cr^{+6} in soil, we only perform the DI leach procedure. • Dioxin/furan.	
Aliquotting and Subsampling	Procedures shall address laboratory practices for the handling, subsampling, and documenting of extraneous materials (e.g., rocks, twigs, vegetation, etc.) present in samples. Subsampling must be consistent with ASTM D6323 and EPA/660/R-03/027. Targeting weights is not allowed. Water samples must be aliquotted by volume using Class A glassware. Do not decant or filter samples, unless required by the method or approved for the project. Sample containers for extractable organics must be solvent rinsed, and the rinse included in the extraction.	*The AFCEE QAPP does not address subsampling in the laboratory.*	The sub-sample shall be representative of the sample being sub-sampled. Use documented procedures and appropriate techniques to obtain representative subsamples. Document client-mandated deviations, additions, or exclusions from the documented sampling procedure in the final report.
Data Qualifier Flags	Flagging should be used only as a last resort. Data should be flagged only after corrective	Flagging criteria are applied when acceptance criteria were not met and corrective action was	*The DOE QSAS does not specify anything in addition to the NELAC standard.*

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	action has been performed. U - Undetected at the LOD – the associated data is the LOD, adjusted by any dilution factor used in the analysis. J - Estimated: The analyte was positively identified; quantitation is an estimation; e.g., matrix interference, below standard, outside the calibration range, between MDL and RL. B - Blank contamination: The analyte was detected above ½ the RL in an associated blank. N - Non-target analyte: The analyte is a tentatively identified compound (TIC) using mass spec. Q - One or more QC criteria (e.g., LCS recovery, surrogate recovery) failed. Qualifiers may be combined when appropriate. See flagging protocols for the LIMS in Attachment 1.	not successful or corrective action was not performed. U - Undetected; analyte was analyzed for but not detected. J - Estimated: analyte was identified, but it is estimated due to discrepancies in meeting certain analyte-specific QC. UJ -Not detected, but result is estimated due to discrepancies in meeting certain analyte-specific QC. B - Blank contamination: analyte found in MB at > ½ RL, and in sample. M - The concentration is estimated due to matrix effect. Q - One or more QC limits failed. F - Analyte was positively identified but concentration is estimated because result is between MDL and RL. T - Tentatively identified compound (mass spec only). See QAPP Table 8.2.2.4-2 for use. Multiple flags can be used. See flagging protocols for the LIMS in Attachment 1.	
Manual Integration	When manual integrations are performed, raw data records shall include the following: • A complete audit trail for those manipulations. • Raw data output showing the results of the manual integration (i.e., chromatograms or manually integrated peaks) • Notation of rationale. • Date • Signature/initials of person performing manual operation (electronic signature is	Use manual integrations judiciously to correct any incorrect integration by the automated instrumentation and not as a routine procedure for the purpose of meeting calibration or method QC acceptance criteria. Perform manual integration only as a corrective action measure. When manual integrations are done, raw data records shall include the following: • A complete audit trail for those manipulations. • Results of both the automated and manual integrations ("before" and "after"	*The DOE QSAS does not specify anything in addition to the NELAC standard.*

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	acceptable). **Identify all manual integrations in the case narrative:** The laboratory provides this information as an attachment to the case narrative.	chromatograms). • Notation of cause. • Justification for the manual integration. • Date • Signature/initials of person performing manual operation. • Primary and secondary reviews may be performed electronically provided all documentation and data integrity are maintained. **Identify all manual integrations in the case narrative:** The laboratory provides this information as an attachment to the case narrative.	
Reporting & Records	Report all results on a dry-weight basis. Project managers must consider those methods that include a drying step in order to avoid double correction for moisture content (LIMS set-up) e.g. Explosives and TOC. Report the date and time of analysis for all tests. Report MDLs and RLs that are adjusted for dilutions with sample results. Report all dilutions. Both date and time of analysis are considered to be essential information and shall be included as part of the analytical record. Identify all samples for which manual integration was necessary. **Version 4.1 Reporting Results:** Guidance: Detection limit (DL) = 2, Limit of Detection (LOD) = 4, Limit of Quantitation (LOQ) = 15, sample is undiluted Sample #1: Analytical result: Not detected; reported result : 4U	Use AFCEE forms in Section 8.8 or equivalent. Report results per AFCEE analytical batch. Report MDLs and results to one decimal place more than RL unless measurement significant figures dictate otherwise. Report soil results on a dry-weight basis. Identify samples that needed manual integration and document manual integrations in the report. All peaks with a response >10% of the nearest IS are potential TICs and must be examined. Concentrations are estimated assuming a response factor of 1 between the TIC and nearest IS. Analyst must review 100% of the data generated. Senior analyst or supervisor must then review 100% of the data. Lab QA must perform a 100% review on 10% of completed data packages. Queries will be performed on a monthly basis to identify AFCEE lots, 10% of the identified lots will be randomly selected for review. Review will be	NELAC requirements apply. Do not include opinions and interpretations unless OK with client, and then only in the case narrative. For any corrections made to quality records, include a justification for the change. Documentation for changes made to data (either hardcopy or electronic) shall include the identification of the person who authorized the change. Obtain written approval from all affected clients prior to disposing of any records associated with DOE analytical data. Opinions and interpretations shall not be included in the report without notification of the client. Opinions and interpretations shall be in the case narrative only.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	Sample #2: Analytical result: 2; reported result : 4J Sample #3: Analytical result: 10; reported result : 10J Sample #4: Analytical result: 15; reported result : 15 Lab QA must perform a 100% review on 10% of completed data packages. Queries will be performed on a monthly basis to identify AFCEE lots, 10% of the identified lots will be randomly selected for review. Review will be conducted utilizing the QA "Data Audit Checklists" available on G:\QA\Edit\FORMS\QA.	conducted utilizing the QA"Data Audit Checklists" available on G:\QA\Edit\FORMS\QA.	
Proficiency Testing	The lab must participate in a PT program as defined by NELAC.	The lab must participate in USEPA Water Supply and Water Pollution Studies or equivalent for state certifications. Satisfactory performance also demonstrates proficiency in methods used for AFCEE samples. The lab must document corrective actions for unacceptable PT results to demonstrate resolution of problems. The lab must also analyze project-specific PT samples submitted on a quarterly basis by AFCEE contractors.	DOE requires successful participation in nationally recognized PE program for one year prior to and throughout a contract. The lab must participate in MAPEP, NELAC for CWA (WP) and SDWA-Water (WS). DMR QA for NPDES is recommended. Suppliers must be approved by PEOP/PEPA administered by NELAC. The lab must make results available to DOE. Reporting an unacceptable value results in a probationary period until the next reporting period for that analyte. "Not reported" is NOT an acceptable result. The lab must correct failures within the next PE program performance cycle. If there are 2 failures in a row, the lab cannot receive samples for the failed method until an acceptable PE score has been achieved. Quick Responses are acceptable. The lab must document causes of PE failure and develop corrective actions within 21 calendar days from receipt of the results.
Corrective Actions	Managers, including the quality manager, are responsible for acting upon corrective action report reviews and are ultimately accountable for	If necessary, complete corrective actions immediately. If acceptance criteria were not met and a corrective action was not successful	Prior to implementation of corrective actions where client data are affected, notify the client of the proposed corrective action.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS

The following table summarizes DoD, AFCEE, and DOE requirements that are generally applicable to all areas of the laboratory.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
Detection Limits Limits of Detection (LOD) Method Detection Limit (MDL) Detection Limit (DL)	Establish MDLs for all matrix-method-analyte combinations. Determine MDLs according to 40 CFR 136B or an acceptable alternative method. If a method employs a cleanup step for only some samples, then determine an MDL for the method with and without the cleanup. Run an **MDLV** check sample at approximately **2X** the current MDL immediately following the MDL study. • The analyte must be reliably detected **and** identified using method-specific criteria. • All requirements for analyte detection must be met, e.g., ion abundance, second column confirmation, or pattern recognition. • If the method does not include confirmation of an analyte, then the MDLV check sample must produce a signal that is 3X the instrument's noise. 10X rule: The ratio of the ***mean recovered concentration*** (not the spiked concentration) must be between 1 - 5 for a water matrix, and between 1 - 10 for other matrices. An annual MDL study is **not** required as long as MDLV check samples are successfully measured **quarterly**. The MDL must be ≤ $^1/_3$ RL. **Version 4.1 DL** Establish DLs for all matrix-method-analyte combinations. Determine DLs according to 40 CFR 136B or an acceptable alternative method.	Establish MDLs for all matrix-method-analyte combinations at initial method setup. Determine MDLs according to 40 CFR 136B. Run an **MDLV** at approximately **2X** the current MDL immediately following an MDL study. • All analytes must be detected and identified by method-specific criteria. • Or the verification check must produce a response that is at least 3X the instrument noise level and greater than the response in the blanks associated with the MDL study. If MDLV fails, then run the MDLV at a higher level and set the MDL at that higher level or re-do the MDL study. 10 X rule: The ratio of the spike amount to the MDL must be <10, otherwise repeat the study using a smaller spike. Demonstrations of MDLs must be provided to AFCEE in their prescribed format before analyzing any of their samples. An annual MDL study is **not** required if MDLVs are run **quarterly**. MDLs must be ≤ ½ the RLs in AFCEE QAPP Section 7 tables.	The LOD is equivalent to MDL. The protocols for determining LODs shall be documented. Establish MDLs for all matrix-method-analyte combinations. Perform an initial MDL study according to 40 CFR 136B. Include **all** sample processing steps of the analytical method in the determination of LOD. The MDL must be updated or verified at a specified frequency (i.e., verified annually). A new MDL study is needed only if there is a significant change that affects the method's sensitivity. The MDL must be ≤ $^1/_3$ RL.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	Version 4.1: LOD Run an **LOD** (equivalent to the MDLV spike) check sample at approximately **2-3X** the current DL (1-4 times for multi-analyte methods) immediately following the DL study. The LOD must be analyzed on each instrument for a given method. This spike concentration establishes the LOD. • The analyte must be reliably detected **and** identified using method-specific criteria. • The LOD must be analyzed on each instrument. • All requirements for analyte detection must be met, e.g., ion abundance, second column confirmation, or pattern recognition. • If the method does not include confirmation of an analyte, then the MDLV check sample must produce a signal that is 3X the instrument's noise. • If the LOD fails, then the LOD must be performed at a higher concentration and pass two consecutive LOD verifications at the higher concentration. The LOD will be set at the higher concentration. An annual DL study is **not** required as long as LOD check samples are successfully measured **quarterly**.		
Reporting Limits (RLs) Limit of Quantitation (LOQ)	RLs are defined by the client and related to project-specific action levels. Must be at least 3 times the MDL. A small number of marginal exceptions may be allowed. Exceptions are allowed for multi-component	Use AFCEE QAPP Section 7 RLs. RL in Section 7 must be at least 2X MDL. Must verify the RL by including a standard ≤ RL as the lowest point on the calibration curve. All results shall be reported ≥ MDL, with an F-flag for results between the MDL and RL to	RL must be > 3X MDL. LOQ is equivalent to PQL. For analyte calibration curves of more than two points, the lowest point above the LOD (MDL) determines the LOQ (RL). The LOQ (RL) must be ≥ 3X LOD (MDL).

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	analytes such as PCBs, toxaphene, GRO, and DRO: The LOQ is not based solely on the LOD (MDL) of the various components, but on the concentration of the mixture at which the pattern becomes recognizable to the analyst. The RL must lie within the calibration range, at or above the LOQ. If a client requires an RL below the lowest standard of the calibration curve and below the LOQ, then method modification is required. For methods that require only one standard (i.e., lower limit of curve is the origin), the RL shall be no lower than the low-level check standard, designed to verify the integrity of the curve at the lower limits. The lowest standard of the calibration establishes the LOQ, which must be ≥ 3X LOD. The quantitation range is defined by the low and high calibration standards. Results reported to the client that are below the RL must be flagged appropriately: ND ≤ MDL: U-flag, report LOD adjusted by any dilution factors used. MDL - RL: J-flag, reported as estimation **Version 4.1 – LOQ** The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. • The LOQ must be set within the calibration range. • The LOQ must be verified quarterly. • The procedure for determining the LOQ must empirically demonstrate precision and accuracy. • If the method is modified, precision and bias at the new LOQ must be demonstrated and reported.	indicate an estimated value. Do not report results < MDL.	Verify the LOQ (RL) annually for each quality system matrix, method, and analyte. Measure a spiked sample with analytes at 1-2 X the LOQ (RL). This is <u>not</u> needed if the LOD is verified annually.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
Initial Calibration Criteria	Include <u>all</u> reported target analytes and surrogates in the initial calibration. Use a minimum of 5 contiguous calibration points for organics and 3 points for inorganics. The RL must lie within the calibration range, at or above the LOQ. Do not exclude any initial calibration points without technical justification. Acceptance Criteria: • Linear regression correlation coefficient ≥0.995 • Maximum %RSD of 20% for each analyte Deviations permitted only with DoD approval. When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted and reanalyzed to bring them within the calibration curve. When sample concentrations fall outside the calibration range, high or low, the resulting data shall be qualified as having estimated values and shall be flagged.	All analytes must be present in initial and continuing calibrations. The minimum number of calibration points is specified in Section 7 of the QAPP. All points used for the calibration must be contiguous. If more than the minimum number of points is used, all standards must be included. The only exception is a standard at either end of the calibration can dropped provided that the minimum number of standards is included. The lowest standard must be at or below the RL for each analyte in the method. Calibrations must meet method-specific criteria in Section 7 of QAPP. All calibration criteria shall satisfy SW-846 requirements at a minimum. For GC and GC/MS, when using RFs, use the average RF from the initial calibration. All results reported in analyzed samples must be within the calibration range.	Samples with concentrations that exceed the calibration range must be diluted to fall within the range, if there is enough sample and prep/analysis will fall within holding time criteria. Sample shall not be initially diluted unless all required project limits can be achieved. Otherwise samples shall be analyzed at a lower dilution. All exceptions must be discussed with the contract technical point of contact. The LOQ = PQL (practical quantitation limit).
Second-Source Standard (Initial Calibration Verification or ICV)	Measure a second-source standard after the ICAL to verify the ICAL (initial calibration verification or ICV). The concentration of the second-source standard shall be at or near the middle of the calibration range. Acceptance criteria must be at least as stringent as those established for the CCV. CCV must pass acceptance criteria before analyzing samples. Second source standards are available for all DoD methods/analytes.	The initial calibration must be verified by a second-source standard (initial calibration verification or ICV). A second-source standard is a standard purchased from a vendor different from that supplying the material used in the initial calibration. It can be used for the CCVs and/or LCSs. Only when there is not a separate vendor, then the use of different lot numbers from the same vendor will be acceptable.	*The DOE QSAS does not specify anything in addition to the NELAC standard.*
Continuing Calibration Verification (CCV)	Verify the initial calibration prior to sample analysis with an acceptable CCV in each analytical batch. As long as the CCV is	Perform CCV daily before sample analysis (unless an initial calibration and second-source standard verification is performed immediately	*The DOE QSAS does not specify anything in addition to the NELAC standard.*

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	acceptable, a new ICAL is not necessary. When reference methods specify a CCV frequency in terms of a per sample basis, this shall be field samples only. However, QC samples must be run with their associated batches. The grouping of QC samples from more than one batch is not acceptable. If the method does specify a frequency, each batch of 20 samples shall be bracketed by CCVs. CCV ≤ middle of the calibration range The CCV may be from the same source as the calibration standards. Compare CCVs to the ICAL. Criteria may include % difference from the mean response factor for the ICAL, minimum response factor checks, and confirmation that the RT is within an acceptable window. The % difference must be <15% of the ICAL for organic methods, and 10% for inorganic methods, unless otherwise specified by the reference method. If CCV fails: • Need project-specific permission to report data • Use Q flag and explain in the case narrative. If initial corrective action attempts fail and the CCV results are still outside established acceptance criteria, and the laboratory chooses to demonstrate the success of routine corrective action through the use of 2 consecutive CCVs, then the concentrations of the 2 CCVs must be at 2 different levels within the original calibration curve. At least one of the CCV standards shall fall below the middle of the initial calibration range. **NOTE:** For dual column methods the CCV **MUST** meet the aforementioned criteria for both columns.	before sample analysis). CCV frequency is specified in Section 7 of the QAPP. Check instrument calibration using ALL target analytes identified in project-specific requirements. If no project-specific analytes are specified, use default analytes listed in Section 7 of QAPP. CCV ≤ middle of the calibration curve. Do not use the continuing calibration verification to update the RFs from the initial calibration. CCVs cannot be used as the LCS, except for methods that do not involve sample preparation (e.g., VOAs).	

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
Control Limits	In order of priority: • Use project-specific limits, if specified. • Use control limits in the QSM (Appendix DoD-D v3 and G v4.1), unless other limits have been approved. • If no DoD limits, then use lab in-house limits. The laboratory limits must be at least as good as the DoD limits where they exist, or as good or better than published limits for similar methods. The lower LCS control limit must be ≥ 10%. Requirements for in-house limits: • Must be statistically derived. Limits at ±3 times the standard deviation of the recovery data are recommended. • Must meet limits specified in the method, if available • Must be updated annually and re-established after major changes in the analytical process (e.g., new instrument, new analyst). • Must be based on at least 30 data points generated under the same analytical process. • Must not exclude failed LCS recovery data and statistical outliers unless there is a documented and scientifically valid reason. • Using control charts to detect trends and prevent out-of-control conditions is recommended. **NOTE:** The TAL Denver QA Office maintains separate LIMS QC programs with verified MDLs and with the appropriate control limits, i.e., project-specific limits, QSM limits, or in-house control limits.	In order of priority: • Use project-specific control limits based on PQOs, if available. • Use the AFCEE LCS control limits in Section 7 of the QAPP. • Use lab in-house limits. • The lab may use in-house limits for the Section 7 methods, but they must be within the AFCEE limits. A number of sporadic marginal exceedances of the LCS control limits are allowed (see "Marginal Exceedances" below). TAL Denver maintains separate LIMS QC programs with verified MDLs and with either AFCEE QAPP limits or in-house control limits, as appropriate (e.g., the A4 QC Program). Other QC Programs may be created as needed. **NOTE:** The TAL Denver QA Office maintains separate LIMS QC programs with verified MDLs and with the appropriate control limits, i.e., project-specific limits, AFCEE QAPP limits or in-house control limits. The LIMS flags out-of-control data based on the limits entered for the QC program. Analysts are responsible for investigating out-of-control data and implementing corrective actions.	In order of priority: • Use acceptance criteria published in the mandated method. • Where there are no published criteria, determine initial criteria and document the method used to establish the limits or utilize client-specified assessment criteria. NELAC marginal exceedances may be used, but for organic analytes only. **NOTE:** The TAL Denver QA Office maintains separate LIMS QC programs with verified MDLs and with the appropriate control limits, i.e., method-specific limits, project-specific limits, or in-house control limits. The LIMS flags out-of-control data based on the limits entered for the QC Program. Analysts are responsible for investigating out-of-control data and implementing corrective actions.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	The LIMS flags out-of-control data based on the limits entered for the QC program. Analysts are responsible for investigating out-of-control data and implementing corrective actions.		
Marginal Exceedances (ME)	MEs apply to methods with more than 10 analytes. • Set the ME limit at 4 standard deviations around the mean. • MEs must be random. • Allowed 5 MEs if > 90 analytes; 4 MEs if 71–90 analytes; 3 MEs if 51–70 analytes; 2 MEs if 31–50 analytes; 1 ME if 11-30 analytes. The same analyte exceeding the LCS control limits 2 out of 3 consecutive LCSs indicates non-random behavior. DoD does not allow any project-specific analytes of concern to exceed its LCS control limits, even marginally. The use of marginal exceedences should be addressed with the client prior to the beginning of a project.	MEs apply to methods with more than 10 analytes. The number of MEs must not exceed 5% of the total number of analytes in the LCS. • Set the ME limit at 4 standard deviations around the mean. • Must be random. • Allowed 5 MEs if > 90 analytes; 4 MEs if 71–90 analytes; 3 MEs if 51–70 analytes; 2 MEs if 31–50 analytes; 1 ME if 11-30 analytes. The same analyte exceeding the LCS control limits 2 out of 3 consecutive LCSs indicates non-random behavior. Monitor the pattern of MEs to ensure random behavior. The use of marginal exceedences should be addressed with the client prior to the beginning of a project.	As required by NELAC, MEs apply to methods with more than 10 analytes. • Set the ME limit at between 3 and 4 standard deviations around the mean. • Must be random. • Allowed 5 MEs if > 90 analytes; 4 MEs if 71–90 analytes; 3 MEs if 51–70 analytes; 2 MEs if 31–50 analytes; 1 ME if 11-30 analytes. The same analyte exceeding the LCS control limits 2 out of 3 consecutive LCSs indicates non-random behavior. Monitor the pattern of MEs to ensure random behavior. The use of marginal exceedences should be addressed with the client prior to the beginning of a project.
Control Charts	The quality manager must ensure continuous improvement through the use of control charts and other method performance indicators. Use of control charts is strongly recommended. Control limits must be continually monitored for shifts in mean recovery, changes in standard deviation, and development of trends.	Use of control charts to track all analytes spiked into the LCS is recommended. Use the control charts to establish in-house control limits. Update charts as needed, e.g., when there is a significant change in analytical system. At a minimum, update charts annually. Review charts each time a point is added so that corrective action is timely.	QA officer must periodically review control charts at a specified frequency for out-of-control conditions and initiate appropriate corrective actions. QA Officer shall be empowered to stop unsatisfactory work, or prevent the reporting of results generated from an out-of-control measurement system.
Batch	When there is no separate preparation method used, e.g., volatiles in water, the batch is defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed	An AFCEE analytical batch (AAB) is a group of samples (not exceeding 20 environmental samples plus associated laboratory QC samples) that are similar in composition (matrix) that are extracted or digested at the same time and with the same lot of reagents and analyzed	A DOE batch is a prep batch or part of a prep batch that is analyzed with the same instrumentation, method sequence, and lots of reagents, and with the manipulations common to each samples within the same time period or in continuous sequential time periods. It is

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	the analysis of 20 environmental samples.	together as a group. The MS and MSD are treated as environmental samples.	preferable that the analytical batch be the prep batch. If equipment restrictions limit the number of samples in any particular step, the samples in the batch are processed continuously and consecutively until the entire batch is competed.
Laboratory Control Sample (LCS)	Include <u>all</u> target analytes in the LCS. For multi-component analytes, the LCS should be spiked with the same constituents as the calibration standard. Analyze 1 LCS per preparation batch. Use evaluation and acceptance criteria in QSM Appendixes DoD-B and DoD-D (version 3) and DoD-F and G (version 4.1). No project-specific analyte of concern can exceed its LCS control limits, even marginally. DoD-defined poor performers, listed below, should not be used to control the batch: 8270 Water 4-nitrophenol benzoic acid phenol phenol-d5 or d6 (surrogate) 8270 Solid 3,3'-dichlorobenzidine benzoic acid 4-chloroaniline 8151 Solid dinoseb 8330 Solid Tetryl	An LCS has failed if ... • A project-specific analyte of concern falls outside the limits. • An LCS has more than the allowed number of MEs. • A single analyte exceeds its ME limits. Once an LCS has failed, corrective action is required.	The LCS is a quality system matrix (see NELAC), known to be free of analytes of interest, spiked with known and verified concentrations of analytes. For soil samples for metals where a digestion is required to get the analyte in solution, use a solid certified reference material (CRM). If a CRM with all requested analytes is not available, use a CRM with a representative amount of analytes of concern. When the suggested solid CRM is not available for the LCS, a matrix spike and LCS pair may be used. Composition of the soil LCS shall be documented in the case narrative. Compare results to acceptance criteria in the reference method, if available, or to lab in-house limits. An LCS failure indicates that the analytical system is "out of control."
LCS Corrective Action	Samples analyzed along with an LCS that is determined to be "out of control" shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.	All exceedances of the LCS control limits, marginal or otherwise, for any analyte, are subject to corrective action: • Attempt to identify the error and find a solution. • Document all findings and corrective action.	Any affected samples associated with an out-of-control LCS shall be reprocessed for reanalysis or the results reported with appropriate data qualifying codes.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
		• Re-prep and reanalyze all AFCEE samples in the affected batch for the failed analytes on a system that is in control or rerun batch with new LCS. • If analyte fails a second time or not enough sample to reanalyze, then all results in the batch for that analyte must be flagged (Q flag). • Document in case narrative.	
Matrix Spike and Duplicate (MS and MSD)	DoD clients should provide a minimum of 3 MS/MSD samples for a new or unknown matrix. In this case, compare the % mean recoveries and standard deviations for each analyte recovered in the new matrix to the DoD LCS means and control limits generated for clean matrices. These should be at least as good as those published in Appendix DoD-D (v3) and G (v4.1). Analyze one MS and MSD using the same environmental matrix collected for the specific DoD project in each prep batch of DoD samples. If there is insufficient sample material, it must be noted in the case narrative. All target analytes must be spiked in the project-specific MS and MSD. The RPD must be calculated as a comparison of measured concentrations. Results for MS and MSD must be evaluated against DoD acceptance criteria for the LCS. Corrective actions for matrix spike failures can vary from project to project and depend on project-specific data quality objectives (DQOs). • Organic cleanup procedures should be considered, i.e., sulfur removal for 8081A and 8082, sulfuric acid washing for 8082, Florisil column cleanup for 8081A, and GPC for biota samples.	Only AFCEE samples shall be used for spiking. Spike each analyte at ≤ midpoint of calibration curve. Use AFCEE Table 7 acceptance limits. If MS or MSD recoveries fail, evaluate to determine whether due to matrix effect or analytical error. Qualify analytes in all related samples and flag per AFCEE QAPP Sections 7 and 8.	In the absence of specific regulatory method criteria for the calculation of the RPD, follow the requirements of SW-846 for the calculation of the RPD.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	• Dilution of the sample can reduce the level of interferences, but will result in higher reporting limits and so should be compared to DQOs. • Re-preparation and reanalysis to confirm matrix interference is not beneficial if the laboratory has objective evidence of matrix interferences. This should be discussed in advance. If reanalysis is required when documented evidence of interference is present, this should be specified in the project documents and communicated to the analysts accordingly.		
Surrogates	Compare surrogate spike recoveries to project-specific limits specified by the client or the QSM limits, if project-specific limits are not available. If DoD limits are not available, use in-house limits.	Add surrogates to all samples, controls, and blanks per the reference Method. No limits are specified in the AFCEE QAPP. When outside of limits, troubleshoot, resolve problems, re-prepare, and reanalyze samples on a system that is in control. Otherwise qualify sample results with a Q flag.	*The DOE QSAS does not specify anything in addition to the NELAC standard.*
Internal Standards	Add internal standards to samples, controls, and blanks per reference Method. No limits are specified in the QSM. Reanalyze any samples associated with out-of-control results.	Add internal standards to samples, controls, and blanks per reference Method. No limits are specified in the AFCEE QAPP. When outside of limits, troubleshoot, resolve problems, re-prepare and reanalyze affected samples on system that is in control. Otherwise qualify results with a Q flag.	*The DOE QSAS does not specify anything in addition to the NELAC standard.*
Method Blanks (MB)	Analyze one blank per batch. Acceptance criteria for MB: • < ½ of the reporting limit for each analyte, • or < $^1/_{10}$ of the regulatory limit, • or < $^1/_{10}$ of the sample results, whichever is greater. For common lab contaminants (defined in TAL Denver's variance requests), the concentration cannot exceed the reporting limit.	Include one blank in every AFCEE batch. The blank should be < MDL, otherwise investigate the source of contamination. MB must be < ½ RL, or < RL for common lab contaminants. If MB fails acceptance criteria: • Take action to correct, minimize, or eliminate the problem. • Reprocess samples if NOT ND and if MB	The method blank shall consist of a quality system matrix (see NELAC) that is similar to the associated samples and is known to be free of the analytes of interest. Analyze at least one blank per batch. Acceptance Criteria: • Any target analyte in MB < RL, or • Target analyte is < $^1/_{10}$ the amount measured in any sample, or

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	If the blank is contaminated: • Investigate and take measures to minimize or eliminate contamination. • Any sample associated with a failed blank must be reprocessed, unless the sample is ND. • If insufficient volume remains to reprocess the sample, then apply a B-flag to all results for the specific analyte(s) in all samples associated with the failed blank. • Document any corrective action. **NOTE:** TAL Denver's LIMS automatically applies a B flag to results when the method blank exceeds the MDL, rather than ½ RL (i.e., we are over-flagging). However, analysts take corrective action only if the blank result exceeds ½ RL.	> $^{1}/_{10}$ of any sample in the batch, or there is evidence the contamination affects the samples. • If no sample remains to reprocess, then apply a B-flag to all affected results. • If analyte is detected in the MB, but not in samples, no flag needed. • Also evaluate MB for any TICs found. • When MB fails, but corrective action is ineffective or not performed, then apply B-flag to data. • Document blank contamination in the case narrative. **NOTE:** TAL Denver's LIMS automatically applies a B flag to results when the method blank exceeds the MDL, rather than ½ RL (i.e., we are over-flagging). However, analysts take corrective action only if the blank result exceeds ½ RL.	• Blank contamination does not affect sample results as per the test method requirements or specific project DQOs. If the blank is contaminated: • Investigate the source of the contamination and take measures to minimize or eliminate the problem. • Reprocess all affected samples or appropriately qualify data if associated MB fails acceptance criteria. • In all cases, the corrective action must be documented in the case narrative. • Contact the client shall to discuss implementation of corrective action.
Sample Duplicates	"Matrix duplicates" are replicate aliquots of the same sample taken through the entire analytical procedure. The matrix duplicate provides a usable measure of precision only when target analytes are found in the selected sample. The frequency of duplicate analysis is determined by the project or as specified in the mandated method. If the known concentration of concern is >5 times the LOQ, a matrix duplicate may be analyzed in place of the MSD. A matrix spike is still required. Duplicate analyses should be performed at a minimum frequency of one per preparatory batch per matrix type. Results are used to assess analytical precision for a given matrix and are expressed as RPD, which must be calculated as a comparison of	One duplicate is required for every 20 field samples. Use AFCEE QAPP Section 7 MS/MSD RPD limits.	Duplicates are identical splits of individual samples that are analyzed by the lab to test for method reproducibility in a given matrix. DOE does not require sample duplicates for most environmental chemistry tests.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	measured concentrations. Acceptance criteria are either specified in the mandated method or established by the lab.		
Holding Time (HT)	Holding time is the time elapsed from the time of sampling to the time of extraction or analysis, as appropriate. Include both date and time of analysis as part of the laboratory report. If the time of the sample collection is not provided, assume the most conservative, i.e., the earliest, time of day. **NOTE:** TAL Denver routinely reports date and time of analysis. However, TAL Denver normally calculates holding time to the same units as expressed in the reference methods. For example, if the holding time is expressed in hours, then TAL Denver measures to the hour; and if expressed in days, then to the day. TAL Denver practice is consistent with the reference methods and the standard practice of EPA data validators. For most projects, this is not considered a variance, TAL Denver ensures that this practice is brought to the attention of project personnel.	Sample prep and analysis must be completed within the method-required HT. HT begins at the time of sample collection. HTs are determined on the basis of days, hours, and minutes. Preparation HT is calculated from the time of sample collection to the time of completion of the sample preparation process as described in the method, prior to any necessary extract cleanup and/or volume reduction. If no prep, analysis HT is calculated from sample collection to completion of all analytical runs, including dilutions, second column confirmations, and any required re-analyses. When sample prep is required prior to analysis, the analysis HT is calculated from the time of prep to completion of all analytical runs. If HT is exceeded, the result must be flagged and identified in the case narrative. Samples not preserved or analyzed in accordance with preservation and HT requirements, must be re-sampled and reanalyzed at the discretion of and at no additional cost to AFCEE.	"Holding time" is the duration between the date and time of sample collection and the dates and times of sample preparation (extraction/distillation) and/or analysis.
Client Approval Needed for Some Preferred Methods	Any modifications to existing method requirements require project-specific approval by DoD personnel. Soxhlet extraction (SW3540) is preferred by the USACE. Written approval is needed to use sonication (SW3550B) instead. The laboratory performs SW3540 only for Method 8141A/B, as required. Although solid-phase extraction (SPE SW3535) is preferred for explosives in water samples, the	Specific screening methods must be selected from AFCEE QAPP Table 6-1 unless a variance is requested. Screening QC are prescribed in Table 6.2-1. All screening data must be flagged with an "S" qualifier. Sample preparation methods are listed in Table 7.1-1. Method SW3535A, SPE extraction of aqueous samples, is recommended, especially for	Laboratory-selected or modified methods must be approved by the DOE client prior to use.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	EPA source method (SW8330) does not mention it. For that reason, written approval is required to use SPE instead of the older salting-out procedure. The USACE requires TAL Denver to advise its clients that the HPLC method for polynuclear aromatic hydrocarbons (SW8310) has a very weak confirmational ability, and that GC/MS selective ion monitoring (SIM) is preferred. TAL Denver performs only the methanol extraction option for 5035/8015B-GRO and 5035/8021B, and is not set up to perform the low-level option. For any DoD QSM projects involving volatile organics by GC, this must be made clear to clients at the project initiation phase and the project RL requirements carefully checked against the routine laboratory MDLs and RLs.	explosives. The EPA source method, SW8330, does not mention it. For that reason, written approval is required to use SPE instead of the older salting-out procedure. Determinative methods are listed in Table 7.2-1. Use listed analytes as defaults when project does not specify analytes. The following AFCEE Tests are not performed at TAL Denver: • Low-level 8021B in soil, we don't have an ARCON autosampler in the GC Department. We perform medium level GRO (methanolic dilution) and achieve the AFCEE RL. • Organophosphorus pesticides 8141A in water, we cannot meet control limits & use different surrogates. • Alkaline digestion 3060A for Cr^{+6} in soil, we only perform the DI leach procedure. • Dioxin/furan.	
Aliquotting and Subsampling	Procedures shall address laboratory practices for the handling, subsampling, and documenting of extraneous materials (e.g., rocks, twigs, vegetation, etc.) present in samples. Subsampling must be consistent with ASTM D6323 and EPA/660/R-03/027. Targeting weights is not allowed. Water samples must be aliquotted by volume using Class A glassware. Do not decant or filter samples, unless required by the method or approved for the project. Sample containers for extractable organics must be solvent rinsed, and the rinse included in the extraction.	*The AFCEE QAPP does not address subsampling in the laboratory.*	The sub-sample shall be representative of the sample being sub-sampled. Use documented procedures and appropriate techniques to obtain representative subsamples. Document client-mandated deviations, additions, or exclusions from the documented sampling procedure in the final report.
Data Qualifier Flags	Flagging should be used only as a last resort. Data should be flagged only after corrective	Flagging criteria are applied when acceptance criteria were not met and corrective action was	*The DOE QSAS does not specify anything in addition to the NELAC standard.*

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	action has been performed. U - Undetected at the LOD – the associated data is the LOD, adjusted by any dilution factor used in the analysis. J - Estimated: The analyte was positively identified; quantitation is an estimation; e.g., matrix interference, below standard, outside the calibration range, between MDL and RL. B - Blank contamination: The analyte was detected above ½ the RL in an associated blank. N - Non-target analyte: The analyte is a tentatively identified compound (TIC) using mass spec. Q - One or more QC criteria (e.g., LCS recovery, surrogate recovery) failed. Qualifiers may be combined when appropriate. See flagging protocols for the LIMS in Attachment 1.	not successful or corrective action was not performed. U - Undetected; analyte was analyzed for but not detected. J - Estimated: analyte was identified, but it is estimated due to discrepancies in meeting certain analyte-specific QC. UJ -Not detected, but result is estimated due to discrepancies in meeting certain analyte-specific QC. B - Blank contamination: analyte found in MB at > ½ RL, and in sample. M - The concentration is estimated due to matrix effect. Q - One or more QC limits failed. F - Analyte was positively identified but concentration is estimated because result is between MDL and RL. T - Tentatively identified compound (mass spec only). See QAPP Table 8.2.2.4-2 for use. Multiple flags can be used. See flagging protocols for the LIMS in Attachment 1.	
Manual Integration	When manual integrations are performed, raw data records shall include the following: • A complete audit trail for those manipulations. • Raw data output showing the results of the manual integration (i.e., chromatograms or manually integrated peaks) • Notation of rationale. • Date • Signature/initials of person performing manual operation (electronic signature is	Use manual integrations judiciously to correct any incorrect integration by the automated instrumentation and not as a routine procedure for the purpose of meeting calibration or method QC acceptance criteria. Perform manual integration only as a corrective action measure. When manual integrations are done, raw data records shall include the following: • A complete audit trail for those manipulations. • Results of both the automated and manual integrations ("before" and "after"	*The DOE QSAS does not specify anything in addition to the NELAC standard.*

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	acceptable). **Identify all manual integrations in the case narrative:** The laboratory provides this information as an attachment to the case narrative.	chromatograms). • Notation of cause. • Justification for the manual integration. • Date • Signature/initials of person performing manual operation. • Primary and secondary reviews may be performed electronically provided all documentation and data integrity are maintained. **Identify all manual integrations in the case narrative:** The laboratory provides this information as an attachment to the case narrative.	
Reporting & Records	Report all results on a dry-weight basis. Project managers must consider those methods that include a drying step in order to avoid double correction for moisture content (LIMS set-up) e.g. Explosives and TOC. Report the date and time of analysis for all tests. Report MDLs and RLs that are adjusted for dilutions with sample results. Report all dilutions. Both date and time of analysis are considered to be essential information and shall be included as part of the analytical record. Identify all samples for which manual integration was necessary. **Version 4.1 Reporting Results:** Guidance: Detection limit (DL) = 2, Limit of Detection (LOD) = 4, Limit of Quantitation (LOQ) = 15, sample is undiluted Sample #1: Analytical result: Not detected; reported result : 4U	Use AFCEE forms in Section 8.8 or equivalent. Report results per AFCEE analytical batch. Report MDLs and results to one decimal place more than RL unless measurement significant figures dictate otherwise. Report soil results on a dry-weight basis. Identify samples that needed manual integration and document manual integrations in the report. All peaks with a response >10% of the nearest IS are potential TICs and must be examined. Concentrations are estimated assuming a response factor of 1 between the TIC and nearest IS. Analyst must review 100% of the data generated. Senior analyst or supervisor must then review 100% of the data. Lab QA must perform a 100% review on 10% of completed data packages. Queries will be performed on a monthly basis to identify AFCEE lots, 10% of the identified lots will be randomly selected for review. Review will be	NELAC requirements apply. Do not include opinions and interpretations unless OK with client, and then only in the case narrative. For any corrections made to quality records, include a justification for the change. Documentation for changes made to data (either hardcopy or electronic) shall include the identification of the person who authorized the change. Obtain written approval from all affected clients prior to disposing of any records associated with DOE analytical data. Opinions and interpretations shall not be included in the report without notification of the client. Opinions and interpretations shall be in the case narrative only.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	Sample #2: Analytical result: 2; reported result : 4J Sample #3: Analytical result: 10; reported result : 10J Sample #4: Analytical result: 15; reported result : 15 Lab QA must perform a 100% review on 10% of completed data packages. Queries will be performed on a monthly basis to identify AFCEE lots, 10% of the identified lots will be randomly selected for review. Review will be conducted utilizing the QA "Data Audit Checklists" available on G:\QA\Edit\FORMS\QA.	conducted utilizing the QA"Data Audit Checklists" available on G:\QA\Edit\FORMS\QA.	
Proficiency Testing	The lab must participate in a PT program as defined by NELAC.	The lab must participate in USEPA Water Supply and Water Pollution Studies or equivalent for state certifications. Satisfactory performance also demonstrates proficiency in methods used for AFCEE samples. The lab must document corrective actions for unacceptable PT results to demonstrate resolution of problems. The lab must also analyze project-specific PT samples submitted on a quarterly basis by AFCEE contractors.	DOE requires successful participation in nationally recognized PE program for one year prior to and throughout a contract. The lab must participate in MAPEP, NELAC for CWA (WP) and SDWA-Water (WS). DMR QA for NPDES is recommended. Suppliers must be approved by PEOP/PEPA administered by NELAC. The lab must make results available to DOE. Reporting an unacceptable value results in a probationary period until the next reporting period for that analyte. "Not reported" is NOT an acceptable result. The lab must correct failures within the next PE program performance cycle. If there are 2 failures in a row, the lab cannot receive samples for the failed method until an acceptable PE score has been achieved. Quick Responses are acceptable. The lab must document causes of PE failure and develop corrective actions within 21 calendar days from receipt of the results.
Corrective Actions	Managers, including the quality manager, are responsible for acting upon corrective action report reviews and are ultimately accountable for	If necessary, complete corrective actions immediately. If acceptance criteria were not met and a corrective action was not successful	Prior to implementation of corrective actions where client data are affected, notify the client of the proposed corrective action.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	the follow-through, verification, and evaluation of these corrective actions.	or not performed, apply the appropriate flagging. Corrective actions can include re-analysis of the affected sample(s), re-sampling and analysis, or change in procedures, depending on the severity of the problem.	Track corrective actions to ensure completion. Perform tracking and trending of lessons learned to prevent the recurrence of nonconformance.
Measuring & Test Equipment	Check balance calibration daily or before use with 2 weights that bracket target weight(s). Balances shall be calibrated annually by a certified technician. Acceptance criteria for analytical balance checks are ±0.1% or ±0.5 mg, whichever is larger, unless method-specific guidance exists. For top-loading balances, see ASTM D 4753. Standard weights must be calibrated every 5 years and require a third-party certificate of acceptance. Monitor refrigerator/freezer temperatures daily. Refrigerators are controlled to 4 ± 2 °C, and freezers to -10 to -20 °C (≤-10°C v4.1). Check the calibration of liquid-in-glass thermometers when received and annually. Check the accuracy of electronic thermometers against a NIST-traceable thermometer quarterly at 2 temperatures that bracket the target temperatures (check at 1 temperature allowed in V4.1). Check the accuracy of mechanical pipettes at least monthly; the volume must be within 3% of the true volume. Check the accuracy of non-volumetric glassware/labware that is used for measuring initial sample and final extract/digestate volumes on a per lot basis at the time of purchase. The volume must be within 3% of the true value. Check the temperature accuracy of drying ovens before and after use and before use only for those used for moisture content. Temperatures	Thermometers used to monitor temperatures of cold storage areas must be calibrated using a NIST-traceable thermometer and correction factors must be applied to temperature readings based on the calibrations.	Balances must be calibrated before initial use and annually thereafter and labeled to that effect by an independent professional technician. Check balances daily or before use with standard weights that bracket the range of use. Document daily balance checks. Monitor and document temperatures daily "Daily" refers to calendar days, not working days. Temperature monitoring data loggers are acceptable. Monitor the conductivity or resistivity of the water from the purification system daily or before use and record the results in a logbook. The laboratory shall have an SOP for reagent and deionized water production that includes (at a minimum): preventive maintenance of water purification equipment, control criteria, and corrective action processes for out-of-spec water. Each person who uses a mechanical volumetric dispensing device for quantitative measurements must verify its accuracy daily prior to use. If the same device is used by more than one person, each person must verify its accuracy before using it.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	the follow-through, verification, and evaluation of these corrective actions.	or not performed, apply the appropriate flagging. Corrective actions can include re-analysis of the affected sample(s), re-sampling and analysis, or change in procedures, depending on the severity of the problem.	Track corrective actions to ensure completion. Perform tracking and trending of lessons learned to prevent the recurrence of nonconformance.
Measuring & Test Equipment	Check balance calibration daily or before use with 2 weights that bracket target weight(s). Balances shall be calibrated annually by a certified technician. Acceptance criteria for analytical balance checks are ±0.1% or ±0.5 mg, whichever is larger, unless method-specific guidance exists. For top-loading balances, see ASTM D 4753. Standard weights must be calibrated every 5 years and require a third-party certificate of acceptance. Monitor refrigerator/freezer temperatures daily. Refrigerators are controlled to 4 ± 2 °C, and freezers to -10 to -20 °C (≤-10°C v4.1). Check the calibration of liquid-in-glass thermometers when received and annually. Check the accuracy of electronic thermometers against a NIST-traceable thermometer quarterly at 2 temperatures that bracket the target temperatures (check at 1 temperature allowed in V4.1). Check the accuracy of mechanical pipettes at least monthly; the volume must be within 3% of the true volume. Check the accuracy of non-volumetric glassware/labware that is used for measuring initial sample and final extract/digestate volumes on a per lot basis at the time of purchase. The volume must be within 3% of the true value. Check the temperature accuracy of drying ovens before and after use and before use only for those used for moisture content. Temperatures	Thermometers used to monitor temperatures of cold storage areas must be calibrated using a NIST-traceable thermometer and correction factors must be applied to temperature readings based on the calibrations.	Balances must be calibrated before initial use and annually thereafter and labeled to that effect by an independent professional technician. Check balances daily or before use with standard weights that bracket the range of use. Document daily balance checks. Monitor and document temperatures daily "Daily" refers to calendar days, not working days. Temperature monitoring data loggers are acceptable. Monitor the conductivity or resistivity of the water from the purification system daily or before use and record the results in a logbook. The laboratory shall have an SOP for reagent and deionized water production that includes (at a minimum): preventive maintenance of water purification equipment, control criteria, and corrective action processes for out-of-spec water. Each person who uses a mechanical volumetric dispensing device for quantitative measurements must verify its accuracy daily prior to use. If the same device is used by more than one person, each person must verify its accuracy before using it.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS

The following table summarizes DoD, AFCEE, and DOE requirements that are generally applicable to all areas of the laboratory.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
Detection Limits Limits of Detection (LOD) Method Detection Limit (MDL) Detection Limit (DL)	Establish MDLs for all matrix-method-analyte combinations. Determine MDLs according to 40 CFR 136B or an acceptable alternative method. If a method employs a cleanup step for only some samples, then determine an MDL for the method with and without the cleanup. Run an **MDLV** check sample at approximately **2X** the current MDL immediately following the MDL study. • The analyte must be reliably detected **and** identified using method-specific criteria. • All requirements for analyte detection must be met, e.g., ion abundance, second column confirmation, or pattern recognition. • If the method does not include confirmation of an analyte, then the MDLV check sample must produce a signal that is 3X the instrument's noise. 10X rule: The ratio of the ***mean recovered concentration*** (not the spiked concentration) must be between 1 - 5 for a water matrix, and between 1 - 10 for other matrices. An annual MDL study is **not** required as long as MDLV check samples are successfully measured **quarterly**. The MDL must be ≤ $^1/_3$ RL. **Version 4.1 DL** Establish DLs for all matrix-method-analyte combinations. Determine DLs according to 40 CFR 136B or an acceptable alternative method.	Establish MDLs for all matrix-method-analyte combinations at initial method setup. Determine MDLs according to 40 CFR 136B. Run an **MDLV** at approximately **2X** the current MDL immediately following an MDL study. • All analytes must be detected and identified by method-specific criteria. • Or the verification check must produce a response that is at least 3X the instrument noise level and greater than the response in the blanks associated with the MDL study. If MDLV fails, then run the MDLV at a higher level and set the MDL at that higher level or re-do the MDL study. 10 X rule: The ratio of the spike amount to the MDL must be <10, otherwise repeat the study using a smaller spike. Demonstrations of MDLs must be provided to AFCEE in their prescribed format before analyzing any of their samples. An annual MDL study is **not** required if MDLVs are run **quarterly**. MDLs must be ≤ ½ the RLs in AFCEE QAPP Section 7 tables.	The LOD is equivalent to MDL. The protocols for determining LODs shall be documented. Establish MDLs for all matrix-method-analyte combinations. Perform an initial MDL study according to 40 CFR 136B. Include **all** sample processing steps of the analytical method in the determination of LOD. The MDL must be updated or verified at a specified frequency (i.e., verified annually). A new MDL study is needed only if there is a significant change that affects the method's sensitivity. The MDL must be ≤ $^1/_3$ RL.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	Version 4.1: LOD Run an **LOD** (equivalent to the MDLV spike) check sample at approximately **2-3X** the current DL (1-4 times for multi-analyte methods) immediately following the DL study. The LOD must be analyzed on each instrument for a given method. This spike concentration establishes the LOD. • The analyte must be reliably detected **and** identified using method-specific criteria. • The LOD must be analyzed on each instrument. • All requirements for analyte detection must be met, e.g., ion abundance, second column confirmation, or pattern recognition. • If the method does not include confirmation of an analyte, then the MDLV check sample must produce a signal that is 3X the instrument's noise. • If the LOD fails, then the LOD must be performed at a higher concentration and pass two consecutive LOD verifications at the higher concentration. The LOD will be set at the higher concentration. An annual DL study is **not** required as long as LOD check samples are successfully measured **quarterly**.		
Reporting Limits (RLs) Limit of Quantitation (LOQ)	RLs are defined by the client and related to project-specific action levels. Must be at least 3 times the MDL. A small number of marginal exceptions may be allowed. Exceptions are allowed for multi-component	Use AFCEE QAPP Section 7 RLs. RL in Section 7 must be at least 2X MDL. Must verify the RL by including a standard ≤ RL as the lowest point on the calibration curve. All results shall be reported ≥ MDL, with an F-flag for results between the MDL and RL to	RL must be > 3X MDL. LOQ is equivalent to PQL. For analyte calibration curves of more than two points, the lowest point above the LOD (MDL) determines the LOQ (RL). The LOQ (RL) must be ≥ 3X LOD (MDL).

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	analytes such as PCBs, toxaphene, GRO, and DRO: The LOQ is not based solely on the LOD (MDL) of the various components, but on the concentration of the mixture at which the pattern becomes recognizable to the analyst. The RL must lie within the calibration range, at or above the LOQ. If a client requires an RL below the lowest standard of the calibration curve and below the LOQ, then method modification is required. For methods that require only one standard (i.e., lower limit of curve is the origin), the RL shall be no lower than the low-level check standard, designed to verify the integrity of the curve at the lower limits. The lowest standard of the calibration establishes the LOQ, which must be ≥ 3X LOD. The quantitation range is defined by the low and high calibration standards. Results reported to the client that are below the RL must be flagged appropriately: ND ≤ MDL: U-flag, report LOD adjusted by any dilution factors used. MDL - RL: J-flag, reported as estimation **Version 4.1 – LOQ** The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. • The LOQ must be set within the calibration range. • The LOQ must be verified quarterly. • The procedure for determining the LOQ must empirically demonstrate precision and accuracy. • If the method is modified, precision and bias at the new LOQ must be demonstrated and reported.	indicate an estimated value. Do not report results < MDL.	Verify the LOQ (RL) annually for each quality system matrix, method, and analyte. Measure a spiked sample with analytes at 1-2 X the LOQ (RL). This is <u>not</u> needed if the LOD is verified annually.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
Initial Calibration Criteria	Include <u>all</u> reported target analytes and surrogates in the initial calibration. Use a minimum of 5 contiguous calibration points for organics and 3 points for inorganics. The RL must lie within the calibration range, at or above the LOQ. Do not exclude any initial calibration points without technical justification. Acceptance Criteria: • Linear regression correlation coefficient ≥0.995 • Maximum %RSD of 20% for each analyte Deviations permitted only with DoD approval. When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted and reanalyzed to bring them within the calibration curve. When sample concentrations fall outside the calibration range, high or low, the resulting data shall be qualified as having estimated values and shall be flagged.	All analytes must be present in initial and continuing calibrations. The minimum number of calibration points is specified in Section 7 of the QAPP. All points used for the calibration must be contiguous. If more than the minimum number of points is used, all standards must be included. The only exception is a standard at either end of the calibration can dropped provided that the minimum number of standards is included. The lowest standard must be at or below the RL for each analyte in the method. Calibrations must meet method-specific criteria in Section 7 of QAPP. All calibration criteria shall satisfy SW-846 requirements at a minimum. For GC and GC/MS, when using RFs, use the average RF from the initial calibration. All results reported in analyzed samples must be within the calibration range.	Samples with concentrations that exceed the calibration range must be diluted to fall within the range, if there is enough sample and prep/analysis will fall within holding time criteria. Sample shall not be initially diluted unless all required project limits can be achieved. Otherwise samples shall be analyzed at a lower dilution. All exceptions must be discussed with the contract technical point of contact. The LOQ = PQL (practical quantitation limit).
Second-Source Standard (Initial Calibration Verification or ICV)	Measure a second-source standard after the ICAL to verify the ICAL (initial calibration verification or ICV). The concentration of the second-source standard shall be at or near the middle of the calibration range. Acceptance criteria must be at least as stringent as those established for the CCV. CCV must pass acceptance criteria before analyzing samples. Second source standards are available for all DoD methods/analytes.	The initial calibration must be verified by a second-source standard (initial calibration verification or ICV). A second-source standard is a standard purchased from a vendor different from that supplying the material used in the initial calibration. It can be used for the CCVs and/or LCSs. Only when there is not a separate vendor, then the use of different lot numbers from the same vendor will be acceptable.	*The DOE QSAS does not specify anything in addition to the NELAC standard.*
Continuing Calibration Verification (CCV)	Verify the initial calibration prior to sample analysis with an acceptable CCV in each analytical batch. As long as the CCV is	Perform CCV daily before sample analysis (unless an initial calibration and second-source standard verification is performed immediately	*The DOE QSAS does not specify anything in addition to the NELAC standard.*

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	acceptable, a new ICAL is not necessary. When reference methods specify a CCV frequency in terms of a per sample basis, this shall be field samples only. However, QC samples must be run with their associated batches. The grouping of QC samples from more than one batch is not acceptable. If the method does specify a frequency, each batch of 20 samples shall be bracketed by CCVs. CCV ≤ middle of the calibration range The CCV may be from the same source as the calibration standards. Compare CCVs to the ICAL. Criteria may include % difference from the mean response factor for the ICAL, minimum response factor checks, and confirmation that the RT is within an acceptable window. The % difference must be <15% of the ICAL for organic methods, and 10% for inorganic methods, unless otherwise specified by the reference method. If CCV fails: • Need project-specific permission to report data • Use Q flag and explain in the case narrative. If initial corrective action attempts fail and the CCV results are still outside established acceptance criteria, and the laboratory chooses to demonstrate the success of routine corrective action through the use of 2 consecutive CCVs, then the concentrations of the 2 CCVs must be at 2 different levels within the original calibration curve. At least one of the CCV standards shall fall below the middle of the initial calibration range. **NOTE:** For dual column methods the CCV **MUST** meet the aforementioned criteria for both columns.	before sample analysis). CCV frequency is specified in Section 7 of the QAPP. Check instrument calibration using ALL target analytes identified in project-specific requirements. If no project-specific analytes are specified, use default analytes listed in Section 7 of QAPP. CCV ≤ middle of the calibration curve. Do not use the continuing calibration verification to update the RFs from the initial calibration. CCVs cannot be used as the LCS, except for methods that do not involve sample preparation (e.g., VOAs).	

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
Control Limits	In order of priority: • Use project-specific limits, if specified. • Use control limits in the QSM (Appendix DoD-D v3 and G v4.1), unless other limits have been approved. • If no DoD limits, then use lab in-house limits. The laboratory limits must be at least as good as the DoD limits where they exist, or as good or better than published limits for similar methods. The lower LCS control limit must be ≥ 10%. Requirements for in-house limits: • Must be statistically derived. Limits at ±3 times the standard deviation of the recovery data are recommended. • Must meet limits specified in the method, if available • Must be updated annually and re-established after major changes in the analytical process (e.g., new instrument, new analyst). • Must be based on at least 30 data points generated under the same analytical process. • Must not exclude failed LCS recovery data and statistical outliers unless there is a documented and scientifically valid reason. • Using control charts to detect trends and prevent out-of-control conditions is recommended. **NOTE:** The TAL Denver QA Office maintains separate LIMS QC programs with verified MDLs and with the appropriate control limits, i.e., project-specific limits, QSM limits, or in-house control limits.	In order of priority: • Use project-specific control limits based on PQOs, if available. • Use the AFCEE LCS control limits in Section 7 of the QAPP. • Use lab in-house limits. • The lab may use in-house limits for the Section 7 methods, but they must be within the AFCEE limits. A number of sporadic marginal exceedances of the LCS control limits are allowed (see "Marginal Exceedances" below). TAL Denver maintains separate LIMS QC programs with verified MDLs and with either AFCEE QAPP limits or in-house control limits, as appropriate (e.g., the A4 QC Program). Other QC Programs may be created as needed. **NOTE:** The TAL Denver QA Office maintains separate LIMS QC programs with verified MDLs and with the appropriate control limits, i.e., project-specific limits, AFCEE QAPP limits or in-house control limits. The LIMS flags out-of-control data based on the limits entered for the QC program. Analysts are responsible for investigating out-of-control data and implementing corrective actions.	In order of priority: • Use acceptance criteria published in the mandated method. • Where there are no published criteria, determine initial criteria and document the method used to establish the limits or utilize client-specified assessment criteria. NELAC marginal exceedances may be used, but for organic analytes only. **NOTE:** The TAL Denver QA Office maintains separate LIMS QC programs with verified MDLs and with the appropriate control limits, i.e., method-specific limits, project-specific limits, or in-house control limits. The LIMS flags out-of-control data based on the limits entered for the QC Program. Analysts are responsible for investigating out-of-control data and implementing corrective actions.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	The LIMS flags out-of-control data based on the limits entered for the QC program. Analysts are responsible for investigating out-of-control data and implementing corrective actions.		
Marginal Exceedances (ME)	MEs apply to methods with more than 10 analytes. • Set the ME limit at 4 standard deviations around the mean. • MEs must be random. • Allowed 5 MEs if > 90 analytes; 4 MEs if 71–90 analytes; 3 MEs if 51–70 analytes; 2 MEs if 31–50 analytes; 1 ME if 11-30 analytes. The same analyte exceeding the LCS control limits 2 out of 3 consecutive LCSs indicates non-random behavior. DoD does not allow any project-specific analytes of concern to exceed its LCS control limits, even marginally. The use of marginal exceedences should be addressed with the client prior to the beginning of a project.	MEs apply to methods with more than 10 analytes. The number of MEs must not exceed 5% of the total number of analytes in the LCS. • Set the ME limit at 4 standard deviations around the mean. • Must be random. • Allowed 5 MEs if > 90 analytes; 4 MEs if 71–90 analytes; 3 MEs if 51–70 analytes; 2 MEs if 31–50 analytes; 1 ME if 11-30 analytes. The same analyte exceeding the LCS control limits 2 out of 3 consecutive LCSs indicates non-random behavior. Monitor the pattern of MEs to ensure random behavior. The use of marginal exceedences should be addressed with the client prior to the beginning of a project.	As required by NELAC, MEs apply to methods with more than 10 analytes. • Set the ME limit at between 3 and 4 standard deviations around the mean. • Must be random. • Allowed 5 MEs if > 90 analytes; 4 MEs if 71–90 analytes; 3 MEs if 51–70 analytes; 2 MEs if 31–50 analytes; 1 ME if 11-30 analytes. The same analyte exceeding the LCS control limits 2 out of 3 consecutive LCSs indicates non-random behavior. Monitor the pattern of MEs to ensure random behavior. The use of marginal exceedences should be addressed with the client prior to the beginning of a project.
Control Charts	The quality manager must ensure continuous improvement through the use of control charts and other method performance indicators. Use of control charts is strongly recommended. Control limits must be continually monitored for shifts in mean recovery, changes in standard deviation, and development of trends.	Use of control charts to track all analytes spiked into the LCS is recommended. Use the control charts to establish in-house control limits. Update charts as needed, e.g., when there is a significant change in analytical system. At a minimum, update charts annually. Review charts each time a point is added so that corrective action is timely.	QA officer must periodically review control charts at a specified frequency for out-of-control conditions and initiate appropriate corrective actions. QA Officer shall be empowered to stop unsatisfactory work, or prevent the reporting of results generated from an out-of-control measurement system.
Batch	When there is no separate preparation method used, e.g., volatiles in water, the batch is defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed	An AFCEE analytical batch (AAB) is a group of samples (not exceeding 20 environmental samples plus associated laboratory QC samples) that are similar in composition (matrix) that are extracted or digested at the same time and with the same lot of reagents and analyzed	A DOE batch is a prep batch or part of a prep batch that is analyzed with the same instrumentation, method sequence, and lots of reagents, and with the manipulations common to each samples within the same time period or in continuous sequential time periods. It is

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	the analysis of 20 environmental samples.	together as a group. The MS and MSD are treated as environmental samples.	preferable that the analytical batch be the prep batch. If equipment restrictions limit the number of samples in any particular step, the samples in the batch are processed continuously and consecutively until the entire batch is competed.
Laboratory Control Sample (LCS)	Include <u>all</u> target analytes in the LCS. For multi-component analytes, the LCS should be spiked with the same constituents as the calibration standard. Analyze 1 LCS per preparation batch. Use evaluation and acceptance criteria in QSM Appendixes DoD-B and DoD-D (version 3) and DoD-F and G (version 4.1). No project-specific analyte of concern can exceed its LCS control limits, even marginally. DoD-defined poor performers, listed below, should not be used to control the batch: 8270 Water 4-nitrophenol benzoic acid phenol phenol-d5 or d6 (surrogate) 8270 Solid 3,3'-dichlorobenzidine benzoic acid 4-chloroaniline 8151 Solid dinoseb 8330 Solid Tetryl	An LCS has failed if ... • A project-specific analyte of concern falls outside the limits. • An LCS has more than the allowed number of MEs. • A single analyte exceeds its ME limits. Once an LCS has failed, corrective action is required.	The LCS is a quality system matrix (see NELAC), known to be free of analytes of interest, spiked with known and verified concentrations of analytes. For soil samples for metals where a digestion is required to get the analyte in solution, use a solid certified reference material (CRM). If a CRM with all requested analytes is not available, use a CRM with a representative amount of analytes of concern. When the suggested solid CRM is not available for the LCS, a matrix spike and LCS pair may be used. Composition of the soil LCS shall be documented in the case narrative. Compare results to acceptance criteria in the reference method, if available, or to lab in-house limits. An LCS failure indicates that the analytical system is "out of control."
LCS Corrective Action	Samples analyzed along with an LCS that is determined to be "out of control" shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.	All exceedances of the LCS control limits, marginal or otherwise, for any analyte, are subject to corrective action: • Attempt to identify the error and find a solution. • Document all findings and corrective action.	Any affected samples associated with an out-of-control LCS shall be reprocessed for reanalysis or the results reported with appropriate data qualifying codes.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
		• Re-prep and reanalyze all AFCEE samples in the affected batch for the failed analytes on a system that is in control or rerun batch with new LCS. • If analyte fails a second time or not enough sample to reanalyze, then all results in the batch for that analyte must be flagged (Q flag). • Document in case narrative.	
Matrix Spike and Duplicate (MS and MSD)	DoD clients should provide a minimum of 3 MS/MSD samples for a new or unknown matrix. In this case, compare the % mean recoveries and standard deviations for each analyte recovered in the new matrix to the DoD LCS means and control limits generated for clean matrices. These should be at least as good as those published in Appendix DoD-D (v3) and G (v4.1). Analyze one MS and MSD using the same environmental matrix collected for the specific DoD project in each prep batch of DoD samples. If there is insufficient sample material, it must be noted in the case narrative. All target analytes must be spiked in the project-specific MS and MSD. The RPD must be calculated as a comparison of measured concentrations. Results for MS and MSD must be evaluated against DoD acceptance criteria for the LCS. Corrective actions for matrix spike failures can vary from project to project and depend on project-specific data quality objectives (DQOs). • Organic cleanup procedures should be considered, i.e., sulfur removal for 8081A and 8082, sulfuric acid washing for 8082, Florisil column cleanup for 8081A, and GPC for biota samples.	Only AFCEE samples shall be used for spiking. Spike each analyte at ≤ midpoint of calibration curve. Use AFCEE Table 7 acceptance limits. If MS or MSD recoveries fail, evaluate to determine whether due to matrix effect or analytical error. Qualify analytes in all related samples and flag per AFCEE QAPP Sections 7 and 8.	In the absence of specific regulatory method criteria for the calculation of the RPD, follow the requirements of SW-846 for the calculation of the RPD.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	• Dilution of the sample can reduce the level of interferences, but will result in higher reporting limits and so should be compared to DQOs. • Re-preparation and reanalysis to confirm matrix interference is not beneficial if the laboratory has objective evidence of matrix interferences. This should be discussed in advance. If reanalysis is required when documented evidence of interference is present, this should be specified in the project documents and communicated to the analysts accordingly.		
Surrogates	Compare surrogate spike recoveries to project-specific limits specified by the client or the QSM limits, if project-specific limits are not available. If DoD limits are not available, use in-house limits.	Add surrogates to all samples, controls, and blanks per the reference Method. No limits are specified in the AFCEE QAPP. When outside of limits, troubleshoot, resolve problems, re-prepare, and reanalyze samples on a system that is in control. Otherwise qualify sample results with a Q flag.	*The DOE QSAS does not specify anything in addition to the NELAC standard.*
Internal Standards	Add internal standards to samples, controls, and blanks per reference Method. No limits are specified in the QSM. Reanalyze any samples associated with out-of-control results.	Add internal standards to samples, controls, and blanks per reference Method. No limits are specified in the AFCEE QAPP. When outside of limits, troubleshoot, resolve problems, re-prepare and reanalyze affected samples on system that is in control. Otherwise qualify results with a Q flag.	*The DOE QSAS does not specify anything in addition to the NELAC standard.*
Method Blanks (MB)	Analyze one blank per batch. Acceptance criteria for MB: • < ½ of the reporting limit for each analyte, • or < $^1/_{10}$ of the regulatory limit, • or < $^1/_{10}$ of the sample results, whichever is greater. For common lab contaminants (defined in TAL Denver's variance requests), the concentration cannot exceed the reporting limit.	Include one blank in every AFCEE batch. The blank should be < MDL, otherwise investigate the source of contamination. MB must be < ½ RL, or < RL for common lab contaminants. If MB fails acceptance criteria: • Take action to correct, minimize, or eliminate the problem. • Reprocess samples if NOT ND and if MB	The method blank shall consist of a quality system matrix (see NELAC) that is similar to the associated samples and is known to be free of the analytes of interest. Analyze at least one blank per batch. Acceptance Criteria: • Any target analyte in MB < RL, or • Target analyte is < $^1/_{10}$ the amount measured in any sample, or

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	If the blank is contaminated: • Investigate and take measures to minimize or eliminate contamination. • Any sample associated with a failed blank must be reprocessed, unless the sample is ND. • If insufficient volume remains to reprocess the sample, then apply a B-flag to all results for the specific analyte(s) in all samples associated with the failed blank. • Document any corrective action. **NOTE:** TAL Denver's LIMS automatically applies a B flag to results when the method blank exceeds the MDL, rather than ½ RL (i.e., we are over-flagging). However, analysts take corrective action only if the blank result exceeds ½ RL.	> $^{1}/_{10}$ of any sample in the batch, or there is evidence the contamination affects the samples. • If no sample remains to reprocess, then apply a B-flag to all affected results. • If analyte is detected in the MB, but not in samples, no flag needed. • Also evaluate MB for any TICs found. • When MB fails, but corrective action is ineffective or not performed, then apply B-flag to data. • Document blank contamination in the case narrative. **NOTE:** TAL Denver's LIMS automatically applies a B flag to results when the method blank exceeds the MDL, rather than ½ RL (i.e., we are over-flagging). However, analysts take corrective action only if the blank result exceeds ½ RL.	• Blank contamination does not affect sample results as per the test method requirements or specific project DQOs. If the blank is contaminated: • Investigate the source of the contamination and take measures to minimize or eliminate the problem. • Reprocess all affected samples or appropriately qualify data if associated MB fails acceptance criteria. • In all cases, the corrective action must be documented in the case narrative. • Contact the client shall to discuss implementation of corrective action.
Sample Duplicates	"Matrix duplicates" are replicate aliquots of the same sample taken through the entire analytical procedure. The matrix duplicate provides a usable measure of precision only when target analytes are found in the selected sample. The frequency of duplicate analysis is determined by the project or as specified in the mandated method. If the known concentration of concern is >5 times the LOQ, a matrix duplicate may be analyzed in place of the MSD. A matrix spike is still required. Duplicate analyses should be performed at a minimum frequency of one per preparatory batch per matrix type. Results are used to assess analytical precision for a given matrix and are expressed as RPD, which must be calculated as a comparison of	One duplicate is required for every 20 field samples. Use AFCEE QAPP Section 7 MS/MSD RPD limits.	Duplicates are identical splits of individual samples that are analyzed by the lab to test for method reproducibility in a given matrix. DOE does not require sample duplicates for most environmental chemistry tests.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	measured concentrations. Acceptance criteria are either specified in the mandated method or established by the lab.		
Holding Time (HT)	Holding time is the time elapsed from the time of sampling to the time of extraction or analysis, as appropriate. Include both date and time of analysis as part of the laboratory report. If the time of the sample collection is not provided, assume the most conservative, i.e., the earliest, time of day. **NOTE:** TAL Denver routinely reports date and time of analysis. However, TAL Denver normally calculates holding time to the same units as expressed in the reference methods. For example, if the holding time is expressed in hours, then TAL Denver measures to the hour; and if expressed in days, then to the day. TAL Denver practice is consistent with the reference methods and the standard practice of EPA data validators. For most projects, this is not considered a variance, TAL Denver ensures that this practice is brought to the attention of project personnel.	Sample prep and analysis must be completed within the method-required HT. HT begins at the time of sample collection. HTs are determined on the basis of days, hours, and minutes. Preparation HT is calculated from the time of sample collection to the time of completion of the sample preparation process as described in the method, prior to any necessary extract cleanup and/or volume reduction. If no prep, analysis HT is calculated from sample collection to completion of all analytical runs, including dilutions, second column confirmations, and any required re-analyses. When sample prep is required prior to analysis, the analysis HT is calculated from the time of prep to completion of all analytical runs. If HT is exceeded, the result must be flagged and identified in the case narrative. Samples not preserved or analyzed in accordance with preservation and HT requirements, must be re-sampled and reanalyzed at the discretion of and at no additional cost to AFCEE.	"Holding time" is the duration between the date and time of sample collection and the dates and times of sample preparation (extraction/distillation) and/or analysis.
Client Approval Needed for Some Preferred Methods	Any modifications to existing method requirements require project-specific approval by DoD personnel. Soxhlet extraction (SW3540) is preferred by the USACE. Written approval is needed to use sonication (SW3550B) instead. The laboratory performs SW3540 only for Method 8141A/B, as required. Although solid-phase extraction (SPE SW3535) is preferred for explosives in water samples, the	Specific screening methods must be selected from AFCEE QAPP Table 6-1 unless a variance is requested. Screening QC are prescribed in Table 6.2-1. All screening data must be flagged with an "S" qualifier. Sample preparation methods are listed in Table 7.1-1. Method SW3535A, SPE extraction of aqueous samples, is recommended, especially for	Laboratory-selected or modified methods must be approved by the DOE client prior to use.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	must comply with method-specific requirements or be within ±5% of set temperature.		

