FINAL
CSSA BASE-WIDE QUALITY ASSURANCE PROJECT PLAN

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LIST OF ACRONYMS AND ABBREVIATIONS

AA	atomic absorption
AFCEE	Air Force Center for Environmental Excellence
AOC	area of concern
ASCII	American Standard Code Information Interchange
ASTM	American Society for Testing and Materials
BFB	bromofluorobenzene
BOD	biological oxygen demand
Br^-	bromide
BTEX	benzene, toluene, ethylbenzene, xylene
C_6	n-hexane
C_6-C_{28}	total petroleum hydrocarbons
C_{35}	n-pentatriacontane
CAS	corrective action system
CCB	continuing calibration blank
CCC	calibration check compound
CCR	Current Conditions Report
CCV	continuing calibration verification
CFR	Code of Federal Register
Cl^-	chloride
CL	control limit
CLP	Contract Laboratory Program
CMI	corrective measure implementation
CMS	corrective measure study
COC	chain-of-custody
COD	coefficient of determination
CSSA	Camp Stanley Storage Activity
CV	calibration verification
DCA	dichloroethane
DCB	dichlorobenzene
DCE	dichloroethylene
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethene
DDT	dichlorodiphenyltrichloroethane
DFTPP	decafluorotriphenylphosphine
DNB	dinitrobenzene
DNT	dinitrotoluene
DOT	Department of Transportation
DQI	data quality indicator
DQO	data quality objective
DRO	diesel range organics

LIST OF ACRONYMS AND ABBREVIATIONS (Continued)

e.g.	for example
EICP	extracted ion current profile
EPA	Environmental Protection Agency
ERPIMS	Environmental Restoration Program Information Management System
F^-	fluoride
FID	flame ionization detector
FSP	field sampling plan
G	glass
GALP	good automated laboratory practices
GC	gas chromatography
GC/MS	gas chromatography/mass spectroscopy
GFAA	graphite furnace atomic absorption
GIS	geographic information system
GRO	gasoline range organics
HAA	haloacetic acid
Handbook	*Handbook for the Installation Restoration Program (IRP) Remedial Investigation and Feasibility Studies (RI/FS)*, September 1993
HCl	hydrochloric acid
HQ	Headquarters
HMX	octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
HNO_3	nitric acid
HPLC	high-performance liquid chromatography
H_2SO_4	sulfuric acid
ICAL	initial calibration
ICP	inductively coupled plasma
ICP-AES	inductively coupled plasma-atomic emission spectroscopy
ICP-MS	inductively coupled plasma-mass spectroscopy
ICS	interference check solution
ICV	initial calibration verification
ID	identification
IDW	investigation derived waste
IM	interim/stabilization measures
IPC	instrument performance check
IRP	Installation Restoration Program
IS	internal standard
LCL	lower control limit
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LIMS	laboratory information management system

LIST OF ACRONYMS AND ABBREVIATIONS (Continued)

LRB	laboratory reagent blank
LTC	Lieutenant Colonel
MCL	maximum contaminant level
MD	matrix duplicate
MDL	method detection limit
MS	matrix spike
MSD	matrix spike duplicate
N/A	not applicable
$Na_2S_2O_3$	sodium thiosulfate
NaOH	sodium hydroxide
NEPA	National Environmental Policy Act
NIST	National Institute of Standards and Technology
NO_2^-	nitrite
NO_3^-	nitrate
NTU	nephelometric turbidity unit
ORP	oxidation-reduction potential
OSHA	Occupational Safety and Health Administration
OVA	organic vapor analyzer
P	polyethylene
PAH	polynuclear aromatic hydrocarbon
PARCC	precision, accuracy, representativeness, completeness, and comparability
PCB	polychlorinated biphenyl
PCE	perchloroethylene or tetrachloroethylene
PE	performance evaluation
PID	photoionization detector
PO_4^{-3}	phosphate
PWS	Public Water System
QA	quality assurance
QAO	Quality Assurance Officer
QAPP	quality assurance project plan
QPR	Quarterly Progress Report
QC	quality control
r	correlation coefficient
R	recovery
RCRA	Resource Conservation and Recovery Act
RDX	hexahydro-1,3,5-trinitro-1,3,5-triazine
RF	response factor
RFI	RCRA Facility Investigation

LIST OF ACRONYMS AND ABBREVIATIONS (Continued)

RI/FS	remedial investigation/feasibility study
RL	Reporting Limit
RMU	rifle management unit
RPD	relative percent difference
RRAD	Red River Army Depot
RRS	Risk Reduction Standards
RRS1	Risk Reduction Standard 1
RRS2	Risk Reduction Standard 2
RSD	relative standard deviation
RRT	relative retention time
RT	retention time
RTW	retention time window
S	soil
SAMHD	San Antonio Metropolitan Health District
SAP	sampling and analysis plan
SD	standard deviation
SO_4^{-2}	sulfate
SOP	standard operating procedure
SOW	statement of work
SPCC	system performance check compound
SQL	sample quantitation limit
SVOC	semivolatile organic compound
SWMU	solid waste management unit
T	California brass
TAC	Texas Administrative Code
TCA	trichloroethane
TCE	trichloroethylene
TCLP	toxicity characteristic leaching procedure
TCMX	tetrachlorometaxylene
THM	trihalomethanes
TIC	tentatively identified compound
TNB	trinitrobenzene
TNT	trinitrotoluene
TNRCC	Texas Natural Resource Conservation Commission
TPDES	Texas Pollutant Discharge Elimination System
TPH	total petroleum hydrocarbon
TRRP	Texas Risk Reduction Program
TSS	total suspended solids
UCL	upper control limit
USACE	United States Army Corps of Engineers
UXO	unexploded ordnance

LIST OF ACRONYMS AND ABBREVIATIONS (Continued)

VOA	volatile organic analysis
VOC	volatile organic compound
W	water
WP	water pollution
WS	water supply
X	observed concentration
XRF	x-ray fluorescence

UNITS OF MEASURE

°C	degree Celsius
%D	percent difference
%R	percent recovery
µg/kg	micrograms per kilogram, (ppb)
µg/L	micrograms per liter, (ppb)
µg/mL	micrograms per milliliter
µL	microliter
g	gram
mg/kg	milligrams per kilogram, (ppm)
mg/L	milligrams per liter, (ppm)
mL	milliliter
mm	millimeter
nm	nanometer
pCi/L	picocuries per liter
pH	degree of alkalinity or acidity
ppb	parts per billion
ppm	parts per million
ppmv	parts per million volume

1.0 INTRODUCTION

The United States Department of the Army is committed to environmental stewardship in all actions as an integral part of the Army mission. Army Regulations 200-1, issued by Headquarters (HQ), Department of the Army, Washington, D.C. on February 21, 1997 provides a brief overview of environmental programs and requirements. It does not provide a complete listing of requirements or detailed guidance on complying with environmental laws and regulations. In addressing environmental issues, readers must consult the applicable laws, regulations, and guidance documents referenced in that regulation. The regulation supplements federal, state, and local environmental laws for preserving, protecting, and restoring the quality of the environment. It also integrates pollution prevention, natural and cultural resources, and the National Environmental Policy Act (NEPA) into the Army Environmental Program.

Camp Stanley Storage Activity (CSSA), located in Boerne, Texas is one of the facilities of the Department of the Army and thereby will carry out the mission of the Army as it applies to the facility.

The United States Environmental Protection Agency (EPA), Region 6, and CSSA entered into a Resource Conservation and Recovery Act (RCRA) Section 3008(h) Administrative Order on consent on May 5, 1999. This Order requires the CSSA to: (1) perform interim/stabilization measures (IM) at the facility to prevent or minimize the further migration of contaminants due to releases of hazardous constituents to the environment, or to mitigate current or potential threats to human health or the environment; (2) perform a RCRA Facility Investigation (RFI) to determine the nature and extent of any release(s) of hazardous waste or hazardous constituents at or from the facility; (3) perform a Corrective Measure Study (CMS) to identify and evaluate alternatives for corrective action(s) to prevent or mitigate any migration of release(s) of hazardous wastes or hazardous constituents at or from the facility, and to collect any other information necessary to support the selection of corrective measures at the facility; and (4) implement the corrective measures [Corrective Measure Implementation (CMI)] selected by the EPA for the facility.

Section IX of the above referenced Administrative Order requires that CSSA follow approved Quality Assurance (QA) and Quality Control (QC) procedures for all sampling and analytical activities. This post-wide Camp Stanley Storage Activity Quality Assurance Project Plan (CSSA QAPP) presents in specific terms the policies, organization, functions, and QA/QC requirements for environmental programs at CSSA. This detailed CSSA QAPP: (1) has been prepared for use by contractors who perform environmental services to ensure the data are scientifically valid and defensible; (2) establishes the analytical protocols and documentation requirements to ensure the samples are collected and analyzed, and the data are reviewed and validated in a specified manner; and (3) provides guidance for using the Data Quality Objective (DQO) process for specific investigations. This CSSA QAPP and a delivery order specific Field Sampling Plan (FSP) shall constitute, by definition, the CSSA Sampling and Analysis Plan (SAP). The SAP will define data quality for a specific project.

The following documents are used as guidance in the preparation of CSSA QAPP:

- EPA Requirements for Quality Assurance Project Plans, EPA/R-5, EPA/240/B-01/003, March 2001.
- EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, EPA/600/R-98/018, February 1998.
- Data Quality Objectives Process for Superfund, Interim Final Guidance, EPA/540/G-93/071, September 1993.
- Guidance for the data quality objective process, EPA QA/G-4, EPA/600R-96/055, August 2000.
- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (U.S. EPA SW846, Third Edition and its first, second, and third update).
- The Air Force Center for Environmental Excellence Quality Assurance Project Plan, version 3.0, March 1998.
- HQ Air Force Center for Environmental Excellence Technical Services Quality Assurance Program, version 1.0, August 1996.
- AFCEE Model Field Sampling Plan, version 1.1, July 1997.
- Title 30, Texas Administrative Code (TAC), Chapter 335, Subchapter S.
- Texas Risk Reduction Program (TRRP), 30 TAC, Chapter 350.
- Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater, EPA/600R-98/128.
- Department of Defense Quality Systems Manual for Environmental Laboratories, Draft version 2, May 2001.
- Environmental Encyclopedia, CSSA, Boerne, Texas.

Other literature references are provided throughout the document as appropriate.

Distribution of the CSSA QAPP shall be controlled by the Environmental Officer of CSSA to ensure that the current version is being used. A sequential numbering system shall be used to identify controlled copies of the QAPP. Controlled copies shall be provided to applicable regulatory agencies, remedial project managers, contractors, and QA coordinators. Whenever revisions are made or material added to the QAPP, a document control system shall be utilized to assure: (1) all parties holding a controlled copy of the QAPP shall receive the revisions/addenda; and (2) outdated material is removed from circulation. The document control system does not preclude making and using copies of the QAPP; however, the holders of controlled copies are responsible for distributing additional material to update any copies within their organizations.

1.1 Contractor Responsibilities

The CSSA QAPP shall be in the possession of the prime contractor's project managers, field teams and laboratory directors, and is a required reading for all staff participating in the CSSA work efforts. All contractors and subcontractors shall be required to comply with the procedures specified in this QAPP in order to maintain validity, comparability, and representativeness of the data produced.

In addition, the prime contractor shall comply with the requirements specified in the HQ Air Force Center for Environmental Excellence, Technical Services Quality Assurance Program, version 1.0, August 1996.
Prior to the beginning of each project, the prime contractor shall follow the DQO process with project team members. The DQOs shall be included in the scoping documents prior to the start of the field operations. When DQOs are refined during project operations, appropriate documentation should be included as addendum to scoping documents.

1.2 Service Center Responsibilities

The Air Force Center for Environmental Excellence (AFCEE) has been selected by CSSA to assist the installation to meet various requirements of its environmental program. The authority to approve quality assurance procedures and variances rests with the Environmental Officer of CSSA, and is not delegated to AFCEE or its representatives. However, CSSA will request technical expertise as needed from AFCEE and its contractors. All deviations from the QAPP shall be documented to CSSA by AFCEE and its contractors and approved or denied based on project DQOs.

1.3 Teaming/Partnership

CSSA, EPA, Texas Natural Resource Conservation Commission (TNRCC), AFCEE and all the contractors shall maintain an open line of communication at all times. As technical issues arise, CSSA shall be informed and they will ensure appropriate personnel on the team are consulted so rational and appropriate decisions can be made.

Subcontractor analytical laboratories must contact the project manager of the prime contractor as soon as technical problems arise and could not be solved at the laboratory level. The prime contractor should work with the laboratory to resolve technical problems related to CSSA data quality and should include AFCEE and CSSA personnel in reaching a resolution to the issues.

2.0 PROJECT MANAGEMENT

2.1 Project Organization

The Installation Commander has delegated the authority to carry out all environmental programs at CSSA to the Environmental Officer. The Environmental Officer works closely with all the regulatory agency managers, and is responsible for all the environmental investigations and cleanup of targeted sites at the installation. Fieldwork is contracted to prime contractors through service centers, like AFCEE and the U.S. Army Corps of Engineers (USACE). Contracting officers and the contracting officer's representatives can direct the prime contractors to perform various tasks per the contract documentation.

The current personnel involved in the environmental programs are:

Post Commander at CSSA is LTC. Jason D. Shirley
Environmental Officer at CSSA is Mr. Brian K. Murphy, CSP
U.S. EPA, Region VI, Remediation Project Manager is Mr. Greg J. Lyssy
U.S. EPA, Region VI, Risk Assessor is Ms. Maria Martinez
TNRCC, Industrial and Hazardous Waste Manager is Mr. Kirk Coulter
TNRCC, Quality Assurance Officer is Ms. Ann Strahl
A Contractor is Parsons Engineering Science, Inc., Austin, TX
Project Manager for Parsons is Ms. Karuna Mirchandani
A Contractor is Foster Wheeler, San Antonio, TX
Program Manager for Foster Wheeler is Mr. Sina Seyedian
Project Manager for Foster Wheeler is Mr. Frank Frey
AFCEE Team Chief is Ms. Teresa DuPriest

The above list of personnel may change in the future and other environmental contractors may be added. All changes and updates will be included in the next revision of the QAPP.

2.1.1 Selection of Subcontractor Analytical Laboratories

The prime contractor for CSSA has the responsibility of selecting one or more analytical subcontractor laboratories to carry out analysis of various sample matrices. In selecting a laboratory, the prime contractor must inform the laboratories of the data quality objectives of the various programs at CSSA and provide the laboratories with the current version of the CSSA QAPP that specifies all the analytical and other technical requirements for the installation. During the selection process, the prime contractor shall review all variance requests and select the laboratories whose variance has the least impact on the DQOs for CSSA.

The prime contractor shall conduct performance and systems audits of the selected laboratory before sending field samples for analysis. CSSA and AFCEE representatives may be present to oversee the prime contractor's systems audit. In addition, all variance requests for the selected laboratory must be pre-approved by AFCEE and/or the appropriate regulatory agencies. The

CSSA environmental officer is a member of the team designated to oversee audits and approve variances.

2.2 Project Background

The preliminary background information can be obtained from Background Information Report, March 1998, in the **Environmental Encyclopedia, Scoping Documents, Volume 1-1: Work Plan.** The Background Study is currently being re-done due to quality control/quality assurance problems with the original analytical data and is in draft form. The revised background study has yet to be approved by regulatory agencies. The above referenced report includes the installation information, history, soils (Ss) geology, groundwater and surface water (W) hydrology and quality, metrology, climate, air quality, biological resources, and summary of previous investigations. The report also includes location, soils classification, geology, and cross-section maps.

2.3 Problem Statement

The Current Conditions Report (CCR), July 1999 in the **Environmental Encyclopedia, Scoping Documents, Volume 1-1: Work Plan** discusses historical analytical data, the status of solid waste management units (SWMUs), areas of concern (AOC), rifle management units (RMUs), and underground storage tanks at CSSA. In addition, there are several Quarterly Progress Reports (QPRs) in **the Environmental Encyclopedia, Scoping Documents, Volume 1-1: Work Plan** that explain various tasks for Interim Measures, RCRA Facility Investigation, Corrective Measures Study, and Corrective Measures Implementation.

The users of the CSSA QAPP are advised to review the latest CCR and QPR before proceeding on investigation or remediation of specific projects, particularly for those sites where work has been performed previously.

2.4 Project Schedule

The RCRA Section 3008(h) Administrative Order describes the work to be performed for: (1) Interim/Stabilization Measures; (2) RCRA Facility Investigation; (3) Corrective Measures Study (CMS); and (4) Corrective Measures Implementation. Each of the above programs has a built in schedule for regulatory approval of Work Plans, and submission of progress reports. A copy of the above referenced Administrative Order is in **Environmental Encyclopedia, Scoping Documents, and Volume 1-1: Work Plan**.

2.5 CSSA Project Quality Objectives

At CSSA, there are a number of different environmental programs that are in progress. The project quality objectives for each one of these programs are different and the objectives may change as each project evolves. Section 2.6 provides general principles of DQOs and how the process should be applied to various programs. Section 2.7 discusses data quality indicators (DQIs) required by regulatory agencies.

2.5.1 Site Closure Program

At CSSA there are SWMUs, AOCs, and RMUs that require investigation and are intended to be closed. A current list of these sites has been submitted to TNRCC (Attachment 2, Letter to TNRCC from Parsons Engineering Science, Inc., July 12, 1999). All closures will be based on a risk reduction approach, as explained in the *Technical Approach Document For Risk Evaluation CSSA, August 2000*. This document is part of the **Environmental Encyclopedia, Volume 1-6.**

An initial evaluation will be conducted to determine if site concentrations of contaminants exceed background concentrations. If site concentrations do not exceed background or reporting limits (RLs) for specific analytes, site closure will be carried out under Risk Reduction Standard 1 (RRS1). If site concentrations exceed background and if the site could be remediated to background, site closure will also be carried out under RRS1. If a site fails to meet the RRSI (i.e., background level) closure standards, CSSA has the option to seek closure under either the Risk Reduction Standards [RRSs (old rules)] or through the Texas Risk Reduction Program (new rules).

The Current Conditions Report 1999, lists copper; lead; zinc; mercury; 2,4-dinitrotoluene (DNT); 2,6-DNT; octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX); and phthalates concentrations at several SWMUs that exceed the TNRCC Risk Reduction Standards 1; and cadmium, lead, copper, perchloroethylene or tetrachloroethylene (PCE), polynuclear aromatic hydrocarbon (PAHs), and 2,4-DNT that exceed TNRCC Risk Reduction Standards 2. Not all sites have been characterized, and future sampling is planned at many SWMUs.

Soil gas surveys conducted at several AOCs and SWMUs showed the presence of PCE, trichloroethylene (TCE), and dichloroethylene (DCE). Additional soil gas surveys may be required at other sites. Future investigations may involve collection and analysis of surface and subsurface soil samples, groundwater and surface water samples, and swipe samples. RMU-1 is currently active and no investigation is planned. Unexploded Ordnance (UXO) will be cleared from RMUs 2, 3, 4, and 5, if necessary, and soil samples will be investigated. UXO clearance will be evaluated on a site-by-site basis. If AOC and RMU sites are found to meet RRS1 criteria after investigation has been completed, closure reports will be prepared according to TNRCC requirements.

The sampling and analyses requirements for various CSSA site closure activities are found in the Environmental Encyclopedia. The Work Plan for SWMU, AOC, and RMU closures at CSSA and all addenda to this Work Plan are in the **Environmental Encyclopedia, Volume 1-1**. The SAP addenda corresponding to the Work Plan addenda are in the **Environmental Encyclopedia, Volume 1-4**.

When groundwater samples are collected from monitoring wells, a complete list of analytes by method SW8260 for volatile organic compounds (VOCs) will be required for the first time a well is sampled. Based on the results of the first round, a reduced analyte list may be approved for subsequent rounds of sampling.

2.5.2 Permit Requirements

CSSA has been granted a Texas Pollutant Discharge Elimination System (TPDES) permit, permit # 03849, for the operation of the wastewater treatment plant and for the treatment of groundwater. The required analytical data for outfall 001 were submitted to TNRCC on December 17, 1999. Analytical requirements are listed in Tables B-1, B-2, B-5, and B-7 of the permit application. Results for all the analytes in the above mentioned tables are required only at the time of permit renewal.

Analytical requirements for Outfall 002 are listed in Attachments A, C and D of the TPDES permit application. Results for all analytes in the above mentioned Attachments are required only at the time of permit application.

The following Tables 2.5.2-1 and 2.5.2-2 list the effluent limitations and monitoring requirements for Outfalls 001 and 002 to comply with the CSSA TPDES permit. All the tests listed in Table 2.5.2-1, except fecal coliform by Method 9221 E, are performed in the laboratory located at Camp Stanley. The results are reported on a monthly effluent report form to TNRCC. A state certified laboratory, San Antonio Metropolitan Health District (SAMHD) Laboratory, San Antonio, Texas, performs the fecal coliform test.

TNRCC has also authorized CSSA to discharge groundwater to Outfall 002 in permit No. 03849. Currently, Outfall 002 is used periodically to treat water generated during installation/development of wells. When Outfall 002 began operation, the flow rate was monitored and samples collected per permit requirements. In addition, Table 2.5.2-2 provides the monitoring requirements for Outfall 002. Compliance testing for TCE and PCE by method SW8260 is required during operation of Outfall 002 and will be performed at an off-post laboratory.

Table 2.5.2-1
TPDES Permit Requirements for Outfall 001

Effluent Characteristic	Analytical Method	Frequency of Testing	Type of Sample
Biological Oxygen Demand (BOD)	Standard Method 5210B	Once per week	Grab
Total Suspended Solids (TSS)	E 160.2	Once per week	Grab
pH	E 150.1	Once per month	Grab
Chlorine, Residual	E 330.5	Five times a week	Grab
Fecal Coliform	Standard Methods 9221 E	Once per month	Grab
Fecal Coliform Screen	Standard Methods 9221 D	Once per week	Grab
Copper, Total (needed only after copper sulfate is used)	Hach 8506	Once per day	Grab

Table 2.5.2-2
TPDES Permit Requirements for Outfall 002

Effluent Characteristic	Analytical Method	Frequency of Testing	Type of Sample
Tetrachloroethylene	SW8260	Twice per week when discharge occurs	Grab
Trichloroethylene	SW8260	Twice per week when discharge occurs	Grab
pH	E 150.1	Once a day	Grab

2.5.3 Drinking Water Permit Requirements

CSSA has several drinking water wells, which it operates and is permitted by TNRCC as a public water system (PWS). The PWS identification (ID) number is 0150117. The drinking water from the wells is used only for the employees of CSSA. As a public water system, CSSA is regulated by safe drinking water regulations and is subject to sanitary surveys by TNRCC.

Primary drinking water regulations require that CSSA conduct periodic monitoring of drinking water for the contaminants listed in Table 2.5.3. The information presented in Table 2.5.3 is obtained from *Drinking Water Standards Governing Drinking Water Quality and Reporting Requirements for Public Water Systems, 30 TAC Chapter 290 Subchapter F, TNRCC, RG-346 September 2000.* Table 2.5.3 also lists the Maximum Contaminant Levels (MCLs), where available, and the frequency of monitoring.

Table 2.5.3
Monitoring Requirements for Drinking Water Contaminants

Analyte	MCL (mg/L)	Frequency
Fecal Coliform	(+) coliform	1/mo
Residual Chlorine	0.2 – 0.5	1/wk
Asbestos	7 million fibers per liter (longer than 10 μm)	1/3yrs 1/9yrs
Inorganic Compounds TAC 290.106		
Antimony Arsenic Barium Beryllium Cadmium Chromium Mercury (inorganic) Selenium Thallium	0.006 0.05 2.0 0.004 0.005 0.1 0.002 0.05 0.002	1/3yrs
NO_3^- [Nitrate (as N)]	10.0	1/1yr
NO_2^- [Nitrite (as N)] Cyanide (free) Fluoride (F^-)	1.0 0.2 4.0	1/3yrs
Lead	0.015	Initial- 2/1yr Secondary-1/3yrs
Synthetic Organic Compounds TAC 290.107(a)		
Alachlor Atrazine Benzo(a)pyrene Carbofuran Chlordane Dalapon Dibromochloropropane Di (2-ethylhexyl) adipate Di (2-ethylhexyl) phthalate	0.002 0.003 0.0002 0.04 0.002 0.2 0.0002 0.4 0.006	4/3yrs (compliance period) 1/3yrs (after compliance period)

Table 2.5.3 (Continued)
Drinking Water Contaminants

Analyte	MCL (mg/L)	Frequency
Synthetic Organic Contaminants (continued)		
Dinoseb Diquat Endothall Endrin Ethylene Dibromide Glyphosate Heptachlor Heptachlor Epoxide Hexachlorobenzene Hexachlorocyclopentadiene Lindane Methoxychlor Oxamyl (Vydate) Pentachlorophenol Picloram PCB Simazine Toxaphene 2,3,7,8-TCCD (dioxin) 2,4,5-TP 2,4-D	0.007 0.02 0.1 0.002 0.00005 0.7 0.0004 0.0002 0.001 0.05 0.0002 0.04 0.2 0.001 0.5 0.0005 0.004 0.003 3X10-8 0.05 0.07	4/3yrs (comp. Period) 1/3yrs (after comp. period)
Volatile Organic Compounds (reduced analyte requirements for CSSA)		
Trichloroethylene Tetrachloroethylene Cis-1,2-Dichloroethylene	0.005 0.005 0.07	4/3yrs (compliance. period) 1/3yrs (after initial period)

Table 2.5.3 (Continued)
Drinking Water Contaminants

Analyte	MCL (mg/L)	Frequency
Disinfection By-products TAC 290.113		
Trihalomethanes (THM) Bromoform Bromodichloromethane Chloroform Dibromochloromethane Haloacetic Acid (HAA) Monochloroacetic acid Trichloroacetic acid Monobromoacetic acid Dibromoacetic acid	Total THM: 0.08 Total HAA: 0.06	1/yr*
Radiological Requirements TAC 290.108		
Radium-226 and Radium-228 Gross Alpha Particle Beta Particle Photon Radioactivity	5 pCi/L 15 pCi/L > 4 millirem/yr > 4 millirem/yr	1/4yrs*
Secondary Constituent Levels TAC 290.118		
Constituent	**Secondary Level**	**Frequency**
Aluminum Chloride (Cl^-) Color Copper Corrosivity Fluoride Foaming Agents Hydrogen Sulfide Iron Manganese Odor pH Silver Sulfate (SO_4^{-2}) Total Dissolved Solids (TDS) Zinc	0.05 – 0.2 mg/L 300.0 mg/L 15 color units 1.0 mg/L non-corrosive 2.0 mg/L 0.5 mg/L 0.05 mg/L 0.3 mg/L 0.05 mg/L 3 threshold odor no. > 7.0 0.1 mg/L 300.0 mg/L 1,000 mg/L 5.0 mg/L	1/3 yr at the point of entry to the distribution system

Table 2.5.3 (Concluded)
Drinking Water Contaminants

Analyte	Action Levels	Frequency
Water Quality Parameters TAC 290.117(f) and 40 CFR 141.82		
pH Alkalinity Calcium Conductivity Temperature	TNRCC recommended values to evaluate effectiveness of corrosion control will be used.	Quarterly required if lead or copper exceeds MCL. At point of entry to the distribution system.

*CSSA staff collects drinking water samples and the samples are sent to the Texas State laboratory for analysis.

mo — Month.
wk — Week.
yrs — Years.
mg/L — milligrams per liter.
pCi/L — picocuries per liter.

2.5.4 Off-Post Well Investigations

The objective of the off-post well investigation is to determine if contamination from identified CSSA source areas has migrated and is impacting groundwater well users. Preliminary investigations have indicated the need to monitor certain wells for specific volatile organic compounds. These compounds are trichloroethylene, tetrachloroethylene, cis-1,2-dichloroethene, and vinyl chloride. Method SW8260 will be used for investigating the presence of the above analytes in off-post well water samples. The reporting limits and the quality control criteria for those analytes in Method SW8260, presented in Section 4.0, are applicable.

If additional investigations are required in the future, the choice of the analytical methods, the list of analytes, and the level of quality control will be directed by the DQOs.

2.6 Data Quality Objectives Process

Data Quality Objectives' process is a systematic process for generating environmental data that will be sufficient for their intended use. This is a seven-step process: (1) State the problem; (2) Identify the decision; (3) Identify the inputs to the decision; (4) Define the boundaries of the study; (5) Develop a decision rule; (6) Specify limits on decision errors; and (7) Optimize the design. The DQO process is iterative, i.e., the seven-step process should be repeated, as needed, based on newly acquired data and/or information. The DQO process should be applied to each program and to each site prior to sampling and analytical activities.

The DQO process is more substantially expanded below with additional details:

Step 1: State the Problem

Purpose: Summarize the contamination problem that will require new environmental data, and identify the resources available to resolve the problem.

Activities:

1. Identify members of the scoping team.
2. Develop/refine the conceptual site model.
3. Define the exposure pathways and exposure scenarios.
4. Specify available resources.
5. Write a brief summary of the contamination problem.

Step 2: Identify the Decision

Purpose: Identify the decision that requires new environmental data to address the contamination problem.

Activities:

1. Identify the key decision for the current phase or stage of the project.
2. Identify alternative actions that may be taken based on the findings of the field investigation.
3. Identify relationships between this decision and any other current or subsequent decisions.

Step 3: Identify the Inputs to the Decision

Purpose: Identify the information needed to support the decision, and specify which inputs require new environmental measurements.

Activities:

1. Identify the information inputs needed to resolve the decision.
2. Identify sources for each information input, and list those inputs that are obtained through environmental measurements.
3. Define the basis for establishing contaminant-specific action levels.
4. Identify potential sampling approaches and appropriate analytical methods.

Step 4: Define the Boundaries of the Study

Purpose: Specify the spatial and temporal aspects of the environmental media that the data must represent to support the decision.

Activities:

1. Define the geographic areas of the field investigation.
2. Define each environmental medium of concern.
3. Divide each medium into strata having relatively homogeneous characteristics.
4. Define the scale of decision-making.
5. Determine the time frame to which the decision applies.
6. Determine when to take samples.
7. Identify practical constraints that may hinder sample collection (reconsider previous steps as necessary).

Step 5: Develop a Decision Rule

Purpose: Develop a logical *if…then…* statement that defines the conditions that would cause the decision-maker to choose among alternative actions.

Activities:

1. Specify the parameter of interest (such as mean, median, maximum, or proportion).
2. Specify the action level for the decision.
3. Combine the outputs of the previous DQO steps into an *if...then...* decision rule that includes the parameter of interest, the action levels, and the alternative actions.

Step 6: Specify Limits on Decision Errors

Purpose: Specify the decision-maker's acceptable limits on decision errors, which are used to establish appropriate performance goals for limiting uncertainty in the data.

Activities:

1. Determine the possible range of the parameter of interest.
2. Define both types of decision errors and identify the potential consequences of each.
3. Specify a range of possible parameter values where the consequences of decision errors are relatively minor (gray region).
4. Assign probability values to points above and below the action level that reflect the acceptable probability for the occurrence of decision errors.
5. Check the limits on decision errors to ensure that they accurately reflect the decision-maker's concern about the relative consequences for each type of decision error.

Step 7: Optimize the Design

Purpose: Identify the most resource-effective sampling and analysis design for generating data that are expected to satisfy the DQOs.

Activities:

1. Review the DQO outputs and existing environmental data.
2. Develop general sampling and analysis design alternatives.
3. For each design alternative, verify that the DQOs are satisfied.
4. Select the most resource-effective design that satisfies all of the DQOs.
5. Document the operational details and theoretical assumptions of the selected design in the SAP

2.7 Data Quality Indicators

The Data Quality Indicators (DQIs) include five parameters: precision, accuracy, representativeness, completeness, and comparability (PARCC). Each parameter, as it applies to CSSA, is explained in subsection below. Formulae for calculating various DQIs are provided in Table 2.7 at the end of this section. For Camp Stanley, the DQIs required will be based on the DQOs and the cost effectiveness for specific projects.

2.7.1 Precision

Precision is the degree of mutual agreement characteristic of independent measurements as a result of repeated application of the process under specified conditions (*Ref. Quality Assurance of Chemical Measurements, Taylor, John K., Lewis Publishers, Inc., Chelsea, Michigan, 48118, 1987*). Camp Stanley environmental programs require analytical and total precision as described below:

Analytical precision is the measurement of the variability associated with multiple determination of the same sample in the laboratory under similar conditions. Analytical precision could be measured either long-term (multiple batches) or for a single analytical batch.

Long-term analytical precision for an analyte in a method can be calculated from multiple determinations of the analyte from laboratory control samples (LCSs) over a period of time. There are several formulas used in the calculation of long-term precision. For Camp Stanley, recoveries of LCS values obtained over a period of time should be used to construct a control chart, and to evaluate long-term analytical precision. Camp Stanley auditors will review long-term precision at the time of review of the laboratory quality assurance documents, at the time of a laboratory audit, or as required.

Control limits (CLs) for each method and analyte are prescribed in various Tables in Section 4.0 of this CSSA QAPP and these limits represent the long-term analytical precision for CSSA programs. The laboratory-established long-term analytical precision is not a reporting requirement for the data packages for CSSA. The laboratory established control limits for each analyte should not be wider than the limits specified in the various Tables in Section 4.0.

Single analytical batch precision can be measured from laboratory duplicates [e.g. LCS and Laboratory Control Sample Duplicate (LCSD)]. However, this is not a requirement for CSSA projects.

Total precision is the measurement of the variability associated with the entire sampling and analysis process. Total precision can be determined from the analysis of field duplicate or matrix duplicate samples. However, total precision is not a requirement for CSSA projects, unless otherwise indicated in the DQO. Field duplicates will be collected only if the DQO requires it. Total precision, if necessary, will be defined in the data quality objectives, based on guidance provided in various Tables in Section 4.0 of the CSSA QAPP. Total precision is calculated as relative percent difference (RPD) and the formula for RPD is presented in Table 2.7.

2.7.2 Accuracy

Accuracy is the degree of agreement of a measured value with the true or expected value of the quantity of concern. The concept of accuracy includes the concept of precision (Taylor, ibid). Accuracy includes components of random error (variability due to imprecision) and systematic error. It therefore reflects the total error associated with a measurement. A measurement is 100% accurate when the value reported does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is measured by comparing the percent recovery (%R) of analytes spiked into a LCS to the spiked concentration (refer to Table 2.7 for a formula). The spiked concentration of an analyte in LCS is considered 100%.

Camp Stanley requires that the accuracy values for each analyte in an analytical batch be reported in data packages. Accuracy values for a batch should be compared to the approved control limits (see tables in Section 4.0) for specified analytes. If an analyte is recovered outside of the approved limits, that analyte values for that batch of samples are unacceptable unless otherwise specified in the CSSA QAPP or the DQO for a project. See Section 4.0 for flagging criteria and Section 9.0 for corrective actions. The decisions to support the qualifier flags must be documented in the case narratives.

2.7.3 Representativeness

Objectives for representativeness are defined for each sampling and analysis task, and are a function of the investigative objectives. Representativeness shall be achieved through use of the standard sampling, and analytical procedures. Representativeness is also determined by appropriate program design, with consideration of elements such as proper well locations, drilling and installation procedures, and sampling locations. Decisions regarding sample/well/boring locations and numbers, and statistical sampling design are documented in Section 3.3 of the AFCEE Model FSP. Numerical value criteria for representativeness could not be defined prior to the start of the project. However, during and at the end of sampling and analytical events, the data should be reviewed with representativeness of the field conditions in mind.

2.7.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions. (EPA QA/G-5). The required completeness for a project will be defined by the DQOs for the project.

For CSSA, valid data is defined as usable data that meets the objectives of the specific project. Completeness is calculated for the aggregation of usable data for each analyte measured for any particular sampling event or other defined set of samples. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of usable results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set.

The laboratory is not required to calculate completeness. The prime contractor shall review the R flagged data for usability for the project and calculate completeness based on the usable data. It is the responsibility of the prime contractor to review the appropriateness of the flags based on the DQOs and guidelines presented in Sections 4.0 and 6.0 of the CSSA QAPP.

The guidelines for completeness are 95 percent for aqueous samples and 90 percent for soil samples. However, completeness requirement will be specified in the DQO.

2.7.5 Comparability

Comparability is the confidence with which one data set can be compared to another data set. The objective for this DQI is to produce data with the greatest possible degree of comparability. The number of matrices that are sampled and the range of field conditions encountered are considered in determining comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units and using standard and comprehensive reporting formats. Complete field documentation using standardized data collection forms shall support the assessment of comparability. Analysis of performance evaluation (PE) samples and reports from audits shall also be used to provide additional information for assessing the comparability of analytical data produced among subcontracting laboratories. Historical comparability shall be achieved through consistent use of methods and documentation procedures throughout the project. Comparability should take into consideration varying field conditions (wet/dry seasons), data produced under different DQOs and involvement of multiple laboratories during the life of a project.

Table 2.7
The Data Quality Indicators

Data Quality Indicators	Symbol	Formula	Definition	Uses
Mean	$\overline{X}$	$\frac{\left(\sum_{i=1}^{n} x_i\right)}{n}$	Measure of central tendency	Used to determine average value of measurements
Standard Deviation	S	$\left(\frac{\Sigma(x_i-\overline{x})^2}{(n-1)}\right)^{1/2}$	Measure of relative scatter of the data	Used in calculating variation of measurements
Relative Standard Deviation (RSD)	RSD	$(S/\overline{X}) \times 100$	Relative standard deviation, adjusts for magnitude of observations	Used to assess precision for replicate results
Percent Difference	%D	$\left(\frac{x_1 - x_2}{x_1}\right) \times 100$	Measure of the difference of 2 observations	Used to assess accuracy
Relative Percent Difference	RPD	$\left(\frac{(x_1 - x_2)}{(x_1 + x_2)/2}\right) \times 100$	Measure of variability that adjusts for the magnitude of observations	Used to assess total and analytical precision of duplicate measurements
Percent Recovery (in LCS)	%R	$\left(\frac{x_{meas}}{x_{true}}\right) \times 100$	Recovery of spiked compound in pure matrix	Used to assess accuracy
Percent Recovery (in a sample)	%R	$\frac{\left(\text{value of spiked sample} - \text{value of unspiked sample}\right)}{\text{Value of added spike}} \times 100$	Recovery of spiked compound in sample matrix	Used to assess matrix effects
Correlation Coefficient	r	See SW8000B Subsection 7.5.2	Evaluation of "goodness of fit" of a regression line	Used to establish linearity of calibration
Coefficient of Determination	COD	See SW8000B Subsection 7.5.3	Evaluation of "goodness of fit" of a polynomial equation	Used for non-linear calibration curve.
Completeness	%	$\frac{\text{Number of usable results}}{\text{Number of possible results}}$	See text in Subsection 2.7.4	To generate a sufficient amount of valid data based on project needs.

x — Observed concentration
n — Number of observations

3.0 SAMPLE MANAGEMENT

Collection procedures for environmental samples, covering all matrices at CSSA are provided in Section 2.0 of the FSP, located in the **Environmental Encyclopedia, Volume 1-4, Sampling and Analysis Plan**. The FSP includes sample handling, identification and labeling, preservation, custody, quality control, record keeping, packaging, and shipping requirements.

According to SW846, Chapter 1, Subsection 3.4.1, the following guidelines are presented to assist the field staff in collection of appropriate number of field QC samples:

Each day of sampling, at least one equipment rinsate should be collected for each matrix sampled. If this frequency is not appropriate for the sampling equipment and method, then the appropriate changes should be clearly identified in the DQO. A trip blank is also recommended for each cooler that contains volatile organic analysis (VOA) samples. In addition, for each sampling batch (20 samples of one matrix type), enough volume should be collected for at least one sample so as to allow the laboratory to prepare one matrix spike (MS) and one matrix duplicate (MD). If there is insufficient sample from one container to prepare both MS and MD, two different samples may be used, one for MS and another for MD.

The following control samples are recommended, unless otherwise specified in the DQO:

- Field duplicate only if specified in the DQO
- Equipment rinsate (one per day per matrix type)
- Trip blank (one per day, volatile organics only)
- Matrix spike (MS, one per batch [20 samples of each matrix type])
- Matrix duplicate (MD, one per batch)

Water samples collected during well installations (installation derived waste and discrete samples) should be analyzed for volatile organic compounds. Three VOA vials are required for each sample.

Investigation Derived Waste (IDW)

Investigation derived wastes consist of soil cuttings and groundwater generated during site investigations. According to TNRCC guidance (see letter to LTC. Shnelling from TNRCC, August 12, 1996 and subsequent correspondence from Mr. Ken Rice, Parson Engineering to Mr. Kirk Coulter, TNRCC, letter dated June 15, 2001) the following procedures will be implemented at CSSA:

- All soil cuttings from subsurface sampling will be bagged until analytical results are received.
- Analytical results from soil cuttings will be compared to TNRCC Risk Reduction Standard 2 (RRS2).

- If the analytical results are below RRS2, the soil will be placed next to the boring from which they were derived.
- If the soil analytical results are higher than RRS2 levels, proper waste classification will be determined as specified under 30 TAC §335 Subchapter R (waste classification) and 40 Code of Federal Regulations (CFR) Part §261 (identification and listing of Hazardous Waste) and disposed of properly in accordance with TNRCC and EPA guidelines.
- Soil cuttings that are suspected of contamination will be placed in approved Department of Transportation (DOT) 55-gallon drums.
- IDW groundwater suspected to be contaminated would be containerized until analytical results are available.
- Once waste classification has been made, the groundwater will be properly disposed of.
- If groundwater is non-hazardous (below RRS2, industrial groundwater protection levels), groundwater will be poured next to the well or boring.
- Decontamination fluids used during investigation will be treated similar to groundwater.

The IDW and discrete samples are used for screening purposes and the laboratory and data package requirements are described in appropriate sections of this QAPP.

3.1 Quality of Sample Containers

Sample containers should be purchased pre-cleaned and treated according to EPA specifications for the methods. Sampling containers that are reused should be decontaminated between uses by the EPA-recommended procedures (*Specifications and Guidance for Contaminant-Free Sample Containers, EPA 540/R-93/051*). Containers should be stored in clean areas to prevent exposure to fuels, solvents, and other contaminants.

3.2 Sample Volumes, Container Types, and Preservation Requirements

Sample volumes, container types, and preservation requirements for the analytical methods performed on CSSA samples are listed in Table 3.2. The required sample volumes, container types, and preservation requirements for analytical methods proposed for project work not listed in Table 3.2 shall be included in an addendum to the FSP and approved by CSSA before use.

The sample collection requirements for water and soils are presented below. For IDW and discrete samples, the requirements are presented in Section 3.0.

If possible, matrix spike determinations should be assigned to samples that are expected to contain low or non-detect analytes of interest. This will enable the laboratory to spike the samples at mid-levels of the linear range and still be able to quantify the spike samples within the linear range.

If possible, matrix duplicate determinations should be assigned to samples that are expected to have quantifiable low to medium concentrations of the analytes of interest. This will enable the laboratory to calculate precision from two measurable quantities.

3.2.1 For Soil and Rock Samples

When Requesting Volatiles Analysis Alone from a Sampling Location:

- For each sample location, two 4-ounce containers are needed. One container should be marked for moisture determination only, and the other for VOC analysis only.
- MS and MD determinations can be accomplished from the container for VOC analysis (the one NOT marked for moisture).

When Requesting Semivolatiles and/or Metals from a Sampling Location:

- One 4-ounce jar filled to the top is needed. From this single container, moisture determination and other analyses can be done.
- When MS and MD are requested for a sample, there is no need to collect additional containers. The material in a 4-ounce jar is sufficient to conduct all required analyses.

When Requesting Volatiles, Semivolatiles, and Metals from a Sampling Location:

- For each sample, two 4-ounce containers are needed: one for moisture (so labeled) and another for other analyses.
- From the one unopened container, perform volatiles analysis (including VOC-MS and/or VOC-MD) first.
- Semivolatile and metal analyses (including the corresponding MS and MD) can be done from either container, after sample aliquots for VOC analysis have been removed.

3.2.2 For Water Matrices Samples

When Requesting Volatiles from a Sampling Location:

- Three VOA vials are required for gas chromatography/mass spectroscopy (GC/MS) (SW8260)
- When MS and MD are required from a sample, three additional VOA vials are needed for each determination.

When Requesting Metals Analysis from a Sampling Location:

- One 500-ml plastic container of water preserved with nitric acid is needed. MS and MD can be done from the sample in the one container.

When Requesting Semivolatile Analysis from a Sampling Location:

- One 1-liter container is needed.
- When MS and MD are both required from a sample, three 1-liter containers are needed.

It is critical that the chain-of-custody (COC) forms identify which field sample container should be used for parent, MS, and MD analysis. For a detail discussion of COC forms, see Section 3.3.

Table 3.2
Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times

Name	Analytical Methods	Container [a]	Preservation [b,c]	Minimum Sample Volume or Weight	Maximum Holding Time
Asbestos	OSHA method ID-191	G	N/A	10 grams	N/A
Alkalinity	E310.1	P, G	4°C	50 mL	14 days
Corrosivity	SW1110	G	None required	10 mL	N/A
Common Anions	SW9056	P, G	None required	50 mL	28 days for Br^-, F^-, Cl^-, and SO_4^{-2}; 48 hours for NO_3^-, NO_2^- and PO_4^{-3}
Cyanide, Total and Amenable to Chlorination	SW9010 SW9012	P, G, T	4°C; NaOH to pH > 12, 0.6 g ascorbic acid	500 mL or 4 ounces	14 days (water and soil)
Filterable Residue	E160.1	P, G	4°C	100 mL	7 days
Nonfilterable Residue	E160.2	P, G	4°C	100 mL	7 days
Hydrogen Ion (pH) (W, S)	SW9040 SW9045	P, G	None required	N/A	Analyze immediately
Ignitability	SW1020	G	None required	10 mL	N/A
Nitrogen, nitrate + nitrite	E353.1	P, G	4°C, H_2SO_4 to pH < 2	500 mL	28 days
Conductance	SW9050	P, G	None required	N/A	Analyze immediately
Temperature	E170.1	P, G	None required	N/A	Analyze immediately
Dissolved Oxygen	E360.1	G	None required	500 mL	Analyze immediately
Turbidity	E180.1	P, G	4°C	N/A	48 hours
Total Organic Carbon	SW9060	P, G, T	4°C, HCl or H_2SO_4 to pH < 2	500 mL	28 days
Chromium (VI)	SW7196	P, G, T	4°C	500 mL or 8 ounces	24 hours (water); 30 days until extraction and 4 days after extraction (soil)

Table 3.2 (Continued)
Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times

Name	Analytical Methods	Container	Preservation	Minimum Sample Volume or Weight	Maximum Holding Time
Mercury	SW7470 SW7471	P, G, T	HNO_3 to pH < 2, 4°C	500 mL or 8 ounces	28 days (water and soil)
Metals (except chromium (VI) and mercury)	SW6010 SW6020 and SW846 AA methods	P, G, T	HNO_3 to pH < 2, 4°C	500 mL or 8 ounces	180 days (water and soil)
Total Petroleum Hydrocarbons (TPHs) –volatile	SW8015 (modified)	G, Teflon™-lined septum, T	4°C, HCl to pH < 2	3 x 40 mL or 4 ounces	14 days (water and soil); 7 days if unpreserved by acid (water)
Total Petroleum Hydrocarbons – extractable	SW8015 (modified)	G, Teflon™-lined septum, T	4°C	1 liter or 8 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Total Petroleum Hydrocarbons	TX1005	G, Teflon™-lined septum, T	4°C, HCl to pH < 2	3 x 40 mL or 4 ounces	14 days (water and soil)
Total Petroleum Hydrocarbons Screen (immunoassay)	SW4030	G, Teflon™-lined septum	4°C	4 ounces	14 days
Organochlorine Pesticides	SW8081	G, Teflon™-lined cap, T	4ºC	1 liter or 4 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Polychlorinated Biphenyls (PCBs)	SW8082 or SW4020 immunoassay	G, Teflon™-lined cap, T	4°C	1 liter or 4 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)

Table 3.2 (Continued)
Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times

Name	Analytical Methods	Container	Preservation	Minimum Sample Volume or Weight	Maximum Holding Time
Organophosphorus Pesticides	SW8141	G, Teflon™-lined cap, T	4°C	1 liter or 4 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Semivolatile Organics	SW8270	G, Teflon™-lined cap, T	4°C, 0.008% $Na_2S_2O_3$	1 liter (amber) or 4 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Volatile Organics	SW8260	G, Teflon™-lined septum, T	4°C, 0.008% $Na_2S_2O_3$ (HCl to pH < 2 for volatile aromatics)[b]	3 x 40 mL or 4 ounces	14 days (water and soil); 7 days if unpreserved by acid (water)
Volatile Organics in Soil Vapor	SW8021 (modified)	Tedlar bag	Ambient temperature	1 liter	72 hours from time of collection
Polynuclear Aromatic Hydrocarbons (PAHs)	SW8310	G, Teflon™-lined cap, T	4°C, store in dark, 0.008% $Na_2S_2O_3$	1 liter or 4 ounces	7 days until extraction and 40 days after extraction (water); 14 days until extraction and 40 days after extraction (soil)
Explosive residues	SW8330	P, G, T	Cool, 4°C	1 liter or 8 ounces	7 days to extraction (water); 14 days to extraction (soil); analyze-within 40 days after extraction

Table 3.2 (Concluded)
Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times

Name	Analytical Methods	Container	Preservation	Minimum Sample Volume or Weight	Maximum Holding Time
TCLP	SW1311	G, Teflon™-lined cap, T	Cool, 4°C	1 liter or 8 ounces	For Volatiles: 14 days to TCLP extraction and 14 days after extraction For Semivolatiles: 14 days to TCLP extraction, 7 days to prep extraction and 40 days after prep extraction For Mercury: 28 days to TCLP extraction and 28 days after extraction For Metals: 180 days to TCLP extraction and 180 days after extraction.
Volatile Organics	TO-14	SUMMA® canister	None	Contact the laboratory for minimum canister pressure requirements	14 days

[a]Polyethylene (P); glass (G); brass sleeves in the sample barrel, sometimes called California brass (T).
[b]No pH adjustment for soil.
[c]Preservation with 0.008 percent $Na_2S_2O_3$ is only required when residual chlorine is present.

3.3 Laboratory Sample Management

Procedures to ensure the custody and integrity of the samples begin at the time of sampling and continue through transport, sample receipt, preparation, analysis and storage, data generation and reporting, and sample disposal. Records concerning the custody and condition of the samples should be maintained in field and laboratory.

Once the samples reach the laboratory, they shall be checked against information on the COC forms for anomalies.

CSSA's COC form contains the following information, and they should be verified on receipt in the laboratory:

- Unique field sample identification including sample location
- Date and time of sampling
- Sample matrix
- Preservative used
- For water samples, specify if the sample is filtered or unfiltered
- Number of containers
- Designation of MS and MD (see Section 3.2)
- Analysis requested, preferably by analytical method number
- Project and shipping information
- Custody transfer signatures with corresponding dates and times for each transfer, from the field to the transporters, and to the laboratories.

The condition, temperature, and appropriate preservation of samples shall be checked and documented on the COC form. Checking an aliquot of the sample using pH paper is an acceptable procedure except for VOCs where an additional sample is required to check preservation. If additional VOA vial is not available, checking the VOC sample at the time of analysis is also acceptable provided the analysis is done within a seven-day holding time. All sample information shall then be entered into a tracking system, and unique laboratory analytical sample identifiers shall be assigned. There is no need to uniquely identify each container for the same sample. If a Laboratory Information Management System (LIMS) is used in the sample processing area, the section supervisor shall verify the information entered into the system. Sample holding time tracking begins with the collection of samples and continues until the analysis is complete. Holding times are specified in Table 3.2.

If the laboratory receives samples improperly preserved, beyond holding times or in any unacceptable manner, the laboratory must contact the project manager of the prime contractor immediately to discuss the issues. The project manager must immediately contact AFCEE (Team Chief and/or AFCEE ERC chemist) to discuss the issues and seek resolution. Decisions made during the discussion must be documented and included as part of the case narratives in the respective data packages and the data should be flagged accordingly. A representative from AFCEE or the prime contractor shall notify CSSA of issues and remedies.

Subcontracted analyses shall be documented with the CSSA COC form. If the primary laboratory uses its own COC form, the prime contractor must approve the form. Procedures ensuring internal laboratory COC shall also be implemented and documented by the laboratory. Specific instructions concerning the analysis specified for each sample shall be communicated to the analysts. Analytical batches shall be created, and laboratory QC samples shall be introduced into each batch.

While in the laboratory, samples shall be stored in limited-access and temperature-controlled areas. Refrigerators, coolers, and freezers shall be monitored for temperature seven days a week. Acceptance criteria for the temperatures of the refrigerators and coolers is 4°C ± 2°C. Acceptance criterion for the temperatures of the freezers shall be less than 0°C. Thermometers that have been calibrated with a National Institute of Science and Technology (NIST)-traceable thermometer shall be used to monitor cold storage areas. As indicated by the findings of the calibration, correction factors shall be applied to each thermometer. Records that include acceptance criteria shall be maintained. Samples for volatile organics' analyses shall be stored separately from other samples, standards, and sample extracts. After analysis, samples shall be stored until disposed of in accordance with applicable local, state, and federal regulations. The laboratory shall maintain disposal records for a minimum of five years.

The laboratory shall maintain standard operating procedures (SOPs) describing sample control and custody, particularly describing how limited access, temperature control, temperature monitoring and other aspects of sample management, are maintained and documented.

4.0 ANALYTICAL METHODS GUIDANCE

This section presents the analytical guidance for CSSA activities and is divided as follows:

- Section 4.0 provides general information.
- Section 4.1 provides information on screening analytical methods.
- Section 4.2 provides information on preparation methods.
- Section 4.3 provides explanation of calibration parameters for definitive data methods. Explanation of quality control parameters for field and the laboratory are provided in Section 5.0.
- Section 4.4 provides specific reporting limits, calibration, and quality control requirements for all definitive data analytical methods.
- Section 4.5 provides information on TPDES required analytical methods.
- Section 4.6 provides information on methods required for monitored natural attenuation (MNA).

SW846 is a guidance document meant to assist analytical chemists and other users in the RCRA program. Methods in SW846 do not need to be implemented exactly as written (see *Current Perspectives in Site Remediation and Monitoring, by D.M. Crumbling and Barry Lesnik, EPA 542-R-01-015, October 2001*). On the contrary, the Superfund program maintains a Contract Laboratory Program (CLP) with highly prescribed methodologies, reporting limits and QA/QC criteria, not for regulatory requirements but for contract requirements.

CSSA is under a RCRA order and will use SW846 methods and not CLP methods. The analytical methods in this section are identified only by their numbers (e.g., 8260 for volatile organic compounds) and do not include the letter designations (e.g., 8260B). The most recent promulgated versions of the method will be specified for a project and will be included in the scoping documents for each project. The new promulgated methods will be included in the CSSA QAPP revisions and the methods in the current CSSA QAPP will be archived.

CSSA will use other appropriate methods for meeting compliance requirements.

Both EPA Region 6 and TNRCC have approved changes in RLs for some analytes in Method SW8260 and these changes have been incorporated in the Table 4.4.4-1. For groundwater, the regulators have also approved a shorter analyte list for Method SW8260 for future investigations. Similarly the approved changes for Method SW6010 and some of the SW7000 methods are incorporated in the respective tables. All approved changes are in **bold** letters in the respective tables.

Although this section presents the methods with a complete analyte list and quality control guidance, the DQOs for a specific project and site will determine the list of analytes required and the level of quality control necessary to make the decisions for that site or project.

CSSA may require additional methods that are not discussed in Section 4.0. Any additional analytical methods should be calibrated and quality controlled according to published method requirements. In the event a new unpublished procedure is being used, the calibration and quality control of the instrument must conform to the manufacturer's protocol. The laboratory must provide calibration and quality control criteria for these methods to the project manager who will obtain approval from the AFCEE Team Chief and CSSA. The approved methods and quality control information will be added to the project scoping documents

Sample requirements for various CSSA projects will be specified in the respective field sampling plans. Container requirements, preservation requirements, storage conditions, and holding times are presented in Table 3.2.

4.1 Screening Analytical Methods

For CSSA, screening analytical methods are defined as those methods that do not require rigorous calibration and quality control activities. Some of the methods are normally carried out in the field (e.g., temperature, pH) and others are carried out in a field or fixed-base laboratory. The screening methods contained in this section are shown in Table 4.1. The field manager should document the field analytical results along with the calibration and quality control information. The chain-of-custody form may require the field manager to document the pH and the temperature of samples.

This section includes brief descriptions of the methods and QC required for screening procedures. The methods and QC procedures were taken from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (U.S. EPA SW846, Third Edition, and its first, second and third update), *Methods for Chemical Analysis of Water and Waste* (U.S. EPA 1979), *ASTM Annual Book of Standards* (1993), and from manufacturers' literature.

CSSA has elected to request less stringent quality control for SW8260 when rapid turnaround (24-hours or less) of results are needed. These analyses are sent to fixed base laboratories that would provide a fast turnaround of results. The QA/QC requirements for these analyses are listed in this section (see Table 4.1.1).

This section also contains brief method descriptions and RLs where applicable.

Table 4.1
Screening Analytical Methods

Method	Parameter
SW1020	Ignitability
SW1110	Corrosivity
SW9040	pH (water)
SW9045	pH (soil)
SW9050	Conductance
SW9060	Total organic carbon
E160.1	Filterable residue
E160.2	Nonfilterable residue
E170.1	Temperature
E180.1	Turbidity
E310.1	Alkalinity
E360.1	Dissolved oxygen
ASTM D1498	Oxidation-reduction potential (ORP)
ASTM D3416	Methane in Soil Gas
SW4020	PCBs by Immunoassay
SW4030	TPH by Immunoassay
SW3550, Section 7.2	Percent Moisture
Organic Vapor	Soil gas screening for halogenated aromatic, and petroleum hydrocarbons
X-Ray Fluorescence (NITON)	Various metals
OSHA Method ID-191	Asbestos screening
SW8021 Screening (Soil Gas)	Aromatic and halogenated volatile organic compounds in Soil Gas Samples

4.1.1 Current EPA Method SW1020-Ignitability

Method 1020 makes use of the Setaflash Closed Tester to determine the flash point of liquids that have flash points between 0° and 110°C and viscosities lower than 150 stokes at 25°C.

4.1.2 Current EPA Method SW1110-Corrosivity

This test exposes steel to liquid waste to determine the corrosivity of the waste.

4.1.3 Current EPA Method SW9040 (water)/SW9045 (soil)–pH

The pH measurements shall be performed for water samples using Method SW9040. The pH measurements of soil samples are performed using Method SW9045. Measurements are determined electrometrically using either a glass electrode in combination with a reference potential, or a combination electrode.

4.1.4 Current EPA Method SW9050–Conductance

Standard conductivity meters are used. Temperature is also reported.

4.1.5 Current EPA Method SW9060–Total Organic Carbon

Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide by either catalytic combustion or wet chemical oxidation. The carbon dioxide formed is then either measured directly by an infrared detector or converted to methane and measured by a flame ionization detector (FID). The amount of carbon dioxide or methane in a sample is directly proportional to the concentration of carbonaceous material in the sample.

Method	Analyte	Water	
		RL	Unit
SW9060	Total organic carbon	1	mg/L

4.1.6 Current EPA Method 160.1–Filterable Residue (Total Dissolved Solids)

A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180 °C.

Method	Analyte	Water	
		RL	Unit
E160.1	Total dissolved solids	10	mg/L

4.1.7 Current EPA Method 160.2–Nonfilterable Residue (Total Suspended Solids)

A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105 °C.

Method	Analyte	Water	
		RL	Unit
E160.2	Total suspended solids	5	mg/L

4.1.8 Current EPA Method 170.1–Temperature

Temperature measurements are made with a mercury-filled centigrade thermometer.

4.1.9 Current EPA Method 180.1–Turbidity

This method is based on a comparison of the light scattered by the sample under defined conditions with the light intensity scattered by a standard reference suspension, the higher the intensity, the greater the turbidity. Turbidity measurements are made in a nephelometer and are reported in terms of nephelometric turbidity units (NTUs). The working range for the method is from 0–40 NTU. Higher levels of turbidity can be measured by diluting the sample with turbidity-free deionized water.

4.1.10 Current EPA Method 310.1–Alkalinity

In this method, an unaltered sample is titrated to an end point of pH 4.5 using hydrochloric or sulfuric acid (H_2SO_4).

Method	Analyte	Water	
		RL	Unit
E310.1	Alkalinity[1]	10	mg/L

[1]Alkalinity measured as calcium carbonate equivalence.

4.1.11 Current EPA Method 360.1–Dissolved Oxygen

An instrumental probe, usually dependent upon an electrochemical reaction, is used for determination of dissolved oxygen in water. Under steady-state conditions, the current or potential can be correlated with dissolved oxygen concentrations.

4.1.12 Current ASTM D1498–Oxidation-Reduction Potential

This method is designed to measure the oxidation-reduction potential in water, which is defined as the electromotive force between a noble metal electrode and a reference electrode when immersed in a solution.

4.1.13 Current ASTM D3416–Methane in Soil Gas

An aliquot of the soil gas sample is introduced into a prechromatographic or stripper column, which removes hydrocarbons other than methane and carbon monoxide. Methane and carbon monoxide are passed through a chromatographic column where they are separated. A flame ionization detector measures the methane. Quantitation is performed by comparing the sample response to the response of a known concentration of methane.