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A/B, AND RSK-175)

The following table summarizes the DoD and AFCEE requirements that are specific to the GC and HPLC SW 846 Methods 8011, 8015, 8021, 8081, 8082, 8141, 8151, 8310, and 8330. These requirements are in addition to or in place of the requirements listed in section 4. The DOE QSAS does not include method-specific requirements and therefore, this table does not include any DOE requirements.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A, AND RSK-175)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
Method 8081 (only) Endrin/DDT Breakdown Check	Performed daily prior to analysis of samples. Degradation must be ≤15% for both Endrin and DDT. Any problems must be corrected. Do not run samples until degradation is ≤15%.	Performed daily prior to analysis of samples. Degradation must be ≤15% for both Endrin and DDT. Any problems must be corrected. Do not run samples until degradation is ≤15%.
Initial Calibration (ICAL)	Perform a minimum 5 point calibration for all analytes prior to sample analysis. Acceptance Criteria options: 1. RSD for each analyte ≤ 20%. 2. Linear least squares regression: r ≥ 0.995. 3. Non-linear regression: coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points must be used for second order). [*This option cannot be used for Method 8082, but is allowed for 8082A.*] Any problems must be corrected and ICAL repeated. No sample can be run until ICAL is successful. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of other Aroclors. Also analyze a mid and low standard for each of the remaining Aroclors for pattern recognition and response factor.	Perform a minimum 5 point calibration for all analytes prior to sample analysis. Any one of the acceptance criteria options below can be applied (except for Method 8082, which may use only Option 1 or 2): 1. Linear: RSD for each analyte ≤ 20%. 2. Linear least squares regression: r ≥ 0.995 for each analyte. 3. Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points must be used for second order). [*This option cannot be used for Method 8082.*] Any problems must be corrected and ICAL repeated. No sample can be run until ICAL is successful. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of other Aroclors. Also analyze a mid and low standard for each of the remaining Aroclors for pattern recognition

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS			
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**	**DOE QSAS**
	must comply with method-specific requirements or be within ±5% of set temperature.		

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A/B, AND RSK-175)

The following table summarizes the DoD and AFCEE requirements that are specific to the GC and HPLC SW 846 Methods 8011, 8015, 8021, 8081, 8082, 8141, 8151, 8310, and 8330. These requirements are in addition to or in place of the requirements listed in section 4. The DOE QSAS does not include method-specific requirements and therefore, this table does not include any DOE requirements.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A, AND RSK-175)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
Method 8081 (only) Endrin/DDT Breakdown Check	Performed daily prior to analysis of samples. Degradation must be ≤15% for both Endrin and DDT. Any problems must be corrected. Do not run samples until degradation is ≤15%.	Performed daily prior to analysis of samples. Degradation must be ≤15% for both Endrin and DDT. Any problems must be corrected. Do not run samples until degradation is ≤15%.
Initial Calibration (ICAL)	Perform a minimum 5 point calibration for all analytes prior to sample analysis. Acceptance Criteria options: 1. RSD for each analyte ≤ 20%. 2. Linear least squares regression: r ≥ 0.995. 3. Non-linear regression: coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points must be used for second order). [*This option cannot be used for Method 8082, but is allowed for 8082A.*] Any problems must be corrected and ICAL repeated. No sample can be run until ICAL is successful. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of other Aroclors. Also analyze a mid and low standard for each of the remaining Aroclors for pattern recognition and response factor.	Perform a minimum 5 point calibration for all analytes prior to sample analysis. Any one of the acceptance criteria options below can be applied (except for Method 8082, which may use only Option 1 or 2): 1. Linear: RSD for each analyte ≤ 20%. 2. Linear least squares regression: r ≥ 0.995 for each analyte. 3. Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points must be used for second order). [*This option cannot be used for Method 8082.*] Any problems must be corrected and ICAL repeated. No sample can be run until ICAL is successful. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of other Aroclors. Also analyze a mid and low standard for each of the remaining Aroclors for pattern recognition

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A, AND RSK-175)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
	Version 4.1: Quantitaion for multi-component analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.	and response factor.
Second-Source Calibration Verification	Measure a second-source standard once after each ICAL. Acceptance limits for all analytes is ± 20% of expected value. Correct any problems and verify second-source standard. Rerun standard. If that fails, correct problem and repeat ICAL. No samples may be analyzed until the second-source calibration verification is successful. **Version 4.1:** Acceptance limits for HPLC Methods: All analytes within ± 15% of expected value.	Measure a second-source standard once after each ICAL. Acceptance limits for all analytes by GC is ± 20% of expected value, and by HPLC, ± 20% of expected value. Correct any problems and verify second-source standard. Rerun standard. If that fails, correct problem and repeat ICAL. No samples may be analyzed until the second-source calibration verification is successful.
RT Window Position	Establish the RT window position for each analyte and surrogate once per ICAL and at the beginning of the analytical shift. Set position using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift.	For GC Methods: Establish the RT window position for each analyte and surrogate for each ICAL and after the initial daily CCV. Set position using the midpoint standard of the initial calibration curve. For HPLC Methods: Establish the RT window position for each analyte and surrogate for each ICAL and at the beginning of the analytical shift. Set position using the midpoint standard of the calibration curve of the value in the calibration verification run at the beginning of the analytical shift (ICV).
RT Window Verification	Verify RT window for each analyte and surrogate for each calibration verification. The analyte must be within the established window. Any problems must be corrected and all samples reanalyzed since the last acceptable RT check. If they fail, redo the ICAL and reset RT window. If CCV, apply a Q-flag to all results for analytes outside the established window. Must have verified RT window at the initial calibration verification. For 8015, check state methods for use of modified RT markers with GRO or DRO.	Verify RT window for each analyte and surrogate for each calibration verification. The analyte must be within the established window. Any problems must be corrected and all samples reanalyzed since the last acceptable RT check. **ICV:** Flagging criteria are not appropriate for initial verification. **CCV:** Apply a Q-flag to all results for the specific analyte(s) in the sample that are outside the established window.
Retention Time (RT)	Perform 72-hour study at method set-up and after major	Perform 72-hour study at method set-up and after major maintenance

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A, AND RSK-175)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
Window Width	maintenance (e.g., column change) to calculate the RT window width for each analyte and surrogate. RT width is ± 3 times the standard deviation for each analyte RT.	(e.g., column change) to calculate the RT window width for each analyte and surrogate. RT width is 3 times the standard deviation for each analyte (each quantitation peak for 8082). **GRO:** Calculate RT based on 2-methylpentane and 1,2,4-trimethylbenzene (per 8015A). **DRO:** Calculate RT based on C10 and C28 alkanes (per 8015B).
Continuing Calibration Verification (CCV)	CCV concentration ≤ midpoint of calibration range. Run at beginning of day, after every 10 field samples, and at the end of the analysis sequence. All analytes must be within ± 20% of expected value from ICAL. Correct any problems, repeat CCV, and reanalyze all samples since last successful CCV. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV if reanalysis is not possible. If %D for an individual analyte > 20%, no samples may be analyzed until the problem has been corrected. **Version 4.1:** Acceptance limits for GC Methods: All analytes within ± 20% of expected value. Acceptance limits for HPLC Methods: All analytes within ± 15% of expected value.	Run after every 10 samples and at the end of the analysis sequence. All analytes must be within ± 20% of expected value (%D). Correct any problems, repeat CCV, and reanalyze all samples since last successful calibration verification. Apply Q-flag to all results for the specific analyte(s) > 20 %D for all associated with the calibration verification.
LCS	Include all analyte(s) in LCS that are required to be reported, including surrogates. Run one LCS per prep batch. Acceptance criteria are specified by DoD, if available. Correct any problems then re-prep and reanalyze the LCS and all associated samples for failed analytes. If insufficient sample, then apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Run one LCS that includes all analytes per analytical batch. Use acceptance criteria in AFCEE QAPP Section 7 tables and AFCEE ME guidance. Correct any problems then reanalyze. If still out, re-prep and reanalyze the LCS and all samples in the affected AFCEE batch. If corrective action fails, apply Q-flag to the specific analyte(s) that are not MEs in all samples in the associated prep batch.
Matrix Spike	Run one per prep batch. Use DoD-specific criteria for LCS. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to	Run one MS/MSD per every 20 Air Force project samples per matrix. Use acceptance criteria for both % recovery (%R) and RPD in AFCEE QAPP Section 7 tables. Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A, AND RSK-175)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
	determine if there is a matrix effect or analytical error.	For specific analyte(s) in all samples collected from the same site matrix as the patent, apply M-flag if (1) MS %R or MSD %R > UCL, (2) MS %R or MSD %R < LCL, or (3) MS/MSD RPD > CL.
MSD or Sample Duplicate	Run one per prep batch. RPD should be ≤ 30%. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference.	Run one MS/MSD per every 20 Air Force project samples per matrix. See above.
Surrogates	Spike all field and QC samples with analytes identified in DoD QSM Appendix DoD-D. Must meet DoD LCS acceptance criteria, otherwise method-specified criteria or lab's in-house criteria. For QC and field samples, correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. For specific analyte(s) in the effected field sample, apply J-flag if acceptance criteria are not met. For 8082 PCB analysis, if both surrogates TCMX and DCBP are analyzed and results are processed for both, then report both even if only one is included in the project work plan.	Add surrogate(s) to every sample, spiked sample, standard, and method blank. Use acceptance criteria in AFCEE QAPP Section 7 tables. Correct any problems, then re-prep and reanalyze the affected samples. If matrix effect is verified, discuss in case narrative. For samples: If %R > UCL for any surrogate, apply J-flag to all positive results for associated analytes. If %R < LCL for any surrogate, apply J-flag to all positive results for associated analytes and UJ-flag to all associated non-detects. If any surrogate recovery is < 10%, apply Q-flag to all results for all associated analytes.
Confirmation of Positive Results	All positive results must be confirmed except toxaphene and technical chlordane in 8081A; and GRO, DRO, and RRO in 8015B. Calibration and QC criteria are the same as for the primary column analysis. The RPD between results for the primary and secondary columns must be ≤ 40%. Apply J-flag if RPD > 40% or Q-flag if sample is not confirmed and discuss in case narrative. Report the higher of the 2 confirmed results unless overlapping peaks are causing erroneously high results then report the unaffected result and document in case narrative.	100% of all positive results must be confirmed except Method 8081A toxaphene and technical chlordane; Method 8015B GRO or DRO; and RSK-175. For Method 8015B, if a significant portion of the peak area observed in the chromatogram does not fall within the C10-C28 DRO range, then the analyst must generate an observation NCM, which the PM will use to qualify the results in the final report case narrative. Calibration and QC criteria are the same as for the primary column analysis. The RPD between results for the primary and secondary columns must be ≤ 40%.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A, AND RSK-175)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
	Version 4.1: If the method does not include reporting requirements, then report the results from the primary column or detector, unless there is a scientifically valid and documented reason for not doing so. Apply J-flag if RPD > 40%. Confirmation is required for all methods except 8015.	Apply J-flag if RPD > 40% from first column result. Apply Q-flag to all results for the specific analyte(s) in the sample not confirmed.
Results Between LOD (MDL) and LOQ (RL) **Version 4.1** Results between DL, LOD, and LOQ	Apply J-flag to all results between LOD and LOQ. **Version 4.1:** Apply J-flag to all results between DL and LOQ.	Apply F-flag to all results between MDL and RL.

REQUIREMENTS FOR ORGANIC ANALYSIS BY HPLC (METHOD 8330B)

The following table summarizes the DoD and AFCEE requirements that are specific to the SW 846 Method 8330B. These requirements are based on in addition to or in place of the requirements listed in section 4. The DOE QSAS does not include method-specific requirements and therefore, this table does not include any DOE requirements.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A, AND RSK-175)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
Soil Drying Procedure	Each sample and batch LCS. The laboratory must have a procedure to determine when the sample is dry to a constant weight. Record date, time, and ambient temperature on a daily basis while drying samples.	NA
Soil Sieving Procedure	Each sample and batch LCS. Weigh the entire sample. Sieve the entire samples with a 10 mesh sieve. Breakup pieces of soil (especially clay) with gloved hands. Do not intentionally include vegetation in the portion of the sample that passes through the sieve unless this is a project specific requirement. Collect and weigh any portion unable to pass through the sieve.	NA
Soil grinding procedure	The laboratory must initially demonstrate that the grinding procedure is capable of reducing the particle size to < 75 µm by passing respresentative portions of ground sample through a 200 mesh sieve (ASTM E11).	NA
Soil grinding blank	Prepared between each sample. A grinding blank using clean solid matrix (such as Ottawa sand) must be prepared and analyzed in the same manner as a field sample. Grinding blanks can be analyzed individually or composited. Acceptance limit is no target analytes detected at greater than ½ LOQ (RL).	
Soil subsampling process	Each sample, duplicate, and batch LCS. Entire ground sample is mixed, spread out on a large flat surface and 30 or more randomly located increments are removed from the entire depth to sum a ~ 10 G subsample.	
Soil sample triplicate	At the subsampling step, one sample per batch.	

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A, AND RSK-175)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
	Three 10 g subsamples are taken from a sample expected to contain the highest levels of explosives within the Quantitation range of the method. The triplicate RSD results must not exceed 20%.	
Aqueous sample preparation	Solid phase extraction is required. The salting-out procedure is not permitted.	
Initial Calibration (ICAL)	Minimum 5 calibration points with the lowest standard at or below the RL (LOQ). Once the calibration curve is established, the lowest calibration standard must be re-analyzed. The signal to noise ratio at the RL must be at least 5:1. Linear regression, acceptance limits $r \geq 0.995$ If using internal standard, acceptance limits RSD ≤ 15%	
Second source calibration verification (ICV)	Immediately following ICAL. All analytes within ± 20% of true value	
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and a the end of the sequence. All target analytes and surrogates within ± 20% of true value.	
Method blank	One per batch No target analytes > ½ RL and greater than 1/10 the amount measured in any sample of 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	
LCS containing all analytes to be reported	One per batch A solid reference material containing all reported analytes must be prepared (e.g. ground and subsampled) and analyzed in exactly the same manner as the field samples. In-house laboratory control limits must demonstrate the laboratory's ability to meet the project MQOs.	
Matrix Spike and matrix spike duplicate (MS/MSD)	One per batch per matrix For matrix evaluation only, therefore is taken post grinding from the same ground sample as the parent subsample is taken. Control limits are the same as for the LCS.	
Confirmation analysis	Required for target analytes detected on the primary column above	

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310, 8330A, AND RSK-175)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
	the LOD. Calibration and QC criteria are the same as for the initial or primary column analysis. Results between primary and secondary column RDP ≤ 40%.	
Results reported between DL and LOQ	Apply J-flag to all results between the DL and LOQ	

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC/MS (METHODS 8260B AND 8270C/D)

The following table summarizes the DoD and AFCEE requirements that are specific to the GC/MS SW 846 Methods 8260 and 8270. These requirements are in addition to or in place of the requirements listed in section 4. The DOE QSAS does not include method-specific requirements and therefore, this table does not include any DOE requirements.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC/MS (METHODS 8260B AND 8270C/D)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
Tuning	Perform tuning prior to calibration and every 12 hours during sample analysis. Use method specified tuning and criteria. If unsuccessful, retune and verify. Rerun any affected samples. No samples may be accepted without a valid tune.	Perform prior to initial calibration and calibration verification. For 8260B, mass spec must be hardware tuned to give an acceptable spectrum for 4-bromofluorobenzene (BFB). The following acceptance criteria (ion abundances for specified masses) apply: Mass 50: 15-40 % of mass 95 Mass 75: 30-60 % of mass 95 Mass 95: base peak, 100 % relative abundance Mass 96: 5-9 % of mass 95 Mass 173: < 2 % of mass 174 Mass 174: > 50 % of mass 95 Mass 175: 5-9 % of mass 174 Mass 176: > 95 %, but < 101 % of mass 174 Mass 177: 5-9 % of mass 176 For 8270C, mass spec must be hardware tuned to give an acceptable spectrum for decafluorotriphenylphosphine (DFTPP). The following acceptance criteria (ion abundances for specified masses) apply: Mass 51: 30-60 % of mass 198 Mass 68: < 2% of mass 69 Mass 70: < 2% of mass 69 Mass 127: 40-60 % of mass 198 Mass 197: < 1 % of mass 198 Mass 198: base peak, 100 % relative abundance Mass 199: 5-9 % of mass 198 Mass 275: 10-30 % of mass 198 Mass 365: > 1 % of mass 198 Mass 441: present, but < mass 443 Mass 442: > 40 % of mass 198 Mass 443: 17-23 % of mass 442 If unsuccessful, retune instrument and verify.
Retention Time (RT)	Perform 72-hour study at method set-up and after major	

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC/MS (METHODS 8260B AND 8270C/D)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
	maintenance (e.g., column change) to calculate the RT width for each analyte and surrogate. RT width is ± 3 times the standard deviation for each analyte RT.	
Method 8270 (only) Breakdown Check (GC Performance Check)	Performed daily prior to analysis of samples. Degradation must be ≤20% for DDT. Any problems must be corrected. Do not run samples until degradation is ≤ 20%. Benzidine and pentachlorophenol should be present at their normal responses and no peak tailing should be observed. **Version 4.1:** Benzidine and pentachlorophenol should be present at their normal responses and peak tailing should not exceed a factor of 2.	Performed daily prior to analysis of samples or calibration standards. Degradation must be ≤20% for DDT. No visible peak tailing for benzidine or pentachlorophenol. As a default, tailing factors should be < 3.0 and 5.0, respectively. Correct any problems, and then repeat performance check.
Initial Calibration (ICAL)	Perform a minimum 5 point calibration for all analytes prior to sample analysis. Acceptance Criteria options: 1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050 2. RSD for RFs for CCCs: VOCs and SVOCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15% Option 2: linear least squares regression r ≥ 0.995 Option 3: non-linear regression coefficient of determination (COD) r^2 ≥ 0.99 (6 points for second order) Any problems must be corrected and ICAL repeated. No sample can be run until ICAL is successful.	Perform a minimum 5 point calibration for all analytes prior to sample analysis. Acceptance Criteria: **SPCCs**: 8260B average RF ≥ 0.30 8270C average RF ≥ 0.050 **CCCs**: %RSD for RFs ≤ 30% **and** one of the options below: Option 1: *linear* - RSD for each analyte < 15% Option 2: *linear* - linear least squares regression r ≥ 0.995 for each analyte Option 3: *non-linear* - COD ≥ 0.99 (6 points for second order) Any problems must be corrected and ICAL repeated. No sample can be run until there is a valid ICAL.
Second-Source Calibration Verification	Measure a second-source standard once after each ICAL. Acceptance limits for all analytes is ± 25% of expected value. Any problems must be corrected. No samples may be analyzed until the second-source calibration verification is successful. **Version 4.1:** All analytes must be within ± 20% of the true value.	Perform a second-source calibration verification once per ICAL. All analytes must be within ± 25% of expected value. Correct any problems and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL. No samples may be analyzed until the calibration has been verified.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC/MS (METHODS 8260B AND 8270C/D)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
RT Window Position	Establish the RT window position for each analyte and surrogate once per ICAL and at the beginning of the analytical shift. Set position using the midpoint standard of the calibration curve.	Establish the RT window position for each analyte and surrogate once per ICAL. Set position using the midpoint standard of the initial calibration curve.
Relative Retention Time (RRT) Verification	Evaluate RRTs for each sample. RRT of each target analyte in each calibration standard must be within ± 0.06 RRT units. Correct any problems, then rerun ICAL.	Evaluate RRTs for each sample. RRT of the analyte must be within ± 0.06 RRT units of ICAL. Correct any problems, then reanalyze all samples analyzed since the last RT check. Apply Q-flag to all results for the specific analyte(s) in the sample that are outside the established window.
Continuing calibration Verification (CCV)	CCV concentration ≤ midpoint of calibration range. Run before sample analysis and every 12 hours of analysis time. The average RF for SPCCs must meet following criteria: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050 The %Difference/Drift for CCCs for both VOCs and SVOCs must meet the following criteria: ≤ 20%. Note that "D" is "difference" when using RFs or "drift" when using regression or non-linear calibration. Correct any problems then rerun the CCV. If that fails, repeat the ICAL. Apply Q-flag if no sample material remains and analyte fails criteria. **Version 4.1:** Acceptance limits all analytes ≤ 20% D	Perform continuing calibration verification (CCV) daily before sample analysis, unless ICAL performed on same day, and after every 12 hours of analysis time. Acceptance critera: **SPCCs:** 8260B: average RF ≥ 0.30 8270C: average RF ≥ 0.050 **CCCs:** ≤ 20 %D All analytes must be within ± 20 %D of expected value from ICAL. (D is "difference" when using RFs, or "drift" when using least squares regression or non-linear calibration.) Correct any problems the rerun CCV. If that fails, repeat ICAL. Apply Q-flag to all results for the specific analyte(s) > 20 %D for all samples associated with the calibration verification.
Internal Standards Verification	Use internal standard in all field samples and standards. RT must be ± 30 seconds from RT of the midpoint standard in the ICAL. EICP area must be within -50% to 100% of ICAL midpoint standard. For any failures, inspect mass spec and GC for malfunctions. Samples analyzed while measurement system was malfunctioning must be reanalyzed. If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging is	Use internal standard in each sample. RT must be ± 30 seconds from RT of the IS in the ICAL mid-point standard. EICP area must be within -50% to +100% of area from IS in ICAL mid-point standard. For any failures, inspect mass spec and GC for malfunctions and make corrections, as appropriate. Samples analyzed while measurement system was malfunctioning must be reanalyzed. Apply Q-flag to all results for analytes associated with a failed IS, unless a

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC/MS (METHODS 8260B AND 8270C/D)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
	not appropriate for failed standards. Sample results are not acceptable without a valid IS verification.	matrix effect can be verified, then apply M-flag.
LCS	Include all analyte(s) in LCS that are required to be reported, including surrogates. Run one LCS per prep batch. Acceptance criteria are specified by DoD, if available. Correct any problems then re-prep and reanalyze the LCS and all associated samples for failed analytes. If insufficient sample or corrective action fails, then apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Run one LCS that includes all analytes per analytical batch. Use acceptance criteria in AFCEE QAPP Section 7 tables and AFCEE ME guidance. Correct any problems then reanalyze. If still out, re-prep and reanalyze the LCS and all samples in the affected AFCEE batch. If corrective action fails, apply Q-flag to the specific analyte(s) that are not MEs in all samples in the associated prep batch.
Matrix Spike	Run one pre prep batch. Use DoD-specific criteria for LCS. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.	Run one MS/MSD per every 20 Air Force project samples per matrix. Use acceptance criteria for both % recovery (%R) and RPD in AFCEE QAPP Section 7 tables. Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs. For specific analyte(s) in all samples collected from the same site matrix as the patent, apply M-flag if (1) MS %R or MSD %R > UCL, (2) MS %R or MSD %R < LCL, or (3) MS/MSD RPD > CL.
MSD or Sample Duplicate	Run one per prep batch. RPD should be ≤ 30%. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference.	Run one MS/MSD per every 20 Air Force project samples per matrix. See above.
Surrogates	Spike all field and QC samples with analytes identified in DoD QSM Appendix DoD-D. Must meet DoD LCS acceptance criteria, otherwise method-specified criteria or lab's in-house criteria. For QC and field samples, correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Add surrogate(s) to every sample, spiked sample, standard, and method blank. Use acceptance criteria in AFCEE QAPP Section 7 tables. Correct any problems, then re-prep and reanalyze the affected samples. If matrix effect is verified, discuss in case narrative. For samples: If %R > UCL for any surrogate, apply J-flag to all positive results for

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC/MS (METHODS 8260B AND 8270C/D)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
	For specific analyte(s) in the effected field sample, apply J-flag if acceptance criteria are not met.	associated analytes. If %R < LCL for any surrogate, apply J-flag to all positive results for associated analytes and UJ-flag to all associated non-detects. If any surrogate recovery is < 10%, apply Q-flag to all results for all associated analytes.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ. **Version 4.1:** Apply J-flag to all results between DL and LOQ.	Apply F-flag to all results between MDL and RL.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC/MS SELECTED ION MONITORING (SIM) METHOD

The following table summarizes the AFCEE requirements that are specific to the GC/MS Selected Ion Monitoring (SIM) Method. The DoD QSM and DOE QSAS do not include method-specific requirements for this method and therefore, this table does not include DoD and DOE requirements.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC/MS SELECTED ION MONITORING (SIM) METHOD	
Requirement	**AFCEE QAPP**
MS Tuning Check	Perform Ms tuning check prior to ICAL and CCV. Use manufacturer's specifications for DFTPP, PFTBA, or other specific compound. Mass assignments should be within ± 0.1 mass units of target values. If check fails, then retune instrument and verify.
Initial Calibration (ICAL)	Perform a minimum 5-point calibration for all analytes prior to sample analysis. Linear RSD for each analyte ≤ 15%; use regression curve for quantitation if RSD exceeds 15%. Linear least squares regression $r^2 \geq 0.99$ for each analyte. Second-order least squares regression COD ≥ 0.99 for each analyte. Correct any problems, then repeat ICAL. Samples may not be analyzed until there is a valid ICAL.
Second-Source Calibration Verification	Perform second-source calibration verification after the ICAL. All analytes must be within ± 25% of expected value. Correct any problems and verify with second-source standard. If that fails, repeat ICAL. Samples may not be analyzed until the calibration has been verified. **Version 4.1**: All analytes within ± 20%
RT Window	The RT window must be verified for each sample. RRT of analyte must be within ± 0.06 RRT units of the ICAL. Correct any problems then reanalyze all samples analyzed since the last RT check. Apply Q-flag to all results for the specific analyte(s) in the sample that are outside the established window.
Calibration Verification (CCV)	Perform calibration verification daily before sample analyses, unless ICAL is performed on the same day, and every 12 hours of analysis time. All analytes must be within ± 20% of expected value. Correct any problems, then rerun CCV. If that fails, repeat ICAL. Apply Q-flag to all results for the specific analyte(s) > 20 %D for all samples associated with the calibration verification.
Internal Standards (IS)	Add IS to each CCV and sample. RT must be ± 30 seconds from RT of IS in the ICAL mid-point standard. EICP area must be within -50% to +100% of the area from IS in ICAL mid-point standard.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC/MS SELECTED ION MONITORING (SIM) METHOD	
Requirement	**AFCEE QAPP**
	For any IS failure, inspect mass spec and GC for malfunctions. All samples analyzed while the system was malfunctioning must be reanalyzed. Apply Q-flag to all results for analytes associated with a failed IS, unless a matrix effect can be verified, then apply M-flag.
LCS	Run one LCS that includes all analytes per analytical batch. Use acceptance criteria in AFCEE QAPP Section 7 tables and AFCEE ME guidance. Correct any problems then reanalyze. If still out, re-prep and reanalyze the LCS and all samples in the affected AFCEE batch. If corrective action fails, apply Q-flag to the specific analyte(s) that are not MEs in all samples in the associated prep batch.
MS/MSD	Run one MS/MSD per every 20 Air Force project samples per matrix. Use acceptance criteria for both % recovery (%R) and RPD in AFCEE QAPP Section 7 tables. Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs. For specific analyte(s) in all samples collected from the same site matrix as the patent, apply M-flag if (1) MS %R or MSD %R > UCL, (2) MS %R or MSD %R < LCL, or (3) MS/MSD RPD > CL.
Surrogates	Add surrogate(s) to every sample, spiked sample, standard, and method blank. Use acceptance criteria in AFCEE QAPP Section 7 tables. Correct any problems, then re-prep and reanalyze the affected samples. If matrix effect is verified, discuss in case narrative. For samples: If %R > UCL for any surrogate, apply J-flag to all positive results for associated analytes. If %R < LCL for any surrogate, apply J-flag to all positive results for associated analytes and UJ-flag to all associated non-detects. If any surrogate recovery is < 10%, apply Q-flag to all results for all associated analytes.
Results Between LOD (MDL) and LOQ (RL)	Apply F-flag to all results between LOD and LOQ. **Version 4.1**: Apply J-flag to all results between DL and LOQ.

REQUIREMENTS FOR ORGANIC ANALYSIS BY HPLC/MS AND HPLC/MS/MS METHODS

The following table summarizes the AFCEE requirements that are specific to HPLC/MS and HPLC/MS/MS methods. The DoD QSM and DOE QSAS do not include method-specific requirements for these methods and therefore, this table does not include DoD and DOE requirements.

REQUIREMENTS FOR ORGANIC ANALYSIS BY HPLC/MS AND HPLC/MS/MS METHODS	
Requirement	**AFCEE QAPP**
Mass Calibration	Perform mass calibration daily, before sample analysis. Tuning standards should span the working range for the analysis. Mass assignments should be within ± 0.3 mass units of target values. If mass calibration fails, then retune instrument and verify.
Initial Calibration (ICAL)	Perform a minimum 5-point calibration for all analytes prior to sample analysis. Linear RSD for each analyte ≤ 20%. Linear least squares regression r ≥ 0.995 for each analyte. Non-linear - COD ≥ 0.99 for each analyte (use 6 points for second order). Correct any problems, then repeat ICAL. Samples may not be analyzed until there is a valid ICAL.
Second-Source Calibration Verification	Perform second-source calibration verification once after each ICAL. All analytes must be within ± 25% of expected value. Correct any problems and verify second-source standard. Rerun second-source verification. If that fails, repeat ICAL. Samples may not be analyzed until the calibration has been verified.
RT Window Position	Establish the RT window position for each analyte and surrogate once per ICAL. Set position using the midpoint standard of the initial calibration curve.
RT Window Verification	Verify RT window for each analyte for each sample. The RRT of the analyte must be within ± 0.06 RRT units of the ICAL; within ± 0.02 RRT units for perchlorate. Correct any problems, then reanalyze all samples analyzed since the last RT check.
Calibration Verification (ICV and CCV)	Run ICV daily, before sample analysis, unless ICAL is performed on same day. Run CCV after every 10 samples and at the end of the analysis sequence. All analytes must be within ± 20 % of expected value (%D). For the ICV, correct any problems and rerun ICV. If that fails, repeat the ICAL. For the CCV, correct any problems then repeat the CCV. Reanalyze all samples since the last successful calibration verification. Apply Q-flag to all results for the specific analyte(s) > 20 %D for all samples associated with the calibration verification.
Internal Standards (IS)	Add IS to each CCV and sample. RT must be ± 30 seconds from RT of IS in the ICAL mid-point standard. Peak area must be within ± 50 % of the area from IS in ICAL mid-point standard.

REQUIREMENTS FOR ORGANIC ANALYSIS BY HPLC/MS AND HPLC/MS/MS METHODS	
Requirement	**AFCEE QAPP**
	For any IS failure, inspect mass spec and HPLC for malfunctions. All samples analyzed while the system was malfunctioning must be reanalyzed. Apply Q-flag to all results for analytes associated with a failed IS, unless a matrix effect can be verified, then apply M-flag.
^{35}Cl : ^{37}Cl Isotope Ratio for Perchlorate (only)	Determine ^{35}Cl : ^{37}Cl isotope ratio for every sample, spiked sample, standard, and method blank. The isotopic ratio must be between 2.35 and 3.85 (± 25% of theoretical). If ratio fails, reanalyze sample. If necessary, re-prep sample and repeat analysis. Apply Q-flag to all results associated with failed isotopic ratio.
LCS	Run one LCS that includes all analytes per analytical batch. Use acceptance criteria in AFCEE QAPP Section 7 tables and AFCEE ME guidance. Correct any problems then reanalyze. If still out, re-prep and reanalyze the LCS and all samples in the affected AFCEE batch. If corrective action fails, apply Q-flag to the specific analyte(s) that are not MEs in all samples in the associated prep batch.
MS/MSD	Run one MS/MSD per every 20 Air Force project samples per matrix. Use acceptance criteria for both % recovery (%R) and RPD in AFCEE QAPP Section 7 tables. Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs. For specific analyte(s) in all samples collected from the same site matrix as the patent, apply M-flag if (1) MS %R or MSD %R > UCL, (2) MS %R or MSD %R < LCL, or (3) MS/MSD RPD > CL.
Surrogates	Add surrogate(s) to every sample, spiked sample, standard, and method blank. Use acceptance criteria in AFCEE QAPP Section 7 tables. Correct any problems, then re-prep and reanalyze the affected samples. If matrix effect is verified, discuss in case narrative. For samples: If %R > UCL for any surrogate, apply J-flag to all positive results for associated analytes. If %R < LCL for any surrogate, apply J-flag to all positive results for associated analytes and UJ-flag to all associated non-detects. If any surrogate recovery is < 10%, apply Q-flag to all results for all associated analytes.
Results Between LOD (MDL) and LOQ (RL)	Apply F-flag to all results between LOD and LOQ.

REQUIREMENTS FOR INORGANIC ANALYSIS (METALS) BY ICP, ICP/MS, AND AA (METHODS 6010B/C, 6020/A, AND 7000 SERIES)

The following table summarizes the DoD and AFCEE requirements that are specific to the ICP, ICP/MS, atomic absorption, and CVAA SW-846 Methods 6010B, 6020, and 7000 series, respectively. The DOE QSAS does not include method-specific requirements and therefore, this table does not include DOE requirements.

REQUIREMENTS FOR INORGANIC ANALYSIS (METALS) BY ICP, ICP/MS, AND AA (METHODS 6010B, 6020, AND 7000 SERIES)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
Instrument Detection Limit Study (ICP & ICP/MS)	Perform an IDL study every 3 months. IDLs must be ≤ MDL. Samples cannot be analyzed without a valid IDL. **Version 4.1:** IDL shall be ≤ LOD	*NOTE: AFCEE QAPP does not specify an IDL for ICP, but only for ICP/MS.* Perform an IDL study at initial setup. IDLs must be ≤ MDL. Samples cannot be analyzed without a valid IDL.
Linear Dynamic Range (LDR) Check Standard (ICP & ICP/MS)	Run an LDR or high-level check standard at least once every 6 months. When calibrating with a single standard and a blank, the daily LDR standard must be analyzed at a concentration greater than any samples analyzed that day Must be within ± 10% of expected value.	Run an LDR or high-level check standard every 3 months. Analyte must be within ± 10% of expected value. Correct any problems then reanalyze of re-set linear range. Apply J-flag to the specific analyte(s) for all results not within linear range.
MS Tuning	Tuning must be performed prior to initial calibration. The following acceptance criteria apply: Mass calibration: ≤ 0.1 amu from the true value Resolution: < 0.9 amu full width at 10% peak height Stability: RSD ≤ 5% for at least 4 replicate analytes If tuning fails, then retune instrument and reanalyze tuning solutions. Do not analyze samples without a valid MS tune.	Tuning must be performed prior to initial calibration. The following acceptance criteria apply: Mass calibration: ≤ 0.1 amu from the true value Resolution: < 0.9 amu full width at 10% peak height Stability: RSD ≤ 5% for at least 4 replicate analytes If tuning fails, then retune instrument and reanalyze tuning solutions. Do not analyze samples without a valid MS tune.
Initial Calibration (ICAL)	**ICP:** Measure a minimum of one high standard and a calibration blank. If more than one standard used, then r must be ≥ 0.995, otherwise no acceptance criteria. **ICP/MS:** If more than one standard used, then r must be ≥ 0.995 **CVAA:** Measure a minimum of 5 standards and a calibration blank; r must be ≥ 0.995. The ICAL must pass before running any samples. **NOTE:** The laboratory currently performs duplicate burns for the	**ICP:** Measure a minimum of one standard and a blank daily, prior to sample analysis. If more than one standard is used, then correlation coefficient must be ≥ 0.995. **ICP/MS:** Measure a minimum of one standard and a blank daily, prior to sample analysis. If more than one standard used, then correlation coefficient must be ≥ 0.995 **CVAA:** Measure a minimum of 5 standards and a calibration blank daily, prior to sample analysis; correlation coefficient must

REQUIREMENTS FOR INORGANIC ANALYSIS (METALS) BY ICP, ICP/MS, AND AA (METHODS 6010B, 6020, AND 7000 SERIES)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
	ICAPES method.	be ≥ 0.995 for linear regression. Samples may not be analyzed until there is a valid ICAL. **NOTE:** The laboratory currently performs duplicate burns for the ICAPES method.
Second-Source Calibration Verification (Initial Calibration Verification or ICV)	Run second-source standard once after each ICAL and prior to sample analysis. Must be within ± 10% of expected value for all analytes. Correct any problems, verify standard, and rerun ICV. If that fails, correct problem and rerun ICAL. Verification must pass before running any samples.	Run ICV daily after ICAL. For ICP/MS, run ICV after ICAL, before beginning a sample run, at a conc other than used for calibration. All analytes must be within ± 10% of expected. Correct any problems, verify standard, and rerun ICV. If that fails, correct problem and repeat ICAL. Verification must pass before running any samples.
Continuing Calibration Verification (CCV)	Run CCV at beginning of day, after every 10 samples, and at the end of the analysis sequence. **ICP:** within ± 10% of expected value **ICP/MS:** within ± 10% of expected value **CVAA:** within ± 20% of expected value Correct any problems, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since last successful CCV. Results cannot be reported without a valid CCV.	Run CCV after every 10 samples and at the end of the analysis sequence. For ICP/MS, conc should be near the middle of the calibration range. **ICP:** all analytes within ± 10% of expected value and RSD of replicate integrations < 5% **ICP/MS:** within ± 10% of expected value **CVAA:** within ± 20% of expected value Correct any problems, then repeat CCV, and reanalyze all samples since last successful calibration verification. Apply Q-flag to all results for the specific analyte(s) > 10 %D for all samples associated with the calibration verification.
Low-Level Calibration Check (ICP & ICP/MS)	Run low-level standard at a concentration ≤ RL daily after one-point ICAL. Must be within ± 20% of expected value. Correct any problems, then reanalyze. Results cannot be reported without a valid low-level check.	Run low-level calibration check standard at a concentration ≤ RL daily, after ICAL. Not required if a ≥ 3-point calibration is performed with the low standard at or below RL. All analytes must be within ± 20% of expected value. Correct any problems, then reanalyze. Results cannot be reported without a valid low-level check.
Calibration Blank	Analyze calibration blank before analyzing samples, after every 10 samples, and at the end of the analysis sequence. Blank contamination must be ≤ 2 x MDL. Correct any problems, then re-prep and reanalyze the calibration blank and 10 previous samples. Apply B-flag to all results for specific analyte(s) in all samples associated with the failed blank.	Analyze calibration blank before beginning a sample run, after every calibration verification (i.e., after every 10 samples), and at the end of an analysis sequence. Blank contamination must be < 2 x MDL. Correct any problems, then re-prep and reanalyze the calibration blank and 10 previous samples. Apply B-flag to all associate positive results for the specific analyte(s) as appropriate.

REQUIREMENTS FOR INORGANIC ANALYSIS (METALS) BY ICP, ICP/MS, AND AA (METHODS 6010B, 6020, AND 7000 SERIES)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
Interference Check Solution (ICS) (ICP & ICP/MS)	Run the ISC at the beginning of an analytical run. **ICS-A:** Absolute value of concentration for non-spiked analytes must be < 2 x MDL (unless element is a verified impurity). **ICS-AB:** Within ± 20% of expected value. Correct any problems and reanalyze ICS. Do not analyze samples without a valid ICS. **NOTE:** TAL Denver has a letter from the ICSA standards manufacturer for many of the elements. The current version of TAL Denver's variance requests for this issue should be included in the Quality Assurance Summary (QAS) for each project.	Run the ISC at the beginning of an analytical run or once during a 12-hour period, whichever is more frequent. **ICS-A:** All non-spiked analytes must be < 2 x MDL (unless element is a verified impurity). **ICS-AB:** Within ± 20% of expected value. Terminate analysis, correct any problems, reanalyze ICS, and reanalyze all affected samples. Do not analyze samples without a valid ICS. **NOTE:** TAL Denver has a letter from the ICSA standards manufacturer for many of the elements. The current version of TAL Denver's variance requests for this issue should be included in the Quality Assurance Summary (QAS) for each project.
LCS	Run one LCS will all analytes required to be reported per prep batch. Recovery must meet DoD QSM limits, if available. **6010 & 7470 Water:** 80-120% (ME limits are 80-120%, except Mo, Se, and Ag, which have limits 75-120%.) **6010 & 7470 Soil:** 80-120%, except Ag 75-120% (ME limits are 80-120%, except Al, SB, Mo, Se, and Zn, which have limits 75-120%, and Ag 70-125%.) Correct any problems, then re-prep and reanalyze LCS and associated samples for failed analytes in all samples in the associated batch. If corrective action fails, apply Q-flag to specific analyte(s) in all samples in associated batch.	Run one LCS will all analytes required to be reported per analytical batch. Recovery must meet recovery and ME limits in AFCEE QAPP Section 7 tables. Correct any problems, then reanalyze. If still out, re-prep and reanalyze the LCS and all samples in the affected AFCEE batch. If corrective action fails, apply Q-flag to specific analyte(s) that are not MEs in all samples in associated prep batch.
Dilution Test	Run dilution test with each prep batch or when a new or unusual matrix is encountered for samples with conc >50 x MDL for ICP, or >25 x MDL for GFAA and CVAA. Five-fold dilution must agree within ± 10% of original determination. If test fails criteria, then perform following corrective action: **ICP:** Perform post-digestion spike (PDS) addition. **GFAA:** Perform recovery test. **CVAA:** Perform matrix spike.	Run dilution test with new sample matrix, at least once per analytical batch, but only for samples with conc ≥ 50 x MDL for ICP, ≥ 100 x MDL for ICP/MS, or >25 x MDL for GFAA and CVAA. Five-fold dilution (1+4) must agree within ± 10% of original determination. If test fails criteria, then perform following corrective action: **ICP:** Perform post-digestion spike (PDS) addition. **ICP/MS:** Perform post-digestion spike (PDS) addition. **CVAA:** None Apply J-flag to all sample results for the specific analyte from the same matrix in the batch if either of the following exist: (1) dilution test not run and batch had analyte conc ≥ 50 x MDL for ICP, ≥ 100 x MDL for

REQUIREMENTS FOR INORGANIC ANALYSIS (METALS) BY ICP, ICP/MS, AND AA (METHODS 6010B, 6020, AND 7000 SERIES)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
		ICP/MS, ≥ 25 x MDL for GFAA or CVAA, OR (2) %D ≥ 10 and PDS (ICP & ICP/MS) or recovery test (GFAA) not performed.
Post-Digestion Spike (PDS) Addition (ICP & ICP/MS)	Perform PDS when dilution test fails or analyte conc in all samples is <50 x MDL for ICP or <100 x MDL for ICP/MS. Spike addition must result in level between 10 - 100 x MDL. Recovery must be within 75 - 125 % of expected result. If PDS fails, then run samples by method of standard addition (MSA) or apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the PDS.	Perform PDS when dilution test fails or if an analyte's conc for all samples in a batch is < 50 x MDL for ICP or <100 x MDL for ICP/MS. Recovery must be within 75 - 125 % of expected results. If PDS fails, check for instrumental problem and reanalyze PDS if appropriate. For ICP/MS, dilute the sample; reanalyze PDS. Apply J-flag to all sample results (for same matrix) for the specific analyte(s) for all samples associated with the PDS. If PDS recovery is < 10%, apply Q-flag to all sample results (for same matrix) for the specific analyte(s) for all samples associated with the PDS.
Recovery Test (GFAA Only)	Perform Recovery Test when dilution test fails or analyte conc in all samples is <25 x MDL. Recovery must be within 85 - 115 % of expected result. If test fails, then run samples by MSA or apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 85 - 115%.	Perform Recovery Test when dilution test fails or analyte conc in all samples in a batch is < 25 x MDL. Recovery must be within 85 - 115 % of expected result. If test fails, then run all samples by MSA. Apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 85 - 115%.
MSA or Internal Standard Calibration	Run MSA or internal standard calibration when matrix interference is suspected. Document use of MSA in case narrative.	
Matrix Spike (MS)	Run one MS per prep batch. Use DoD acceptance criteria for LCS. If MS fails, consult project-specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.	Run one MS/MSD per every 20 Air Force project samples per matrix. Use acceptance criteria for both % recovery (%R) and RPD in AFCEE QAPP Section 7 tables. Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs. For specific analyte(s) in all samples collected from the same site matrix as the patent, apply M-flag if (1) MS %R or MSD %R > UCL, (2) MS %R or MSD %R < LCL, or (3) MS/MSD RPD > CL.
MSD or Sample Duplicate	Analyze one MSD or sample duplicate per prep batch per matrix. RPD between duplicates must be ≤ 20%. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance	Run one MS/MSD per every 20 Air Force project samples per matrix. See above.

REQUIREMENTS FOR INORGANIC ANALYSIS (METALS) BY ICP, ICP/MS, AND AA (METHODS 6010B, 6020, AND 7000 SERIES)		
Requirement	**DoD QSM v3 and v4.1**	**AFCEE QAPP**
	criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference.	
Internal Standard (IS) (ICP/MS Only)	Add IS to every sample. IS intensity must be within 30 - 120% of intensity of the IS in the ICAL. Perform corrective action as described in Method 6020.	Add IS to every sample. IS intensity must be within 30 - 120% of intensity of the IS in the ICAL. Perform corrective action as described in Method 6020. Do not report results unless sample has a valid IS response.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.	Apply F-flag to all results between MDL and RL.

REQUIREMENTS FOR WET CHEMISTRY (METHODS 7196, 9010/9012, AND 9056)

The following tables summarize the DoD and AFCEE requirements that are specific to Wet Chemistry Methods 7196, 9010/9012, and 9056. The DOE QSAS does not include method-specific requirements and therefore, this table does not include DOE requirements.

REQUIREMENTS FOR COLORIMETRIC HEXAVALENT CHROMIUM ANALYSIS (METHOD 7196)		
Requirement	**DoD QSM v3**	**AFCEE QAPP**
Reference Blank (Reagent Water)	Analyze before beginning standards or sample analysis. Use for blank subtraction of standards, field samples, and QC samples. For turbid field samples, a turbidity blank must be used instead of the reference blank.	Analyze once prior to daily ICAL or sample analysis. Used for blank subtraction of standards, and field and QC samples. For turbid field samples, a turbidity blank must be used instead of the reference blank.
ICAL	Perform ICAL daily prior to sample analysis using a minimum of 3 standards plus a calibration blank. Correlation coefficient r must be ≥ 0.995. Do not analyze samples unless ICAL passes criteria.	Perform ICAL daily prior to sample analysis using a minimum of 3 standards plus a calibration blank. Correlation coefficient r must be ≥ 0.995 for linear regression. Do not analyze samples unless ICAL passes criteria.
Second-Source Calibration Verification (ICV)	Run ICV before beginning a sample run. Value of ICV must be within ± 10% of expected value. Correct any problems and verify second-source standard. Rerun ICV. If that fails, correct any problems and repeat ICAL. Reanalyze all samples since last successful calibration.	Run ICV before beginning a sample run. Value of ICV must be within ± 10% of expected value. Correct any problems and verify second-source standard. Rerun ICV. If that fails, correct any problems and repeat ICAL. Samples may not be analyzed until the calibration has been verified.
Continuing Calibration Verification (CCV)	Run CCV after every 15 samples and at the end of the analysis sequence. Value must be within ± 10% of expected value. Correct any problems then repeat CCV and reanalyze all samples since the last successful calibration verification.	Run CCV after every 15 samples and at the end of the analysis sequence. Value must be within ± 10% of expected value. Correct any problems then repeat CCV and reanalyze all samples since the last successful calibration verification. Apply Q-flag to all samples associated with the calibration verification.
Verification Check (Matrix Spike)		Analyze one matrix spike (verification check) once for every sample matrix analyzed. Spike recovery must be 85-115%. The verification check is performed to ensure the lack of reducing condition and/or interference from matrix. If check indicates interference, dilute and reanalyze sample. Persistent interference indicates the need to use an alternate method.
MSD or Sample Duplicate	For aqueous matrix, run one MS/MSD pair per every 10 samples per matrix. For solid matrix, run one MS/MSD pair per prep batch per matrix.	Run one MS/MSD per every 20 Air Force project samples per matrix. Use acceptance criteria for both % recovery (%R) and RPD in AFCEE

REQUIREMENTS FOR COLORIMETRIC HEXAVALENT CHROMIUM ANALYSIS (METHOD 7196)		
Requirement	**DoD QSM v3**	**AFCEE QAPP**
	RPD between duplicates must be ≤ 20% for aqueous matrix, and ≤ 30% for solid matrix. Apply J-flag for the specific analyte(s) in parent sample if acceptance criteria not met. Contact client to determine if additional measures are desired.	QAPP Section 7 tables. Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs. For specific analyte(s) in all samples collected from the same site matrix as the patent, apply M-flag if (1) MS %R or MSD %R > UCL, (2) MS %R or MSD %R < LCL, or (3) MS/MSD RPD > CL.
Pre-digestion Matrix Spikes Method 3060 Solid Matrix Only	Analyze one soluble and one insoluble pre-digestion MS pre prep batch prior to analysis. MS recoveries must be within 75 - 125%. Correct any problems and rehomogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results. If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.	Apply F-flag to all results between MDL and RL.

REQUIREMENTS FOR CYANIDE ANALYSIS (METHODS 9010/9012)		
Requirement	**DoD QSM v3**	**AFCEE QAPP**
ICAL	Run initial calibration, using at least 6 standards and a calibration blank, daily prior to sample analysis. Correlation coefficient r must be ≥ 0.995 for linear regression. Do not analyze samples unless ICAL passes criteria.	Run initial calibration, using at least 6 standards and a calibration blank, daily prior to sample analysis. Correlation coefficient must be ≥ 0.995 for linear regression. Correct any problems and repeat ICAL. Do not analyze samples unless ICAL passes criteria.
Distilled Standards	Analyze one high and one low distilled standard once per multipoint calibration. Values must be within ± 15% of true value. Correct any problems, then repeat. Do not analyze samples until distilled standards have passed.	Analyze one high and one low distilled standard once per multipoint calibration. Values must be within ± 15% of true value. Correct any problems, then repeat. Do not analyze samples until distilled standards have passed.

REQUIREMENTS FOR CYANIDE ANALYSIS (METHODS 9010/9012)		
Requirement	**DoD QSM v3**	**AFCEE QAPP**
Second-Source Calibration Verification	Analyze second-source calibration verification check standard once after each multipoint calibration. Value must be within ± 15% of expected value. Correct any problems and verify standard. Rerun verification. If that fails, correct any problems and repeat ICAL.	Analyze second-source calibration verification standard once after each ICAL. Value must be within ± 15% of expected value. Correct any problems and verify standard. Rerun verification. If that fails, correct any problems and repeat ICAL. Samples may not be analyzed until the calibration has been verified.
MS/MSD	For 9010, run one MS/MSD pair per prep batch per matrix. For 9012, run one MS/MSD pair per every 10 samples per matrix. Use DoD LCS QC acceptance criteria. RPD between MS and MSD must be ≤ 20%. Apply J-flag for the specific analyte(s) in parent sample if acceptance criteria not met. Contact client to determine if additional measures are desired.	Run one MS/MSD per every 20 Air Force project samples per matrix. Use acceptance criteria for both % recovery (%R) and RPD in AFCEE QAPP Section 7 tables. Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs. For specific analyte(s) in all samples collected from the same site matrix as the patent, apply M-flag if (1) MS %R or MSD %R > UCL, (2) MS %R or MSD %R < LCL, or (3) MS/MSD RPD > CL.
Sample Duplicate	Analyze duplicate sample once per every 20 samples. % Difference between duplicates must be ± 20% of sample. Correct any problems and reanalyze sample and duplicate. Apply Q-flag if sample cannot be rerun or reanalysis does not correct problem.	Analyze duplicate sample once per every 20 project samples. % Difference between duplicates must be ± 20% of sample. Correct any problems and reanalyze sample and duplicate. Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.	Apply F-flag to all results between MDL and RL.

REQUIREMENTS FOR COMMON ANIONS ANALYSIS (METHOD 9056)		
Requirement	**DoD QSM v3**	**AFCEE QAPP**
ICAL	Run initial calibration for all analytes, using at least 3 standards and a calibration blank, prior to sample analysis. Correlation coefficient r must be ≥ 0.995 for linear regression. Do not analyze samples unless ICAL passes criteria.	Run initial calibration for all analytes, using at least 3 standards and a calibration blank, prior to sample analysis. Calibration must comply with one of the options below: Option 1: Linear - RSD for each analyte must be ≤ 10%. Option 2: Linear - least squares regression r must be ≥ 0.995 for each analyte.

REQUIREMENTS FOR COMMON ANIONS ANALYSIS (METHOD 9056)		
Requirement	**DoD QSM v3**	**AFCEE QAPP**
		Option 3: Non-linear - COD must be ≥ 0.99 (use 6 points for second order) Correct any problems and repeat ICAL. Do not analyze samples unless there is a valid ICAL.
Second-Source Calibration Verification	Analyze second-source calibration verification check standard once after each multipoint calibration. Value for all analytes must be within ± 10% of expected value. Correct any problems and verify standard. Rerun verification. If that fails, correct any problems and repeat ICAL.	Analyze second-source calibration verification standard once after each ICAL. Value for all analytes must be within ± 10% of expected value. Correct any problems and verify standard. Rerun verification. If that fails, correct any problems and repeat ICAL. Samples may not be analyzed until the calibration has been verified.
RT Window Position	Establish RT window position for each analyte once per multipoint calibration. Position shall be at midpoint of ICAL curve.	Establish RT window position for each analyte once per ICAL and at the beginning of the analytical shift. Position shall be at the midpoint standard of ICAL curve or the value in the calibration verification run at the beginning of the analytical shift.
Retention Time (RT) Window Width	Calculate RT window width for each analyte after method set-up and after major maintenance (e.g., column change). RT width is ± 3 times standard deviation for each analyte over 24-hour period.	Calculate RT window width for each analyte after method set-up and after major maintenance (e.g., column change). RT width is ± 3 times standard deviation for each analyte RT over 24-hour period.
RT Window Verification	RT window verification is performed for each CCV. Each analyte peak must fall within the established window. Correct any problems, then reanalyze all samples since the last RT check. If they fail, redo the ICAL and reset RT window.	RT window is verified for each analyte for each CCV. Each analyte peak must fall within the established window. Correct any problems, then reanalyze all samples analyzed since the last acceptable RT check. If the RT verification fails for the ICV, flagging is not appropriate. If the RT verification fails for the CCV, apply Q-flag to all results for the specific analyte(s) in the samples that are outside the established window.
Initial Calibration Verification (ICV)	Perform ICV daily before sample analysis, when eluent is changed, and with every batch of samples. All analytes must be within ± 10% of expected value AND RTs must be within established windows. Correct any problems, rerun ICV. If that fails, then repeat ICAL.	Perform ICV daily before sample analysis (unless the ICAL was performed on the same day), and when eluent is changed. All analytes must be within ± 10% of expected value AND RTs must be within established windows (see above). Correct any problems, rerun ICV. If that fails, then repeat ICAL.
Continuing Calibration Verification (CCV)	Run midrange CCV standard after every 10 samples and at the end of the analytical sequence. Instrument response must be within ± 10% of expected value. Correct any problems, then repeat CCV and reanalyze all samples since last successful calibration verification. Apply Q-flag to all results for the specific analyte(s) in all samples	Run CCV standard after every 10 samples and at the end of the analytical sequence. All analytes must be within ± 10% of expected value AND RTs must be within established windows (see above). Correct any problems, then repeat CCV and reanalyze all samples since last successful calibration verification. Apply Q-flag to all results for the specific analyte(s) > 10 %D for all

REQUIREMENTS FOR COMMON ANIONS ANALYSIS (METHOD 9056)		
Requirement	**DoD QSM v3**	**AFCEE QAPP**
	since the last acceptable calibration verification.	samples associated with the calibration verification.
MS/MSD	Run one MS/MSD pair per prep batch per matrix. Use DoD LCS QC acceptance criteria. RPD between MS and MSD must be ≤ 20%. Apply J-flag for the specific analyte(s) in parent sample if acceptance criteria not met. Contact client to determine if additional measures are desired.	Run one MS/MSD per every 20 Air Force project samples per matrix. Use acceptance criteria for both % recovery (%R) and RPD in AFCEE QAPP Section 7 tables. Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs. For specific analyte(s) in all samples collected from the same site matrix as the patent, apply M-flag if (1) MS %R or MSD %R > UCL, (2) MS %R or MSD %R < LCL, or (3) MS/MSD RPD > CL.
Sample Duplicate	Analyze one sample duplicate per every 10 samples. % Difference between sample and its duplicate must be ≤ 10%. Correct any problems and reanalyze sample and duplicate. If corrective action fails, apply Q-flag to specific analyte(s) in the sample.	Analyze one sample duplicate per every 10 samples. % Difference between sample and its duplicate must be ≤ 10%. Correct any problems and reanalyze sample and duplicate. If corrective action fails, apply J-flag to specific analyte(s) in the sample.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.	Apply F-flag to all results between MDL and RL.

REQUIREMENTS FOR PERCHLORATE ANION BY ION CHROMATOGRAPHY (METHOD 314.0)	
Requirement	**AFCEE QAPP**
MCT Determination	Perform MCT determination at initial setup and once per 12-month period. Calculate $PD_{A/H}$ for the perchlorate peak at increasing onc of mixed common anion solution. The MCT is the matrix conductance where the $PD_{A/H}$ exceeds 20 %. Option 1: Least squares regression - plot $PD_{A/H}$ versus matrix conductance. COD r^2 must be > 0.95. Option 2: Use the conductance level of the highest mixed anion solution that yeilded a $PD_{A/H}$ value < 20%. Samples may not be analyzed without a valid MCT.
ICAL	Run initial calibration for all analytes, using at least 5 standards, prior to sample analysis. Calibration must comply with one of the options below: Option 1: Linear - mean RSD must be ≤ 15%.