4.1.14 Current EPA Method SW4020–Screening for Polychlorinated Biphenyls by Immunoassay

Soil samples are screened for total polychlorinated biphenyls (PCBs) using immunoassay test kits. A mini methanol extraction of the soil sample is performed, and the extract and an enzyme conjugate reagent are added to immobilized antibodies. The enzyme conjugate competes with the PCBs in the sample for binding to immobilized anti-PCB antibodies. The test is interpreted by comparing the response produced by the sample to the response produced by a standard.

4.1.15 Current EPA Method SW4030–Screening for Petroleum Hydrocarbons by Immunoassay

Soil samples are screened for levels of total petroleum hydrocarbons using TPH test kits. A mini extraction of the soil sample is performed, and the extract and an enzyme conjugate reagent are added to immobilized antibodies. The enzyme conjugate competes with hydrocarbons for binding to immobilized anti-hydrocarbon antibodies. The test is interpreted by comparing the response produced by the sample to the response produced by a standard.

4.1.16 SW3550, Section 7.2–Percent Moisture

Percent moisture is determined for solid samples undergoing analysis for inorganic and organic analytes. The sample is weighed, dried, and then reweighed. Percent moisture is calculated as:

$$\frac{\text{Initial Weight - Dried Weight}}{\text{Initial Weight}} \times 100 = \%\ \text{moisture}$$

The moisture content is used to calculate results for soil samples on a dry weight basis using the calculation presented below:

$$\frac{\text{Result of analysis on wet weight basis}}{1 - (\%\ \text{Moisture}/100)} = \text{Result of analysis on a dry weight basis}$$

All soil and sediment results and MDLs shall be reported on a dry weight basis.

For soil and sediment samples, method recommended amounts should be weighed from each sample for analytical purposes. There is no need to adjust the weight of the sample based on moisture content in order to obtain the same reporting limit for all samples. If the laboratory encounters special situations where deviation from the above rule is deemed necessary, the laboratory should contact the prime contractor to obtain variance. Samples for analysis and for moisture determination may be weighed from the same container on the same day.

4.1.17 Real-Time Portable Organic Vapor Analyzers (OVAs)

Two types of portable analyzers shall be used to perform real-time non-specific analyses of hydrocarbon vapors. The instruments include a FID and a photoionization detector (PID) organic vapor monitor. One or more of these instruments may be used at a specific site, depending on the contaminant species of interest. When used together, the instruments provide complementary information because they are sensitive to different types of hydrocarbon vapors.

The portable analyzers shall be used as a screening tool to help determine the optimum locations for the collection of samples. Field data recorded on the COC forms give the laboratory analysts an indication of the approximate concentration of contaminants and aid in calculating dilution factors before analysis. Additionally, the real-time instruments are used to aid in selecting the proper level of personal protective equipment and monitoring air emissions during sampling activities. The comparability of results obtained from the PID and FID instruments can be considered only to be within the variability of this type of screening instrument. Comparability is greatest when the instruments are calibrated with the same standards and operated within similar concentration ranges.

The FID uses the principle of hydrogen flame ionization to detect and measure total hydrocarbon vapors. The FID has a dynamic operating range from 1 part per million volume (ppmv) to 10 ppmv or 1 ppmv to 100,000 ppmv, depending on the instrument, and provides a non-specific response to total hydrocarbons. If concentrations exceed the range of the instrument, a dilution probe shall be attached to the FID to allow elevated vapor concentrations to be measured. The instrument is highly sensitive to compounds such as methane, benzene, and acetone; but is less sensitive to alcohols and halogenated compounds.

During operation, a sample is drawn into the probe and transmitted to the detection chamber by an internal pumping system. Inside the chamber, the sample is exposed to a hydrogen flame that ionizes the organic vapors. As the organic vapors burn, the ions produced are collected on an electrode in the chamber, and a current proportional to the hydrocarbon concentration is generated. This current is measured and displayed on the meter.

The PID uses a photo ionization detector to detect and measure mainly aromatic hydrocarbon vapors. The instrument has an operating range of 0-2,000 parts per million (ppm). During operation, a gas sample is drawn into the probe and past an ultraviolet light source by an internal pumping system. Contaminants in the sample are ionized, producing an instrument response if their ionization potential is equal to or less than the ionizing energy supplied by the lamp. The radiation produces a free electron for each molecule of ionized contaminant, which generates a current directly proportional to the number of ions produced. This current is measured and displayed on the meter. The PID measures the total value for all species present with ionization potentials less than or equal to that of the lamp.

4.1.18 Metals Screening by X-Ray Fluorescence (XRF)

Hand held XRF analyzers may be used to detect metals contamination in soils. Niton Corporation has several analyzers available for lead-based paint testing, soils and air filter testing, and for rapid identification of metals and metal alloys. Based on the objectives of screening a specific niton analyzer, may be chosen from the following:

- XL-300 Series Analyzer is used for lead-based paint testing, on-site analysis of dust wipes, air filters, paint chips, and soil.
- XL-700 Series Multi-Element Analyzer is used for multi-element testing of soils, air filters, and thin-film samples. It is available with or without lead-based paint testing.
- XL-800 Series Analyzer is designed for rapid identification of metal alloys, ideal for scrap metal sorting.

In the event one of these analyzers are used in CSSA for screening purposes, the calibration and quality control of the chosen instrument must comply with manufacturer's protocol. EPA has incorporated SW6200 method for testing of metals in soils and sediments by XRF. If applicable, the SW6200 method should be consulted to incorporate required calibration and quality control requirements.

4.1.19 Asbestos Screening in Bulk Materials by OSHA Method ID-191

This method uses polarized light microscopy techniques including phase-polar illumination and central-stop dispersion microscopy. Identification of a particle as asbestos requires that it be asbestiform. A microscopist experienced in distinguishing asbestiforms from other patterns or arrangements should perform the analysis.

4.1.20 Aromatic and Halogenated Volatile Organic Compounds in Soil Vapor Samples

This is a mobile laboratory screening method for benzene, toluene, ethylbenzene, xylene (BTEX); tetrachloroethylene; trichloroethylene; cis-1,2-dichloroethene; trans-1,2-dichloroethene; and vinyl chloride. For CSSA a modified SW8021B is being used by DHL Analytical, Inc. CSSA has the option to use other mobile analytical laboratories. The method is used for analyzing soil vapors and uses photoionization and Hall electrolytic conductivity detectors, connected in series. No second column confirmation is required.

4.1.21 Calibration and QC Procedures for Screening Methods

Table 4.1.1 presents the calibration and QC procedures for each method. These requirements, as well as the corrective actions criteria, are included. In this table, the first two columns designate the method number and the class of analytes that may be determined by the method. The third column lists the method-required calibration and QC elements. The fourth column designates the minimum frequency for performing each calibration and QC element. The fifth column designates the acceptance criteria for each calibration and QC element. The sixth column

designates the corrective action in the event that a calibration or QC element does not meet the acceptance criteria.

Table 4.1.1
Summary of Calibration and QC Procedures for Screening Methods

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
SW9045	pH (soil)	2-point calibration with pH buffers that bracket the expected sample pH	Once per day at the beginning of testing	± 0.05 pH unit on repeat measurement of the calibration buffers	Check with new buffers; if still out, repair meter; repeat calibration check
		pH 7 buffer	At each sample location	± 0.1 pH unit	Recalibrate
		Duplicate sample	10% of field samples	± 0.1 pH unit	Correct problem, repeat measurement. If still out, repeat calibrations and reanalyze samples
SW9040	pH (water)	2-point calibration with pH buffers that bracket the expected sample pH	Once per day at the beginning of testing.	± 0.05 pH unit on repeat measurement of the calibration buffers	Check with new buffers; if still out, repair meter; repeat calibration check
		PH 7 buffer	At each sample location	± 0.1 pH units	Recalibrate
SW9050	Conductance	Calibration with KCl standard	Once per day at beginning of testing	± 5%D	If calibration is not achieved, check meter, standards, and probe; recalibrate
		Field duplicate	10% of field samples	± 5%RPD	Correct problem, repeat measurement

Table 4.1.1 (Continued)
Summary of Calibration and QC Procedures for Screening Methods

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
E170.1	Temperature	Calibrate field or laboratory thermometers against NIST certified thermometer	Initially calibrate all new thermometers. Recalibrate all thermometers once every 12 months.	Temperature of the measuring devise must match the NIST thermometer measurement.	Apply correction factors to individual thermometers.
E180.1	Turbidity	Calibration with one formazin standard per instrument range used	Once per day at beginning of testing	Follow manufacturer's protocol for calibration.	If calibrations is not achieved, check meter; replace if necessary, recalibrate.
		Split sample	Once per batch	RPD ≤ 20%	Correct problem, repeat measurement.
None	Organic Vapor Concentrations (FID and PID)	3 point calibration	Monthly	$r \geq 0.995$	Recalibrate; check instrument and, if necessary, replace
		Calibration verification and check	Daily at beginning and end of day	Response ± 20% of expected value	Correct problem, recalibrate
SW9060	Total Organic Carbon	Method blank	Daily or one per batch, whichever is more frequent	< RL	Clean system; reanalyze blank. Repeat until analyte < RL
		Field duplicate	10% of field samples	RPD ≤ 20%	Repeat measurement
E160.1	Filterable Residue	Split sample	Once per batch	RPD ≤ 20%	Correct problem, repeat measurement
E160.2	Nonfilterable Residue	Split sample	Once per batch	RPD ≤ 20%	Correct problem, repeat measurement

Table 4.1.1 (Continued)
Summary of Calibration and QC Procedures for Screening Methods

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
ASTM D1498	Oxidation-reduction Potential (ORP)	Sensitivity verification	Daily	ORP should decrease when pH is increased	If ORP increases, correct the polarity of electrodes. If ORP still does not decrease, clean electrodes and repeat procedure.
		Calibration with one standard	Once per day	Two successive readings ± 20 millivolts	Correct problem, recalibrate
		Field duplicate	10% of field samples	± 10 millivolts	Correct problem, repeat measurement
SW1110	Corrosivity	Duplicate	10% of field samples	RPD ≤ 20%	Correct problem, repeat measurement
E310.1	Alkalinity	Split sample	Once per batch	RPD ≤ 20%	Correct problem, repeat measurement
E360.1	Dissolved oxygen	Field duplicate	Once per batch	RPD ≤ 20%	Correct problem, repeat measurement
SW4020	PCBs by immunoassay	Split sample	Once per batch	RPD ≤ 20%	Correct problem, repeat measurement
SW4030	Petroleum hydrocarbons by immunoassay	Split sample	Once per batch	RPD≤ 20%	Correct problem, repeat measurement
ASTM D3416	Methane in soil gas	Three point calibration	Daily, prior to sample analysis	r ≥ 0.995	Recalibrate
		Method blank	Daily or one per batch, whichever is more frequent	< RL	Clean system; reanalyze blank and repeat until all analytes < RL
		Duplicate	1 per batch or 10%	RPD ≤ 20%	Analyze third aliquot: if still out, flag data with J

Table 4.1.1 (Concluded)
Summary of Calibration and QC Procedures for Screening Methods

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
SW8021 Screen for soil gas	Volatile Organic Compounds	Minimum of 3-point initial calibration (bracket expected range of concentrations)	Once at the beginning of the field effort	$r \geq 0.995$	Repeat initial calibration
		CCV at the midpoint standard	Each day before sample analysis and at a min. rate of 10%	75-125% recovery	Perform corrective action and recalibrate
		Blank	At the beginning of each batch	< RL	Perform corrective actions and repeat blank analysis
		Sample duplicate	Once per batch	RPD ≤ 30%	Reanalyze both samples. Discuss in case narrative
		Retention time windows (RTWs)	At the beginning of the field effort and as needed.	± 3SD for each analyte retention time from a 72-hours study	None
		Retention time (RT) check from daily calibration verification	At the beginning of each day	Daily RT should be within the established window for each analyte. Update daily retention time window according to Subsection 4.3.7 of the CSSA QAPP	None
		MDL study	Once per 12 months	Establish according to Subsection 4.3.1	None
SW8260 IDW and discrete samples	Volatile organic compounds	Initial five point calibration	See Table 4.4.4-3		
		Calibration verification	See Table 4.4.4-3		
		Method Blank	See Table 4.4.4-3		
		LCS	See Table 4.4.4-3		

4.2 Preparation Methods

Extraction and digestion procedures for liquid and solid matrices presented in this section are outlined in Table 4.2.

Table 4.2
Extraction and Digestion Procedures

Method	Parameter
SW1311	Toxicity Characteristic Leaching Procedure (TCLP)
SW3005	Acid Digestion of Water Samples for Metals Analysis
SW3010	Acid Digestion of Aqueous Samples and Extracts for Metals Analysis
SW3015	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Metals Analysis
SW3020	Acid Digestion of Aqueous Samples and Extracts for Metals Analysis
SW3050	Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis
SW3051	Microwave Assisted Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis
SW3060	Alkaline Digestion for Hexavalent Chromium
SW3510	Separatory Funnel Liquid-Liquid Extraction
SW3520	Continuous Liquid-Liquid Extraction
SW3535	Solid-Phase Extraction
SW3540/ SW3541	Soxhlet Extraction
SW3550	Ultrasonic Extraction
SW5021	Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis
SW5030	Purge and Trap

4.2.1 Method SW1311–Toxicity Characteristic Leaching Procedure

Current Method SW1311 is used to prepare samples for determination of the concentration of organic (semivolatile and volatile) and inorganic constituents that are leachable from waste or other material.

QC is accomplished by preparing a toxicity characteristic leaching procedure blank at a rate of one blank for every 20 extractions conducted in the extraction vessel. Additional extract is prepared so one MS is performed for each waste type (samples of similar waste types shall be batched together). One MS must be analyzed in each CSSA analytical batch. These QA measures are in accordance with the requirements of EPA Method SW1311.

4.2.2 Current Method SW3005–Acid Digestion of Water Samples for Metals Analysis

This method is an acid digestion procedure used to prepare water samples for metals analysis. The digested samples are analyzed for total recoverable and dissolved metals determination by inductively coupled plasma (ICP).

For analysis of total recoverable metals, the entire sample is acidified at collection time.

For analysis of dissolved metals, upon collection the samples are filtered then acidified.

4.2.3 Current Method SW3010–Acid Digestion of Aqueous Samples and Extracts for Metals Analysis

Current Method SW3010 prepares aqueous or waste samples for total metals determination by ICP. The samples are vigorously digested with acid and then diluted.

4.2.4 Current Method SW3015–Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Metals Analysis

This method is used to prepare aqueous or waste samples that contain suspended solids, for total metals determination by graphite furnace atomic absorption (GFAA) spectroscopy or ICP. The samples are digested with acid and heated in a microwave.

4.2.5 Current Method SW3020–Acid Digestion of Aqueous Samples and Extracts for Metals Analysis

Current Method SW3020 prepares aqueous or waste samples for total metals determination by GFAA or ICP. The samples are vigorously digested with acid and then diluted.

4.2.6 Current Method SW3050–Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis

Current Method SW3050 is applicable to the preparation of sediment, sludge, and soil samples for metals analysis by ICP or, for some metals, by GFAA. A sample is digested then refluxed with acid. A separate aliquot of the sample is dried for a total solids and/or percent moisture determination.

4.2.7 Current Method SW3051–Microwave Assisted Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis

Current Method SW3051 is applicable to the preparation of sediment, sludge, and soil samples for metals analysis by GFAA or ICP. The samples are digested with acid and heated in a microwave. A separate aliquot of the sample is dried for a total solids and/or percent moisture determination.

4.2.8 Current Method SW3060–Alkaline Digestion for Hexavalent Chromium

Current Method SW3060 is applicable to spectrophotometry. The samples are digested with sodium hydroxide.

4.2.9 Current Method SW3510-Separatory Funnel Liquid-Liquid Extraction

Current Method SW3510 is designed to quantitatively extract nonvolatile and Semivolatile Organic Compounds (SVOCs) from liquid samples using standard separatory funnel techniques. The sample and the extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup and detection methods are described in the organic analytical method used to analyze the extract.

4.2.10 Current Method SW3520-Continuous Liquid-Liquid Extraction

Current Method SW3520 is a procedure for isolating organic compounds from aqueous samples and is designed for extraction solvents with greater density than the sample.

4.2.11 Current Method SW3535-Solid-Phase Extraction

Current Method SW3535 is a procedure for isolating organic compounds from aqueous samples using solid-phase extraction media.

4.2.12 Current Method SW3540/SW3541-Soxhlet Extraction

Current Method SW3540 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. Method SW3541 is an automated Soxhlet extraction. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

4.2.13 Current Method SW3550-Ultrasonic Extraction

Current Method SW3550 is a procedure for extracting nonvolatile and SVOCs from solids such as soils and sludges. The sonication process ensures intimate contact of the sample matrix with the extraction solvent.

4.2.14 Current Method SW5021-Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis

Current Method SW5021 is a general-purpose method for the preparation of VOCs in soils, sediments, and solid wastes by gas chromatography (GC) or GC/Mass Spectrometry (MS) analysis.

4.2.15 Current Method SW5030-Purge and Trap

Current Method SW5030 describes sample preparation and extraction for the analysis of VOCs. The method is applicable to nearly all types of samples, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, and water. Results may vary due to the large variability and complexity of matrices of solid waste samples.

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

4.3 Calibration Parameters for Definitive Data

This section contains descriptions of parameters that are required for the calibration of instruments. Where applicable, formulas for calculations are included. Parameters described in this section are applicable to all methods. The next section, Section 4.4, provides additional CSSA method-specific requirements for calibration, quality control, and flagging. Section 5.0 describes all quality control parameters for field and laboratory.

4.3.1 Method Detection Limits (MDLs)

The method detection limit is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero.

Laboratories participating in CSSA work effort shall demonstrate the MDLs for each instrument, including confirmatory columns, method of analysis, analyte, and matrix (i.e., water and soil) using the following instructions:

(1) Estimate the MDL using one of the following:
   (a) the concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5
   (b) the concentration equivalent of three times the standard deviation (SD) of replicate measurement of the analyte in reagent water
   (c) the region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).

(2) Prepare a standard in reagent water (ASTM Type II water) at a concentration between one and five times the estimated method detection limit and analyze seven aliquots of the standard. Ottawa sand instead of reagent water should be used for soil methods. Glass beads of 1 mm diameter or smaller may be used instead of water for determining MDLs for metals in solid matrix.
(3) Determine the standard deviation (s) for each analyte from the seven replicate analyses.

(4) Determine the MDL for each analyte as follows:

$$MDL = 3.14(s)$$

(note: 3.14 is the one-sided t-statistic at the 99 percent confidence level appropriate for determining the MDL using seven samples)

(5) If the spike level used in step two is more than ten times the calculated MDL, repeat the process using a smaller spiking level.

Where multiple instruments are used, the MDL used for reporting purposes shall represent the least sensitive instrument.

NOTE: Code of Federal Regulations 40 CFR, 136, appendix B or Chapter 1 of SW846 methods has not set frequency requirements for revalidating the method detection limits. However, for CSSA the laboratory shall revalidate MDLs whenever there are repairs that affect the sensitivity of the instrument.

The laboratory shall provide results of MDL studies to CSSA at the beginning of the project (i.e., before project samples are analyzed) and upon request, in the format specified in Section 6.0. The MDL study results need not be included with every CSSA data package.

All results shall be reported at or above the MDL values. No numerical results shall be reported below the MDL. Results less than or equal to the MDL shall be reported as the MDL value and flagged with a "U".

CSSA is aware that MDLs calculated according to the above procedure use either ASTM Type II water or Ottawa Sand and are not representative of the sensitivities that could be obtained with field samples of varying matrices.

4.3.2 Reporting Limits

The laboratories participating in CSSA work effort shall compare the results of the experimental MDLs to RLs for each analyte in methods that are listed in Section 4.4 or RLs defined by the project DQOs. The MDL may not be more than one-half the corresponding RL. The laboratories shall also verify RLs by including a standard at or below the RL as the lowest point on the calibration curve.

TNRCC has approved soil and water reporting limits for analytes of concern for volatile and semivolatile organic compound and for metals. These RLs are applicable to all CSSA projects and for all the laboratories participating in these projects. The tables in Section 4.4 of this QAPP list all the approved RLs in **bold** letters.

The prime contractor shall compare the RLs to the action levels of specific projects and assure CSSA that RLs are at or below one-half of the action levels. Variance to elevate RLs will not be granted if they exceed action levels, unless they are pre-approved by regulatory agencies.

4.3.3 Sample Quantitation Limits (SQLs)

SQL is MDL adjusted to reflect sample-specific action such as dilution or use of a smaller sample aliquot for analysis due to matrix effects or high concentration of some analytes (see p. 49, *Guidance for Data Useability in Risk Assessment, (Part A), Final, April 1992, Office of Emergency Response, U.S. Environmental Protection Agency, Washington, DC*). SQLs may be required for some samples during the risk assessment process.

If SQLs are required for a specific project for risk assessment or other purposes, the project scoping documents (Work Plan and Quality Assurance Project Plan) will specify the requirement. The prime contractor has the responsibility to provide the SQLs in the data summary, verification and validation reports, and shall identify all SQLs in the technical report data tables.

Analytical adjustments, such as dilution of a sample for quantitation of extremely high level of one chemical, could result in non-detects for other chemicals in that same sample, even though these chemicals may have been present at trace quantities in the undiluted sample. In this case, the original and dilution results should be submitted for risk assessment purposes, unless otherwise stated in the project DQOs. Appropriate flags should be assigned to invalid results in the original and dilution results.

For TCLP leachates, dilution analysis may be performed due to the nature of the leachates, provided the elevated RLs are below the TCLP Leachate Maximum Concentration criteria.

4.3.4 Instrument Calibration

This section on instrument calibration will cover both the initial and the continuing calibration requirements for CSSA. The requirements are as follows:

Initial Calibration

- Analytical instruments shall be calibrated with **all the analytes of concern required for a particular project** in accordance with the specifications of the analytical methods.
- The initial calibration acceptance criteria for each method are specified in the respective tables in Section 4.4.
- The initial calibration must include a minimum number of standard concentrations required by the method including a standard at or below the corresponding RL. The requirements for each method are specified in the respective tables in Section 4.4.
- If a standard at or below the RLs could not be included as part of the initial calibration curve, then a RL verification must be done after the initial calibration (see Section 4.3.2)

- If more than the required minimum number of standard concentrations is used in the initial calibration, all standard concentrations must be included in calculating the acceptance of the initial curve.
- If the highest concentration for an analyte exceeds the linearity for that analyte, the laboratory may delete the highest concentration point and recalculate the acceptance with all the remaining points. All results for field samples shall be reported only within the calibration linearity range.
- No middle data point in the calibration curve shall be excluded in the calculation of the acceptance of the linearity of the curve.
- Calibration stock solutions may be prepared in the laboratory or obtained from a vendor. *When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source* (see sections on calibration standard preparation in the organic Methods SW846).
- For calibration of multi-response analytes, refer to the individual methods in Section 4.4.
- Immediately after the completion of the initial calibration, a traceability standard at the mid-point of the curve should be analyzed (see the section on traceability for details).
- Records of standard preparation and instrument calibration shall be maintained.
- Records shall unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results.

Calibration Verification

- The initial calibration shall be checked at frequencies specified in the methods or in various tables in Section 4.4.
- The acceptance criteria for the calibration verification are provided in the methods or in the tables in Section 4.4
- The average response factors (RFs) of the initial multi-point calibration shall not be updated with the RF value from the calibration verification.
- The continuing calibration verification standard should be from the primary calibration standards. The mid-level standard from the calibration curve should be used. This prevents unnecessary variables introduced in verifying the original calibration.

4.3.5 Traceability

The concept of traceability is applicable to two areas in the field of analytical chemistry: 1) to establish the purity of calibration standards; and 2) to trace the working standard identification to stock standards or neat (pure) materials through record keeping.

To establish the purity of calibration materials used for all CSSA projects, they should be traceable to national reference standards (EPA QA/G-5, Section B7.5). Traceability to national reference standards can be achieved in several ways:

- Inorganic standard materials from National Institute of Standards and Technology (NIST) could be routinely used as calibration materials if economically feasible. No further traceability for establishing purity is required.
- If inorganic standard materials are purchased from commercial vendors or prepared in the laboratory from commercially obtained neat (pure) materials or stock standards, the purity should be checked against NIST materials once during initial multipoint calibration. The commercial or laboratory prepared standards should be within ± 10% of the NIST value. If the commercial vendor has already checked the purity of the material against NIST standards and is able to provide a certificate of analysis, no additional checks are required to establish the purity in the laboratory.
- For organic calibration standards, NIST or EPA standard materials should be used to check the purity of commercially obtained or laboratory prepared standard materials once during initial multipoint calibration. The commercial or laboratory prepared standards should be within ± 20% for GC and high-performance liquid chromatography (HPLC) methods and ± 25% for GC/MS methods of the national reference materials.
- If NIST or EPA has approved an independent agency to certify the purity of calibration standards, such certified calibration standards can be used without further verification. However, a certificate of purity from the approved certifying agency must be kept on file and available for external review.

When no national reference standards or an approved certifying agency is available, commercially obtained calibration standards should be checked against similar standards from another commercial vendor (second source) once during initial calibration. The acceptance criteria, as stated earlier, should be ± 10% for inorganic and ± 20% for GC and HPLC organic standards, and ± 25% for GC/MS organic standards (acceptance criteria for second source verification are from *Department of Defense Quality System Manual for Environmental Laboratories, Draft version 2, May 2001*).

The second aspect of traceability is established through record keeping relating to standards preparation and use. Analysts should identify the stock standards by vendor, lot number, analyte identity, concentration of the analyte, and other relevant information of stock standards in a standards preparation log. The label from the stock standards may be affixed to the particular page of the standards preparation log. The date of preparing the working standards, the dilutions made, the diluents used, and the expiration date of the working standards shall be included in the log. The working standards shall be identified by unique numbers that are traceable to other analytical records. Other analytical records include, but are not limited to, instrument calibration records, sample preparation records, and analytical run logs.

4.3.6 Internal Standards (ISs)

Internal standards are certain compounds added to sample extracts after preparation or extraction of a sample. ISs shall be added to calibration standards, environmental samples, controls, and blanks in accordance with the method requirements. They are used in an IS calibration method to correct sample results affected by column injection losses, purging losses, or viscosity effects.

For CSSA projects, ISs are required for Methods SW8260 (Table 4.4.4-3) and SW8270 (Table 4.4.5-3). Specific ISs are listed in both methods. However, other compounds can be selected if the retention times (RTs) are similar to the compounds being detected by the GC/MS. If the ISs selected for these methods are different than the ones listed in the respective Tables in Section 4, a variance must be obtained for the use of the ISs. Most of the compounds of interest in the analytical methods should be between 0.8-1.2 relative to the retention time of one of the ISs.

Internal standards retention times and area counts must be checked for each calibration verification. If the retention times varied more than ± 30 seconds or the area counts either below 50% or above 100% of the established limits, samples should not be analyzed. Corrective action must be performed and the system must be brought back to control (see Tables 4.4.4-3 and 4.4.5-3).

The retention times and the area counts for the ISs for each sample should be reported in the CSSA FORM O-9. When the IS results are outside of the acceptance limits for retention times or the area counts, corrective actions shall be performed. After the system problems have been resolved and system control has been reestablished, all samples analyzed while the system was malfunctioning shall be reanalyzed. If corrective actions are not performed or are ineffective, the appropriate validation flag, as described in Tables 4.4.4-3 and 4.4.5-3, shall be applied to the sample results.

Guidelines for flagging sample data when corrective actions were ineffective or when holding times are exceeded for repeat analyses are specified below. These guidelines should be used, provided there were no other quality control problems in that analytical batch.

- When area count for an IS is higher than the high limit (> +100%), all results of analytes quantified using that IS are likely to be biased low. All positives associated with that IS should be qualified with a J flag. All non-detects associated with that IS should be flagged R.
- When an area count for an IS is lower than the low limit (< -50%), all results of analytes quantified using that IS are likely to be biased high. All positives associated with that IS should be flagged J. All non-detects associated with that IS should not be flagged.

4.3.7 Retention Time and Retention Time Windows

Retention time windows are used in GC and HPLC analysis for qualitative identification of analytes. Absolute retention time windows are calculated from replicate analyses of a standard on multiple days. The procedure and calculation method are given in SW846 Method 8000B.

For GC and HPLC methods, the daily retention times of each analyte in the method are checked from the calibration verification standards for that day or the analytical batch. If the daily retention time of an analyte falls within the established absolute retention time window, the daily window is calculated based on that day's retention time and using the +/- 3SD used in establishing the absolute retention time window.

When the daily retention time for an analyte is outside of the established absolute window, corrective action shall be performed. After the system problems have been resolved and system control has been reestablished, reanalyze all samples analyzed since the last acceptable retention time check. If corrective actions are not performed, the appropriate validation flag, as described in tables in Section 4.4, shall be applied to the sample results.

For GC/MS methods, the retention times of each analyte should be within 0.8-1.2 relative to one of the internal standards used for the quantitation of that analyte. The relative retention time (RRT) is established for each analyte based on the above rule. The RRT for each analyte is calculated for that day or for an analytical batch from the corresponding calibration verification standard and the RRT for each analyte should be ± 0.06 RRT units.

In addition, the retention times of the internal standards should be checked during and after data acquisition. The retention times of the internal standards should not be more than 30 seconds from the retention times of these standards from the mid-point calibration standard from the most recent initial calibration event.

4.3.8 Confirmation

Confirmation of analyte identification and concentration is required for GC and HPLC methods. Quantitative confirmation of results is required when concentrations are at or above the RL for samples analyzed by GC or HPLC, unless otherwise specified for the method in Section 4.0, and shall be completed within the method-required holding times. For GC methods, a second column is used for confirmation. For HPLC methods, a second column or a different detector is used. Confirmation column or detector must be calibrated and quality controlled like the primary column or detector. If one result is significantly higher (e.g., > 40%), the chromatograms should be evaluated to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, the baseline parameters established by the instrument data system (or operator) during peak integration should be evaluated. If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result. This approach is conservative relative to protection of the environment. The data user should be advised of the disparity between the results on the two columns. The case narrative must explain the actions taken and the rationale for selecting a result.

If holding times are exceeded and the analyses are performed, the results shall be flagged according to the procedures as described in Sections 4.0.

4.3.9 Stability of Calibration and Reagent Materials

The standards and chemicals used in the laboratory shall be within the manufacturer's expiration dates. Expired materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. In addition, the following expiration policy shall be followed: The expiration dates for solutions in ampules

shall not exceed the manufacturer's expiration date. Expiration dates for laboratory-prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions.

The laboratory shall label standards and QC materials with expiration dates.

4.3.10 Supplies and Consumables

The laboratory shall inspect supplies and consumables prior to their use in analysis. The materials description in the methods of analysis shall be used as a guideline for establishing the acceptance criteria for these materials. An inventory and storage system for these materials shall assure use before manufacturers' expiration dates and storage under safe and chemically compatible conditions.

4.4 Definitive Data Analytical Methods

A brief description and three tables for each method are included in the following subsections. The first table presents the RLs for each analyte in the method. The RLs are presented for both soil and water matrices. The second table presents the acceptance criteria for the accuracy of spiked analyte and surrogate recoveries. This table also presents the acceptance criteria for the precision of matrix and field duplicate recoveries. The third table presents the calibration and QC procedures for each method. Corrective actions and data flagging criteria are also included in this table.

In the third table, the first two columns designate the method number and the class of analytes that may be determined by the method. The third column lists the CSSA-required calibration and QC elements. The fourth column designates the minimum frequency for performing each calibration and QC element. The fifth column designates the acceptance criteria for each calibration and QC element. The sixth column designates the corrective action in the event that a calibration or QC element does not meet the acceptance criteria. The last column designates the data flagging criteria that shall be applied in the event that the method-required calibration and QC acceptance criteria are not met.

Table 4.4
List of Definitive Data Methods for CSSA

Method	Parameter
TX1005	Total Petroleum Hydrocarbons (C_6-C_{28}) by GC
SW8021 (modified) for soil gas	Aromatic and Halogenated Volatile Compounds in soil gas (Mobile Lab) by GC
SW8081	Organochlorine Pesticides by GC
SW8082	Polychlorinated Biphenyls by GC
SW8260	Volatile Organic Compounds by GC/MS
SW8270	Semivolatile Organic Compounds by GC/MS
SW8310	Polynuclear Aromatic Hydrocarbons by HPLC
SW8330	Explosive Residues by HPLC
SW6010	Trace Metals by ICP
SW6020	Trace Metals by ICP/MS
SW7060	Arsenic by GFAA
SW7131	Cadmium by GFAA
SW7191	Chromium by GFAA
SW7196	Chromium, Hexavalent by Colorimetric Method
SW7421	Lead by GFAA
SW7470	Mercury by Cold Vapor for Aqueous Samples
SW7471	Mercury by Cold Vapor for Soil Samples
E314.0	Perchlorate by IC

4.4.1 Total Petroleum Hydrocarbons by TNRCC Method 1005 (Revision 03)

This is a GC method designed to determine petroleum hydrocarbons from n-hexane (C_6) to n-pentatriacontane (C_{35}) in soil and water. This range includes gasoline range organics (GRO), kerosene, diesel range organics (DRO) and portions of the heavier fuel and lubricating oils. The analysis may be truncated at n-octacosane (nC_{28}) when the environmental medium of concern does not contain heavier hydrocarbon boiling range. The method uses flame ionization detector as the mode of detection, is used to separate two ranges, nC_6-nC_{12} and $>nC_{12}$ to nC_{28}, and a third range $>nC_{28}$ to nC_{35} when applicable.

TPH for each range C_6-$C_{10,}$ > C_{10}-$C_{28,}$ and $>C_{28}$ -C_{35} will be analyzed simultaneously but quantified separately, as part of the whole. TPHs are identified by their chromatographic fingerprint patterns. Other compounds (see Section 3.0 of the method) may alter the fingerprint pattern for identification and affect the quantitation of TPH. If a clear identification could not be made, discuss the issue with the prime contractor and CSSA. If the fingerprint is recognizable, but the quantitation is questionable, report the results with a J flag as estimated, and include an explanation in the case narrative.

Table 4.4.1-1
RLs for TPH by TNRCC Method 1005

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
TNRCC 1005	TPH	5.0	mg/L	50.0	mg/kg

Table 4.4.1-2
QC Acceptance Criteria for TNRCC Method 1005

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
TNRCC 1005	TPH	75-125	≤ 20	75-125	≤ 20
	Surrogates				
For C6-C12 Range, **choose one:** Trifluoromethylbenzene 1-chlorooctane		70-130		70-130	
For >C12 range, choose one: 1-chlorooctadecane, 2-fluorobiphenyl o-terphenyl		70-130		70-130	

Table 4.4.1-3
Summary of Calibration and QC Procedures for TNRCC Method 1005

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria[b]
TNRCC 1005	TPH	Five point initial calibration	Initial calibration prior to sample analysis	Linear regression: r >0.995 or Average response factor: %RSD ≤ 25%	Recalibrate	Apply R to all results associated with failed initial calibration
		Calibration verification	Daily before sample analysis and after every 10 samples	Within ± 25% of the expected value	Correct the problem and repeat initial calibration	Apply R to all specific fractions associated with the failed daily calibration
		Method blank	Once for every 20 samples	No TPH detected ≥ RL	Correct the problem. Reprep and analyze method blank and all the samples associated with the contaminated blank	Apply B to all positive results associated with the contaminated blank
		LCS	Once for every 20 samples	QC acceptance criteria in Table 4.4.1-2	Correct the problem. Reprep and analyze the LCS and all the samples associated with the failed LCS	If %R > UCL, apply J to all positive results. If %R < LCL, apply J to all positive results and apply R to all non-detects.
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.1-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.1-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.

Table 4.4.1-3 (Concluded)
Summary of Calibration and QC Procedures for TNRCC Method 1005

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria[b]
TNRCC 1005	TPH	MDL study	Once per 12 months	MDL should be ≤ 1/2 RL	Revalidate MDLs	Project samples should not be analyzed until the MDLs are validated
		Results reported between MDL and RL	None	None	None	None
		Retention time window check	Once per analytical batch	± 0.1 minute of the established retention time (see Section 9.3.4 of the method)	Correct problem, reestablish RT window and reanalyze all samples analyzed since the last retention time check	Apply R flag to each fraction if no corrective actions were done
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analysis of QC check samples	Once per analyst	QC acceptance criteria in Table 4.4.1-2	Recalculate the results. Correct the problem. Repeat four QC check samples	Apply R to all results for all samples analyzed by the analyst.

4.4.2 Current Method SW8081-Organochlorine Pesticides

Organochlorine pesticides in water and soil samples are analyzed using Method SW8081. This analytical method involves the extraction of the samples. The pesticides are then separated and quantified by GC using electron capture detection. Reporting limits for this method are presented in Table 4.4.2-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.2-2 and 4.4.2-3.

A second-column confirmation is not required for the analysis of toxaphene or chlordane.

Table 4.4.2-1
RLs for Method SW8081

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Organochlorine Pesticides SW8081	α-BHC	0.35	µg/L	0.019	mg/kg
	β-BHC	0.23	µg/L	0.033	mg/kg
	δ-BHC	0.24	µg/L	0.011	mg/kg
	γ-BHC (Lindane)	0.25	µg/L	0.020	mg/kg
	α-Chlordane	0.80	µg/L	0.015	mg/kg
	γ-Chlordane	0.37	µg/L	0.015	mg/kg
	4,4'-DDD	0.50	µg/L	0.042	mg/kg
	4,4'-DDE	0.58	µg/L	0.025	mg/kg
	4,4'-DDT	0.81	µg/L	0.036	mg/kg
	Aldrin	0.34	µg/L	0.022	mg/kg
	Dieldrin	0.44	µg/L	0.035	mg/kg
	Endosulfan I	0.30	µg/L	0.021	mg/kg
	Endosulfan II	0.40	µg/L	0.024	mg/kg
	Endosulfan Sulfate	0.35	µg/L	0.036	mg/kg
	Endrin	0.39	µg/L	0.036	mg/kg
	Endrin Aldehyde	0.50	µg/L	0.016	mg/kg
	Heptachlor	0.40	µg/L	0.020	mg/kg
	Heptachlor Epoxide	0.32	µg/L	0.021	mg/kg
	Methoxychlor	0.86	µg/L	0.057	mg/kg
	Toxaphene	0.50	µg/L	0.57	mg/kg

Table 4.4.2-2
QC Acceptance Criteria for Method SW8081

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW8081	α-BHC	75–125	≤ 30	65–135	≤ 50
	β-BHC	51–125	≤ 30	41–133	≤ 50
	δ-BHC	75–126	≤ 30	65–136	≤ 50
	γ-BHC (Lindane)	73–125	≤ 30	63–130	≤ 50
	α-Chlordane	41–125	≤ 30	31–135	≤ 50
	γ-Chlordane	41–125	≤ 30	31–133	≤ 50
	4,4-DDD	48–136	≤ 30	38–146	≤ 50
	4,4-DDE	45–139	≤ 30	35–149	≤ 50
	4,4-DDT	34–143	≤ 30	25–153	≤ 50
	Aldrin	47–125	≤ 30	37–126	≤ 50
	Dieldrin	42–132	≤ 30	32–142	≤ 50
	Endosulfan I	49–143	≤ 30	39–153	≤ 50
	Endosulfan II	75–159	≤ 30	65–169	≤ 50
	Endosulfan Sulfate	46–141	≤ 30	36–151	≤ 50
	Endrin	43–134	≤ 30	33–144	≤ 50
	Endrin Aldehyde	75–150	≤ 30	65–160	≤ 50
	Heptachlor	45–128	≤ 30	35–138	≤ 50
	Heptachlor Epoxide	53–134	≤ 30	43–144	≤ 50
	Methoxychlor	73–142	≤ 30	63–152	≤ 50
	Toxaphene	41–126	≤ 30	31–136	≤ 50
	Surrogates				
	DCBP	34–133		25–143	
	TCMX	45–125		35–135	

Table 4.4.2-3
Summary of Calibration and QC Procedures for Method SW8081

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8081	Organochlorine pesticides	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	Option 1 linear-:%RSD ≤ 20% for each analyte	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				Any analyte >20% RSD use linear option 2 or non-linear option 3 below for that analyte		
				Linear option 2: least square regression r> 0.995		
				Non-linear – COD ≥ 0.990 (6 points for second order, 7 points for third order)		
		Second source calibration verification	Once per five-point initial calibration	All analytes within ± 20% of the primary standard values	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window	Absolute RT windows established during method validation	± 3 SD for each analyte RT from a 72 hour study. See Section 7.6 of method SW8000B	Not applicable (N/A)	Not applicable
		Retention time check for each analyte	Beginning of each shift using calibration verification standard	RT for each analytes must fall within established absolute RT window	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis	All analytes within ± 15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration

Table 4.4.2-3 (Continued)
Summary of Calibration and QC Procedures for Method SW8081

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8081	Organochlorine pesticides	Calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within ± 15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification
		Breakdown check (Endrin and DDT)	Daily prior to analysis of samples and at the beginning of every 12 hour shift	Degradation ≤ 15%	Repeat breakdown check	If endrin breakdown fails apply J to all endrin, endrin ketone and endrin aldehyde results. If DDT breakdown fails, apply J to all DDT, DDE and DDD.
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.2-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte(s) in all samples in the associated analytical batch

Table 4.4.2-3 (Continued)
Summary of Calibration and QC Procedures for Method SW8081

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8081	Organochlorine pesticides	LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4.4.2-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4.4.2-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply R to all non-detects If any surrogate recovery is < 10%, apply R to all results
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.2-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.2-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.

Table 4.4.2-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW8081

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8081	Organochlorine pesticides	Confirmation (excluding toxaphene and chlordane)	All positive analytes from the first column	RPD 40%	None	Apply J to specific analytes if RPD >40%. See Subsection 4.3.8 of CSSA QAPP
		MDL study	Once per 12 month period	MDLs shall be ≤ ½ the RLs in Table 4.4.2-1	Revalidate MDLs	Project samples should not be analyzed until the MDLs are validated
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all the records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.3 Current Method SW8082-Polychlorinated Biphenyls

PCBs in water and soil samples are analyzed using Method SW8082. This analytical method involves the extraction of the samples. The PCBs are then separated and quantified by GC using electron capture detection or electrolytic conductivity detection. Reporting Limits for this method are presented in Table 4.4.3-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.3-2 and 4.4.3-3.

For analysis of PCBs, the initial five-point calibration and second source calibration verification shall contain PCBs 1016 and 1260. Retention times shall be verified for both PCBs during the initial five-point calibration. The daily calibration, initial calibration verification, and the calibration verification may be done using only a mixture of PCB-1016 and PCB-1260. Single standards of other five PCBs are required to aid the analyst in pattern recognition and to calculate response factors of the other five PCBs. If a PCB is present (i.e., above the MDL), report the result of the PCB using the response factors from the initial five-point calibration or from the appropriate response factors of the individual PCB. The LCS and MS may only be spiked with the 1016/1260 mix. A second-column confirmation is not required.

Table 4.4.3-1
RLs for Method SW8082

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
PCBs	PCB-1016	1.00	µg/L	0.70	mg/kg
	PCB-1221	1.00	µg/L	0.70	mg/kg
	PCB-1232	1.00	µg/L	0.70	mg/kg
	PCB-1242	1.00	µg/L	0.70	mg/kg
	PCB-1248	1.00	µg/L	0.70	mg/kg
	PCB-1254	1.00	µg/L	0.70	mg/kg
	PCB-1260	1.00	µg/L	0.70	mg/kg

Table 4.4.3-2
QC Acceptance Criteria for Method SW8082

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW8082	PCB-1016	54–125	≤ 30	44–127	≤ 50
	PCB-1221	41–126	≤ 30	31–136	≤ 50
	PCB-1232	41–126	≤ 30	31–136	≤ 50
	PCB-1242	39–150	≤ 30	29–160	≤ 50
	PCB-1248	41–126	≤ 30	31–136	≤ 50
	PCB-1254	29–131	≤ 30	25–141	≤ 50
	PCB-1260	41–126	≤ 30	31–136	≤ 50
	Surrogate				
	DCBP	34–133		25–143	

Table 4.4.3-3
Summary of Calibration and QC Procedures for Method SW8082

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8082	PCBs	Five-point initial calibration for PCBs 1016 and 1260	Initial calibration prior to sample analysis	Option 1 linear: %RSD ≤ 20% for each analyte	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				Any analyte > 20% RSD use linear option 2 or non linear option 3 for that analyte		
				Linear option 2: least square regression r > 0.995		
				Non-linear option 3: COD ≥ 0.990 (6 points for second order, 7 points for third order)		
		Second source calibration verification	Once per five-point initial calibration	Each PCB ± 20% of the primary standard value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window for PCB 1016/1260 mix	Each initial calibration and calibration verifications	± 3 times standard deviation for each quantitation peak retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification for PCB 1016/1260 mix	Daily, before sample analysis	All analytes within ± 15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration verification for PCB 1016/1260 mix	After every 20 samples and at the end of the analysis sequence	All analytes within ± 15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

Table 4.4.3-3 (Continued)
Summary of Calibration and QC Procedures for Method SW8082

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8082	PCBs	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.3-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte(s) in all samples in the associated analytical batch
		LCS (1016/1260 mix)	One LCS per analytical batch	QC acceptance criteria, Table 4.4.3-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4.4.3-2	Correct problem then re-extract and analyze sample	For the samples; if the %R > UCL for the surrogate apply J to all positive results if the %R < LCL for the surrogate, apply J to all positive results, apply R to all non-detects If the surrogate recovery is < 10%, apply R to all results

Table 4.4.3-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW8082

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8082	PCBs	MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.3-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.3-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		MDL study	See section 4.3.1 of the CSSA QAPP	MDLs shall be ≤ ½ the RLs in Table 4.4.3-1	Revalidate MDLs	Project samples should not be analyzed until the MDLs are validated
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.4 Current Method SW8260-Volatile Organics

Volatile (purgeable) organics in water and soil samples are analyzed using Method SW8260. This method uses a capillary column GC/MS technique. Volatile compounds are introduced into the GC by purge and trap (SW5030). An inert gas is bubbled through the water samples (or a soil-water slurry for soil samples) to transfer the purgeable organic compounds from the liquid to vapor phase. Soil samples with higher contaminant levels are extracted using methanol before purging. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is back flushed and heated to desorb the purgeable organics onto a capillary GC column where they are separated and then detected with a mass spectrometer. The analytes detected and RLs (using a 25 mL purge) for this method are listed in Table 4.4.4-1. The calibration, quality control and flagging requirements are listed in Tables 4.4.4-2 and 4.4.4-3.

The mass spectrometer is tuned every 12 hours to give an acceptable spectrum for Bromofluorobenzene (BFB). The tuning acceptance criteria are given in the following list as ion abundance for each specified mass:

- mass 50 15 percent to 40 percent of mass 95
- mass 75 30 percent to 60 percent of mass 95
- mass 95 base peak, 100 percent relative abundance
- mass 96 5 percent to 9 percent of mass 95
- mass 173 less than 2 percent of mass 174
- mass 174 greater than 50 percent of mass 95
- mass 175 5 percent to 9 percent of mass 174
- mass 176 greater than 95 percent, but less than 101 percent of mass 174
- mass 177 5 percent to 9 percent of mass 176

If a particular instrument tune fails, corrective action must be taken, and the retune must be in control before analyzing CSSA samples.

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.4-2 and 4.4.4-3.

Table 4.4.4-1
RLs for Method SW8260

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
VOCs SW8260	1,1,1,2-Tetrachloroethane	0.5	µg/L	0.003	mg/kg
	1,1,1-Trichloroethane	0.8	µg/L	0.004	mg/kg
	1,1,2,2-Tetrachloroethane (SPCC)	0.5	µg/L	0.0025	mg/kg
	1,1,2-Trichloroethane	1.0	µg/L	0.005	mg/kg
	1,1-Dichloroethane (SPCC)	0.4	µg/L	0.002	mg/kg
	1,1-Dichloroethene (CCC**)**	**1.2**	**µg/L**	0.006	mg/kg
	1,1-Dichloropropene	1.0	µg/L	0.005	mg/kg
	1,2,3-Trichlorobenzene	0.5	µg/L	0.004	mg/kg
	1,2,3-Trichloropropane	3.2	µg/L	0.02	mg/kg
	1,2,4-Trichlorobenzene	0.5	µg/L	0.004	mg/kg
	1,2,4-Trimethylbenzene	1.3	µg/L	0.007	mg/kg
	1,2-Dichloroethane	0.6	µg/L	0.003	mg/kg
	1,2-Dichlorobenazene	0.3	µg/L	0.002	mg/kg
	1,2-Dibromo-3-chloropropane	2.6	µg/L	0.01	mg/kg
	1,2-Dichloropropane (CCC)	0.4	µg/L	0.002	mg/kg
	1,2-Ethylene dibromide	0.6	µg/L	0.003	mg/kg
	1,3,5-Trimethylbenzene	0.5	µg/L	0.003	mg/kg
	1,3-Dichlorobenzene	1.2	µg/L	0.006	mg/kg
	1,3-Dichloropropane	0.4	µg/L	0.002	mg/kg
	1,4-Dichlorobenzene	0.3	µg/L	0.002	mg/kg
	1-Chlorohexane	0.6	µg/L	0.003	mg/kg
	2,2-Dichloropropane	3.5	µg/L	0.02	mg/kg
	2-Chlorotoluene	0.4	µg/L	0.002	mg/kg
	4-Chlorotoluene	0.6	µg/L	0.003	mg/kg
	Benzene	0.4	µg/L	0.002	mg/kg
	Bromobenzene	0.3	µg/L	0.002	mg/kg
	Bromochloromethane	0.4	µg/L	0.002	mg/kg
	Bromodichloromethane	**0.8**	**µg/L**	0.004	mg/kg
	Bromoform (SPCC)	1.2	µg/L	0.006	mg/kg
	Bromomethane	1.1	µg/L	0.005	mg/kg
	Carbon tetrachloride	2.1	µg/L	0.01	mg/kg
	Chlorobenzene (SPCC)	0.4	µg/L	0.002	mg/kg
	Chloroethane	1.0	µg/L	0.005	mg/kg
	Chloroform (CCC)	**0.3**	**µg/L**	0.002	mg/kg
	Chloromethane (SPCC)	1.3	µg/L	0.007	mg/kg
	Cis-1,2-Dichloroethene	**1.2**	**µg/L**	0.006	mg/kg
	Cis-1,3-Dichloropropene	1.0	µg/L	0.005	mg/kg
	Dibromochloromethane	**0.5**	**µg/L**	0.003	mg/kg
	Dibromomethane	2.4	µg/L	0.01	mg/kg
	Dichlorodifluoromethane	1.0	µg/L	0.005	mg/kg
	Ethylbenzene (CCC)	0.6	µg/L	0.003	mg/kg
	Hexachlorobutadiene	1.1	µg/L	0.005	mg/kg
	Isopropylbenzene	0.5	µg/L	0.008	mg/kg
	m & p-Xylene	1.3	µg/L	0.007	mg/kg
	Methylene chloride	**2.0**	**µg/L**	0.005	mg/kg
	n-Butylbenzene	1.1	µg/L	0.005	mg/kg

Table 4.4.4-1 (Concluded)
RLs for Method SW8260

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
VOCs SW8260	n-Propylbenzene	0.4	µg/L	0.002	mg/kg
	Naphthalene	1.0	µg/L	0.02	mg/kg
	o-Xylene	1.1	µg/L	0.005	mg/kg
	p-Isopropyltoluene	1.2	µg/L	0.006	mg/kg
	Sec-Butylbenzene	1.3	µg/L	0.007	mg/kg
	Styrene	0.5	µg/L	0.0025	mg/kg
	Trichloroethene	**1.0**	**µg/L**	0.01	mg/kg
	Tert-Butylbenzene	1.4	µg/L	0.007	mg/kg
	Tetrachloroethene	**1.4**	**µg/L**	0.007	mg/kg
	Toluene (CCC)	1.1	µg/L	0.005	mg/kg
	Trans-1,2-Dichloroethene	**0.6**	**µg/L**	0.003	mg/kg
	Trans-1,3-Dichloropropene	1.0	µg/L	0.005	mg/kg
	Trichlorofluoromethane	0.8	µg/L	0.004	mg/kg
	Vinyl chloride (CCC)	**1.1**	**µg/L**	0.009	mg/kg

The reporting limits listed in the above table have been approved by TNRCC.

The analytes in the above table that are in **bold** have been approved by EPA and TNRCC as the reduced list of target analytes for groundwater analysis at CSSA under the EPA 3008(h) order.

Table 4.4.4-2
QC Acceptance Criteria for Current Method SW8260

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)	Assoc. IS
SW8260	1,1,1,2-Tetrachloroethane	72–125	≤ 20	62–108	≤ 30	2
	1,1,1-Trichloroethane	75–125	≤ 20	65–135	≤ 30	1
	1,1,2,2-Tetrachloroethane	74–125	≤ 20	62-125	≤ 30	3
	1,1,2-Trichloroethene	75–127	≤ 20	65–135	≤ 30	1
	1,1-Dichloroethane	72–125	≤ 20	62–135	≤ 30	1
	1,1-Dichloroethene	70-130	≤ 20	65–135	≤ 30	1
	1,1-Dichloropropene	75–125	≤ 20	65–135	≤ 30	1
	1,2,3-Trichlorobenzene	75–137	≤ 20	65–147	≤ 30	3
	1,2,3-Trichloropropane	75–125	≤ 20	65–135	≤ 30	3
	1,2,4-Trichlorobenzene	75–135	≤ 20	65–145	≤ 30	3
	1,2,4-Trimethylbenzene	75-125	≤ 20	65–135	≤ 30	3
	1,2-Dichloroethane	68–127	≤ 20	58–137	≤ 30	1
	1,2-Dichlorobenzene	75-125	≤ 20	65–135	≤ 30	3
	1,2-Dibromo-3-chloropropane	59–125	≤ 20	49–135	≤ 30	3
	1,2-Dichloropropane	70–125	≤ 20	60–135	≤ 30	1
	1,2-Ethylene dibromide	75-125	≤ 20	65–135	≤ 30	2
	1,3,5-Trimethylbenzene	72-125	≤ 20	62–135	≤ 30	3
	1,3-Dichlorobenzene	75-125	≤ 20	65–135	≤ 30	3
	1,3-Dichloropropane	75-125	≤ 20	65–135	≤ 30	2
	1,4-Dichlorobenzene	75-125	≤ 20	65–135	≤ 30	3
	1-Chlorohexane	75-125	≤ 20	65–135	≤ 30	2
	2,2-Dichloropropane	75-125	≤ 20	65–135	≤ 30	1
	2-Chlorotoluene	73–125	≤ 20	63–135	≤ 30	3
	4-Chlorotoluene	74–125	≤ 20	64–135	≤ 30	3
	Benzene	75–125	≤ 20	65–135	≤ 30	1
	Bromobenzene	75–125	≤ 20	65–135	≤ 30	3
	Bromochloromethane	73–125	≤ 20	63–135	≤ 30	1
	Bromodichloromethane	75–125	≤ 20	65–135	≤ 30	1
	Bromoform	75–125	≤ 20	65–135	≤ 30	2
	Bromomethane	65–125	≤ 20	62–135	≤ 30	1
	Carbon Tetrachloride	62–125	≤ 20	52–135	≤ 30	1
	Chlorobenzene	75–125	≤ 20	65–135	≤ 30	2
	Chloroethane	65–125	≤ 20	55–135	≤ 30	1
	Chloroform	74–125	≤ 20	64–135	≤ 30	1
	Chloromethane	65-125	≤ 20	65–135	≤ 30	1
	Cis-1,2-Dichloroethene	75–125	≤ 20	65–135	≤ 30	1
	Cis-1,3-Dichloropropene	74–125	≤ 20	64–135	≤ 30	1
	Dibromochloromethane	73–125	≤ 20	63–135	≤ 30	2
	Dibromomethane	69–127	≤ 20	59–137	≤ 30	1
	Dichlorodifluoromethane	50-150	≤ 20	50-150	≤ 30	1
	Ethylbenzene	75-125	≤ 20	65–135	≤ 30	2
	Hexachlorobutadiene	75–125	≤ 20	65–135	≤ 30	3
	Isopropylbenzene	75–125	≤ 20	65–135	≤ 30	3
	M, & p-Xylenes	75–125	≤ 20	65–135	≤ 30	2
	Methylene chloride	75–125	≤ 20	65–135	≤ 30	1

Table 4.4.4-2 (Concluded)
QC Acceptance Criteria for Current Method SW8260

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)	Assoc. IS
SW8260	n-Butylbenzene	75–125	≤ 20	65–135	≤ 30	3
	n-Propylbenzene	75–125	≤ 20	65–135	≤ 30	3
	Naphthalene	75–125	≤ 20	65–135	≤ 30	3
	o-Xylene	75–125	≤ 20	65–135	≤ 30	2
	p-Isopropyltoluene	75–125	≤ 20	65–135	≤ 30	3
	Sec-Butylbenzene	75–125	≤ 20	65–135	≤ 30	3
	Styrene	75–125	≤ 20	65–135	≤ 30	2
	Trichloroethene	71–125	≤ 20	61–135	≤ 30	1
	Tert-butylbenzene	75-125	≤ 20	65–135	≤ 30	3
	Tetrachloroethene	71–125	≤ 20	61–135	≤ 30	2
	Toluene	74–125	≤ 20	64–135	≤ 30	1
	Trans-1,2-Dichloroethene	75–125	≤ 20	65–135	≤ 30	1
	Trans-1,3-Dichloropropene	66–125	≤ 20	56–135	≤ 30	1
	Trichlorofluoromethane	67–125	≤ 20	57–135	≤ 30	1
	Vinyl Chloride	46–134	≤ 20	36–144	≤ 30	1
	Surrogates					
	Dibromofluoromethane	75-125		65–135		
	Toluene-D8	75–125		65–135		
	4-Bromofluorobenzene	75–125		65–135		
	1,2-Dichloroethane-D4	62–139		52–149		
	Internal Standards					
	Fluorobenzene	See Table 4.4.3-3 And Section4.3.6				1
	Chlorobenzene-D5					2
	1,4-Dichlorobenzene-D4 (DCB)					3

Table 4.4.4-3
Summary of Calibration and QC Procedures for Current Method SW8260

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8260	Volatile Organics	Five-point initial calibration for all analytes of concern	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.30[c] and %RSD for RFs for CCCs ≤ 30% and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				Option 1 linear- %RSD ≤ 15% for each analyte		Apply R to all results for specific analyte(s) for all samples associated with out of control calibration
				Any analyte >15% RSD use linear option 2 or non-linear option 3 below for that analyte	See section 7.5.1.1 of SW8000B	
				Option 2 linear – least squares regression r > 0.995		
				Option 3 non-linear – COD ≥ 0.990 (6 points for second order, 7 points for third order)		
		Second source calibration verification	Once per five-point initial calibration	All analytes within ± 25% of the primary standard values. Approved variances: 1,1-DCE, bromomethane and chloromethane within ± 30%, and dichlorodifluoro methane within ± 50%	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration

Table 4.4.4-3 (Continued)
Summary of Calibration and QC Procedures for Current Method SW8260

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8260	Volatile Organics	Relative retention time	RRT established during method validation	Each analyte should be within 0.8-1.2 relative to one of the internal standards. See Subsection 7.3.2.2 of SW8260B	Correct the problem	Not applicable
		Retention time for each analyte	For each shift from the calibration verification standard	RRT of the analyte within ± 0.06 RRT units of the established RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.30[c]; and CCCs ≤ 20% difference (when using RFs) or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within ± 20% of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.4-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst

Table 4.4.4-3 (Continued)
Summary of Calibration and QC Procedures for Current Method SW8260

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8260	Volatile Organics	Internal standards check	In each calibration verification and in each sample	Retention time ±30 seconds from retention time of the mid-point std. In the ICAL. EICP area within – 50% to + 100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	See Section 4.3.6 Area count >+100%, apply J for positives and R for non-detects. <-50%, apply J for positives only.
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte(s) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4.4.4-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte(s) in all samples in the associated analytical batch: if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.4-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if: (1) %R for MS > UCL; or (2) %R for MS < LCL

Table 4.4.4-3 (Concluded)
Summary of Calibration and QC Procedures for Current Method SW8260

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8260	Volatile Organics	MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.4-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4.4.4-2 **Medium-level soil samples will not be reextracted for the failure of one surrogate. The sample extract will be reinjected (approved variance).**	Correct problem then reextract and analyze sample	For the samples: if the %R > UCL for a surrogate, apply J to all positive results if the %R < LCL for a surrogate, apply J to all positive results; apply R to all non-detect results If any surrogate recovery is < 10%, apply R to all results
		MDL study	See section 4.3.1 of the CSSA QAPP	MDLs shall be ≤ ½ the RLs in Table 4.4.4-1	Revalidate MDLs	Project samples should not be analyzed until the MDLs are validated
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all the records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.
[c]Except > 0.10 for bromoform, and > 0.10 for chloromethane and 1,1-dichloroethane (DCA)

4.4.5 Current Method SW8270-Semivolatile Organics

Semivolatile organics (also known as base/neutral and acid extractables) in water and soil samples are analyzed using Method SW8270. This technique determines quantitatively the concentration of a number of SVOCs. Samples are extracted and both base/neutral and acid extracts are then concentrated through evaporation. Compounds of interest are separated and quantified using a capillary column GC/mass spectrometer. The RLs are listed in Table 4.4.5-1.