REQUIREMENTS FOR PERCHLORATE ANION BY ION CHROMATOGRAPHY (METHOD 314.0)	
Requirement	**AFCEE QAPP**
	Option 2: Linear - least squares regression r must be > 0.995. Option 3: Non-linear - COD must be ≥ 0.99 (use 6 points for second order). Correct any problems and repeat ICAL. Do not analyze samples unless there is a valid ICAL.
Second-Source Calibration Verification	Analyze second-source calibration verification standard once per each ICAL, upon re-establishing calibration, and quarterly. Instrument response must be within ± 10% of expected value. Correct any problems and verify standard. Rerun verification. If that fails, correct any problems and repeat ICAL. Samples may not be analyzed until the calibration has been verified.
Instrument Performance Check (IPC)	Run IPC daily, before sample analysis. Conductance must be within 10% of original value (original value within ± 10% of MCT). $PD_{A/H}$ must be < 25%. Instrument response must be within ± 20% of expected response. RT shifts > 5% from previous analysis, or overall RT < 80% of original recorded value. If conductance of IPC solution does meet acceptance criteria, then prepare fresh IPC. Re-determine MCT or correct problem and reanalyze IPC. If response and/or RTs fail acceptance criteria, then correct problems, clean or replace column. Samples may not be analyzed until the IPC criteria have been met.
Initial Calibration Verification (ICV/ICCS)	Perform ICV daily before sample analysis (unless the ICAL was performed on the same day), and when eluent is changed. Instrument response must be within ± 25% of expected value using a standard at or below the RL. Correct any problems, rerun ICV/ICCS. If that fails, then repeat ICAL.
Calibration Verification (CCV/CCCS and ECCS)	Run CCV standard after every 10 samples (CCCS) and at the end of the analytical sequence (ECCS). Instrument response must be within ± 15% of expectred response, alternately using separate mid- and high-level standards. Correct any problems, then repeat calibration verification. Reanalyze all samples since last successful calibration verification. Apply Q-flag all samples associated with the calibration verification.
Pretreated Laboratory Reagent Blank (LRB)	The LRB is required in an analytical batch that includes samples that have been pretreated to reduce the common anion levels. Perchlorate must be < ½ RL. Correct any problems, then re-prep and analyze pretreated LRB and all samples processed with the contaminated blank. Apply B-flag to all associated positive results as appropriate.
MS/MSD	Run one MS/MSD per every 20 Air Force project samples per matrix. Use acceptance criteria for both % recovery (%R) and RPD in AFCEE QAPP Section 7 tables. Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs. For specific analyte(s) in all samples collected from the same site matrix as the patent, apply M-flag if (1) MS %R or MSD %R > UCL, (2) MS %R or MSD %R < LCL, or (3) MS/MSD RPD > CL.
RL Verification	Perform RL verification at initial setup and once per 12-month period. Instrument response must be within ± 30% of expected response for a mixed common anion solution containing perchlorate at the RL. Conductance must be within ± 10% of the MCT. If verification fails, then lower the MCT by 10% and repeat the RL verification. Samples may not be analyzed without a valid RL verificaiton.

REQUIREMENTS FOR PERCHLORATE ANION BY ION CHROMATOGRAPHY (METHOD 314.0)	
Requirement	**AFCEE QAPP**
Results Between LOD (MDL) and LOQ (RL)	Apply F-flag to all results between MDL and RL.

REQUIREMENTS FOR PERCHLORATE ANALYSIS FOR DoD ENVIRONMENTAL RESTORATION/CLEANUP PROJECTS

The following table summarizes the DoD requirements for the determination of perchlorate in environmental matrices as specified in the DoD Perchlorate Handbook. It should be noted that the DoD has mandated that ion chromatographic EPA Methods 314.0 and 314.1 are not appropriate for sampling and analysis associated with environmental restoration /cleanup or range assessment activities. Only methods employing mass spectrometry are to be used for these project. TestAmerica Denver uses ion chromatography coupled with electrospray tandem mass spectrometry (IC/MS/MS). The TestAmerica Denver method is defined in SOP DV-LC-0024, which complies with the *DoD Standard Operating Procedure for the Analysis of Perchlorate in Water, Soils and Solid Wastes Using Ion Chromatography / Electrospray / Mass Spectrometry (IC/MS or IC/MS/MS)*. The following table includes only those requirements that are not already defined under the general requirements section (Section -).

REQUIREMENTS FOR PERCHLORATE ANALYSIS FOR DoD ENVIRONMENTAL RESTORATION/CLEANUP	
Requirement	**AFCEE QAPP**
Mass Tuning	Optimize setting of mass spec daily before sample analysis. Tuning standards should contain the analytes of interest. Must meet acceptance criteria in laboratory SOP (DV-LC-0024). If tuning fails, then retune instrument. If tuning will not meet acceptance criteria, perform mass calibration and retune. Samples may not be analyzed without acceptable tuning.
Mass Calibration	A valid mass calibration is required prior to any sample analysis. Update calibration on as-needed basis (e.g., QC failures, ion masses show large deviations from known masses, major instrument maintenance is performed, or the instrument is moved). Calibration range must bracket the ion masses of interest without greatly exceeding the range. Use the most recent mass calibration for the analytical run. Use same mass calibration for all data files in an analytical run. Verify mass calibration by acquiring a full scan continuum mass spectrum of a perchlorate stock standard. Perchlorate ion should be within ± 0.3 m/z of masses 99, 101, and 107. If mass calibration fails, recalibrate. No samples may be analyzed under a failing mass calibration.
Retention Time (RT) Window Width	Perform 72-hour study to establish RT window width at the time of method setup and after major maintenance (e.g., column change). Width is set at ± 3 standard deviations for each analyte RT. **NOTE:** The method uses an internal standard, and therefore, retention times expressed relative to the retention time of the internal standard, i.e., relative retention time or RRT. The 72-hour study is not appropriate for a method that uses an internal standard and

REQUIREMENTS FOR PERCHLORATE ANALYSIS FOR DoD ENVIRONMENTAL RESTORATION/CLEANUP	
Requirement	**AFCEE QAPP**
	RRTs, and is therefore not included in DV-LC-0024.
RT Window Position	Set RT window position once per ICAL and at the beginning of the analytical shift. Set position using the mid-point standard of the calibration curve of the value in the CCV run at the beginning of the analytical shift. **NOTE:** The method uses an internal standard, and therefore, RRTs. The DoD SOP for perchlorate stipulates RRT criteria of 1.0 ± 2%, which is included in DV-LC-0024.
ICAL	Run initial calibration using at least 5 standards daily, prior to sample analysis. The calibration must be linear and shall not be forced through the origin. The concentration corresponding to the absolute value of the calibration curve's Y-intercept must be ≤ LOD. The calibration curve must comply with one of the options below: Option 1: Linear - RSD for each analyte (including MRL) must be must be ≤ 20%. Option 2: Linear - least squares regression r must be ≥ 0.995. Correct any problems then repeat ICAL. Do not analyze samples unless there is a valid ICAL.
Second-Source Calibration Verification (SSCV)	Analyze second-source calibration verification standard at the mid-point of the calibration range once after each ICAL. Perchlorate value must be within ± 10% of expected value. Correct any problems and verify second-source standard. Rerun SSCV. If that fails, correct any problems and repeat ICAL. Samples may not be analyzed until the calibration has been verified.
Initial Calibration Verification Standard (ICV)	Run ICV standard at the mid-point of the calibration range after the daily ICAL. % Difference must be ≤ 15% relative to initial value. Correct any problems and rerun ICV. If that fails, correct problem and repeat ICAL. No samples may be run until calibration has been verified.
Continuing Calibration Verification (CCV)	Analyze mid-level CCV standard after every 10 samples. All samples should be bracketed by the analysis of a standard demonstrating that the system was capable of accurately detecting and quantifying perchlorate. % Difference must be ≤ 15% relative to initial value. Correct any problems and rerun CCV and all samples analyzed since the last successful CCV. If that fails, apply Q-flag to all results in all samples since the last acceptable calibration verification, if reanalysis is not possible.
Limit of Detection Verification Standard (LODV)	Analyze LODV standard before and directly after every batch of samples. Spike LODV at approximately 2 x LOD. It can be analyzed after every 10 samples to reduce the reanalysis rate. Recovery must be within ± 30 % of true value. If a sample with perchlorate conc at or between LOD and RL is bracketed by a failing LODV, it must be reanalyzed. A sample with conc above the RL can be reported. Correct any problems and rerun the LODV and all samples analyzed since the last successful LODV. If that fails, and reanalysis is not possible, Q-flag all results in all samples since the last acceptable calibration verification.
Interference Threshold Study	Perform an interference threshold study at initial setup and when major changes occur in the method's operating procedures (e.g., addition of cleanup procedures, column changes, mobile phase changes, etc.). Measure the threshold of common suppressors (chloride, sulfate, carbonate, bicarbonate) that can be present in the system without affecting the quantitation of perchlorate. The threshold is the conc of the common suppressors where perchlorate recovery falls outside an 85-115% window.

REQUIREMENTS FOR PERCHLORATE ANALYSIS FOR DoD ENVIRONMENTAL RESTORATION/CLEANUP	
Requirement	**AFCEE QAPP**
	NOTE: The IC/MS/MS method uses a labeled perchlorate as an internal standard. If the matrix of a sample contains enough interfering species to adversely affect the quantitation of perchlorate, the internal standard recovery would fail. However the level of anions needed to cause this to happen would also plug up the system and seriously damage the instrumentation. Consequently, the threshold study used a maximum concentration of 5000 mg/L for each anion, which is much greater than could be expected in any water samples submitted to the lab. The perchlorate recovery at this level was unaffected.
Interference Check Sample (ICS)	Extract one ICS with every batch of 20 or fewer samples. The ICS shall contain perchlorate at the RL and interfering anions at the concentration determined by the interference threshold study. The ICS is used to verify the method performance at the matrix conductivity threshold (MCT). At least one ICS must be analyzed daily. Recovery of perchlorate must be within ±30 % of true value. RT must be monitored. Correct any problems and then reanalyze all samples in the batch. If poor recovery from the cleanup filters is suspected, a different lot of filters must be used to re-extract all samples in the batch. If column degradation is suspected, a new column must be calibrated before the samples can be reanalyzed. No samples may be reported that are associated with a failing ICS. **NOTE:** As noted above, the method uses a labeled perchlorate internal standard, which is sufficient for evaluating the effects of interfering species. Consequently, the ICS is analyzed with each initial calibration to demonstrate the ability of the method to accurately determine perchlorate at low levels in the presence of interfering species.
Laboratory Control Sample (LCS)	Run one LCS per prep batch to undergo same pretreatment steps as samples. Recovery must be within laboratory-generated limits, which must be no wider than 85 to 115%. Correct any problems, then re-prep and reanalyze the LCS and all associated samples. If corrective action fails, apply Q-flag to all samples in the associated prep batch.
Matrix Spikes (MS)	Run one MS per 20 samples per matrix. Spike aliquot of selected sample at the RL. Recovery must be within 75 - 125%. If acceptance criteria are not met, apply J-flag to parent sample. If MS results are outside the limits, the data must be evaluated to determine the source of the difference and to determine if these is a matrix effect or analytical error.
Matrix Spike Duplicates or Laboratory Duplicates (MS/MSD)	Run one MSD per 20 samples per matrix. Spike aliquot of selected sample at the RL. Recovery must be within 75 - 125%. RPD must be < 20%. If acceptance criteria are not met, apply J-flag to parent sample. If MS results are outside the limits, the data must be evaluated to determine the source of the difference and to determine if these is a matrix effect or analytical error.
Laboratory Reagent Blank (LRB)	Analyze LRB prior to calibration and after samples with overrange conc of perchlorate and after each batch is analyzed. Concentration of perchlorate in LRB must be < ½ RL. If LRB is contaminated, reanalyze LRB until no carryover is observed and all samples processed since the contaminated blank. Apply B-flag to all results not preceded by an acceptable reagent blank if reanalysis is not possible.
^{35}Cl / ^{37}Cl Isotope Ratio	Determine ration for every sample, spiked sample, standard, and method blank. Monitor for both the parent ion at mass 99/101 and the daughter ion at mass 83/85 for tandem MS methods, or just 99/101 for MS only. The ratio must fall within 2.3 to 3.8 (the theoretical ratio is 3.06). If ratio falls outside of limits, then rerun sample. If sample was not pretreated, the sample should be re-extracted using cleanup procedures.

REQUIREMENTS FOR PERCHLORATE ANALYSIS FOR DoD ENVIRONMENTAL RESTORATION/CLEANUP	
Requirement	**AFCEE QAPP**
	If, after cleanup, the ration still fails, use alternative techniques to confirm the presence of perchlorate (i.e., a post spike sample, dilution to reduce any interferences, etc.). Data should be qualified as estimated with a J-flag and should be noted in the case narrative.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.

1. Variances and clarifications to the QSM v3

TestAmerica Denver will work with our DoD clients to develop a QAPP that meets data quality objectives for the project and that meets the laboratory's capabilities.

In a few situations, TestAmerica Denver is not able to meet the requirements in the DoD QSM, and in other situations the requirements are unclear and may be subject to a range of interpretations. To avoid misunderstandings and potential project failures, the laboratory will supply the document tiled "TestAmerica Denver Technical Approach to the DoD QSM" during the project setup phase, which details the issues requiring variances or clarifications. The current document is saved to TestAmerica Denver's Public Outlook folders, and an example document is as follows:

TestAmerica Denver requests the following variances and clarifications to the DoD QSM Version 3 Final, January 2006.

Variance Requests

1. Method Blanks – Appendix DOD-B Tables
   QSM 3 Requirement: No analytes detected > ½ RL, flag failures with a B data qualifier
   Proposal: TestAmerica Denver's LIMS system automatically applies a B flag to results when the method blank exceeds the MDL, rather than ½ RL (i.e., we are over-flagging). However, the laboratory is able to produce reports which correct this level of flagging for most analytical data. Currently, the laboratory's Wet Chemistry data is over-flagged. For all data, the laboratory is providing corrective action to the ½ RL level.

2. Holding Times – Section 5.10.2.g, Grey Box 75
   QSM 3 Requirement: For DoD work, both date and time of analysis are considered to be essential information, regardless of the length of the holding time, and shall be included as part of the laboratory report.
   Proposal: The laboratory does routinely report date and time of analysis. However, TestAmerica Denver normally calculates holding time to the same units as expressed in the methods. For example, if the holding time is expressed in hours, then we measure to the hour; and if expressed in days, then we measure to the day. We believe our practice is consistent with the methods and the standard practice of EPA data validators. For most projects this is not considered a variance, but we want to ensure that this practice is brought to the attention of project personnel.

Clarifications

TestAmerica Denver believes that the following items do not constitute variances, but are issues open to a range of interpretations. Our goal here is to clarify all potential technical issues at the outset of projects to ensure that there are no misunderstandings. If these clarifications need to be submitted to DoD for approval, we are prepared to defend them.

1. Marginal Exceedances for LCS Failures - Section D.1.1.2.1.e, Grey Box D-9
   QSM 3 Requirement: DoD does not allow any project-specific analytes of concern to exceed its LCS control limits, even marginally.
   Clarification: The phrase "project-specific analytes of concern" is not defined and is unclear. Based on discussions with Dr. Joseph Solsky and Dr. Chung-Rei Mao at the USACE/CX branch, TestAmerica Denver understands that the intent was not to imply all target analytes. Instead, it was meant to apply to analytes that are identified site contaminants and are identified as primary drivers for environmental decisions. For example, if vinyl chloride had already been identified as a special analyte of concern, a risk-driver, and this is discussed in the approved project documents, then vinyl chloride cannot exceed the LCS control limits, even marginally. In the more common situation where special analytes of concern have not been identified (e.g., investigation phase of work), the concept of marginal exceedances can be applied when evaluating LCSs for all target analytes. TestAmerica has requested written verification from USACE, confirming that we understand this concept correctly.

2. Acceptable Corrective Action for Noncompliant CCV – Section 3.5.5.10.e & Grey Box 59
   Sect. 3.5.5.10.3: If the continuing calibration verification results obtained are outside established acceptance produce [sic] a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive calibration verifications, or a new initial instrument calibration must be performed.
   Grey Box 59 Adds: If initial corrective action attempts fail and the CCV results are still outside established acceptance criteria, and the laboratory chooses to demonstrate the success of routine corrective action through the use of two consecutive CCVs, then the concentration of the two CCVs must be at two different levels within the original calibration curve.
   Clarification: TestAmerica Denver does not employ the additional attempt to verify calibration as described in Grey Box 59. Instead, the laboratory makes only one attempt to pass consecutive CCVs at the same concentration. If this attempt fails, the laboratory would take appropriate corrective actions and recalibrate.

3. Calibration Verification Control Limits for Organics - Section 5.5.10, Grey Box 58, Tables B-2 & B-3
   Grey Box 58: For DoD, the percent drift/percent difference of the CCV standard shall be less than 15% of the initial calibration for organic methods....
   Table B-2: The acceptance criterion for GC and HPLC methods is given as "All analytes within + 20% of expected value from the ICAL...."
   Table B-3: The acceptance criterion for GC/MS methods is < 20% D for CCC compounds.

Clarification: The statement in Section 5, Grey Box 58, contradicts the more detailed QC requirements in the tables of Appendix DOD-B. It appears that when the tables were changed, Grey Box 58 was not revised to match the tables. We understand this oversight to be an editorial error and that the acceptance criteria are those given in the tables.

4. Reporting Dilutions for Organic Methods – Appendix DOD-A, Item 4, 16th bullet
QSM 3 Requirement: If the lab analyzes multiple dilutions of a sample, the dilution, less diluted results, and the neat analytical results must all be reported.
Clarification: TestAmerica Denver often uses screening techniques to determine appropriate dilution levels. Screening can include visual observation of the samples or sample extracts in instances where high concentrations of organic compounds are evident (e.g., non-aqueous liquid phase). Static headspace, GC/FID, is used to screen all samples for volatile organic analysis.
   a) If the dilution is overestimated by the screening technique (i.e., peak/signal is too low), the lab will conduct additional analyses to produce lower detection limits.
   b) The laboratory may not be able to analyze less dilute samples if significant levels of non-target and/or target compounds are present at concentrations high enough to prevent further dilutions.
   c) In order to protect our instruments form failure and contamination, initial analysis of sample dilutions based on screening data will be used to document any samples containing concentrations high enough to prevent further dilutions.
   d) Screening data will be included in the data package.
TestAmerica Denver will make every attempt to minimize dilutions and to analyze extracts without dilution if it can be done without damaging the analytical equipment.

5. Common Laboratory Contaminants – Section D.1.1.1, d, Grey Box D-5
QSM 3 Requirement: If the method blank contamination exceeds one-half the reporting limit, then the laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria. The concentrations of common laboratory contaminants shall not exceed the reporting limit.
Clarification: The common laboratory contaminants for TestAmerica Denver comprise the following:

Dissolved Gases (RSK-175):	methane
Volatile Organics (8260 & 8021):	acetone, 2-butanone, carbon disulfide, and methylene chloride
GC/MS Semivolatiles (8270):	bis-2-ethylhexylphthalate, di-n-octyl phthalate
Metals (6010 & 6020):	Ca, Fe, K, Mg, Na, Si, Zn
(6020 only):	Sn

6. RPD Calculation – Section D.1.1.3.1.d, Grey Box D-13
   QSM 3 Requirement: For DoD, RPD must be calculated as a comparison of measured concentrations.
   Clarification: For most DoD projects, TestAmerica Denver's analytical reports are produced by a CLP-style report generator that does calculate the RPD as defined. However, some clients prefer the presentation on the reports generated directly from our LIMS, and if aliquots for the unspiked sample and/or replicate spikes (e.g., MS/MSD) are different then the RPD calculation will produce a different RRD value because it is based on recovered amounts, rather than recovered concentrations.

7. Analysis of Volatile Organics & GRO in Soil
   Clarification: TestAmerica Denver's GC section can only perform the methanol extraction, high-level option, of the SW-846 Method 5035. The reporting limits supplied during project setup reflect this fact. TestAmerica Denver's GC/MS section is able to perform both the high-level and the low-level options of Method 5035.

8. Analysis TOC in Soil
   Clarification: TestAmerica Denver is not currently supporting this method. Projects requiring this analysis would include arrangements for subcontracting with an approved laboratory. The laboratory is purchasing a new TOC soil instrument, and this analytical capability will be made available after instrument qualification.

variances for afcee qapp 4.0

TestAmerica Denver has reviewed the AFCEE QAPP, Version 4.0, and has identified requirements that TestAmerica Denver either cannot meet as written or that need further clarification. TestAmerica Denver has documented variances and clarifications in the document titled "TestAmerica Denver Technical Approach to AFCEE QAPP Version 4.0." The current document is saved to TestAmerica Denver's Public Outlook folders, and an example document is as follows:

TestAmerica Denver
Technical Approach to AFCEE QAPP Version 4.0

TestAmerica Denver has reviewed the AFCEE QAPP, Version 4.0. TestAmerica Denver understands that the AFCEE QAPP, Version 4.0 may be a guidance document to be supplemented by a project-specific QAPP. As of this writing, TestAmerica Denver has not received a project-specific QAPP. We propose the following clarifications to and variances from the AFCEE QAPP, Version 4.0.

Technical Requirements

Method Blank Evaluation Guidance – Section 8.2.1.6
   Requirement: For method blanks, the source of contamination shall be investigated and measures taken to correct, minimize, or eliminate the problem if the

concentration exceeds one-half the RL. (Use the RL for common laboratory contaminants.)

Proposed: Based on TestAmerica Denver's long-term method blank results, we propose the following compounds as the list of common laboratory contaminants:

Dissolved Gases (RSK175) – methane
Volatile Organics (8260B) – acetone, 2-butanone, methylene chloride
GC/MS Semivolatiles (8270C) – bis-2-ethylhexylphthalate, di-n-octyl phthalate
Metals (6010B, 6020) – Ca, Fe, K, Mg, Na, Si, Zn
Metals (6020 only) - Sn

Justification: AFCEE allows consideration of common lab contaminants in method blanks. The intent of this list is to specify which compounds will be evaluated as common laboratory contaminants.

Data Qualifier Flags for Method Blanks – Table 8.2.2.4-2, and Section 7 Tables

Requirement: Flag results for analytes detected > ½ RL using a B flag qualifier.

Proposed: Flag results for analytes detected > MDL and take corrective action when the blank results exceed ½ RL.

Justification: This request results from a limitation of our LIMS system. There will be no impact on data quality, as the laboratory will "over flag" data.

Soil Gases in Water by RSK-175 - Table 7.2.1.10-1

Requirement: A second-source calibration verification must be analyzed once after each ICAL.

Proposed: TestAmerica Denver proposes to use a different lot from the same vendor (Scott Specialty Gases), rather than a true second-source standard.

Justification: Section 4.5.4 of the AFCEE QAPP states that, "When a project requires analyses for which there is not a separate vendor source available, the use of different lot numbers from the same vendor will be acceptable to verify calibration."

CCV Acceptance for Method 8260B – Table 7.2.2.4-1

Requirement: In addition to percent difference (%D) criteria for CCCs, all analytes must be within + 20% D of expected value from ICAL.

Proposed: Allow + 35% D for the following compounds that are known poor performers (reference USEPA National Functional Guidelines for Organic Data Review) - acetone, 2-butanone, carbon disulfide, chloromethane,2-hexanone, methylene chloride, 4-methyl-2-pentanone, 1,2-dibromo-3-chloropropane, and naphthalene.

Justification: TestAmerica Denver's control charts for these compounds demonstrate that there will be a high frequency of failures (and resulting reanalyses) for these compounds without the requested variance.

CCV Acceptance for Extractable Organics – Tables 7.2.2.4-1, 7.2.5.3-1, and 7.2.1.10-1

Requirement: Table 7.2.2.4-1: In addition to percent difference (%D) criteria for CCCs, all analytes must be within + 20% D of expected value from ICAL.
Tables 7.2.5.3-1 and 7.2.1.10-1: All analytes must be within + 20% D of expected value from ICAL.

Proposed: Allow + 30% D for the following compounds that are known poor performers (reference DoD QSM): 4-nitrophenol (8270C), benzoic acid (8270C), phenol

(8270C), phenol-d5 (8270C), 3,3'-dichlorobenzidine (8270C), 4-chloroaniline (8270C), dinoseb (8151A), tetryl (8330)

Justification: TestAmerica Denver's control charts for these compounds demonstrate that there will be a high frequency of failures (and resulting reanalyses) for these compounds without the requested variance.

IS Compounds for PAHs by 8270-SIM – Table 7.2.3.1-2

Requirement: Use at least 2 of the following IS compounds: anthracene-d10, benzo(a)anthracene-d12, or perylene-d12.

Proposed: TestAmerica Denver currently uses the following 3 IS compounds: acenaphthene-d10, phenanthrene-d10, and chrysene-d12.

Justification: TestAmerica Denver uses deuterated analogues of three target compounds that elute throughout the chromatogram, which follows the intent of the QAPP. We need to procure new standard mixes, establish performance characteristic, and revise our SOP before we incorporate the IS named in the QAPP.

RT Limits for LC/MS 8321A Methods - Table 7.2.6.4-1

Requirement: Relative retention time (RRT) for each analyte must be within + 0.06 RRT units of the ICAL (within + 0.02 RRT units for perchlorate)

Proposed: Retention time window width will be 3 times the standard deviation for each analyte, based on the retention time from a 72-hour study.

Justification: The proposed criterion is taken directly from the SW846 HPLC methods. The HPLC used for Method 8321A is the same as the HPLC used for SW846 Methods 8310 and 8330. There is no reason that the HPLC would experience tighter RRTs simply because a different detector is used after separation.

CCV Acceptance for 8321A Explosives & 8321A Herbicides – Table 7.2.6.4-1

Requirement: All analytes must be within + 20% D of expected value from ICAL.

Proposed: Allow + 30% D for these two methods.

Justification: TestAmerica Denver's CCV control data for these compounds demonstrate that there will be a high frequency of failures (and resulting reanalyses) for these compounds without the requested variance.

Reporting Limits (RLs) – Tables in Section 7

Requirement: RLs for selected compounds are given in the table below.

Proposed: TestAmerica Denver proposes higher RLs for selected compounds, as shown in the table below.

Justification: TestAmerica Denver proposes higher RLs for selected compounds because: (1) our method detection limit (MDL) is greater than one half the AFCEE RL; and/or (2) the calibration linearity requirements are not achieved at the AFCEE RL (i.e., results are not quantitatively reliable at that level).

Compound, Matrix	Method	TAL Denver MDL	AFCEE RL	TAL Denver Proposed RL
Methylene Chloride, water (ug/L)	SW8260B	0.2	1.0	2.0
Toxaphene, water (ug/L)	SW8081A	0.4	1.0	2.0
Benzene, water (ug/L)	SW8021B	0.3	0.2	0.6
MCPA, water (ug/L)	SW8151A	150	100	300

Compound, Matrix	Method	TAL Denver MDL	AFCEE RL	TAL Denver Proposed RL
Arsenic, soil (mg/kg)	SW6020	0.02	0.3	0.5
Selenium, water (mg/L)	SW6020	0.0003	0.002	0.005
soil (mg/kg)		0.03	0.2	0.5
Iron, soil (mg/kg)	SW6010B	3.8	3.0	10

Spike Acceptance Limits

Requirement: AFCEE spike acceptance limits for selected compounds are given in the table below.

Proposed: TestAmerica Denver proposes wider spike acceptance limits for selected compounds, as shown in the table below.

Justification: TestAmerica Denver's proposed acceptance limits are based on three standard deviation values from the mean recovery. These limits are calculated from intralaboratory historical data. Exceptions are requested only in those cases where historical data demonstrate that there will be a high frequency of random failures for these compounds without the requested variance.

Analyte	AFCEE Water Accuracy Limit (%R)	TAL Denver Proposed Water Accuracy Limit (%R)	AFCEE Soil Accuracy Limit (%R)	TAL Denver Proposed Soil Accuracy Limit (%R)
Method SW8015B Table 7.2.1.2-2, page 7-15				
Jet Fuel (JP4, JP8, or JetA)	61-143	30-102	---	---
Method SW8151A Table 7.2.1.8-2, page 7-27				
Dichloroprop	68-122	46-122	72-142	47-142
Dinoseb	28-115	20-115	20-131	10-131

Reporting Requirements

Holding Time Compliance -- Section 4.5.1

Requirement: Holding times are determined on the basis of days, hours, minutes. If the time of the sample collection is not provided, the laboratory must assume the most conservative (i.e., the earliest time of day).

Proposed: TestAmerica Denver proposes to determine holding times as given in the promulgated method, which is given as days or hours.

Justification: TestAmerica Denver's proposal is compliant with the promulgated method. TestAmerica Denver's LIMS cannot track holding times to the minute.

Laboratory Data Reporting Requirements -- Section 8.2.1.1

Requirement: Soil samples shall have results reported on a dry weight basis. There are two options:

a) A wet weight aliquot of sample equivalent to the method specified dry weight aliquot should be taken for analysis. Alternatively,
b) the lab may choose to use a consistent wet weight aliquot that is expected to be large enough to compensate for the moisture in the sample and use this as a consistent weight. RLs are project specific requirements and are NOT adjusted for sample moisture.

Proposed: Testamerica Denver proposes to weigh the routine amount of sample or to weigh an additional 10%-20% for all samples. Reporting limits will be elevated accordingly, to compensate for percent moisture and the additional amount of sample.

Justification: Similar to option b above, TestAmerica Denver's LIMS has been programmed to adjust sample concentration, RLs, and MDLs for percent moisture. The extra 10-20% of sample is to ensure that reported RLs are < project RLs. If there are particularly wet samples to be collected, e.g., sediments with free liquid, we would need to discuss how to aliquot and analyze the samples in any event.

AFCEE Forms -- Section 8.8

Requirement: AFCEE forms shall be included in the project QAPP and used unless a variance is requested and approved in advance and the forms included in the project QAPP, to be used by the contractor, can be verified to contain at a minimum the information requested on the AFCEE forms.

Proposed: TestAmerica Denver proposes to substitute its Level IV report and CLP-like forms.

Justification: TestAmerica Denver's LIMS cannot produce AFCEE QAPP 4.0 forms. Our Level IV report and CLP-like forms include all of the information contained in the AFCEE forms. We propose an equivalent format that provides all essential information required to perform external data validation.

Attachment 1
Flagging Protocols for LIMS

Program	Enter In Quantims	Remaps to	R02 footnote	Use For
DOD QSM version 3 and 4.1	AF	J	Analyte was detected, but result is estimated due to a QC failure	Surrogate Failures, MS/MSD failures.
	J	J	Estimated result, Result is less than RL.	Automatically applied by Quantims
	COL/CHI	J	Agreement between results are not within +/- 40%	Use for >40% RPD between columns
	E	J	Estimated Result. Result concentration exceeds the calibration range.	Use for result over range. Remap to J, some clients still like E even though the QAPP says J. In that case, you must use "O".
	O	E	Estimated Result. Result concentration exceeds the calibration range.	Use for result over range. Remap to E. Use this flag for those clients that still like to see the "E" qualifier.
	B	B	Analyte was detected in the associated method blank.	Automatically applied by Quantims
	Q	Q	One or more QC criteria failed	Use for LCS, CCV, and internal standard failures
AFCEE 3.1	AF	J	Analyte was detected, but result is estimated due to a QC failure	Surrogate failures, LCS failures
	M	M	Matrix spike recovery is outside control limits.	Matrix spike failures, internal standard failures
	R	R	The data is rejected	Internal standard failures, missed HT, CCV failures, surrogate <10%
	E	J	Estimated Result. Result concentration exceeds the calibration range.	Use for result over range. Remap to J, some clients still like E even though the QAPP says J. In that case, you must use "O".
	O	E	Estimated Result. Result concentration exceeds the calibration range.	Use for result over range. Remap to E. Use this flag for those clients that still like to see the "E" qualifier.
	B	B	Analyte was detected in the associated method blank.	Automatically applied by Quantims
	J	F	Estimated result, Result is less than RL.	Automatically applied by Quantims
AFCEE 4.0	AF	J	Analyte was detected, but result is estimated due to a QC failure	Surrogate failures, LCS failures
	M	M	Matrix spike recovery is outside control limits.	Matrix spike failures, internal standard failures
	R	R	The data is rejected.	Internal standard failures, missed HT, CCV failures, surrogate <10%
	E	E,J	Estimated Result. Result concentration exceeds the calibration range.	Use for result over range. May remap to J or E, some clients still like E even though the QAPP says J.
	B	B	Analyte was detected in the associated method blank.	Automatically applied by Quantims
	J	F	Estimated result, Result is less than RL.	Automatically applied by Quantims

Attachment 2
Associated Surrogates and Internal Standards for 8270C SIM

Analyte	Associated IS	Associated Surrogate
Acenaphthene	1	1,2
Acenaphthylene	1	1,2
Anthracene	2	3
Benzo(a)anthracene	3	3
Benzo(a)pyrene	3	3
Benzo(b)fluoranthene	3	3
Benzo(k)fluoranthene	3	3
Benzo(g,h,i)perylene	3	3
Chrysene	3	3
Dibenzo(a,h)anthracene	3	3
Fluoranthene	2	3
Fluorene	1	1,2
Indeno(1,2,3-c,d)pyrene	3	3
Naphthalene	1	1,2
Phenanthrene	2	3
Pyrene	2	3
Surrogates		
Nitrobenzene-d5	1	1
2-Fluorobiphenyl	1	2
Terphenyl-d14	2	3
Internal Standards		
Acenaphthene-d10	1	-
Phenanthrene-d10	2	-
Chrysene-d12	3	-
*In the event that a compound not covered by this table is requested the associated internal standard/surrogate will be used as required by the method.		

Attachment 3
Associated Surrogates and Internal Standards – 8270

Analyte	Associated IS	Associated Surrogate
1,2,4-Trichlorobenzene	2	4
1,2-DCB	1	4
1,3-DCB	1	4
1,4-DCB	1	4
2,4-DNT	3	2
2,6-DNT	3	2
2-Chloronaphthalene	3	2
2-Methylnaphthalene	2	2
2-Nitroaniline	3	2
3,3'-Dichlorobenzidine	5	2
3-Nitroaniline	3	2
4-Bromophenyl phenyl ether	4	2
4-Chloroaniline	2	4
4-Chlorophenyl phenyl ether	3	2
4-Nitroaniline	3	2
Acenaphthene	3	6
Acenaphthylene	3	6
Anthracene	4	6
Benzo(a)anthracene	5	6
Benzo(a)pyrene	6	6
Benzo(b)fluoranthene	6	6
Benzo(g,h,i)perylene	6	6
Benzo(k)fluoranthene	6	6
Benzyl alcohol	1	4
Bis(2-chloroethoxy)methane	2	4
Bis(2-chloroethyl)ether	1	4
Bis(2-chloroisopropyl) ether	1	4
Bis(2-ethylhexyl)phthalate	5	2
Butyl benzyl phthalate	5	2
Chrysene	5	6
Di-n-butyl phthalate	4	2
Di-n-octyl phthalate	5	2
Dibenzo(a,h)anthracene	6	2
Dibenzofuran	3	2
Diethyl phthalate	3	2
Dimethyl phthalate	3	2
Fluoranthene	4	6
Fluorene	3	6
Hexachlorobenzene	4	2
Hexachlorobutadiene	2	4
Hexachloroethane	1	4
Indeno(1,2,3-c,d)pyrene	5	6
Isophorone	2	4
N-nitrosodi-n-propylamine	1	4

N-Nitrosodiphenylamine	4	2
Naphthalene	2	6
Nitrobenzene	2	4
Phenanthrene	4	6
Pyrene	5	6
2,4,5-Trichlorophenol	3	1
2,4,6-Trichlorophenol	3	1
Analyte	Associated IS	Associated Surrogate
2,4-Dichlorophenol	2	3
2,4-Dimethylphenol	2	3
2,4-Dinitrophenol	3	1
2-Chlorophenol	1	3
2-Methylphenol	1	3
2-Nitrophenol	2	3
4,6-Dinitro-2-methylphenol	4	1
4-Chloro-3-methylphenol	2	1
4-Methylphenol	1	3
4-Nitrophenol	3	1
Benzoic Acid	2	3
Pentachlorophenol	4	1
Phenol	1	5
Surrogates		
2,4,6-Tribromophenol	3	1
2-Fluorobiphenyl	3	2
2-Fluorophenol	1	3
Nitrobenzene-d5	2	4
Phenol-d5	1	5
Terphenyl-d14	5	6
Internal Standards		
1,4-Dichlorobenzene-d4	-	-
Naphthalene-d8	-	-
Acenaphthene-d10	-	-
Phenanthrene-d10	-	-
Chrysene-d12	-	-
Perylene-d12	-	-

*In the event that a compound not covered by this table is requested the associated internal standard/surrogate will be used as required by the method.

Attachment 4
Associated Surrogates and Internal Standards for 8260B

Analyte	Associated IS	Associated Surrogate
1,1,1,2-Tetrachloroethane	2	2
1,1,1-TCA	1	1
1,1,2,2-Tetrachloroethane	3	3
1,1,2-TCA	1	4
1,1-DCA	1	1
1,1-DCE	1	1
1,1-Dichloropropene	1	4
1,2,3-Trichlorobenzene	3	3
1,2,3-Trichloropropane	3	3
1,2,4-Trichlorobenzene	3	3
1,2,4-Trimethylbenzene	3	3
1,2-DCA	1	4
1,2-DCB	3	3
1,2-Dibromo-3-chloropropane	3	3
1,2-Dichloropropane	1	4
1,2-EDB	2	2
1,3,5-Trimethylbenzene	3	3
1,3-DCB	3	3
1,3-Dichloropropane	2	2
1,4-DCB	3	3
1-Chlorohexane	2	2
2,2-Dichloropropane	1	1
2-Chlorotoluene	3	3
4-Chlorotoluene	3	3
Acetone	1	1
Benzene	1	4
Bromobenzene	3	3
Bromochloromethane	1	1
Bromodichloromethane	1	4
Bromoform	2	2
Bromomethane	1	1
Carbon Tetrachloride	1	4
Chlorobenzene	2	2
Chloroethane	1	1
Chloroform	1	1
Chloromethane	1	1
cis-1,2-DCE	1	1
cis-1,3-Dichloropropene	1	4
Dibromochloromethane	2	2
Dibromomethane	1	4
Dichlorodifluoromethane	1	1
Ethylbenzene	2	2
Hexachlorobutadiene	3	3
Isopropylbenzene	3	3

m,p-Xylene	2	2
Methylene Chloride	1	1
Methyl t-butyl ether (MtBE)	1	1
MEK (2-Butanone)	1	1
MIBK (methyl isobutyl ketone)	1	4
n-butylbenzene	3	3
n-Propylbenzene	3	3
Analyte	Associated IS	Associated Surrogate
Naphthalene	3	3
o-Xylene	2	2
p-Isopropyltoluene	3	3
sec-Butylbenzene	3	3
Styrene	2	2
TCE	1	4
tert-butylbenzene	3	3
Tetrachloroethene	2	2
Toluene	1	4
trans-1,2-DCE	1	1
trans-1,3-Dichloropropene	1	4
Trichlorofluoromethane	1	1
Vinyl Chloride	1	1
Surrogates		
Dibromofluoromethane	1	1
Toluene-d8	2	2
4-Bromofluorobenzene	3	3
1,2-DCA-d4	1	4
Internal Standards		
Fluorobenzene	1	-
Chlorobenzene-d5	2	-
1,4-Dichlorobenzene-d4	3	-
*In the event that a compound not covered by this table is requested the associated internal standard/surrogate will be used as required by the method.		

Attachment 5 – Target Analyte Lists for DoD Version 4.1 Projects
Volatile Organics by SW846 Method 8260

Volatile Organic Compound	CAS #	Comments
Acetone	67-64-1	
Benzene	71-43-2	
Bromobenzene	108-86-1	
Bromochloromethane	74-97-5	
Bromodichloromethane	75-27-4	
Bromoform	75-25-2	
Bromomethane	74-83-9	
2-Butanone (MEK)	78-93-3	
n-Butylbenzene	104-51-8	
Sec-Butylbenzene	135-98-8	
Tert-butylbenzene	98-06-6	
Carbon Disulfide	75-15-0	
Carbon Tetrachloride	56-23-5	
Chlorobenzene	108-90-7	
Chlorodibromomethane	124-48-1	
Chloroethane	75-00-3	
Chloroform	67-66-3	
Chloromethane	74-87-3	
2-Chlorotoluene	95-49-8	
4-Chlorotoluene	106-43-4	
1,2-Dibromo-3-chloropropane	96-12-8	
1,2-Dibromoethane (EDB)	106-93-4	
Dibromomethane	74-95-3	
1,2-Dichlorobenzene	95-50-1	See also 8270
1,3-Dichlorobenzene	541-73-1	See also 8270
1,4-Dichlorobenzene	106-46-7	See also 8270
Dichlorodifluoromethane	75-71-8	
1,1-Dichloroethane	75-34-3	
1,2-Dichloroethane	107-06-2	
1,1-Dichloroethene	75-35-4	
Cis-1,2-Dichloroethene	156-59-2	
Trans-1,2-Dichloroethene	156-60-5	
1,2-Dichloropropane	78-87-5	
1,3-Dichloropropane	142-28-9	
2,2-Dichloroproane	594-20-7	
1,1-Dichloropropene	563-58-6	
Cis-1,3-Dichloropropene	10061-01-5	
Trans-1,3-Dichloropropene	10061-02-6	
Ethylbenzene	100-41-4	
2-Hexanone	591-78-6	
Hexachlorobutadiene	87-68-3	
Isopropylbenzene	98-82-8	
p-Isopropyltoluene	99-87-6	
Methylene Chloride	75-09-2	
4-Methyl-2-pentanone (MIBK)	108-10-1	
Methyl tert-butyl ether (MTBE)	1634-04-4	
Naphthalene	91-20-3	See also 8270 and 8310
n-Propylbenzene	103-65-1	
Styrene	100-42-5	
1,1,1,2-Tetrachloroethane	630-20-6	
1,1,2,2-Tetrachloroethane	79-34-5	
Tetrachloroethene	127-18-4	
Toluene	108-88-3	
1,2,3-Trichlorobenzene	87-61-6	
1,2,4-Trichlorobenzene	120-82-1	
1,1,1-Trichloroethane	71-55-6	

1,1,2-Trichloroethane	79-00-5	
Trichloroethene	79-01-6	
Trichlorofluoromethane	75-69-4	
1,2,3-Trichloropropane	96-18-4	
1,2,4-Trimethylbenzene	95-63-6	
1,3,5-Trimethylbenzene	108-67-8	
Vinyl chloride	75-01-4	
o-Xylene	96-47-6	
m,p-Xylene	108-38-3/ 106-42-3	
Xylene (total)[1]	1330-20-7	
4-Bromofluorobenzene	460-00-4	Surrogate
Dibromofluoromethane	1868-53-7	Surrogate
1,2-Dichloroethane-d4	17060-07-0	Surrogate
Toluene-d8	2037-26-05	Surrogate

[1] Data may be reported on a project specific basis as Total Xylene, however, for the purpose of the DoD QSM, it will be analyzed and reported as m,p-xylene and o-xylene.

Attachment 5 – Target Analyte Lists for DoD Version 4.1 Projects
Semi-Volatile Organics by SW846 Method 8270

Semi-Volatile Organic Compound	CAS#	Comments
Acenaphthene	83-32-9	See also 8310
Acenaphthylene	208-96-8	See also 8310
Anthracene	120-12-7	See also 8310
Benzidine	92-87-5	
Benzoic acid [2,3]	65-85-0	
Benz[a]anthracene	56-55-3	See also 8310
Benzo[b]fluoranthene	205-99-2	See also 8310
Benzo[k]fluoranthene	207-08-9	See also 8310
Benzo[ghi]perylene	191-24-2	See also 8310
Benzo[a]pyrene	50-32-8	See also 8310
Benzyl alcohol	100-51-6	
Bis(2-chloroethoxy)methane	111-91-1	
Bis(2-chloroethyl)ether	111-44-4	
Bis(2-chloroisopropyl)ether	108-60-1	
Bis(2-ethylhexyl)phthalate	117-81-7	
4-Bromophenyl phenyl ether	101-55-3	
Butyl benzyl phthalate	85-68-7	
Carbazole	86-74-8	
4-Chloroaniline	106-47-8	
4-Chloro-3-methylphenol	59-50-7	
2-Chloronapthalene	91-58-7	
2-Chlorophenol	95-57-8	
4-Chlorophenyl phenyl ether	7005-72-3	
Chrysene	218-01-9	See also 8310
Dibenz[ah]anthracene	53-70-3	See also 8310
Dibenzofuran	132-64-9	
Di-n-butylphthalate	84-74-2	
1,2-Dichlorobenzene	95-50-1	See also 8260
1,3-Dichlorobenzene	541-73-1	See also 8260
1,4-Dichlorobenzene	106-46-7	See also 8260
3,3'-Dichlorobenzidine [2]	91-94-1	
2,4-Dichlorophenol	120-83-2	
2,6-Dichlorophenol	87-65-0	
Diethyl phthalate	84-66-2	
2,4-Dimethylphenol	105-67-9	
Dimethyl phthalate	131-11-3	
4,6-Dinitro-2-methylphenol	534-52-1	
2,4-Dinitrophenol	51-28-5	
2,4-Dinitrotoluene	121-14-2	
2,6-Dinitrotoluene	606-20-2	
1,2-Diphenylhydrazine	122-66-7	
Di-n-octylphthalate	117-84-0	
Fluoranthene	206-44-0	See also 8310
Fluorene	86-73-7	See also 8310
Hexachlorobenzene	118-74-1	
Hexachlorobutadiene	87-68-3	
Hexachloroethane	67-72-1	
Indeno[123-cd]pyrene	193-39-5	See also 8310
Isophorone	78-59-1	
2-Methylnaphthalene	91-57-6	
2-Methylphenol	95-48-7	
3-Methylphenol/4-Methylphenol	108-39-4/ 106-44-5	
Naphthalene	91-20-3	See also 8260, 8310
2-Nitroaniline	88-74-4	
3-Nitroaniline	99-09-2	

4-Nitroaniline	100-01-6	
Nitrobenzene	98-95-3	See also 8330
2-Nitrophenol	88-75-5	
4-Nitrophenol [3]	100-02-7	
N-nitroso-dimethylamine	62-75-9	
N-nitrosodiphenylamine	86-30-6	
N-nitrosodi-n-propylamine	621-64-7	
N-nitrosopyrrolidine	930-55-2	
Pentachlorophenol	87-86-5	
Phenanthrene	85-01-8	See also 8310
Phenol [3]	108-95-2	
Pyrene	129-00-0	See also 8310
1,2,4-Trichlorobenzene	120-82-1	See also 8260
2,4,5-Trichlorophenol	95-95-4	
2,4,6-Trichlorophenol	88-06-2	
2-Fluorophenol	367-12-4	Surrogate
Phenol-d5 [3]	13127-88-3	Surrogate
Nitrobenzene-d5	4165-60-0	Surrogate
2-Fluorobiphenyl	321-60-8	Surrogate
2,4,6-Tribromophenol	118-79-6	Surrogate
Terphenyl-d14	1718-51-0	Surrogate

[2] Poor performing analyte for the solid matrix. Must be in the calibration standard but data indicate it may not consistently produce quantitative data.

[3] Poor performing analyte for the water matrix. Must be in the calibration standard but data indicate it may not consistently produce quantitative data.

Attachment 5 – Target Analyte Lists for DoD Version 4.1 Projects
Organophosphorus Pesticides by SW846 Method 8141

Organophosphorus Pesticide	CAS#	Comments
Azinphos-methyl	86-50-0	
Bolstar (Sulprofos)	35400-43-2	
Chlorpyrifos	2921-88-2	
Coumaphos	56-72-4	
Demeton-o	298-03-3	
Demeton-s	126-75-0	
Diazinon	333-41-5	
Dichlorvos (DDVP)	62-73-7	
Disulfoton	298-04-4	
Ethoprop	13194-48-4	
Fensulfothion	115-90-2	
Fenthion	55-38-9	
Merphos	150-50-5	
Naled	300-76-5	
Parathion, methyl	298-00-0	
Phorate	298-02-2	
Ronnel	299-84-3	
Stirophos (Tetrachlorvinphos)	961-11-5	
Tokuthion (Protothiofos)	34643-46-4	
Trichlornate	327-98-0	
Chlormefos	24934-91-6	Surrogate
Triphenylphosphate	115-86-6	Surrogate

Chlorinated Herbicides by SW846 Method 8151

Chlorinated Herbicide	CAS#	Comments
2,4-D	94-75-7	
2,4-DB	94-82-6	
2,4,5-TP	93-72-1	
2,4,5-T	93-76-5	
Dalapon	75-99-0	
Dicamba	1918-00-9	
Dichlorprop	120-36-5	
Dinoseb	88-85-7	
MCPA	94-74-6	
MCPP	93-65-2	
2,4-Dichlorophenylacetic acid (DCAA)	19719-28-9	Surrogate

Attachment 5 – Target Analyte Lists for DoD Version 4.1 Projects
Polynuclear Aromatic Hydrocarbons by SW846 Method 8310 and 8270 SIM

Polynuclear Aromatic Compound	CAS#	Comment
Acenaphthene	83-32-9	
Acenaphthylene	208-96-8	
Anthracene	120-12-7	
Benz[a]anthracene	56-55-3	
Benzo[a]pyrene	50-32-8	
Benzo[b]fluoranthene	205-99-2	
Benzo[k]fluoranthene	207-08-9	
Benzo[ghi]perylene	191-24-2	
Chrysene	218-01-9	
Dibenzo[ah]anthracene	53-70-3	
Fluoranthene	206-44-0	
Fluorene	86-73-7	
Indeno[123-cd]pyrene	193-39-5	
Naphthalene	91-20-3	
Phenanthrene	85-01-8	
Pyrene	129-00-0	
Decafluorobiphenyl	434-90-2	Surrogate 8310
2-Fluorobiphenyl	321-60-8	Surrogate 8270
Nitrobenzene-d5	4165-60-0	Surrogate 8270
Terphenyl-d14	1718-51-0	Surrogate 8270

Explosives by SW846 8330 and 8321

Explosive Compound	CAS#	Comment
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	
Hexahydro-1,3,5-trinitor-1,3,5-triazine (RDX)	121-82-4	
1,3,5-Trinitrobenzene	99-35-4	
1,3-Dinitrobenzene	99-65-0	
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	479-45-8	
Nitrobenzene	98-95-3	See also 8270
2,4,6-Trinitrotoluene (TNT)	118-96-7	
4-amino-2,6-dinitrotoluene	19406-51-0	
2-amino-4,6-dinitrotoluene	35572-78-2	
2,4-Dinitrotoluene	121-14-2	See also 8270
2,6-Dinitrotoluene	606-20-2	See also 8270
2-Nitrotoluene	88-72-2	
3-Nitrotoluene	99-08-1	
4-Nitrotoluene	99-99-0	
Pentaerithrityl tetranitrate (PETN)	78-11-5	
Nitroglycerin	9010-02-0	
3,5-Dinitroaniline	618-87-1	
1,2-Dinitrobenzene	528-29-0	Surrogate

Attachment 5 – Target Analyte Lists for DoD Version 4.1 Projects
Organochlorine Pesticides by SW846 Method 8081

Organochlorine Pesticide	CAS#	Comment
Aldrin	309-00-2	
alpha-BHC	319-84-6	
beta-BHC	319-85-7	
Delta-BHC	319-86-8	
Gamma-BHC	58-89-9	
Alpha-Chlordane	5103-71-9	
Gamma-Chlordane	5103-74-2	
4,4'-DDD	72-54-8	
4,4'-DDE	72-55-9	
4,4'-DDT	50-29-3	
Dieldrin	60-57-1	
Endosulfan I	959-98-8	
Endosulfan II	33213-65-9	
Endosulfan sulfate	1031-07-8	
Endrin	72-20-8	
Endrin aldehyde	7421-93-4	
Endrin ketone	53494-70-5	
Heptachlor	76-44-8	
Heptachlor epoxide	1024-57-3	
Hexachlorobenzene	118-74-1	See also 8270
Methoxychlor	72-43-5	
Toxaphene	8001-35-2	
Tetracholoro-m-xylene	877-09-8	
Decachlorobiphenyl	2051-24-3	

Polychlorinated Biphenyls by SW846 Method 8082

PCB Compound	CAS#	Comment
Aroclor 1016	12674-11-2	
Aroclor 1221	11104-28-2	
Aroclor 1232	11141-16-5	
Aroclor 1242	53469-21-9	
Aroclor 1248	12672-29-6	
Aroclor 1254	11097-69-1	
Aroclor 1260	11096-82-5	
Aroclor 1262	37324-23-5	
Aroclor 1268	11100-14-4	
Tetrachloro-m-xylene	877-09-8	Surrogate
Decachlorobiphenyl	2051-24-3	Surrogate

Attachment 5 – Target Analyte Lists for DoD Version 4.1 Projects
Metals by ICP, ICPMS, and CVAA
(based on SW846 methods 6010, 6020, and 7000 series)

Metal	CAS#	Comment
Aluminum	7429-90-5	6010
Antimony	7440-36-0	6010/6020
Arsenic	7440-38-2	6010/6020
Barium	7440-39-3	6010/6020
Beryllium	7440-41-7	6010/6020
Cadmium	7440-43-9	6010/6020
Calcium	7440-70-2	6010
Chromium	7440-47-3	6010/6020
Cobalt	7440-48-4	6010/6020
Copper	7440-50-8	6010/6020
Iron	7439-89-6	6010
Lead	7439-92-1	6010/6020
Magnesium	7439-95-4	6010
Manganese	7439-96-5	6010/6020
Mercury	7439-97-6	7470/7471
Molybdenum	7439-98-7	6010/6020
Nickel	7440-02-0	6010/6020
Potassium	7440-09-7	6010
Selenium	7782-49-2	6010/6020
Silver	7440-22-4	6010/6020
Sodium	7440-23-5	6010
Thallium	7440-28-0	6010/6020
Tin	7440-31-5	6020
Uranium	7440-61-1	6020
Vanadium	7440-62-2	6010/6020
Zinc	7440-66-6	6010/6020

Other Inorganics Target Analyte List
(based on SW846 7000 series, 9010, 9012, and 9056)

Inorganic Compound	CAS#	Comment
Bromide	24959-67-9	9056
Chloride	16887-00-6	9056
Chromium, hexavalent	18540-29-9	7196
Cyanide	57-12-5	9010/9012/9014
Fluoride	16984-48-8	9056
Nitrate	14797-55-8	9056
Nitrite	14979-65-0	9056
Phosphate	14265-44-2	9056
Sulfate	14808-79-8	9056

ATTACHMENT B

LABORATORY QUALITY ASSURANCE MANUAL

Title Page:

Quality Assurance Manual
Approval Signatures

Robert C Hanisch — 6/15/09

Laboratory Director – Robert C. Hanisch — Date

Karen Kuoppala — 06-15-09

Quality Manager – Karen Kuoppala — Date

Susan Decker — 6/15/09

Organic Operations Manager/Technical Director – Susan Decker — Date

Richard Clinkscales — 6/15/09

Inorganic Operations Manager/Technical Director – Richard Clinkscales — Date

Cover Page:

Quality Assurance Manual

TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002
303-736-0100
303-431-7171

www.testamericainc.com

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Title Page:

Quality Assurance Manual
Approval Signatures

Laboratory Director – Robert C. Hanisch	Date
Quality Manager - Karen Kuoppala	Date
Organic Operations Manager/Technical Director – Susan Decker	Date
Inorganic Operations Manager/Technical Director – Richard Clinkscales	Date

SECTION 2

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

SOPs AND POLICIES REFERRED TO IN THE QA MANUAL

SOP/Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-003	Management of Change Procedure
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-005	Calibration Curves (General)
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-L-P-001	Record Retention
CW-F-P-002	Authorization Matrix
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-004	Controlled Purchases Policy

SECTION 3

INTRODUCTION
(NELAC 5.1 - 5.3)

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Denver'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. Each TestAmerica 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 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (1999). In addition, the policies and procedures outlined in this manual are compliant with the various accreditation and certification programs listed in Appendix 6. The relevant NELAC section is included in the heading of each QAM section.

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.
- EPA SW-846, *Test Methods for the Evaluation of Solid Waste, 3rd Edition,* September 1986; Update I, July 1992; Update II, September 1994; and Update III, December 1996, Update IV, January 2008.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th and 21st Edition.
- *U.S. Department of Energy*, *Quality Systems for Analytical Services*, Revision 2.4, October 2008.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 4.1, April 2009.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan(QAPP),* Version 4.0.02, May 2006.
- Nuclear Regulatory Commission (NRC) quality assurance requirements.
- 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 TestAmerica Denver 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 5 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

TestAmerica Denver analyzes thousands of environmental and industrial samples every month. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge 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 process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses 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 water, industrial waste, and soil 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 requirements. All methods performed by TestAmerica Denver 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 manual is reviewed annually by the QA Manager and laboratory personnel to assure that it reflects current practices and meets the requirements of TestAmerica Denver's clients and regulators. 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. The updates will be reviewed by the QA Manager, Laboratory Director/Manager, Technical Director(s), relevant operational staff and Corporate Quality Assurance (if a change is made to the Corporate template) and then incorporated into the document in periodic updates. The QAM is based on a Corporate QAM Template that is prepared and reviewed annually by the Corporate Quality Department. Necessary changes are coordinated by the Corporate Quality Department and distributed electronically to each laboratory for inclusion in the laboratory specific QA Manuals.

Laboratory-specific QAM changes are approved and documented through the Management of Change process (Refer to SOP No. DV-QA-028P, Management of Change Procedure).

3.4.2 Control

This manual is considered confidential within TestAmerica and may not be altered in any manner by other than a duly appointed representative from TestAmerica. If the document has been provided to external users or regulators, it is for the exclusive purpose of reviewing TestAmerica Denver's quality systems and shall not be used in any other way without the written permission of an appointed representative of TestAmerica. The procedure for control of distribution is incorporated by reference to TestAmerica Denver policy DV-QA-001P, "Preparation and Management of Standard Operating Procedures (SOPs) and Other Controlled Documents".

The order of precedence in the event of a conflict between policies is outlined in Section 5.3 of this Quality Assurance Manual.

Figure 3-1.

Example - Format for a QA/QC Policy Memorandum

Corporate (or Laboratory) QA/QC Policy Memorandum # _____

Effective Date: _____ Expiration Date: When Appropriate QAM Section is Revised

Corporate: *(Only needed for Corporate Memorandum – Delete if Laboratory)*

COO - West	Date	Vice-President, QA and EHS	Date
COO - East	Date		

Local:

Organic Operations Manager Approval Technical Director	Date	Quality Assurance Approval	Date
Laboratory Director Approval	Date	Inorganic Operations Manager Approval Technical Director	Date

1. **Purpose**

2. **Procedure**

3. **Attachments**

4. **References/Cross References**

SECTION 4

ORGANIZATION AND MANAGEMENT
(NELAC 5.4.1)

4.1 OVERVIEW

TestAmerica Denver is part of a national network of laboratories known as TestAmerica. This Quality Assurance Manual (QAM) is applicable to the TestAmerica Denver laboratory only.

TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002
Federal ID# CO0026

The Corporate organization chart can be found in Figure 4-1 and the laboratory's organization chart can be found in Appendix 2. The locations of other TestAmerica labs are as follows:

TestAmerica Anchorage
TestAmerica Austin
TestAmerica Bangkok, Thailand
TestAmerica Buffalo
TestAmerica Burlington
TestAmerica Cedar Falls
TestAmerica Chicago
TestAmerica Connecticut
TestAmerica Corpus Christi
TestAmerica Dayton
TestAmerica Edison
TestAmerica Honolulu
TestAmerica Houston
TestAmerica Irvine
TestAmerica King of Prussia
TestAmerica Knoxville
TestAmerica Los Angeles
TestAmerica Mobile
TestAmerica Nashville
TestAmerica North Canton
TestAmerica Ontario
TestAmerica Pensacola
TestAmerica Phoenix
TestAmerica Pittsburgh
TestAmerica Portland
TestAmerica Richland
TestAmerica San Francisco
TestAmerica Savannah
TestAmerica Seattle
TestAmerica Spokane

TestAmerica St. Louis
TestAmerica Tacoma
TestAmerica Tallahassee
TestAmerica Tampa
TestAmerica Valparaiso
TestAmerica Watertown
TestAmerica West Sacramento
TestAmerica Westfield

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 define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management.

4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of TestAmerica Denver. All employees have access to the QAM and are responsible for knowing the content of this manual and 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.

4.2.2 President/Chief Executive Officer (CEO)

The President/CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the Chairman/CEO, the President/CEO establishes the overall quality standard and data integrity program for the Analytical Division, providing the necessary leadership and resources to assure that the standard and integrity program are met.

4.2.3 Chief Operating Officer (COO)

The COO serves as the ranking executive for all respective analytical laboratory operational functions and reports to the President/CEO of the Analytical Division. The COO is responsible for the daily management of all analytical laboratories, long-term planning and development of technical policies and management plans. The COO ensures the attainment of corporate objectives through the selection, development, motivation, and evaluation of top management personnel. The COO approves all operating budgets and capital expenditures. The COO signs-off on the final QAM template that contains company policies for implementing the Quality Program.

4.2.4 General Manager (GM)

Each GM reports directly to the COO. Each GM has full responsibility for the overall administrative and operational management of their respective laboratories. The GM's responsibilities include allocation of personnel and resources, long-term planning, setting goals, and achieving the financial, business, and quality objectives of TestAmerica. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews. The GM is 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 Client and Technical Services

The Vice President (VP) of Client and Technical Services reports directly to the President/CEO and is responsible for offerings to clients including quality assurance, environmental health and safety, risk management, technical assistance, legal compliance and contract administration. The VP of Client and Technical Services provides support and direction to the Executive Director and Directors of these areas, and supports the COO in decisions regarding long term planning, resource allocation and capital expenditures.

4.2.6 Vice President of Client and Technical Services

The Vice President (VP) of Client and Technical Services who manages the Quality Assurance and Environmental, Health and Safety Programs, reports directly to the CEO. With the aid of the other Senior Management Team members, Laboratory Directors/Managers, Quality Directors, EHS Director, QA Managers and EHS Coordinators, the VP of Client and Technical Services has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and Environmental, Health and Safety Program within TestAmerica. Additional responsibilities include:

- Review of QA/QC aspects of Corporate Documents, national projects and expansions or changes in services.
- Maintenance of data investigation records that are reported to Corporate Management.
- Working with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation, along with the Quality Directors, of a monthly report that includes quality metrics across the Analytical Division and a summary of any quality related initiatives and issues.
- With the assistance of other Corporate Senior Management Team members and the EHS Director, development and implementation of the TestAmerica Environmental, Health and Safety Program.

The VP of Client and Technical Services is also responsible for offerings to clients including risk management, technical assistance, legal compliance, and contract administration. The VP of Client and Technical Services provides support and direction to the Managers of these areas, and supports decisions regarding long term planning, resource allocation and capital expenditures.

4.2.7 Quality Directors (Corporate)

The Quality Directors report to the VP of Client and Technical Services. Together with the VP, the Quality Directors have the responsibility for the establishment, general overview and maintenance of the Laboratory's Quality Assurance Program within TestAmerica. The Quality Directors are responsible for:

- Coordination/preparation of the Corporate QM Template that is used by each laboratory to prepare its own laboratory-specific QAM.

- Maintenance of data investigation records that are reported to Corporate Management.
- Oversight of the QA/QC programs within each laboratory. This includes a final review of each laboratory-specific QAM and receipt of each laboratory's QA monthly report.
- Review of QA/QC aspects of national projects.
- Assistance with certification activities.

4.2.8 Ethics and Compliance Officers (ECOs)

TestAmerica has designated a senior member of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – VP of Client and Technical Services. The ECO is involved when data investigations occur. The ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECO ensures 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 ECO monitors and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the CEO, COO, Laboratory Director/Manager 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 ECO 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.

4.2.9 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.

4.2.10 Environmental Health and Safety Directors (EHSDs) (Corporate)

The EHSD reports directly to the VP of Client and Technical Services. The EHSD is 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.

· Development and execution of the company Environmental Health and Safety Internal Audit program.

· 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.