The mass spectrometer is tuned every 12 hours to give an acceptable spectrum for decafluorotriphenylphosphine (DFTPP). The tuning acceptance criteria are given in the following list as ion abundance for each specified mass:

- mass 51 30 percent to 60 percent of mass 198
- mass 68 less than 2 percent of mass 69
- mass 70 less than 2 percent of mass 69
- mass 127 40 percent to 60 percent of mass 198
- mass 197 less than 1 percent of mass 198
- mass 198 base peak, 100 percent relative abundance
- mass 199 5 percent to 9 percent of mass 198
- mass 275 10 percent to 30 percent of mass 198
- mass 365 greater than 1 percent of mass 198
- mass 441 present, but less than mass 443
- mass 442 greater than 40 percent of mass 198
- mass 443 17 percent to 23 percent of mass 442

If a particular instrument tune fails, corrective action must be taken, and the retune must be in control before analyzing CSSA samples.

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.5-2 and 4.4.5-3.

Table 4.4.5-1
RLs for Method SW8270

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Semivolatile Organics Base/Neutral Extractables SW8270	1,2,4-Trichlorobenzene	10.0	µg/L	0.7	mg/kg
	1,2-DCB	10.0	µg/L	0.7	mg/kg
	1,3-DCB	10.0	µg/L	0.7	mg/kg
	1,4-DCB (CCC)	10.0	µg/L	0.7	mg/kg
	2,4-DNT	10.0	µg/L	0.7	mg/kg
	2,6-DNT	10.0	µg/L	0.7	mg/kg
	2-Chloronaphthalene	10.0	µg/L	0.7	mg/kg
	2-Methylnaphthalene	10.0	µg/L	0.7	mg/kg
	2-Nitroaniline	50.0	µg/L	3.3	mg/kg
	3-Nitroaniline	50.0	µg/L	3.3	mg/kg
	3,3'-Dichlorobenzidine	20.0	µg/L	1.3	mg/kg
	4-Bromophenyl phenyl ether	10.0	µg/L	0.7	mg/kg
	4-Chloroaniline	20.0	µg/L	1.3	mg/kg
	4-Chlorophenyl phenyl ether	10.0	µg/L	0.7	mg/kg
	4-Nitroaniline	50.0	µg/L	3.3	mg/kg
	Acenaphthylene	10.0	µg/L	0.7	mg/kg
	Acenaphthene (CCC)	10.0	µg/L	0.7	mg/kg
	Anthracene	10.0	µg/L	0.7	mg/kg
	Benz (a) anthracene	10.0	µg/L	0.7	mg/kg
	Benzo (a) pyrene (CCC)	10.0	µg/L	0.7	mg/kg
	Benzo (b) fluoranthene	10.0	µg/L	0.7	mg/kg
	Benzo (g,h,i) perylene	10.0	µg/L	0.7	mg/kg
	Benzyl alcohol	20.0	µg/L	1.3	mg/kg
	Bis (2-chloroethoxy) methane	10.0	µg/L	0.7	mg/kg
	Bis (2-chloroethyl) ether	10.0	µg/L	0.7	mg/kg
	Bis (2-chloroisopropyl) ether	10.0	µg/L	0.7	mg/kg
	Bis (2-ethylhexyl) phthalate	10.0	µg/L	0.7	mg/kg
	Butyl benzylphthalate	10.0	µg/L	0.7	mg/kg
	Chrysene	10.0	µg/L	0.7	mg/kg
	Di-n-butylphthalate	10.0	µg/L	0.7	mg/kg
	Di-n-octylphthalate (CCC)	10.0	µg/L	0.7	mg/kg
	Dibenz (a,h) anthracene	10.0	µg/L	0.7	mg/kg
	Dibenzofuran	10.0	µg/L	0.7	mg/kg
	Diethyl phthalate	10.0	µg/L	0.7	mg/kg
	Dimethyl phthalate	10.0	µg/L	0.7	mg/kg
	Fluoranthene (CCC)	10.0	µg/L	0.7	mg/kg
	Fluorene	10.0	µg/L	0.7	mg/kg
	Hexachlorobenzene	10.0	µg/L	0.7	mg/kg
	Hexachlorobutadiene (CCC)	10.0	µg/L	0.7	mg/kg
	Hexachlorocyclopentadiene (SPCC)	10.0	µg/L	0.7	mg/kg
	Hexachloroethane	10.0	µg/L	0.7	mg/kg
	Indeno (1,2,3-c,d) pyrene	10.0	µg/L	0.7	mg/kg
	Isophorone	10.0	µg/L	0.7	mg/kg
	N-Nitrosodiphenylamine	10.0	µg/L	0.7	mg/kg

Table 4.4.5-1 (Concluded)
RLs for Method SW8270

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Semivolatile Organics Base/Neutral Extractables SW8270	n-Nitrosodi-n-propylamine (SPCC)	10.0	µg/L	0.7	mg/kg
	Naphthalene	10.0	µg/L	0.7	mg/kg
	Nitrobenzene	10.0	µg/L	0.7	mg/kg
	Phenanthrene	10.0	µg/L	0.7	mg/kg
	Pyrene	10.0	µg/L	0.7	mg/kg
Semivolatile Organics Acid Extractables SW8270	2,4,5-Trichlorophenol	50.0	µg/L	3.3	mg/kg
	2,4,6-Trichlorophenol (CCC)	10.0	µg/L	0.3	mg/kg
	2,4-Dichlorophenol (CCC)	10.0	µg/L	0.3	mg/kg
	2,4-Dimethylphenol	10.0	µg/L	0.3	mg/kg
	2,4-Dinitrophenol (SPCC)	50.0	µg/L	3.3	mg/kg
	2-Chlorophenol	10.0	µg/L	0.3	mg/kg
	2-Methylphenol	10.0	µg/L	0.3	mg/kg
	2-Nitrophenol (CCC)	10.0	µg/L	0.3	mg/kg
	4,6-Dinitro-2-methylphenol	50.0	µg/L	3.3	mg/kg
	4-Chloro-3-methylphenol (CCC)	20.0	µg/L	1.3	mg/kg
	4-Methylphenol	10.0	µg/L	0.3	mg/kg
	4-Nitrophenol (SPCC)	50.0	µg/L	1.6	mg/kg
	Benzoic acid	50.0	µg/L	1.6	mg/kg
	Pentachlorophenol (CCC)	50.0	µg/L	3.3	mg/kg
	Phenol (CCC)	10.0	µg/L	0.3	mg/kg

Table 4.4.5-2
QC Acceptance Criteria for Current Method SW8270

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)	Assoc. IS	Assoc. Sur.
SW8270	1,2,4-Trichlorobenzene	44–142	≤ 20	34–152	≤ 30	2	4
	1,2-DCB	42–155	≤ 20	32–135	≤ 30	1	3
	1,3-DCB	36–125	≤ 20	26–135	≤ 30	1	3
	1,4-DCB	30–125	≤ 20	25–135	≤ 30	1	3
	2,4-DNT	39–139	≤ 20	29–149	≤ 30	3	4
	2,6-DNT	51–125	≤ 20	41–135	≤ 30	3	4
	2-Chloronaphthalene	60–125	≤ 20	50–135	≤ 30	3	4
	2-Methylnaphthalene	41–125	≤ 20	31–135	≤ 30	2	5
	2-Nitroaniline	50–125	≤ 20	40–135	≤ 30	3	2
	3,3'-Dichlorobenzidine	29–175	≤ 20	25–175	≤ 30	5	6
	3-Nitroaniline	51–125	≤ 20	41–135	≤ 30	3	2
	4-Bromophenyl phenyl ether	53–127	≤ 20	43–137	≤ 30	4	1
	4-Chloroaniline	45-136	≤ 20	35-146	≤ 30	2	5
	4-Chlorophenyl phenyl ether	51–132	≤ 20	41–142	≤ 30	3	4
	4-Nitroaniline	40–143	≤ 20	30–153	≤ 30	3	2
	Acenaphthylene	47–125	≤ 20	37–135	≤ 30	3	4
	Acenaphthene	49–125	≤ 20	39–135	≤ 30	3	4
	Anthracene	45–165	≤ 20	35–175	≤ 30	4	1
	Benz (a) anthracene	51–133	≤ 20	41–143	≤ 30	5	6
	Benzo (a) pyrene	41–125	≤ 20	31–135	≤ 30	6	6
	Benzo (b) fluoranthene	37–125	≤ 20	27–135	≤ 30	6	6
	Benzo (g,h,i) perylene	34–149	≤ 20	25–159	≤ 30	6	6
	Benzyl alcohol	35–125	≤ 20	25–135	≤ 30	1	3
	Bis (2-chloroethoxy) methane	49–125	≤ 20	39–135	≤ 30	2	5
	Bis (2-chloroethyl) ether	44–125	≤ 20	34–135	≤ 30	1	3
	Bis (2-chloroisopropyl) ether	36–166	≤ 20	26–175	≤ 30	1	3
	Bis (2-ethylhexyl) phthalate	33–129	≤ 20	25–139	≤ 30	5	6
	Butyl benzyl phthalate	26–125	≤ 20	25–135	≤ 30	5	6
	Chrysene	55–133	≤ 20	45–143	≤ 30	5	6
	Di-n-butyl phthalate	34–126	≤ 20	25–136	≤ 30	4	1
	Di-n-octyl phthalate	38–127	≤ 20	28–137	≤ 30	5	6
	Dibenz (a,h) anthracene	50–125	≤ 20	40–135	≤ 30	6	6
	Dibenzofuran	52–125	≤ 20	42–135	≤ 30	3	4
	Diethyl phthalate	37–125	≤ 20	27–135	≤ 30	3	4
	Dimethyl phthalate	25–175	≤ 20	25–175	≤ 30	3	4
	Fluoranthene	47–125	≤ 20	37–135	≤ 30	4	1
	Fluorene	48–139	≤ 20	38–149	≤ 30	3	2
	Hexachlorobenzene	46–133	≤ 20	36–143	≤ 30	4	1
	Hexachlorobutadiene	25–125	≤ 20	25–135	≤ 30	2	5
	Hexachlorocyclopentadiene	41–125	≤ 20	31–135	≤ 30	3	2
	Hexachloroethane	25–153	≤ 20	25–163	≤ 30	1	3
	Indeno (1,2,3-c,d) pyrene	27–160	≤ 20	25–170	≤ 30	5	6
	Isophorone	26–175	≤ 20	25–175	≤ 30	2	5
	n-Nitrosodi-n-propylamine	37–125	≤ 20	27–135	≤ 30	1	3
	n-Nitrosodiphenylamine	27–125	≤ 20	25–135	≤ 30	4	1
	Naphthalene	50–125	≤ 20	40–135	≤ 30	2	5

Table 4.4.5-2 (Concluded)
QC Acceptance Criteria for Current Method SW8270

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)	Assoc. IS	Assoc. Sur.
SW8270	Nitrobenzene	46–133	≤ 20	36–143	≤ 30	2	4
	Phenanthrene	54–125	≤ 20	44–135	≤ 30	4	1
	Pyrene	47–136	≤ 20	37–146	≤ 30	5	6
	2,4,5-Trichlorophenol	25–175	≤ 20	25–175	≤ 30	3	1
	2,4,6-Trichlorophenol	39–128	≤ 20	29–138	≤ 30	3	1
	2,4-Dichlorophenol	46–125	≤ 20	36–135	≤ 30	2	5
	2,4-Dimethylphenol	45–139	≤ 20	35–149	≤ 30	2	5
	2,4-Dinitrophenol	30–151	≤ 20	25–161	≤ 30	3	4
	2-Chlorophenol	41–125	≤ 20	31–135	≤ 30	1	3
	2-Methylphenol	25–125	≤ 20	25–135	≤ 30	1	3
	2-Nitrophenol	44–125	≤ 20	34–135	≤ 30	2	4
	4,6-Dinitro-2-Methyl Phenol	26–134	≤ 20	25–144	≤ 30	4	1
	4-Chloro-3-Methyl Phenol	44–125	≤ 20	34–135	≤ 30	2	5
	4-Methylphenol	33–125	≤ 20	25–135	≤ 30	1	3
	4-Nitrophenol	25–131	≤ 20	25–141	≤ 30	3	2
	Benzoic Acid	25–162	≤ 20	25–172	≤ 30	2	5
	Pentachlorophenol	28–136	≤ 20	38–146	≤ 30	4	1
	Phenol	25–125	≤ 20	25–135	≤ 30	1	5
SW8270	**Surrogates**						
	2,4,6-Tribromophenol	25–134		25–144			1
	2-Fluorobiphenyl	43–125		34–135			2
	2-Fluorophenol	25–125		25–135			3
	Nitrobenzene-D5	32–125		25–135			4
	Phenol-D6	25–125		25–135			5
	Terphenyl-D14	42–126		32–136			6
	Internal Standards						
	1,4-Dichlorobenzene-D4	See Table 4.4.5-3					1
	Naphthalene-D8						2
	Acenaphthalene-D10	And					3
	Phenanthrene-D10						4
	Chrysene-D12	Section4.3.6					5
	Perylene-D12						6

Table 4.4.5-3
Summary of Calibration and QC Procedures for Current Method SW8270

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8270	Semivolatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
				Option 1 linear- %RSD ≤ 15% for each analyte		
				Any analyte >15% RSD use linear option 2 or non-linear option 3 below for that analyte	See section 7.5.1.1 of SW8000B	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				Option 2 linear – least squares regression r > 0.995		
				Option 3 non-linear – COD ≥ 0.990 (6 points for second order, 7 points for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ± 25% of the primary standard values	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample

Table 4.4.5-3 (Continued)
Summary of Calibration and QC Procedures for Current Method SW8270

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8270		Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050; and CCCs ≤ 20% difference (when using RFs) or drift (when using least squares regression or non-linear calibration) 3,3-dichlorobenzidine ≤35% (approved variance)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within ± 20% of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration verification
	Semivolatile Organics	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.5-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Internal standards check	In each calibration verification and in each sample	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL. EICP area within - 50% to + 100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	See Section 4.3.6 Area count >+100%, apply J for positives and R for non-detects. <-50%, apply J for positives only
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte(s) in all samples in the associated analytical batch

Table 4.4.5-3 (Continued)
Summary of Calibration and QC Procedures for Current Method SW8270

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8270		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4.4.5-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte(s) in all samples in the associated analytical batch; If the LCS %R > UCL, apply J to all positive results If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
	Volatile Organics	MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.5-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.5-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.

Table 4.4.5-3 (Concluded)
Summary of Calibration and QC Procedures for Current Method SW8270

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8270		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4.4.5-2	Correct problem then re-extract and analyze sample	For the samples; if the %R > UCL for a surrogate, apply J to all positive results of analytes associated with the surrogate If the %R < LCL for a surrogate, apply J to all positive results of analytes associated with the surrogate, apply R to all non-detect results of analytes associated with the surrogate If any surrogate recovery is < 10%, apply R to all results of analytes associated with the surrogate
		MDL study	See section 4.3.1 of the CSSA QAPP	Detection limits established shall be ≤ ½ the RLs in Table 4.4.5-1	Revalidate MDLs	Project samples should not be analyzed until the MDLs are validated
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.6 Current Method SW8310–Polynuclear Aromatic Hydrocarbons

Method SW8310 is used to determine the concentration of parts per billion (ppb) levels of selected polynuclear aromatic hydrocarbons in groundwater and soils by HPLC. Samples are extracted then analyzed by direct injection. Detection is by ultraviolet and fluorescent detectors. RLs are listed in Table 4.4.6-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.6-2 and 4.4.6-3.

Table 4.4.6-1
RLs for Current Method SW8310

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Polynuclear Aromatic Hydrocarbons SW8310	Acenaphthene	18.0	µg/L	1.2	mg/kg
	Acenaphthylene	23.0	µg/L	1.54	mg/kg
	Anthracene	6.6	µg/L	0.44	mg/kg
	Benzo (a) anthracene	0.13	µg/L	0.009	mg/kg
	Benzo (a) pyrene	0.23	µg/L	0.015	mg/kg
	Benzo (b) fluoranthene	0.18	µg/L	0.012	mg/kg
	Benzo (g,h,i) perylene	0.76	µg/L	0.05	mg/kg
	Benzo (k) fluoranthene	0.17	µg/L	0.011	mg/kg
	Chrysene	1.5	µg/L	0.1	mg/kg
	Dibenzo (a,h) anthracene	0.3	µg/L	0.02	mg/kg
	Fluoranthrene	2.1	µg/L	0.14	mg/kg
	Fluorene	2.1	µg/L	0.14	mg/kg
	Indeno (1,2,3-c,d) pyrene	0.43	µg/L	0.03	mg/kg
	Naphthalene	18.0	µg/L	1.2	mg/kg
	Phenanthrene	6.4	µg/L	0.42	mg/kg
	Pyrene	2.7	µg/L	0.18	mg/kg

Table 4.4.6-2
QC Acceptance Criteria for Current Method SW8310

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW8310	Acenaphthene	43–130	≤ 30	33–140	≤ 50
	Acenaphthylene	49–125	≤ 30	39–135	≤ 50
	Anthracene	54–125	≤ 30	44–135	≤ 50
	Benzo (a) Anthracene	39–135	≤ 30	29–145	≤ 50
	Benzo (a) Pyrene	52–125	≤ 30	42–135	≤ 50
	Benzo (b) Fluoranthene	31–137	≤ 30	25–147	≤ 50
	Benzo (g,h,i) Perylene	53–125	≤ 30	43–135	≤ 50
	Benzo (k) Fluoranthene	60–129	≤ 30	50–139	≤ 50
	Chrysene	59–134	≤ 30	49–144	≤ 50
	Dibenzo (a,h) Anthracene	51–125	≤ 30	41–135	≤ 50
	Fluoranthene	42–125	≤ 30	32–135	≤ 50
	Fluorene	53–125	≤ 30	43–135	≤ 50
	Indeno (1,2,3-c,d) Pyrene	55–125	≤ 30	45–135	≤ 50
	Naphthalene	43–125	≤ 30	33–135	≤ 50
	Phenathrene	52–129	≤ 30	42–139	≤ 50
	Pyrene	55–125	≤ 30	45–135	≤ 50
	Surrogates				
	Terphenyl-D14 Decachlorobiphenyl Triphenylene (Choose one surrogate. Provide historical control limits for AFCEE/CSSA approval.)				

Table 4.4.6-3
Summary of Calibration and QC Procedures for Current Method SW8310

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8310	PAHs	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	Linear :RSD for each analyte ≤ 20%	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				Any analyte >20% RSD, use linear option 2 or non-linear option 3 below for that analyte		
				linear – least squares regression r > 0.995		
				non-linear – COD ≥ 0.990 (6 points for second order, 7 points for third order)		
		Second source calibration verification	Once per five-point initial calibration	All analytes within ± 20% of the primary standard values	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis	All analytes within ± 15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration

Table 4.4.6-3 (Continued)
Summary of Calibration and QC Procedures for Current Method SW8310

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8310	PAHs	Calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within ± 15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.6-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte(s) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4.4.6-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte(s) in all samples in the associated analytical batch; If the LCS %R > UCL, apply J to all positive results If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

Table 4.4.6-3 (Continued)
Summary of Calibration and QC Procedures for Current Method SW8310

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8310	PAHs	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4.4.6-2	Correct problem then reextract and analyze sample	For the samples; If the %R > UCL for any surrogate, Apply J to all positive results If the %R < LCL for any surrogate, Apply J to all positive results, Apply R to all non-detects If any Surrogate recovery is < 10%, apply R to all results
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.6-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.6-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		Confirmation[C]	All positive analytes	RPD 40%	None	Apply J to specific analytes if RPD >40% See Subsection 4.3.8 of the CSSA QAPP
		MDL study	See section 4.3.1 of the CSSA QAPP	MDLs shall be ≤ ½ the RLs in Table 4.4.6-1	Revalidate MDLs	Project samples should not be analyzed until the MDLs are validated

Table 4.4.6-3 (Concluded)
Summary of Calibration and QC Procedures for Current Method SW8310

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all the records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.
[c]Use a second column or different detector.

4.4.7 Current Method SW8330–Explosive Residues

Current Method SW8330 provides HPLC conditions for the detection of ppb levels of certain explosive residues in a water, soil, and sediment matrix. Prior to using this method, appropriate sample preparation techniques must be used.

Aqueous samples of low concentrations are extracted by a salting-out procedure with acetonitrile and sodium chloride. An aliquot of the extract is separated on a C-18 reverse-phase column, determined at 254 nanometers (nm), and confirmed on a cyanide reverse-phase column.

Aqueous samples of higher concentration can be diluted, filtered, separated on a C-18 reverse-phase column, determined at 254 nm, and confirmed on a cyanide reverse-phase column.

Soil and sediment samples are extracted in an ultrasonic bath and filtered before chromatography, separated on a C-18 reverse-phase column and confirmed on a cyanide reverse phase column as in aqueous samples.

RLs are listed in Table 4.4.7-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.7-2 and 4.4.7-3.

Table 4.4.7-1
RLs for Current Method SW8330

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Explosive Residues SW8330	1,3,5- trinitrobenzene (TNB)	7.3	µg/L	0.25	mg/kg
	1,3- DNB	4.0	µg/L	0.25	mg/kg
	2,4,6- trinitrotoluene (TNT)	6.9	µg/L	0.25	mg/kg
	2,4-DNT	5.7	µg/L	**0.5**	mg/kg
	2,6-DNT	9.4	µg/L	0.26	mg/kg
	HMX	13.0	µg/L	2.2	mg/kg
	m-Nitrotoluene	7.9	µg/L	**0.6**	mg/kg
	Methyl-2,4,6-trinitrophenylnitramine	44.0	µg/L	0.65	mg/kg
	Nitrobenzene	7.0	µg/L	0.26	mg/kg
	o-Nitrotoluene	12.0	µg/L	0.25	mg/kg
	p-Nitrotoluene	8.5	µg/L	**0.5**	mg/kg
	RDX	14.0	µg/L	1.0	mg/kg

Table 4.4.7-2
QC Acceptance Criteria for Current Method SW8330

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW8330	1,3,5-TNB	75-142	≤30	65-152	≤50
	1,3-DNB	75-125	≤30	65-135	≤50
	2,4,6-TNT	75-128	≤30	65-138	≤50
	2,4-DNT	75-125	≤30	65-135	≤50
	2,6-DNT	75-129	≤30	65-139	≤50
	HMX	74-137	≤30	64-147	≤50
	m-Nitrotoluene	60-134	≤30	50-144	≤50
	Methyl 2,4,6-Trinitrophenylnitramine	44-142	≤30	34-152	≤50
	Nitrobenzene	29-134	≤30	25-144	≤50
	o-Nitrotoluene	75-129	≤30	65-139	≤50
	p-Nitrotoluene	42-150	≤30	32-160	≤50
	RDX	75-132	≤30	65-142	≤50
	Surrogates[a] [a] Choose an analyte and its LCS limit from the method that is not expected to be present in the sample as the surrogate.				

Table 4.4.7-3
Summary of Calibration and QC Procedures for Current Method SW8330

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8330	Explosives	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	Linear: RSD ≤ 20% for each analyte.	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
				Any analyte > 20% RSD use linear option 2 or non linear option 3 for that analyte		
				linear – least squares regression r > 0.995		
				non-linear – COD ≥ 0.990 (6 points for second order, 7 points for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ± 20% of the primary standard values	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis	All analytes within ± 15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration

Table 4.4.7-3 (Continued)
Summary of Calibration and QC Procedures for Current Method SW8330

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8330	Explosives	Calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within ± 15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.7-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte(s) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4.4.7-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte(s) in all samples in the associated analytical batch; If the LCS %R > UCL, apply J to all positive results If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

Table 4.4.7-3 (Continued)
Summary of Calibration and QC Procedures for Current Method SW8330

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW8330	Explosives	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4.4.7-2	Correct problem then reextract and analyze sample	For the samples; If the %R > UCL for any surrogate, Apply J to all positive results If the %R < LCL for any surrogate, Apply J to all positive results, Apply R to all non-detects. If any Surrogate recovery is < 10%, apply R to all results
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.7-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.7-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		Confirmation[C]	All positive analytes	RPD 40%	None	Apply J to specific analytes if RPD>40%. See Subsection 4.3.8 of the CSSA QAPP
		MDL study	See section 4.3.1 of the CSSA QAPP	MDLs shall be ≤ ½ the RLs in Table 4.4.7-1	Revalidate MDLs	Project samples should not be analyzed until the MDLs are validated

Table 4.4.7-3 (Concluded)
Summary of Calibration and QC Procedures for Current Method SW8330

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all the records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.
[c]Use a second column or different detector.

4.4.8 Current Method SW6010-Trace Elements (Metals) by Inductively Coupled Plasma-Atomic Emission Spectroscopy for Water and Soil

Samples are analyzed for trace elements or metals using Method SW6010 for water and soils. Analysis for most metals requires digestion of the sample. Following digestion, the trace elements are determined simultaneously or sequentially using Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES). The elements and corresponding RLs for this method are listed in Table 4.4.8-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.8-2 and 4.4.8-3.

Table 4.4.8-1
RLs for Current Method SW6010

Parameter/Method	Analyte	Water		Soil	
		RL	**Unit**	**RL**	**Unit**
ICP Metals SW6010	Aluminum	0.2	mg/L	22.0	mg/kg
	Antimony	0.05	mg/L	10.0	mg/kg
	Arsenic	0.03	mg/L	40.0	mg/kg
	Barium	**0.005**	**mg/L**	1.0	mg/kg
	Beryllium	0.005	mg/L	1.0	mg/kg
	Cadmium	0.007	mg/L	0.50	mg/kg
	Calcium	1.1	mg/L	100	mg/kg
	Chromium	**0.01**	**mg/L**	20	mg/kg
	Cobalt	0.006	mg/L	10.0	mg/kg
	Copper	**0.01**	**mg/L**	2.0	mg/kg
	Iron	0.20	mg/L	3.0	mg/kg
	Lead	0.025	mg/L	10.0	mg/kg
	Magnesium	0.10	mg/L	100	mg/kg
	Manganese	**0.005**	**mg/L**	2.0	mg/kg
	Molybdenum	0.015	mg/L	3.0	mg/kg
	Nickel	**0.01**	**mg/L**	2.0	mg/kg
	Potassium	**1.00**	**mg/L**	600	mg/kg
	Selenium	0.03	mg/L	3.0	mg/kg
	Silver	0.01	mg/L	1.0	mg/kg
	Sodium	1.0	mg/L	10.0	mg/kg
	Thallium	0.08	mg/L	6.0	mg/kg
	Vanadium	0.01	mg/L	1.0	mg/kg
	Zinc	**0.05**	**mg/L**	**5.0**	**mg/kg**

Table 4.4.8-2
QC Acceptance Criteria for Current Method SW6010

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW6010	Aluminum	75-125	≤ 20	75-125	≤ 20
	Antimony	75-125	≤ 20	75-125	≤ 20
	Arsenic	75-125	≤ 20	75-125	≤ 20
	Barium	75-125	≤ 20	75-125	≤ 20
	Beryllium	75-125	≤ 20	75-125	≤ 20
	Cadmium	75-125	≤ 20	75-125	≤ 20
	Calcium	75-125	≤ 20	75-125	≤ 20
	Chromium	75-125	≤ 20	75-125	≤ 20
	Cobalt	75-125	≤ 20	75-125	≤ 20
	Copper	75-125	≤ 20	75-125	≤ 20
	Iron	75-125	≤ 20	75-125	≤ 20
	Lead	75-125	≤ 20	75-125	≤ 20
	Magnesium	75-125	≤ 20	75-125	≤ 20
	Manganese	75-125	≤ 20	75-125	≤ 20
	Molybdenum	75-125	≤ 20	75-125	≤ 20
	Nickel	75-125	≤ 20	75-125	≤ 20
	Potassium	75-125	≤ 20	75-125	≤ 20
	Selenium	75-125	≤ 20	75-125	≤ 20
	Silver	75-125	≤ 20	75-125	≤ 20
	Sodium	75-125	≤ 20	75-125	≤ 20
	Thallium	75-125	≤ 20	75-125	≤ 20
	Vanadium	75-125	≤ 20	75-125	≤ 20
	Zinc	75-125	≤ 20	75-125	≤ 20

Table 4.4.8-3
Summary of Calibration and QC Procedures for Current Method SW6010

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6010	ICP Metals	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	N/A	N/A	Apply R to all results for specific analyte(s) for all samples associated with the calibration if calibration not done
		RL check standard	After each initial calibration	70-130%	Reestablish reporting limit for specific metals	Contact the prime contractor. (See section 9.0 for resolution.)
		Initial calibration verification (second source)	Daily after initial calibration	All analytes within ± 10% of the primary standard values	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration blank	After every calibration verification	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples	Apply B to all positive results for specific analyte(s) in all samples associated with the blank
		Calibration verification (Instrument Check Standard)	After every 10 samples and at the end of the analysis sequence	All analyte(s) within ± 10% of expected value and RSD of replicate integrations < 5%	Repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.8-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte(s) in all samples in the associated analytical batch

Table 4.4.8-3 (Continued)
Summary of Calibration and QC Procedures for Current Method SW6010

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6010	ICP Metals	Interference check solution (ICS)	At the beginning of an analytical batch	Within ± 20% of expected value	Terminate analysis; correct problem; reanalyze ICS; reanalyze all affected samples	Apply R to all results for specific analyte(s) in all samples associated with the ICS
		LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 4.4.8-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte(s) in all samples in the associated analytical batch; If the LCS %R > UCL, apply J to all positive results If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution test See Subsection 5.2.8 of CSSA QAPP	Each analytical batch	1+4 dilution must agree within ± 10% of the original determination	See Subsection 5.2.8 of CSSA QAPP	See Subsection 5.2.8 of CSSA QAPP
		Post digestion spike	See Subsection 5.2.8 of CSSA QAPP	Recovery within 75-125% of expected results	Correct problem then reanalyze post digestion spike addition	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition If post digestion spike addition recovery is < 10%, apply R to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition

Table 4.4.8-3 (Concluded)
Summary of Calibration and QC Procedures for Current Method SW6010

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6010	ICP Metals	MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.8-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.8-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		MDL study	See section 4.3.1 of the CSSA QAPP	MDLs shall be ≤ ½ the RLs in Table 4.4.8-1	Revalidate MDLs	Project samples should not be analyzed until the MDLs are validated
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all the records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.9 Current Method SW6020-Trace Elements (Metals) by Inductively Coupled Plasma Mass Spectrometry for Water and Soil

Samples are analyzed for trace elements or metals using Method SW6020 for water and soils. Analysis for total (i.e., acid leachable) metals requires digestion of the sample. Following digestion, the trace elements are determined simultaneously or sequentially using Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS). The elements and RLs for this method are listed in Table 4.4.9-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.9-2 and 4.4.9-3.