4.2.11 Laboratory Director

TestAmerica Denver's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. 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:

- Provides one or more technical directors for the appropriate fields of testing. The name(s) of the Technical Director will be included in the national database. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing. The role of the Technical Director at TestAmeria Denver is fulfilled by the Laboratory Director or appointed designee(s).
- Ensures 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.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.

- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures 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.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Director(s), and the Operations Manager as direct reports.

4.2.12 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025.

- The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:
- 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.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate 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 are temporarily suspended following the procedures outlined in Section 13.
- Monitoring standards of performance in quality control and quality assurance.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Reviews training for effectiveness and implements changes as needed.

4.2.13 Quality Assurance Specialist

The Quality Assurance Specialist performs several roles. The QA Specialist reports to the facility QA Manager. The QA Specialist is responsible for QA documentation and involvement in the following activities:

- Assist 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.
- Facilitate 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.
- Assist the QA Manager in the preparation of new SOP's and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- Manages the performance testing (PT) studies, coordinates follow up studies for failed analytes and works with QA Manager and Laboratory Staff to complete needed corrective action reports.
- Personnel training records review and maintenance.
- Document control maintenance.

- Assists the Quality 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.
- Manages certifications and accreditations.
- Monitors for compliance the following QA Metrics: Temperature Monitoring of refrigeration units and incubators; thermometer calibrations; balance calibrations; eppendorf/pipette calibrations; and proper standard/reagent storage.
- Periodic checks on the proper use and review of instrument logs.
- Initiate the Mint-miner data file review process for organic instrumentation. Maintain tracking sheet of activity.
- Initiate the annual Instrument review.
- Assist in the technical review of data packages which require QA review.

4.2.14 Quality Assurance Assistant

The Quality Assurance Assistant performs several roles. The QA Assistant reports to the facility QA Manager. The QA Assistant is responsible for QA documentation and involvement in the following activities:

- Assist 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.
- Serves as a project manager for proficiency testing samples and other QC samples. Processes and reports QC samples as routine samples to appropriate agencies.
- Assist the QA Manager in maintaining the laboratory's reference data to keep it current and accurate.
- Prepares certification applications for states as directed by QA Manager.
- Personnel training records review and maintenance.
- Document control maintenance.
- Assisting departments in generating MDL spreadsheets and calculations, reviewing MDL studies submitted to QA.
- Assisting in contol limit gneration.
- Ensuring maintenance of records archives.
- Maintaining historical indicies foo all technical records including SOPs, QC records, laboratory data, etc.

4.2.15 Technical Director

The Technical Director(s) report(s) directly to the Laboratory Director. The role of the Technical Director at TestAmerica Denver is fulfilled by the Operations Managers or appointed designee(s). He/she is accountable for all analyses and analysts with respect to ISO 17025. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and second- and third-generation instrumentation. Specific responsibilities include, but are not limited to:

- 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 vs. 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.
- Captains department supervisors to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with supervisors and QA Manager.

4.2.16 LIMS Administrator

The LIMS Administrator reports to corporate IT. In the pursuit of his/her duties, he/she:

- Establishes and maintains the laboratory information system (LIMS) for tracking all samples in the laboratory.
- Updates and enhances LIMS.
- Develops expertise in the requirements described in Good Automated Laboratory Practices (GALP)-EPA 2185, 1995 Edition, in order to ensure compliance.
- Programs and tests software modifications/changes.
- Coordinates testing to ensure that all LIMS software accurately performs its intended functions. Testing is performed and documented after installation or when modifications/ changes are made.
- Maintains historical files of software, software operating procedures (manuals), software changes/modifications (Change Log) and software version numbers.
- Maintains log of repairs and service performed on LIMS hardware.
- Develops and verifies security practices to assure the integrity of LIMS data. Identifies threats, potential threats, and future threats.
- Maintains awareness of any environmental conditions of the facility housing the LIMS that may compromise LIMS raw data and informs management.
- LIMS database back-up once daily.

4.2.17 LAN Analyst

The LAN Analyst reports to the LIMS Administrator. Specific responsibilities include, but are not limited to:

- Working with corporate IT to solve problems and standardize laboratory IT equipment and processes
- Monitoring and supporting office automation so LAN is operational for internal and external communications
- Troubleshooting problems throughout the laboratory relating to computers, software, telephones, and other electronic equipment
- Managing software and hardware for all computer applications to give users legal and operational equipment to perform daily tasks
- Responsible for new user setup on network, LIMS, telephone, and voice mail
- Maintaining tape backups for multiple computer servers
- Providing after hour on-call support to keep network and PCs functioning properly
- Analyzing server log files for errors to look for potential problems with file servers
- Installing or upgrading computers and other equipment

4.2.18 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. He/She reports directly to the Laboratory Director. He/She acts as the Technical Director in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Director and QA Manager and in compliance with regulatory requirements.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

4.2.19 Radiation Safety Officer

The Radiation Safety Officer (RSO) is responsible for implementing TestAmerica Denver's radiation safety program. The RSO reports directly to the Technical Director. The RSO's duties consist of:

- Manage the personnel radiation dosimetry program
- Maintains the Radioactive Materials License and radionuclide inventory
- Monitors laboratory operation for compliance with the Radiation Safety Manual
- Training, documenting, and evaluating the TestAmerica Denver personnel for handling radioactive material
- Creating, releasing, and decontaminating of Radiological Control Areas (RCAs)
- Monitoring and tracking of radioactive materials
- Conducting the radioactive material waste disposal program in accordance with State and Federal regulations
- Maintaining all records related to the radiation safety program

4.2.20 Employee Health and Safety Coordinator

The EH&S Coordinator is responsible for administering the EH&S program that provides a safe, healthy working environment for all employees and the environment. The Employee Health and Safety Coordinator (EH&S Coordinator) reports directly to the Laboratory Director and the corporate Environmental Health and Safety Director. He/She monitors all areas for unsafe conditions, acts, and potential hazards. Specific responsibilities include, but are not limited to:

- Staying current with the hazardous waste regulations

- Continuing training on hazardous waste issues
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste
- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica’s medical consultants.

4.2.21 Hazardous Waste Specialist

The Hazardous Waste Specialist is responsible for coordinating and implementing the divisional hazardous waste program to ensure compliance with all federal, state, local laws, and company policies. The Hazardous waste specialist reports to the EH&S Coordinator. The duties consist of:

- Staying current with the hazardous waste regulations
- Conducts weekly inspections of satellite accumulation areas and all hazardous waste storage areas
- Operates and maintains on-site wastewater treatment system
- Coordinates the proper storage, packing and disposal of laboratory wastes according to Department of Transportation (DOT) and Resource Conservation and Recovery Act (RCRA) regulations

- Maintains waste disposal records
- Coordinates spill response activities including documentation for waste storage areas

4.2.22 Waste Disposal Technician

The Waste Disposal Technician is responsible for proper disposal of spent chemicals, process waste, and unused laboratory samples used in the laboratory according to corporate, federal, state, and local guidelines. The Waste Disposal Technician reports to the Hazardous Waste Specialist and EH&S Coordinator. The duties consist of:

- Packaging hazardous waste for transport per DOT, RCRA and TSCA guidelines
- Identifying waste streams and maintaining satellite accumulation areas
- Packages expired chemicals for shipment or disposal
- Tracks volume of waste generated for reporting to corporate and EPA
- Prepares and tracks implementation of the Waste Minimization Plan
- Empties satellite containers into bulk containers and returns to the laboratory for reuse

4.2.23 Department Manager

Department Managers report to the Operations Manager. At TestAmerica Denver there are two levels of Department Managers (I or II). The level designation is based on the level of experience. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training, development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.
- Encourage 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.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Director, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Director, Operations Manager, and/or Laboratory Director.

- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/She is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct 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.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.24 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The Analyst position at TestAmerica Denver is divided into levels. These levels range from Analyst I to Analyst V. The level designation is based on experience, expertise, and responsibilities. The responsibilities of the analysts are listed below:

- Perform analyses 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.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Director, and the QA Manager. These improvements, if approved, will be incorporated. 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.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.25 Laboratory Technician

Laboratory Technicians are responsible for the preparation of samples and performing all tasks assigned to them by the group leader or supervisor. The Laboratory Technician position at TestAmerica Denver is divided into three levels. These levels are Laboratory Technician I, Laboratory Technician II, and Laboratory Technician III. The level designation is based on experience, expertise, and responsibilities. The responsibilities of the Laboratory Technician are listed below:

- 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
- Report 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 Director, and/or the QA Manager or member of QA staff.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.26 Laboratory Assistant

The Laboratory Assistant position is an entry-level position to learn basic laboratory technician skills. The Laboratory Assistant reports to their group leader or supervisor. The Laboratory Assistants duties include the following:

- Assisting the Laboratory Technicians in preparation of samples for analysis
- Preparing routine forms and reports
- Collecting and preparing materials and supplies for the laboratory
- Assisting technicians in conducting routine analysis

4.2.27 Sample Control Manager

The Sample Control Manager reports to the Project Management Manager. The responsibilities are outlined below:

- Direct the logging of incoming samples into the LIMS
- Ensure the verification of data entry from login
- Provide daily assessments of sample receipts
- Monitor the preparation and shipment of bottle kits to clients

- Oversee the receipt, log in, and storage of samples
- Schedules couriers for sample pickup from customer sites

4.2.28 Sample Control Technician

The Sample Control Technician reports to the Sample Control Manager. The Sample Control Technician position at TestAmerica Denver is divided into levels. These levels range from Sample Control Technician I to Sample Control Technician IV. The level designation is based on experience and responsibilities of the Technician. The Sample Control Technician responsibilities include the following:

- Receive and unload samples or consignments in accordance with DOT regulations
- Verify samples against the Chain of Custody (COC)
- Log in sample into the LIMS to assign a lot number for tracking purposes and distribute the paperwork to the Project Managers and Department Managers
- Label samples with lot number assigned and deliver the samples to the appropriate labs for analysis daily
- Monitor freezer and cooler temperatures daily to confirm that the readings are within SOP guidelines
- Ship all subcontracted samples to designated lab in accordance with DOT regulations as needed

4.2.29 Shipping/Maintenance Technician

The Shipping/Maintenance Technician reports to the Sample Control Manager and the Project Management Manager. The Shipping/Maintenance Technician duties include the following:

- Maintaining the inventory control system
- Receiving and distributing incoming supplies
- Preparing and shipping bottle sampling kits to clients or on-site crews
- Maintaining bottle and cooler inventory
- Packing in-house samples for shipment to other laboratories

4.2.30 Courier

The Courier reports to the Sample Control Manager and the Project Management Manager. The Courier's duties include the following:

- Picking up and delivering samples and reports to clients and the laboratory
- Receiving and signing the chain of custody for samples
- Preparing and shipping bottle sampling kits to clients or on-site crews
- Performing preventative maintenance on company vehicles

4.2.31 Project Management Manager

The Project Management Manager reports to the Laboratory 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 functions of this position are outlined below:

- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.
- Responsible to ensure that clients receive the proper sampling supplies.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to 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.
- Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

4.2.32 Project Manager

The Project Managers report to the Project Management Manager and serve as liaisons between the laboratory and its clients. At TestAmerica Denver there are two levels of Project Managers (I or II). The level designation is based on experience, expertise, and responsibilities. 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

4.2.33 Project Management Assistant

The Project Management Assistant reports to the Project Management Manager and designated Project Manager. The Project Management Assistant assists the Project Manager in servicing the client's needs and communicating those needs to the laboratory. The Project Management Assistant's responsibilities include:

- Collating data reports, expanded deliverables, CLP data packages and electronic data deliverables (EDD's) for delivery to clients.
- Writing case narratives accompanying data packages to communicate anomalies to clients
- Entering data from subcontracted laboratories
- Proof reading and filing data reports received from the laboratory
- Assisting Project Managers in changing compound lists, TAT, and setting up tables in Word or Excel
- Monitoring report due dates for timely delivery
- Invoicing completed data packages
- Generating credit or debit invoices to ensure proper payment
- Copying and paginating reports

4.2.34 Support Supervisor

The Support Supervisor reports to the Laboratory Director and Project Management Manager. He/She is responsible for ensuring the timely and correct shipment of data reports to clients. He/She oversees the data review and data packaging groups. In addition, he/she:

- Coordinates work projects with project managers
- Supervises the review of data packages and authorizes its release
- Oversees the completion, mailing, and archiving of data reports

- Supervises the review of data packages for compliance with any Quality Assurance Program Plan (QAPP)

4.2.35 Data Review Analyst

The Data Review Analyst reports to the Support Supervisor. The Data Review Analyst is responsible for the reviewing of analytical data for contract compliance, completeness, and appropriate documentation. In addition, the Data Review Analyst performs the following:

- Reviews routine and non-routine data as recorded/produced by instrumentation
- Looks for discrepancies/inconsistencies with other project related results
- Assures contract compliance and compliance with client expectations have been met
- Checks data for compliance with the QAPP

4.2.36 Data Packaging Technician

The Data Packaging Technician reports to the Support Supervisor. The Data Review Analyst is responsible for preparing complete and accurate client report packages in accordance with contract compliance. Data Review Technicians perform the following duties:

- Compiling of data packages
- Paginating of data packages
- Creating hard copy deliverables
- Entering of data needed for final reports into the appropriate database
- Printing of final reports

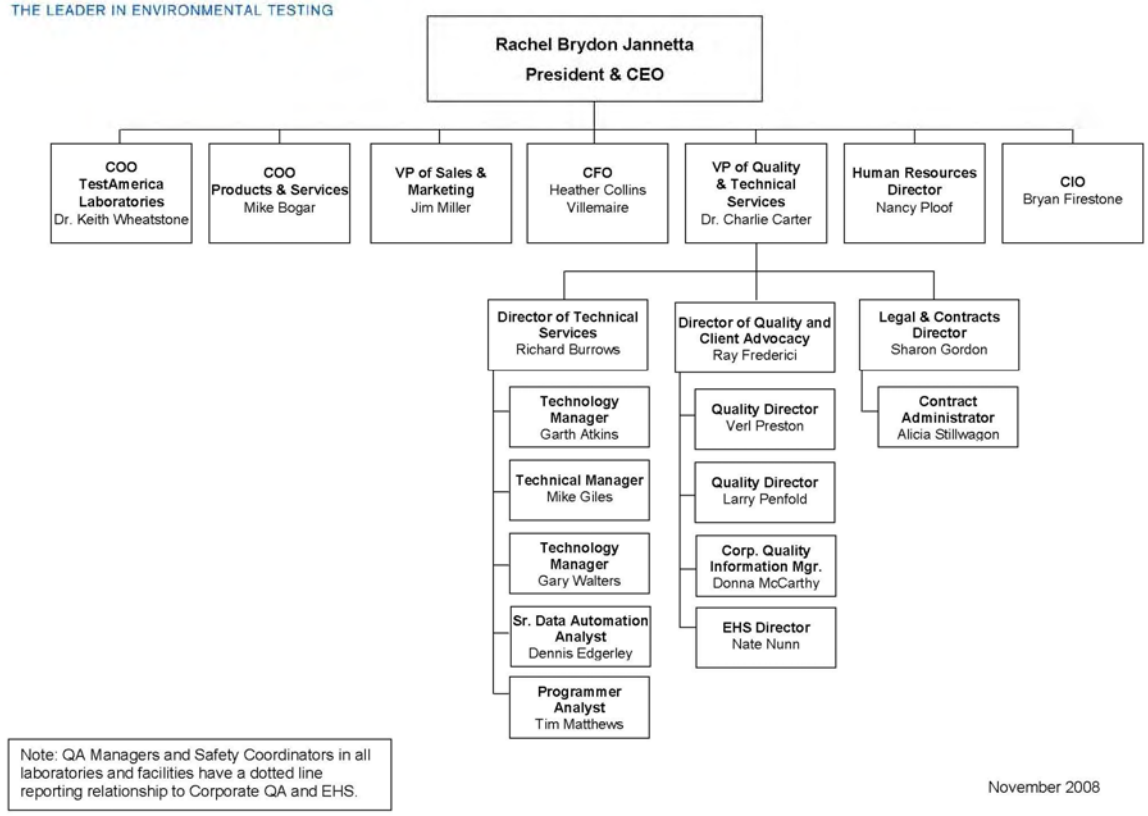
4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel Title	Key Personnel	Deputy
Laboratory Director	Robert C. Hanish	Brett VanDelinder
QA Manager	Karen Kuoppala	John Morris
Organic Operations Manager Organic Technical Director	Susan Decker	Richard Clinkscales
Inorganic Operations Manager Inorganic Technical Director	Richard Clinkscales	Susan Decker
Project Management Manager	Brett VanDelinder	Pat McEntee
Organic MS Manager	William Rhoades	Susan Decker
Organic GC Manager	Dennis Jonsrud	Susan Decker
Metals Manager	Doug Gomer	Richard Clinkscales
Wet Chemistry Manager	Dave Elkin	Richard Clinkscales
LCMS Manager	Andria Lenoble	Susan Decker
Support Supervisor	Bernice Parra	Beth Miller
EHS Coordinator	Adam Alban	Robert Fayard, Bret Roberts
Radiation Safety Officer	Andrew Meyer	Adam Alban

Figure 4-1.

Corporate Organization Chart



SECTION 5

QUALITY SYSTEM
(NELAC 5.4.2)

5.1 QUALITY POLICY STATEMENT

The management of TestAmerica and TestAmerica Denver are committed to providing data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols described in this manual.

In all aspects of the laboratory and business operations, management is dedicated in maintaining the highest ethical standards. An Ethics Policy sign-off can be viewed in Appendix 1. Training on ethical and legal responsibilities is provided annually and each employee signs off annually on the policy as a condition of employment.

It is TestAmerica's Policy to continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. The company recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.

TestAmerica Denver strives to provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at TestAmerica Denver 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 7 elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Policy No. CA-L-P-001) and employee ethics statements (Appendix 1).
- An Ethics and Compliance Officer (ECO).
- 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. (SOP No. CA-L-S-001)
- Procedures and guidance for recalling data if necessary (SOP No. CA-L-S-001).
- An effective external and internal monitoring system that includes procedures for internal audits (Section 16).

As an American Council of Independent Laboratories (ACIL) member, all TestAmerica laboratories adhere to the following ACIL Code of Ethics:

- 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 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 SUPPORTING DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents prepared by the laboratory and company management:

- Quality Assurance Manual (QAM) Template
- 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
- Corporate TestAmerica QA/QC Policy Memorandums (Refer to Section 3.4).
- Laboratory QA/QC Policy Memorandums (Refer to Section 3.4).

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- TestAmerica QA/QC Policy Memorandum - Corporate
- Laboratory QA/QC Policy Memorandum
- Quality Assurance Manual
- Corporate SOPs and Policies
- Laboratory SOPs and Policies

- Other (Work Instructions (WI), memos, flow charts, etc.)

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 characterize 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. The calculation of precision is described in Section 25.

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. The calculation of accuracy is described in Section 25.

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, and by the degree to which approval from the US EPA or other pertinent regulatory agencies is obtained for any procedure for which significant modifications have been made.

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), interelement 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 prepares a Reference Data Summary (aka. Browser Report) that summarizes the precision and accuracy acceptability limits for analyses performed at TestAmerica Denver. This summary is updated each time new limits are generated and is obtained with the use of the QC Browser software/program. The new and previous limits are listed in a table format along with the control chart data generated from TestAmerica Denver's TraQar Control Limits program. The limits, control charts, and any notations pertaining to the data are compiled into a package that contains the effective date. The control limit data package is then scanned and stored in the QA/Read/Control Limits folder on the L drive. 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, TestAmerica Denver has developed limits from evaluation of data from similar matrices. Criteria for the development of control limits are contained in Section 25.

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. TestAmerica Denver routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The control charting process is defined in detail in SOP DV-QA-003P. If a method defines the QC limits, the method limits are used. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Department 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 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 Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate limits are determined for a specific time period as defined in SOP DV-QA-003P. The resulting ranges are entered in LIMS.

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. Refer to SOP DV-QA-003P for a description of the control charting process and evaluation of trending.

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 17). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6

DOCUMENT CONTROL
(NELAC 5.4.3)

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 at each laboratory Facility:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

The Corporate staff posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These are collectively termed "Official Documents" and encompass the Policies and Procedures that all facilities are required to employ. These official documents are only considered controlled when they are read on the company 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 official documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory specific SOP DV-QA-0010, Document Control provides additional information for TestAmerica Denver 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. Discussion on records control is described in Section 15.

The maintenance of purchasing data is discussed in Section 9.

The maintenance of sales and marketing contracts is discussed in Section 7.

6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a control system for each document include a unique name and number, the number of pages of the item, the effective date, revision number and the

laboratory's name. The QA Manager or designee is responsible for the maintenance of the system and maintains the items in the QA Office.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a department manager submits an electronic or hardcopy 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 the official document on file. The official document is provided as needed to those using it. Controlled documents shall be available at all locations where the operational activity described in the document is performed (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 annually and revised as appropriate. Changes to documents occur when a procedural change warrants a revision of the document.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP No. DV-QA-001P. Requirements for TestAmerica corporate quality documents are described in Corporate SOP no. CW-Q-S-001. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department as described in SOP DV-QA-0005, Document Archiving Procedure. Electronic copies are stored on the Public server in the QA folder for the applicable revision under G:\QA\READ\SOPS\ESOPS\ALL.

For changes to SOPs, refer to SOP No. DV-QA-001P, Preparation and Management of Standard Operating Procedures (SOPs).

Forms, worksheets, work instructions, white papers, protocols, and information are organized by department and document type in the QA office. Electronic versions are kept on the Public server in the QA folder under G:\QA\READ\SOPS\Word Docs. The procedure for the care of these documents is in SOP DV-QA-001P.

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 as described in Section 15.

SECTION 7

REVIEW OF WORK REQUEST

7.1 OVERVIEW

TestAmerica 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 TestAmerica'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 TestAmerica'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 regulatory and client 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 lab'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 TestAmerica'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 review process 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 Customer Service Manager (CSM) is considered adequate. The CSM 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. 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 National Account Director, 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 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):

- Legal & Contracts Director
- General Manager
- The Laboratory Project Manager
- Customer Service Representative
- The Laboratory Operations Manager
- Laboratory and/or Corporate Technical Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- 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 National Account Director, Legal Contracts Director, or local account representative 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 Legal & Contracts Director maintains copies of all signed contracts. TestAmerica Denver's Customer Service Department maintains copies of all signed contracts 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. See Figure 7-3 for contract review forms.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory CSM and the Lab Director/Manager. Contracts filed by the CSM group are filed in locked fire proof cabinets.

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. The PM keeps a phone log of conversations with the client.

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, TestAmerica Denver assigns a PM to each client. The PM is the first point of contact for the 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. The bid document form in figure 7-3 is used to disseminate information from the CSM staff to the PM.

PM's are the direct 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 the 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. Unique or large programs generally have a Quality Assurance Summary prepared by the PM. This summary is posted on the outlook folders for anyone in the lab to access. The Quality Assurance Summary documents all requirements that are non-standard.

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 also communicated to the laboratory management during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department 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).

TestAmerica 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.

Figure 7-3.

Contract Review Requirements Checklist

CONTRACT NO.:_____ **DATE:**_____

	Exception Criteria	Comments
☐	The contract value is over $100K.	
☐	Payment terms are over 90 days, or payment terms requested indicate that TAL will be paid when the client is paid, with no maximum time limit.	
☐	A waiver of subrogation by TAL or our insurance company is required.	
☐	The warranty clause does not refer to TAL quality documents or the "standards of a competent professional in this industry."	
☐	Remedies for breach of warranty include resampling costs paid for by TAL.	
☐	The indemnification clause is very broad and can include liability for consequential damages.	
☐	There is a liquidated damages or penalty clause.	
☐	FAR flow down clauses impose cost accounting standards or defective pricing liability.	
☐	There is an organizational conflict of interest clause.	
☐	Insurance limits are over TAL's: a. General Liability - $2,000,000, Limits Requested _____ b. Automobile Liability - $1,000,000, Limits Requested _____ c. Workers Compensation–Other than statutory limit, Limits Requested _____ d. Employer's Liability - $1,000,000, Limits Requested _____ e. Professional/Pollution Liability - $5,000,000, Limits Requested _____ f. Umbrella Liability - $4,000,000, Limits Requested _____	

REVIEWER:_____

Figure 7-3.
Contract Summary Form

Prepared By:		**Contract No.:**		**Date:**	
This Summary is for:	(check one) ☒ **Completed contract** ☐ **Contract/proposal review due by:**				
	(check one) ☐ **Client contract** ☒ **Subcontract** ☐ **Teaming Agreement** ☐ **Vendor Contract**				
The estimated value of the Contract over its life ($000) is:		**Signed Original Contract Location:**		**Term of Agreement:**	
Contracting Party:					
Ultimate Client:					
Date of Contract:		**Project/Program Name/Location:**			
Responsible TAL Contacts:	**Sales:**	**PM/Technical:**	**Contract Reviewer(s):**		
Primary TAL Location(s):		**Secondary TAL Location(s):** (List All)			
Contracting Party Technical Contact:					
Address:					
Telephone:		**Fax:**			
Contracting Party Contracts Contact:					
Address:					
Telephone:		**Fax:**			
Type of Work: (check all that apply)	☐ **Lab Testing** ☐ **Consulting** ☐ **On-site Lab** ☐ **On-site Field Support** ☐ **Courier Service** ☐ **Includes work to be Subcontracted** **Work is** ☐ **Environmental or** ☐ **Not Environmental**				
Contract Type: (check all that apply)	☐ **MSA** ☐ **BOA** ☐ **Project-Specific** ☐ **Work Order under MSA or BOA** ☐ **Direct with Fed Gov't** ☐ **Fed Gov't Subcontract** ☐ **Direct State/Local Gov't** ☐ **State/Local Gov't Subcontract** ☐ **Commercial Client** ☐ **E/C Firm**				

Pricing:	☐ **Included** ☐ **Not Included**		
Standard TAL Term & Conditions?	☐ **Yes** ☐ **No**	Required Routine TAT:	☐ **EDD** **(# of Days)** _____ ☐ **Hard Copy** **(# of Days)** _____ ☐ **Business Days** ☐ **Calendar Days**
Reporting Formats Required:	☐ **Standard** ☐ **Standard + raw data** ☐ **Full CLP-Like** ☐ **Batch QC** ☐ **Project-specific QC**		
EDD Formats Required:	☐ **EDD** ☐ **CDROM** ☐ **iQ**		

Client Forecasts Required	☐ **Yes** ☐ **No**	**If yes, how much advance notice?**	
QAPP or Lab-standard Requirements?	☐ **QAPP** ☐ **Lab-Standard**	**Certification Required: (Describe)**	
Liquidated Damages or penalties?	☐ **Yes** ☐ **No**	**If Yes, Summarize:**	
Payment Terms		**Sample Disposal:**	☐ **Not stated or** ☐ **Must retain for Select One and/or** ☐ **Must get client approval for disposal**
Record Retention Requirement?	☐ **Yes** ☐ **No**	**If Yes, Summarize:**	
Special Invoicing Requirements?	☐ **Yes** ☐ **No**	**If Yes, Summarize:**	
How are Change Orders Handled?			
Other Special Requirements/ Comments/Notes: **TOPIC**	***COMMENTS***		

Figure 7-3.

Contract File

Company Name: _____

Site: _____

Contract Number _____

Contract Type
☐ C=Contract
☐ CO=Change Order
☐ DO=Delivery Order
☐ Mod=Modification
☐ MSA=Master Service Agreement
☐ PO=Purchase Order
☐ SC=Subcontract
☐ TO=Task Order
☐ WO=Work Order
☐ WR=Waiver/Release

Effective Date: _____

Expiration Date: _____

NTE Value: _____

Quote #: _____

Payment Terms: _____

Action	Completed	Date
Prepare Contract Summary	☐	
Legal Review	☐	
New Clients Only - Accounting Department Approval	☐	
TAL Execute Contract	☐	
Signed Contract to Client (Waiting on Executed Copy) ☐ **OR Signed Contract Received From Client** ☐	☐	
Fully Executed Contract Received from Client ☐ **OR Fully Executed Contract Returned to Client** ☐ ☐ **PDF-Email** ☐ **Original** ☐ **FAX**	☐	
Scan to Network (Fully Executed Contract)	☐	
Provide Contract Copy to Project Manager	☐	
Request Insurance Certificate	☐	

PM: _____

Final Lien Release Required ☐

Log Contract

TAL Denver Spread Sheet	☐
TAL Corporate Spread Sheet	☐
TAL Denver Signature Log	☐

Comments:

Figure 7-3. Con't

BID DOCUMENTATION FORM

ORIGINATOR:_____ DATE:_____

PROJECT NAME:_____ SITE:_____ _____

QUOTE NO.:_____/_____CLIENT CODE:_____

PRICING: ☐ CPL ☐ NATIONAL CONTRACT☐ LOCAL CONTRACT ☐ CPL MODIFIED
☐ EXISTING TA CONTRACT/PROJECT: TA LAB:_____

TA CONTRACT/PO NO.:_____

CREDIT CARD: ☐ AMEX ☐ Master Card☐ VISA
☐ Cardholder's name, account number, expiration date _____

CLIENT STATUS: ☐ Gold ☐ Gold Exception ☐ Standard ☐ Phase I ☐ Phase II

EMF FEE: ☐ Yes ☐ No

Minimum Log in Fee: ☐ Yes $___ ☐ No

CONTRACT/PO NO.:_____

PAYMENT TERMS: ☐ 30 Days ☐ 45 Days ☐ 60 Days ☐ 90 Days ☐ Other_____

PROJECT MANAGER ADD ALL METHODS/QUANTITIES FOR FORECAST ☐
PROJECT MANAGER MONITOR CONTRACT FOR NTE VALUE ☐ $_____k NTE, ___% THRESHOLD

PROJECT TYPE: ☐ Commercial ☐ State ☐ Federal:_____

QA/REGULATORY OVERSIGHT: ☐ EPA☐ USACE ☐ AFCEE ☐ DOE☐ STATE ☐ NONE

REGULATORY AREA:☐ RCRA/RFI/GW ☐ NPDES/CWA/WW ☐ SDWA/DW ☐ TSCA
☐ CERCLA
☐ drinking water compliance monitoring

CERTIFICATIONS/
APPROVALS: ☐ STATE_____☐ USACE*** ☐ AFCEE*** ☐ DoD QSM
Self Declaration
☐ NELAP☐ Other_____ ☐ DOE/Radioactive Materials License
☐ None
****USACE and AFCEE do not perform laboratory audits or issue laboratory certifications. TAL DEN does support these programs.*

TAL ACCOUNT EXECUTIVE:_____ TAL PROJECT MANAGER:_____

☐ AE Input Provided (See Attachment or Notes) ☐ No AE Input Received

CLIENT CONTACT:_____PHONE:_____

FAX:_____MOBILE:_____

E-MAIL:_____

COMPANY:_____

REPORT TO: _____

REPORT TO: _____

ORIGINAL INVOICE TO: _____
+ CoC

_____

DUPLICATE INVOICE TO: _____
+ CoC

_____

PROJECT
MANAGER: _____PHONE:_____

FAX: _____E-MAIL:_____

PROJECT
CHEMIST: _____PHONE:_____

FAX: _____E-MAIL:_____

FIELD
CONTACT: _____PHONE:_____

FAX: _____E-MAIL:_____

START DATE:_____DURATION:_____

PROJECT TESTS BY MATRIX

METHOD	WATER	SOIL	WASTE	BIOTA	AIR	COMMENTS

BOTTLE ORDER REQUIREMENTS:

SHIP TO: _____

_____

DELIVERY DUE DATE: _____

RUSH SHIPPING BILLABLE ☐ YES *(5 BUSINESS DAY'S NOTICE, MINIMUM)*
TWO WAY SHIPPING PAID -- PROVIDE FX RETURN LABELS ☐

COURIER SERVICE REQUESTED ☐ YES *(40mi. radius, $20 per trip)*

SAMPLING MATERIALS: ☐ COC Forms ☐ Labels ☐ Custody Seals ☐ USDA Permit
☐ PPQ Form 550 Stickers
☐ Quarantined Soil Stickers (DV-QA-0019, NY, MD, NC, SC, GA, FL, AI, MS, LA, AR, TX)
VOA Vials ☐ Preserved ☐ Unpreserved
☐ Trip Blanks ☐ Temperature Blanks

☐ Encore Samplers ☐ Terra Core Samplers
☐ 3 EnCore/sample, $30 ☐ 1 Terra Core kit/sample, $15
☐ EnCore T Handle (*no cost, rental*) ☐ percent moisture jar

SAMPLING FREQUENCY: ☐ Single Event ☐ Weekly ☐ Monthly ☐ Quarterly
☐ Semi-Annual ☐ Annual ☐ _____

RADIOACTIVITY: known radioactivity at site: ☐ NO ☐ Yes
☐ µCi levels ☐ mCi levels (if yes, contact RSO)
☐ prescreening required (always, if radioactivity suspected)

QAPP/SOW: ☐ AFCEE 3.1 ☐ AFCEE 4.0.02 ☐ USACE Shell ☐ DoD QSM V3
☐ TECQ TRRP ☐ Project/Client Specific (See Attachment) ☐ None

☐ MDL current
☐ need to request MDL from QA Department and Operations Manager
☐ MDLV required
☐ need to request MDLV from QA Department and Operations Manager

TAT REQUIREMENTS (BUSINESS DAYS): ☐ E-MAIL___ ☐ CD _____ ☐ HARDCOPY___ ☐ EDD___

SERVICE REQUIREMENTS: ☐ Email Sample Confirmation Report (R03 or S37)+ CoC + Sample Receiving Checklist
☐ Notify client of all nonconformances within 1 business day of occurrence

HARDCOPY DELIVERABLES: ☐ Standard (level II) ☐ CLP-Like Forms (level III) ☐ Raw Data (level IV)
☐ MI Summary Forms for All Organics Methods Other:_____

☐ MULTIPLE REPORTS ISSUED/REISSUED
☐ LEVEL IV HARDCOPY REPORT, $40 EACH
☐ LEVEL III HARDCOPY REPORT, $25 EACH

☐ Airbill ☐ Original Chain of Custody ☐ Sample Receiving Checklist
☐ Glossary of Terms, Qualifiers ☐ _____

EDDs: ☐ QUA 08 ☐ ERPIMS 4.0 ☐ None ☐ Client-Specific _____
☐ Specifications Attached

SACs: ☐ TAL DEN Standard, short spike list, standard data flags
(01)

☐ AFCEE 3.1 QAPP, AFCEE spike list, AFCEE flags
(9G)

☐ AFCEE 4.0.02 QAPP, AFCEE spike list, AFCEE flags (A4)
☐ Comprehensive spike list for organics, IDLs for metals, non-verified MDLs , standard data flags (9H)
☐ DoD QSM V3, comprehensive / full spike list for organics, verified MDLs,QSM data flags (Q3)
☐ Drinking Water Compliance, client requires notification for MCL exceedance (DW)
☐ Need New SAC? _____

PROJECT QC: ☐ Batch MS/MSDs ☐ Project-Specific MS/MSDs
☐ See CoC for client designation ☐ Lab designate MS/MSD
☐ MS/MSD billable at unit cost ☐ MS/MSD gratis
☐ report batch QC if available ☐ do not report batch QC

☐ LCS ☐ LCSD always ☐ LCSD if no MS/MSD available

☐ MS/MSD -- for AFCEE/QSM: Method SW8081A requires toxaphene and single component pesticides if these are target compounds

☐ LCS/MS/MSD Standard Spike List ☐ LCS/MS/MSD Full Spike List (Attached)

☐ DRO LCS/MS/MSD required ☐ MO/RRO LCS/MS/MSD required

☐ Standard QC Limits ☐ Project-Specific QC Limits (Attached)

☐ Field Blanks ☐ Field Duplicates ☐ Laboratory Duplicates

☐ Custom Calibration/Calibration Verification Requirements (Attached)

☐ Project-Specific QC Evaluation Criteria (Attached)

PROJECT PARAMETERS/
☐ Standard Method List (Attached) ☐ Project-Specific List (Attached)

GC/MS TICs needed? ☐ Yes ☐ No
VOC fraction: 10 / _____
SVOC fraction: 20 / ____

☐ Report Soil on Dry Weight Basis ☐ Report Soil on As-Received Basis
☐ Weigh out additional amount to compensate for dry weight correction (AFCEE)

☐ Report to MDL ☐ Report to RL

Multiple dilutions required to be analyzed and reported? ☐ Yes ☐ No
☐ Analyst select and report preferred value for each target compound, provide multiple Form Is with preferred values only, EDD will reflect preferred value for each target compound
(MUST CHOOSE THIS OPTION FOR SEDD 2A OR ADR EDD)
☐ Analyst report multiple values for each target compound, provide multiple Form Is with multiple values for every compound, EDD will reflect multiple values for each target compound
☐ ANALYTICAL DILUTION > 10X, EXTRACTED SAMPLE. 50% SURCHARGE, EACH SAMPLE.
☐ ANALYTICAL DILUTION > 10X, DAI/P&T SAMPLE. 70% SURCHARGE, EACH SAMPLE.

Metals digestion, water samples: ☐ Total ☐ Total Recoverable ☐ Potentially Dissolved

☐ Dissolved
☐ field filtered
☐ lab filter/preserve upon receipt

SPECIAL TECHNICAL REQUIREMENTS:

Method 8260B:	Acrolein, Acrylonitrile or 2-Chloroethyl vinyl ether required?	☐ Yes	☐ No	
	Unpreserved analysis required?	☐ Yes	☐ No	
	Client apprised of impact on results?	☐ Yes	☐ No	
	7-Day holding time specified in special instructions?	☐ Yes	☐ No	
Method 624	Acrolein, Acrylonitrile or 2-Chloroethyl vinyl ether required?	☐ Yes	☐ No	
	3-Day holding time specified in special instructions?	☐ Yes	☐ No	
Method 524.2:	Unpreserved analysis required?	☐ Yes	☐ No	
	Client apprised of 24 hour HT for unpreserved samples?	☐ Yes	☐ No	
DEN-WC-0048H (Hydrazines)	Client apprised of 48 hour HT for laboratory filtration and preservation of water samples?	☐ Yes	☐ No	
	Client apprised of requirement for unchlorinated water sample?	☐ Yes	☐ No	
	Client apprised of potentially elevated soil RLs due to required dilutions?	☐ Yes	☐ No	
TX1005	PM apprised of requirement to store soil samples at –12° C?	☐ Yes	☐ No	

PFOA/APFO
- ☐ Report target compound as PFOA (perfluorooctanoic acid)
- ☐ Report target compound as APFO (ammonium perfluorooctanoate)
- ☐ FOSA -- requires separate preparation and analysis -- for aqueous samples

BTEX/GRO
- ☐ analyze separately, using two SACs -- BTEX requires 2nd column confirmation; GRO quantified from gasoline standard
- ☐ analyze together, using "XU" SAC -- BTEX does not require 2nd column confirmation; GRO quantified from synthetic HC standard

SW5035 Sampling
- ☐ EnCore Sampler required
  - ☐ 48h HT to freezing or methanol preservation
  - ☐ 7d HT to freezing or methanol preservation
  - ☐ 14d HT to freezing or methanol preservation
- ☐ Terra Core Sampler required

- ☐ methanol preservation required (ML)
- ☐ sodium bisulfate preservation required (LL)
- ☐ DI water preservation required (LL)

Hexachlorophene by Method SW8270C -- Appendix IX analyses

- ☐ client advised that 40CFR Part 264 advises PQL = 10ug/L; TAL Denver's estimated PQL = 300ug/L
- ☐ client advised that TAL DEN analyzes a single-point standard at 1000ug/L, estimates a DL of 30-330ug/L, and has no MDL value for this compound (compound subject to non-reproducible performance)
- ☐ PM needs to include disclaimer in case narrative

Antimony -- Digestion of Soil Samples by Method SW3050B / SW6020

- ☐ client advised that alternate digestion procedure exists for antimony, which improves solubility and recovery of antimony from soil matrices (Section 7.5)
- ☐ client declined alternate digestion for antimony (Section 7.5)
- ☐ client requested alternate digestion procedure for antimony (Section 7.5)

Metals Analysis

- ☐ Beryllium by ICP/AES only
- ☐ "QO" method Code only for SW6010B
- ☐ "AS" method Code only for EPA200.7
- ☐ Cations by ICP/AES only
- ☐ New ICP/MS instrument is operated in collision cell mode. This instrument may not be used for drinking water compliance monitoring (per Method EPA 200.8). If samples are analyzed for drinking water compliance monitoring and Method EPA 200.8 is required, then include this text in Special Instructions: "EPA 200.8 Drinking Water -- collision cell instrument may ***not*** be used to analyze samples."

Fluoride by 340.2

- ☐ Client notified that the lab does not perform distillation – needed for wastewater
- ☐ If for wastewater compliance, EPA 300 is used, subcontract lab, or the client already has history of comparability for distillation vs. no distillation & ISE.

SW8330B / MIS Preparation *cf*. SW8330A / preparation

- ☐ SW8330B preparatory procedure: lay out contents of entire container (~1kg); air dry, at least overnight; remove rocks, vegetation; use mortar & pestle to break up clods, disaggregate soil; pass soil through 10 mesh sieve; use mechanical grinder (TBD); take ≥ 30 subsamples; create 10g aliquot
- ☐ SW8330B: Ring & puck grinding required prior to explosives analysis (e.g., samples collected from a firing point at a firing range)
- ☐ SW8330B: ball mill grinder required prior to explosives analysis (e.g., samples collected from an impact zone at a firing range)
- ☐ SW8330B: no mechanical grinding required prior to explosives analysis (e.g., samples collected from a depot or ammunition plant)

- ☐ SW8330A preparatory procedure: lay out contents of entire container (~50g); air dry, at least overnight; remove rocks, vegetation; use mortar & pestle to break up clods, disaggregate soil; pass soil through 30 mesh sieve; create 2g aliquot

Hybridized AFCEE / QSM

- ☐ additional target compounds -- QA, CSM, and OPS need to review and comment
- ☐ additional QA/QC criteria -- QA, CSM, and OPS need to review and comment
- ☐ CSM needs to submit variances/comments to Client, document agreement
- ☐ request new SAC

AFCEE / QSM SPECIAL INSTRUCTIONS

- ☐ mercury digestion procedure, soil samples: add glass beads

LC/MS/MS or IC/MS/MS Analyses

- ☐ Is client aware of high salt content in samples (≥1µM)? (Na+ and K+ cations cause ionization suppression, creation of salt adducts, salt build up on capillary thread and cone >>> instrument failure, inconsistent results)

SAMPLE/EXTRACT STORAGE AND WASTE DISPOSAL

☐ Lab refrigerate samples and extracts 30 / 60 / 90 / ___ days after invoice

☐ Lab dispose of samples and extracts 30 / 60 / 90 / ___ days after invoice

☐ Return samples and extracts 30 / 60 / 90 / ____ days after invoice

DATA RETENTION

☐ 5 / 7 / 10 years after invoice

PROJECT KICKOFF MEETING

☐ Need to schedule with departments: ☐ Organics ☐ Metals ☐ Wet Chemistry ☐ Reporting ☐ Log In ☐ QA

SUBCONTRACTED TESTS:

TEST	MINIMUM SAMPLE AMOUNT	SAMPLE CONTAINER/ PRESERVATIVE	UNIT COST ($)

SUBCONTRACT VENDOR: _____ _____

VENDOR POC: _____

PHONE:_____FAX:_____

ADDRESS: _____

_____

_____

SATURDAY DELIVERY: ☐ YES ☐ NO ☐ VENDOR ADDRESS ☐ HOLD AT CARRIER

VENDOR QUOTE NO.: _____ VENDOR QUOTE DATE:_____

VENDOR TAT (BUSINESS DAYS) _____ VENDOR DELIVERABLES:_____

ADDITIONAL INFORMATION:

SECTION 8

SUBCONTRACTING OF TESTS
(NELAC 5.4.5)

8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the corporate network. The phrase "work sharing" refers to internal transfers of samples between company 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 we must 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 the SOP on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

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 NELAC/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-NELAC accredited work where required. Refer to TestAmerica Denver's SOP DV-QA-0027 for laboratory specific procedures.

For DOD projects the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories are evaluated following the procedures outlined below and as seen in Figure 8-1. The subcontractor laboratory must receive written project-specific approval from the DoD client before any samples are analyzed.

The QSM has 5 specific requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.
2. Subcontractor laboratories must be approved by the specific DoD Component laboratory approval process.
3. Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of PT samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.
4. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
5. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

Project Managers (PMs), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing 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.

Note: TestAmerica Denver discloses, in all work proposals/contracts, the laboratories that could be used as a subcontract laboratory. In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work. It is required to have written approval from the client, whether it be email or in the contract itself, for all subcontract work.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM, Regional Account Executive (RAE), or Customer Service Manager (CSM) 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:

- The first priority is to attempt to place the work in a qualified network laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with the company (in JD Edwards): A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation for the requested tests prior to sending samples.
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC, A2LA, State and/or Federal accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All intra-company 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. Refer to SOP No. CA-C-S-001, Work Sharing Process.

When the potential sub-contract laboratory has not been previously approved, CSMs, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director/Manager. The Laboratory Director/Manager requests that the QA Manager begin the process of approving the subcontract 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).

8.2.1 The QA Manager must ensure that the Preliminary Evaluation Documentation Checklist (Figure 8-1) has been completed and have supporting documentation on file prior to initiation of any work. This does not apply to other TestAmerica facilities. A letter or e-mail is sent to the lab requesting the following information:

8.2.1.1 If a lab is NELAC or A2LA accredited,

8.2.1.1.1 Copy of necessary certifications verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accredit-able (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.

8.2.1.1.2 Insurance Certificate. This is required by TestAmerica's Chief Financial Officer

8.2.1.1.3 USDA soil permit if available**

8.2.1.2 For Laboratories accredited by other agencies with an auditing program:

8.2.1.2.1 Copy of necessary certifications verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accredit-able (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.

8.2.1.2.2 Insurance Certificate. This is required by TestAmerica's Chief Financial Officer

8.2.1.2.3 USDA soil permit if available**

8.2.1.2.4 Description of Ethics and Data Integrity Plan.

8.2.1.2.5 The most recent 2 sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action.

8.2.1.2.6 State Audit with Corrective Action Response

8.2.1.2.7 Example final report to confirm format is compliant and provides the necessary information. Minimally, it must be determined that Batch QC results are included in the laboratory reports and data is appropriately qualified.

8.2.1.2.8 A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review (it may need to be sent elsewhere for evaluation). This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously [minimum of 6 months] are grandfathered in.)

8.2.1.2.9 DoD work includes additional requirements as described in Section 8.1 above.

8.2.1.3 For laboratories performing tests that are unaccredited or accredited by an agency without an audit program:

8.2.1.3.1 A copy of their Quality Assurance Manual (controlled if possible). Ensure data quality limits for relevant methods are acceptable and that training procedures are adequate.

8.2.1.3.2 Copy of necessary certifications (if available) verifying that the required approvals are current. Ensure that all needed analytes are included; some may not be accredit-able (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.

8.2.1.3.3 Insurance Certificate. This is required by TestAmerica's Chief Financial Officer.

8.2.1.3.4 USDA soil permit if available**

8.2.1.3.5 Evidence of a current SOP per method. A copy of the first page and signature page of the SOP is acceptable. A table of contents including effective dates may also be acceptable. The SOP can be examined if an on-site audit is performed.

8.2.1.3.6 Description of Ethics and Data Integrity Plan.

8.2.1.3.7 The most recent 2 sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action.

8.2.1.3.8 Example final report to confirm format is compliant and provides the necessary information. (minimally, it must be determined that Batch QC results are included in the laboratory reports and data is appropriately qualified.

8.2.1.3.9 Statement of Qualification (SOQ) or summary list of Technical Staff and Qualifications – position, education and years of experience.

8.2.1.3.10 DoD work includes additional requirements as described in Section 8.1 above.

8.2.1.3.11 A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review (it may need to be sent elsewhere for evaluation). This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously [minimum of 6 months] are grandfathered in.)

8.2.2 Once the information is received by the QA Manager, it is evaluated for acceptability and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site along with the associate documentation and notify the finance group for JD Edwards.

**USDA permit is required if soils less than three feet deep from New York, North Carolina, South Carolina, Georgia, Florida, Tennessee, Alabama, Mississippi, Louisiana, Arkansas, Texas, Oklahoma, New Mexico, Arizona, California, Hawaii, or outside the continental U. S. are to be analyzed. These samples require special shipping measures; check with the EHS Department. It may be necessary to heat-treat the samples before shipping if the subcontract laboratory does not have a USDA permit; however, some analytes/tests may be irrelevant after heat treatment.

8.2.3 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. The company does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.4 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contract Department. Any problems identified will be brought to Corporate QA attention.

- 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 must be posted using the Vendor Performance Report (Form No. CW-F-WI-009)**.**
- Information must 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. The QA Manager will notify all network laboratories and Corporate QA 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 Lab Directors/Managers, QA Managers and Sales Directors.

8.3 OVERSIGHT AND REPORTING

The PM (or RAE or CSM) must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE or CSM) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented, with the initial setup of each project or annual basis, on a Verification of Subcontract Lab Status (Figure 8-2) and the form is retained in the project folder. For network laboratories, certifications can be viewed on the company website.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within the network.

The PM will communicate with the subcontracted laboratory to monitor the status of the analyses, facilitate successful execution of the work and ensure the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC 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 network work sharing laboratory may be transferred electronically and the results reported by the network 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 Laboratory Director/Manager may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, Corporate QA must be informed, and the QA Manager will be required to verify adequacy of proficiency scores and certifications. The laboratory must also request a copy of the raw data to support the analytical results for the first project submitted to the subcontract laboratory unless the laboratory has NELAC accreditation. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. The QA Manager will request full documentation and qualify the subcontractor under the provisions above. The approval process should be completed within 30 calendar days of subcontracting.

Figure 8-1.
Example - Preliminary Evaluation Documentation Checklist

Laboratory Under Evaluation:	
Client/Project For Which the Lab Will Be Subcontracted:	
Type of Analytical Services Required: ___Inorganic ___Radiochemistry ___General ___Organic _____Physical Testing _____Microbiological	**Type of Sample Matrices Required:** ___Drinking Water ___Waste Water ___Groundwater ___Mixed Waste ___Hazardous Waste

Item	**Yes**	**No**	**NA**	**Comments**
1. Which parameters will be subcontracted to this laboratory? List all:				
Did the subcontractor submit the following items and are they acceptable:				
2. Was a most recent audit, of requested parameters, performed by a state or federal agency, NELAP or other related third party audit submitted? Did the laboratory pass the state or the federal agency, NELAP, or other related third party audit?				
a. Was the Corrective action response sent to the state for federal agency? Was the laboratory corrective action response sufficient to address the problems found by the auditor?				
3. Were the two most recent PE samples for the requested parameters submitted?				
a. Did the PE samples pass criteria? If not, was the laboratory's corrective action response sufficiently explanatory?				
4. From the list of equipment submitted, does the auditor feel that sufficient equipment is available for performing the subcontracted analysis? Are equipment appropriate of the required test(s)?				
5. Was the laboratory QA manual submitted? Does the laboratory have a valid QA program and a QA manual?				
a. Are all subcontracted methods referenced in the QA manual?				

Laboratory Under Evaluation:	
Client/Project For Which the Lab Will Be Subcontracted:	
Type of Analytical Services Required: ___Inorganic ___Radiochemistry ___General ___Organic _____Physical Testing _____Microbiological	**Type of Sample Matrices Required:** ___Drinking Water ___Waste Water ___Groundwater ___Mixed Waste ___Hazardous Waste

Item	Yes	No	NA	Comments
b. Do reporting limits; referenced methods numbers; sample containers, preservations and holding times; summary of method calibrations; laboratory quality control samples/criteria; and preventive maintenance referenced in the QA manual. If not, list the missing key elements:				
6. Were MDLs and reporting limits (RLs) submitted? Are they acceptable? From the MDLs and RLs submitted, can the potential subcontractor routinely meet the required RLs for the listed parameters?				
7. Are required local state agency certifications for laboratory testing available, current, and acceptable?				
8. Does the laboratory use EPA approved standard methods? Does the laboratory have the necessary SOPs to perform the required analyses?				
9. Does the laboratory meet client/project-specific analytical and QA requirements?				
10. Was an example of a standard client sample data report for the above parameters submitted? Is it acceptable?				
11. From the documentation presented by the potential subcontractor, does the QA auditor reviewing the data feel that the subcontractor can be used? If response is no, explain why?				
12. Has the auditor discussed these reasons with the TestAmerica Denver laboratory management, that requested the laboratory, and are the concerns shared by TestAmerica Denver management?				
13. Does the auditor feel that an on-site laboratory audit of the potential subcontractor is required?				
a. Has a date and time been set for the on-site audit?				

Laboratory Under Evaluation:	
Client/Project For Which the Lab Will Be Subcontracted:	
Type of Analytical Services Required: ___Inorganic ___Radiochemistry ___General ___Organic _____Physical Testing _____Microbiological	**Type of Sample Matrices Required:** ___Drinking Water ___Waste Water ___Groundwater ___Mixed Waste ___Hazardous Waste

Item	**Yes**	**No**	**NA**	**Comments**
14. If radioactive materials involved, Radioactive Materials License and Radiation Protection Program.*				
*Any questions, contact the Corporate Health & Safety Director.				
Additional Comments:				
Prepared By:	**Date:**			
Reviewed By:	**Date:**			

Figure 8-2.
Example - Verification of Subcontract Lab Status.

TestAmerica Denver is responsible to our clients for on-going assurance that subcontracted analytical services meet TestAmerica Denver's expectations for quality. As part of this program, we require on-going verification that the following statements are true. Please return the completed form with the final report to TestAmerica Denver.

Laboratory Name: _____

	True	False	N/A	Comments
Your laboratory continues to hold current certifications as applicable to the requested fields of testing?				
Your laboratory has successfully completed PT samples for at least 2 of the last 3 of the requested fields of testing?				
Your laboratory has successfully completed method detection limits for the requested fields of testing within the last 12 months?				
There are no changes in equipment that affect the laboratory's capability to perform the requested fields of testing?				
There are no changes in qualified personnel that affect the laboratory's capability to perform the requested fields of testing?				
All testing is performed at the location to which the samples were delivered?				
Your laboratory does not have any OSHA, DOT, DoE, DoD, or EPA citations or pending investigations?				

Completed by: _____ on _____.
Name

SECTION 9

PURCHASING SERVICES AND SUPPLIES
(NELAC 5.4.6)

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 the specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Capital expenditures are made in accordance with the Controlled Purchases Procedure, CW-F-S-004. Only one quote is required where the item being purchased is a sole source product, Examples of sole source capital expenditures are laboratory test equipment, client specified purchases and building leases. A minimum of two quotes is required where the opportunity exists to source from more than one vendor. All documentation related to the purchase of capital items will be maintained in the individual CapEx files located in Corporate Purchasing. Data will be held in accordance with the record retention policy.

TestAmerica will enter into formal contracts with vendors when it is advantageous to do so. Contracts will be signed in accordance with the Authorization Matrix Policy, CW-F-P-002. Examples of items that are purchased through vendor contracts are laboratory instruments, consumables, copiers and office supplies. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. 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.

Non-capital expenditure items are purchased through the requisition and approval process in JD Edwards or through other TestAmerica authorized methods (approved web-sites, purchasing cards). Labs have the ability to select from the approved vendors in JD Edwards.

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

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents must meet with the requirements of the specific method and testing procedures for which they are

being purchased. Solvents and acids are pre-tested in accordance with Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001, *Verification and Storage of Chemical Standards*, SOP No. DV-QA-0015, and the TestAmerica Addendum to S-T-001, SOP No. S-T-001 DEN-1.

9.3.1 Purchasing

The nature of the analytical laboratory demands that all material used in any of the procedures is 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. The Department Manager should complete the Purchase Request Order Form (Figure 9-1) when requesting reagents, standards, or supplies.

The analyst must provide the item number, item description, package size, and the quantity needed. The Department Manager completes the purchase request order form and provides it to the Shipping/Maintenance Technician. The Shipping/Maintenance Technician places the order with the corporate office, which in turn places the order with the vendor.

9.3.2 Receiving

It is the responsibility of the Shipping/Maintenance Technician to receive the shipment. It is the responsibility of the Shipping/Maintenance Technician to date the material when received for the vendor storage and purchasing area. If the material received was ordered directly by the lab for laboratory use, the analyst that placed the order is responsible for dating the material when received. Once the ordered reagents or materials are received, the shipping/maintenance technician compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are maintained and updated by the EH&S officer and 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

There are many different grades of analytical reagents available to the analyst. All 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, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of 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 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.

- An expiration date can not be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.

- Expiration dates can be extended if the dry chemical 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 is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained within each department.

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. The minimum total pressure must be 100 psig. The tank regulators are set at 100 psig, when the tank pressure goes at/below 100 psig the automatic system switches to a tank with higher pressure, and then the empty tank must be replaced. 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 conductivity of less than 1mmho/cm (or resistivity of greater than 1.0 megaohm-cm) at 25°C. The conductivity is checked and recorded daily. If the water's conductivity is less than the specified limit, the Laboratory Director 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 water (or other similar quality) 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.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

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. Table 9-1 details specific storage instructions for reagents and chemicals. Section 22 discusses conditions for standard storage.

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 Operations Manager and/or the Laboratory Director/Manager. If they agree with the request the procedures outlined in 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, approved by corporate (CapEx), and the order is given to the corporate office to place the actual order.

Upon receipt of a new or used piece of equipment, it is given a short name, such as HP-20, added to the equipment list described in Section 21 that is maintained by the QA Department and IT must be notified so that can be linked for back-ups. The instrument name is then added into the LIMS system for data recording purposes. 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 (see Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department as specified in the laboratory's procedure for software verification (see SOP S-ITQ-007). Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained within the department that the equipment/instrument is located.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 21. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers/Laboratory Director.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). The level of control used in the selection process is dependent on the anticipated spend 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 (CW-F-WI-009).

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 (CW-F-WI-007 – refer to Figure 9-2).

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 Laboratory Director are consulted with vendor and product selection that have an impact on quality.

Figure 9-1.

Purchase Order Request Form

TestAmerica Denver
Purchase Order Request Form

Vendor Name	Vendor #	Item Description	Item #	Qty.	U/M	Unit Cost	Total	Billing Acct. Number	Requested Delivery Date	Requested By
							$0.00			
							$0.00			
							$0.00			
							$0.00			
							$0.00			
							$0.00			
							$0.00			
			Total	**0**		**Total**	**$0.00**			

Department #

Order Placed By:_____

Group Leader Approval:_____

Date: _____

Manager Approval_____

Req Creation Date:_____

Type of Shipping	
Overnight Rush (1-day)	
Rush 2 Day (2-days)	
Ship Ground (5-7 days)	
Ship For Sure - (Date)	

If type of shipping is not designated the order will ship ground.
Rush orders processed late will need an extra day for delivery.
Please fill out form in its entirety.
Ordering days are Tues. and Thurs. before 10 am.

Accounting Codes:

58100 - Building MX
60000 - Glassware
61000 - Sample Bottles
62500 - Consumable Lab Supplies
63000 - Solvents/Chemicals
63000.001 - Standards
64000 - Gases
71000 - MX and Repairs (Contract)
71100 - MX & Repairs (Non-Contract)
77000 - Office Supplies

Table 9-1.

Storage of Reagents and Chemicals

Chemical	Storage Requirements
Concentrated Acids and Bases	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Bulk Dry Chemicals	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Working Solutions containing Organic Compounds	Stored as per method recommendation/ requirement. They are generally stored refrigerated at 4°C± 2°C.
Working Solutions containing only Inorganics	Stored at room temperature; refrigeration is optional, but recommended.
Flammable Solvents	Stored in solvent cabinets at room temperature.
Non-Flammable Solvents	Stored separately from the flammable solvents in cabinets at room temperature.

Figure 9-2
Example – JD Edwards Vendor Add Request Form

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

JD Edwards Vendor Add Request Form

Vendor name:	Lab location and individual making request:
Vendor address (remit to):	Vendor phone:
Vendor address (remit to):	Vendor fax:
Contact name:	Product / service provided:

Reason for Vendor Addition: Check all reasons that apply

☐ Cost Reduction	Estimated Annual Savings $
☐ Replace Current Vendor	Reason?
	Vendor being Replaced?
☐ New Product / Service	Describe:
☐ ISO Approved (Required for Aerotech / P&K only)	

Small Business:

Does this vendor help us to meet our small business objectives: _____

If yes, which category: _____

Personal and Ethical Considerations:

Is there any personal conflict of interest with a TestAmerica employee and the vendor listed above? _____

_____

Have ethical considerations been taken into account in your evaluation of this vendor?_____

_____

Can this product be sourced from another TestAmerica facility?_____

Please complete form and email to NCPurchasing@testamericainc.com or fax to (330) 966-9275.

I approve the addition of this vendor:

_____ _____

Purchasing Manager - Patrick Eckman Corporate Controller - Leslie Bowers

Form No. CW-F-WI-007

SECTION 10

SERVICE TO THE CLIENT
(NELAC 5.4.7)

10.1 OVERVIEW

TestAmerica Denver 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 discussed in Section 5. The laboratory has procedures to ensure confidentiality to clients (Section 16 and 26).

Note: ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

10.2 SPECIAL SERVICES

The laboratory's standard procedures for reporting data are described in Section 26. When requested the following special services are provided:

- The laboratory will provide the client or the client's representative reasonable access to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- The laboratory will work with client-specified third party data validators as specified in the client's contract.
- The laboratory will provide the client with all requested information pertaining to the analysis of their samples. An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

10.3 CLIENT COMMUNICATION

Project managers are an important communication link to the clients. The lab shall inform its clients of any delays in project completion as well as any non-conformances in either sample receipt (refer to Section 24) or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

The QA Manager or Technical Director are available to discuss any technical questions or concerns that the client may have.

10.4 REPORTING

The laboratory will work with the client to produce any special communication reports required by the contract.

10.5 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica Denver participates in the American Council of Independent Laboratories (ACIL) Seal of Excellence program. This program includes the submission of a survey to laboratory clients. The clients send their responses directly to ACIL.

TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 11

COMPLAINTS
(NELAC 5.4.8)

11.1 OVERVIEW

TestAmerica Denver believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that helps to continually improve processes and improving 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, 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 dealing with both external and internal complaints.

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 13 (Corrective Actions) and is documented following SOP DV-QA-013P, Customer Complaints. It is the laboratory's goal to provide a satisfactory resolution to complaints in a timely and professional manner.

11.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process and the documentation of the complaint.

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 likely hood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving 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.

11.3 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 13. In addition, Corporate management, Sales and Marketing and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 13.

11.4 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 17)

1

SECTION 12

CONTROL OF NON-CONFORMING WORK
(NELAC 5.4.9)

12.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, 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 13).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. Refer to SOP DV-QA-0031, Nonconformance and Corrective Action System for the procedure to handle such situations.

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 20. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Department 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 NELAC (or the analytical method) requirements and the reason. Data being reported to a non-NELAC state would need to note the change made to how the method is normally run.

12.2 RESPONSIBILITIES AND AUTHORITIES

SOP No. CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall, outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of the company's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director or Department Manager, with approval from the QA Manager may exceptionally 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

2
described in Section 13 and in SOP DV-QA-0031, Nonconformance and Corrective Action System. 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 laboratory management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, the Department Manager, the Manager of the PM staff, and the Operations Manager. 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) and Quality Director within 24 hours.

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/Manager, QA Manager, ECOs, COO's – East and West, General Managers and the Quality Directors – East and West 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.

12.3 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.

SOP No. CA-L-S-001 distinguishes between situations when it would be appropriate for the laboratory QA Manager and Laboratory Director/Manager (or his/her designee) to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting (Section 13) in lieu of the data recall determination form contained in SOP No. CA-L-S-001.

12.4 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 (Section 13).

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.

12.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

3

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 12.2, Paragraph 5 above.

Prior to suspension/restriction, confidentiality will be respected, and the problem and the required corrective and preventive action will be stated in writing and presented to the Laboratory Director/Manager.

The Laboratory Director/Manager 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 13 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 General Manager 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/Manager to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (i.e., 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, QA Manager, Department Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, the Director of Client Services and Sales and Marketing should 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 as described in Section 13.