Table 4.4.9-1
RLs for Method SW6020

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
ICP Metals SW6020	Aluminum	0.02	mg/L	2.0	mg/kg
	Antimony	0.001	mg/L	0.10	mg/kg
	Arsenic	0.02	mg/L	2.0	mg/kg
	Barium	0.003	mg/L	0.30	mg/kg
	Beryllium	0.003	mg/L	0.30	mg/kg
	Cadmium	0.002	mg/L	0.20	mg/kg
	Chromium	0.004	mg/L	0.40	mg/kg
	Cobalt	0.0008	mg/L	0.08	mg/kg
	Copper	0.006	mg/L	0.60	mg/kg
	Lead	0.002	mg/L	0.20	mg/kg
	Manganese	0.002	mg/L	0.20	mg/kg
	Nickel	0.002	mg/L	0.20	mg/kg
	Silver	0.002	mg/L	0.20	mg/kg
	Thallium	0.0002	mg/L	0.02	mg/kg
	Zinc	0.025	mg/L	2.5	mg/kg

Table 4.4.9-2
QC Acceptance Criteria for Method SW6020

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW6020	Aluminum	80–120	≤ 15	80–120	≤ 25
	Antimony	80–120	≤ 15	80–120	≤ 25
	Arsenic	80–120	≤ 15	80–120	≤ 25
	Barium	80–120	≤ 15	80–120	≤ 25
	Beryllium	80–120	≤ 15	80–120	≤ 25
	Cadmium	80–120	≤ 15	80–120	≤ 25
	Chromium	80–120	≤ 15	80–120	≤ 25
	Cobalt	80–120	≤ 15	80–120	≤ 25
	Copper	80–120	≤ 15	80–120	≤ 25
	Lead	80–120	≤ 15	80–120	≤ 25
	Manganese	80–120	≤ 15	80–120	≤ 25
	Nickel	80–120	≤ 15	80–120	≤ 25
	Silver	80–120	≤ 15	80–120	≤ 25
	Thallium	80–120	≤ 15	80–120	≤ 25
	Zinc	80–120	≤ 15	80–120	≤ 25

Table 4.4.9-3
Summary of Calibration and QC Procedures for Method SW6020

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6020	ICP/MS Metals	MS tuning sample	Prior to initial calibration and calibration verification	See section 5.8 of method SW6020	Retune instrument then reanalyze tuning solution	Apply R to all results for all analytes for all samples associated with the MS tuning
		Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	N/A	N/A	Apply R to all results for specific analyte(s) for all samples associated with the calibration if calibration not done
		RL check standard	After each initial calibration	70-130%	Reestablish reporting limits for specific metals	Contact the prime contractor. (See section 9.0 for resolution.)
		Calibration blank	Before beginning a sample run, after every 10 samples and at end of the analysis sequence	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples	Apply B to all positive results for specific analyte(s) in all samples associated with the blank
		Calibration verification (second source)	Before beginning a sample run, after every 10 samples and at the end of the analysis sequence	All analyte(s) within ± 10% of the primary standard values	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4 9-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst

Table 4.4.9-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW6020

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6020	ICP/MS Metals	Method blank	One per analytical batch	No analyte detected ≥ RL	Correct problem reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte(s) in all samples in the associated analytical batch
		Interference check solutions (ICS-A and ICS-AB)	At the beginning and end of an analytical run or once per 12-hour period, whichever is more frequent.	ICS-A All non-spiked analytes < RL ICS-AB Within ± 20% of true value	Terminate analysis; locate and correct problem; reanalyze ICS; reanalyze all affected samples	Apply R to all results for specific analyte(s) in all samples associated with the ICS
		LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 4.4.9-2	Correct problem reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution test See Subsection 5.2.8 of CSSA QAPP	Each analytical batch	1+4 dilution must agree within ± 10% of the original determination	See Subsection 5.2.8 of CSSA QAPP	See Subsection 5.2.8 of CSSA QAPP
		Post digestion spike addition	See Subsection 5.2.8 of CSSA QAPP	Recovery within 75-125% of expected results	Dilute the sample; reanalyze post digestion spike addition	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition

Table 4.4.9-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW6020

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW6020	ICP/MS Metals	MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.9-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL or
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.9-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		MDL study	Every three months	MDLs shall be ≤ ½ the RLs in Table 4.4.9-1	Revalidate MDLs	Project samples should not be analyzed until the MDLs are validated
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.10 Method SW7060–Arsenic by GFAA

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in micro liter amounts. An electric current heats the graphite tube. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the arsenic. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analysis are listed in Table 4.4.10-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.10-2 and 4.4.10-3.

Table 4.4.10-1
RLs for Method SW7060

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7060	**Arsenic**	0.005	mg/L	0.5	mg/kg

Table 4.4.10-2
QC Acceptance Criteria for Method SW7060

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW7060	Arsenic	74-120	≤ 15	74-120	≤ 25

Table 4.4.10-3
Summary of Calibration and QC Procedures for Method SW7060

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7060	Arsenic	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration verification	Once per initial daily multipoint calibration	Analyte within ± 10% of the primary standard value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected ≥ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all positive results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within ± 20% of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.10-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte in all samples in the associated analytical batch

Table 4.4.10-3 (Continued)
Summary of Calibration and QC Procedures for Method SW7060

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7060	Arsenic	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 4.4.10-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte in all samples in the associated analytical batch; If the LCS %R > UCL, apply J to all positive results If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution test See Subsection 5.2.8 of CSSA QAPP	Each analytical batch	1+4 dilution sample result must be ± 10% of the undiluted sample result	See Subsection 5.2.8 of CSSA QAPP	See Subsection 5.2.8 of CSSA QAPP
		Recovery test See Subsection 5.2.8 of CSSA QAPP	See Subsection 5.2.8 of CSSA QAPP	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.10-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL

Table 4.4.10-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW7060

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7060	Arsenic	MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.11-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		MDL study	See section 4.3.1 of the CSSA QAPP	MDL should be ≤ ½ RL in table 4.4.10-1	Revalidate MDLs	Project samples should not be analyzed until MDLs are validated
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all the records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.11 Method SW7131– Cadmium by GFAA

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. An electrical current heats the graphite tube resistively. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the Cadmium. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analyzes are listed in Table 4.4.11-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.11-2 and 4.4.11-3.

Table 4.4.11-1
RLs for Method SW7131

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7131	**Cadmium**	**0.001**	**mg/L**	0.1	mg/kg

Table 4.4.11-2
QC Acceptance Criteria for Method SW7131

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW7131	Cadmium	80-122	≤ 15	80-122	≤ 25

Table 4.4.11-3
Summary of Calibration and QC Procedures for Method SW7131

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7131	Cadmium	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second source calibration verification	Once per initial daily multipoint calibration	Analyte within ± 10% of the primary standard value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected ≥ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all positive results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within ± 20% of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.12-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte in all samples in the associated analytical batch

Table 4.4.11-3 (Continued)
Summary of Calibration and QC Procedures for Method SW7131

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7131	Cadmium	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 4.4.11-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte in all samples in the associated analytical batch; If the LCS %R > UCL, apply J to all positive results If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution test See Subsection 5.2.8 of CSSA QAPP	Each analytical batch	1+4 dilution sample result must be ± 10% of the undiluted sample result	See Subsection 5.2.8 of CSSA QAPP	See Subsection 5.2.8 of CSSA QAPP
		Recovery test See Subsection 5.2.8 of CSSA QAPP	See Subsection 5.2.8 of CSSA QAPP	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.11-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.11-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		MDL study	See section 4.3.1 of the CSSA QAPP	MDL should be ≤ 1/2 RL in Table 4.4.11-1	Revalidate MDLs	Project samples should not be analyzed until MDLs are validated

Table 4.4.11-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW7131

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all the records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.12 Method SW7191– Chromium by GFAA

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the Chromium. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analyzes are listed in Table 4.4.12-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.12-2 and 4.4.12-3.

Table 4.4.12-1
RLs for Method SW7191

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7191	Chromium	0.005	mg/L	0.5	mg/kg

Table 4.4.12-2
QC Acceptance Criteria for Method SW7191

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW7191	Chromium	80-121	≤ 15	80-121	≤ 15

Table 4.4.12-3
Summary of Calibration and QC Procedures for Method SW7191

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7191	Chromium	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration verification	Once per initial daily multipoint calibration	Analyte within ± 10% of the primary value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected ≥ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all positive results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within ± 20% of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.13-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte in all samples in the associated analytical batch

Table 4.4.12-3 (Continued)
Summary of Calibration and QC Procedures for Method SW7191

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7191	Chromium	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 4.4.12-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution test See Subsection 5.2.8 of CSSA QAPP	Each analytical batch	1+4 dilution sample result must be ± 10% of the undiluted sample result	See Subsection 5.2.8 of CSSA QAPP	See Subsection 5.2.8 of CSSA QAPP
		Recovery test See Subsection 5.2.8 of CSSA QAPP	See Subsection 5.2.8 of CSSA QAPP	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.12-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL

Table 4.4.12-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW7191

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7191	Chromium	MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.12-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		MDL study	See section 4.3.1 of the CSSA QAPP	MDLs shall be ≤ ½ the RLs in Table 4.4.12-1	Revalidate MDLs	Project samples should not be analyzed until the MDLs are validated
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.13 Method SW7196-Hexavalent Chromium (Colorimetric)

Dissolved hexavalent chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, may be determined colorimetrically. RLs for this method are listed in Table 4.4.13-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.13-2 and 4.4.13-3.

Table 4.4.13-1
RLs for Method SW7196

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7196	Hexavalent Chromium	0.5	mg/L	1.0	mg/kg

Table 4.4.13-2
QC Acceptance Criteria for Method SW7196

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW7196	Hexavalent Chromium	86–117	≤ 15	86–117	≤ 25

Table 4.4.13-3
Summary of Calibration and QC Procedures for Method SW7196

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7196	Hexavalent Chromium	Multipoint calibration curve (minimum three standards and a blank)	Initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to the specific analyte result for all samples associated with the calibration
		Second-source calibration verification	After each new stock standard preparation	Analyte within ± 10% of the primary standard value	Correct problem then repeat initial calibration	Apply R to the specific analyte result for all samples associated with the calibration
		Calibration verification	After every 15 samples and at the end of the analysis sequence	Chromium within ± 20% of expected value	Correct problem then repeat initial calibration and reanalyze all samples since last successful calibration	Apply R to the specific analyte result in all samples since the last acceptable calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.13-2	Recalculate results; locate and fix problem with system and then rerun demonstration	Apply R to the specific analyte result for all samples analyzed by the analyst
		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 reanalyze sample persistent interference indicates the need to use and alternate method	Apply R to the specific analyte result for all samples analyzed since the last acceptable verification check
		MDL study	See section 4.3.1 of the CSSA QAPP	MDLs shall be ≤ ½ the RLs in Table 4.4.13-1	None	Apply R to all specific analyte results for all samples analyzed

Table 4.4.14-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW7196

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7196	Chromium	Method blank	One per analytical batch	No analyte detected > RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to the specific analyte result for all samples in the associated analytical batch
		LCS	One LCS per analytical batch	QC acceptance criteria, Table 4.4.13-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.14-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.13-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.

[a]All corrective actions associated with CSSA project work shall be documented, and all records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.14 Method SW7421– Lead by GFAA

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted as appropriate. Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. An electrical current heats the graphite tube resistively. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the Lead. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. RLs for this analysis are listed in Table 4.4.14-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.14-2 and 4.4.14-3.

Table 4.4.14-1
RLs for Method SW7421

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7421	**Lead**	**0.005**	mg/L	0.5	mg/kg

Table 4.4.14-2
QC Acceptance Criteria for Method SW7421

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW7421	Lead	74-124	≤ 15	74-124	≤ 25

Table 4.4.14-3
Summary of Calibration and QC Procedures for Method SW7421

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7421	Lead	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration verification	Once per initial daily multipoint calibration	Analyte within ± 10% of the primary standard value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected ≥ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all positive results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within ± 20% of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.14-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte in all samples in the associated analytical batch

Table 4.4.14-3 (Continued)
Summary of Calibration and QC Procedures for Method SW7421

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7421	Lead	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 4.4.14-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte in all samples in the associated analytical batch; If the LCS %R > UCL, apply J to all positive results If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Dilution test See Subsection 5.2.8 of CSSA QAPP	Each analytical batch	1+4 dilution sample result must be ± 10% of the undiluted sample result	See Subsection 5.2.8 of CSSA QAPP	See Subsection 5.2.8 of CSSA QAPP
		Recovery test See Subsection 5.2.8 of CSSA QAPP	See Subsection 5.2.8 of CSSA QAPP	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.14-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL

Table 4.4.14-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW7421

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7421	Lead	MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.14-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		MDL study	See section 4.3.1 of the CSSA QAPP	MDLs shall be ≤ ½ the RLs in Table 4.4.14-1	None	Apply R to all specific analyte results for all samples analyzed
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all the records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.15 Method SW7470/SW7471–Mercury by Manual Cold-Vapor Technique

Water and soil samples are analyzed for mercury using Methods SW7470 and SW7471, respectively. This method is a cold-vapor, flameless atomic absorption (AA) technique based on the absorption of radiation by mercury vapor. Mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an AA spectrophotometer. Mercury concentration is measured as a function of absorbance. The RLs for these methods are listed in Table 4.4.15-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.15-2 and 4.4.15-3.

Table 4.4.15-1
RLs for Method SW7470/SW7471

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7470 (W) SW7471 (S)	**Mercury**	**0.001**	**mg/L**	0.1	mg/kg

Table 4.4.15-2
QC Acceptance Criteria for Method SW7470/SW7471

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW7470/SW7471	Mercury	77–120	≤ 15	77–120	≤ 25

Table 4.4.15-3
Summary of Calibration and QC Procedures for Method SW7470/SW7471

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7470 SW7471	Mercury	Initial multipoint calibration (minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second source calibration verification	Once per initial daily multipoint calibration	Analyte within ± 10% of the primary standard value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected ≥ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all positive results for the specific analyte in all samples associated with the blank
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within ± 20% of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.15-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all positive results for the specific analyte in all samples in the associated analytical batch

Table 4.4.15-3 (Continued)
Summary of Calibration and QC Procedures for Method SW7470/SW7471

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7470 SW7471	Mercury	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 4.4.15-2	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA analytical batch	For specific analyte in all samples in the associated analytical batch; If the LCS %R > UCL, apply J to all positive results If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.4.15-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL

Table 4.4.15-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW7470/SW7471

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW7470 SW7471	Mercury	MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.16-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		MDL study	See section 4.3.1 of the CSSA QAPP	MDLs shall be ≤ ½ the RLs in Table 4.4.15-1	None	Apply R to all specific analyte results for all samples analyzed
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with CSSA project work shall be documented, and all the records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.4.16 Method 314.0–Perchlorate Anion

This method addresses the determination of the perchlorate anion in water samples, as well as in aqueous extracts of soil samples using ion chromatography.

A large (approximately 1.0 mL) volume of sample is introduced into an ion chromatograph. Perchlorate is separated and measured using a system comprised of an ion chromatographic pump, sample injection valve, guard column, analytical column, suppressor device, and conductivity detector.

The method requires the use of a conductivity detector to monitor sample matrix conductivity and to determine if sample pretreatment is required. Pretreatment must be performed whenever the conductivity exceeds the laboratory determined Matrix Conductivity Threshold (MCT) and can consist of dilution and/or use of specific pretreatment cartridges or columns designed to remove matrix interferences. The MCT is the matrix conductance where the calculated Area to Height (A/H) ratio percent difference ($PD_{A/H}$) for the perchlorate peak exceeds 20%.

An analytical batch is a sequence of samples, which are analyzed within a 30-hour period and include no more than 20 field samples. An analytical batch must also include all required QC samples, which do not contribute to the maximum field sample total of 20. The required QC samples include:

- Instrument Performance Check (IPC) Standard
- Method Blank
- Second Source Verification
- Laboratory Control Sample
- Continuing Calibration Verification
- Matrix Spike
- Matrix Duplicate

RLs are listed in Table 4.4.16-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.4.16-2 and 4.4.16-3.

Table 4.4.16-1
RLs for Method 314.0

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
314.0	Perchlorate	4.0	µg/L	50	µg/kg

Table 4.4.16-2
QC Acceptance Criteria for Method 314.0

Method	Sample	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
314.0	Perchlorate	80 -120	≤ 15	70 - 130	≤ 30

Table 4.4.16-3
Summary of Calibration and QC Procedures for Method 314.0

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
314.0	Perchlorate	3-points for one order of magnitude and 5 points for 2 orders of magnitude	Initial calibration prior to sample analysis	*option 1 linear*- RSD ≤15% *option 2 linear* – least squares regression r > 0.995	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
		Second-source calibration verification	Once per multipoint calibration, upon reestablishing calibration, quarterly	Instrument response within ± 10% of expected value	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
		Instrument Performance Check	Daily, before sample analysis	Conductance within 10% of original value (original value within ± 10% of MCT)	Prepare fresh IPC solution	Apply R to all results for the sample
				$PD_{A/H}$ < 25%, instrument response within ± 20% of expected response	Redetermine MCT or correct problem and reanalyze IPC	
				Retention time shifts < 5%, or overall retention time < 80% of original recorded value	Correct problem, clean or replace column	
		Initial calibration verification	Daily, before sample analysis or when eluent is changed	Instrument response within ± 25% of expected value using a standard at or below the RL	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration

Table 4.4.16.3-3 (Continued)
Summary of Calibration and QC Procedures for Method 314.0

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
314.0	Perchlorate	Calibration verification	After every 10 samples and at the end of the analysis sequence	Instrument response within ± 15% of expected response, alternately using separate mid and high level standards	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results in all samples since the last acceptable calibration verification
		Method blank	One per analytical batch	Perchlorate must be ≤ ½ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results in all samples in the associated analytical batch
		Pretreated laboratory reagent blank	Required in any analytical batch which includes samples that have been pretreated to reduce the common anion levels	Perchlorate must be ≤ ½ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results in all samples in the associated analytical batch
		Laboratory Control Sample	One LCS per analytical batch following the ICCS	Instrument response within ± 15% of expected response	Correct problem then reprep and analyze the LCS and all samples in the affected CSSA batch	For all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Matrix Spike	One per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 4.4.16-2	none	For all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.4.16-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.

Table 4.4.16-3 (Concluded)
Summary of Calibration and QC Procedures for Method 314.0

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
314.0	Perchlorate	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.4.16-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		MCT determination	At initial set-up, once per 12 month period	Calculate $PD_{A/H}$ for the perchlorate peak at increasing concentrations 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, ($r^2 > 0.95$) *option 2* – Use the conductance level of the highest mixed anion solution which yielded a $PD_{A/H}$ value < 20%	Samples cannot be analyzed without a valid MCT
		RL verification	At initial set-up, once per 12 month period	Instrument response within ± 30% of expected response for a mixed common anion solution containing perchlorate at the RL and conductance within ± 10% of the MCT	Lower the MCT by 10% and repeat the RL verification	Samples cannot be analyzed without a valid RL verification
		MDL study	At initial set-up, and See section 4.3.1 of the CSSA QAPP	Detection limits established shall be ≤ ½ the RLs in Table 4.4.16-1	none	Apply R to all results for in all samples analyzed
		Results reported between MDL and RL	None	None	None	None

[a]All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not

4.5 Description of TPDES Required Analytical Methods

This section describes the analytical methods needed for TPDES permit application and for monitoring effluents in outfall 001 and 002 to satisfy TPDES complying requirements. A short description of the analytical method, and calibration and quality control requirements are presented below.

4.5.1 Biological Oxygen Demand (BOD)

The method consists of filling with sample, to overflowing, an airtight bottle and incubating it at a specified temperature for five days. Dissolved oxygen is measured initially and after incubation. BOD is computed from the difference between the initial, and final dissolved oxygen.

If grab samples are analyzed within two hours of collection, cold storage is unnecessary. Keep samples refrigerated or in an iced cooler (temperature 2-6° C) if analysis will be performed after two hours. For TPDES, permit-reporting samples must be analyzed within six hours of collection.

4.5.2 Total Suspended Solids (non-filterable residue) by Method EPA 160.2

A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to a constant weight at 103-105 ^{o} C.

Method	Analyte	Water	
		RL	Unit
E160.2	Total suspended solids	5	mg/L

4.5.3 pH by Method EPA 150.1

The pH of a sample is determined electrometrically using a combination electrode.

Table 4.5.3
Summary of Calibration and Quality Control Requirements

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
EPA 150.1	pH (water)	2-point calibration with pH buffers that bracket the expected sample pH	Once per day at the beginning of testing.	± 0.05 pH unit on repeat measurement of the calibration buffers	Check with new buffers; if still out, repair meter; repeat calibration check

4.6 Description of Analytical Methods Required for Monitoring Natural Attenuation

At CSSA, the primary focus is to monitor natural attenuation of chlorinated solvents in groundwater. Analytical methods needed to monitor natural attenuation are listed in this Table 4.6.1. However, not all listed methods may be necessary for each round of sampling. The methods required for each sampling event will be included in the DQOs. Many of the individual method descriptions and required calibration and quality control information have been presented in earlier sections of this CSSA QAPP.

Table 4.6
Analytical Methods for Monitoring Natural Attenuation

Analytical Parameter	Method of Analysis	Field/Fixed Base Laboratory	Comments
Alkalinity	Hach method 8221	Field, on–post laboratory	See Subsection 4.1.10
Arsenic	SW6010	Fixed base	To determine if anaerobic biological activity is solubilizing arsenic from the aquifer material. See Subsection 4.4.8
Conductivity	E120.1/SW9050, direct reading meter	Field	See Subsection 4.1.4
Dissolved Oxygen	DO Meter EPA 360.1	Field	Concentrations less than 1 mg/L generally indicate anaerobic pathway. See Subsection 4.1.11
Nitrate	SW9056	Fixed Base	See Subsection 4.6.2
Nitrite	SW9056	Fixed Base	See Subsection 4.6.2
Dissolved Ferrous Iron	Colorimetric (Hach #8146)	Field, on-post laboratory	
Sulfate	Colorimetric (Hach #8051)	Fixed Base	See Subsection 4.6.2
Hydrogen Sulfide	Hach method 8131	Field, on-post laboratory	

Table 4.6 (Concluded)
Analytical Methods for Monitoring Natural Attenuation

Analytical Parameter	Method of Analysis	Field/Fixed Base Laboratory	Comments
Dissolved Methane, Ethane, Ethene, Carbon dioxide	RSKSOP-175	Fixed Base	GC method for head space analysis of dissolved gases. Ethane and ethene data are used to evaluate biological transformation of chlorinated solvents.
pH	E150.1/SW9040	Field	See Subsection 4.1.3
Redox Potential	ASTM D1498		See Subsection 4.1.12
Chlorinated Hydrocarbons	SW8260	Fixed Base	See Subsection 4.4.3
Temperature	E170.1	Field	See Subsection 4.1.8
Total Organic Carbon	SW9060	Fixed Base	Used to classify plume and to determine if reductive dechlorination is possible in the absence of anthropogenic carbon

4.6.1 Method RSK-175 –Soil Gases (Volatile Organics) in Water

Soil gases in water are sampled and analyzed using method RSK-175. This method uses a high resolution GC coupled to one or more appropriate detectors (CSSA requires the use of a mass-selective detector). The analytes detected and RLs for this method are listed in Table 4.6.1-1.

Calibration—The mass spectrometer is tuned daily to give an acceptable spectrum for BFB. The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 50 15 percent to 40 percent of mass 95
- mass 75 30 percent to 60 percent of mass 95
- mass 95 base peak, 100 percent relative abundance
- mass 96 5 percent to 9 percent of mass 95
- mass 173 less than 2 percent of mass 174
- mass 174 greater than 50 percent of mass 95
- mass 175 5 percent to 9 percent of mass 174
- mass 176 greater than 95 percent, but less than 101 percent of mass 174
- mass 177 5 percent to 9 percent of mass 176

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.6.1-2 and 4.6.1-3.

Table 4.6.1-1
RLs for Method RSK-175

Analyte	Water	
	RL	Unit
Methane	5	µg/L
Ethane	5	µg/L
Ethene	5	µg/L

Table 4.6.1-2
QC Acceptance Criteria for Method RSK-175

Analyte	Accuracy Water (% R)	Precision Water (% RPD)
Methane	60-120	≤ 20
Ethane	65-115	≤ 20
Ethene	65-115	≤ 20

Table 4.6.1-3
Summary of Calibration and QC Procedures for Method RSK-175

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
RSK-175	Volatile Organics	Initial multipoint calibration minimum 3 standards	Initial calibration prior to sample analysis	%RSD for all calibration analytes ≤ 30% or linear regression correlation coefficient r≥ 0.995	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration verification (one point)	Daily, before sample analysis and every 12 hours of analysis time	All calibration analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.6.1-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description	Retune instrument and verify	Apply R to all results for all samples associated with the tune
		ISs	Immediately after or during data acquisition for the calibration verification standard.	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL. EICP area within -50% to +100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	See Section 4.3.6 Area count >+100%, apply J for positives and R for non-detects. <-50%, apply J for positives only
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) above the RL in all samples in the associated analytical batch

Table 4.6.1-3 (Concluded)
Summary of Calibration and QC Procedures for Method RSK-175

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
RSK-175	Volatile Organics	LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4.6.1-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected AFCEE analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		MDL study	See section 4.3.1 of the CSSA QAPP	Detection limits established shall be ≤ ½ the RLs in Table 4.6.1-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	None	None	none	Apply F to all results between MDL and RL

[a]All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

4.6.2 Current Method SW9056–Common Anions

This method addresses the sequential determination of the anions chloride, fluoride, bromide (Br^-), nitrate, nitrite, phosphate(PO_4^{-3}), and sulfate in the collection solutions from the bomb combustion of solid waste samples, as well as water samples.

A small volume of combustate collection solution or other water sample is injected into an ion chromatograph to flush and fill a constant volume sample loop. The sample is then injected into a stream of elutent.

The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn (guard) column and a separator column, are packed with a low-capacity, strongly basic anion exchanger. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppressor column that reduces the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical-conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

RLs are listed in Table 4.6.2-1. The calibration, QC, corrective action, and data flagging requirements are given in Tables 4.6.2-2 and 4.6.2-3.

Table 4.6.2-1
RLs for Method SW9056

Analyte	Water		Soil	
	RL	Unit	RL	Unit
Bromide	0.5	mg/L	0.5	mg/kg
Chloride	1.0	mg/L	1.0	mg/kg
Fluoride	1.0	mg/L	1.0	mg/kg
Nitrate	1.0	mg/L	1.0	mg/kg
Nitrite	1.0	mg/L	1.0	mg/kg
Phosphate	1.0	mg/L	1.0	mg/kg
Sulfate	1.0	mg/L	1.0	mg/kg

Table 4.6.2-2
QC Acceptance Criteria for Method SW9056

Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
Bromide	86–112	≤ 20	86–112	≤ 30
Chloride	91–111	≤ 20	91–111	≤ 30
Fluoride	86–114	≤ 20	86–114	≤ 30
Nitrate	90–110	≤ 20	90–110	≤ 30
Nitrite	88–116	≤ 20	88–116	≤ 30
Phosphate	87–110	≤ 20	87–110	≤ 30
Sulfate	88–115	≤ 20	88–115	≤ 30

Table 4.6.2-3
Summary of Calibration and QC Procedures for Method SW9056

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW9056	Common anions	Multipoint calibration for all analytes (minimum 3 standards and one calibration blank)	Initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per multipoint calibration	All analytes within ±10% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time over 8 hour period	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis or when elutent is changed	All analytes within ±10% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Calibration verification	After every 10 samples and at the end of the analysis sequence	Instrument response within ±5% of expected response	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

Table 4.6.2-3 (Continued)
Summary of Calibration and QC Procedures for Method SW9056

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW9056	Common anions	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 4.6.2-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 4.6.2-2	Correct problem then reprep and analyze the LCS and all samples in the affected AFCEE analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Duplicate	One per every 10 samples	%D ≤10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results

Table 4.6.2-3 (Concluded)
Summary of Calibration and QC Procedures for Method SW9056

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]	Flagging Criteria[b]
SW9056	Common anions	MS	One MS per every 20 samples per matrix	QC acceptance criteria, Table 4.6.2-2	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1) %R for MS > UCL or (2) %R for MS < LCL
		MD	One MD per every 20 samples per matrix	Precision (%RPD) criteria in Table 4.6.2-2	None	Results >RLs and RPD outside of CL, apply J for all positive results for specific analytes in the same matrix in the analytical batch.
		MDL study	See section 4.3.1 of the CSSA QAPP	Detection limits established shall be ≤ ½ the RLs in Table 4.6.2-1	None	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	None	None	None	Apply F to all results between MDL and RL

[a]All corrective actions associated with CSSA project work shall be documented, and all records shall be maintained by the laboratory.
[b]Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

5.0 QUALITY CONTROL AND QUALITY ASSURANCE REQUIREMENTS

This section describes definitions, general principles of QA and QC and specific QA/QC requirements for field and analytical laboratories. Some of the specifics for various analytical methods are listed in tables in Section 4.0.

Quality control requirements for projects are specified in the respective DQOs. The number of field blanks, field duplicates, and field instrument QC may vary from project to project. Similarly, analytical QC requirements vary depending on project DQOs. Initial investigation may require a complete list of method analytes to be tested while ongoing investigations may allow a reduced analyte list, less stringent QC, and control limits. Data that are being produced for risk assessment may require accurate determination of detection and quantitation limits. This section describes various QC parameters for field and analytical efforts and should be taken into consideration when specific project DQOs are being identified and defined.

Definitions

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 (EPA QA/G-5).

Quality Control: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. The aim is to provide quality that is satisfactory, adequate, dependable, and economical (EPA QA/G-5).

5.1 Field Quality Control

This section explains various blanks that are to be collected along with normal environmental samples. The rationale for collecting these blanks and the recommended frequencies of collection are explained in the following sections. The frequency of collection of blanks is based on the type of investigation and is usually provided in respective statements of work.

5.1.1 Ambient Blank

The ambient blank consists of American Society for Testing and Materials (ASTM) Type II reagent grade water (or equivalent grade of deionized water normally used in the laboratory to prepare standards and reagents) poured into a VOC sample vial (containing appropriate amount of HCl for preservation) at the sampling site (in the same vicinity as the associated samples). It is handled like an environmental sample and transported to the laboratory for analysis. Ambient blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes.

Ambient blanks are used to assess the potential introduction of contaminants from ambient sources (e.g., active runways, engine test cells, gasoline motors in operation, etc.) to the field samples during sample collection. Ambient blanks shall be collected downwind of possible

VOC sources. The recommended frequency of ambient blank collection is one blank per VOC contaminated site per day, unless otherwise specified in the DQO.