SECTION 13

CORRECTIVE ACTION
(NELAC 5.4.10)

13.1 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 13-1).

13.2 DEFINITIONS

- **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 are contained in the method specific SOPs. 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 is not only a correction made to the immediate event, but a change in process, procedure or behavior that is required to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

13.3 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 for investigation.
- 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 (see more on client complaints in Section 11).

13.3.1 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
- Holding Time Violations
- Observations

13.3.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Failed or Unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic Reporting / Calculation Errors

13.4 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.

13.4.1 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. Table 13-1 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 Department Manager, QA Manager (or QA designee), or Technical Director is consulted. The laboratory may also consult the technical contacts designated in the company for assistance.

13.4.2 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.

13.4.3 Monitoring of the Corrective Actions

- The Department Manager and QA Manager is responsible to ensure that the corrective action taken was effective.

- Ineffective actions will be documented and re-evaluated until acceptable resolution is achieved. Department 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 a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 17). 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.

13.4.4 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. (Section 16 includes additional information regarding internal audit procedures.)
- 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.

13.5 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 12 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCM or CAR, refer to SOP DV-QA-0031, Nonconformance and Corrective Action System.

Table 13-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs, SOP DV-QA-024P, Requirements for Federal Programs, or Appendix 4.

Table 13-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table 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, SOP DV-QA-003P, SOP DV-QA-024P, and Appendix 4, QAM Sections 20 and 21, and SOP CA-L-S-001 (Internal Investigation of Potential Data Discrepancies and Determination for Data Recall). All corrective actions are reviewed at a minimum monthly 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 a written NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

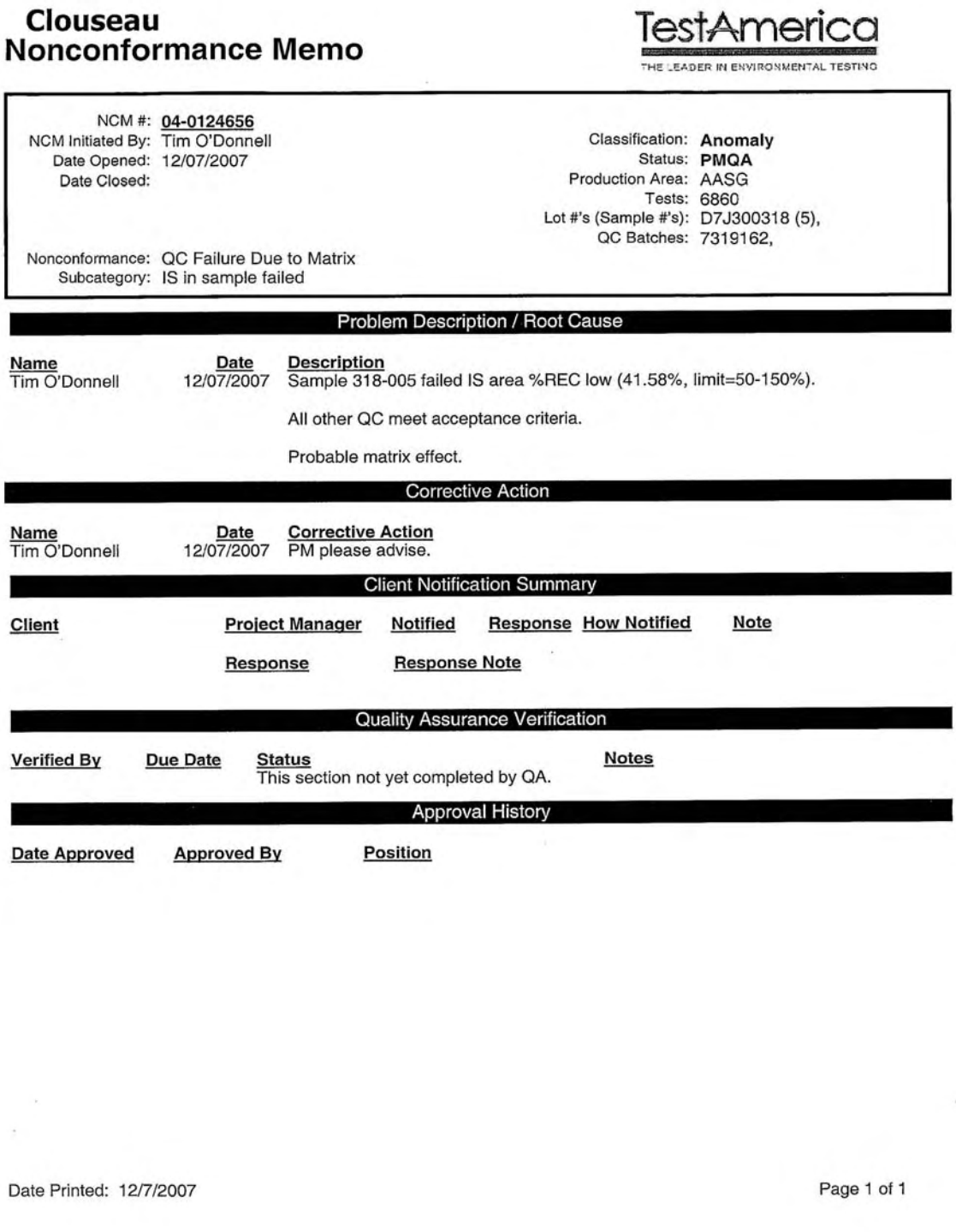
13.6 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall be crossed-out, and not erased, deleted, made illegible, or otherwise 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 13-1.
Example Non-Conformance Memo



Clouseau
Nonconformance Memo

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

NCM #: 04-0124656
NCM Initiated By: Tim O'Donnell
Date Opened: 12/07/2007
Date Closed:

Classification: **Anomaly**
Status: **PMQA**
Production Area: AASG
Tests: 6860
Lot #'s (Sample #'s): D7J300318 (5),
QC Batches: 7319162,

Nonconformance: QC Failure Due to Matrix
Subcategory: IS in sample failed

Problem Description / Root Cause

Name	Date	Description
Tim O'Donnell	12/07/2007	Sample 318-005 failed IS area %REC low (41.58%, limit=50-150%).
		All other QC meet acceptance criteria.
		Probable matrix effect.

Corrective Action

Name	Date	Corrective Action
Tim O'Donnell	12/07/2007	PM please advise.

Client Notification Summary

Client	Project Manager	Notified	Response	How Notified	Note	Response	Response Note

Quality Assurance Verification

Verified By	Due Date	Status	Notes
		This section not yet completed by QA.	

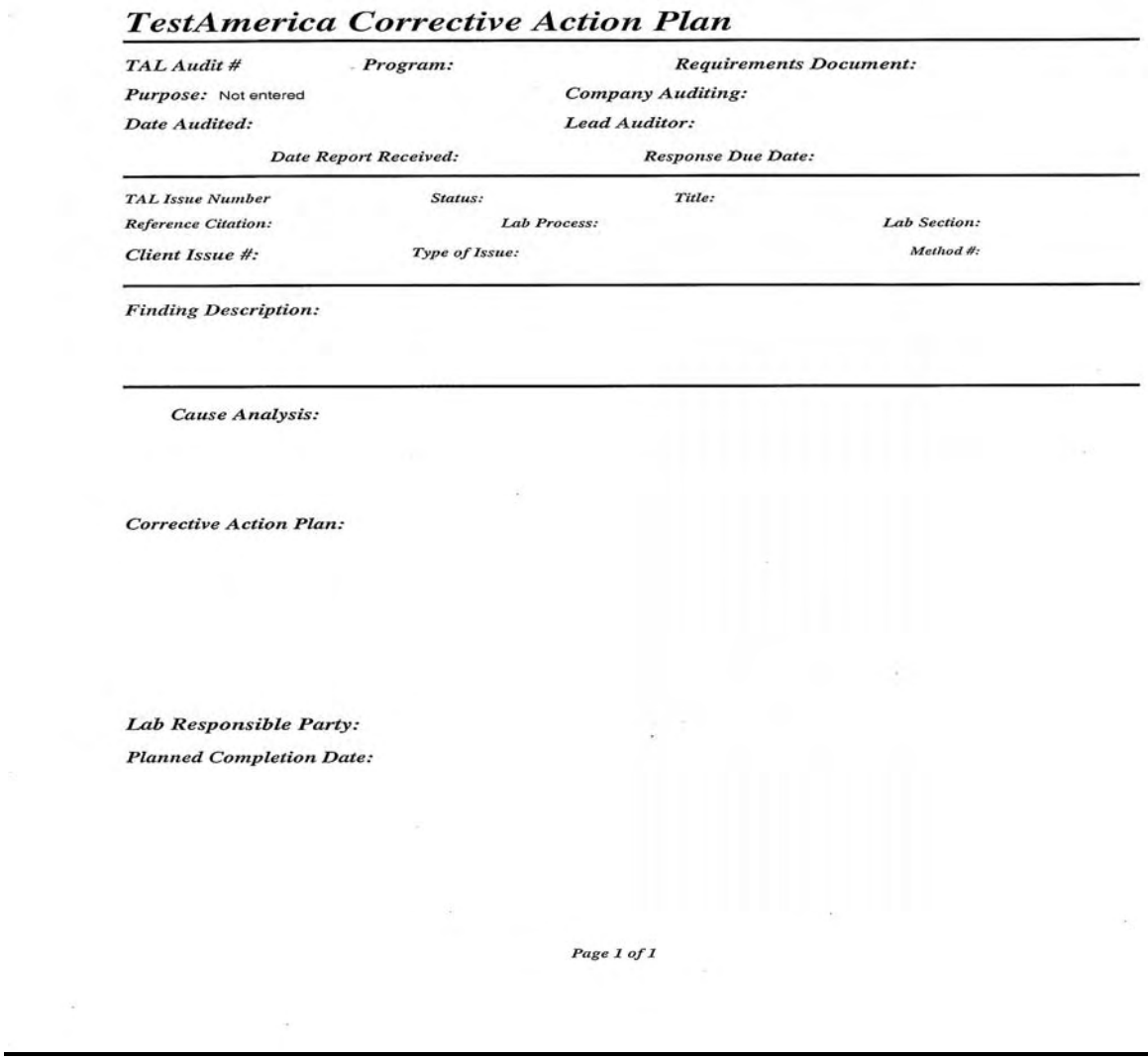
Approval History

Date Approved	Approved By	Position

Date Printed: 12/7/2007

Page 1 of 1

Figure 13-1. Con't
Example - Corrective Action Report



TestAmerica Corrective Action Plan

TAL Audit # *Program:* *Requirements Document:*

Purpose: Not entered *Company Auditing:*

Date Audited: *Lead Auditor:*

Date Report Received: *Response Due Date:*

TAL Issue Number *Status:* *Title:*

Reference Citation: *Lab Process:* *Lab Section:*

Client Issue #: *Type of Issue:* *Method #:*

Finding Description:

Cause Analysis:

Corrective Action Plan:

Lab Responsible Party:

Planned Completion Date:

Page 1 of 1

Figure 13-1. Con't
Example Open Corrective Action Summary Table

TestAmerica Denver

Summary of Open Federal Audits

LabName	AuditDate	Audit#	ProgName	Doc	CoAuditing	RcvdDate	DueDate
Denver	10/9/2006	63	Internal CA	NELAC	STL Denver	9/20/2006	10/9/2006
Denver	10/9/2006	74	External CA	Other	Clean Harbors/S	9/28/2006	10/31/2006
Denver	10/9/2006	64	Internal CA	NELAC	STL Denver	10/9/2006	10/9/2006
Denver	10/24/2006	71	Internal Audit	Other	STL Denver	10/24/2006	10/24/2006
Denver	10/26/2006	81	State Audit	Other	State of Arizona	11/29/2006	1/16/2007
Denver	11/7/2006	72	Internal Audit	Other	STL Denver	11/7/2006	11/10/2006
Denver	11/27/2006	76	Internal CA	Other	STL Denver	11/27/2006	11/28/2006
Denver	11/30/2006	78	Client Audit	Other	USGS	11/30/2006	12/5/2006
Denver	12/13/2006	86	AFCEE	AFCEE 4.0	EQM	1/9/2007	2/9/2007
Denver	1/17/2007	83	PT Failures	NELAC	STL Denver	1/16/2007	1/19/2007
Denver	4/27/2007	103	Internal Audit	Other	STL Denver	4/27/2007	5/4/2007
Denver	5/10/2007	113	Client Audit	NELAC	Parsons	5/10/2007	5/11/2007
Denver	5/11/2007	111	Internal Audit	AFCEE 4.0	STL Denver	5/4/2007	5/11/2007
Denver	5/16/2007	117	Client Audit	QSM V. 3	USACE	5/21/2007	6/4/2007
Denver	7/11/2007	123	Client Audit	Other	SM Stoller		7/13/2007
Denver	7/30/2007	127	PT Failures	NELAC	ERA	7/30/2007	8/13/2007
Denver	8/15/2007	131	State Audit	Other	State of WV	9/11/2007	9/26/2007
Denver	8/23/2007	133	Client Audit	QAPjP	ENSR	10/3/2007	10/30/2007
Denver	8/30/2007	129	State Audit	Other	State of Colorad	9/5/2007	9/28/2007

Table 13-1. Con't

General Corrective Action Procedures

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Initial Instrument Blank *(Analyst)*	- Instrument response < ½ RL	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards *(Analyst, Supervisor)*	- Correlation coefficient > 0.99. - Standard concentrations should bracket reporting limit. - % 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, Supervisor)*	- % Recovery within control limits as defined in the method SOPs.	- 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 as defined in the method SOPs. SOP DV-QA-027P has additional information for GC analyses.	- 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 LIMS.	- 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. See SOP DV-QA-003P for detailed corrective actions.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in LIMS.	See SOP DV-QA-003P for detailed corrective actions.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean (limits stored in LIMS).	See SOP DV-QA-003P for detailed corrective actions.
Method Blank (MB_ *(Analyst, Data Reviewer)*	< Reporting Limit [1]	See SOP DV-QA-003P for detailed corrective actions.

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Proficiency Testing (PT) Samples *(QA Manager, Department Manager/Supervisor)*	- 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, Department Manager/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.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager, QA Manager, Corporate QA, Corporate Management)	- SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001 and DV-QA-019P.
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). See SOP DV-QA-013P.
QA Monthly Report (Refer to Section 17 for an example) (QA Manager, Lab Director/Manager, Department Supervisors/Managers)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Health and Safety Violation (Safety Officer, Lab Director/Manager, Department Supervisor/Manager)	- 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 reporting limit (several programs require controlling to ½ the RL, see SOP DV-QA-024P for Federal Program Requirements). Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone, phthalates, zinc, iron, copper, and lead **provided** they appear in similar levels in the reagent blank and samples. 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 14.0

PREVENTIVE ACTION
(NELAC 5.4.11)

14.1 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 continuous process improvement activity 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 TestAmerica Denver's commitment to its Quality Assurance (QA) program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc..

The monthly Quality Assurance Metrics Report shows performance indicators in all areas of the 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. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's Corrective Action process (Section 13) 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 provides a valuable mechanism for identifying preventive action opportunities.

14.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process, management review, and the Management of Change process (see below).

Note: There may be varying levels of formality and documentation during the preventive action process due to the simplicity/complexity of the action taken.

14.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 17). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

14.2 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 SOP CA-Q-S-003, Management of Change.

SECTION 15.0

CONTROL OF RECORDS
(NELAC 5.4.12)

TestAmerica Denver maintains a record 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.

15.1 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 15-1. Quality records are maintained by the Quality Assurance (QA) Manager in a combination system of a paper filing and database system, which is backed up as part of the regular network 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 the Department Manager or their designee.

Table 15-1. Record Index[1]

Technical Records	Official Documents	QA Records	Project Records	Administrative Records
Retention: 5 Years from analytical report issue*	5 Years from document retirement date*	5 Years from archival* **Data Investigation:** 5years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)	5 Years from analytical report issue*	**Personnel:** 7 Years (HR Records must be maintained as per Policy CW-L-P-001) **Finance:** See Accounting and Control Procedures Manual
Raw Data Logbooks[2] Standards Certificates Analytical Records Lab Reports	Quality Assurance Manual (QAM)	Internal and External Audits/ Responses	Sample receipt and COC Documentation	Finance and Accounting
	Work Instructions	Certifications	Contracts and Amendments	EH&S Manual, Permits, Disposal Records
	SOPs	Corrective/Preventive Action	Correspondence	Employee Handbook
	Manuals	Management Reviews	QAPP	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)
		Method & Software Validation, Verification data	SAP	
		Data Investigation	Telephone Logbooks	Administrative Policies
	Policies		Lab Reports	Technical Training Records

[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 15-2.

All records are legible and stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or the Iron Mountain data storage facility that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** The laboratory retains analytical records for 2 months on-site at the laboratory and client reports for 6 months, after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless other wise 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 15-2 have lengthier retention requirements and are subject to the requirements in Section 15.1.3. Policy CW-L-P-001 (Record Retention) provides additional information on record retention requirements.

15.1.1 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 15-2, 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. The specific requirements for the length of retention of documents are listed in the statement of work in the contract set up between the client and the laboratory. The laboratory then marks the Iron Mountain storage box with the longer time of storage.

Table 15-2. Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
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
Navy Facilities Engineering Service Center (NFESC)	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

15.1.2 All records are held secure and in confidence. Records maintained at the laboratory are located in Arvada**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Logs are maintained in each storage box to note removal and return of records.

15.1.3 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, see section 20.12.1 'Computer and Electronic Data Related Requirements. For more information, refer to SOP DV-QA-025P, Electronic Data Backup.

15.1.4 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 (Records stored off site should be accessible within 2 days of a request for such records). 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 chain of custody 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 per the Data Archiving SOP No. DV-QA-0005. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run long 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, entered into the LIMS or the standards log program for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. 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.

- Also refer to Section 20.13.1 ‘Computer and Electronic Data Related Requirements’**.**

15.2 TECHNICAL AND ANALYTICAL RECORDS

15.2.1 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 (refer to Section 15.1). 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 performance of each analysis and checking of results.

15.2.2 Observations, data and calculations are recorded at the time they are made and are identifiable to the specific task.

15.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. 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 (previous discussions relate where most of this information is maintained – specifics may be added below):

- laboratory sample ID code**;**
- Date of analysis and time of analysis is 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 the method specific logbook or benchsheet.
- 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, incubation periods or subculture, 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 in the LIMS, on specific analytical report formats, and in client specific QAPPs and QASs**.**

15.3 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

15.3.1 Sample Handling Records

Sample handling and tracking is discussed in Section 24. 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.

15.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. See Table 15-1.

15.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

15.5.1 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 to the accrediting body upon request.

15.5.2 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.

15.5.3 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**.**

15.5.4 TestAmerica Denver has a record management system 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 within a given analysis and/or instrument. No analysis and/or instrument have more than one active notebook at a time, so all data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially by method and analysis date. Standards are maintained in the Standards Log program – no logbooks are used to record that data**.**

15.5.5 Records are considered archived when moved off-site. Access to archived hard-copy information is documented with an access log and in/out records is used in archived boxes to note data that is removed and returned. 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**.**

15.5.6 In the event that the laboratory transfers ownership or goes out of business, TestAmerica Denver 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.

15.5.7 Records Disposal

15.5.7.1 Records are removed from the archive and disposed 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.

15.5.7.2 Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

15.5.7.3 If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required. [Refer to Policy No. CW-L-P-001 (Records Retention).]

SECTION 16

AUDITS
(NELAC 5.4.13)

16.1 OVERVIEW

Audits measure laboratory performance and insure compliance with accreditation/certification and project requirements. Audits specifically provide management with an on-going assessment of the quality of results produced by the laboratory, including how well the policies and procedures of the QA system and the Ethics and Data Integrity Program are being executed. They are also instrumental in identifying areas where improvement in the QA system will increase the reliability of data. There are two principle types of audits: Internal and External. Internal audits are performed by laboratory or corporate personnel. External audits are conducted by regulators, clients or third-party auditing firms. In either case, the assessment to program requirements is the focus.

Table 16-1. Audit Types and Frequency

Internal Audits	**Description**	**Performed by**	**Frequency**
	Analyst & Method Compliance	QA Department or Designee	- 100% of all methods over a two year period. - 100% of all analysts annually.
	Instrument	QA Department or Designee	100% of all organic instruments and any inorganic chromatography instruments. Annually.
	Final Report	QA Department or Designee	- 1 complete report each month.
	Support Systems	QA Department or Designee	- Annual for entire labs support departments & equipment (e.g., thermometers, balances), can be divided into sub-sections over the course of the year.
	Performance Audits (Double-Blind PTs)	Corporate QA, Laboratory QA Department or Designee	- As needed.
	Special	QA Department or Designee	- As Needed
External Audits	**Description**	**Performed by**	**Frequency**
	Program / Method Compliance	Regulatory Agencies, Clients, accreditation organizations	- As required by program and/or clients needs
	Performance Audits	Provided by a third party.	- As required by a client or regulatory agency. Generally provided semi-annually through the analysis of PT samples.

16.2 INTERNAL AUDITS

Annually, the laboratory prepares a schedule of internal audits to be performed throughout the year. As previously stated, these audits verify and monitor that operations continue to comply with the requirements of the laboratory's QA Manual and the Corporate Ethics Program, the

DoD Quality Systems Manual, and other Federal Programs. A schedule of the internal audits is maintained by the QA Manager in the *Internal Audit Workbook*. An example can be found in Figure 16-1.

It is the responsibility of the QA Manager to plan and organize audits in consideration of the laboratory work load and the department personnel schedules so that all pertinent personnel and operations are thoroughly reviewed. When designees (other than QA department personnel & approved by the QA Manager), perform audits, the QA Manager shall insure that these persons do not audit their own activities except when it can be demonstrated that an effective audit will be carried out. In general, the auditor:

- is neither the person responsible for the process being audited nor the immediate supervisor of the person responsible for the project/process.
- Is free of any conflicts of interest.
- Is free from bias and influences that could affect objectivity.

Laboratory personnel (e.g., supervisors and analysts) may assist with both method and support system audits as long as the items listed in the above paragraph are observed. These audits are conducted according to defined criteria listed in the checklists of the *Internal Audit Workbook.* These personnel must be approved by the QA Manager; and must complete the audit checklists in their entirety. This process introduces analyst experience and insight into the laboratory's auditing program.

The auditor must review the previous audit report and identify all items for verification of corrective actions. A primary focus will be dedicated to the ability of the laboratory to correct root-cause deficiencies and that the corrective action has been implemented and sustained as documented.

Refer to SOP DV-QA-0029, Independent QA Data Review for details on TestAmerica Denver's internal lab audit process.

16.2.1 Systems

An annual systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems, client and State requirements. This audit is performed in portions throughout the year through method, analyst, instrument, work order/final report and support system audits. Audits are documented and reported to management within 1 week of their performance. Systems audits cover all departments of the facility, both operational and support. The multiple audits are compiled into one systems audit package at the end of the year (*Internal Audit Workbook*).

16.2.1.1 Method, Analyst, Instrument and Work Order/Final Report Audits

Procedures for the method compliance, analyst, instrument and work order/final report audits are incorporated by reference to SOP No. CA-Q-S-004, Method Compliance and Data Authenticity Audits. These audits are not mutually exclusive. For example, the performance of a method audit will also cover multiple analysts and instruments. The laboratory's goal is to annually review all analysts and instruments as described in SOP No. CA-Q-S-004. The

laboratory will also audit all methods within a two year time period and audit a minimum of one Work Order/Final Report from receiving through reporting on a monthly basis.

16.2.1.2 Support Systems

Support system audits are performed to ensure that all departments & ancillary equipment are operating according to prescribed criteria. Support system audits include the review of both non-analytical and operational departments. Support equipment audits (e.g., metrology items) include the review of balance calibrations, weight calibrations; water quality testing, etc.. Non-analytical may include sample receiving and bottle preparation. These types of support audits ensure that the operations are being performed to support ethical data as well as ensuring the accuracy & precision of the utilized equipment.

These audits can be performed in portions throughout the year or in one scheduled session. However, the audit schedule must document that these aspects are reviewed annually. Many of the metrology systems are considered to be surveillance activities that can be monitored by QA personnel or delegated to specified department personnel. These surveillance activities are performed on a semi-annual basis unless issues warrant a greater frequency or previous audits continually showing no deficiencies allow the frequency to be reduced to once a year.

An example audit checklist can be found in Figure 16-2. Instructions for reporting findings are included in the *Internal Audit Workbook*. In general, findings are reported to management within 1 week of the audit and a response is due from management within 30 days.

16.2.2 Performance Audits

Corporate QA may arrange for double blind PT studies to be performed in the laboratories. Results are given to Management and Corrective actions of any findings are coordinated at each facility by the QA Managers and Laboratory Directors/Managers. These studies are performed on an as needed basis. They may be performed when concerns are raised regarding the performance of a particular method in specific laboratories, periodically to evaluate methods that may not normally be covered in the external PT program or may be used in the process of developing best practices. The local QA Manager may also arrange for PT studies on an as needed basis. (Refer to Section 16.3.2 for additional information on Performance Audits.)

16.2.3 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.

16.3 EXTERNAL AUDITS

TestAmerica facilities are routinely audited by clients and external regulatory authorities. 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. The department managers 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. This time frame is generally 30 days.

Be aware that NELAC requires that the audit response report be acceptable to the primary accrediting authority after the second submittal. The lab shall have accreditation revoked for all or any portion of its scope of a accreditation for any or all fields of testing, a method, or analyte within a field of testing if it is not corrected.

TestAmerica Denver 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.

16.3.1 Confidential Business Information (CBI) Considerations

During on-site audits, on-site 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 2003 NELAC standards.

16.3.2 Performance Audits

The laboratory is involved in performance audits conducted semi-annually through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Water Pollution studies, Water Supply studies, Soil and Hazardous Waste studies, DMRQA studies, and project specific or client requested studies.

- It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Further, 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.

- PTs generally do not have holding times associated with them. In the absence of any holding time requirement, it is recommended that the holding time begin when the PT sample is prepared according to the manufacturers instructions. Holding times should apply to full volume PT samples only if the provider gives a meaningful "sampling date". If this is

not provided, it is recommended that the date/time of opening of the full volume sample be considered the beginning of holding time.

- Login will obtain the COC information from the documentation provided with the PTs with review by QA or other designated staff.

- Vials will be prepared as required in the instruction set provided with the samples. After preparation to full volume the sample may be spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis.

- PT samples will not undergo multiple preps, multiple runs, multiple methods (unless being used to evaluate multiple methods), multiple dilutions, UNLESS this is what would be done to a normal client sample (e.g. if a client requests, as PT clients do, that we split VOA coeluters, then dual analysis IS normal practice).

- The type, composition, concentration and frequency of quality control samples analyzed with the PT samples shall be the same as with routine environmental samples.

- Instructions may be included in the laboratory's SOPs for how low level samples are analyzed, including concentration of the sample or adjustment of the normality of titrant. When a PT sample falls below the range of the routine analytical method, the low-level procedure may be used.

- No special reviews shall be performed by operation and QA, UNLESS this is what would be done to a normal client sample. To the degree that special report forms or login procedures are required by the PT supplier, it is reasonable that the laboratory WOULD apply special review procedures, as would be done for any client requesting unusual reporting or login processes.

- 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.

16.4 AUDIT FINDINGS

Internal or External Audit findings should be documented using the corrective action process and database (refer to Section 13). The laboratory is expected to prepare a response to audit findings within 30 days of receipt of an audit report unless the report specifies a different time frame. The response may include action plans that could not be completed within the 30 day timeframe. In these instances, a completion date must set and agreed to by operations management and the QA Manager.

Responsibility for developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

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.

The procedures must be in accordance to SOP No. CA-L-S-001, Internal Investigations of Data Discrepancies and Determination of Data Recall.

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.

Figure 16-1.

Example - Internal Audit Workbook

Laboratory: TAL Denver

Internal Audit Schedule 2007

***Schedule to be completed 4/2007 for remainder of the year.**

Area Audited	Type	Cycle	SOP Reference	Comments	Scheduled	Audited	Closed
Balances	System	6 mo	DEN-QA-0014	CHRISTINA	5/7/2007	5/1/2007	5/1/2007
					12/7/2007	9/19/2007	9/19/2007
Temperature Logs/Thermometers	System	6 mo	DEN-QA-0001 & DEN-(	MARIA	5/7/2007	5/15/2007	5/15/2007
					12/7/2007	12/10/2007	12/10/2007
Sample Storage and Disposal	System	1 yr	DEN-QA-0003	MIKE	7/1/2007		
Maintenance Logs	System	6 mo	QA-008	CHRISTINA	5/7/2007	5/1/2007	5/1/2007
					12/7/2007		
Holding Blanks for Volatile Ref/Freezers (where required)	System	6 mo	DEN-QA-0013	Although blanks are tracked routinely, a six-month review of all VOA blanks will be	4/6/2007	4/6/2007	4/6/2007
Lab Water Quality Testing	System	6 mo	DEN-QA-0026	See audit database a	4/7/2007	5/17/2007	5/17/2007
					11/7/2007		
Sample Control (Log In)	System	1 yr	DEN-QA-0003	MIKE	7/1/2007		
Shipping Procedures	System	1 yr	DEN-QA-0017	CHRISTINA	6/1/2007		
Computer Operations (LIMS)	System	1 yr	S-ITQ-001	MIKE	7/1/2007		
SOP Distribution System	System	1 yr	QA-001	MARIA	8/1/2007		
Archiving of Paper Records	System	1 yr	DEN-QA-0005	CHRISTINA	8/1/2007	5/30/2007	5/30/2007
Statistical Process Control	System	1 yr	QA-003	MIKE	8/1/2007	8/14/2007	8/14/2007
Electronic Archiving	System	1 yr	QA-025	MARIA	9/1/2007		
Data Review System	System	1 yr	QA-012	CHRISTINA	9/1/2007	9/10/2007	9/26/2007
Final Report Generation	System	1 yr	DEN-QA-0022	CHRISTINA	9/1/2007	10/19/2007	11/2/2007
Standards/Reagents	System	6 mo	DEN-QA-0015	MIKE	5/7/2007	5/1/2007	5/1/2007
					12/7/2007	10/22/2007	11/2/2007
Manual Integration	System	1 yr	DPOL-QA-011	MIKE	10/1/2007		
Corrective Action System	System	1 yr	DEN-QA-0031	CHRISTINA	10/1/2007	11/6/2007	
Training Records	System	6 mo	DEN-QA-0024	MARIA	5/7/2007	6/28/2007	6/28/2007
					12/7/2007	11/7/2007	11/7/2007
MDLs	System	1 yr	QA-005	CHRISTINA	11/1/2007		
SOPs	System	1 yr	QA-001	MARIA	11/1/2007		
Purchasing/Procurement	System	1 yr	STL.PG-001	MIKE	11/1/2007		
Pipette/Diluter/Dispenser Calibration Check	System	6 mo	DEN-QA-0008	MIKE	5/7/2007	7/9/2007	7/9/2007
					12/7/2007		
Subcontract Lab Approval	System	1 yr	DEN-QA-0027	CHRISTINA	11/1/2007	11/21/2007	
Customer Complaint System	System	1 yr	QA-013	MARIA	11/1/2007		
Annual Systems Audit	System	1 yr	NA	Larry Penfold	January 7-10		
Methods	Method	2 yr					

Figure 16-2.

Example – Internal Audit System Checklist: Corrective Actions

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica <Location>

INTERNAL AUDIT - Corrective Actions

[Printed Name(s) or Date(s)]

(Summary Page)

Area Audited:	
Auditor:	
Date:	
Persons Contacted During Audit:	
Date Reported to Department Manager:	
Reported To:	
Date Reported to Lab Director/Manager:	
Reported To:	
Date Response Due:	
Response Received and Accepted by QA Manager:	
Associated Corrective Action Report Number(s):	
Scheduled Follow-up:	

Item	Requirement	Ref.	Y	N	NA	Evidence/Comments	Follow Up
1	Does the laboratory have a corrective action program in place?	5.4.10.1					
2	Does the laboratory have a current corrective action SOP or is this information in the QA Manual?	5.4.10.1					
3	Do all laboratory personnel have documented training and access to initiate corrective actions?	5.4.10.1					
4	Are causes clearly identified by department, staff name, scope of issue (how many reports affected)?	5.4.10.6					
5	Is a root cause for the issue identified?	5.4.10.2					
6	Is a corrective action (plan) clearly described?						
7	Was the corrective action fully implemented?						
8	Is documentation (if applicable) completed as specifed by the corrective action (training, revised SOP, etc)						
9	Has a follow-up assessment been conducted to verify the corrective action was successful?						
10	Are corrective actions reviewed on a regular basis by management?	5.4.10.6a 5					
11	Is there a defined distribution flow for corrective action notification, review, closure, and follow-up?	5.4.10.6a					
12	Are non-conformances reviewed on a regular basis and used, if necessary, to initiate root cause corrective actions?						
13	Does the lab have a documented procedure for QC corrective action (i.e., documented within each method / parameter SOP or in the QA Manual)?	4.10.1					
14	***Verify Corrective Actions from previous systems audits. List Items:***						
15							
16							
17							

Auditor Signature:_____

Primary Reference(s): Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices
NELAC Standard, June 2003
DoD Quality Systems Manual, Version 3, January 2006
EPA Manual for the Certification of Laboratories Analyzing Drinking Water

Figure 16-2. Con't
Example Internal Lab Section Audit Checklist

TestAmerica Denver

Organic Preparation QA Data Audit Checklist

Date Audited:_____ Batch Number(s) Audited:_____

Method:_____ Auditor:_____

Analyst(s):_____

Evaluation	Acceptance Criteria	Acceptable (Y/N/NA)?	*Comments*
IDOC on file?	Required for each analyst		
Is internal COC complete?	All required info entered.		
Is original handwritten version of benchsheet available?	Original records must be kept 5 yrs		
Is Data Recording Policy followed?	Entries in ink, single line cross-out, date & initials		
Method and/or SOP# clearly indicated?	Entry must be made		
Personnel clearly indicated?	Everyone involved must be listed		
Sample pH entered?	Entry must be made for most tests		
Times on & off for extraction recorded?	CLLE & Soxhlet need it		
All standards traceable?	Std #s required		
All reagents traceable?	Lot #s required		
Nonconformances recorded?	See NCM SOP		
NCMs described accurately in case narrative?	All NCMs must be communicated to client		
All required fields entered?	Per method SOP		
2nd-level review documented?	Name or initials & date		

Overall Comments:

Corrective Action Required:

A copy of this report will be maintained in the Quality Assurance office.

Auditor Signature _____ Date _____

L:/QA/Read/Audit/Data Audit/QA Data Review Forms/Organic Prep
9/10/07 version

SECTION 17

MANAGEMENT REVIEWS
(NELAC 5.4.14)

17.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director for review and comments. The final report shall be submitted to the Operations Manager as well as the appropriate Quality Director and General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. At a minimum, the report content will contain the items listed below. During the course of the year, the Laboratory Director/Manager, General Manager 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 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. The report is presented to the Senior Management Team and General Managers by the VP of Client and Technical Services.

The TestAmerica QA Report template is comprised of a discussion of three key QA issues facing the laboratory and ten specific sections (Figure 17-1):

- **Metrics:** Describe actions or improvement activities underway to address any outlying quality metrics.
- **SOPs:** Report SOPs that have been finalized and report status of any outstanding SOP reviews.
- **Corrective Actions:** Describe highlights and the most frequent cause for report revisions and corrective/preventive action measures underway. Include a discussion of any recalls handled at the lab level utilizing CA-Q-WI-003, *Data Recall Report*. Corporate SOP CA-L-S-001, *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* describes the process in detail. Include a section for client feedback and complaints. Include both positive and negative feedback. Describe the most serious client complaints and resolutions in progress.
- **MDLs and Control Limits:** Report which MDLs/ MDL verifications are due. Report the same for Control Limits.
- **Audits:** Report Internal and External Audits that were conducted. Include all relevant information such as which methods, by whom, corrective actions needed by when and discuss unresolved audit findings.
- **Performance Testing (PT) Samples:** Report the PT tests that are currently being tested with their due dates, report recent PT results by study, acceptable, total reported and the month and year.
- **Certifications:** Report on any certification programs being worked on by due date, packages completed. Describe any issues, lapses, or potential revocations.

- **Regulatory Updates:** Include information on new state or federal regulations that may impact the laboratory. Report new methods that require new instrumentation, deletion of methods, changes in sampling requirements and frequencies etc…
- **Miscellaneous:** Include any issues that may impact quality within the laboratory. This section is also used to communicate the status on any Management of Change Request Forms (CRFs) that have missed targeted due dates.
- **Next Month:** Report on plans for the upcoming month.
- **Lab Director Comments Section:** This section gives the Laboratory Director/Manager the opportunity to comment on issues discussed in the report and to document plans to resolve these issues. Unresolved issues that reappear in subsequent monthly reports must be commented on by the Laboratory Director/Manager.
- **Metrics:** The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The VP-QA/EHS prepares 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 Analytical Division Senior Management Team and General Managers.

17.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Operations Manager, Department Managers, and QA Manager) conducts an annual review 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. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director/Manager. 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 can not be solved by the lab and report them to Corporate IT.

This review uses information generated during the preceding year to assess the “big picture” by ensuring that routine quality actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review (refer to Section 17.1) 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 Management team 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),
- Review of the ACIL seal of excellence program performance.
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

The annual review includes the previous 12 months. Based on the annual review, 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)].

The QA Manual is also reviewed at this time and revised to reflect any significant changes made to the quality systems.

17.3 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. The Corporate Data Investigation/ Recall SOP shall be followed (SOP No. CA-L-S-001). 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.

The CEO and COO receive a monthly report compiled form from the Quality Directors summarizing any current data integrity or data recall investigations as described in SOP No. CA-L-S-001. The General Manager's are also made aware of progress on these issues for their specific labs.

Figure 17-1.

Example - QA Monthly Report to Management

QUALITY REPORT TO MANAGEMENT

LABORATORY: TAL Denver
PERIOD COVERED: November 2007
PREPARED BY: QA Manager DATE: December 10, 2007
DISTRIBUTED TO: Corporate QA, Lab Director, Program Manager, Operations Manager

THREE KEY ISSUES FOR MONTH:
1. Working through QAM update, scheduled to be complete 12/15/07.
2. DOE acceptance of corrective action report received.
3. Owe Corporate Federal QA Manager limits/SOPs/MDLs for FUDS Contract.

1. METRICS
Data submitted for WP153 and soil study 60.

2. SOPs

Please see the SOP tracking database, and weekly QA % currency updates.

The following SOPs were finalized (or reviewed for accuracy):
Reviewed/Revised in October:

DV-OP-0013 Mutli-increment Sampling
DV-OP-0013 Multi-increment Sampling for Metals only
DV-GC-0020 Chlorinated Pesticides by 8081

2. CORRECTIVE ACTION

Highlights:
Received DOE acceptance for CAR

Revised Reports:
Please see the attached metrics.

Data Investigations/Recalls (Corporate Data Investigation/Recall SOP) :
none.

Client Feedback and Complaints:
1.) Several client complaints were received regarding TAT. Reduced TAT is occurring as lab backlog drops.
2.) The PM and lab received compliments from Mactec for performance on the DFC work.

4. MDLs AND CONTROL LIMITS

MDLs Due:
Please see the MDL tracking database and Denver QA HelpDesk Records.

# of MDLs in QA pending review/update:1
# of MDLs in QA being reviewed: 0

The GCMS lab is working on MDLs for APIX SVOC compounds.

CSLP MDLs are completed and will be turned in to QA this week.
Meeting was held with GC, GCMS, and Organic Extractions this week to prepare MDL schedule.

Control Limits Due:

5. AUDITS

INTERNAL AUDITS
Electronic Data back-up:

A CAPEX has been placed to replace computers that require removal of the drive for backup. The IT staff estimates a 30 day time frame for completion of the software program that will run each night and perform backups for LCMS and some of the other instruments currently requiring manual backup. This issue will be closed when that program is completed.

EXTERNAL AUDITS
Response for Navy audit due 12/13//07.

6. PT SAMPLES

The following PT samples are now in house (Due Dates):
WP153
Soils study #60

7. CERTIFICATIONS

Certification Packages Being Worked On (Include Due Date):
Arizona

Describe any issues, lapses, or potential revocations.

8. REGULATORY UPDATE
Lab still updating quotes and notifying clients of Method Update Rule (MUR) changes.

9. MISCELLANEOUS
On-time delivery is poor due to lab backlog. Average for the month ≈50%.

10. NEXT MONTH
The lab will be audited by Larry Penfold January 7-10.

LAB DIRECTOR COMMENTS AND PLANNED CORRECTIVE ACTIONS:	
LAB DIRECTOR REVIEW:	DATE:

Figure 17-2.

Example - Laboratory Metrics Categories

 # Reports for month
 # Reports revised due to lab error
% Revised Reports
 # Reports Reviewed by QA
# of Data Recall Investigations
Cummulative # Days Data Recalls Open
 # Client Complaints
 # Client Compliments
# of Technical Data Audits Planned
# of Technical Data Audits Perfromed
% of Technical Data Audits Performed
# of Planned Department Quality Systems Audits
# of Planned Department Quality Sytems Audits Complete
% Annual Internal Systems Audits Complete
Total Number of Audit Findings (internal and external)
# of Audit Findings Past Due
2008 Open Audit Findings
# of PT analytes participated and received scores
# of PT analytes not acceptable
% PT Cummulative Score
 # PT Repeat Analyte Failures Cummulative
Total Number of Corrective Actions
# of Corrective Actions Past Due
% Corrective Action Items Past Due
# of SOPs
# of SOP with Procedure Compliance Review/Revision Past Due
 # Methods or Administrative Procedures without approved SOPs
% SOP Complete
Date of last Comprehensive Ethics Training
 # Staff > 90 Days from Hire Date AND have not received Comprehensive Ethics Training

Total Number of MDLs/MDLVs Required
 # MDLs/MDLVs Past Due
% MDLs/MDLVs Complete
Training Documentation Records (good>90%, Fair 70-90%,or Poor <70%)
Hold Time Violations due to lab error
Total Access Update Status (good, fair, poor)
Total Access Certification PDFs current (good, fair, poor)
Method Certification Losses (performance or audit issues)
Last NELAC Audit Date
QAM Effective Date
Last Management QS Review Date

SECTION 18

PERSONNEL
(NELAC 5.5.2)

18.1 OVERVIEW

TestAmerica'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 Appendix 2.

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.

18.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

TestAmerica makes every effort to hire analytical staff that posses 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. There are competent analysts and technicians in the industry who have not earned a college degree. 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, DO, Redox, 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, Herbicides, 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
Department 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
Department Manager – **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

Specialty	Education	Experience
Department Manager – Microbiology	Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of 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 Department 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.

18.3 TRAINING

TestAmerica 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 – Initial Training	Prior to work in designated area	All
Environmental Health & Safety	Refer to EH&S Manual	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	1 week 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 20.

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 20).
- 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). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in the Laboratory Training SOP (DV-QA-0024).

18.4 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, 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 an Ethics Policy No. CA-L-P-001 and an Ethics Statement/Agreement (Appendix 1). All initial and annual training is documented by signature on the signed Ethics Policy and Code of Ethical Conduct 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 (Appendix 1)
- 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 19

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS
(NELAC 5.5.3)

19.1 OVERVIEW

TestAmerica Denver is a 54,000 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.

19.2 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 effect 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.

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 (refer to Section 12).

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

The lab is equipped with a generator to maintain temperature on the sample refrigerators in the event of a power outage. The laboratory walk-in refrigerators are monitored around the clock and linked to an alarm system, which notifies the appropriate personnel of any temperature outages.

19.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Sample grinding and sample analytical areas.
- Organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.
- Waste disposal and sample/extract handling 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.

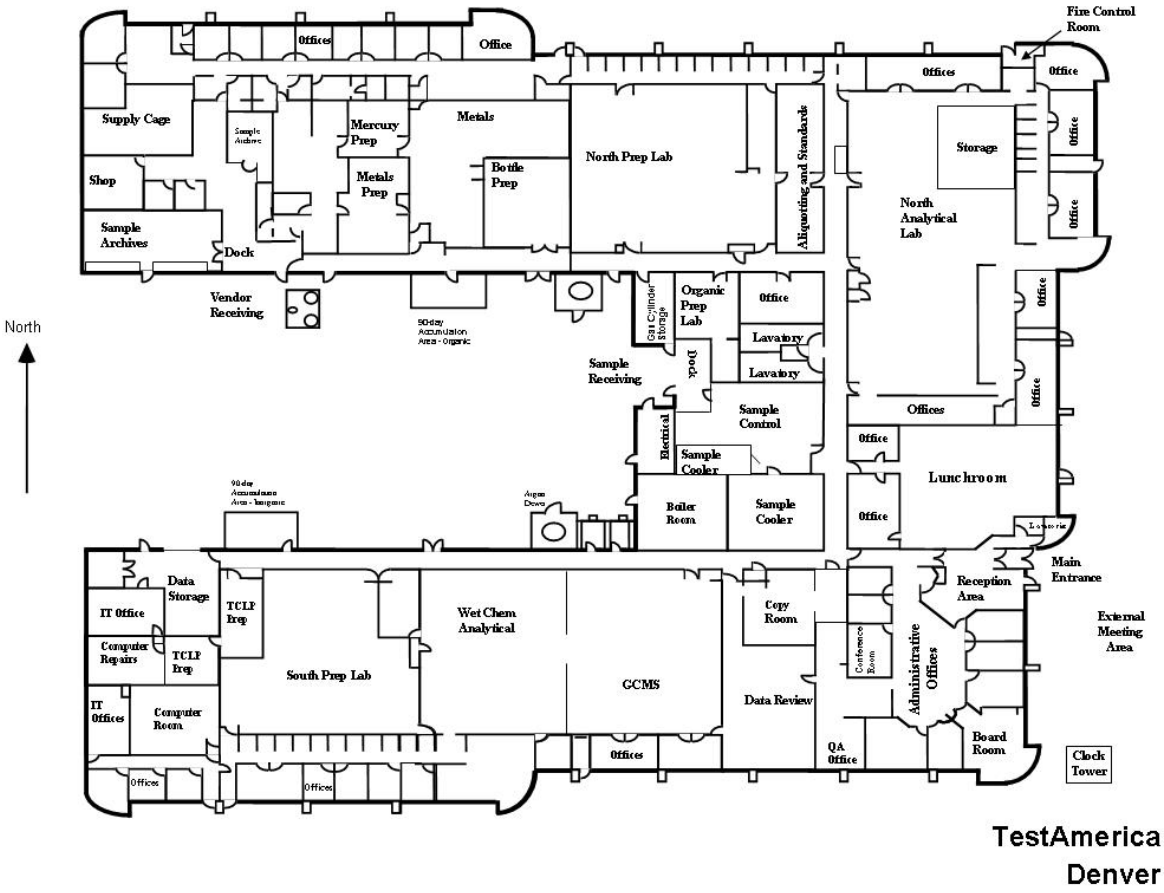
Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

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.

Refer to Standard Methods, 20th Ed., 9020B, Section 2 for specific requirements for microbiological laboratory facility requirements.

19.4 FLOOR PLAN



TestAmerica
Denver

19.5 BUILDING SECURITY

Building security cards and alarm codes are distributed to employees as necessary.

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 TestAmerica Denver. 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 20.0

TEST METHODS AND METHOD VALIDATION
(NELAC 5.5.4)

20.1 OVERVIEW

TestAmerica Denver 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.

20.2 STANDARD OPERATING PROCEDURES (SOPs)

TestAmerica Denver 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 (refer to Section 6 on Document Control):

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for preparation, review, revision and control are incorporated by reference to SOPs: CW-Q-S-002 (Writing a Standard Operating Procedure (SOP) and SOP DV-QA-001P.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP. Refer to the corporate SOP CW-Q-S-002 "Writing a Standard Operating Procedure" for content and requirements of technical and non-technical SOPs and DV-QA-001P, Preparation and Management of Standard Operating Procedure.

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.

20.4 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, etc.), 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.

20.4.1 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.

In general, TestAmerica Denver follows procedures from the referenced methods shown below in 20.4.1.1.

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.

20.4.1.1 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:

- *Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *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*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th 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, April 2008.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *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*

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.

20.4.2 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.

20.4.2.1 A demonstration of capability is performed whenever there is a change in instrument type, method or personnel.

20.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Operations Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures (refer to Section 15, Control of Records).

20.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit 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).
- The reporting limit is set at or above the first standard of the curve for the analyte.
- 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.*
- Refer to Section 12 (Control of Non-Conforming Work).

20.4.3 Initial Demonstration of Capability (IDOC) Procedures

20.4.3.1 Refer to SOP DV-QA-0024, Employee Training.

A certification statement (see Figure 20-1 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.

20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP/Methods Manual (Section 20.2) 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. The information included in the checklist below (Figure 20-2) is needed before samples are accepted for analysis by a new method.

20.6 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. (From 2003 NELAC Standard)

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. SOP DV-QA-024P contains information for the federal program requirements.

20.6.1 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.

20.6.1.1 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.

20.6.1.2 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. The laboratory determinations of MDLs are described in Section 20.7.

20.6.1.3 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 level at which both the presence of an analyte and its concentration can be reliably determined. 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. See SOP DV-QA-024P for specific relationships for work performed under the DoD QSM version 4.1.

20.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

20.6.1.5 Determination of Range

Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

20.6.1.6 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.

20.6.1.7 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.

20.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in SOP DV-QA-0024, Employee Training. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

20.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B. 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 (refer to 20.7.10). 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. This low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate student t-value is used. TestAmerica Denver's SOP procedures are outlined in detail in SOP DV-QA-003P, Determination of Method Detection Limits for Chemical Tests.

20.7.1 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B. For details, refer to SOP DV-QA-003P, Determination of Method Detection Limits for Chemical Tests.

MDL's are initially performed for each individual instrument and non-microbiological method analysis. Unless there are requirements to the contrary, the laboratory will use the highest calculated MDL for all instruments used for a given method as the Mng purposes. This MDL is not required for methods that are not readily spiked (e.g. pH, turbidity, etc.). Titration and gravimetric methods where there is no additional preparation involved, the MDL is based on the lowest discernable unit of measure that can be observed.

20.7.2 MDL's must be run against acceptable instrument QC, including ICV's and Tunes. This is to insure that the instrument is in proper working condition and falsely high or low MDL's are not calculated.

20.7.3 Use only clean matrix which is free of target analytes (e.g.: Laboratory reagent water, Ottawa Sand) unless a project specific MDL is required in a field sample matrix.

20.7.4 The Reporting Limit (also may be referred to as Limit of Quantitation or LOQ) should generally be between 2 and 5 times the MDL (see SOP DV-QA-024P for federal program requirements). If the MDL is being performed during method development, use this guideline to determine the Reporting Limit for the analysis. If a sample is diluted, the reported MDL is adjusted according to the dilution factor.

20.7.5 If the MDL is < 1/10 of the spike concentration for more than 10% of the analytes in the method (< 1/5 of spike recovered for DoD for water samples) the MDL must be repeated (including extraction or digestion) using a lower spike level unless the % recovery is < 50% or > 150% of the "true value". Note: The concentration of the spike will be at a level below the calibration range.

20.7.6 The calculated MDL cannot be not greater than the spike amount.

20.7.7 If the most recent calculated MDL does not permit qualitative identification of the analyte then the laboratory may use technical judgment for establishing the MDL (e.g., calculate what level would give a qualitative ID, compare with IDL (20.8), spike at a level where qualitative ID is determined and assign that value as MDL, minimum sensitivity requirements, Standard deviation of method blanks over time, etc.). Refer to SOP DV-QA-003P for details.

20.7.8 Each of the 7 spikes must be qualitatively identifiable (e.g., appear in both columns for dual column methods, characteristic ions for GCMS mass spectra, etc). Manual integrations to force the baseline for detection are not allowed.

20.7.9 The initial MDL is calculated as follows:

$$MDL = t_{(n-1,\ 1-a\ =\ 0.99)} \times (\text{Standard Deviation of replicates})$$

where $t_{(n-1,\ 1-a\ =\ 0.99)} = 3.143$ for seven replicates.

20.7.10 Subsequent to the initial MDL determination, periodic MDL verification, confirmation or determinations may be performed by the procedure in 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices (e.g., method blanks over time, single standard spikes that have been subjected to applicable sample prep processes, etc.). Refer to SOP DV-QA-003P for details.

20.7.11 Because of the inherent variability in results outside of the calibration range, TestAmerica does not recommend the reporting of results below the lowest calibration point in a curve; however, it is recognized that some projects and agencies require the reporting of results below the RL. Any result that falls between the MDL and the Reporting limit, when reported, will be qualified as an estimated value.

20.7.12 Detections reported down to the MDL must be qualitatively identified.

20.7.13 MDLs and Reporting limits are adjusted in LIMs based on moisture content. Adjustments for sample aliquot size are made if the aliquot used is less than 80% or more than 120% of the standard aliquot, or if it is required for a given project.

20.8 INSTRUMENT DETECTION LIMITS (IDL)

20.8.1 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.

20.8.2 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.

20.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

20.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

20.9.1 Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified or see section 20.6 for other options. 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. 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 (See 20.6). MDLs must be verified at least annually (see SOP DV-QA-024P for federal program frequency requirements).

20.9.2 When a Reporting limit is established, it must be initially verified by the analysis of a low level standard or QC sample (LCS at 1-2 the reporting limit) and annually thereafter. Unless there are requirements to the contrary the acceptance criteria is $\pm$ 50%. The annual requirement is waved for methods that have an annually/quarterly verified MDL.

20.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis 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.

For GC, HPLC and IC methods, there must be sufficient separation between analyte peaks so as to not misidentify analytes. In the mid-level standard, the distance between the valley and peak height cannot be any less than 25% of the sum of the peak heights of the analytes. This also applies to GCMS in the case where the two compounds share the same quantitation ion.

Note: Some analytes do not separate sufficiently to be able to identify or quantitate them as separate analytes (e.g. m-xylene and p-xylene) and are quantitated and reported as a single analyte (e.g. m,p-xylenes).

Once the analyst has determined that the instrument is in optimum working condition through calibration and calibration verification procedures, he or she uses a mid-range calibration or calibration verification standard to establish the retention times for each of the individual analytes in a method. The analyst makes three injections of the same standard over a 72-hour (24 hr period for 300.0) period, tabulating the retention times for each analyte for each of the three injections. The width of retention time window is normally the average absolute retention time ± 3 Standard Deviations (see SOP DV-QA-024P for federal program requirements). A peak outside the retention time window will not be identified by the computer as a positive match of the analyte of interest.

It is possible for the statistically calculated RT window to be too tight and need to be adjusted based on analyst experience. In these instances method default retention time windows may be used (e.g., for 8000 series methods a default of 0.03 minutes may be used, and EPA CLP 0.05 minutes is used). The same concept is applied when any peak outside of that window will not be identified by the computer as a positive match.

The calibration verification standard at the beginning of a run may be used to adjust the RT for an analyte. This is essentially re-centering the window but the size of the window remains the same. The RTs are verified when all analytes are within their RT windows and are properly identified.

20.11 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, spectrochemical, and specific electrode response factors.

20.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

20.12.1 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 analytical result" (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 result is believed to lie within at least a 95% confidence level with the coverage factor k=2.

20.12.2 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.

20.12.3 The 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). 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.

20.12.4 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 a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l.

20.12.5 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.

20.13 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.

20.13.1 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 SOPs P-I-006, Virus Protection Policy, P-I-008, internet Security Policy, and P-I-003 Computer Systems Account and Naming Policy. The laboratory is currently running Quantims 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 IBM DB-2 which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

20.13.1.1 **Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, 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.

  Note: "Commercial off-the-shelf software in use within the designed application range is considered to be sufficiently validated." *From NELAC 2003 Standard.* However, laboratory specific configurations or modifications are validated prior to use.

- In order to assure accuracy, all data entered or transferred into the LIMS data system goes through a minimum of two levels of review.
- The QA department performs random data audits to ensure the correct information has been reported.
- Changes to reports are documented in a Non-Conformance Memo. Details are specified in SOP DV-QA-019P, Result and Report Revisions.

- Analytical data file security is provided through three policies.
  - The first policy forbids unauthorized personnel from using laboratory data acquisition computers.
  - The second policy is the implementation of network passwords and login names that restrict directory access.
  - The third layer is maintained through the LIMS and includes the use of username/password combinations to gain access to the LIMS system, the fact that all data in the LIMS is associated with the user to added/reviewed the data, and the restriction of review authority of data.
- All software installations will be in accordance with any relevant copyright licensing regulations.
- All software installed on any computer within the laboratory must be approved by the Information Technology Department regional support technician assigned to the laboratory. Shrink-wrapped or otherwise sealed OEM software that is directly related to instrument usage does not need approval but the Information Technology department must be notified of the installation.
- Anti-virus software shall be installed on all servers and workstations. The anti-virus software shall be configured to check for virus signature file and program updates on a daily basis and these updates will be pushed to all servers and workstations. The anti-virus software will be configured to clean any virus-infected file if possible, otherwise the file will be deleted. Disks and CDs brought from any outside source that are not OEM software must be scanned for viruses before being accessed.

- **Interlab LIMS Permissions Policy**
  - PURPOSE - The purpose of this policy is to provide a mechanism for maintaining the integrity of information contained in each laboratory's LIMS while providing the necessary access for information sharing to staff at other laboratory facilities.

- DEFINITIONS - Host Laboratory: The laboratory facility that 'owns' the LIMS system or 'hosts' a project/job.
- POLICIES

  (a) All permissions for the laboratory's LIMS system must only be granted by a representative of that laboratory.
  - If someone outside of the host lab needs permissions for Project Management or other uses, they must go through the Lab Director or his/her designated representative.
  - Permissions must never be granted without the knowledge of the host laboratory.

  (b) Only laboratory analytical or QA staff from the home laboratory may have edit permissions for laboratory analysis data.

  (c) Any changes made in laboratory's LIMS system:
  - Must be documented and traceable.
  - If made by staff of an affiliate lab, written permission from the home lab to make the changes (email approval is sufficient) is required.
  - No corrections may be made in another laboratories system without their knowledge.

  (d) Data qualifiers in laboratory reports must only be corrected, edited, etc. by the staff at the host laboratory.

  (e) Full analytical data "View" only permissions may be granted to outside Project Management and Sales staff. Search permissions may also be granted so status may be checked.

  (f) All qualifiers must be approved by QA staff before adding to standard reference tables. In addition, changes to qualifiers in the LIMS master list must be approved by corporate QA.

20.13.1.2 Ensure Information Availability: Protection against loss of information or service through scheduled back-ups, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

- Insured by timely backup procedures on reliable backup media, stable file server network architecture, and UPS protection
- UPS Protection: Each fileserver is protected by an appropriate power protection/backup unit. In the event of a power outage, there is approximately 15-30 minutes of up-time for the servers prior to shutdown. This allows for proper shutdown procedures to be followed with the fileservers.
- File Server Architecture
  - All files are maintained on multiple Windows 2000 or newer servers which are secured physically in the Information Technology office. Access to these servers is limited to members of the Information Technology staff.
  - All supporting software is maintained for at least 5 years from the last raw data generated using that software. [Length of time is dependent on local regulations or client requirements (e.g., OVAP requires 10 years).]
- System Back-up Overview and Procedures
  - Data from both servers and instrument attached PC's are backed up and purged in compliance with the corporate back-up policy.

- A Maintenance Plan has been defined to create a daily archive of all data within the LIMS database to a backup location. This backup is initiated automatically by either the database or back-up system.
- Backup tapes will be stored in compliance with the corporate Data Backup Policy. Backup verifications are carried out in accordance with the corporate Data Backup Policy.
- Instrument data back-ups are verified on a periodic basis by the QA department when performing electronic data audits. The audit takes place on data that has been moved to a back-up location ensuring that it has been moved. Refer to SOP DV-QA-025P, Electronic Data Backup.

20.13.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

- All servers are located in a secure area of the IT department offices. Access to the servers is limited to IT staff members, lab directors, the President and Vice President of Operations. Individuals with access at TestAmerica Denver are: Wendlee Fischler, Michael Sara, Mark Dean, Damien Kaaz, Conner Sargent, Stephen Madrid, Jeff Woodruff, Nathan Mead, and Joanne Thomas.
- The company website contains SSL (Secure Socket Layer) encryption for secure website sessions and data transfers.
- The reporting portion of the LIMS system requires a project manager to enter their unique password anytime they create a report that displays a signature on it (.PDF).
- Electronic documents such as PDF files and electronic data deliverables will be made available to clients via the secure web site. The logon page for this web site contains an agreement that the customer must accept before they will be logged on which states that the customer agrees not to alter any electronic data made available to them.

20.13.2 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. Details for data review at TestAmerica Denver are defined in SOP DV-QA-0020, Data Review.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then entry into the LIMS is verified by the second level reviewer. The review checklists are signed by both the analyst and second level reviewer to confirm the accuracy of the manual entry(s) as well as review the data for technical accuracy. Refer to SOP DV-QA-0020, Data Review for details of the review process.

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices and TestAmerica Denver SOP DV-QA-0033.

Analytical results are reduced to appropriate concentration units specified by the PM in LIMS, 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 second level review staff. Calculations

and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

20.13.2.1 All raw data must be retained in the batch folder and computer file (if appropriate). All information pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed and initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

20.13.2.2 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. The units "mg/L" and "mg/kg" are the same as "parts per million (ppm)". The units "µg/L" and "µg/kg" are the same as "parts per billion (ppb)." Some low level methods utilized primarily for aqueous samples are reported in "ng/L", which are the same as "parts per trillion" (ppt). For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1%.

- Several environmental methods, such as color, turbidity, conductivity, use very specific, non-concentration units to report results (e.g., NTU, umhos/cm etc).
- Occasionally, the client requests that results be reported in units which take into account the measured flow of water or air during the collection of the sample. When they provide this information, the calculations can be performed and reported.

20.13.2.3 Refer to SOP DV-QA-004P, Rounding and Significant Figures for details regarding the number of significant figures to report for each step in the process.

20.13.2.4 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.

20.13.2.5 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 prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

20.13.3 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 13.
- 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 Department Manager/QA Manager at the facility. The QA Department controls all worksheets following the procedures in Section 6.

20.13.4 Review / Verification Procedures

Review procedures are out lined in several SOPs (DV-QA-0003, Sample Management and Chain of Custody, DV-QA-0020, Data Review, and DV-QA-0022, Package Assembly), 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, SOP DV-QA-0033, Acceptable Manual Integration Practices. The general review concepts are discussed below, more specific information can be found in the SOPs.

20.13.4.1 The data review process at TestAmerica Denver starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information. Refer to SOP DV-QA-0003.

20.13.4.2 The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add data qualifiers if applicable (see Appendix 7 for list of common data qualifiers). To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. TestAmerica Denver performs second level review on all batches, verifying 100% of data manually entered into LIMS and at least 10% of data that is automatically uploaded to the LIMS. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. 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

20.13.4.3 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager, Technical Manager, or Department Manager for further investigation. Corrective action is initiated whenever necessary. SOP DV-QA-018P, *Repeat Analysis and Reporting* provides detail on this process.

20.13.4.4 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

20.13.4.5 As a final review prior to the release of the report, the Project Manager reviews the report 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 chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met. The following are some examples of chemical relationships that are reviewed (if data is available):

- Total Results are $\geq$ Dissolved results (e.g. metals)
- Total Solids (TS) $\geq$ TDS or TSS
- TKN $\geq$ Ammonia
- TKN $\geq$ total organic nitrogen
- TKN = ammonia + total organic nitrogen
- Total Phosphorus $\geq$ Orthophosphate
- COD $\geq$ TOC
- Total cyanide $\geq$ Amenable Cyanide
- TDS $\geq$ individual anions
- TDS $\geq$ total alkalinity
- TDS $\geq$ hardness
- Hexavalent chromium $\leq$ total chromium

20.13.4.6 Some federal programs require independent review of a percentage of the report packages by the QA Department (see SOP DV-QA-024P). The Project Manager then signs the final report. (*Also see section 26 on Reporting Results*). When complete, the report is sent out to the client.

20.13.4.7 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 20-3.

20.13.5 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 SOP CA-Q-S-002 and SOP DV-QA-0033, *Acceptable Manual Integration* Practices as the guidelines**.**

20.13.5.1 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.

20.13.5.2 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 principals and policy and is grounds for immediate termination.

20.13.5.3 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.

20.13.5.4 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.

Figure 20-1. Example - Demonstration of Capability

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Analyst Demonstration of Capability

Certification Statement

8270C-SIM - 8270C-SIM
SOP:
Matrix: Water

Date: 11-Dec-07

STL - Denver laboratory
4955 Yarrow Street
Arvada, CO 80002
(303) 736-0100

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 TestAmerica Quality Assurance Plan, has met the Initial or Ongoing Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification following the TestAmerica SOP
3. A copy of the laboratory-specific SOP is available for all personnel on-site.
4. The data associated with the initial/ongoing demonstration of capability are true, accurate, complete and self-explanatory (*). These data are attached to this certification statement.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Comments/Observations:

Analyst's Name	Signature	Date
Technical Director's Name	Signature	Date
QA Manager's Name	Signature	Date

* *True: Consistent with supporting data.*
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices..
Complete: Includes the results of all supporting performance testing.
Self-explanatory: Data properly labeled and stored so that the results are traceable and require no additional explanation.

12/11/2007 *Page 1 of 4*

Figure 20-2.

Example - New Method / Additional Analyte Checklist

New Method / Additional Analyte Checklist

The following items are **required** to be completed prior to the acceptance of client samples. Fill in any blanks that do not apply with "NA". Provide associated instrument QC when samples or QC samples are analyzed (includes run log).

New Method _____ Added Analytes _____

1_____ Standard Operating Procedure

- Note: For additional analytes, a **ROMD [or whatever an internal communication memo is named in your lab]** can be used to add the analytes, include RL and matrix.
  - _____ Analysis SOP
  - _____ Preparation SOP
  - _____ SOP for any other relevant process
  - _____ Pages from any applicable logbooks (instrument, standards, etc)

2_____Evaluation of Selectivity. As applicable: e.g. Retention Time Window Study, second column confirmation, Interelement correction checks, spectral or fluorescence profiles, etc.

3_____ Initial Calibration Curve (Include Tune verification or similar (e.g. degradation checks) if applicable)

4_____ Method Detection Limit (MDL) Study (summary and raw data)

- _____ Water
- _____ Soil
- _____ Other

5_____ Real Sample and MS, MSD ***(CA ELAP Requirement)***

- Tap Water for water only methods
- Local Soil sample for SW-846 methods (if applying for soil or soil/water)
- Local water sample may be used in lieu of tap water if it is a non- drinking water method
- Does not have to contain the target analytes

6_____ Reporting Limit Verification standard

- Spike a blank matrix at the RL and process through the entire method. MDL study should be able to be used if recovery is good. Note the spike level(s) and recovery(~~y~~ies)

7_____ Demonstration of Capability (DOC) per analyst (Precision and Accuracy (P&A) verification)

- 4 LCS for each matrix – most acceptance criteria are in the methods. The MDL study may be used if DOC criteria are met.
- Non-Standard methods – 3 x (1 LCS at LOQ-25%, 50%, 75% of the calibration range + Blank) prepared each day. (see NELAC Chpt 5, appendix C.3.3 (b))

8_____ Acceptable PT sample(s) if available

Notes: PT sample required for all new methods
PT sample required for all new analytes under NELAP

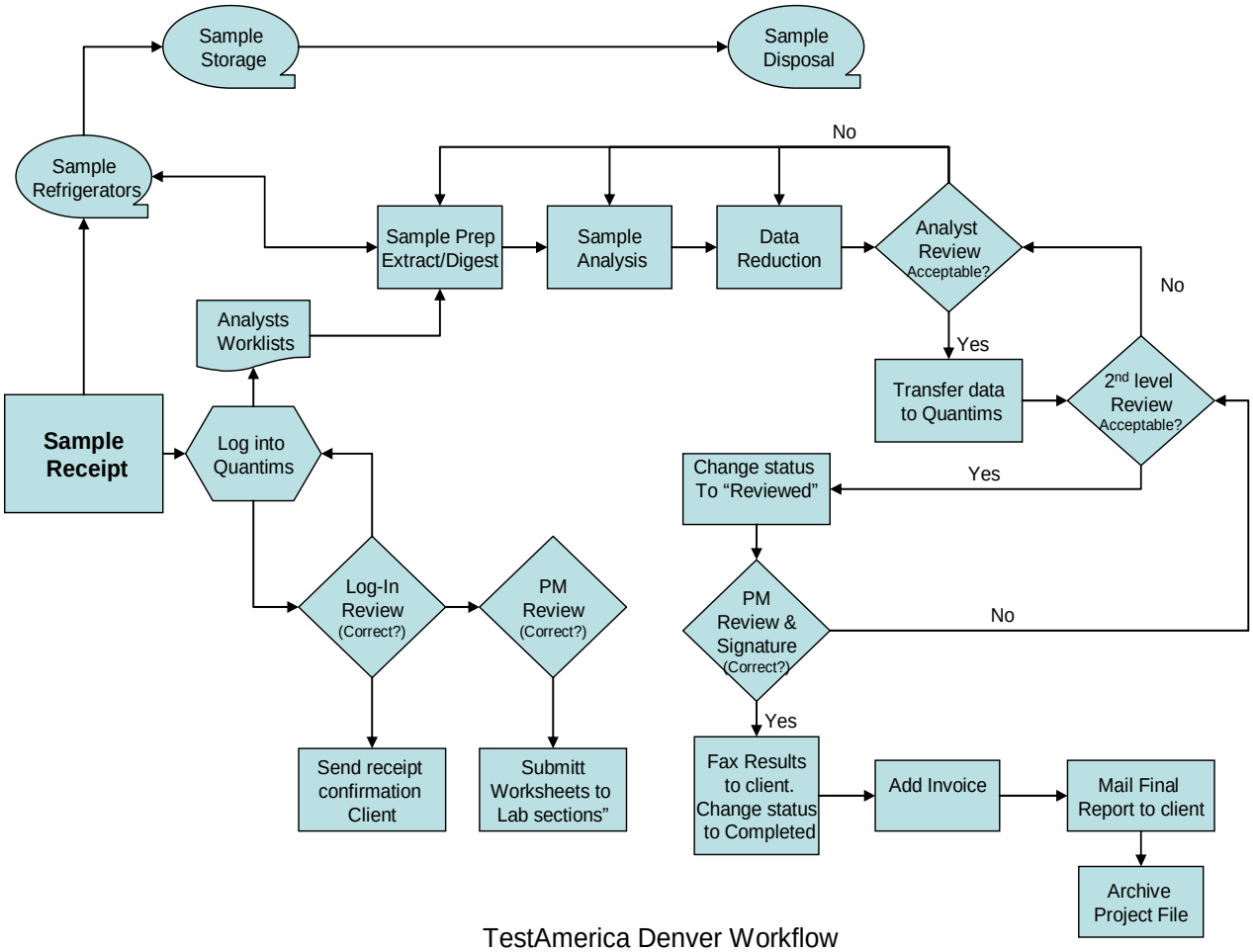
Submitted by _____ Date _____

9_____ Certification/Approval from Regulatory Agency where available.

QA Review / Acceptance _____ **Date** _____

Figure 20-3.

Work Flow



TestAmerica Denver Workflow

SECTION 21

EQUIPMENT (AND CALIBRATIONS)
(NELAC 5.5.5)

21.1 OVERVIEW

TestAmerica 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 method SOPs, in SOP DV-QA-024P for federal programs, and in Appendix 4. A list of laboratory equipment and instrumentation is presented in Table 21-1*.*

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

21.2 PREVENTIVE MAINTENANCE

21.2.1 TestAmerica Denver follows a well-defined 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.

21.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, 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.

21.2.2.1 Calibrations, routine maintenance, and adjustments are part of the analysts' and Department Managers' responsibilities. However, service contracts may be in place for some instruments to cover any major repairs.

21.2.2.2 High purity gases, reagents, and spare parts are kept on hand to minimize repair time and optimize instrument performance.

21.2.3 Table 21-2 summarizes the schedule for routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be 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.)

21.2.4 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.

21.2.4.1 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.

21.2.4.2 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.).

21.2.4.3 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.

21.2.5 In addition, the maintenance records contain:

- The identification of the instrument/equipment **(**instrument's Serial Number and Model Number)
- The date the instrument/equipment was put into use.
- If available, the condition when the instrument was received (e.g. new, used, reconditioned).
- Required maintenance is listed in the maintenance logbooks, as well as any maintenance performed.

21.2.6 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 (refer to Sections 12 and 13).

21.2.7 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 using the procedures outlined in Section 8.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

21.3 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, incubators, 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.

21.3.1 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 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 annually by an outside calibration laboratory to NIST standards.

All balances are serviced annually by a 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. Refer to SOP DV-QA-0014, *Balance Calibration Check*.

21.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to $\pm$ 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 logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

21.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, electronic thermometers, digital probes and thermocouples are calibrated quarterly refer to SOP DV-QA-0001, *Thermometer Calibration Procedure*.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer has increments of 0.2 ºC, and has a range applicable to all 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 incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in SOP DV-QA-0001, *Thermometer Calibration Procedure.*

21.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day on a continual basis. Refer to SOP DV-QA-0012, *Monitoring Refrigerator Temperature and Power Failure Contingency Plan.*

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and < 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks posted on or near the device.

21.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are checked for accuracy at least quarterly.

The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.

These devices are given unique identification numbers, and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis. Any device not regularly verified can not be used for any quantitative measurements. Refer to SOP DV-QA-0008, *Calibration and Verification of Mechnical Pipettes.*

21.3.6 Autoclaves

TestAmerica Denver uses an autoclave for sterilization of microbiological equipment and used media only. All information regarding the autoclave is maintained in the Autoclave, Coliform lot,

and Monthly check logbook. The information recorded includes the date, contents, maximum temperature, total run time and the analyst's initials.

Demonstration of sterilization of the autoclave is performed each time of use with a Diack sterilization monitor, a maximum reading thermometer, and temperature sensitive tape. On a monthly basis, spore strips are used for the determination of effective sterilization.

The autoclaves timing device is checked on a monthly basis against a clock/watch and the actual time elapsed is documented.

Any maintenance that is performed on the autoclave (internally or by service contract) is recorded in the maintenance section of the check logbook.

21.4 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. Detailed information regarding calibration models and calculations can be found in SOP CA-Q-S-005.

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 13).

Note: Instruments are calibrated initially and as needed after that and at least annually.

21.4.1 CALIBRATION STANDARDS

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. However, the general procedures are described below.

21.4.1.1 For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods. If a reference or mandated method does not specify the number of calibration standards, the minimum number is three, not including blanks or a zero standard. All of the standard solutions are prepared using Class A volumetric

glassware, calibrated pipettes, and/or microsyringes and appropriate laboratory quality solvents and stock standards.

21.4.1.2 Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. TestAmerica Denver uses Veritas Standards Log software for standards tracking. It is maintained for each department, containing concentration, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.

21.4.1.3 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).

21.4.1.4 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 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The lowest calibration standard must be at or below the reporting limit.

21.4.1.5 Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration will then involve the analysis of each of these sets of the appropriate number of standards.

21.4.1.6 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 Disodium Iminodiacetate (IDA) analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

21.4.2 CALIBRATION FOR ORGANIC METHODS (GC, HPLC, GC/MS)

21.4.2.1 Many of the organic analytical methods utilize an internal standard calibration (GCMS and some GC). Because of the complex nature of the multipeak chromatograms produced by the method, some instruments necessitate the use of external standard calibration (most GC and HPLC). Surrogate compounds are included in the calibration processes for all appropriate organic analyses. For more details on the calibration types listed below, refer to SOP No. CA-Q-S-005, Calibration Curves.

21.4.2.2 Once the operating parameters have been established according to the method, each instrument is calibrated for the appropriate method. The analyst prepares five or more standard solutions at various concentrations containing all of the analytes of interest, internal standards, and surrogates that are appropriate for the method. Note: There are a several EPA methods that have different requirements and are exceptions (e.g. EPA 547) where a minimum of 3 calibration standards are prepared and analyzed.

21.4.2.3 The standard solutions are introduced into the instrument in the same manner as samples are; whether it be by direct injection, by headspace analysis, or by purge and trap. The calibration factor (CF) for methods that use external standards, and the response factor (RF) for methods that use internal standards are calculated for the five standards.

- External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the response to the amount of analyte in the calibration standard is defined as the Calibration factor (CF).

- Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC and HPLC methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. The internal standard solution will contain one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution (e.g., not all internal standards need to be at the same concentration in this solution). The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. The volume of

the solution spiked into sample extracts should be such that minimal dilution of the extract occurs (e.g., 10 uL of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations).

An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard should produce an instrument response (e.g., area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This should result in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP No. CA-Q-S-005, Calibration Curves, for specific calculations.

21.4.2.4 Policies regarding the use of calibration standard results for creating the calibration curve are as follows:

- A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting level must be elevated to be the lowest calibration standard used for calibration.

- The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
- Mid-calibration standards may not be excluded unless an obvious reason is found, i.e., cracked vial, incorrectly made, etc. The failed standard should be re-run immediately and inserted into the initial calibration. If not useful, recalibration is required.

21.4.2.5 Percent RSD Corrective Action

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

21.4.2.5.1 The first step is generally to check the instrument operating conditions. This option will apply in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

21.4.2.5.2 If the RSD for any analyte is greater than the acceptance criteria in the applicable analytical method or SOP, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

21.4.2.5.3 A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

Note: When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

21.4.2.6 Alternatively, the least squares regression may be used to determine linearity. A five point line must result in a correlation coefficient (r) of 0.990 or better using the least squares method to be considered acceptable. In many cases it may be preferred that the curves be forced through zero (not to be confused with including the origin as an additional data point, which is not allowed). See SOP DV-QA-024P for requirements for federal programs.

Note: EPA method 8000B does not allow forcing through zero however the agency has revaluated this position and has since changed this stance to allow forcing through zero. In addition, from EPA Method 8000C: "However, the use of a linear regression or forcing the regression through zero may NOT be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards.").

21.4.2.7 Instead of a linear curve model (either Average RF or least squares regression), a second order curve (Quadratic) may be used (and preferred) as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (COD or r^2) for the quadratic curve must be at least 0.99 for it to be considered acceptable. For more details on the calculations see Calibration Curve SOP CA-Q-S-005. Some limitations on the use of Quadratic Curve fits:

21.4.2.7.1 Care MUST be exercised to assure that the results from this equation are real, positive, and fit the range of the initial calibration.

21.4.2.7.2 They **may not** be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).

21.4.2.7.3 They **may not** be used to compensate for detector saturation. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1^{st} order fit or average RF.

21.4.3 Calibration for Inorganic Analyses

EPA Method 7000 from EPA SW-846 is a general introduction to the quality control requirements for metals analysis. For inorganic methods, quality control measures set out in the individual methods and in the *Standard Methods for the Examination of Water and Wastewater* (20th Edition) may also be included. Standard Operating Procedures for the analysis and the quality control documentation measures are kept in the analyst group's reference binders, as well as posted on the network at G:\QA\Read\SOPs\ESOPs.

In general, inorganic instrumentation is calibrated with external standards. Some exceptions would be Inductively Coupled Plasma (ICP), Inductively Coupled Plasma Mass Spec (ICPMS), and Ion Chromatography Mass Spec (ICMS). These analyses may use an internal standard to compensate for viscosity or other matrix effects. While the calibration procedures are much the same for inorganics as they are for organics, CF's or RF's are not used. The calibration model in 21.4.2.6 is generally used for most methods, however in some instances the model from section 21.4.2.7 may be used. A correlation coefficient (r) of 0.995 or greater must be used to accept a calibration curve generated for an inorganic procedure. Correlation coefficients are determined by hand-held scientific calculators or by computer programs and documented as part of the calibration raw data. Coefficients of calibration curves used for quantitation must be documented as part of the raw data. Curves are not allowed to be stored in calculator memories and must be written on the raw data for the purposes of data validation. Detailed information regarding calibration models and calculations can be found in SOP CA-Q-S-005.

21.4.3.1 "Calibrations" for titrimetric analyses are performed by standardizing the titrants against a primary standard solution. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (20th Edition) for more information.

21.4.3.2 Spreadsheets that are used for general chemistry calculations must have all cells containing calculations locked to prevent accidental changes to the calculations.

21.4.3.3 Instrument technologies (e.g. ICP) with validated techniques from the instrument manufacturer or other methods using a zero point and single point calibration require the following:

21.4.3.3.1 The instrument is calibrated using a zero point and a single point calibration standard.

21.4.3.3.2 The linear range is established by analyzing a series of standards, one at the reporting limit (RL).

21.4.3.3.3 Sample results within the established linear range do not need to be qualified.

21.4.3.3.4 The zero point and single standard is run daily with each analytical batch.

21.4.3.3.5 A standard at the RL is analyzed daily with each analytical batch and must meet established acceptance criteria.

21.4.3.3.6 The linearity is verified at a frequency established by the manufacturer or method. See SOP DV-MT-0012, *ICP Analysis for Trace Metals by Methods 6010 and 200.7.*

21.4.4 Calibration Verification

The calibration relationship established during the initial calibration must be verified at periodic intervals as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. 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.

Note: The process of calibration verification referred to 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, and is not appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses.

21.4.4.1 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 or standard that can be injected within 12 hours of the beginning of the shift.

21.4.4.2 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.

21.4.4.3 The acceptance limits for calibration verifications can be found in each method SOP. As a rule of thumb: GCMS $\pm$ 20%, GC and HPLC $\pm$ 15%, Inorganics: $\pm$ 10 or 15%. Actual methods may have wider or tighter limits; see the method SOP for specifics.

21.4.4.4 If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the initial calibration to quantitate sample results.

21.4.4.5 If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

21.4.4.5.1 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.

21.4.4.5.2 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, for some methods a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit specific details for utilizing this option are described in SOP DV-QA-27P, *Standardized CCV Criteria for GC and HPLC.*

21.4.4.6 Verification of Linear Calibrations

Calibration verification for linear 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. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP. 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.

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{(\text{CF(v) or RF(v)}) - (\text{Avg. CF or RF})}{(\text{Avg. CF or RF})} \times 100$$

Where: CF(v) or RF(v) = CF or RF from verification standard

Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result} - \text{True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

21.4.4.7 Verification of a Non-Linear Calibration

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations described in 21.4.4.6 above.

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.

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.

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. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

21.5 POLICY ON 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.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it will not be reported as a TIC. If the compound is reported on the same form as true TICs, it must 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.

21.5.1 Use the following guidelines for making tentative identifications

21.5.1.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

21.5.1.2 The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

21.5.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

21.5.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

21.5.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

The concentration of any non-target analytes identified in the sample (see above) should be estimated. The same formulae as calibrated analytes should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

Note: The above guidelines above are from EPA SW846 III edition, method 8260B.
For general reporting if TICs are requested, the ten (10), largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard will be termed “Tentatively Identified Compounds” (TICs). More or fewer TICs may be identified based on client requirements.

21.5.2 TIC Reporting Limits

In general Reporting limits cannot be specified because of the unknown nature of the TIC. Any reporting limit that is reported can only be evaluated as an estimate as the quantitation is based on the assumption that the TIC responds exactly as the IS responds which is most likely not the case. In general, it is not recommended to set a Reporting limit at too low of a concentration as it gives a false impression.

21.6 POLICY ON 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.

21.6.1 The concentration of the BFB or DFTPP must be at or below the concentrations that are referenced in the analytical methods. Part of the purpose of the tune is to demonstrate

sensitivity and analyzing solutions at higher concentrations does not support this purpose. Tune failures may be due to saturation and a lower BFB/DFTPP concentration may be warranted.

21.6.2 Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex +/- 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

21.6.3 **Other Options or if Auto Tune Fails**:

21.6.3.1 Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. In this case, manually identify and average the apex peak +/- 1 scan and background correct as in 21.6.4 above. This is consistent with EPA 8260 and 8270.

21.6.3.2 Or the scan across the peak at one half peak height may be averaged and background corrected. This is consistent with Standard Methods 6200, EPA 624 and EPA 625.

21.6.3.3 Adjustments such as adjustments to the repeller and ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as all of the subsequent injections in the 12 hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than 2 tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in instrument log.

21.6.3.4 A single scan at the Apex (only) may also be used for the evaluation of the tune. For SW 846 and EPA 600 series methods, background correction is still required.

21.6.3.5 Cleaning the source or other maintenance may be performed and then follow steps for tune evaluation above. Note: If significant maintenance was performed, see methods 8000B or 8000C then the instrument may require recalibration prior to proceeding.

21.6.4 Tune evaluation printouts must include the chromatogram and spectra as well as the Tune evaluation information. In addition, the verifications must be sent directly to the printer or pdf file (no screen prints for DFTPP or BFB tunes). This ability should be built into the instrument software.

21.6.5 All MS tune settings must remain constant between running the tune check and all other samples. It is recommended that a separate tune method not be used, however a separate method may be used as long as the MS conditions between the methods are the same as the sample analysis method and tracked so any changes that are made to the analysis method are also made to the tune method.

Table 21-1.

TestAmerica Denver Equipment and Instrumentation

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
ICP	Thermo Jarrell Ash (020) S/N 225390	61E Trace	1994	Yes	6010B, 200.7
	Thermo Jarrell Ash (016) S/N 389590	61E Trace	1997	Yes	6010B, 200.7
	Thermo Fischer (025) S/N 20062004	ICP 6500	2006	Yes	6010B, 200.7
	Thermo Fischer (026) S/N 20063207	ICP 6500	2006	Yes	6010B, 200.7
ICP/MS	Agilent ICP-MS (024) S/N JP51201530	7500 Series	2006	Yes	6020, 200.8
	Perkin Elmer SCIEX (004) S/N 305970360	ELAN 6000	1997	Yes	6020, 200.8
Mercury Analyzer	Cetac CVAA (023) S/N 030504QTA	M-7500	2005	Yes	7470, 7471A, 245.1, 245.2
	Perkin Elmer (019) S/N 4025	FIMS FIAS 400	1996	Yes	7471A, 7470, 245.1, 245.2
Ion Chromatograph	Dionex (IC3) S/N 98040510	DX-120	1997	Yes	300.0, 9056
	Dionex (IC4) S/N 056537	AS 50	2000	Yes	Hydrazine, MMH, UDMH
	Dionex (IC5) S/N 0106180	LC 20	2002	Yes	300.0, 314.0, 9056
	Dionex (IC6) S/N 03100162	ICS 2000	2003	Yes	300.0, 9056
	Dionex (IC7) S/N 03100161	ICS 2000	2003	Yes	300.0, 314.0, 9056
	Dionex (IC8) S/N 08020954/08020762	ICS 2000 RFIC	2008	Yes	300.0, 314.0, 9056
	Dionex (IC9) S/N 08020888	ICS 3000	2008	Yes	300.0, 314.0, 9056
TOC	LECO (LEC) S/N 3097	C632 (Solid)	2007	Yes	5310B, 9060
	Shimadzu (SHI3) S/N H51404335027	TOC-V_{CPN}	2005	Yes	415.1, 9060, 5310B
	Shimadzu (SHI2) S/N 414445340	TOC-VCSH	2004	Yes	415.1, 9060, 5310B

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
TOX	MCI S/N 43F30588	TOX-10	1987	No	9020B, 9021, 9023
	Thermo Euroglass (Thermo 1) S/N 993752	ECS 1200	1997	Yes	9020B, 9021, 9023
	Thermo Scientific (Thermo 2) S/N 993728	ECS 1200	2004	Yes	9020B, 9021, 9023
UV/VIS	Thermo UV1 S/N 114403	UV1	2004	Yes	365.1
	Alpkem (Alp1) S/N 908893427	A002393	1997	Yes	325.2, CN, Phenol
	Alpkem (Alp2) S/N 917893398	A002393	1997	Yes	353.2, NH_3/TKN, 351.2, 351.3
	Konelab S/N P0518697	Model 20	2003	Yes	365.1, 365.3, 375.4, ASTM D516-02
	Astoria Pacific Analyzer S/N 200052	Astoria 2	2005	Yes	351.2, 353.2, 365.1
Ion Analyzer	Orion Research S/N PX94A	EA940	1985	No	340.2, 4500-F C, RedOx Potential
Autotitrator (pH, Alkalinity, Conductance)	Man-Tech (AT2)	PC – Titrate PC-1000	2000	Yes	9040B, 9045C, 150.1, 2320B, 310.1, 310.2, 2510B, 9050A, 120.1
PH Meter	Thermo Orion	PerpHecTROSS Sure Flow pH/ ATC 93700	2003	No	9040B, 9045C, 150.1, 4500-H B
	Thermo Orion S/N: TVT71A	SA 720	2005	No	1311
Dissolved Oxygen Meter	YSI S/N 08D100984	5100	2008	No	405.1, 5210B
	YSI S/N 02G0238	5100	2008	No	405.1, 5210B
Turbidimeter	HF Scientific S/N 104008	Micro 100	2001	No	180.1
Flashpoint	Herzog S/N 043291648	Pensky Martens Model MP-329	2003	No	1010, ASTM D93

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
Spectrophotometer	HACH S/N 990200012321	DR/2010	2007	No	354.2, 376.2, 410.4, 7196A, 3500 Fe D, 3500 Cr D, 4500 S^{-2} D
GC/MS Semivolatiles	Hewlett-Packard (B) S/N US00007283	6890 – GC 5973 – MSD	1999	Yes	8270C, 625
	Hewlett-Packard (D) S/N US00007319	6890 – GC 5973 – MSD	1996	Yes	8270C, 625
	Agilent Technologies (F) S/N US00036181	6890 – GC 5973N – MSD	1996	Yes	8270C SIM
	Agilent Technologies (K) S/N CN10332028	6890N – GC 5973 – MSD	2003	Yes	8270C, 8270C SIM, 625
	Agilent Technologies (G2) S/N CN10421078	6890N – GC 5973 – MSD	2004	Yes	8270C Best Practice
	Agilent Technologies (G4) S/N CN10438087	6890N – GC 5973 Inert – MSD	2004	Yes	8270C Best Practice
	Hewlett-Packard (Q) (S/N US0000021949	6890 – GC 5973 – MSD	2001	Yes	8270C, 625
	Hewlett-Packard (Y) S/N US00007291	6890 – GC 5973 – MSD	1996	Yes	8270C, 625
	Agilent Technologies (G5) S/N CN10605078	6890N – GC 5975 – MSD	2006	Yes	8270C, 8270C SIM, 625
GC/MS Volatiles	Agilent Technologies (C) S/N US00007315	6890N – GC 5973 – MSD	2002	Yes	8260B
	O·I Analytical S/N 14049	4552 – Purge & Trap 4660 - Concentrator			
	Hewlett-Packard (E) S/N 3336A60699	5890II – GC 5972 – MSD	1997	Yes	8260B-Water
	Tekmar	ALS 2016 S/N 90163026 LSC 2000 S/N 90151004			
	Hewlett-Packard (H) S/N 3336A60700	5890II – GC 5972 – MSD	1994	Yes	8260B-Waters
	O·I Analytical S/N 14052	4552 – Purge & Trap 4660 - Concentrator			
	Hewlett Packard (P) S/N US00007321	6890 - GC 5973 – MSD	1999	Yes	8260B
	O·I Analytical	4552 – Purge & Trap 4660 - Concentrator			

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
	Hewlett-Packard (G) S/N 3336A56276	5890 Series II - GC 5972 - MSD	1996	Yes	8260B
	Varian S/N 12751	Archon Purge & Trap O·I 4560 - Concentrator			
	Hewlett-Packard (J) S/N 3336A60701	5890II – GC 5972 – MSD	1994	Yes	8260B
	Varian S/N 12726	Archon Purge & Trap O·I 4560 - Concentrator			
	Agilent Technologies (R1) S/N LN10524033	6890N - GC 5973 Inert – MSD	1994	Yes	8260B/524
	O·I Analytical S/N 14043	4552 – Purge & Trap 4660 - Concentrator			
	Hewlett-Packard (R2) S/N 336A53965	5890II - GC 5972 – MSD	1995	Yes	8260B
	O·I Analytical S/N 14383	4552 – Purge & Trap 4660 - Concentrator			
	Hewlett-Packard (S) S/N 3336A60702	5890II – GC 5972 – MSD	1994	Yes	8260B/624
	Varian S/N 12750	Archon Purge & Trap O·I 4560 - Concentrator			
	Hewlett-Packard (Z) S/N 3336A60013	5890II – GC 5972 – MSD	1996	Yes	8260B-Waters, 524
	O·I Analytical	DPM-16 – Purge & Trap S/N C429411174 O·I 4560 – Concentrator S/N H416460186			
	Agilent Technologies (GC/MS1) S/N CN10420009	6890N – GC 5973 – MSD	2004	Yes	8260B Waters
	O·I Analytical S/N 14593	4552 – Purge & Trap 4660 - Concentrator			

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
	Hewlett-Packard (I) S/N 2643A11361	5890 – GC	2003	Yes	Volatile Screening
	Tekmar Dohrmann Headspace Autosampler S/N US03038002	7000			
	Hewlett-Packard (T) S/N 2750A14928	5890 Series II – GC	2001	Yes	Volatile Screening
	Tekmar Dohrmann Headspace Autosampler S/N US01198005	7000HT			
GC Semivolatiles	Hewlett-Packard (A) S/N 2750A16891	5890 Dual FID	1987	Yes	8015 Alcohol
	Hewlett-Packard (C) S/N US00029514	6890 Dual ECD	1999	Yes	608, 8081A
	Hewlett-Packard (D) S/N DE00020818	6890 Dual NPD	1997	Yes	614, 8141A
	Agilent Technologies (D2) S/N US10521035	6890N Dual NPD	2004	Yes	614, 8141A
	Hewlett-Packard (E) S/N 3121A35858	5890II Dual ECD	1992	Yes	504.1, 8011
	Hewlett-Packard (M) S/N US00024143	6890 Dual ECD	1999	Yes	615, 8151A
	Agilent Technologies (P1) S/N US10418019	6890N Dual ECD	2004	Yes	608, 8081A
	Agilent Technologies (P2) S/N US10418024	6890N Dual ECD	2004	Yes	608, 8081A

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
	Agilent Technologies (P3) S/N US10418023	6890N Dual ECD	2004	Yes	608, 8082
	Hewlett-Packard (R) S/N 3336A55030	5890II Dual ECD	1994	Yes	8151
	Hewlett-Packard (T) S/N 2536A05971	5890 Dual NPD	1999	Yes	607, 8070A
	Hewlett-Packard (U) S/N US00063217	5890II Single FID	1999	Yes	8015B DRO
	Hewlett-Packard (V) S/N 2631A08686	5890 Dual ECD	1990	Yes	8081 (limited use)
	Hewlett-Packard (W) S/N 3126A36250	5890II Dual ECD	1990	Yes	608, 8082
	Hewlett-Packard (Z2) S/N 3336A51924	5890II Dual FID	1990	Yes	8015B DRO
GC Volatiles	Hewlett-Packard (B) S/N 3019A28634	5890 Series II Dual PID / FID	1990	Yes	8021 GRO
	Tekmar	LSC 2000 Concentrator S/N 90142014\ALS 2016 P & T S/N 89108007			
	Hewlett-Packard (F)	5890II Dual ELCD	1990	Yes	Retired/ Parts only
	Tekmar	LSC 2000 Concentrator S/N 88305008 ALS 2016 P & T S/N 90129029			
	Hewlett-Packard (H)	5890A Dual PID Single FID	1988	Yes	8015, 8021B Aromatics, 8021B GRO
	Tekmar	LSC 2000 Concentrator S/N 90100002/ALS 2016 P & T S/N 88145007			

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
	Hewlett-Packard (J) S/N US00026194	6890 Dual FID	1997	Yes	RSK-175
	Tekmar Dohrmann US02296004	HS Autosampler 7000 HT			
	Hewlett-Packard (K) S/N 2843A19497	5890A Dual PID Single FID	1988	Yes	8015, 8021B Aromatics, 8021B GRO
	Tekmar	LSC 2000 Concentrator S/N 92098003 ALS 2016 Purge & Trap S/N 92101007			
	Hewlett-Packard (L) S/N 2336A00164	5890A FID	1988	Yes	8015B GRO
	Tekmar	LSC 2000 Concentrator S/N 89283001 ALS 2016 Purge & Trap S/N 90121028 ALS 2032 Purge & Trap S/N 94300004			
	Hewlett-Packard (P) S/N 2518A05337	5890A Dual PID Single FID	1990	Yes	8015B, 8021B Aromatic, 8021B GRO
	Tekmar	LSC 2000 Concentrator S/N 89310005 ALS 2016 Purge & Trap S/N 90100036			
	Agilent Technologies (S-1) S/N US10341120	6890 Dual PID/ Dual ELCD	2003	Yes	8021B
	O I Analytical S/N 14046	4552 – Purge & Trap 4660 - Concentrator			
	Hewlett-Packard (Y) S/N 2843A19484	5890A PID/FID	1988	Yes	Screen only

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
	Tekmar	LSC 3000 Concentrator S/N 93132006 ALS 2016 Purge & Trap S/N 91112002 ALS 2032 Purge & Trap S/N 88145006			
HPLC	Hewlett-Packard (G) S/N DE91609974	1100 Multiple wavelength UV/ Fluorescence detectors	1999	Yes	8310
	Agilent Technologies (Q) S/N DE11120993 (Quat Pump)	1100 Multiple wavelength UV/ Fluorescence detectors	2001	Yes	8330
	Agilent Technologies (X3) S/N DE33236507 (Quat Pump)	1100 Multiple wavelength UV/ Fluorescence detectors	2004	Yes	8330
HPLC/MS/MS	Micromass/Waters 2790 HPLC Inlet S/N VB118 (LCMS1) plus Dionex AS50 Autosampler, LC30 Chromatography Oven, CD25 Conductivity Detector	Quattro Ultima	2000	Yes	8321A, 6860
	Micromass/Waters Acquity UPLC Inlet (LCMS3) S/N VAA188	Quattro Premier XE	2004	Yes	8321A
	Micromass/Shimadzu 10 Avp HPLC Inlet (LCMS2) plus Shimadzo Inlet SIL-10AD, Shimadzo UV-VIS Detector SPD-10A, Dionex Ion Chromatography ICS 2000 S/N VB304	Quattro Ultima	2001	Yes	8321A

Instrument Type	Manufacturer	Model	Purchase Date	Auto-sampler	Method Performed
	Micromass/Waters 2695 HPLC Inlet (LCMS4) S/N QAA632	Quattro Micro AP1	2006	Yes	8321A
GCMS	Agilent Technologies (GCMS X4) S/N CN10438076	6890N-GC 5973-MSD	2004	Yes	Custom
CI/MS/MS	Varian (CIMS1) S/N 1200-680	1200L MS/MS CP-3800 GC	2004	Yes	Low Level NDMA

Support Equipment

Instrument Type	Manufacturer	Model	Quantity	Location
Centrifuge	Sorvall Legend T	Sorvall Legend T	1	Metals
Hot Block	Environmental Express	SC100	11	Metals
Sonic Bath	Bransonic	Bransonic	1	Metals
Hot Block	Thermo Scientific Precision	Thermo Scientific Precision	2	Metals
Incubator	Fisher Scientific	Low Temperature Incubator	1	Wet Chemistry

Instrument Type	Manufacturer	Model	Quantity	Location
Incubator	Thermo Electron Corporation	Thermo Electron Corporation	1	Wet Chemistry
TOX Sample Preparation	Microcoulometric Titration System	Microcoulometric Titration System	5	Wet Chemistry
Cyanide Digestor	Westco Scientific Instruments, Inc.	Westco Scientific Instruments, Inc.	1	Wet Chemistry
Centrifuge	Beckman	Beckman G- D-G	1	Wet Chemistry
COD Digestor	HACH	DRB 200	1	Wet Chemistry
Digestion System w/ Controller	A I Scientific	AIM 600/AIM 500	1	Wet Chemistry
Solvent Evaporator w/Digital Temperature Control System	UA-SYS	UA-SYS Heating System S-EVAP KD	1	Wet Chemistry
Oil & grease Machine w/ SPE-DEX 3000 Controller/ Speed VAP II 9000 Solvent Evaporation System VAC Generator	Horizon Technology	3000 XL	1	Wet Chemistry
Cool Flow 25 NES Lab Kontes w/Midi Vap 2000	Scientific Glassware Instruments	Scientific Glassware Instruments	1	Wet Chemistry

Instrument Type	Manufacturer	Model	Quantity	Location
Oven	VWR	1370 GD	1	Wet Chemistry
Oven	VWR	1370 G	1	Wet Chemistry
Oven	VWR	1370 FM	1	Wet Chemistry
Oven	Fisher Scientific	Fisher Scientific	1	Wet Chemistry
Oven	Yamato	Mechanical Convection Oven DKN 810	1	Wet Chemistry
Centrifuge	IEC Clinical	IEC Clinical	1	Mass Spectrometry
Oven	VWR	1320	1	North Prep
Oven	Welbit	Welbit	1	North Prep
Oven	Fisher Scientific	Fisher Scientific	1	North Prep
Turbo Vap	Caliper Life Science	Turbo Vap II	3	North Prep

Instrument Type	Manufacturer	Model	Quantity	Location
Seperatory Funnel Rotators	Ap & R Machine Tool	Ap & R Machine Tool	5	North Prep/South Prep
Lab Ultra	ELGA	Pure lab Ultra	1	North Prep
Microwave Extraction	CEM Corporation	MARSXpress Xtraction	1	North Prep
Sonicator	Fisher Scientific	550 Sonic Dismembrator	2	North Prep
Sonicator	Heat Systems	Sonicator Ultrasonic Processor XΔ	1	North Prep
Sonicator	Misonix	Sonicator 3000	2	North Prep
Sonicator	Heat Systems	W-385	2	North Prep
Drying Oven	Blue M	Temp-O-Loy Amecling Oven	1	North Prep
N-Evap	Organomation Associates, Inc.	N-Evap II Nitrogen Evaporator	3	North Prep
Turbo Vap	Zymark	Turbo Vap A	1	North Prep

Instrument Type	Manufacturer	Model	Quantity	Location
Water Bath	Waterlow	Waterlow	2	North Prep
Muffle Furnance	Lindberg	Lindberg	1	South Prep
Shaker	New Brunswick Scientific	Innova 2100	1	MS VOA
Sonicator	Branson	Branson 2210	1	South Prep
Balance	Mettler	PE300	1	MS VOA Hood #36
Balance	Sartorious	PT600	1	MS VOA Hood #37
Balance	Ohaus	GT4100	1	Wet Chem
Balance	Mettler	PE160	1	Wet Chem
Balance	Mettler	PM4600	1	Digestions
Balance	Mettler	PC4400	1	Wet Chem

Instrument Type	Manufacturer	Model	Quantity	Location
Balance	Mettler	AE240	1	Wet Chem
Balance	Mettler	PM4000	1	Wet Chem
Balance	Sartorious	1602	1	Wet Chem
Balance	Mettler	PE600	1	GC SVOA Hood #31
Balance	Mettler	AE2400	1	Standards and Aliquoting Room Hood #58
Balance	Mettler	PM4000	1	North Prep
Balance	Mettler	PJ3600	1	Wet Chem
Balance	Mettler	PM4600	1	North Prep
Balance	Mettler	PM4000	1	Metals
Balance	Mettler	PE3600	1	Wet Chem

Instrument Type	Manufacturer	Model	Quantity	Location
Balance	Mettler	PJ3600	1	Metals
Balance	Mettler	PM400	1	Metals
Balance	Mettler	AE160	1	Metals
Balance	Mettler	PE6000	1	Wet Chem
Balance	Mettler	PC400	1	GC SVOA Hood #32
Balance	Mettler	PM4000	1	Wet Chem
Balance	Mettler	AE163	1	South Prep
Balance	Mettler	AE260	1	MS VOA Standards Prep Room
Balance	Ohasu	TS4005	1	Wet Chem
Rotary Agitation Apparatus				TCLP Prep

Table 21-2.

Example: Schedule of Routine Instrument Maintenance

Instrument	Procedure	Frequency
Cetak and Perkin Elmer Mercury Analyzers	• Check silica gel in drying tube • Change Lamp • Clean cell and aspirator in aqua regia • Check pump tubing and pump flow • Check Waste Container • Fill reductant bottle with 10% Stannous Chloride and check acid reagent	As needed As needed Monthly Daily Daily Daily
ICP	• Check pump tubing • Fill Argon humidifier with water • Check fluid level in waste container • Clean or replace air filters • Check torch for residue • Check nebulizer flow • Clean nebulizer and drain chamber • Fill rinse solution/ IS solution • Replace capillary tubing/sipper probe • Check internal fluid reservoir • Change internal cooling fluid	Daily Weekly Daily As needed Daily Daily As needed Daily As needed Monthly Yearly
ICP MS	• Change pump tubing • Check level of tuning solution • Check waste container • Load printer with paper • Check air filters • Replace coolant on chiller • Clean or change nebulizer • Clean or replace torch • Replace capillary tubing • Change oil in vacuum pumps • Remove and clean cones	Daily Daily Daily Daily Monthly Bi-annually As needed As needed As needed As needed As needed
UV-Vis Spectrophotometer	• Clean ambient flow cell • Precision check/alignment of flow cell • Wavelength verification check	As required As required Semi-annually
Colorimetric Analyzer	• Clean detector • Clean filters • Check tubing • Clean sample probe shaft • Clean pump, diluter, and XYZ sampler. • Lubricate pump roller	Daily Daily Daily Daily Monthly Semi-annually

Instrument	Procedure	Frequency
Ion Chromatograph	• Check plumbing for leaks	Daily
	• Check gases	Daily
	• Check pump pressure	Daily
	• Checkeluent level	Daily
	• Check conductivity meter	Daily
	• De-gas pump head when flow is erratic	As needed
	• Change analytical columns and bed supports guard	As needed
		As needed
	• Check and replace any damaged/dis-colored tubing	As needed
		As needed
	• Clean conductivity cell	
	• Lubricate left hand position	
Total Organic Halide Analyzer	• Check electrodes/polish if needed	Daily
	• Replace dehydrating fluid /electrolyte fluid	Daily
	• Clean quartz boat	Daily
	• Perform cell performance check	Daily
	• At the end of each day of use, wash out the absorption module, empty the electrolyte and fill chamber with DI water, empty dehydrator tube	Daily
	• Clean or replace pyrolysis tube	As needed
	• Clean titration cell	As needed
	• Replace reference electrode fluid	As needed
	• Change quartz wool	As needed
	• Replace o-rings and seals	As needed
Hewlett Packard GC/MS	• Check inlet pressure	Daily
	• Check temperature of inlet, detector, verify temperature program	Daily
	• Check Septa and clean injection port	Daily
	• Check carrier gas supply	Daily
	• Check tune parameters	Daily
	• Check oil levels in mechanical pumps and the diffusion pump if the vacuum is unsufficient	As needed
	• Replace electron multiplier	As needed
	• Clean Source	As needed
	• Replace filaments	As needed
	• Change rough pump oil and exhaust filters	Annually
	• Relubricate the turbomolecualr pump-bearing wick	Annually
Gas Chromatograph	• Check carrier gas supply	Daily
	• Check temperatures of inlet, detectors, verify temperature program	Daily
	• Check septa clean injection port or replace injection port liner and cut column if needed	As needed
	• Reactivate carrier gas drying agents	As needed
	• Replace or repair flwo controllers if constant flow cannot be mainatined	As needed

Instrument	Procedure	Frequency
Electron Capture Detector (ECD)	• Detector wipe test (Ni-63) • Detector cleaning	Semi-annually As needed
Flame Ionization Detector (FID)	• Detector cleaning	As needed
Nitrogen Phosporus Detector (NPD)	• Replace bead • Replace ceramic rings	As needed As needed
Photoionization Detector (PID)	• Change O-rings • Clean lamp window	As needed As needed
HPLC	• Check level of eluent vessels • Check gas supply • Change pump seals • Change the column frit • Change fuses in power supply • Filter all samples • Change autosampler rotor or oil autosampler slides • Change or backflush columns	Daily Daily Semi-annually or as required As needed As needed Daily As needed As needed
APCI/ESI LC/MS/MS	• Check solvent reservoirs • Verify that pump is primed and operating pulse free • Verify temperatures for capillary heater/vaporizer heater • Verify pressure of manifold/fore-pump • Verify that corona and multiplier are functional • Clean Lenses • Clean skimmer • Replace column • Oil autosampler • Change autosampler filters • Replace sample inlet tube • Replace fused silica tubing at ESI interface • Replace rough pump oil • Replace turbo pump oil • Vaccum system components including fans and fan covers	Daily Daily Daily Daily Daily As needed As needed As needed As needed As needed As needed As needed Semi-annually Annually Annually
Balances	• Class “S” traceable weight check • Clean pan and check if level • Field service	Daily, when used Daily At least Annually
Sonicator	• Inspect probe for etching/pitting • Tune sonicator assembly • Dissasemble and clean probe tips	Daily Weekly As needed
Conductivity Meter	• Standardize with KCL • Conductivity cell cleaning • Check probes and cables	Daily As needed As needed

Instrument	Procedure	Frequency
Flash Point Tester	• Check stirrer • Check tubing • Check gas supply • Check thermometer against NIST thermometer	Daily Daily Daily Daily, when used
Digestion Block	• Check with NIST thermometer	Annually
Turbidimeter	• Check light bulb • Inspect cells • Clean housing	Daily, when used Monthly Monthly
Deionized/Distilled Water	• Conductivity check • System cleaning • Replace cartridge & large mixed bed resins	Daily As needed As needed
Drying Ovens	• Temperature monitoring • Temperature adjustments	Daily As required
Refrigerators/ Freezers	• Temperature monitoring • Temperature adjustment • Defrosting/cleaning	Daily As required As required
pH/Specific Ion Meter	• Calibration/check slope • Clean electrode	Daily As required
BOD Incubator	• Temperature monitoring • Coil and incubator cleaning	Daily Monthly
Centrifuge	• Check brushes and bearings	Every 6 months or as needed
Water baths	• Temperature monitoring • Water replaced	Daily Monthly or as needed

Table 21-3.

Periodic Calibration

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Analytical Balance	Accuracy determined using A2LA-accredited NIST weights. Minimum of 3 weights bracketing the weight of interest. Inspected and calibrated by A2LA accredited person annually.	Daily	± 0.2%	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service.
Top Loading Balance	Accuracy determined using-A2LA-accredited NIST weights. Minimum of 2 weights bracketing the weight of interest. Inspected and calibrated by A2LA accredited person annually.	Daily	± 0.5%	Clean. Replace.
A2LA-accredited NIST Weights	Accuracy determined by accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
NIST-Traceable Thermometer	Accuracy determined by A2LA-accredited weights and measurement laboratory.	5 years	As per certificate.	Replace.
Thermometer	Against NIST-traceable thermometer	Yearly at appropriate temperature range for intended use	± 1.2°C	Replace
Minimum-Maximum Thermometers	Against NIST-traceable thermometer	Yearly	± 1.5°C	Replace
InfraRed Temperature Guns	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 1.5°C	Repair/replace
Dial-type Thermometers	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 1.5°C	Replace
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, check again in two hours.	2.7 ± 1.7°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Freezer	Temperature checked using NIST-traceable thermometer	Daily. If out of range, check again in two hours.	(-10)-(-20)°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Oven	Temperature checked using NIST-traceable thermometer.	When in use.	104 ± 1°C (drying) 180 ± 2°C (TDS)	Adjust. Replace.
Incubator	Temperature checked using NIST-traceable thermometer.	When in use. For microbi-ology, twice daily when in use.	BOD: 20 ± 1.0°C Micro: 35 ± 0.5°C	Adjust. Replace.
Water Bath	Temperature checked using NIST-traceable thermometer.	When in use.	± 2°C	Adjust. Replace.
Volumetric Dispensing Devices (Eppendorf ® pipette, automatic dilutor or dispensing devices)	One delivery by weight. Using DI water, dispense into tared vessel. Record weight with device ID number.	Monthly	± 2% Calculate accuracy by dividing weight by stated volume times 100 for percent.	Adjust. Replace.
Glass Microliter Syringes	None	Accuracy must be initially de-monstrated if syringe was not received with a certifi-cate attesting to established accuracy.	± 2%	Not applicable.
Conductivity Meter	Cell impedance calibrated with three KCl standards.	Each use.	$r \geq 0.99$	Recalibrate.
Deionized Water	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Weekly	<10 µmhos/cm^2	Record on log. Report discrepancies to QA Director.

Table 21-3
Preventive Maintenance Procedures
For Laboratory Equipment

Instrument/ Equipment Type	Maintenance	Frequency
Gas Chromatograph	Replace Gas line dryers and filters	As needed*
	Replace Gas cylinders	As needed*
	Check or adjust column gas flow and/or detector make-up flow	As needed*
	Replace Injection port Septa	Daily*
	Replace Injection port liners/re-silonize liners	GC(MSVOA); GC/MS SVOC, Daily*
	Replace injection port liner o-ring	GC, As needed; GC/MS, Daily*
	Replace inlet seal and ring	GC, As needed, GC/MS, Daily*
	Replace column ferrules	GC, As needed; *
	Clip column (injector and detector end)	GC, As needed; GC/MS, Daily*
	Replace syringes on autosamplers	As needed*
	Replace heated-zones heaters and sensors	As needed*
	Replace inlet assembly	As needed*
	Empty solvent rinse and solvent waste vials (on autosampler tower)	Daily or as needed
	Replace column	As needed*
Flame Ionization Detector (FID)	Clean/replace jet	As needed*
	Clean collector	As needed*
	Check and/or adjust gas flows	As needed*
Photoionization Detector (PID)	Clean window	As needed*
	Replace o-ring seat	As needed*
	Replace Lamp	As needed*
	Check and/or adjust gas flows	As needed*
	Adjust Lamp power supply intensity	As needed*
Mass Spectrometer (MS)	Clean source, replace source parts, replace filaments	As needed*
	Clean analyzer	As needed*
	Replace electron multiplier	As needed*
	Clean or replace glass jet separator, replace transfer line from jet separator to MS	As needed*
	Change rough pump oil	After each source cleaning
	Refill calibration compound (PFTBA) vial	As needed
Purge and Trap Equipment	Refill rinse water supply/Empty rinse water waste	Weekly or as needed
	Refill spiking solutions vials	As needed
	Rinse sparge tubes	Daily
	Clean or replace 6-port valve	As needed*
	Replace Transfer lines (from Autosampler to LSC and from LSC to GC)	As needed*
	Adjust gas flows and pressures	As needed
	Perform leak check	As needed
Inductively Coupled Plasma, Atomic Emission Spectrometer (ICP-AES)	Replace Peristaltic pump tubing	As needed*
	Clean autosampler, change tubing	As needed*
	Clean nebulizer and torch assembly	As needed*
	Replace nitrogen and argon tanks	As needed*
	Refill rinse water receptacle	Daily
	Empty waste receptacle	Daily
	Check for internal standard and sample flow through peristaltic pump tubing	As often as possible
	Replace internal standard solution receptacle	As needed
	Operate and check vents	Daily
	Perform Hg alignment	Daily*

Instrument/ Equipment Type	Maintenance	Frequency
	Check water level and water filter on recirculating-cooling unit, refill and replace filter	Check daily, refill and replace as needed
	Check purge windows	Daily, replace as needed
	Replace nebulizer and o-rings	As needed*
	Replace torch	As needed*
	Drain air compressor	Weekly
	Replace mixing chambers	As needed*
	Clean or replace air filters	Weekly
	Check pneumatic filters	Weekly, replace as needed
	Perform wave calibration (UV and Vis)	Quarterly*
	Calibrate Detector	Quarterly*
High Pressure Liquid Chromatography (HPLC)	Replace pre-column filter	As needed*
	Refill Solvent reservoirs	Daily or as needed
	Reverse column and rinse with solvents	Daily or as needed*
	Replace column	As needed*
	Clean solvent reservoir filters	As needed*
	Replace ball-valve cartridges on high pressure pump	As needed*
	Replace DAD flow cell windows	As needed*
	Check system solvent pressure	Daily
pH Meters	Clean or replace electrode	As needed
	Refill electrode electrolyte	As needed
Balance	Clean pan and platform	After each use
	Check Level bubble	Daily
	Check calibration	Daily
	Cleaning and calibration by authorized service	Annually
Conductivity Meter	Clean probe	As needed
Dissolved Oxygen Meter	Replace membrane	As needed
	Clean probe	As needed
ZHE vessels	Replace o-rings and screens	As needed
ZHE and TCLP Tumblers	Check Rotation Rate	Yearly
Spectrophotometers	Clean and check tubing	As needed
Burettes and Pipets	Clean and check calibration	Monthly
Thermometers	Check calibration	Annually, Quarterly for Digitals and IR Thermometer*
Ovens	Check and/or adjust temperature, record temperature on log sheet	Daily
Refrigerators and Freezers	Check and/or adjust temperature, record temperature on log sheet	Daily
	Defrost freezers	As needed
OI Alpkem/Astoria, Flow Injection Analyzer	Replace tubes on autodilutor	As needed*
	Clean autosample surfaces	As needed
	Spray silicone on cloth and rub on pump rollers	As needed
	Clean or replace o-rings and ports on valves	As needed*
	Clean union and T's on manifold and replace o-rings on manifold	As needed
	Dry and clean detector surfaces	As needed
	Replace flow cell o-rings and flares	As needed*
	Replace manifold tubing	As needed*
	Adjust pump timing	As needed
APCI/ESI LC/MS/MS	Change filters in Autosampler	As needed*
	Change Pump Seals	As needed*
	Rinse Capillary with MeOH	As needed*
	Rinse and clean corona needle	As needed*
	Replace fused silca tubing at ESI interface	As needed*
	Replace sample inlet tube in APCI	As needed*
	Clean lenses	As needed*

SECTION 22

MEASUREMENT TRACEABILITY
(NELAC 5.5.6)

22.1 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. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), at a minimum, 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. The following definitions are provided by the American Association for Laboratory Accreditation (A2LA):

"Traceability is the property of a measurement result whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, each step in the chain having stated uncertainties." There are six essential elements:

- An unbroken chain of comparison
- A calculated measurement uncertainty for each step in the chain to allow for an overall uncertainty calculation
- Documentation of each step in each calibration report
- All steps in the chain are performed by individuals with evidence of technical competence and accredited by a recognized accreditation body
- Reference to International Standard (SI) units
- Recalibration at appropriate intervals to preserve traceability

Calibration is defined as "determining and documenting the deviation of the indication of a measuring instrument (or the stated value of a material measure) from the conventional 'true' value of the measurand."

Uncertainty is defined as "a parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measurand." Measurement of Uncertainty is discussed is Section 20 of this QA Manual.

22.2 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), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

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 Denver 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 Denver 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.

22.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, and ISO/IEC with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. (Refer to Section 9 for additional information on purchasing). 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 IDA 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 Table 9-1 in Section 9 for general storage requirements and SOP DV-QA-0015 for additional storage information. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

22.4 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 SOP No. 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 maintained by the appropriate group until they are permanently archived by QA. 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 DV-QA-0015, Verification and Storage of Calibration Standards.

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.

22.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's Standards software, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the Standards program.

- 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 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.

22.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID – assigned in the Standards log software.
- Special Health/Safety warnings if applicable

22.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- 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)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, 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 raw data.

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; and 3) according to requirements in SOP DV-QA-0015, Verification and Storage of Calibration Standards.

SECTION 23.0

SAMPLING
(NELAC 5.5.7)

23.1 OVERVIEW

TestAmerica Denver 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. On occasion, the lab will supply personnel to assist with the duties mentioned above. In that case,the laboratory staff must adhere to the site specific health and safety plan as provided by the client.

23.2 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. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

23.2.1 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

23.2.2 Preparing Container Orders

When new containers arrive at the laboratory, the date of receipt is recorded on the packing list received with them for retained documentation. Periodically, containers are evaluated for cleanliness based upon their intended parameter sample analysis. Upon request, the containers are then sent to clients for use in collecting samples. The shipping date, type and number of containers are maintained on file by the lab. Shipping personnel insure that container stock is rotated so that "first in" is "first out." When a client requests containers, a client services representative creates a container request in LIMS; it is then stored permanently in LIMS with a unique container order number. Copies of the container request are printed for the shipping department. One copy goes to the client with the containers; one copy is filed in the shipping department.

The laboratory also provides EnCore, TerraCore or other soil sampling devices when requested.

If containers are provided directly to the client from the manufacturer or from other sources, the laboratory will not be responsible for any of the above records.

23.3 FIELD QUALITY CONTROL (QC)

Common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the site specific Quality Assurance Project Plan (QAPP) or by the client. TestAmerica provides trip blanks for VOC analysis with the sample containers for all volatile organic analyses. Blanks generated in the field will be analyzed along with the field samples (exception soil samples where the blank is aqueous).

23.3.1 Equipment Blank / Rinsate Blank - The equipment blank, sometimes referred to as a rinsate blank, is a sample of the water used to decontaminate sampling equipment. The source water should be as free of target analytes as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and will also be affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank should begin when the equipment is rinsed and the water is collected.

23.3.2 Field Blank - The field blank is water that is as free of target analytes as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination. The sampling time for the field blank should be when the blank is prepared in the field.

23.3.3 Trip Blank - The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers. They are filled with organic-free water (the source of the organic free water is the same source of water used to prepare volatile standards, method blanks, LCS and sample dilutions), sealed and taken into the field with the empty containers which will be used for sampling. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method. Unless otherwise specified, the sampling time for the trip blank is the time of receipt at the laboratory (When the “Trip” ends).

23.3.4 Field Duplicates - Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

23.4 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (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. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However there are some programs that 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.

23.4.1 Semi-Volatile - Holding times for sample preparation for semi-volatile organics are measured from the sampling date (and time where applicable) until the day (and time where applicable) solvent contacts the sample. Holding times for analysis are measured from the date (and time where applicable) of initiation of extraction to the time of injection into the gas chromatograph.

23.4.2 Volatiles - Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date and time of injection into the gas chromatograph.

23.4.3 Inorganics - For inorganic and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date (and time where applicable).

23.5 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times (refer to Tables 23-1 to 23-3) 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.

23.6 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. In that regard the following guidelines apply to analysts:

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.

Refer to SOPs DV-QA-0023, *Subsampling* and SOPs DV-OP-0013 and DV-OP-0014.

23.6.1 For multiphasic samples, the client should instruct the laboratory as to the intent of the testing and how to handle the sample. If the entire sample is to be accounted for, and the phases do not mix easily with inversion/stirring, such that a representative aliquot can be taken, the analyst should record the percent by volume of each phase. The analysis must be conducted on each phase separately; the final results can either be reported separately or combined mathematically, weighting the individual phase results by volume. One exception to this procedure is the situation addressed in the TCLP and SPLP methods for wastes containing free liquids. However, if the leachate and final filtrate are not miscible, it is necessary to combine mathematically the concentrations of the two (or more) solutions by volume.

Tables 23-1 to 23-3 detail holding times, preservation and container requirements, and sample volumes for SDWA and NPDES methods. The sample volumes are intended to be a minimal amount to perform the method, the containers that are used may be of larger size.

Note: the holding times are program specific and different programs may have different holding times for equivalent methods (e.g., there are difference in Holding times for many Organic analytes between SDWA and NPDES. RCRA methods may also be different.)

Table 23-1.

Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Acidity	Water	100 mL	2310 B	250 mL plastic or glass, Cool, 4°C, 14 days	---	Not Applicable
	Solid[5]	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Alkalinity	Water	100 mL	2320B	250 mL plastic or glass, Cool, 4°C, 14 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Ammonia	Water	400 mL	350.1	500 mL plastic or glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Biochemical Oxygen Demand (BOD)	Water	200 mL	5210 B	1000 mL plastic or glass, Cool, 4°C 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Bromide	Water	100 mL	300.0[7]	250 mL plastic or glass, No preservative required, 28 days	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not	---	Not Applicable	---	Not Applicable

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
		Applicable				
Chemical Oxygen Demand (COD)	Water	100 mL	410.4	250 mL glass or plastic, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Chloride	Water	50 mL	300.0[7] 4500-Cl C,E	250 mL plastic or glass, No preservative required, 28 days	9056	Method 9056: Cool, 4°C, analyze ASAP after collection.
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Chromium (Cr^{+6})	Water	100 mL	3500 Cr-D	Method 218.4: 200 mL plastic or glass, Cool, 4°C, 24 hours Method 3500 Cr-D: 200 mL quartz, TFE, or polypropylene HNO_3 to pH <2 Cool, 4°C Analyze ASAP after collection	7196A	200 mL plastic or glass, Cool, 4°C, 24 hours
	Solid	Not Applicable	---	Not Applicable	7196A	250 mL plastic or glass, 30 days to digestion, 96 hours after digestion
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

Table 23-1.
Inorganic Sample Containers, Preservatives, and Holding Times – con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Color	Water	100 mL	2120 B	250 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Conductivity	Water	100 mL	120.1	200 mL glass or plastic, Cool, 4°C, 28 days	9050A	200 mL glass or plastic, Cool, 4°C, 24 hours
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Cyanide (Amenable)	Water	IL	335.4	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours	9010B/ 9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days
	Solid	50g	---	Not Applicable	9010B/ 9012A	Not Specified
	Waste	50g	---	Not Applicable	9010B/ 9012A	Not Specified
Cyanide (Total)	Water	IL	335.4	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours	9010B/ 9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid[6] Cool, 4°C, 14 days
	Solid	50g	--	Not Applicable	9010B 9012A	8 or 16 oz glass Teflon-lined lids, Cool, 4°C, 14 days

Table 23-1.

Inorganic Sample Containers, Preservatives, and Holding Times – con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Cyanide (Total) (continued)	Waste	50g	--	Not Applicable	9010B/ 9012A	8 or 16 oz glass Teflon-lined lids, Cool, 4°C
Flashpoint (Ignitability)	Liquid	Not Applicable	---	Not Applicable	1010	No requirements, 250 mL amber glass, Cool, 4°C is recommended
	Solid	Not Applicable	--	Not Applicable	---	Not Applicable
	Waste	Not Applicable	--	Not Applicable	---	Not Applicable
Fluoride	Water	300 mL	300.0[7] 4500-F C, C-97	500 mL plastic, No preservation required, 28 days	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Hardness (Total)	Water	50 mL	2340B	250 mL glass or plastic, HNO_3 to pH < 2, 6 months	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Iron (Ferrous)	Water	100 mL	3500-Fe D	1 liter glass or polyethylene container, 6 months **This test should be performed in the field.**	-	Not Applicable
	Solid	Not Applicable	-	Not Applicable	-	Not Applicable
	Waste	Not Applicable	-	Not Applicable	-	Not Applicable

Table 23-1.

Inorganic Sample Containers, Preservatives, and Holding Times – con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Methylene Blue Active Substances (MBAS) (Surfactant)	Water	100 mL	5540-C-00	250 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Nitrate	Water	100 mL	300.0[7] 353.2	Method 300.0: 250 mL plastic or glass, Cool, 4°C, 48 hours. Method 352.1: 250 mL plastic or glass, Cool, 4°C, 48 hours.	9056	Method 9056: Cool, 4°C, analyze ASAP after collection Method 9210: Cool, 4°C Preserve by adding 1 mL of 1M boric acid solution per 100 mL of sample
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	9210	Not Specified
Hydrazines	Water	100 mL	---	Preserve at lab to pH =2 within 48 hours of collection. Hold time 28 days.	---	Preserve at lab to pH =2 within 48 hours of collection. Hold time 28 days.
	Solid	10 grams	---	4 oz jar Cool, 4°C	---	4 oz jar Cool, 4°C
Nitrite	Water	50 mL	300.0[7] 353.2	250 mL plastic or glass Cool, 4°C, 48 hours	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

Table 23-1.

Inorganic Sample Containers, Preservatives, and Holding Times – con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Nitrate-Nitrite	Water	100 mL	4500-NO3 F	250 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Ortho-phosphate	Water	50 mL	300.0[7] 365.3	100 mL plastic or glass, Filter on site Cool, 4°C, 48 hours	9056	Cool, 4°C, analyze ASAP collection
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
pH	Water	50 mL	150.1 4500-H^+ B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.**	9040B	100 mL plastic or glass. Analyze immediately. **This test should be performed in the field.**[8]
	Solid	Not Applicable	---	Not Applicable	9045C	4 oz glass or plastic, Cool, 4°C, Analyze as soon as possible.[8]
	Waste	Not Applicable	---	Not Applicable	9045C	4 oz glass or plastic, Cool, 4°C, Analyze as soon as possible.[8]
Phenolics	Water	100 mL	420.4	500 mL glass, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	9066	1 liter glass recommended, Cool, 4°C, H_2SO_4 to pH < 4, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	9065	Not Specified

Table 23-1.