When an analyte is detected in an ambient blank, the specific analyte(s) in all the samples associated with that ambient blank (for the same matrix and sampling date) will be flagged according to flagging convention in Table 6.1-2.

5.1.2 Equipment Blank

An equipment blank is a sample of ASTM Type II reagent grade water (or equivalent grade of deionized water normally used in the laboratory to prepare standards and reagents) poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis. Equipment blanks collected for VOCs must be appropriately preserved.

Equipment blanks are used to assess the effectiveness of equipment decontamination procedures.

The recommended frequency of collection for equipment blanks is one per site, unless otherwise specified in the DQO. Equipment blanks shall be collected immediately after the equipment has been decontaminated. The blank shall be analyzed for all laboratory analyses requested for the environmental samples collected at the site.

When an analyte is detected in the equipment blank the appropriate validation flag, as described in Table 6.1-2, shall be applied to all sample results from samples collected with the affected equipment.

5.1.3 Trip Blank

The trip blank consists of VOC sample vials (2 or 3) filled in the laboratory with ASTM Type II reagent grade water (or equivalent grade of deionized water normally used in the laboratory to prepare standards and reagents), transported to the sampling site, handled like an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes. Trip blanks from the laboratory must be properly preserved before they are sent to the field. A trip blank should be included when two or more samples are shipped in a cooler. The number of trip blanks needed should be discussed at the time of establishing the DQOs.

Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. The purpose of the trip blanks is to evaluate cross contamination of samples during transportation and storage, particularly when highly contaminated samples are stored or transported along with low level contaminated samples.

If a large number of trip blanks are stored in the field and used as needed, caution should be exercised as to the storage conditions of these vials. These vials must be stored away from sources of potential external contamination.

When an analyte is detected in the trip blank, the appropriate validation flag, as described in Table 6.1.2 shall be applied to all the positive results for the samples shipped in the same cooler.

One trip blank shall accompany each cooler of samples sent to the laboratory for analysis of VOCs.

5.1.4 Field Duplicates

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. Splitting from a single sample after collection (e.g. from the same bailer, or the same collection device) is not allowed. Two samples collected from one composite mix will not be considered as field duplicates. For CSSA field duplicates are not required unless specified in project DQOs.

Results from the field duplicates are used to assess Total Precision (both sampling and analysis). In order to calculate Total Precision, both the laboratory and prime contractor staff must be able to identify the samples as duplicates. Field duplicates must be identified as duplicates but given different identification numbers on the containers and on the chain-of-custody forms. The chain-of-custody form must request all the specified analyses for both the samples.

The frequency of collection for field duplicates varies and should not exceed 5% of the samples for any field event. When an extended sampling event occurs and the sampling techniques have been assessed by the prime contractor and the CSSA Environmental Officer to be satisfactory, a lower percentage of field duplicates may satisfy the DQOs and may be cost effective.

The sample collection team should select field samples from either known or suspected contaminated areas of a site and designate them as field duplicates. It is important to note that if Total Precision, established by field duplicates, exceeds the DQO limits for precision for a given matrix, the results of field samples may be used as estimated values. If the Total Precision for a set of duplicates could not be calculated because no analytes were detected above MDLs in both samples (i.e., non-detects), the data do not require an R flag. The case narrative should state that the duplicates were analyzed and a numerical precision value could not be determined through no fault of the sampling or laboratory staff.

5.2 Analytical Quality Control

QC elements relevant to screening data are presented in Section 4.1. This section presents QC requirements that shall be followed during all analytical activities for fixed-base, mobile, and field laboratories producing definitive data. The purpose of this QC program is to produce data of known quality that satisfy the project objectives. This program provides a mechanism for ongoing control and evaluation of data quality measurements through the use of QC materials.

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 the first and the 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 (see *Department of Defense Quality Systems Manual for Environmental Laboratories, version 1, October 2000, page 52*).

Each preparation batch must include, as a minimum, one method blank and one laboratory control sample that are similar in matrix as the field samples. Method blanks, equipment blanks, and laboratory control samples are for quality control purposes and are not considered environmental samples. A matrix spike, a matrix duplicate, and a field duplicate may be added if required by the DQOs and submitted along with the field environmental samples. Matrix spike, matrix duplicate, and field duplicates are considered environmental samples and are included as part of the 20 sample preparation batch.

If samples that are prepared together in a batch are analyzed in more than one analytical batch, each analytical batch must include the method blank and the laboratory control sample that were part of the preparation batch. Samples requiring dilutions that are analyzed in a subsequent analytical batch do not require the original method blank and LCS as long as the original recoveries were in control. Samples requiring dilutions that are analyzed in a subsequent batch will need a calibration verification. However, the second batch may require a new set of method blank and LCS if samples from another preparation batch are included.

If an analytical batch includes samples prepared in more than one preparation batch, method blanks and laboratory control samples from each preparation batch must be included to validate the results of each sample matrix.

The identity of each analytical batch shall be unambiguously reported with the analyses so that a reviewer can identify the QC samples and the associated environmental samples.

The type of QC samples and the frequency of use of these samples are discussed below and in the method-specific subsections of Section 4.0.

5.2.1 Laboratory Control Sample

The laboratory control sample is analyte-free water for aqueous analyses or Ottawa sand for soil analyses (except metals where glass beads of 1mm diameter or smaller may be used) spiked with all analytes listed in the QC acceptance criteria tables in Section 4.0 of this QAPP if full analyte list is warranted, or a shorter target analyte list approved for the project. For CSSA groundwater studies, a shorter list of analytes have been approved for Methods SW8260B and SW6010B. However, the project manager and the laboratory must review the project-specific objectives and include either a short or a complete list of analytes in the LCS. Each analyte in the LCS shall be

spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. The LCS shall be carried through the complete sample preparation and analysis procedure.

The LCS is used to evaluate each preparation and analytical batch and to determine if the method is in control. One LCS shall be included in every preparation batch. If more than one LCS is analyzed in an analytical batch, results from all LCSs analyzed shall be reported.
The performance of the LCS is evaluated against the QC acceptance limits given in the tables in Section 4.0.

Whenever an analyte in the LCS is outside the acceptance limit, corrective action shall be performed and documented in the case narratives. After the system problems have been resolved and system control has been reestablished, all samples in the analytical batch shall be reanalyzed for the out-of-control analyte(s). When an analyte in the LCS exceeds the upper or lower control limit and the corrective action was ineffective, the laboratory should discuss the issue with the prime contractor, AFCEE/ERC, and CSSA personnel. Appropriate validation flag, as described in Sections 4.0, shall be applied to all affected results after obtaining approval from AFCEE and CSSA.

5.2.2 Matrix Spike

A matrix spike is an aliquot of a sample spiked with known concentrations of all analytes of interest for the specific project. Aliquots from one sample container should be used for the parent and MS analysis (see Section 3.2 for container requirements). For aqueous VOA analysis, when a 25 ml purge is needed, three VOA vials should be collected as a minimum.

Project-specific DQOs will specify the analytes of interest needed. For CSSA groundwater studies, a reduced list of analytes can be used, as in the case of LCS, for Methods SW8260 and SW6010. However, all the method analytes are needed for all newly installed wells or off-post wells that have not been previously sampled, or if the DQOs require them. The spiking occurs prior to sample preparation and analysis. Each analyte in the MS shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. Only CSSA samples shall be used for spiking. It is the responsibility of the prime contractor and the field staff to designate MS samples on the chain-of-custody forms. If field staff fails to designate an MS in the chain of custody form, a laboratory representative must contact the project manager and have appropriate samples identified for the MS.

The MS is used to document the bias of an analytical method due to sample matrix. CSSA does not use MS to control the accuracy or precision of the analytical process.

Currently, it is known that there are three different zones of groundwater at CSSA. Monitoring wells that are placed in different zones may provide information on the different matrices of groundwater. When knowledge of groundwater matrices is available, the number of MSs needed to evaluate the effect of matrix on analytical method may be reduced and should be specified for a project DQOs.

The performance of the MS is evaluated against the QC acceptance limits given in the Tables in Section 4.0.

SW846 methods consider the use of MS as a recommendation and not a requirement. If matrix effect is noted, the resulting qualified data are usable to make decisions. Data should not be corrected for matrix interference. It is not clear that the matrix effect is taken into any calculation in remedial design, remedial action, or risk assessment. CSSA, therefore, considers the use of MS as a secondary measure of quality.

5.2.3 Matrix Duplicate

A matrix duplicate is an aliquot of a sample taken from one container and analyzed like the parent sample. Matrix duplicate results are compared to the parent sample results and measure of matrix precision is established by calculating the %RPD and comparing the values to the respective precision values listed in various Tables in Section 4.0. A matrix duplicate is required for every preparation batch (20 CSSA samples or less), unless otherwise indicated in the DQOs.

If %RPD values are outside of the control limits, particular analytes in the samples from the same matrix and analyzed in the same analytical batch shall be qualified with a J flag only when the result is greater than the RL. If a numerical precision value could not be calculated, the case narrative should discuss the event.

5.2.4 Surrogate

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but that are not normally found in environmental samples.

Surrogates are used to evaluate extraction efficiency and laboratory performance of individual samples.

Surrogates shall be added to environmental samples, controls, and blanks, according with the method requirements, or according to project DQOs.

Whenever a surrogate recovery is outside the acceptance limit, corrective action must be performed. After the system problems have been resolved and system control has been reestablished, affected samples must be prepared and analyzed. If corrective actions are not performed or are ineffective, the laboratory should follow the guidelines specified in Section 9.0 of this QAPP. An appropriate validation flag, as described in Sections 4.0, shall be applied to the sample results after approval from AFCEE and CSSA.

5.2.5 Interference Check Solution

The interference check solution, used in inductively coupled plasma (ICP) analyses, contains both interfering and analyte elements of known concentrations. The ICS is used to verify

background and interelement correction factors. The ICS is analyzed at the beginning of an analytical batch for SW6010 and at the beginning of an analytical batch or once every 12-hour period, whichever is more frequent for SW6020.

When the interference check solution results are outside of the acceptance limits stated in the respective Tables in Section 4.0, corrective action shall be performed. After the system problems have been resolved and system control has been reestablished, reanalyze the ICS. If the ICS result is acceptable, reanalyze all affected samples. If corrective action is not performed or the corrective action was ineffective, the laboratory should follow the guidelines specified in Section 9.0 of this QAPP. An appropriate validation flag, as described in Sections 4.0, shall be applied to all affected results after approval from the AFCEE and CSSA.

5.2.6 Method Blank

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank shall be carried through the complete sample preparation and analytical procedure.

The method blank is used to document contamination resulting from the analytical process. A method blank shall be included in every CSSA preparation batch.

The presence of analytes in a method blank at concentrations equal to or greater than the RL indicates a need for corrective action. Corrective action shall be performed to eliminate the source of contamination prior to proceeding with analysis. After the source of contamination has been eliminated, all samples in the analytical batch shall be prepared and reanalyzed. No analytical data shall be corrected for the presence of analytes in blanks. When an analyte is detected in the method blank and in the associated samples and if corrective actions are not performed or are ineffective, the laboratory must contact the project manager to discuss the issue. See Section 9.0 for guidelines for obtaining CSSA and AFCEE approval. Approved validation flag, as described in Sections 4.0 shall be applied to the sample results.

Results of the method blank shall be reported in the appropriate CSSA forms in Section 6.0. If the blank results are less than the corresponding RL, it shall be reported as < RL numerical value (e.g., < 0.03 mg/L for arsenic in water).

5.2.7 Holding Time Compliance

All sample preparation and analysis shall be completed within the method-required holding times. The holding time for a sample begins at the time of sample collection. Some methods have separate holding time requirements for extraction and analysis (e.g., Methods SW8081, SW8270, etc.). The extraction holding time is calculated from the time of sample collection to the time of completion of the sample extraction process as described in the applicable method, prior to any necessary extract cleanup and/or volume reduction procedures. The analysis holding time is calculated from the time of extraction completion to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalysis.

If no extraction is required, the analysis holding time is calculated from the time of sample collection to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalyses. In methods requiring sample preparation prior to analysis, if holding times are exceeded and the analyses are performed, the results shall be flagged R.

5.2.8 Dilution Tests

A review of past analytical results on CSSA water samples (approximately 90 samples) indicate that only 1.1% of cadmium and lead samples required dilution. Other metals, arsenic, barium, chromium, copper, nickel and zinc did not require dilutions. CSSA no longer requires dilution tests for water samples. For soil samples (over 600 samples) dilutions were needed to obtain accurate results. The percentages of dilutions needed ranged from 11.58% for chromium to 43.42% for lead. CSSA requires that dilution tests be performed for soil samples. The following guidelines are provided:

- After completion of an analytical batch, if all the CSSA samples are < 25xMDL for that metal in the SW7000 methods, or <50xMDL for SW6010, or <100xMDL for SW6020, no dilution test is needed for CSSA samples in that batch.
- If one or more samples are above the limits for that specific method, select one sample and perform dilution test. At a minimum a five-fold dilution of the digestate is required. If the result of the diluted sample is within 10% of the undiluted sample result, no further action is required.
- If the result of the diluted sample exceeds the above acceptance criteria, matrix interference is indicated. Evaluate the results of the matrix spike performed on a sample in that batch. If no matrix interference is observed, the results in this batch should be qualified with a J flag. If matrix interference is observed, the results of all the samples for that analyte should be qualified with M flag.
- If the dilution test failed and if no matrix spike sample was included in that batch, qualify the results of the analyte for all samples in that batch with M flag.
- If the atomic absorption instrument measures more than one metal simultaneously, the rules discussed above applies only for that metal exceeding >25xMDL.
- Under normal analytical conditions, CSSA does not require a recovery test, a post-digestion test, or a method of standard addition. However, if DQO requires a high degree of accuracy in decision-making, a recovery test, a post-digestion test, and/or a method of standard addition should be performed. This requirement should be specified in the DQO for that project.

5.2.9 Quality Control Charts

Analytical performance in the laboratory should be monitored for each method through the use of trend analysis of the behavior of analytes over a period of time. This is best determined by the use of control charts. As a quality assurance function, periodic reviews of control charts will

identify degradation or trends in the analytical system allowing the analysts to take effective actions to prevent out-of-control events.

CSSA recommends that, as a minimum, trend analysis be performed for system performance check compounds (SPCCs), calibration check compounds (CCCs), and analytes of interest in the laboratory control samples for a specified project.

5.2.10 Manual Integration

The laboratories participating in the CSSA environmental analysis should maintain a SOP for manual integration of chromatographic peaks. The SOP should specify when integrations conducted by the analytical instruments are unreliable and unacceptable, how the problems should be resolved by the analyst, approved by the section supervisor and documented. The documentation must include the justification and rationale for performing manual integration. The section supervisor must approve the technical acceptability of the manual integration. The reviewers of the data package, be it Quality Assurance Officer (QAO), project manager, or the prime contractor's data reviewer, must be satisfied that the manual integration event was justified and properly performed. CSSA requires that all manual integration activities are documented and included in the respective case narratives. Both instrument integrated and manually integrated chromatograms should be included with the case narrative as proof of acceptable integration.

Some examples of unreliable instrument peak integration include co-elution of analytes, unusual tailing of peaks, drastic changes in baseline, and incompletely resolved peaks. There may be a number of other instances that may warrant a manual integration of the area of peaks. All analysts performing chromatographic analysis must be trained in the proper way of performing manual integration and when not to perform manual integration.

5.2.11 Analytical Variance Requests

The prime contractor must assure CSSA that chosen analytical subcontractor laboratories are able to meet the requirements specified in the CSSA QAPP. There may be instances where a laboratory may require certain variances from the specifications. All variance requests from the laboratories must be submitted in writing to the prime contractor prior to the start of a project. The prime contractor must review laboratory documentation and justification provided by the laboratory to assess the validity of the variance. If the justification is substantiated, then the prime contractor should evaluate the effect of the variance against the data quality objectives of the project.

The prime contractor should submit its assessment (with supporting documentation) to CSSA for approval. No variance shall be granted without CSSA approval.

All approved variances should be included as an appendix to the project-specific SAP.

5.3 Quality Control for Automated Laboratory

This section describes guidance for Good Automated Laboratory Practices (GALP) for assuring the reliability of laboratory data (see *EPA 2185-Good Automated Laboratory Practices, Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations, 1995 Edition*).

Laboratory Information Management System is a system of hardware and software that collects and manages data. Automated laboratory systems that record data but do not allow changes to the data are not LIMS. The ability to affect changes to original observations or measurements is the factor in determining whether the automated laboratory system is a LIMS. If data entering automated laboratory systems can be manipulated or changed in any way by the action of a person prior to being recorded, then that automated laboratory system is a LIMS.

The prime contractor shall review the LIMS for conformance with the above referenced EPA guidance; that the data transformed from analytical instruments to the LIMS are correct; that manual entries are double checked; that LIMS output are reviewed and approved; and that LIMS is secure from tampering. The LIMS security should prevent changes to the data by unauthorized persons.

EPA's GALP guidance is built on six principles (see *Chapter 2, GALP implementation guidance of EPA 2185, 1995*).

1. Laboratory management must provide a method of assuring the integrity of all LIMS data.
2. The formulas and decision algorithms employed by LIMS must be accurate and appropriate.
3. A critical control element is the capability to track LIMS raw data entry, modification, and recording to the responsible person.
4. Consistent and appropriate change controls capable of tracking LIMS operations and software are vital elements in the control process.
5. Procedures must be established and documented for all users to follow. Control of even the most carefully designed and implemented LIMS will be thwarted if the user does not follow these procedures.
6. The risk of LIMS failure requires that procedures be established and documented to minimize and manage failures.

EPA's GALP guidance discusses the implementation of the six principles as they relate to: a) laboratory management; b) personnel; c) quality assurance unit; d) LIMS raw data; e) software; f) security; g) hardware; h) comprehensive testing; i) records retention; j) facilities; and k) standard operating procedures. The users of the CSSA QAPP are required to read the EPA's GALP and implement the principles as appropriate. A short summary of each topic is provided below:

a) **Laboratory Management** shall ensure that personnel understand the functions they are to perform on the LIMS; that a quality assurance unit monitors LIMS activities; that facilities, personnel, and resources are adequate and available; that corrective actions are promptly taken; and that all applicable GALP provisions are followed.
b) **Personnel** shall have adequate education, training, and experience to perform assigned LIMS functions, and be available for timely operation of LIMS.
c) **Quality Assurance Unit** is independent of LIMS personnel and reports directly to laboratory management; has immediate access to LIMS data, SOPs and other records; audits LIMS at periodic intervals to ensure the integrity; determines that no deviation from SOPs were made; and reports any problems to laboratory management.
d) **Raw data** are identified and documented with time, dates, and personnel responsible for recording data. Instruments transmitting raw data are uniquely identified and when data are recorded, the time and date are documented. Procedures and practices to verify the accuracy of raw data are documented and included in the SOPs.
e) **Software:** SOPs shall be prepared for development, testing and quality assurance, change control, version control, and a historical file. This guidance is applicable to new systems and/or existing and commercially available systems.
f) **Security:** Laboratory management shall ensure that security practices to assure the integrity of data are adequate and that threat assessments have been performed. The integrity objective provides owners and users of laboratory data assurance that data are reliable and accurate. The availability objective provides protection against loss of information or services. The confidentiality objective addresses those situations where disclosure of data is undesirable and/or unlawful. The laboratory management shall evaluate tangible (facilities, hardware, software, supplies, documentation, and data) and intangible (personnel, reputation, motivation, morale, goodwill, and opportunity) assets and determine threats to assets. The threats may be natural disasters, unrestricted access to LIMS, or fraud. Safeguards must be in place for stand-alone, networked, or data center computing.
g) **Hardware** must be of adequate design and capacity and a description must be documented and maintained. SOPs for installation and maintenance, and records of repair and non-routine maintenance shall be kept.
h) **Comprehensive Testing:** In order to ensure on-going reliability, performance and accuracy, comprehensive testing of LIMS shall be conducted at least once every 24 months.
i) **Records Retention:** The laboratory management and prime contractor shall ensure that LIMS data and documentation are retained according to CSSA contract requirements.
j) **Facilities:** The environmental conditions of the facility housing the LIMS are regulated to protect against LIMS data loss.
k) **Standard Operating Procedures** are periodically reviewed to ensure that they accurately describe the current procedures and are updated as appropriate. Laboratory management must authorize changes.

6.0 DATA MANAGEMENT

Data management involves several important stages that include data transformation, review, verification, and validation. This section describes the processes for review, verification, and validation and provide formats for data submittal. Data storage, retrieval, and security are discussed in this section. Both the analytical laboratory and the prime contractor shall follow the requirements in this section, unless otherwise stated in project-specific documents. Applicable terms are defined in respective sections. Where there are no clear definitions available, the process is explained.

Data Reporting:

- MDLs and sample results shall be reported to one decimal place more than the corresponding RL.
- Soil/sediment samples shall have results reported on a dry weight basis.
- Analyte results below the corresponding MDLs are considered non-detects. The non-detect result should be entered with the numerical value of the MDL with a U flag in all the CSSA report forms.
- Method blank results at or below the corresponding RLs are considered non-detects. Non-detect method blank values should be reported "<RL" in the CSSA report forms.
- RLs should be adjusted for dilutions.

Data Packages:

Packages for **VOC screening level data** for IDW and discreet samples shall contain, at a minimum, calibration verification, method blank, and LCS data for the analytical batch in which CSSA field samples are analyzed. The acceptability of the initial calibration data should be discussed in the case narrative. The date and the initial calibration identification should be referenced in the calibration verification data sheet. Alternatively, the data sheets for the initial calibration may be included. Matrix spikes, matrix duplicates, and field duplicates are not required for these samples. The data need not be presented in the CSSA forms as long as the acceptance criteria for various QC samples are presented in the data package. Normally, only a reduced approved list of analytes should be reported, however, if any unexpected analytes are positively identified, the laboratory should contact the prime contractor, who in turn will discuss the issue with CSSA. CSSA will decide on the reporting requirements of the unexpected analytes.

This section for the definitive data management is organized as follows:

- Section 6.1 discusses the analytical laboratory's data review requirements
- Section 6.2 discusses the Prime Contractor's data verification requirements
- Section 6.3 discusses the Prime Contractor's data validation requirements
- Section 6.4 discusses the CSSA approval process of the data packages
- Section 6.5 discusses the electronic copy requirements for CSSA
- Section 6.6 discusses the hard copy requirements for CSSA

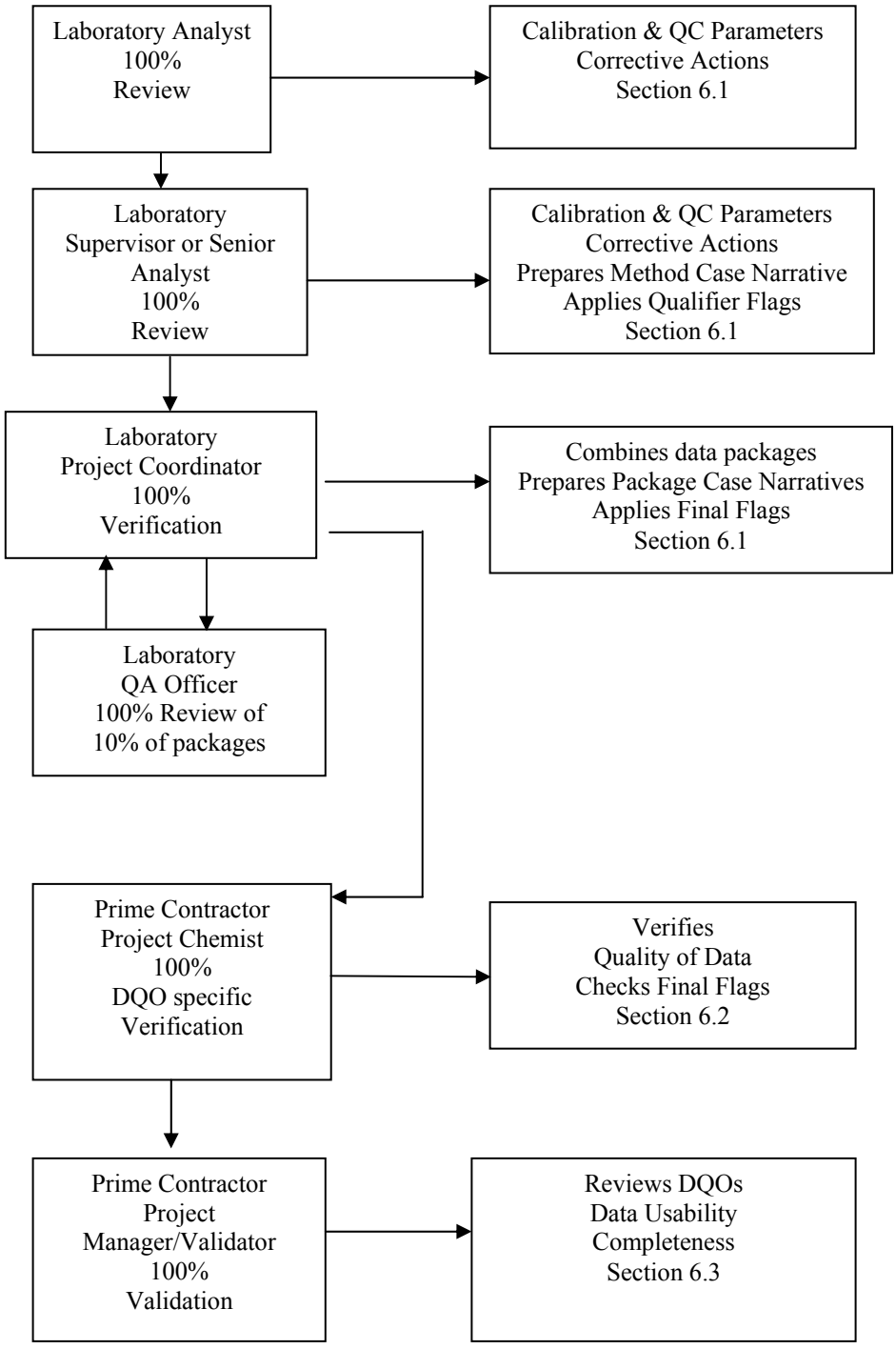

Figure 6.0 Data Review, Verification, and Validation Process

6.1 Data Review in the Laboratory

Data review is the process of evaluating analytical data against stated calibration, accuracy, precision, and other parameters listed in various tables in Section 4.0 of the CSSA QAPP. If specific guidance is not provided in Section 4.0 of the QAPP, applicable instrument or product manufacturer's protocol or analytical method requirements should be used. The objective of data review process (see Figure 6.0) is to evaluate the quality of the data.

In the laboratory analytical sections, the analyst performing the tests shall review 100 % of the data. After the analyst review has been completed, a senior analyst or the section supervisor shall review 100% of the data independently. All corrective action reports pertaining to the data package shall be reviewed, and if corrective actions were ineffective, appropriate qualifier flags shall be applied to the data by the supervisor or the senior analyst. Qualifier flags are provided in various tables in this section.

Data qualifiers shall be added or, if applied by a software package, reviewed by the laboratory supervisor of the respective analytical section, after the first and second level of laboratory data reviews have been performed. Analytical batch comments shall be added to the first page of the definitive data report packages to explain any nonconformance or other issues.

Each section supervisor shall submit the data to the laboratory project manager who is responsible for combining all the laboratory data into a package to be submitted to the prime contractor. The project manager performs a verification of the quality of the entire data package after it has been assembled and prepares a case narrative for the package. The verification includes confirmation of the quality of the submitted data from various sections, evaluating the corrective action reports, and assigning the final flag where necessary. This final qualifier shall reflect the most severe qualifier that was applied to the data, i.e., all data will have only one data qualifying flag associated with it. The allowable final data qualifiers for definitive data and the hierarchy of data qualifiers, listed in order of the most severe through the least severe, are *R, M, F, J, B*, and *U.*

The one exception to these data flagging criteria rules applies to the tentatively identified compounds (TICs) that are identified only in the GC/MS methods. These TICs numerical results will always be qualified with a T flag.

The laboratory project manager will assure that data are presented in the appropriate forms specified in this section and if electronic deliverables are requested by CSSA, they are in the appropriate formats specified in project documents. If both hard copy and electronic deliverables are required, the project manager must verify that data are identical in both formats.

The laboratory QA section shall perform a 100 % review of 10 % of the completed data packages. The QA section shall evaluate the accuracy of calibration, quality control, flags, case narrative, and corrective action reports during verification. If QA review identifies any problems that are not covered by previous reviewers, the laboratory project manager must be notified for proper corrective actions.

Table 6.1-1
Data Qualifiers

Qualifier	Description
J	The analyte was positively identified, the quantitation is an estimation.
U	The analyte was analyzed for, but not detected. The associated numerical value is at or below the MDL.
R	The data did not meet calibration and/or QC criteria.
B	The analyte was found in an associated blank, as well as in the sample.
M	A matrix effect was present.
T	Tentatively identified compound (using GC/MS)

Table 6.1-2
General Flagging Conventions

QC Requirement	Criteria	Flag	Flag Applied To
Holding Time	Time exceeded for extraction or analysis	R	All analytes in the sample
Ambient Blank (VOC samples only)	Analyte(s) detected ≥ RL	B	The specific analyte(s) in all samples with the same matrix, collected from the same site and sampling date
Trip Blank (VOC samples only)	Analyte(s) detected ≥ RL	B	The specific analyte(s) in all samples shipped in the same cooler as the blank
Equipment Blank	Analyte(s) detected ≥ RL	B	The specific analyte(s) in all samples with the same sampling date as the equipment blank
Field duplicates	Field duplicates > RLs and RPD outside CL	J for the positive results	The specific analyte(s) in all samples collected on the same sampling date
Sample Preservation/ Collection	Preservation/collection requirements not met	R for all results	All analytes in the sample
Sample Storage	< 2°C or > 6°C	J for the positive results R for the nondetect VOCs	All analytes in the sample

It is important to remember that data review is an iterative process and the data packages may be sent to previous reviewers for additional information or clarification. The laboratory must have a system of documenting each stage of the review and verification process. The system should be such that an external reviewer must be able to follow the identity of the persons reviewing the data, the dates of review and changes or modifications made during the review.

6.2 Data Verification by the Prime Contractor

Data verification is defined as, "confirmation by examination and provision of objective evidence that specified requirements have been fulfilled" (EPA QA/G-5, Appendix B).

The prime contractor's chemist shall review the entire definitive data report package and apply the final data qualifiers. Initially, the prime contractor must review the flags applied by the laboratory for accuracy as specified by Tables in Section 4.0 and by Tables 6.1-1 and 6.1-2. The prime contractor may use various checklists during the verification process to document all the verification activities. However, these checklists should not be included as part of the data packages. CSSA reserves the right to request these completed checklists when needed. All changes to the data or flags must be explained in the Data Verification Report, and the QA summary section of the technical reports.