Inorganic Sample Containers, Preservatives, and Holding Times – con't

Analytical		Minimum Sample	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
Parameters	**Matrix**	**Size[1]**	**Method**	**Requirements**	**Method**	**Requirements**
Phosphate	Water	50 mL	365.3	Not Applicable	9056	Cool, 4°C, analyze ASAP collection
	Solid	Not Applicable	---	Not Applicable	9056	Not Applicable
	Waste	Not Applicable	---	Not Applicable	9056	Not Applicable
Phosphorus (Total)	Water	50 mL	365.3	100 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Reactivity (Cyanide and Sulfide)	Liquid	10 g	---	Not Applicable	Chapter 7 Section 7.3.3.2 and 7.3.4.2	10 oz amber glass, Cool, 4°C, no headspace, analyze as soon as possible.
	Solid	10 g	---	Not Applicable	Chapter 7 Section 7.3.3.2 & 7.3.4.2	10 oz amber glass, Cool, 4°C, no headspace, analyze as soon as possible.
	Waste	10 g	---	Not Applicable	Chapter 7 Section 7.3.3.2 and 7.3.4.2	10 oz amber glass, Cool, 4°C, no headspace, analyze as soon as possible.
Settleable Solids	Water	1000 mL	2540 F	1000 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Specific Conductance	Water	50 mL	2510 B	250 mL plastic or glass, Cool, 4°C, 24 hours	9050A	250 mL plastic or glass, Cool, 4°C, 28 days

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Specific Conductance – Con't	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Sulfate (SO_4)	Water	100 mL	300.0[7] 375.2	100 mL plastic or glass, Cool, 4°C, 28 days	9056 9038	Method 9056: Cool, 4°C, analyze ASAP collection Method 9038: 200 mL plastic or glass, Cool, 4°C, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	100 mL	---	Not Applicable	9038	200 mL plastic or glass, Cool, 4°C, 28 days
Sulfide	Water	100 mL	4500-S2 D-00	500 mL plastic or glass, Cool, 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days	9030B/ 9034	500 mL plastic, no headspace, Cool, 4°C, Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution, 7 days
	Solid	50 g	---	Not Applicable	9030B 9034	Cool, 4°C, fill surface of solid with 2N Zinc acetate until moistened, store headspace-free
	Waste	50 g	---	Not Applicable	9030B 9034	Cool, 4°C, fill surface of solid with 2N Zinc acetate until moistened, store headspace-free

Table 23-1.

Inorganic Sample Containers, Preservatives, and Holding Times – con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Sulfite (SO_3)	Water	100 mL	4500-SO3 B-00	100 mL plastic or glass, No preservative required, analyze immediately **This test should be performed in the field.**	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total Dissolved Solids (Filterable)	Water	100 mL	2540 C	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total Kjeldahl Nitrogen (TKN)	Water	500 mL	4500-N	500 mL plastic or glass, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total Organic Carbon (TOC)	Water	100 mL	5310-B,C,D	100 mL plastic or glass, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	9060	100 mL glass or 40 mL VOA vials, Cool, 4°C, H_2SO_4 or HCl to pH < 2, 28 days
	Solid	Not Applicable	---	Not Applicable	9060	Not Specified
	Waste	Not Applicable	---	Not Applicable	9060	Not Specified

Table 23-1.

Inorganic Sample Containers, Preservatives, and Holding Times – con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method	Requirements
Total Organic Halides (TOX)	Water	100 mL	---	Method 5320B: 500 mL amber glass, Teflon®-lined lid, Cool, 4°C, HNO_3 to pH <2, no headspace, 14 days Method 450.1: 500 mL amber glass, Teflon®-lined lid, Cool, 4°C, HNO_3 to pH <2, no headspace, 28 days	9020B	500 mL amber glass, Teflon®-lined lid, Cool, 4°C, H_2SO_4 to pH < 2, no headspace, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total Solids	Water	100 mL	2540 B	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total Suspended Solids (Nonfilterable)	Water	100 mL	2540 D	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Turbidity	Water	50 mL	180.1	250 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable

Table 23-1.

Inorganic Sample Containers, Preservatives, and Holding Times – con't

Analytical		Minimum Sample	NPDES[2], [3], [7]		RCRA (SW846)[3], [4]	
Parameters	**Matrix**	**Size[1]**	**Method**	**Requirements**	**Method**	**Requirements**
Volatile Solids	Water	100 mL	160.4	250 mL plastic or glass, Cool, 4°C, 7 days ---	Not Applicable	Water
Water Content	Solid	NA	---	Not Applicable	---	Not Applicable
	Waste	NA	---	Not Applicable	---	Not Applicable
	Water	NA	---	Not Applicable	---	Not Applicable
	Solid	10 g	---	Refer to specific method used	---	Refer to specific method used
	Waste	10 g	---	Refer to specific method used	---	Refer to specific method used
Metals (excludes Hg)	Water	100 mL	200 series	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months	6010B, 6020	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months
	Solid	200 g	200 series	8 or 16 oz glass or polyethylene container storage at 4 °C	6010B, 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	200 g	200 series	Not Applicable	6010B, 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
Mercury (CVAA)	Water	100 mL	245.1	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days
	Solid	200 g	245.5	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days	7471A	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days (CORP-MT-0007)
	Waste	200 g	--	Not Applicable	7471A	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days (CORP-MT-0007)

Footnotes

(1) Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
(2) National Pollutant Discharge Elimination System - MCAWW, March 1983.
(3) Holding times are calculated from date of collection.
(4) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA, (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
(5) Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.
(6) Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.
(7) Method not listed in 40 CFR Part 136.
(8) If not done in the field (ASAP) per the method and requested by client, analyze in lab within 48 hours.

Table 23-2

Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2, 3]		RCRA (SW846)[3, 4]	
			Method	Requirements	Method[6]	Requirements
Aromatic Volatiles	Water	40 mL	602	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2
	Solid[5]	5 g or 25 g	--	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate).Cool, 4°C (See Note 12 Page 136 for holding time.)
	Waste	5 g or 25 g	--	Not Applicable	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis.

Table 23-2

Organic Sample Containers, Preservatives, and Holding Times – con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	Method	NPDES[2, 3]		RCRA (SW846)[3, 4]
				Requirements	Method[6]	Requirements
Aromatic Volatiles (continued)	Waste	5 g or 25 g	--	Not Applicable	8021B	Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C. (See Note 12 Page 136 for holding time.)
Halogenated Volatiles By GC	Water	40 mL	601	Not Applicable	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days
	Solid[5]	5 g or 25 g	601		8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C. (See Note 12 Page 136 for holding time.)

Table 23-2

Organic Sample Containers, Preservatives, and Holding Times – con't

Analytical		Minimum Sample	NPDES[2], [3]		RCRA (SW846)[3], [4]	
Parameters	**Matrix**	**Size[1]**	**Method**	**Requirements**	**Method[6]**	**Requirements**
Nitrosamines	Water	1L	607 [10]	1 liter amber glass with Teflon®-lined lid, Sodium thiosulfate or ascorbic acid if residual chlorine present, Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8070A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
Nitrosamines	Soil	30 g	--		8070A	4 or 8 oz glass widemouth with Teflon®-lined lid, Cool 4 °C, Extraction, 14 days Analysis, 40 days of the start of the extraction
Herbicides	Water	1L	615 [10]	1 liter amber glass with Teflon®-lined lid, Sodium thiosulfate or ascorbic acid if residual chlorine present, Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8151A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	--	Not Applicable	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid, Cool 4 °C, Extraction, 14 days Analysis, 40 days of the start of the extraction
Nitroaromatics	Water	0.5L	--	Not Applicable	8330	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	25 g	---	Not Applicable	8330	4 or 8 oz glass widemouth with Teflon®-lined lid Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction

Table 23-2

Organic Sample Containers, Preservatives, and Holding Times – con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Nitroaromatics (continued)	Waste	25 g	---	Not Applicable	8330	4 or 8 oz glass widemouth with Teflon®-lined lid Cool, 4 °C, Extraction, 14 days Analysis, 40 days of the start of the extraction
Organo-phosphorus Pesticides	Water	1L	---	Not Applicable	8141A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	30 g	---	Not Applicable	8141A	4 or 8 oz glass widemouth with Teflon®-lined lid Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	30 g	---	Not Applicable	8141A	4 or 8 oz glass widemouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
PAHs by GC and HPLC	Water	1L	610	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present. Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8310	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction

Table 23-2

Organic Sample Containers, Preservatives, and Holding Times – con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
PAHs by GC and HPLC (continued)	Solid	30 g	---	Not Applicable	8310	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	30 g	---	Not Applicable	8310	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C Extraction, 14 days Analysis, 40 days of the start of the extraction
Pesticides/ PCBs	Water	1L	608	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8081A 8082	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	30 g	---	Not Applicable	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	30 g	---	Not Applicable	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C Extraction, 14 days Analysis, 40 days of the start of the extraction

Table 23-2

Organic Sample Containers, Preservatives, and Holding Times – con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Petroleum Hydrocarbons/Oil and Grease	Water	1L	413.1 413.2 418.1	1 liter glass, Cool, 4°C, HCl to pH <2, 28 days	9070	1 liter glass with Cool, 4°C, HCl to pH <2, 28 days
	Solid	---	---	Not Applicable	9071A	8 oz. glass with Teflon®-lined lid, Holding Time not specified
	Waste	---	---	Not Applicable	9071A	8 oz. glass with Teflon®-lined lid, Holding Time not specified
	Water	1 L	1664[7]	1 liter glass, Cool, 0-4°C HCl or H_2SO_4 to pH <2 28 days	---	---
	Solid	30 g	1664[7]	8 or 16 oz. wide mouth glass jar, Cool, 0-4°C, 28 days	---	---
	Waste	---	---	Not Applicable	---	---

TABLE 23-2

Organic Sample Containers, Preservatives, and Holding Times – Con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Semivolatiles	Water	1L	625	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction, 7 days Analysis, 40 days	8270C	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, within 40 days of extraction
	Solid	30 g	---	Not Applicable	8270C	8 or 16 oz glass wide mouth with Teflon-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction
	Waste	30 g	---	Not Applicable	8270C	8 or 16 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction
Volatile Organics	Water	40 mL	624	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2[8]	8260B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2[9]

TABLE 23-2

Organic Sample Containers, Preservatives, and Holding Times – Con't

Analytical Parameters	Matrix	Minimum Sample Size[1]	NPDES[2], [3]		RCRA (SW846)[3], [4]	
			Method	Requirements	Method[6]	Requirements
Volatile Organics (continued)	Solid[5]	5 g or 25 g	--	Not Applicable	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C. (See Note 12 Page 136 for holding time.)
	Waste	5 g or 25 g	--	Not Applicable	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C. (See Note 12 Page 136 for holding time.)

TABLE 23-2

Organic Sample Containers, Preservatives, and Holding Times Footnotes

Footnotes

(1) Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

(2) National Pollutant Discharge Elimination System - 40 CFR Part 136, Appendix A.

(3) Holding times are calculated from the date of collection.

(4) Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).

(5) Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.

(6) Only one determination method is listed when separate methods are required for preparation and analysis.

(7) Method 1664 was promulgated by the EPA with an effective date of June 14, 1999.

(8) For acrolein and acrylonitrile the pH should be adjusted to 4-5. This pH adjustment is not required if acrolein is not measured. Samples requiring analysis of acrolein that received no pH adjustment must be analyzed within three days of sampling.

(9) For acrolein and acrylonitrile the pH should be adjusted to 4-5.

(10) Method not listed in 40 CFR Part 136.

(11) Should only be used in the presence of residual chlorine.

(12) Depending on regulatory programs, EnCore™ samplers may be preserved for up to 14 days from sampling by freezing at -5 to -12°C until analysis. Alternatively the EnCore™ sample may be transferred to a 40-ml VOA vial and preserved by freezing at -5 to -12°C until analysis. Some regulatory agencies may require 4 or 8 oz glass with Teflon®-lined lid, Cool 4°C, 14 days. This technique is not recommended, but will be supported where required. (Preservation and holding times are subject to client specifications.)

TABLE 23-3
Sample Containers, Preservatives, and Holding Times for TCLP[(1)] and SPLP[(2)]

Analytical Parameters	Matrix	Minimum Sample Size[(3)]	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 28 days	Glass or polyethylene 28 days
Metals (except mercury)	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

Footnotes

(1) TCLP = Toxicity Characteristic Leaching Procedure

(2) SPLP = Synthetic Precipitation Leaching Procedure

(3) Smaller sample size is adequate for solid samples or individual fractions. A combined volume of 32 oz. is recommended for semivolatiles and metals. A separate 4 oz. container should always be used for the volatile fraction. Volatile fractions should be stored with minimal headspace.

SECTION 24

HANDLING OF SAMPLES
(NELAC 5.5.8)

Sample management procedures at TestAmerica Denver ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

24.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and can be 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 24-1.

24.1.1 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 24-1). 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.

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. 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. Samples are only considered to be received by lab when personnel at the laboratory 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.

24.1.2 Legal / Evidentiary Chain-of-Custody

All samples are tracked through the sample utility software program "STU" to ensure internal chain of custody and cradle to grave tracking of each sample container. If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal (Figure 24-2), retain the shipping record with the COC, and an internal COC for analysts to fill out and sample disposal record from STU (Figures 24-3 and 24-4) will be included in the data package.

24.2 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. Refer to SOP DV-QA-0003, *Sample Management and Chain of Custody*.

24.2.1 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 Condition Upon Receipt Anomaly Form (CUR Figure 24-6) 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.

24.2.1.1 Inspection of samples include a check for (see Figure 24-5):

- Complete documentation to include sample identification, location, date and time of collection, collector's name, preservation type, sample type and any additional comments concerning the samples.
- Complete sample labels to include unique identification in indelible ink.
- Use of appropriate sample containers (see Section 23)

- Adherence to holding times as specified in the test method and/or summarized in Section 23.
- Adequate sample volume for required analyses (see Section 23).
- Damage or signs of contamination to sample container. Volatile vials are also inspected for headspace

24.2.1.2 Using the infrared temperature gun, check and record the temperature of the samples (use temperature blanks if present) to verify appropriate thermal preservation. Record the temperature on both the chain of custody (Figure 24-1) and the sample receiving checklist (Figure 24-5).

- Samples shall be deemed acceptable if arrival temperature is just above freezing and less than or equal to 6.0° C, or ≥ -20° C if shipped frozen (encores). Samples that are hand-delivered immediately after collection may not be at the required temperatures; however, if there is evidence that the chilling process has begun, such as the arrival on ice, the samples shall be considered acceptable. This will be documented on the CUR (Figure 24-6).

- If the samples were shipped in ice and solid ice is still present and in direct contact with samples, report the samples as "received on ice." Direct contact means samples must be surrounded by ice cubes or crushed ice. Ice present in a plastic bottle or other container does not constitute direct contact. Samples shipped with only “blue ice” may not be reported as “received on ice”.

24.2.1.3 Verify sample preservation as specified in the test method. Check for correct pH as specified in the test method. The results are documented on the CUR form (Figure 24-5). In the case of volatiles it is recorded after analysis on the instrument run log. Chlorine is checked at the time of analysis on samples requiring extractable organics, BOD, TOX, cyanide, fluoride, ammonia, TKN, CBOD and Nitrate; presence or absence is recorded. The need for a residual chlorine check is noted on the sample receiving checklist by the project manager during the cooler greeting process.

24.2.1.4 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.

24.2.1.5 If samples are received without a COC, TestAmerica will provide a generic COC form to be completed by the client when the samples are brought to the laboratory. The client is always provided with a copy of the completed COC form for their records.

24.2.1.6 If analyses with short holding times are requested, the dates and times are inspected to ensure that holding times have not already expired.

24.2.1.7 Only department of transportation (DOT) trained staff may receive samples, so it is imperative that samples are dropped during normal working hours, or special arrangements are made with the project manager. If an attempt is made to drop

samples after hours without arrangements to have DOT trained staff available, the laboratory staff will be unable to accept them.

24.2.1.8 Any deviations from the checks described in Section 24.2.1 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 criteria (Section 24.3) 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.

24.2.2 Sample Log-in

All samples that are received by the laboratory are logged into the LIMS and the Sample Transfer Utility program (STU) to allow the laboratory to track and evaluate sample progress. Each group of samples that are logged in together (typically one project from a given client/sampling event) is assigned a unique job number. Within each job, each sampling point (or sample) receives a unique number. Sample numbers are generated sequentially over time, and are not re-assigned. A sample may be composed of more than one bottle since different preservatives may be required to perform all analyses requested. Even if multiple containers are received for a single sample, each container is uniquely identified with an 6-digit workorder number added to the sample number. The LIMS generates sample labels that are attached to each bottle for a given sample.

Each job/set of samples is logged into LIMS with a minimum of the following information:

- Client Name, Project Name, Address, Phone, Fax, Report to information, invoice to information (most of this information is “default information” that is stored in the LIMS).
- Date and time sampled;
- Date and time received;
- Job and/or project description, sample description;
- Sample matrix, special sample remarks;
- Reporting requirements (i.e., QC level, report format, invoicing format);
- Turn-around-time requirements;
- Parameters (methods and reporting limits or MDLs are default information for a given parameter)

24.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 24-5) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- Cooler seals intact;
- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method;
- sample holding times must be adhered to;
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- 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. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

24.4 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 suitable for the sample matrix, except metals sample containers which may be stored unrefrigerated. 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, document the transfer of containers in STU and place them on carts, analyze the sample, and return the remaining sample to the refrigerator from which it originally came, documenting the return in STU. Empty containers are stored in the sample archive area until disposal, this transfer is documented in STU. All samples are kept in the refrigerators until the project is invoiced. At this time, the samples will be retained for an additional thirty days, either in the refrigerators, or in the sample archive area. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues. Upon disposal, the drum number used for disposal is logged into STU.

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.

24.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in a designated area. For any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, the analyst will notify login staff so the hazardous sample is properly labeled as such. The sample itself is clearly marked with a label reading “HAZARDOUS”, “PCBs” or “FOREIGN SOIL”. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm. All foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility, refer to SOP DV-QA-0019, *Quarantine Soils Procedure* for more detail.

24.6 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. 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.

24.7 SAMPLE DISPOSAL

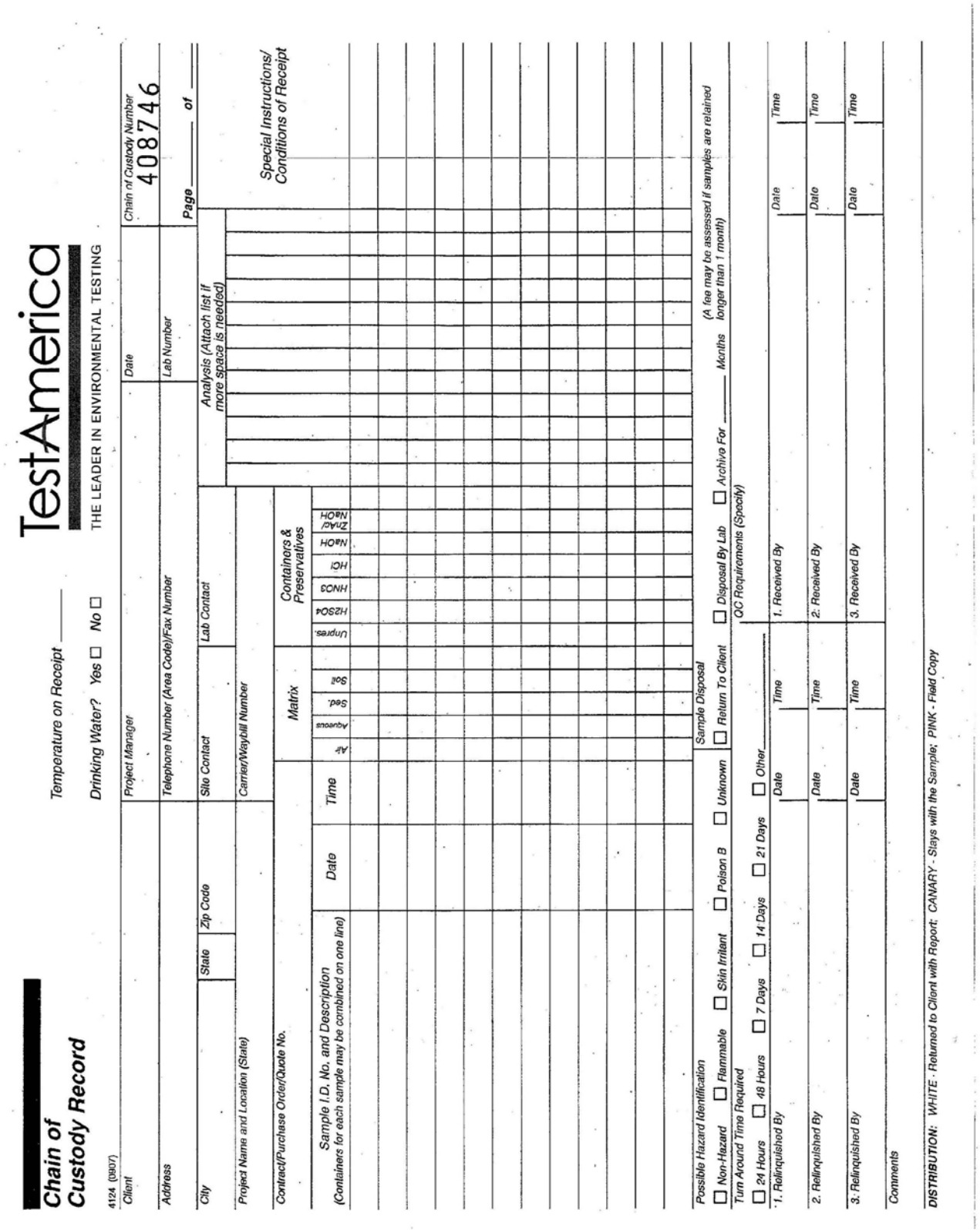
Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after 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: *DV-HS-0005, Excess Sample Material Management.* All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than six weeks 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 of samples is kept on file. The STU software allows tracking for each sample container from the time of sample receipt through the disposal process, including such detail as the identifying number of the waste drum used for disposal. 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 Hazardous Waste Manifest will be prepared to document the

disposal of each drum, see Figure 24-7 for labeling of drums for disposal. Additional detail is in SOP DV-HS-0004, *Hazardous Waste Manifesting*.

Figure 24-1.

TestAmerica Denver: Chain of Custody (COC)



Chain of Custody Record

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

4124 (0907)

Temperature on Receipt _____

Drinking Water? Yes ☐ No ☐

Chain of Custody Number 408746

Page _____ of _____

Client | Project Manager | Date

Address | Telephone Number (Area Code)/Fax Number | Lab Number

City | State | Zip Code | Site Contact | Lab Contact | Analysis (Attach list if more space is needed)

Project Name and Location (State) | Carrier/Waybill Number

Contract/Purchase Order/Quote No. | Matrix | Containers & Preservatives

Special Instructions/ Conditions of Receipt

Sample I.D. No. and Description (Containers for each sample may be combined on one line) | Date | Time | Air | Aqueous | Sed. | Soil | Unpres. | H2SO4 | HNO3 | HCl | NaOH | ZnAc/ NaOH

Possible Hazard Identification

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal

☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months (A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required

☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other _____

QC Requirements (Specify)

1. Relinquished By | Date | Time | 1. Received By | Date | Time

2. Relinquished By | Date | Time | 2. Received By | Date | Time

3. Relinquished By | Date | Time | 3. Received By | Date | Time

Comments

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

Figure 24-2.

Example: Custody Seal

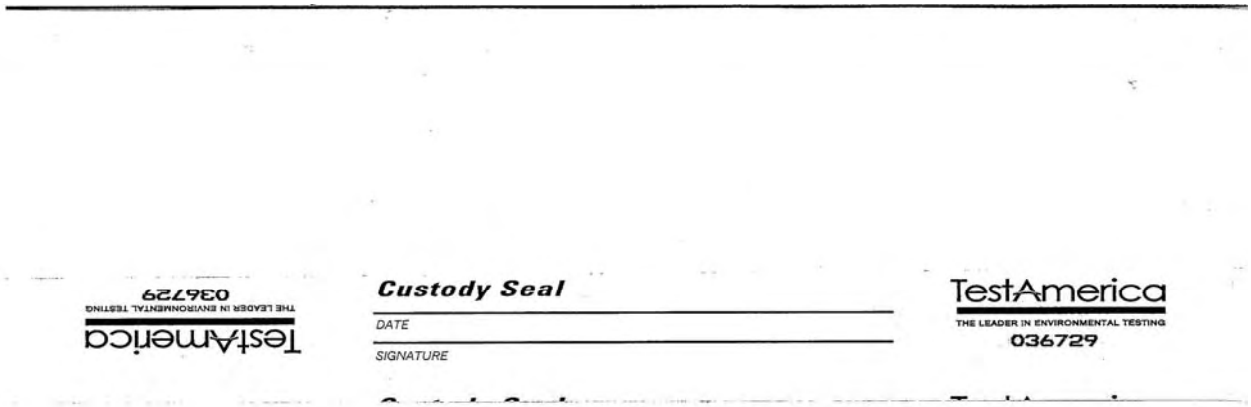


Figure 24-3.
Example: Internal Chain of Custody

Internal Chain-of-Custody ***TestAmerica Denver***

TestAmerica Lot #	**Initial Receipt Date**	**Sample Custodian Initials**
	Samples Stored in Sample Receiving Walk-In	
Sample #s	Location in Walk-In (or Satellite area for strict internal COC samples)	Matrix

Sample #s	Department	Test(s)	Matrix	Date/Time Out	Analyst Initials	Date/Time In	Analyst Initials

Satellite Area Sample Transfers

Satellite Area		**Sample Numbers**	**Transfer Date**	**Storage Location**	**Received by**	**2nd Transfer Date**	**2nd Storage Location**	**Archive Date**	**Archived By**
Aquatic Toxicology									
Metals (aqueous, non-AFCEE)									
GC Volatiles	Water								
	Solid								
MS Volatiles	Water								
	Solid								

C=Comsumed, B=Broken, T=Transfer, LA=Liter Amber, LP=Liter Poly, MDP=Multiple Day Preparation

Figure 24-4.
Example: Disposal Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Test America - Denver
4955 Yarrow Street
Arvada, CO 80002

Sample Transfer Audit Report

LotID	ClientSampID	ContainerID	EventID	ClientName	ClientCd	Quote	TranferType	TransferTime	UserName	StorageLoc	DrumNewLoc	ContType
D7J230212-001	TF2-MW-01	J9L8R-001	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-001	TF2-MW-01	J9L8R-001	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-001	TF2-MW-01	J9L8R-002	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-001	TF2-MW-01	J9L8R-002	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-001	TF2-MW-01	J9L8R-003	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-001	TF2-MW-01	J9L8R-003	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-002	TF2-MW-02	J9L8W-001	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-002	TF2-MW-02	J9L8W-001	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-002	TF2-MW-02	J9L8W-002	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-002	TF2-MW-02	J9L8W-002	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-002	TF2-MW-02	J9L8W-003	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-002	TF2-MW-02	J9L8W-003	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-003	TF2-MW-03	J9L8X-001	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-003	TF2-MW-03	J9L8X-001	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-003	TF2-MW-03	J9L8X-002	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-003	TF2-MW-03	J9L8X-002	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-003	TF2-MW-03	J9L8X-003	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-003	TF2-MW-03	J9L8X-003	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-004	TF2-MW-06	J9L81-001	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-004	TF2-MW-06	J9L81-001	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-004	TF2-MW-06	J9L81-002	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-004	TF2-MW-06	J9L81-002	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-004	TF2-MW-06	J9L81-003	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-004	TF2-MW-06	J9L81-003	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-005	TF2-MW-10	J9L85-001	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-005	TF2-MW-10	J9L85-001	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-005	TF2-MW-10	J9L85-002	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-005	TF2-MW-10	J9L85-002	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-005	TF2-MW-10	J9L85-003	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-005	TF2-MW-10	J9L85-003	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-006	TF2-MW-11M	J9L86-001	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-006	TF2-MW-11M	J9L86-001	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-006	TF2-MW-11M	J9L86-002	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-006	TF2-MW-11M	J9L86-002	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-006	TF2-MW-11M	J9L86-003	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	
D7J230212-006	TF2-MW-11M	J9L86-003	57366		404697	55443	Relocate	12/05/2007 14:11	Chavez, Lawrence	NF	A09	
D7J230212-006	TF2-MW-11M	J9L86-004	51414		404697	55443	Login	10/23/2007 13:07	Bindel, Aaron	NF	NA	

12/12/2007 18:50 Page 1 of 2

Figure 24-5. Sample Receiving Checklist Page 1

Lot #:_____ Date/Time Received:_____

Company Name & Sampling Site:_____

PM to Complete This Section:	*Yes*	*No*		*Yes*	*No*
Residual chlorine check required:	☐	☐	Quarantined :	☐	☐

Quote #:

Special Instructions:

Time Zone:
• EDT/EST • CDT/CST • MDT/MST • PDT/PST • OTHER

Unpacking Checks:

Cooler #(s): _____ _____ _____ _____ _____ _____ _____ _____

Temperatures (°C): _____ _____ _____ _____ _____ _____ _____ _____

N/A	*Yes*	*No*		*Initials*
☐	☐	☐	1. Cooler seals intact? (N/A if hand delivered) If no, document on CUR.	_____
	☐	☐	2. Chain of custody present? If no, document on CUR.	
	☐	☐	3. Bottles broken and/or are leaking? If yes, document on CUR.	
	☐	☐	4. Multiphasic samples obvious? If yes, document on CUR.	
	☐	☐	5. Proper container & preservatives used? (ref. Attachment D of SOP# DEN-QA-0003) If no, document on CUR.	
☐	☐	☐	6. pH of all samples checked and meet requirements? If no, document on CUR.	
	☐	☐	7. Sufficient volume provided for all analysis requested? (ref. Attachment D of SOP# DEN-QA-0003) If no, document on CUR, and contact PM before proceeding.	
	☐	☐	8. Did chain of custody agree with labels ID and samples received? If no, document on CUR.	
☐	☐	☐	9. Were VOA samples without headspace? If no, document on CUR.	
☐	☐	☐	10. Were VOA vials preserved? Preservative ☐HCl ☐4±2°C ☐Sodium Thiosulfate ☐ Ascorbic Acid	
	☐	☐	11. Did samples require preservation with sodium thiosulfate?	
☐	☐	☐	12. If yes to #11, did the samples contain residual chlorine? If yes, document on CUR.	
☐	☐	☐	13. Sediment present in dissolved/filtered bottles? If yes, document on CUR.	
☐	☐	☐	14. Is sufficient volume provided for client requested MS, MSD or matrix duplicates? If no, document on CUR, and contact PM before proceeding.	
	☐	☐	15. Receipt date(s) > 48 hours past the collection date(s)? If yes, notify PA/PM.	
	☐	☐	16. Are analyses with short holding times requested?	
	☐	☐	17. Was a quick Turn Around (TAT) requested?	

Figure 24-5. Sample Receiving Checklist Page 2

Lot #_____

Login Checks: *Initials*

N/A	*Yes*	*No*		
	☐	☐	18. Sufficient volume provided for all analysis requested? (ref. Attachment D of SOP# DEN-QA-0003) If no, document on CUR, and contact PM before proceeding.	_____
☐	☐	☐	19. Is sufficient volume provided for client requested MS, MSD or matrix duplicates? If no, document on CUR, and contact PM before proceeding.	
	☐	☐	20. Did the chain of custody includes "received by" and "relinquished" by signatures, dates, and times?	
☐	☐	☐	21. Were special log in instructions read and followed?	
☐	☐	☐	22. Were AFCEE metals logged for refrigerated storage?	
	☐	☐	23. Were tests logged checked against the COC? Which samples were confirmed? _____	
☐	☐	☐	24. Was a Rush form completed for quick TAT?	
☐	☐	☐	25. Was a Short Hold form completed for any short holds?	
	☐	☐	26. Were special archiving instructions indicated in the General Comments? If so, what were they?	

_____

Labeling and Storage Checks: *Initials*

_____

☐	☐	☐	28. Was the subcontract COC signed and sent with samples to bottle prep?	
	☐	☐	29. Were sample labels double-checked by a second person?	
☐	☐	☐	30. Were sample bottles and COC double checked for dissolved/filtered metals by a second person?	
	☐	☐	31. Did the sample ID, Date, and Time from label match what was logged?	
☐	☐	☐	32. Were stickers for special archiving instructions affixed to each box and to the ICOC? See #27	
☐	☐	☐	33. Were AFCEE metals stored refrigerated?	

Document any problems or discrepancies and the actions taken to resolve them on a Condition Upon Receipt Anomaly Report (CUR).

FIGURE 24-6 CONDITION UPON RECEIPT ANOMALY REPORT (CUR)

TestAmerica Denver
Condition Upon Receipt Anomaly Report (CUR)

Lot No : _____ Date/Time:_____

Client : _____ Initiated by: _____

Affected Samples COC#_____

Client ID	Lab ID	Analyses Requested

CONDITION/ANOMALY/VARIANCE (CHECK ALL THAT APPLY):

❑COOLERS
- ❑Received, No Chain of Custody (COC)
- ❑Not Received but COC(s) Available
- ❑Leaking
- ❑Other: _____

❑ TEMPERATURE (greater than 6° C)
- ❑Cooler Temp_____
- ❑Temperature Blank _____

❑CONTAINERS
- ❑Leaking
- ❑Broken
- ❑Extra
- ❑Without Labels
- ❑VOA Vials with Headspace _____mm
- ❑Other: _____

❑CUSTODY SEALS (COOLER(S)/CONTAINER(S)
- ❑None
- ❑ Not Intact
- ❑Other: _____

❑CHAIN OF CUSTODY (COCs)
- ❑Not relinquished by Client; No date/time Relinq.
- ❑ Incomplete Information
- ❑Other: _____

❑ CONTAINER LABELS
- ❑Not the same ID/info as in COC
- ❑ Incomplete

❑ID COLLECTION ❑Time ❑Date ❑PRESERVATIVE
- ❑ Markings/Info smeared or illegible
- ❑Torn
- ❑Other: _____

❑ SAMPLES

❑Samples NOT RECEIVED but listed on COC ------ ❑will be noted on COC ❑Client to send samples with new COC
- ❑Samples received but NOT LISTED on COC
- ❑Logged based on Label Information
- ❑Logged based on info from other samples on COC
- ❑ Logged according to Work Plan
- ❑Logged on HOLD UNTIL FURTHER NOTICE
- ❑ Other: _____ _____

- ❑ Trip Blank received, not on COC, _____vials received
- ❑ Mislabeled as to tests, preservatives, etc.
- ❑Holding time expired
- ❑ Improper container used
- ❑Not preserved / Improper preservative used
- ❑ Improper pH _____ ❑ Lab to preserve sample
- ❑ Insufficient quantities for analysis

Comments:_____
_____
_____

Corrective Action:
- ❑ Client Informed: verbally on: _____By: _____: In writing on: _____ By: _____
- ❑ Sample(s) processed "as is". _____
- ❑ Sample(s) on hold until: _____ If released, notify: _____

Sample Control Supervisor Review: _____ Date: _____

Project Management Review: _____ Date: _____

FIGURE 24-7 Labeling for Waste Disposal

Shipping Label Requirements for Waste

Waste Code	Waste Stream	Drum Type	Label information	DOT Label
A	**Expired Extract Vials**	**Steel- 0pen Head**	**RQ WasteSolids containing Flammable Liquids, n.o.s (Hexane, Acetone, Methanol), 4.1, UN3175, PGII, (D001)**	**Flammable Solid, Class 4.1**
B	**Waste Dichlormethane**	**Steel- Bung Top**	**Waste Dichloromethane, 6.1, UN1593, PG III, (Methylene Chloride), F002**	**Toxic, Class 6.1**
C	**Flammable Solvent**	**Steel-Bung Top**	**RQ Waste Flammable Liquids, n.o.s. (Hexane, Acetone), 3, UN1993, PG II, (D001)**	**Flammable Liquid, Class 3**
D	**Sodium Sulfate**	**Steel-Open Head**	**Non DOT Regulated Material, (Sodium Sulfate)**	**None**
E	**Aqueous Alkaline**	**HDPE-Bung Top**	**RQ, Waste Corrosive Liquids, basic, Inorganic, n.o.s. (Sodium Hydroxide), 8, UN3266, PG II, (D002)**	**Corrosive, Class 8**
F	**Aqueous Acidic**	**HPDE-Bung Top**	**RQ Waste Corrosive Liquid, Acidic, Inorganic, n.o.s. (Sulfuric Acid, Hydrochloric Acid), 8, UN3264, PG II (D002)**	**Corrosive, Class 8**
G	**Aqueous Acidic**	**HDPE-Bung Top**	**Pending Characterization/Process Knowledge**	**Pending Characterization/process knowledge**
H	**Aqueous Acidic**	**HDPE-Bung Top**	**RQ Waste Corrosive Liquid, Acidic, Inorganic, n.o.s. (Sulfuric Acid, Hydrochloric Acid), 8, UN3264, PG II (D002)**	**Corrosive, Class 8**
I	**COD Vials**	**HDPE- Open Head**	**RQ Waste Sulfuric Acid Solution (Sulfuric acid, Chromium, Mercury, Silver) 8, UN1830, PG II, (D002,D007,D009,D011)**	**Corrosive, Class 8**
J	**Aqueous Acidic**	**HDPE-Bung Top**	**Pending Characterization/Process Knowledge**	**Pending Characterization/process knowledge**
M	**Miscellaneous Waste**	**Variable**	**Pending Characterization/process knowledge**	**Pending Characterization/process knowledge**
O	**Used Pump Oil**	**HDPE-Bung Top**	**Non-RCRA Regulated Material, (Pump Oil)**	**None**
P	**Solid Laboratory Waste**	**Steel- Open Head**	**Environmentally Hazardous Substances, Solid, n.o.s. 9, UN3077, PG III, (Soil, Anhydrous, Rubber Gloves)**	**Miscellaneous Dangerous Goods, Class 9**

Shipping Label Requirements for Waste

Waste Code	Waste Stream	Drum Type	Label information	DOT Label
S	**Excess Sample – Solid**	**Steel- Open Head**	**Non DOT Regulated Material, (Soil Samples)**	**Pending Characterization/process knowledge**
W	**Excess Sample – Aqueous**	**HDPE-Bung Top**	**Pending Characterization/process knowledge**	**Pending Characterization/process knowledge**
RAD followed by the Waste Code Listed Above	**Radioactive (RAD) –Could Apply to Any of the Waste Streams Listed Above**	**Per 49 CFR 171 –173 and TSDF**	**Per 49 CFR 171 –173 and TSDF**	**Per 49 CFR 171-173 and TSDF**

Note: If characterization determines a waste is hazardous, labeling shall meet the requirements of 49 CFR 171-180. This table does not supersede 49 CFR 171-180.

SECTION 25.0

ASSURING THE QUALITY OF TEST RESULTS
(NELAC 5.5.9)

25.1 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 21, 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.

25.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. 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.

25.3 NEGATIVE CONTROLS

25.3.1 **Method Blanks** are used to assess preparation and analysis for possible contamination during the preparation and processing steps.

25.3.2 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.

25.3.3 The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

25.3.4 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.

25.3.5 Evaluation criteria and corrective action for method blanks is defined in the specific standard operating procedure for each analysis. Generally, corrective action is taken if the concentration of a target analyte in the blank is at or above the reporting limit.

- The source of contamination is investigated

- Measures are taken to minimize or eliminate the source of the contamination
- Affected samples are reprocessed or the results are qualified on the final report.

25.3.6 **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.

25.3.7 **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.

25.3.8 **Trip Blanks** are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank 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. Trip Blanks are also sometimes referred to as Travel Blanks.

25.3.9 **Field Blanks** 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)

25.3.10 **Equipment Blanks** 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. (NELAC)

25.3.11 **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 (refer to section 24.4 and SOP DV-QA-0013, *Refrigerator Blank and Trip Blank Monitoring*).

25.3.12 **Field blanks**, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. When known, blanks 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".

25.3.13 Negative Controls for Microbiological Methods

Microbiological Methods utilize a variety of negative controls throughout the process to ensure that false positive results are not obtained. These controls are critical to the validity of the microbiological analyses. Some of these negative controls are: Sterility checks of media are analyzed for each lot of pre-prepared media, ready-to-use media and for each batch of medium prepared by the laboratory.

25.3.13.1 Filtration blanks are run at the beginning and end for each sterilized filtration unit used in a filtration series.

25.3.13.2 Sterility checks on sample containers are performed on at least one container per lot of purchased, pre-sterilized containers. Container sterility checks are performed using non-selective growth media.

25.3.13.3 Sterility checks are performed on each batch of pre-prepared dilution water. All checks are performed using non-selective growth media.

25.3.13.4 Sterility checks are also performed on at least one filter from each new lot of membrane filters using non-selective growth media.

25.3.13.5 Negative culture controls demonstrate that a media does not support the growth of non-target organisms and ensures that there is not an atypical positive reaction from the target organisms. Prior to the first use of the media, each lot of pre-prepared selective media or batch of laboratory prepared selective media is analyzed with at least one known negative culture control as appropriate to the method.

25.4 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), 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.

25.4.1 Method Performance Control - Laboratory Control Sample (LCS)

25.4.1.1 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.

25.4.1.2 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 may reported as the LCS.

25.4.1.3 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.).

25.4.1.4 As stated in the opening of this section, the LCS goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).

25.4.1.5 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.

25.4.1.6 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.

25.4.1.6.1 For methods that have 1-10 target analytes, spike all components.

25.4.1.6.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

25.4.1.6.3 For methods with more than 20 target analytes, spike at least 16 components.

25.4.1.6.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

25.4.1.6.5 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.

25.4.1.7 **Accuracy Calculation**: Percent Recovery (%R) Calculation (applies to LCS, CCV, Surrogates, and Matrix Spikes.

$$\%R = \frac{AV}{TV} \times 100$$

Where: AV = Analyzed Value
TV = True Value

25.4.2 Positive Controls for Microbiological Methods

Prior to the first use of the media, each lot of pre-prepared media is tested with at least one pure culture of known positive reaction.

25.5 SAMPLE MATRIX CONTROLS

25.5.1 Matrix Spikes (MS)

25.5.1.1 The Matrix spike is 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.

25.5.1.2 An MS is essentially a sample fortified with a known amount of the test analyte(s). 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.

25.5.1.3 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. 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, a representative number of the listed components (see LCS analytes 25.4.1.6 above) may 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.

25.5.1.4 The percent recovery calculation for matrix spikes is essentially the same as the calculation shown in 25.4.1.7 except that:

AV = Sp – Sa

Where: Sp = Spike result

Sa = Sample result

25.5.2 Surrogate Spikes

25.5.2.1 Surrogate Spikes 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.

25.5.2.2 Surrogate compounds 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 (also refer to Section 25.5). 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.

25.5.3 Duplicates

25.5.3.1 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. Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. 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.

25.5.3.2 **Precision Calculation** (Relative Percent Difference - RPD)

$$RPD = \frac{|S - D|}{\frac{(S + D)}{2}} \times 100$$

Where: S=Sample Concentration
D=Duplicate Concentration

25.5.4 Internal Standards

25.5.4.1 In most organic analyses, internal standards are spiked into all environmental and quality control samples (including the initial calibration standards). An internal standard is also used with some metals analyses. It is typically added to sample extracts after the extraction (post-prep). The acceptance criteria in most methods are 50% to 200% of the responses in the mid-point of the corresponding calibration curve. Consult the method-specific SOPs for details on the internal standard compounds, calculations and acceptance criteria.

25.5.4.2 When the internal standard recoveries fall outside these limits, if there are not obvious chromatographic interferences, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets internal standard recovery criteria, the second run is reported (or both are reported if requested by the client).

25.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

25.6.1 Each individual analyte in the LCS, MS, or Surrogate Spike are evaluated against the control limits as published in the test method. Where there are no established acceptance criteria, the laboratory calculates control limits with the use of control charts or, in some cases, utilizes client project specific or regulatory mandated 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.

25.6.2 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 (e.g. EPA SW846 8000 series methods). Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

25.6.2.1 The lab should consider the effects of the spiking concentration control limits, and to avoid censoring of data. The acceptance criteria for recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling data to generate control limits.

25.6.2.2 Not only should the results all be from a similar matrix, but the spiking levels should also be approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results should all be generated using the same set of extraction, cleanup and analysis techniques. For example, results from solid samples extracted by ultrasonic extraction are not mixed with those extracted by Soxhlet.

25.6.2.3 The laboratory should try and avoid discarding data that do not meet a preconceived notion of acceptable performance. This results in a censored data set, which, when used to develop acceptance criteria, will lead to unrealistically narrow criteria. For a 99% confidence interval, 1 out of every 100 observations likely will still fall outside the limits. For methods with long analyte lists this may mean occasional failures every batch or two. While professional judgment is important in evaluating data to be used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. However, data points shall be discarded if they were the result of human or mechanical error or sample concentration exceeded spike level by > 4x.

25.6.3 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 20-30 data points (more points are preferred).

25.6.3.1 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).

25.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method.

25.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable.

25.6.3.4 The maximum acceptable recovery limit will be 150%.

25.6.3.5 The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

25.6.3.6 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.

25.6.3.7 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. Refer to SOP DV-QA-003P for details.

25.6.4 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 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

25.6.4.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

25.6.4.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

25.6.4.3 Or, for NELAC and Department Of Defense (DOD) work, there are an allowable number of 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

25.6.4.3.1 Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).

25.6.4.3.2 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.

25.6.4.3.3 Though marginal excedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

25.6.5 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 Appendix 4 and in Section 13.

25.6.6 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.

25.7 METHOD DETECTION LIMITS (MDLs)

MDLs, calculated as described in Section 20.7, are updated or verified annually, or more often if required by the method.

25.8 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

25.8.1 The laboratory has written procedures to assure the accuracy of the test method including calibration (see Section 21), use of certified reference materials (see Section 22) and use of PT samples (see Section 16).

25.8.2 A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 20.

25.8.3 Use of formulae to reduce data is discussed in the method standard operating procedures and in Section 21.

25.8.4 Selection of appropriate reagents and standards is included in Section 9 and 22.

25.8.5 A discussion on selectivity of the test is included in Section 5.

25.8.6 Constant and consistent test conditions are discussed in Section 19.

25.8.7 The laboratories sample acceptance policy is included in Section 24.

25.8.8 A listing of the type of test result correlations that are looked at during report review (e.g. Total Chromium should be greater or equal to Hexavalent Chromium) is included in Section 20.13.4.5.

SECTION 26.0

REPORTING RESULTS
(NELAC 5.5.10)

26.1 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 a conflict between the client requested formats and accreditation requirements or data usability information, accreditation requirements and data usability information will take precedence over client requests. 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 20.

26.2 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, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

26.2.1 A report title (e.g. Analytical Report For Samples) with a “sample results” column header.

26.2.2 The report cover page is printed on company letterhead, which includes the laboratory name, address and telephone number.

26.2.3 A unique identification of the report (e.g. lot 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: The total number of pages is indicated at the front of each report.

26.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

- 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 (eg. Sampling information).

26.2.5 The name and address of client and a project name/number, if applicable.

26.2.6 Client project manager or other contact

26.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

26.2.8 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.

26.2.9 Date reported or date of revision, if applicable.

26.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

26.2.11 Reporting limits.

26.2.12 Method detection limits (if requested)

26.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

26.2.14 Sample results.

26.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

26.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 26.2.4 – Item 2 regarding additional addenda).

26.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

26.2.18 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

26.2.19 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

26.2.20 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not. For Example:

"The results included in this report have been reviewed for compliance with the laboratory QA/QC plan and meet all requirements of NELAC. All data have been found to be compliant with laboratory protocol and any exceptions are noted below. "

26.2.21 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.

26.2.22 When Soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

26.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

26.2.24 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 "partial report", and that a complete report will follow once all of the work has been completed.

26.2.25 Any out of network subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All in-network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

26.3 REPORTING LEVEL OR REPORT TYPE

TestAmerica Denver 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 I is a report with the features described in Section 26.2 above.
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, 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. A Level II report is not included, unless specifically requested. No raw data is provided.
- 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 diskette deliverable form. 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 26.7.

26.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Denver offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, SEDD, NWIS, Dbase, GISKEY, Text Files, and a number of client specific formats.

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.

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.

26.4 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. Refer to Appendix 7 for a list of the laboratory's standard footnotes and qualifiers.

26.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

26.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

26.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

26.4.4 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.

26.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If TestAmerica Denver is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in Section 8.

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 the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

26.6 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.

26.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

26.7 FORMAT OF REPORTS

The format of reports are designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

26.8 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 13). Refer to SOP DV-QA-019P, *Result and Report Revisions*.

When the report is re-issued, a notation of "revised report ", is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the revision. *For Example*: *Report was revised on 11/3/07 to include toluene in sample NQA1504 per client's request*

26.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

26.9.1 Sample Reanalysis Policy

Because there is a certain level of uncertainty with any analytical measurement a sample 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 arrangements for reanalysis protocols can be established.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 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 and reanalyze the sample a third time for confirmation if sufficient sample is available.

- 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.

26.9.2 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.

26.9.3 Multiple Reports

TestAmerica does not issue multiple reports for the same workorder 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.

TESTAMERICA
ETHICS POLICY No. CA-L-P-001

Refer to CA-L-P-001 for complete policy.

TestAmerica
EMPLOYEE ETHICS STATEMENT

I understand that TestAmerica is committed to ensuring the highest standard of ethical and professional conduct in all business activities. The Company and its employees will comply with all applicable laws, regulations, and policies. We will ensure the highest standards of quality and integrity of the data and services we provide to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform, the data I report in connection with my employment at the Company, and all business activites, I agree that:

- *I will not intentionally report data values that are inconsistent with the actual values observed or measured.*
- *I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations.*
- *I will not intentionally misrepresent another individual's work as my own or represent my own work as someone else's.*
- *I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers; I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the laboratory's Standard Operating Procedures, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.*
- *I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers. I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions.*
- *I will not, through acts of commission, omission, erasure, or destruction improperly report measurement standards, quality control data, test results, or conclusions; nor will I intentionally alter or omit dates, dollar values, or other business related information in order to achieve desired financial results.*
- *I shall not compare or disclose results for any Performance Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica laboratory, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.*
- *I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Quality Assurance Manager. The Quality Assurance Manager will initial and date the information and return a copy to me. I shall not condone any accidental or intentional reporting of inauthentic data by other*

employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of Management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.

- *I understand that if any supervisor, manager, or representative of TestAmerica management instructs, requests, or directs me to perform any of the aforementioned improper laboratory practices, or if I am in doubt or uncertain as to whether or not such laboratory practices are proper, I will not comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Lab Director, all supervisors and managers with direct line reporting relationship between me and the Lab Director, and the local Quality Assurance representative, excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, I may contact Corporate Quality Assurance / Ethics Compliance Officer(s) for assistance.*
- *I understand the critical importance of accurately reporting data, measurements, and results, whether initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or retained by TestAmerica for subsequent internal use;*
- *I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the TestAmerica family of companies.*
- *I shall not accept gifts of a value that would adversely influence judgment.*
- *I shall avoid conflicts of interest and report any potential conflicts to the management (e.g. employment or consulting with competitors, clients, or vendors).*
- *I shall not participate in unfair competition practices (e.g. slandering competitors, collusion with other labs to restrict others from bidding on projects).*
- *I shall not misrepresent certifications and status of certifications to clients or regulators.*
- *I shall not intentionally discharge wastes illegally down the drain or onto the ground.*
- *I shall not take any action, personally, or on behalf of the Company, which violates any applicable law, regulation, or internal policy, or which causes the Company to incur financial risk or loss or causes the Company to report incorrect financial information.*
- *I understand that any attempt by management or an employee to circumvent these policies will be subject to disciplinary action.*

As a TestAmerica employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

I understand that all of my dealings as an employee must be in compliance with applicable Federal and State laws, including safety regulations, environmental regulations, accounting rules, and employment laws, such as Drug Free Workplace Act and anti-discrimination and harassment legislation.

I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination of my employment. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE _____ Date _____

Supervisor/Trainer: _____ Date _____

TestAmerica
CONFIDENTIALITY AND PROPRIETARY INFORMATION AGREEMENT

TestAmerica and their predecessors, in their businesses, have developed and use commercially valuable technical and non-technical information and to guard the legitimate interests of TestAmerica and its clients, it is necessary to protect certain information as confidential and proprietary.

I, _____ , understand and acknowledge that during the term of my employment by TestAmerica, I will be privy to and entrusted with certain confidential information and trade secrets of TestAmerica and its clients.

Confidential information and trade secrets include, but are not limited to: customer and client lists; price lists; marketing and sales strategies and procedures; operational and equipment techniques; standard operating procedures; business plans and systems; quality control procedures and systems; special projects and technological research, including projects, research and reports for any government entity or client; client's plans and processes; client's manner of operation; the trade secrets of clients; client's data; vendor or supplier pricing; employee lists and personal information, and any other records, data, files, drawings, inventions, discoveries, applications, or processes which are not in the public domain.

I agree as follows:

1. I will not in any way, during the term of my employment, or at any time thereafter, except as authorized in writing by the Legal Department of TestAmerica or the client where client data is involved, disclose to others, use for my own benefit, remove from TestAmerica's premises (except to the extent off-site work is approved by my supervisor), copy or make notes of any confidential information and/or trade secrets of TestAmerica or its clients, excepting only that information which may be public knowledge. Technical and business information of any previous employer or other third party which I may disclose to TestAmerica shall be limited to that which was acquired legitimately and disclosed to me without restriction as to secrecy.

2. I agree that all inventions (whether or not patentable) conceived or made by me during the period of my employment by TestAmerica shall belong to TestAmerica, provided such inventions grow out of my work for TestAmerica and are related to the business of TestAmerica. I agree to disclose and assign such inventions to TestAmerica. In California, this provision shall not apply to any invention which qualifies fully under Section 2870 of the California Labor Code.

3. On termination of my employment from TestAmerica, I will deliver to TestAmerica all documents, records, notes, data, memoranda, files, manuals, equipment and things of any nature which relate in any way to confidential information and/or trade secrets of TestAmerica or its clients and which are in my possession or under my control.

4. I agree that during the period of my employment and for one (1) year from and after the termination (for any reason) of my employment with TestAmerica, I shall not directly or indirectly (without first obtaining the written permission of TestAmerica), recruit for employment, or induce to terminate his or her employment with TestAmerica, any person who is an active employee of TestAmerica on the last day of my employment with TestAmerica.

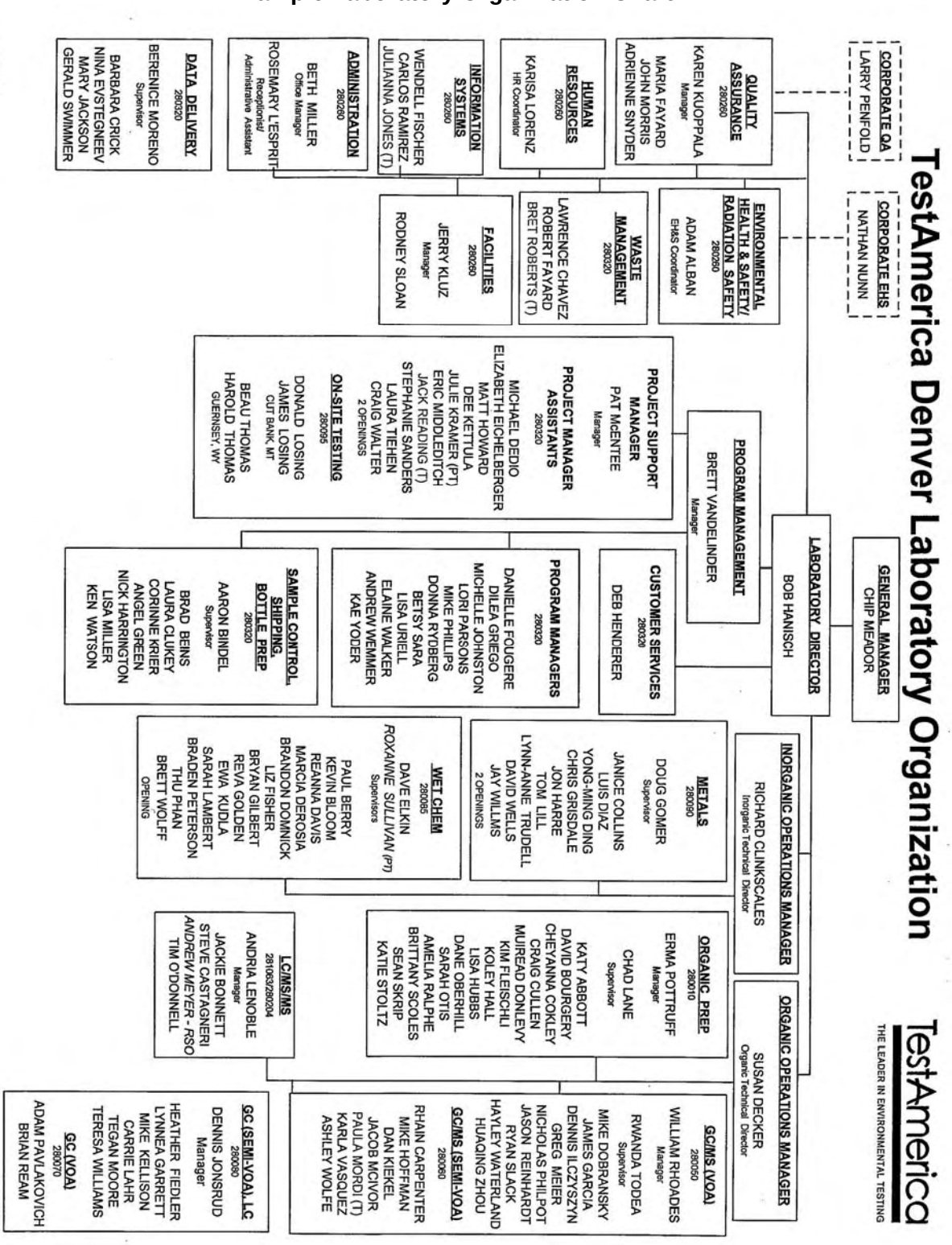
5. I acknowledge that if I were to breach any provision of this Confidentiality Agreement, money damages will be inadequate, and I hereby agree that TestAmerica shall be entitled, where appropriate, to specific performance and/or injunctive relief (i.e. to require me to comply with this Agreement). I further acknowledge that the willingness of TestAmerica to hire me or to continue my employment constitutes full and adequate consideration for the agreements, and obligations to which I have agreed as set forth in this document.

I have executed this Agreement, intending to be legally bound.

_____	_____	_____
Printed Name	*Signature*	*Date*

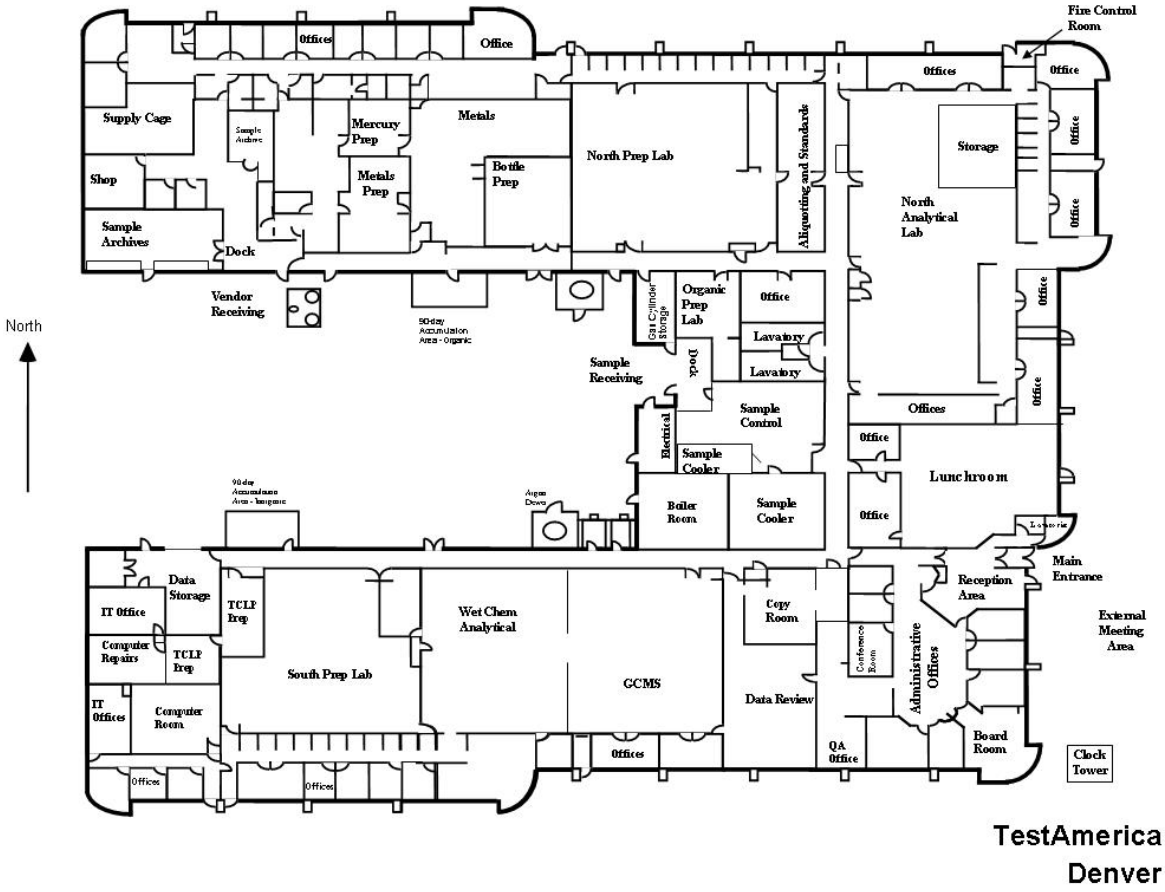
Appendix 2.

Example Laboratory Organization Chart



Appendix 3.

Laboratory Floor Plan



Criteria in Appendix 4 are to be used for general guidance. Method or Program specific criteria take precedence.
For methods not listed (SW6020, SW8321, SW6860, Hydrazine) refer to the analytical SOPs.

Appendix 4: Summary of Calibration and QC Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[3]	Corrective Action[4]
SW8081 SW8082 SW8141 SW8151	Minimum five-point initial calibration for all target analytes[2]	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	Linear regression correlation coefficient $r^2 \geq 0.99$, $r \geq 0.995$. RSD of CF ≤ 20%	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source	Once immediately following initial calibration	All target analytes within 15% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value and within the RT Window[7].	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Breakdown check (Endrin and DDT)[1]	Before sample analysis	Degradation ≤15% for either Endrin or DDT.	Inlet/column maintenance; repeat breakdown check and re-analyze all samples since last successful breakdown check.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected ≥ RL	Correct problem then re-prep[6] and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch, not to exceed 20 samples in a batch.	See Control Limits in LIMS or Clouseau	Re-prep[6] and analyze the LCS and all samples in the affected analytical batch
	Surrogate(s)	Every sample, spike, standard, and method blank	See Control Limits in LIMS or Clouseau	Check system, re-inject, re-extract[6]
	MS/MSD	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits in LIMS or Clouseau	None (LCS is used to determine if data is acceptable).
	Second-column confirmation	100% for all positive results Only applies to 8082 for specific programs (see SOP DV-QA-024P for federal program Requirements)	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. If questions, see the supervisor or technical director.
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 --8081A only
2 – Method 8082, a five-point calibration is only analyzed for Aroclors 1016 and 1260.
3 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
4 - All abnormalities must be noted in a NCM.
6 - If unable to re-extract the samples because of insufficient sample volume or holding time has expired a NCM must be generated.
7 - The mean of all calibrated compounds may be used, but all compounds above the 15% must be documented in a NCM.

Appendix 4: Summary of Calibration and QC Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[3]	Corrective Action[4]
EPA608 EPA615	Minimum three-point (preferably five) initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	**RSD of CF ≤ 10%** Linear regression - correlation coefficient r ≥ 0.99, r ≥ 0.995.	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, and at the end of the analysis sequence	All analytes within **15% of expected value** and within the RTW.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Breakdown check (Endrin and DDT)[1]	Before sample analysis	Degradation ≤15% for either Endrin or DDT.	Inlet/column maintenance; repeat breakdown check and re-analyze all samples since last successful breakdown check.
	Method blank	One per analytical prep batch, **not to exceed 10 samples in a batch**.	No analytes detected ≥ RL	Correct problem then re-prep[7] and analyze method blank and all samples processed with the contaminated blank
	LCS all analytes	One per prep batch, **not to exceed 10 samples in a batch.**	See Control Limits in LIMS or Clouseau	Re-prep[7] and analyze the LCS and all samples in the affected analytical batch
	Surrogate(s)	Every sample, spiked sample, standard, and method blank	See Control Limits in LIMS or Clouseau	Check system, re-inject, re-extract[7]
	MS	One per batch per matrix, 10%, if insufficient sample for MS, then an additional LCS will be analyzed.	See Control Limits in LIMS or Clouseau	All target compounds should be reported, and any compounds that are outside criteria must be within criteria in the LCS.
	Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or as needed.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. If questions, see the supervisor or technical director.
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

3 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
4 - All abnormalities must be documented in a NCM.
6 - Report all target compounds identified in the method blank above the MDL.
7 - If unable to re-extract the samples because of insufficient sample volume or holding time has expired, then a NCM must be generated

Appendix 4: Summary of Calibration and QC Procedures for GC/MS Organics

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
SW8260 SW8270	Check of mass spectral ion intensities[1], i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours	Refer to criteria listed in the method SOP for Tune criteria, including DDT, Benzidine and Pentachlorophenol requirements for 8270.	Retune the instrument and verify (instrument maintenance may be needed).
SW8260	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	SPCCs average RF $\geq$ 0.30 or 0.1 depending on the compound and %RSD for RFs for CCCs $\leq$ 30% and all other target analytes %RSD for RF $\leq$ 15%.	Correct problem then repeat initial calibration
SW8270			SPCCs average RF $\geq$ 0.050 and %RSD for RFs for CCCs $\leq$ 30% and all other target analytes %RSD for RF $\leq$ 15%.	Correct problem then repeat initial calibration
			option (if %RSD is > 15%)–linear regression $r^2 \geq 0.99$, $r \geq 0.995$.	If the calibration is not considered linear by either %RSD or linear regression, then correct the problem and re-calibrate.
SW8260 SW8270	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following five-point initial calibration	All analytes within 25% of expected value	Correct problem then repeat initial calibration
	Relative Retention time window	Each sample	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
SW8260	Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF $\geq$ 0.30 or 0.1 depending on the compound; and	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
SW8270			SPCCs average RF $\geq$ 0.050; and	
SW8260 SW8270	Continuing calibration check		CCCs: $\leq$20% difference (when using RFs) or drift (when using least squares regression). **All other target compounds ≤20%, up to 5 non-CCC target compounds, may fail this requirement provided the % difference is ≤ 40%.**	
SW8260 SW8270	Method blank	One per analytical prep batch	No analytes detected $\geq$ RL	Correct problem then re-prep[5] and analyze method blank and all samples processed with the contaminated blank

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
SW8260 SW8270	Internal Standards	Every sample/standard and blank	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples. (See federal programs SOP DV-QA-024P for program specific requirements)	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the technical director for advice).
	LCS	One per prep batch, not to exceed the 20 samples in a batch.	See Control Limits in LIMS or Clouseau	Correct problem then re-prep[5] and analyze the LCS and all samples in the affected analytical batch
	MS/MSD	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits in LIMS or Clouseau	None (the LCS is used to evaluate to determine if the batch is acceptable).
	Surrogate(s)	Every sample, spike, standard, and blank	See Control Limits in LIMS or Clouseau	Check system, re-analyze, re-prep[5]
SW8260	pH check	All 8260 water samples.	pH ≤2.	If the pH is > 2, then a NCM must be generated
SW8260	Residual chlorine check (North Carolina samples only)	Each sample.	Residual chlorine should be negative.	If the residual chlorine is positive, then document in a NCM.
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 – SW8260B requires BFB; SW8270C requires DFTPP
2 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
3 - All abnormalities must be documented in a NCM.
4 - Report all target compounds identified in the method blank above the MDL.
5 - If unable to re-prep samples because of insufficient sample volume or the holding time has expired, then a NCM must be generated.

Appendix 4: Summary of Calibration and QC Procedures for GC/MS Organics

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
EPA624 EPA625	Check of mass spectral ion intensities[1] (i.e. Tune)	Prior to initial calibration or Continuing calibration verification every 12 hours.	Refer to criteria listed in the method SOP for Tune requirements including DDT, Benzidine and Pentachlorophenol criteria for 625.	Retune instrument and verify instrument maintenance may be needed.
	Five- point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	%RSD < 35%, if %RSD is > 35% then linear regression is used (for linear regression $r^2 \geq 0.99$), $r \geq 0.995$.	If the calibration is not considered linear by either %RSD or linear regression, then correct problem then repeat initial calibration
	Initial calibration verification (ICV), 20 ug/L, must be from a 2nd source. May be the same as the LCS.	Immediately following initial calibration	See analytical SOP.	Correct problem then repeat initial calibration
	Relative Retention time window	Each sample	Retention time (RT) of the analyte within 30 seconds of the RT (± 0.25 min. RTW is used) of the target.	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
EPA625	Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time.	All calibration analytes within 20% of expected value	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
EPA624 EPA625	Method blank	One per prep batch (not to exceed 20 samples per batch).	No analytes detected ≥ RL	Correct problem then re-prep[5] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes.	One per prep batch (not to exceed 10 samples per batch) or daily.	See Control Limits in LIMS or Clouseau	Correct problem then re-prep[5] and analyze the LCS and all samples in the affected analytical batch
	MS	One per batch of 10 per matrix, if insufficient sample for MS, then a duplicate LCS will be analyzed.	See Control Limits in LIMS or Clouseau	All target compounds should be reported, and any compound that is outside criteria must be within criteria in the LCS.
	Surrogate(s)	Every sample, spiked sample, standard, and method blank	See Control Limits in LIMS or Clouseau	Correct problem then re-prep[5] and analyze sample
EPA624 EPA625	Internal Standards	Every sample/standard	Retention time ±30 seconds from retention time of the mid-point std. in the CCV/ICAL (sample/standard). EICP area within -50% to +100% of ICAL mid-point std for the CCV and –50% to +100% of the prior CCV for the samples.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the technical director for advice).
EPA624	pH check	All 624 samples after analysis	pH should be ≤ 2.	If the pH is > 2, then document in a NCM.
EPA624	Residual chlorine check (North Carolina samples only)	All samples after analysis	Residual chlorine should be negative.	If the residual chlorine is positive, then document in a NCM.

Method	QC Check	Frequency	Acceptance Criteria[2]	Corrective Action[3]
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 – 624 requires BFB; 625 requires DFTPP
2 - This is summary of the acceptance criteria, refer to the method SOP for specific or more information.
3 - All abnormalities must be documented in a NCM
4 - Report all target compounds identified in the method blank above the MDL.
5 - If unable to re-prep samples because of insufficient sample volume or holding time has expired, then generate a NCM

Appendix 4: Summary of Calibration and QC Procedures for Method SW8310

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8310	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	CF RSD for each analyte ≤20% or mean RSD for all analytes ≤20%, with all compounds above 20% commented in LIMS with each sample. linear – $r^2 \geq 0.99$, $r \geq 0.995$.	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Retention time verification	Update at start of run or daily	All standards within window	Correct problem then re-analyze all samples analyzed since the last retention time check
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value	Correct problem then reprocess or repeat initial CCV and re-analyze all samples since last successful CCV.
	Method blank	One per prep batch (not to exceed more than 20 samples per batch).	No analytes detected ≥ ½ RL	Correct problem then re-prep[4] and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch (not to exceed more than 20 samples per batch).	See Control Limits in LIMS or Clouseau	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Surrogate	Every sample, spike, standard, and method blank	See Control Limits in LIMS or Clouseau	Check system, re-inject, re-extract[4]
	MS/MSD	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits in LIMS or Clouseau	None (LCS is used to determine if the batch is acceptable).
	Confirmation	100% for all positive results (use response of both detectors)	Same as for initial or primary analysis. Comment LIMS if >40% difference in compound response between detectors.	Same as for initial or primary analysis.
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific information or more information.

2 - All abnormalities must be documented in a NCM.

3 - Report all target compounds identified in the method blank above the MDL.

4- If unable to re-extract because of insufficient sample volume or the holding time has expired, then a NCM must be generated.

Appendix 4: Summary of Calibration and QC Procedures for Method EPA610 (HPLC)

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
EPA610 (HPLC)	Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF of each analyte **<10%,** $r^2 \geq$ 0.99, r ≥ 0.995, or linear regression.	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Retention time verification	Update at start of run or daily	All standards within window	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
	Continuing calibration verification (CCV)	Before sample analysis and at the end of the analysis sequence	All analytes within 15% of expected value	Correct problem then repeat initial CCV and re-analyze all samples since last successful CCV.
	Method blank	One per prep batch **(not to exceed more than 10 samples per batch).**	No analytes detected ≥ ½ RL or MDL, whichever is greater[3]	Correct problem then re-prep[4] and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes	One per prep batch **(not to exceed more than 10 samples per batch).**	See Control Limits in LIMS or Clouseau	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Surrogate	Every sample, spiked sample, standard, and method blank	See Control Limits in LIMS or Clouseau	Check system, re-inject, re-extract[4]
	MS	One per batch per matrix, if insufficient sample for MS, then an additional LCS will be analyzed.	See Control Limits in LIMS or Clouseau	All target compounds should be reported, and any compound that is outside criteria must be within criteria in the LCS.
	Confirmation	100% for all positive results (use response of both detectors)	Same as for initial or primary analysis. Comment LIMS if >40% difference in compound response between detectors.	Same as for initial or primary analysis
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific information or more information.
2 - All abnormalities must be noted in a NCM.
3 - Report all target compounds identified in the method blank above the MDL.
4- If unable to re-extract because of insufficient sample volume or the holding time has expired, then a NCM must be generated.

Appendix 4: Summary of Calibration and QC Procedures for Method SW8330

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8330	Five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF of each analyte ≤20% or mean RSD for all analytes ≤20%, with all compounds above 20% commented in LIMS with each sample. linear – $r^2 \geq 0.99$, $r \geq 0.995$	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Retention time verification	Update at start of run or daily	All standards within RT window	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value	Correct problem then repeat initial CCV and re-analyze all samples since last successful CCV.
	Method blank	One per prep batch not to exceed more than 20 samples per batch.	No analytes detected ≥ ½ RL	Correct problem then re-prep[4] and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch (not to exceed more than 20 samples per batch).	See Control Limits in LIMS or Clouseau	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Surrogate	Every sample, spike, standard, and blank	See Control Limits in LIMS or Clouseau	Check system, re-inject, re-extract[4]
	MS/MSD	One per batch per matrix	See Control Limits in LIMS or Clouseau	None (LCS is used to determine if the batch is acceptable).
	Confirmation	100% for all positive results; 2nd column (lunacolumn) confirmation	Same as for initial or primary analysis. Comment LIMS if >40% difference in compound response between detectors.	Same as for initial or primary analysis
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
2 - All abnormalities must be documented in a NCM.
3 - Report all target compounds identified in the method blank above the MDL.
4 - If unable to re-extract sample because of insufficient sample volume or expired holding time, then a NCM must be gnerated.

Appendix 4: Summary of Calibration and QC Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
EPA504.1 SW8011	Five-point initial calibration for all target analytes (calibration standards should be prepped as the samples).	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF of each analyte ≤ 20% **RSD of CF < 10% for Method 8011** Linear – $r^2 \geq 0.99$, $r \geq 0.995$	Correct problem then repeat initial calibration
	Initial calibration verification (ICV) must be from a 2nd source.	Immediately following initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value and within the RTW.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected ≥ RL	Correct problem then re-prep[4] and analyze method blank and all samples processed with the contaminated blank
	LCS	One per prep batch, not to exceed 20 samples in a batch.	See Control Limits Manual	Re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Surrogate	Every sample, spike, standard, and method blank	See Control Limits Manual	Check system, re-inject, re-extract[4]
	MS/MSD	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits Manual	None (LCS is used to determine if data is acceptable).
	Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis. If the relative % difference of results between the 2 columns is greater than 40%, a comment should be placed in LIMS.
	Retention time window calculated for each analyte (see section 9 for how to calculate RTW's).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or technical director.
	MDL check standard	Each week that samples are analyzed.	Detected	Correct problem and re-analyze samples.

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information.
2 - All abnormalities must be documented in a NCM.
3 - Report all target compounds identified in the method blank above the MDL.
4 - If unable to re-extract the samples because of insufficient sample volume or holding time has expired, then a NCM must be generated.

Appendix 4: Summary of Calibration and QC Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8021 SW8015[5]	Five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	RSD of CF ≤ 20% Linear – least squares regression r^2 ≥ 0.99, r ≥ 0.995	Correct problem then repeat initial calibration
	Initial calibration verification (ICV), must be from a 2nd source.	Immediately following five-point initial calibration	All analytes within 15% of expected value	Correct problem then repeat initial calibration
	LCS for all analytes must be from a 2nd source.	One per prep batch, not to exceed 20 samples in a batch.	See Control Limits Manual	Re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 15% of expected value and within the RTW.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.
	Method blank	One per analytical prep batch, not to exceed 20 samples in a batch.	No analytes detected ≥ RL	Correct problem then re-prep[4] and analyze method blank and all samples processed with the contaminated blank
	Surrogate	Every sample, spiked sample, standard, and method blank	See Control Limits Manual	Check system, re-analyze, re-prep[4]
	MS/MSD	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	See Control Limits Manual	None (LCS is used to determine if data is acceptable).
	GC/MS confirmation.	At the clients request or analyst judgment.		
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or technical director.
8021	pH Check	All water samples after analysis.	pH should be less than 2.	If pH is > 2, then place a comment on the benchsheet and in LIMS.
8021	Residual chlorine check (North Carolina samples only)	All water samples after analysis.	Residual chlorine should be negative.	If residual chlorine is positive, document in a NCM.
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 - This is a summary of the acceptance criteria, refer to the method SOP for specific or more information. 2 - All abnormalities must be documented in a NCM.
3 - Report all target compounds identified in the method blank above the MDL.
4 - If unable to re-prep the samples because of insufficient sample volume or holding time has expired, then a NCM must be generated.
5 - For GRO and DRO, see state specific SOP/Method for acceptance criteria. If there is not a specific method for that state, then follow the acceptance criteria in this table.

Appendix 4: Summary of Calibration and QC Procedures for GC Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
EPA601 EPA602	Minimum three-point (preferably five) initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	**RSD of CF < 10%** **RSD of RF < 10%** $r^2 \geq 0.99$, $r \geq 0.995$	Correct problem then repeat initial calibration
	Initial calibration verification (ICV), 20 ug/L, must be from a 2nd source. May be the same as the LCS.	Once immediately following initial calibration	Reference 601/602 table in Section 5 ("Q" in EPA method).	Correct problem then repeat initial calibration
	LCS for all analytes	One per prep batch, **not to exceed 10 samples in a batch.**	See Control Limits Manual	Re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Method blank	One per analytical prep batch, **not to exceed 10 samples in a batch.**	No analytes detected ≥ RL	Correct problem then re-prep[4] and analyze method blank and all samples processed with the contaminated blank
	Surrogate(s)	Every sample, spiked sample, standard, and method blank	See Control Limits in LIMS or Clouseau	Check system, re-analyze, re-prep[4]
	MS	One per batch of 10 per matrix, if insufficient sample for MS, then an additional LCS will be analyzed.	See Control Limits in LIMS or Clouseau	All target compounds should be reported, and any compound that is outside criteria must be within criteria in the LCS.
	GC/MS confirmation.	At clients request or analyst judgment.		
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run (or as needed).	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or technical director.
	pH check	All samples after analysis.	pH should be ≤ 2.	If pH is> 2, then place a comment on the benchsheet, in the PIPE database, and in LIMS.
	Residual chlorine check (North Carolina samples only)	All samples after analysis.	Residual chlorine should be negative.	If residual chlorine is positive, document in a NCM.
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 – This is a summary of the acceptance criteria, refer to the method SOP for specific or more information. 2 – All abnormalities must be noted on the data, the benchsheet, in the PIPE database, and in LIMS.
3 – Report all target compounds identified in the method blank above the MDL.
4 – If unable to re-prep the samples because of insufficient sample volume or holding time has expired, then a NCM must be generated.

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
All	MDL	Sudy performed annually, per instrument. When significant change is made to a method.	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.
All	MDLV	Verify the MDL quarterly or an MDL study must be performed annually	Within 95% confidence level	Re-evaluate MDL standard used and MDL; see Technical Director.
SW8321 Carbamates (DV-LC-0016)	CCV. Continuing calibration verification	After every 10 samples	70 - 130%	Recalibrate the instrument
	HT. Holding Time	All samples	Water: 7 days Soil: 14 days Analysis: 40 days	Contact PM. Client may need to resample or want to run out of hold.
	ICAL. Seven point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration prior to each batch.	Coefficient of calibration $\geq$0.990 linear. >0.990 2nd order. RSD <15%	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria
	ICV. IInitial calibration verification from a 2nd source. If a 2nd source is not available for PFCs, then a 2nd lot number may be used.	Immediately following Initial Calibration Blank (ICB) and immediately following minimum five-point initial calibration	All analytes within 70-130% recovery	Correct problem then repeat initial calibration
	IS. Internal Standard recovery.	All samples and standards	50 - 200%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.
	LCS. Laboratory Control Sample	One per prep batch, not to exceed the 20 samples in a batch.	+/- 3 standard deviations around the mean	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Mass calibration with PEG or other appropriate material bracketing mass calibration range	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu for all compounds	Re-calibrate

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
	MB. Method Blank	Each batch	<1/2 RL	The source of contamination must be be investigated, corrected, and then B-flagged should contamination still be present
	Tune (while infusing). Check mass of spectral ion intensities.	Prior to initial calibration	+/- 0.5 amu for all compounds	Calibrate the mass spec
SW8321 Explosives (DV-LC-0010)	CCV. Continuing calibration verification	50 µg/L and 100 µg/L after every 10 samples	70 - 130%	Recalibrate the instrument
	HT. Holding Time	All samples	Water: 7 days Soil: 14 days Analysis: 40 days	Contact PM. Client may need to resample or want to run out of hold.
	ICAL. Seven point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	Coefficient of calibration $\geq$0.990 linear. >0.990 2nd order. RSD <15%	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria
	ICV. Initial calibration verification from a 2nd source.	Immediately following Initial Calibration Blank (ICB) and immediately following minimum five-point initial calibration	All analytes within 70-130% recovery	Correct problem then repeat initial calibration
	IS. Internal Standard recovery.	All samples and standards	RDX-^{13}C3, 1,3-Dinitrobenzene-d4, and 2,6-Dinitrotoluene-d3 50 - 200%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.
	LCS. Laboratory Control Sample	One per prep batch, not to exceed the 20 samples in a batch.	70 - 130%	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Mass calibration with PEG or other appropriate material bracketing mass calibration range	If large amounts of drift are observed in the tune.	+/- 0.5 amu for all compounds	Re-calibrate

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8321 Explosives (DV-LC-0010)	MB. Method Blank	Each batch	<1/2 RL	The source of contamination must be be investigated, corrected, and then B-flagged should contamination still be present
	Surrogate	All samples and standards	Nitrobenzene-d5: 30 - 120%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.
	Tune (while infusing). Check mass of spectral ion intensities.	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu for all compounds	Calibrate the mass spec
SW8321 Herbicides (DV-LC-014)	CCV. Continuing calibration verification	After every 10 samples	70 - 130%	Recalibrate the instrument
	HT. Holding Time	All samples	**Water: 7 days (direct inject!)** Soil: 14 days Analysis: 40 days	Contact PM. Client may need to resample or want to run out of hold.
	ICAL. Seven point initial calibration for target analyte	Initial calibration prior to sample analysis. Perform instrument re-calibration prior to each batch.	Coefficient of calibration >0.990 2nd order. RSD <20%	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria
	ICV. IInitial calibration verification from a 2nd source.	Immediately following Initial Calibration Blank (ICB) and immediately following minimum five-point initial calibration	All analytes within 75 - 125% recovery	Correct problem then repeat initial calibration
	IS. Internal Standard recovery.	All samples and standards	Typically 50 - 200% AFCEE 70 - 130%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.
	LCS. Laboratory Control Sample	One per prep batch, not to exceed the 20 samples in a batch.	DCAA 25 - 125%	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8321 Herbicides (DV-LC-0014)	Mass calibration with PEG or other appropriate material bracketing mass calibration range	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu for all compounds	Re-calibrate
	MB. Method Blank	Each batch	<1/2 RL	The source of contamination must be be investigated, corrected, and then B-flagged should contamination still be present
	Tune (while infusing). Check mass of spectral ion intensities.	Prior to initial calibration	+/- 0.5 amu for all compounds	Calibrate the mass spec
SW8321 Hexachlorophene (DV-LC-0022	CCV. Continuing calibration verification	After every 10 samples	70 - 130%	Recalibrate the instrument
	HT. Holding Time	All samples	**Water: 7 days (direct inject!)** Soil: 14 days Analysis: 40 days	Contact PM. Client may need to resample or want to run out of hold.
	ICAL. Seven point initial calibration for target analyte	Initial calibration prior to sample analysis. Perform instrument re-calibration prior to each batch.	Coefficient of calibration $\geq$0.990 linear. >0.990 2nd order. RSD <20%	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria
	ICV. IInitial calibration verification from a 2nd source. If a 2nd source is not available for PFCs, then a 2nd lot number may be used.	Immediately following Initial Calibration Blank (ICB) and immediately following minimum five-point initial calibration	All analytes within 70-130% recovery	Correct problem then repeat initial calibration
	IS. Internal Standard recovery.	All samples and standards	50 - 200%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8321 Hexachlorophene (DV-LC-0022)	LCS. Laboratory Control Sample	One per prep batch, not to exceed the 20 samples in a batch.	70 - 130%	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Mass calibration with PEG or other appropriate material bracketing mass calibration range	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu	Re-calibrate
	MB. Method Blank	Each batch	<1/2 RL	The source of contamination must be be investigated, corrected, and then B-flagged should contamination still be present
	Tune (while infusing). Check mass of spectral ion intensities.	Prior to initial calibration	+/- 0.5 amu	Calibrate the mass spec
EPA1625 NDMA (DV-LC-0019)	CCV. Continuing calibration verification	After every 10 samples	75 - 125%	Recalibrate the instrument
	HT. Holding Time	All samples	Water: 7 days Soil: 14 days Analysis: 40 days	Contact PM. Client may need to resample or want to run out of hold.
	ICAL. Seven point initial calibration for target analyte	Initial calibration prior to sample analysis. Perform instrument re-calibration prior to each batch.	Coefficient of calibration $\geq$0.990 linear. >0.990 2nd order. RSD <20%	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria
	ICV. IInitial calibration verification from a 2nd source.	Immediately following Initial Calibration Blank (ICB) and immediately following minimum five-point initial calibration	80 - 120%	Correct problem then repeat initial calibration
	IS. Internal Standard recovery.	All samples and standards	18 - 142%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
EPA1625 NDMA (DV-LC-0019)	LCS. Laboratory Control Sample	One per prep batch, not to exceed the 20 samples in a batch.	Per SOP: 70 - 130% Per QuantIMS: 68 - 124%	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Mass calibration with PEG or other appropriate material bracketing mass calibration range	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu	Re-calibrate
	MB. Method Blank	Each batch	<1/2 RL	The source of contamination must be be investigated, corrected, and then B-flagged should contamination still be present
	Surrogate	All samples and standards	Per SOP: 20 - 150% Per QuantIMS: 18 - 142%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.
	Tune (while infusing). Check mass of spectral ion intensities.	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu	Calibrate the mass spec
SW6860 Perchlorates (DV-LC-0024)	CCV. Continuing calibration verification	2 (mid range and low range) per 10 samples.	Mid range 85 - 115% recovery Low range	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check
	HT. Holding Time	All samples	Water: 28 days Soil: 28 days Analysis: 28 days	Contact PM. Client may need to resample or want to run out of hold.
	ICAL. Seven point initial calibration for target analyte	As needed, after maintenance or major changes such as IC column type.	Coefficient of calibration $\geq$0.995 linear. >0.990 2nd order. RSD <15%	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW6860 Perchlorates (DV-LC-0024)	ICS. Interference Check Sample	Each batch	May vary per client. See control limits in LIMS, DoD handbook, or SOP. 70 - 130% typical	Check the calibration standards and instrument conditions (may need to replace column). Repeat ICAL.
	ICV. IInitial calibration verification from a 2nd source.	Immediately following Initial Calibration Blank (ICB) and immediately following minimum five-point initial calibration	85 -115% recovery of perchlorate	Correct problem then repeat initial calibration
	IS. Internal Standard recovery.	All samples and standards	50 - 150%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.
	Isotope Ratio. 35CI/37CI (If tandem MS, this monitors both the parent ion at masses 99/101 and the daughter ion at masses 83/85)	All samples, spiked samples, standards and method blanks	2.3 - 3.8	Re-extract using cleanup procedures. If still fails, use post-spiking procedure or dilution. If dilution, results are J-flagged for DoD samples.
	LCS. Laboratory Control Sample (Perch: conc level=RLV)	One per prep batch, not to exceed the 20 samples in a batch.	May vary per client. See Control Limits in LIMS or Clouseau. (DoD is 85 - 115%)	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	LODV. Limit of Detection Verification	Before and after every batch	70 - 130% recovery	Check instrument settings and calibration. Rerun the LODV and all samples since the last successful LODV. Q-flag if still unacceptable.
	LRB. Laboratory Reagent Blank	Immediately prior to initial calibration	<1/2 RL	Repeat until no carryover

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW6860 Perchlorates (DV-LC-0024)	Mass calibration with PEG or other appropriate material bracketing mass calibration range	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu	Re-calibrate
	MB. Method Blank	Each batch	<1/2 RL	The source of contamination must be be investigated, corrected, and then B-flagged should contamination still be present
	MS/MSD. Matrix Spike and Matrix Spike Duplicate samples	One per batch per matrix, if insufficient sample for MS/MSD, then a LCS/LCSD will be analyzed.	May vary per client and matrix. See Control Limits in LIMS or Clouseau	None (the LCS is used to evaluate to determine if the batch is acceptable).
	RLV. Reporting Limit Verification	Same concentration as the LCS, each initial calibration	70 - 130% recovery	Recalibrate and rerun all associated samples
	Tune (while infusing). Check mass of spectral ion intensities.	Prior to initial calibration	+/- 0.5 amu	Calibrate the mass spec
SW8321 PFCs (DV-LC-0012)	ICB. Initial Calibration Blank	Immediately following initial calibration	<Method Detection Limit and <0.1 µg/L on column	Identify source of contamination, clean, and repeat initial calibration
	CCV. Continuing calibration verification	After every 10 samples	80 - 120%	Recalibrate the instrument
	ICAL. Seven point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	Coefficient of calibration ≥0.990 linear. >0.990 2nd order. RSD <15%	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria
	ICV. IInitial calibration verification from a 2nd source. If a 2nd source is not available for PFCs, then a 2nd lot number may be used.	Immediately following Initial Calibration Blank (ICB) and immediately following minimum five-point initial calibration	All analytes within 70-130% recovery	Correct problem then repeat initial calibration

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8321 PFCs (DV-LC-0012)	IS. Internal Standard recovery.	All samples and standards	50 - 200%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.
	LCS. Laboratory Control Sample	One per prep batch, not to exceed the 20 samples in a batch.	+/- 3 standard deviations around the mean	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Mass calibration with PEG or other appropriate material bracketing mass calibration range	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu for all compounds	Re-calibrate
	MB. Method Blank	Each batch	<1/2 RL	The source of contamination must be be investigated, corrected, and then B-flagged should contamination still be present
	Tune (while infusing). Check mass of spectral ion intensities.	Prior to initial calibration	+/- 0.5 amu for all compounds	Calibrate the mass spec
SW8321 PFOA and PFOS (DV-LC-0012)	Surrogate(s)	Every sample, spike, standard, and blank	See Control Limits in LIMS or Clouseau	Check system, re-analyze, re-prep[5]
	CCV. Continuing calibration verification	After every 10 injections	80 - 120%	Recalibrate the instrument
	Closing CCV. Closing calibration curve verification	Close of each analytical session	0.5 ug/L: 70 - 130%	Instrument must be recalibrated and samples reanalyzed
	HT. Holding Time	All samples	Water: 7 days Soil: 14 days Analysis: 40 days	Contact PM. Client may need to resample or want to run out of hold.
	ICAL. Seven point initial calibration for target analyte	Initial calibration prior to sample analysis. Perform instrument re-calibration prior to each batch.	Coefficient of calibration $\geq$0.990 linear. >0.990 2nd order. RSD <15%	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8321 PFOA and PFOS (DV-LC-0012)	ICV. IInitial calibration verification from a 2nd source.	Immediately following Initial Calibration Blank (ICB) and immediately following minimum five-point initial calibration	80 - 120%	Correct problem then repeat initial calibration
	IS. Internal Standard recovery	All samples and standards	50 - 200%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.
	Isotope Ratio.	All samples, spiked samples, standards and method blanks	80 - 120%	Re-extract using cleanup procedures. If still fails, use post-spiking procedure or dilution. If dilution, results are J-flagged for DoD samples.
	LCS. Laboratory Control Sample	One low LCS and one mid LCS per batch	+/- 3 standard deviations around the mean	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Mass calibration with PEG or other appropriate material bracketing mass calibration range	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu for all compounds	Re-calibrate
	MB. Method Blank	Each batch	<1/2 RL	The source of contamination must be be investigated, corrected, and then B-flagged should contamination still be present
	RLV. Reporting Limit Verification	Same concentration as the LCS, each initial calibration	70 - 130% recovery	Recalibrate and rerun all associated samples
	Tune (while infusing). Check mass of spectral ion intensities.	Prior to initial calibration	+/- 0.5 amu for all compounds	Calibrate the mass spec
SW8321 Picric Acid (DV-LC-0025)	CCV. Continuing calibration verification	After every 10 samples	70 - 130%	Recalibrate the instrument

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8321 Picric Acid (DV-LC-0025)	HT. Holding Time	All samples	Water: 7 days Soil: 14 days Analysis: 40 days	Contact PM. Client may need to resample or want to run out of hold.
	ICAL. Seven point initial calibration for target analyte	Initial calibration prior to sample analysis. Perform instrument re-calibration prior to each batch.	Coefficient of calibration ≥0.990 linear. >0.990 2nd order. RSD <15%	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria
	ICV. IInitial calibration verification from a 2nd source.	Immediately following Initial Calibration Blank (ICB) and immediately following minimum five-point initial calibration	70 - 130%	Correct problem then repeat initial calibration
	IS. Internal Standard recovery.	All samples and standards	50 - 200%	Re-extract if no matrix effect is obvious. Dilute if matrix effect is obvious.
	LCS. Laboratory Control Sample	One per prep batch, not to exceed the 20 samples in a batch.	70 - 130%	Correct problem then re-prep[4] and analyze the LCS and all samples in the affected analytical batch
	Mass calibration with PEG or other appropriate material bracketing mass calibration range	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu	Re-calibrate
	MB. Method Blank	Each batch	<1/2 RL	The source of contamination must be be investigated, corrected, and then B-flagged should contamination still be present
	Tune (while infusing). Check mass of spectral ion intensities.	Prior to initial calibration	+/- 0.5 amu	Calibrate the mass spec
SW8321 Warfarin	CCV. Continuing calibration verification	After every 10 samples	80 - 120%	Recalibrate the instrument

Appendix 4: Summary of Calibration and QC Procedures for LCMS Organics

Method	QC Check	Frequency	Acceptance Criteria[1]	Corrective Action[2]
SW8321 Warfarin	ICAL. Seven point initial calibration for target analyte	Initial calibration prior to sample analysis. Perform instrument re-calibration prior to each batch.	Coefficient of calibration ≥0.990 linear. >0.990 2nd order. RSD <15%	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria
	ICV. IInitial calibration verification from a 2nd source. If a 2nd source is not available for PFCs, then a 2nd lot number may be used.	Immediately following Initial Calibration Blank (ICB) and immediately following minimum five-point initial calibration	All analytes within 70-130% recovery	Correct problem then repeat initial calibration
	Mass calibration with PEG or other appropriate material bracketing mass calibration range	As needed (failed tune criteria), after major maintenance, minimum of annually.	+/- 0.5 amu for all compounds	Re-calibrate
	MB. Method Blank	Each batch	<1/2 RL	The source of contamination must be be investigated, corrected, and then B-flagged should contamination still be present
	Tune (while infusing). Check mass of spectral ion intensities.	Prior to initial calibration	+/- 0.5 amu for all compounds	Calibrate the mass spec

1- This is a summary of the acceptance criteria, refer to the method SOP for specific information or more information.

2 - All abnormalities must be documented in a NCM.
3 - Report all target compounds identified in the method blank above the MDL.
4- If unable to re-extract because of insufficient sample volume or the holding time has expired, then a NCM must be generated.

Appendix 4: Summary of Calibration and QC Procedures for Method SW6010

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6010	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank (CB)	After every continuing calibration verification	Must be <3 times the IDL or the average of 3 CB must be <3 times the IDL.	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value and RSD of replicate integrations <5%	Repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICS)	At the beginning of an analytical run	Within 20% of expected value	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	LCS	One per prep batch	See Control Limits in LIMS or Clouseau	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	MS/MSD	One per batch per matrix	See Control Limits in LIMS or Clouseau	None
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 25% of expected results	Correct problem then re-analyze post digestion spike addition

1 – Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration and QC Procedures for Method SW6020

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW6020	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A
	Second-source calibration verification (ICV)	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Calibration blank (CB)	After every continuing calibration verification	Must be <RL	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10% of expected value	Repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected ≥ RL. Some programs require <1/2 RL.	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICS)	At the beginning of an analytical run	Within 20% of expected value	Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	LCS	One per prep batch	See Historical Limits in LIMS	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	MS/MSD	One per batch per matrix	See Historical Limits in LIMS	None
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.
	Dilution test	One per batch	1:5 dilution must agree within 10% of the original determination	N/A
	Post digestion spike addition	One per batch	Recovery within 25% of expected results	N/A

1 – Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration and QC Procedures for Method SW7196

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW7196	Initial calibration (minimum three standards and a blank)	Initial calibration prior to sample analysis.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Immediately following initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Beginning and after every 10 samples and at the end of the analysis sequence	All analytes within 20% of expected value	Correct problem then repeat initial calibration and re-analyze all samples since last successful calibration
	Verification check to ensure lack of reducing condition and/or interference	Once for every sample matrix analyzed	Spike recovery between 85-115%	If check indicates interference, dilute and re-analyze sample persistent interference indicates the need to use and alternate method
	Method blank	One per prep batch	No analytes detected $\geq$ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	MS/MSD	One per 20 samples per matrix	See Control Limits in LIMS or Clouseau	none
	LCS	One per batch	See Control Limits in LIMS or Clouseau	Re-prep, re-analyze all affected samples.
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration and QC Procedures for Method SW7470/SW7471

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW7470 SW7471	Initial calibration (minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration. If calibration fails again, re-digest the entire digestion batch.
	Second-source calibration verification (ICV)	Immediately following initial daily calibration	Analytes within 10% of expected value	Correct problem then repeat initial calibration. If calibration fails again, re-digest the entire digestion batch.
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ MDL	Correct problem then re-digest and re-analyze calibration and entire digestion batch
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	Analytes within 20% of expected value	Correct problem then repeat all QC and samples since last successful calibration. If the CCV fails again upon reanalysis, reprep the entire digestion batch.
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank, all samples, and QC processed with the contaminated blank
	LCS	One per prep batch	See Control Limits in LIMS or Clouseau	Correct problem then re-prep and analyze the LCS, all samples, and QC in the affected analytical batch
	Dilution test; five-fold dilution test	Each preparatory batch	Five times dilution sample result must be ±10% of the undiluted sample result	Perform post digestion spike addition
	Recovery test	When dilution test fails	Recovery within 85-115% of expected results	Dilute the sample; re-analyze post digestion spike addition
	MS/MSD	One per batch per matrix	See Control Limits in LIMS or Clouseau	None
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 - Report all targets identified in the method blank above the MDL**.**

Appendix 4: Summary of Calibration and QC Procedures for Method SW9010/SW9012/SW9014

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW9010 SW9012 SW9014	Initial calibration (six standards and a calibration blank)	Initial daily calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration
	Distilled standards (one high and one low)	Once per calibration	Analytes within 10% of true value	Correct problem then repeat distilled standards
	Second-source calibration verification (ICV)	Immediately following initial daily calibration	Analytes within 15% of expected value	Correct problem then repeat initial calibration
	Continuing calibration verification (CCV)	Beginning and after every 10 samples and at the end of the analysis sequence	Analytes within 15% of expected value	Correct problem then repeat initial Continuing calibration verification and re-analyze all samples since last successful Continuing calibration verification
	Method blank	One per prep batch	No analytes detected $\geq$ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	LCS	One per batch per matrix	See Control Limits in LIMS or Clouseau	Re-prep, re-run affected samples
	MS/MSD	One per batch per matrix	See Control Limits in LIMS or Clouseau	None
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

Appendix 4: Summary of Calibration and QC Procedures for Mercury

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA245.1	Initial calibration (minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	$r^2 \geq 0.99$, $r \geq 0.995$ for linear regression	Correct problem then repeat initial calibration
	Second-source calibration verification (ICV)	Immediately following five-point initial calibration	Analyte within 5% of expected value	Correct problem then repeat initial calibration
	Calibration blank	Once per initial daily calibration	No analytes detected ≥ MDL	Correct problem then re-analyze calibration blank and all samples associated with blank
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	Analyte within 10% of true value	Correct problem then repeat calibration and re-analyze all samples and QC since last successful calibration
	LCS	One per prep batch	All analytes within 15% of expected value	Correct problem then re-prep and analyze the LCS, all samples, and QC in the affected analytical batch
	Matrix Spike/Matrix Spike Duplicate	One per batch or 10 samples	All analytes within 30% of expected value	None
	Method Blank	One per batch	No analytes > RL	Reprep
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration and QC Procedures for ICP Metals

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA200.7	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A
	Second-source calibration verification (ICV)	Each calibration	Value of all analytes within 5% of expected value	Correct problem then repeat initial calibration
	Linear Dynamic Range	Once annually	All analytes within 10% of expected value	Calibration range lowered to meet LDR results
	Calibration blank	After every Continuing calibration verification	No analytes detected ≥ MDL	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10%	Repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICS)	At the beginning of an analytical run, daily		Terminate analysis; correct problem; re-analyze ICS; re-analyze all affected samples
	LCS	One per prep batch	All analytes within 15% of expected value	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Dilution test	Each new sample matrix	1:5 dilution must agree within 10% of the original determination	Perform post digestion spike addition
	Post digestion spike addition	When dilution test fails	Recovery within 25% of expected results	Correct problem then re-analyze post digestion spike addition
	Matrix Spike/Matrix Spike Duplicate	One per batch of 20 samples	All analytes within 30% of expected value	None
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 - Report all targets identified in the method blank above the MDL**.**

Appendix 4: Summary of Calibration and QC Procedures for ICPMS Metals

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA200.8	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis.	N/A	N/A
	Second-source calibration verification (ICV)	Each calibration	Value of all analytes within 10% of expected value	Correct problem then repeat initial calibration
	Linear Dynamic Range	Quarterly	All analytes within 10% of expected value. LDR set at 90% for samples	Calibration range lowered to meet LDR results
	Calibration blank	After every Continuing calibration verification	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All analytes within 10%	Repeat calibration and re-analyze all samples since last successful calibration
	Method blank	One per prep batch	No analytes detected ≥ RL	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank
	Interference check solution (ICS)	N/A	N/A	N/A
	LCS	One per prep batch	All analytes within 15% of expected value. See historical limits in LIMS	Correct problem then re-prep and analyze the LCS and all samples in the affected analytical batch
	Dilution test	N/A	N/A	NA
	Post digestion spike addition	N/A	N/A	NA
	Matrix Spike/Matrix Spike Duplicate	One per batch of 20 samples	All analytes within 30% of expected value. See historical limits in LIMS	None
	MDL verification	Quarterly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

1 - Report all targets identified in the method blank above the MDL.

Appendix 4: Summary of Calibration and QC Procedures for Gravimetric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SM2540 C (TDS) SM2540 D (TSS) SM2540 B (TS) EPA160.4, SM2540E* (TVS)* ASTM D5057* (Density/ Specific Gravity)*	Verification standard– single standard (if available)	Each batch	±10%	Repeat
	Method blank	Each batch	No analytes detected ≥ RL	Repeat, rerun
	Duplicate	Each batch, less than 20	±20%	None
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

***Analysis is performed at TestAmerica Denver but does not have any check standard available.**

Appendix 4: Summary of Calibration and QC Procedures for Titrimetric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SM2310B: Acidity. Alkalinity. SM2320: HCO3-, CO3-2. SM4500-CO2 C: CO2. SM4500SO3: Sulfite 4500S^{2}F, 9030\9034: Sulfide SM4500CL C: Chloride 2340B or C: Hardness	Verification standard– single standard (if available)	Each batch	±10%	Repeat, check
	Method blank	Each batch	No analyte detected ≥ report limit	Repeat batch
	Duplicate	Each batch	±20%	None
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

Appendix 4: Summary of Calibration and QC Procedures for Spectrophotometric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA350.1: NH_3. EPA410.4: COD. SW7196, SM3500Cr: Cr+6 EPA335.4, 9010, 9012, SM4500CN : CN. SM4500S^{-2} D: Sulfide SM5310B,9060: TOC. SM4500NO_2B: Nitrite SM3500Fe D: Ferrous Iron SM4500CL E: Chloride EPA420.1, 420.4: Phenol EPA351.2: TKN EPA353.2: Nitrate, NO2+NO3 EPA365.1: Total Phos, O-Phos ASTMD516-02: Turb. Sulfate EPA180.1: Turbidity.	Calibration curve – minimum 5 point	Initial. Perform re-calibration once per year minimum.	RSD <10%, $r^2 \geq 0.99$, $r \geq 0.995$	Recalibrate
	Independent calibration verification – mid-level, second-source required (ICV)	Immediately following initial calibration.	±10%	Recalibrate
	Continuing calibration verification (CCV)	Beginning, every 10 samples, and at end of sequence	±10%	Correct, recalibrate
	Method blank	Each use	No analyte detected ≥ report limit	Reprep, rerun
	MS/MSD	Each batch, less than 20	±20% Or: historical or client specified where applicable	None
	LCS	Each batch	± 10% Or: historical or client specified where applicable	Rerun
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

Appendix 4: Summary of Calibration and QC Procedures for Electrometric Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SM5210B: BOD[1], CBOD[1]. SM2510B, SW9050: Cond. SW9023: EOX. SM4500F-C: Flouride. SM4500H$^+$B, SW9040/9045: pH. SM5310B, SW9020,9076: TOX. EPA365.3: ORP[1]	Calibration Curve – minimum of 5 standards	Initial Calibration. Perform re-calibration once per year minimum	±10%, $r^2 \geq 0.99$, $r \geq 0.995$.	Recalibrate
	Independent calibration verification (second source) (ICV)	Immediately after initial calibration	±10%	Recalibrate
	Continuing calibration verification (CCV)	Beginning, every 10 samples, and end of batch	±10%	Rerun
	Method blank NA for pH	Each batch	No analyte detected ≥ report limit	Reprep
	LCS	Each batch	±10% Or: historical or client specified where applicable	Rerun batch
	MS/MSD	Each batch	± 20% Or: historical or client specified where applicable	None
	Duplicate	When spike not available	±20%	None
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

[1]Calibration curve does not apply.

Appendix 4: Summary of Calibration and QC Procedures for Ion Chromatographic Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA300 & SW9056: Bromide Chloride Chlorate Fluoride Nitrate Nitrite Sulfate.	Calibration Curve – Minimum 5-point calibration	Initial calibration. Perform instrument re-calibration once per year minimum.	RSD ± 10%, $r^2 \geq 0.99$, $r \geq 0.995$.	Recalibrate
	Calibration verification (ICV), second source	Immediately following initial calibration	±10%	Recalibrate
	Continuing calibration verification (CCV)	Each use, beginning, every 10 samples, end of batch	± 10%	Rerun affected samples
	Method blank	Each batch	No analyte detected ≥ report limit	Rerun batch
	LCS	Each batch	±10% Or: historical or client specified where applicable	Rerun batch
	MS/MSD[1]	Each batch	±20% Or: historical or client specified where applicable	None, use LCS
	Duplicate	Each batch	±30%	None
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

[1]Only applies to EPA300, SW9056.

Appendix 4: Summary of Calibration and QC Procedures for Oil & Grease Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA1664 SW9070. SW9071.	Verification standard (NA for 1664)	Single standard	±10% PAR standard	Rerun
	Method blank	Each batch	No analyte detected ≥ report limit	Repeat batch
	LCS	Each batch	See Control Limits Manual	Repeat batch
	MS/MSD	Each batch	See Control Limits Manual	None, use LCS
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

Appendix 4: Summary of Calibration and QC Procedures for Physical Analyses

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
SW1010: Flash Point. SM2120B*: Color* SW9095*: Paint Filter*. SM2540F*: Settleable Solids*. .	Method blank	Each batch	No analyte detected ≥ report limit	Repeat, rerun
	Two standards for Flash Point 1 Known for Settleable Solids Method-specific standards for Color.	Each batch	Flashpoint LCS ± 2º F	Rerun batch
	Duplicate	Each batch	±20%	None
	MDL verification (NA for flashpoint and paint filter)	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

***Analysis is performed at TestAmerica Denver but does not have any check standard available.**

Appendix 4: Summary of Calibration and QC Procedures for Perchlorate

Method	QC Check	Frequency	Acceptance Criteria	Corrective Action
EPA314.1: Perchlorate	Calibration Curve – Minimum 5-point calibration	Initial calibration. Perform instrument re-calibration once per year minimum.	r ≥ 0.995.	Recalibrate
	Calibration verification (ICV), second source	Immediately following initial calibration	±10%	Recalibrate
	Initial Performance Check (IPC)	Each batch	±20%	Recalibrate
	Initial Calibration Check Standard (ICCS)	Each batch	±25%	Recalibrate
	Initial Calibration Blank (ICB)	After initial calibration	No analyte detected ≥ report limit	
	Continuing calibration verification (CCV)	Each use, beginning, every 10 samples, end of batch	± 10%	Rerun affected samples
	Method blank	Each batch	No analyte detected ≥ report limit	Rerun batch
	LCS	Each batch	±15%	Rerun batch
	MS/MSD[1]	Each batch	±20% RPD 15%	Document in NCM
	MDL verification	Minimum yearly	Detectible	Re-evaluate MDL standard used and MDL; see Technical Director.

Appendix 5. Glossary/Acronyms

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. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority:
The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

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)

Aliquot, aliquant:
A measured portion of a sample taken for analysis.

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. (NELAC)

arithmetic mean

The arithmetic mean ($\bar{x}$) is the average of a set of values. It is equal to the sum of the observed values divided by the number of observations. Also called "average".

$$\overline{X} = \frac{\sum_{i=1}^{n} x_i}{n}$$

where: $\overline{X}$ = the mean
x_i = the i^{th} data value
n = number of data values

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 NELAC). (NELAC)

Assessment Criteria:
The measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)

Assessment Team:
The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)

Assessor:
One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC)

Audit:
A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch:
Environmental samples which 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 to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Benchmarking:
A step-by-step method of improving performance by identifying and studying best practices and comparing them to industry practices.

Bias:
A systematic (consistent) error in test results. Bias is expressed as the difference between the population mean and the true or reference value, or as estimated from sample statistics, the difference between the sample average and the reference value.

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)

Blind Sample:
A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration:
To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve:
The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

calibration factor (CF):
The ratio of the instrument response of an analyte to the amount injected. CFs are used in external standard calibrations.

$$CF = \frac{Total\ Area\ of\ Peak}{Mass\ Injected}$$

Calibration Method:
A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:
A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):
A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain of Custody:
An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:
The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

coefficient of variation (relative standard deviation)
A measure of precision (relative dispersion). It is equal to the standard deviation (*s*) divided by the mean ($\overline{X}$) and multiplied by 100 to give a percentage value.

$$\boldsymbol{CV\ (RSD)} = \left(\frac{s}{\overline{X}} \right) \times 100$$

collocated samples:
Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. The results will indicate sampling as well as analytical variability.

Comparability:
Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):
The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

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. (NELAC)

completeness
Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed. It is usually expressed as a percentage:

$$\%Completeness = \frac{V}{n} \times 100$$

where: V = number of measurements judged valid
n = total number of measurements

composite

A sample composed of two or more increments.

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. NELAC 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
(NELAC)

Conformance:
An affirmative indication or judgement 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)

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)

correlation coefficient
The correlation coefficient (r) is a determination of how closely data "fits" a straight line. It is a number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers. A correlation coefficient of +1 (usually calculated to three decimal places or 1.000) means the data falls exactly on a straight line with positive slope. A correlation coefficient of -1 (or -1.000) means the data falls exactly on a straight line with negative slope.

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). (NELAC)

Data Reduction:
The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

data quality objective (DQO)
Data quality objectives (DQOs) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application (EPA 1994). Typically, DQOs are identified during project scope and development of sampling and analysis plans. In this QA manual, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory. EPA has established six primary analytical DQOs for environmental studies: precision, accuracy, representativeness, completeness, comparability, and detectability.

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

degrees of freedom
The number of independent deviations used in calculating an estimate of the standard deviation.

Detection Limit:
The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

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)

Environmental Detection Limit (EDL):
The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

Equipment Blank:
Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

error
The difference between an observed or measured value and its true value.

External Standard Calibration:
Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA):
The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)

Federal Water Pollution Control Act (Clean Water Act, CWA):
The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

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 Testing:
NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Finding:
An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (NELAC)

Holding Times (Maximum Allowable Holding Times):
The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Inspection:
An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Internal Standard:
A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

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 Response:
Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

Laboratory:
A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

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. 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), there is no LCS. 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.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:
Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

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 of Detection (LOD):
An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Manager (however named):
The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

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 matrix or Saline/Estuarine source. 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: 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. (NELAC)

Matrix Spike (spiked sample or fortified sample):

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 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 selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):

A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

measurement
The process or operation of ascertaining the extent, degree, quantity, dimensions, or capability with respect to a standard.

median
The middle value of a set of data when the data set is ranked in increasing or decreasing order.

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. (NELAC)

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)

National Environmental Laboratory Accreditation Conference (NELAC):
A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP):
The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

NELAC Standards:
The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)

outlier

A result excluded from the statistical calculations due to being deemed "suspicious" when applying the "Grubbs Test" (or equivalent).

parameter
In statistical analysis, a constant or coefficient that describes some characteristic of a population (e.g., standard deviation, mean, regression coefficients). In analytical chemistry, a chemical or physical attribute of a sample that is being measured, i.e., an analyte (e.g., chemical concentration, temperature, pH, etc.).

percent difference
The difference between two values, expressed as a percent of the first value.

$$\%D = \frac{X_1 - X_2}{X_1} \times 100\%$$

where: %D = percent difference
X_1 = first value
X_2 = second value

percent recovery
A measure of accuracy determined from the comparison of a reported spike value to its true spike concentration.

$$\%R = \frac{\textbf{\textit{observed conc.} – \textit{sample conc.}}}{\textbf{\textit{true spike conc.}}} \times 100\%$$

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. (NELAC)

Performance Based Measurement System (PBMS):
A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

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. (NELAC)

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. (NELAC)

Preservation:
Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

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. (NELAC) [2.1]

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. (NELAC)

Proficiency Test Sample (PT):
A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Quality Assurance:
An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

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 which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:
An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. 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. (EPA-QAD)

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. (NELAC)

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 (ANSI/ASQC-E-41994)

Quantitation Limits:
The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range:
The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Reagent Blank (method reagent blank):
A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method:
A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Relative percent different (RPD)
Statistic for evaluating the precision of a replicate set. For replicate results:

$$RPD = \left[\frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2} \right)} \right] \times 100$$

where: X_1 = first observed concentration
X_2 = second observed concentration

Relative response factor (RRF)
A measure of the relative mass spectral response of a compound compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. Because a RRF is the comparison of two responses, it is a unitless number. RRFs are determined by the following equation:

$$RRF = \frac{A_x}{A_{IS}} \times \frac{C_{IS}}{C_x}$$

where: A = area of the characteristic ion measured
C = concentration

IS = internal standard
x = analyte of interest

Replicate Analyses:
The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Reporting limit (RL)
One of two types of reporting limit conventions within STL Denver. The Reporting Limit (RL) is a uniform, STL-wide reporting limit based on an evaluation of the PQLs at STL laboratories and the expected method performance in routine water and soil matrices. Project Specific Reporting Limits (PSRLs) are reporting limits that are defined by project requirements.

Representativeness
Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. At each STL laboratory, every effort must be made to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before subsampling.

reproducibility
The precision, usually expressed as a standard deviation, measuring the variability among results of measurements of the same sample at different laboratories.

Requirement:
Denotes a mandatory specification; often designated by the term “shall”. (NELAC)

Resource Conservation and Recovery Act (RCRA):
The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the “cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal. (NELAC)

Safe Drinking Water Act (SDWA):
The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Duplicate:
Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic): The 2nd 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 2nd 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:
(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity:
The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

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.

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. 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, a representative number (at a minimum 10%) of the listed components may 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.. (NELAC)

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard addition
The procedure of adding known increments of the analyte of interest to a sample to cause increases in detection response to subsequently establish, by extrapolation of the plotted responses, the level of the analyte of interest present in the original sample.

Standard deviation
A measure of the dispersion about the mean of the elements in a population. The square root of the variance of a set of values:

$$s = \sqrt{\frac{\sum_{i=1}^{n} \left(x_i - \overline{X}\right)^2}{n-1}}$$

where: s = standard deviation
= sum of

X = observed values
n = number of observations

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

Standardized Reference Material (SRM):
A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named):
The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

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 Director:
Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test:
A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method:
An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Toxic Substances Control Act (TSCA):

The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:
The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

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.

United States Environmental Protection Agency (EPA):
The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation:
The process of substantiating specified performance criteria. (EPA-QAD)

Verification:
Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell:
A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Acronyms:

A2LA – American Association for Laboratory Accreditation
ASTM – American Society for Testing and Materials
BOD – Biological Oxygen Demand
BS – Blank Spike
BSD – Blank Spike Duplicate
CAR – Corrective Action Report
CCC – Calibration Check Compound
CCV – Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
COD – Chemical Oxygen Demand
CRS – Change Request Form
CUR – Condition Upon Receipt
DFTPP – Decafluorotriphenylphosphine
DOC – Demonstration of Capability
DOE – Department of Energy
DOT – Department of Transportation
DoD – Department of Defense
DQO – Data Quality Objectives
DU – Duplicate
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HDPE – High Density Polyethylene
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICS – Interference Check Sample
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
ISO – International Organization for Standardization
LCL – Lower Control Limit
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
MDL – Method Detection Limit
MS – Matrix Spike
MSA – Method of Standard Additions
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NELAC - National Environmental Laboratory Accreditation Conference
NELAP - National Environmental Laboratory Accreditation Program
NCM – Non-conformance Memo
NIST – National Institute of Standards Technology
NPDES – National Pollutant Discharge Elimination System

Acronyms con't:

PAH – Polyanuclear Aromatic Hydrocarbon
PCB – Polychlorinated biphenyl
PDS – Post Digestion Spike
PM – Project Manager
PQL – Practical Quantitation Limit
PSRL – Project Specific Reporting Limit
PT – Performance Testing
QAM – Quality Assurance Manual
QAPP – Quality Assurance Project Plan
QAS – Quality Assurance Summary
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RCRA – Resource Conservation and Recovery Act
RF – Response Factor
RFP – Request for Proposal
RL – Reporting Limit
RPD – Relative Percent Difference
RRF – Relative Response Factor
RSD – Relative Standard Deviation
RSO – Radiation Safety Officer
SD – Standard Deviation
SDG – Sample Delivery Group
SOP - Standard Operating Procedure
SOW – Statement of Work
SPCC – System Performance Check Compound
SPLP – Synthetic Precipitation Leaching Procedure
SRM – Standard Reference Material
TCLP – Toxicity Characteristic Leaching Procedure
TIC – Tentatively Identified Compound
TAT – Turn-Around-Time
TKN – Total Kjeldahl Nitrogen
TOC – Total Organic Carbon
TOX – Total Organic Halides
UCL – Upper Control Limit
UPS – Uninterruptible Power Supply
USEPA – United States Environmental Protection Agency
VOA – Volatiles
VOC – Volatile Organic Compound
WS – Water Supply
WP – Water Pollution

Appendix 6.

Laboratory Certifications, Accreditations, Validations

TestAmerica Denver maintains certifications, accreditations, certifications, and validations 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:

Organization	Certificate Number	Organization	Certificate Number
AFCEE	None	Nevada	CO0026
Alabama	40730	New Jersey	CO004
Alaska	UST-30	New Mexico	None
Arizona	AZ0713	North Carolina	358
Arkansas	88-0687	North Dakota	R-034
California	2513	Oklahoma	8614
Colorado	CO0026	Oregon	CO200001
Connecticut	PH-0686	Pennsylvania	68-00664
Florida	E87667	RAM License	Colorado 486-03
Georgia – DW	962	South Carolina	72002001
Georgia – NP & Soils	None	Tennessee	TN02944
Idaho	CO00026	USACE	Self Declared
Illinois	007726	USDA	S-60617
Iowa	370	Texas	T104704183
Kansas	E-10166	Utah	Quans5
Louisiana	02096	Washington	C1284
Maine	CO0002	Wisconsin	999615430
Maryland	268	West Virginia	354
Minnesota	11175AA		

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.

Claims of Accreditation Status

TestAmerica Denver has agreed to make only valid claims as to its accreditation/certification status by any authority by ensuring that the expiration dates are not exceeded and the method-specific scope or parameter lists are supportable, as required by each. Any false claims would be reported to that authority. The agreement covers the use of the authority's name, such as "Authority-Accredited," logo, or certificate number. The only valid proof of accreditation/certification is the current certificate and scope of the authority. It is the responsibility of the laboratory to make these documents available to all staff, and it is the staff's duty to reference only the current documents.

A report with scope and non-scope analytes may only be presented on the same report if the non-accredited results are clearly and unambiguously identified. No report with non-scope analytes may be associated with the logo, “Authority accredited” phrase, or the certificate number. Only the analytes specified by a unique method are valid within the scope. There shall be no intentional misleading of the users of the laboratory’s services in this regard.

No opinions and/or interpretations based on results outside the laboratory’s scope may be presented on a document referenced by “Authority-accredited, the logo, or the certificate number. If these are made, they must be written in a separate letter which is not endorsed by the authority.

The “Authority-accredited” logo may only be affixed to equipment calibrated by a laboratory that is accredited by the authority. If calibration labels contain the logo, they must also show the calibration laboratory’s name or its certificate number, the instrument’s unique identification, the date of the last calibration, and a cross-reference to the last calibration certificate.

Should the company decide to use the “Authority-accredited” logo in marketing activities, no misrepresentation may occur. Only reference to the accredited scope at a specific laboratory site is allowed. If any “Authority-accredited” language is used in proposals or quotations, any non-scope analytes must be clearly denoted as not accredited by that authority. The same is true for any use of laboratory letterhead with the “Authority-accredited” wording or logo. The logo may not be affixed to any material, item, product, part, or packaging, thereby implying accreditation status to that piece. In literature, any use of the logo must be positioned adjacent to the accredited laboratory’s name and clearly state that the presence of the logo does not imply certification/approval of the products tested. At no time may the logo appear to suggest that a person is accredited. Misrepresentation of accreditation status is never allowed and must be reported if it occurs. If in doubt, the idea of the logo’s use may be presented to the authority for approval.

If accreditation is terminated or suspended, the laboratory will immediately cease to use the “Authority-accredited” wording, the logo, or the certificate number reference in any way and inform clients impacted by the change.

Appendix 7. Data Qualifiers - Standard

Qualifier	Definition
*	Surrogate or Relative Percent Difference (RPD) is outside control limits.
A	Spiked analyte recovery is outside control limits.
B	Organics: Method blank contamination. The associated method blank contains the target analyte at a reportable level. Inorganics: Estimated result. Result is less than the RL
COL	More than 40% difference between the primary and confirmation detector results. The lower of the two results is reported.
DIL	The concentration is estimated or not reported due to dilution.
E	Estimated result. Result concentration exceeds the calibration range.
G	Inorganics: Elevated reporting limit. The reporting limit is elevated due to matrix interference.
J	Organics: Estimated result. Result is less than RL Inorganics: Method blank contamination. The associated method blank contains the target analyte at a reportable level.
L	Serial dilution of a digestate in the analytical batch indicates that physical and chemical interferences are present
N	Spiked analyte recovery is outside stated control limits.
NC	The recovery and/or RPD were not calculated.
ND	The analyte was not detected at the MDL concentration and with a measurable degree of confidence can be said not to be present at or above the RL concentration.
P	Relative percent difference (RPD) is outside stated control limits.
Q	Elevated reporting limit. The reporting limit is elevated due to high analyte levels.
V	General Chemistry: Elevated reporting limit due to limited sample volume.
Wa	Post digestion spike recovery fell between 40-85% due to matrix interference.
Wb	Post digestion spike recovery fell between 115-150% due to matrix interference.
I	Percent recovery is estimated since the results exceeded the calibration range.
T1	A tentatively identified compound that did not generate a spectral match of 80% or greater. Typically called “unknown”
T2	A tentatively identified compound with a spectral match of 80% or better
T3	A tentatively identified compound that was calibrated for by the lab, but not on the client target analyte list.
IC	Diluted due to high inorganic chloride.

This is not an exhaustive list of qualifiers. All qualifiers are defined on each data sheet. Client specific qualifiers may also be used, and would also be defined on the data sheet.

Appendix 7 con't. Data Qualifiers – AFCEE 4.0

Qualifier	Definition
J	The analyte was positively identified; the quantitation is estimated due to discrepancies in meeting certain analyte-specific quality control criteria.
U	The analyte was analyzed for, but not detected. The associated numerical value is at or below the MDL.
F	The analyte was positively identified but the associated numerical value is above the MDL and below the RL.
R	The data are rejected due to deficiencies in the ability to analyze the sample and meet QC criteria.
Q	One or more quality control criteria (for example, LCS recovery, surrogate spike recovery, etc.) failed.
B	The analyte was found in an associated blank, as well as in the sample.
M	A matrix effect was present.
NC, MSB	The recovery and RPD were not calculated because the sample amount was greater than four times the spike amount.
NC, DIL	The recovery was not calculated because the sample was diluted four times or greater.
N	Inorganics: Spiked analyte recovery is outside stated control limits.
A	Organics: Spiked analyte recovery is outside stated control limits.
*	Surrogate or LCS is outside control limits.
UJ	The analyte was not detected; however, the result is estimated due to discrepancies in meeting certain analyte specific quality control criteria.

Appendix 7 con't. Data Qualifiers – DoD QSM Version 3 and 4.1

Qualifier	Definition
U	Undetected at the limit of detection. The associated data value is the limit of detection, adjusted by any dilution factor used in the analysis.
J	Estimated: The analyte was positively identified; the quantitation is estimated (for example, matrix interference, outside the calibration range).
B	Blank contamination: The analyte was detected in the associated method blank at a concentration greater than one-half the reporting limit.
B	Metals Forms 3, 5B and 9 (ICB, CCB, Post-Digestion Spike and Serial Dilution): Analyte was detected above the method detection limit but below the reporting limit.
Q	One or more quality control criteria (for example, LCS recovery, surrogate recovery) failed. Data usability should be carefully assessed by the project team.
A	Spiked analyte recovery is outside control limit.
MSB	The recovery and RPD were not calculated because the sample amount was greater than four times the spike amount.
NC DIL	The recovery and RPD were not calculated due to dilution.
N	Inorganics: Spiked analyte recovery is outside stated control limits.
A	Organics: Spiked analyte recovery is outside stated control limits.
*	Surrogate or LCS is outside control limits.