In the case of matrix interference, the laboratory will follow the guidelines specified in appropriate Tables in Section 4.0. However, the prime contractor must apply M flags to additional samples from the same site and same matrix, where applicable.

6.3 Data Validation by the Prime Contractor

Validation is defined as "confirmation by examination and provision of objective evidence that the particular requirements for a specific **intended use** have been fulfilled" (EPA QA/G-5, Appendix B). Validation is distinguished from verification in that verification establishes the **quality** of data while validation establishes the **usability** of the data. For this reason, the person validating the data must have prior knowledge of the DQOs for the specific project and, if possible, communicate with the users to obtain a complete understanding of the intended use of the data.

For CSSA drinking water well samples, 100% validation is required. This includes wells CS-1, CS-9, CS-10, and all wells sampled as part of the off-post monitoring program. Because these wells supply drinking water to the public and have potential to impact human health, valid and defensible data are required.

For groundwater monitoring wells, validation requirements will vary according to specific projects and the requirements will be specified in the DQOs

Validation should be performed by the prime contractor's data validation personnel that are different from the ones that verified the data. However, the project manager and the project chemist who have knowledge of the project DQOs should be consulted.

If a third party validation is required, this requirement should be stated in the DQOs and must specify the percentage of data that require validation. DQOs must also specify applicable federal or state guidelines and any modifications to the guidelines to be used for validation.

The USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (EPA540/R-99/008, October 1999) or for Inorganic Data Review (*EPA-540/R-94-013, February 1994*) should be used only if there is a regulatory requirement.

The following guidelines are offered to the person performing validation:

- Review the case narratives pertaining to the data packages and establish that corrective actions were performed.
- Review all approved laboratory variances that are applicable to the project.
- Review all qualifier flags based on acceptance criteria.
- Review the data verification report from the prime contractor.
- Ascertain if the representativeness objective for the project was achieved.
- Review qualified data for usability for the specific project. At times there may be a need to use data that does not meet technical criteria. Use of these data does not constitute either a new requirement standard or full acceptance of the data. Any decision to utilize such data is strictly to facilitate the progress of the project requiring the availability of the data.
- Some individual samples, by the nature of the sample itself may inhibit the attainment of specifications. Appropriate allowances must be made for these sample results.
- Awareness of previous investigations for the specific projects and pre-existing data gaps.
- Calculate completeness of sample and analytical data collection to check against the objectives for the project.
- Identify data gaps based on completeness and non-conformance events.
- Evaluate if the data gaps prevent CSSA to make decisions intended in DQOs.
- Exercise professional judgment based on DQOs if specific technical criteria is either unavailable or could not be used.
- Document all evaluations, calculations, rejections, recommendations, and provide rationale for all specific validation actions.
- Submit a validation report and include an executive summary, if appropriate.

6.4 CSSA Approval Process

CSSA chemists or the designated consultant chemists shall verify at least 10% of the data packages submitted by the prime contractor. The purpose of the review is to confirm that the quality of data has been established by the prime contractor's verification process and to obtain an early understanding of the data usability. A complete validation report by the prime contractor explaining data usability would be included in the Technical Report.

These data packages shall contain the following information:

- Chain-of-custody forms

- Field and laboratory cross reference identification (e.g., CSSA FORM I-1)
- Relevant CSSA forms for reporting calibration and quality control events for all methods in a data package. Unless otherwise specified, raw data (instrument printout, chromatograms etc.) are not required for a package. CSSA reserves the right to obtain specific raw data packages when necessary.
- As a minimum, CSSA forms to report instrument tune, holding times, initial calibration, second source verification, calibration verification, method blanks, laboratory control samples, matrix spikes, surrogates, and other method-specified quality control information should be provided.
- Relevant sample analytical data sheets for each method (see CSSA Form I-2 or O-2)
- Case narratives for each data package by analytical by method. Case narratives should include information that adversely affect the quality of the data, or state that no problems were encountered. Problems relating to insufficient sample volumes, sample dilutions, missed holding times, out-of-control events and corrective actions should be discussed.
- Corrective action reports specific to each package.
- Data verification report. The verification report is a summary of the prime contractor's assessment of the quality of the data. It is NOT a copy of a checklist used during the verification process. The verification report must explain any changes to qualified results (flags) and any professional judgment rationale used during the verification process.
- A project summary report. The project summary report should associate the data package to a specific project (background study, quarterly groundwater monitoring, soil-vapor extraction, soil gas survey, etc.) The report should include a short summary of previous findings, the DQO for the project, risk drivers, and regulatory action levels. A discussion of project reporting limits and their relationship to the action levels should be included.

6.5 Electronic Reports

The prime contractor shall provide an electronic deliverable report in the Environmental Restoration Program Information Management System (ERPIMS) format as specified by the SOW for the project.

ERPIMS is a data management system designed to accommodate all types of data collected for Installation Restoration Program projects. Specific codes and data forms have been developed to allow consistent and efficient input of information to the system. The database information shall be provided by the prime contractor via American Standard Code Information Interchange (ASCII) files in specified ERPIMS format on 3.5" floppy diskettes. The information transferred shall include all required technical data such as site information; well characteristics; and hydrogeologic, geologic, physical, and chemical analysis results. Electronic data reporting formats and requirements are given in the most current version of the *ERPIMS Data Loading Handbook*. The electronic deliverable shall also conform to the standards for CSSA's Geographical Information System (GIS) chemistry data.

6.6 Hard Copy Reports

Hard copies of the analytical reports are used to document site investigation, site closure, and compliance with various environmental programs. Site investigation and closure documentation must meet requirements specified in either RRS or TRRP standards, depending on which investigation and/or

closure method is being utilized. Compliance samples will undergo less scrutiny and a lower level of documentation and reporting. Hard copies of all data packages and other reports should be delivered to CSSA after completion of validation and after the packages/reports have been accepted by the CSSA service centers.

The hardcopy data reports shall conform to the formats identified in this section.

Screening data report packages may vary from project to project. In general, quick turnaround of analytical data reports may be required. The format and the required calibration and quality control information will be specified in the DQOs and will be communicated to the analytical laboratory participating in the efforts. Therefore, standard forms for screening data are not prescribed in the CSSA QAPP.

A definitive data inorganic report package shall consist of the following CSSA forms: COC and Forms I-1 to I-10 (as appropriate) for each analytical batch of inorganic analyses performed.

A definitive data organic report package shall consist of the following CSSA forms: COC and Forms O-1 to O-13 (as appropriate) for each analytical batch of organic analyses performed.

A definitive data wet chemistry report package shall consist of the following CSSA forms: COC and W-1 to W-10 (as appropriate) for each analytical batch of wet chemistry analyses performed.

For mercury analysis, Form I-3A shall be substituted for Form I-3 in the inorganic report package.

MDL forms should be included when DQO requires them.

INSTRUCTIONS FOR COMPLETING CSSA REPORT FORMS

The following instructions shall be used in completing the CSSA report forms for screening and definitive data. The bold lettering identifies the fields on the CSSA report forms.

Use as many sheets as necessary. Sheets may be duplicated with only those sections necessary to be completed (i.e., you do not have to duplicate previously reported information from one sheet to the next). Sequentially number the sheets at the bottom of the page if more than one sheet is necessary.

*Reporting Dilutions: Justification for diluting samples shall be provided in the comments section on the appropriate form (I-2, O-2, or W-2). If the result for any analyte is outside the calibration range (i.e., greater than the highest calibration standard), the sample shall be diluted appropriately and reanalyzed. Results from the undiluted and diluted sample shall be reported on the appropriate form (I-2, O-2, or W-2). The results of the analysis of the diluted sample shall be reported with the dilution noted on the report form and the MDL and RL adjusted for the dilution. The results of the diluted sample will be usable provided there are no QC problems with this analytical batch. Under these conditions the undiluted results should be flagged R.

ALL INORGANIC, ORGANIC, AND WET CHEMISTRY FORMS

The field identifications for all the forms are explained below. They are arranged in alphabetical order.

AB#: enter the unique CSSA analytical batch number (see Section 5.2 of the CSSA QAPP for a definition of a batch)

Acceptance Criteria: enter the acceptance criteria required to be met (see QAPP Section 4.0)

Amt. Spiked: enter the amount of spike added to the matrix

Analyte: enter all analyte names in the same order as listed in the tables in QAPP Section 4.0.

Blank ID: enter the unique identification number for the blank.

CCB #1 ID: (used for 6010B analysis) enter the identification number for the first Continuing Calibration Blank (CCB) (the same ID number will be found in the run sequence log, e.g., CCB960603-1)

CCB #2 ID: (used for 6010B analysis) enter the identification number for the second CCB (the same ID number will be found in the run sequence log, e.g., CCB960603-2)

CCB #3 ID: (used for 6010B analysis) enter the identification number for the third CCB (the same ID number will be found in the run sequence log, e.g., CCB960603-3)

CCV #1 ID: enter the unique identification number for the first calibration verification (CCV) such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., CCV960603-1)

CCV #2 ID: enter the unique identification number for the second CCV such that the CCV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., CCV960603-2)

COD (optional): if a non-linear calibration is used for the calibration of an analyte, enter the coefficient of determination

Comments: enter any comments

Compound: enter BFB or DFTPP, as appropriate

Concentration: enter the numeric result

Concentration Units: enter the appropriate units (i.e., mg/L or mg/kg dry weight)

Confirm: enter the numeric result from the confirmation column/detector

Contract #: enter the contract number and delivery order number under which the analytical work is being performed (e.g., F21625-94-D-8005/0001)

Continuing Calibration Blank 1: enter a numeric result for the first continuing calibration blank run

Continuing Calibration Blank 2: enter a numeric result for the second continuing calibration blank run

Continuing Calibration Blank 3: enter a numeric result for the third continuing calibration blank run

Control Limits: enter the control limits required to be met (see QAPP Section 4.0).

%D: enter the percent difference between the expected and found

Date: enter the date, in the format DD-MMM-YY (e.g., 6 Jun 96)

Date Samples Collected/Prepared/Analyzed: enter the appropriate dates in the format DD-MMM-YY (e.g., 3 Jun 96)

Date Analyzed: enter the date the sample was analyzed by the laboratory in the format DD-MMM-YY (e.g., 6 Jun 96)

Date Analysis Started: enter the date the sample analysis was started in the format DD-MMM-YY (e.g., 6 Jun 96)

Date Analysis Completed: enter the date the sample analysis was completed in the format DD-MMM-YY (e.g., 6 Jun 96)

Dilution: enter the dilution (if applicable, e.g., 5, 10 etc.)

Duplicate Sample Result: enter the numeric result of the duplicate sample. Soil sample results must be entered only on a dry weight basis. If an analyte was not detected above the MDL, leave this column blank.

Expected: enter the expected result (i.e., the concentration of the calibration material).

Flag: enter the appropriate qualifier flag according requirements specified in various tables in Sections 4.0 and 6.0 of the CSSA QAPP.

Found, Found 1, Found 2: enter the measured result. Found: enter the ICV result; Found 1: corresponds to the first CCV run; Found 2: corresponds to the second CCV run, etc.

ICV ID: enter the unique identification number for the ICV such that the ICV could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., ICV960603-1)

Ion Abundance Criteria: enter the criteria for the specific mass (see QAPP Subsections 4.4.4 and 4.4.5)

Initial Calibration ID: enter the unique identifying number given to the initial calibration event used in the determination of the sample results

Initial Calibration Date: enter the appropriate date in the format DD-MMM-YY (e.g., 3 Jun 96)

Initial Calibration Blank ID: enter the identification number for the calibration blank (the same ID number will be found in the run sequence log, e.g., CB960603

Initial Calibration Blank: enter a numeric result for the calibration blank

Injection Date/Time: enter the date (in the format DD-MMM-YY) and time (in 24-hour format) of the performance check

Instrument ID: enter the instrument identifier (e.g., the serial number or other identifying number/name)

Internal Std: (used for 8260B and 8270C analysis) enter the name of the internal standard(s) used

Lab ID: enter the laboratory sample ID, if applicable

Lab Name: enter the laboratory name (e.g., Garland Labs, Inc.)

Lab Sample ID: enter the unique identifying number given to the sample by the laboratory that corresponds to the Field Sample ID

LCS ID: enter the unique identification number for the laboratory control sample such that the LCS could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., LCS960603)

Mass: enter the mass of the ion used for tuning (see QAPP Section 4.0)

Matrix: enter the sample matrix (e.g., water, soil)

Max. Holding Time: enter the maximum allowable holding time in days for specific analytes

MDL: enter the laboratory derived method detection limit

Mean %RSD: enter the mean of the RSDs of all analytes for those analytes not using a least squares regression or non-linear calibration

Method Blank ID: enter the unique identifying number given to the method blank (the same ID number will be found in the run sequence log, e.g., MB960603)

MS ID: enter the unique identification number for the matrix spike such that the MS could be traced back to the source material used for spiking (the same ID number will be found in the run sequence log e.g., MS960603)

Name: name of person completing data package

Parent Field Sample ID: enter the field sample ID of the parent sample (the sample spiked for the MS)

Parent Sample Result: enter the numeric result of the parent sample. Soil sample results must be entered only on a dry weight basis. If an analyte was not detected above the MDL, leave this column blank

Post/Command: enter USA/AMC/TACOM/RRAD/CSSA

Prime Contractor: enter the name of the prime contractor (e.g., RDS, Inc.)

Q or Qualifier: enter a "*" for any calibration that was not acceptable as per QAPP Section 4.0 and for any RFs not meeting minimum requirements for SPCCs and/or CCCs.

QC Sample ID: enter the unique identification number for each QC sample.

r: enter the correlation coefficient

%R: enter the percent recovery

% Relative Abundance: enter the percent relative abundance as the result of the tune

Replicate 1,2,3,4,5,6,7: enter the result of the replicate

RF1, RF2, RF3: enter the response factor corresponding to the standard with the same number

%RPD: enter relative percent difference between the sample and duplicate, as appropriate

%RSD: enter the per cent relative standard deviation of the response factors

RL: enter the project CSSA reporting limit as stated in the QAPP or approved variance for each analyte

Signature: signature of person completing data package

% Solids: enter the % solids

2nd Source ID: enter the unique identifier for the 2nd source standard such that the standard could be traced back to its source material (the same ID number will be found in the run sequence log, e.g., 2S960603)

Spike Added: enter the amount of spike added to the parent sample

Spiked Sample Result: enter the numeric result of the MS. Soil sample results must be entered only on a dry weight basis.

Std1, Std2, Std3: enter the concentration of the standard

Std ID: enter the unique identification number of the standard

Std. Dev.: enter the standard deviation of the seven replicates

Surrogate: enter the name of the surrogate(s) used

Time Analysis Started: enter the time the sample analysis was started in 24-hour format (e.g., 0900, 2130)
Time Analysis Completed: enter the time the sample analysis was completed in 24-hour format (e.g., 0900, 2130)

Time Held Analysis: enter the time in days elapsed between the date extracted and the date analyzed

Time Held Ext.: enter the time in days elapsed between the date collected and the date extracted

Title: title of person completing data package

Units: enter the appropriate units (e.g., µg/L, mg/kg, degrees C)

CHAIN-OF-CUSTODY FORM

COC#: enter a unique number for each chain-of-custody form

Ship to: enter the laboratory name and address

Carrier: enter the name of the transporter (e.g., FedEx) or hand carried

Air bill#: enter the air bill number or transporter tracking number (if applicable)

Project Name: enter the project name (e.g., Banks CSSA RI/FS)

Sampler Name: enter the name of the person collecting the samples

Sampler Signature: signature of the person collecting the samples

Send Results to: enter the name and address of the prime contractor

Field Sample ID: enter the unique identifying number given to the field sample (includes MS, MD, field duplicate, and field blanks)

Date: enter the year and date the sample was collected in the format M/D (e.g., 6/3)

Time: enter the time the sample was collected in 24-hour format (e.g., 0900)

Matrix: enter the sample matrix (e.g., water, soil)

Pres.: enter the preservative used (e.g., HNO3) or "none"

Filtered/Unfilt.: enter "F" if the sample was filtered or "U" if the sample was not filtered

# of Containers: enter the number of containers (i.e., jars, bottles) associated with the sample

MS: enter "X" if the sample is designated the MS

Analyses Requested: enter the method name of the analysis requested (e.g., SW6010B)

Comments: enter comments

Sample Condition Upon Receipt at Laboratory: enter any problems with the condition of any sample(s)

Cooler Temperature: enter the internal temperature of the cooler, upon opening, in degrees C

Special Instructions/Comments: enter any special instructions or comments

Released by (Sig): enter the signature of the person releasing custody of the samples

Company Name: enter the company name employing the person releasing/receiving custody

Received by (Sig): enter the signature of the person receiving custody of the samples

Date: enter the date in the format M/D/YY (e.g., 6/3/96) when the samples were released/received

Time: enter the time in 24-hour format (e.g., 0900) when the samples were released/received

CSSA
INORGANIC ANALYSES DATA PACKAGE

Analytical Method: _____ AB #: _____

Lab Name: _____ Contract #: _____

Post/Command: USA/AMC/TACOM/RRAD/CSSA Prime Contractor: _____

Field Sample ID	Lab Sample ID
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Comments:_____
_____
_____

I certify this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

CSSA
INORGANIC ANALYSES DATA SHEET 2
RESULTS

Analytical Method: _____ Preparation Method: _____ AB #: _____

Field Sample ID: _____ Lab Sample ID: _____

Matrix: _____ % Solids: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Date Collected: _____ Date Prepared: _____ Date Analyzed: _____

Concentration Units (mg/L or mg/kg dry weight): _____

Analyte	MDL	RL	Concentration	Dilution	Flag

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSES DATA SHEET 3
INITIAL MULTIPOINT CALIBRATION

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Instrument ID: _____ Concentration Units mg/L

Analyte	Std1	RF1	Std2	RF2	Std3	RF 3	r	Q

r — correlation coefficient
Note: Form I-3 may be expanded to include more than 3 standards and RFs.

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSES DATA SHEET 3A
MERCURY INITIAL MULTIPOINT CALIBRATION

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Instrument ID: _____ Concentration Units mg/L

Analyte	Std1	RF1	Std2	RF2	Std3	RF3	Std4	RF4	Std5	RF5	r	Q
Mercury												

r – correlation coefficient

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSES DATA SHEET 4
CALIBRATION VERIFICATION

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

2nd Source ID: _____ 2nd Source Verification Date:_____

Instrument ID: _____ ICV ID: _____

CCV #1 ID: _____ CCV #2 ID: _____

Concentration Units mg/L

	2nd Source Calibration Verification			Initial Calibration Verification			Continuing Calibration Verification					
Analyte	**Expected**	**Found**	**% D**	**Expected**	**Found**	**% D**	**Expected**	**Found 1**	**% D**	**Found 2**	**% D**	**Q**

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSES DATA SHEET 4 (Continued)
CALIBRATION VERIFICATION

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

CCV #3 ID: _____ CCV #4 ID: _____

CCV#5 ID: _____ Instrument ID: _____

Concentration Units mg/L

	Continuing Calibration Verification								Q
Analyte	**Expected**	**Found 3**	**%D**		**Found 4**	**%D**	**Found 5**	**%D**	

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSES DATA SHEET 5
BLANKS

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Initial Calibration Blank ID: _____ CCB #1 ID: _____

CCB #2 ID: _____ CCB #3 ID: _____

Method Blank ID:_____ Concentration Units (mg/L or mg/kg): _____

Analyte	**Initial Calibration Blank**	**Continuing Calibration Blank**			**Method Blank**	**RL**	**Q**
		1	2	3			

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSES DATA SHEET 6
LABORATORY CONTROL SAMPLE

Analytical Method:_____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

LCS ID: _____ Concentration Units (mg/L or mg/kg): ____

Analyte	Expected	Found	%R	Control Limits	Q

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSES DATA SHEET 7
MATRIX SPIKE SAMPLE RECOVERY

Analytical Method: _____ AB#:_____

Initial Calibration ID: _____ Initial Calibration Date: _____

Parent Field Sample ID: _____ MS ID: _____

Concentration Units (mg/L or mg/kg): _____

Analyte	Parent Sample Result	Spike Added	Spiked Sample Result	%R	Control Limits %R	Q

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSIS DATA SHEET 8
MATRIX DUPLICATES OR FIELD DUPLICATES

Analytical Method: _____ Units: _____

Parent Sample ID: _____ Duplicate Sample ID: _____

Analyte/Test	Sample Result	Duplicate Sample Result	%RPD	Acceptance Criteria	Flag

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSES DATA SHEET 9
HOLDING TIMES

Analytical Method: _____

Field Sample ID	Lab Sample ID	Date Collected	Date Analyzed	Max. Holding Time (days)	Time Held (days)	Q

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSES DATA SHEET 9A
HOLDING TIMES FOR TCLP

Field Sample ID	Lab Sample ID	Date Collected	Date prepared	Date Analyzed	Max. Prep. Holding Time (days)	Time Held for Prep. (days)	Max. Analy. Holding Time (days)	Time Held for Analy. (days)	Q

Comments: _____
_____
_____

CSSA
INORGANIC ANALYSES DATA SHEET 10
INSTRUMENT ANALYSIS SEQUENCE LOG

Analytical Method: _____ AB#_____

Instrument ID #: _____

Lab Sample ID/Std ID/Blank ID/QC Sample ID	Date Analysis Started	Time Analysis Started	Date Analysis Completed	Time Analysis Completed

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA PACKAGE

Analytical Method:_____ AB #: _____

Lab Name: _____ Contract #: _____

Post/Command: USA/AMC/TACOM/RRAD/CSSA Prime Contractor: _____

Field Sample ID	Lab Sample ID
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Comments: _____
_____
_____

I certify this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

CSSA
ORGANIC ANALYSES DATA SHEET 2
RESULTS

Analytical Method: _____ Preparation Method: _____ AB #: _____

Field Sample ID: _____ Lab Sample ID: _____ Matrix: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Date Collected:_____ Date Prepared: _____ Date Analyzed: _____

% Solids: _____ Concentration Units: (µg/L or mg/kg dry weight)

Analyte	MDL	RL	Concentration	Dilution	Confirm	Flag

Surrogate	Recovery	Control Limits	Qualifier

Internal Std	Qualifier

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 3A
INITIAL MULTIPOINT CALIBRATION

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date:_____

Instrument ID: _____ Concentration Units µg/L

Analyte	Std1	RF1	Std2	RF2	Std3	RF3	Std4	RF4	Std5	RF5	Std6	RF6	Std7	RF7

All analytes and surrogates should be listed in this form. SPCCs are marked with* and CCCs are marked with #

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 3B
INITIAL MULTIPOINT CALIBRATION

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Instrument ID: _____ Concentration Units µg/L

Analyte	%RSD	r	COD	Q

All analytes and surrogates should be listed in this form. SPCCs are marked with* and CCCs are marked with #

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 4
SECOND SOURCE CALIBRATION VERIFICATION

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date:_____

2nd Source ID: _____ Date of 2nd Source Analysis:_____

Instrument ID: _____ Concentration Units µg/L

Analyte	Expected	Found	%D	Q

All analytes and surrogates should be listed in this form. SPCCs are marked with* and CCCs are marked with #

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 5
CALIBRATION VERIFICATION

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Instrument ID: _____ CCV #1 ID: _____

CCV #2 ID: _____ CCV#ID: _____

Analyte	CCV#1		CCV #2		CCV #3		Q
	RF	%D	RF	%D	RF	%D	

All analytes and surrogates should be listed in this form. SPCCs are marked with* and CCCs are marked with #

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 6
BLANK

Analytical Method: _____ AB #: _____

Concentration Units (µg/L or mg/kg): _____ Method Blank ID: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Analyte	Method Blank	RL	Q

Surrogate	Recovery	Control Limits	Qualifier

Internal Std	Qualifier

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 7
LABORATORY CONTROL SAMPLE

Analytical Method:_____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

LCS ID: _____ Concentration Units (µg/L or mg/kg): _____

Analyte	Expected	Found	%R	Control Limits	Q

Surrogate	Recovery	Control Limits	Qualifier

Internal Std	Qualifier

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 8
MATRIX SPIKE SAMPLE RECOVERY

Analytical Method: _____ AB#:_____

Concentration Units (µg/L or mg/kg): _____ % Solids: _____

Parent Field Sample ID: _____ MS ID: _____

Analyte	Parent Sample Result	Spike Added	Spiked Sample Result	%R	Control Limits %R	Q

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 9
INTERNAL STANDARD AREA AND RT SUMMARY

Analytical Method: _____ AB#: _____

Instrument ID: _____ Date of Analysis: _____

	IS_1				IS_2				IS_3			
	Area Count	**Q**	**RT**	**Q**	**Area Count**	**Q**	**RT**	**Q**	**Area Count**	**Q**	**RT**	**Q**
MID STD. OF ICAL												
UPPER LIMIT												
LOWER LIMIT												
SAMPLE NO.												

AREA UPPER LIMIT = +100% of internal standard area.
AREA LOWER LIMIT = -50% of internal standard area.
RT UPPER LIMIT = +0.50 minutes of internal standard RT.
RT LOWER LIMIT = -0.50 minutes of internal standard RT.
Q Column is used to flag values outside QC limits with an asterisk.

CSSA
ORGANIC ANALYSIS DATA SHEET 10
MATRIX DUPLICATES OR FIELD DUPLICATES

Analytical Method: _____ Contract #: _____

Units: _____ Parent Sample ID: _____ Duplicate Sample ID: _____

Analyte/Test	Sample Result	Duplicate Sample Result	%RPD	Acceptance Criteria	Q

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 11
HOLDING TIMES

Analytical Method: _____ AB #: _____

Preparation Method: _____

Field Sample ID	Date Collected	Date Extracted	Max. Holding Time Ext.	Time Held Ext.	Date Analyzed	Max. Holding Time Analysis	Time Held Analysis	Q

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 12
INSTRUMENT ANALYSIS SEQUENCE LOG

Analytical Method: _____ AB#:_____

Instrument ID #: _____

Field Sample ID/Std ID/ Blank ID/QC Sample ID	Date Analysis Started	Time Analysis Started	Date Analysis Completed	Time Analysis Completed

Comments: _____
_____
_____

CSSA
ORGANIC ANALYSES DATA SHEET 13
INSTRUMENT PERFORMANCE CHECK
(BFB or DFTPP)

Analytical Method: _____ Instrument ID:_____

Compound: _____ Injection Date/Time: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Mass	Ion Abundance Criteria	% Relative Abundance	Q

CSSA
WET CHEM ANALYSES DATA PACKAGE

Analytical Method: _____ AB #: _____

Lab Name: _____ Contract #: _____

Post/Command: USA/AMC/TACOM/RRAD/CSSA Prime Contractor: _____

Field Sample ID	Lab Sample ID

Comments: _____
_____
_____

I certify this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

CSSA
WET CHEM ANALYSES DATA SHEET 2
RESULTS

Analytical Method:_____ AB #: _____

Field Sample ID: _____ Lab Sample ID: _____

Matrix: _____ % Solids: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Date Collected: _____ Date Prepared: _____ Date Analyzed: _____

Concentration Units: mg/L, mg/kg dry weight, other units)

Analyte	MDL	RL	Concentration	Dilution	Flag

Comments: _____
_____
_____

CSSA
WET CHEM ANALYSES DATA SHEET 3
INITIAL MULTIPOINT CALIBRATION

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

Instrument ID: _____ Concentration Units: mg/L

Analyte	Std1	RF1	Std2	RF2	Std3	RF3	r	Q

Note: This form may be expanded to accommodate additional standards and response factors.
r – correlation coefficient

Comments: _____
_____
_____

CSSA
WET CHEM ANALYSES DATA SHEET 4
CALIBRATION VERIFICATION

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date: _____

2nd Source ID: _____ 2nd Source Verification Date: _____

Instrument ID: _____ CCV #1 ID: _____

CCV #2 ID: _____ Concentration units: mg/L

	2nd Source Calibration Verification			Continuing Calibration Verification					
Analyte	Expected	Found	%D	Expected	Found 1	%D	Found 2	%D	Q

Comments: _____
_____

CSSA
WET CHEM ANALYSES DATA SHEET 5
BLANKS

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration Date:_____

Calibration Blank ID: _____ Method Blank ID: _____

Concentration Units: _____

Analyte	Calibration Blank	Method Blank	RL	Q

Comments: _____
_____
_____

CSSA
WET CHEM ANALYSES DATA SHEET 6
LABORATORY CONTROL SAMPLE

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration date: _____

LCS ID: _____ Concentration Units (mg/L or mg/kg): _____

Analyte	Expected	Found	%R	Control Limits	Q

Comments: _____

CSSA
WET CHEM ANALYSES DATA SHEET 7
MATRIX SPIKE SAMPLE RECOVERY

Analytical Method: _____ AB #: _____

Initial Calibration ID: _____ Initial Calibration date: _____

Parent Field Sample ID: _____ MS ID: _____

% Solids: _____ Concentration Units (mg/L or mg/kg): _____

Analyte	Parent Sample Result	Spike Added	Spiked Sample Result	%R	Control Limits %R	Q

Note: The parent sample result column should be left blank if the result is at or below MDL

Comments: _____
_____
_____

CSSA
WET CHEM DATA SHEET 8
MATRIX DUPLICATES OR FIELD DUPLICATES

Analytical Method: _____

Units: _____ Parent Sample ID: _____ Duplicate Sample ID: _____

Analyte	Sample Result	Duplicate Sample Result	%RPD	Acceptance Criteria	Q

Comments: _____
_____
_____

CSSA
WET CHEM ANALYSES DATA SHEET 9
HOLDING TIMES

Analytical Method:_____

Field Sample ID	Date Collected	Date Analyzed	Max. Holding Time (days/hours)	Time Held (days/hours)	Q

Note: For short holding times of collection and analysis should be included in appropriate columns. If the site of collection and analytical laboratory are in different geographical time zones, appropriate compensation should be made for time differential

Comments: _____
_____
_____

CSSA
WET CHEM ANALYSES DATA SHEET 10
INSTRUMENT ANALYSIS SEQUENCE LOG

Analytical Method: _____ AB#:_____

Instrument ID #: _____

Field or lab sample ID/Std ID/ Blank ID/QC Sample ID	Date Analysis Started	Time Analysis Started	Date Analysis Completed	Time Analysis Completed

Comments: _____
_____
_____

CSSA
MDL STUDY REPORT FORM

Analytical Method: _____ Analysis Date:_____

Analysis Date: _____ Instrument ID: _____

Concentration Units:_____

Analyte	Amt. Spiked	Replicate							Std. Dev.	MDL
		1	2	3	4	5	6	7		

MDL FORM Method _____
Page ____ of ____

CSSA
CHAIN-OF-CUSTODY RECORD

COC#: _____

Ship to:	Project Name:	Send Results to:
	Sampler Name:	
Carrier: Airbill #:	Sampler Signature:	

Field Sample ID	Date	Time	Matrix	Pres	Filtered/Unfilt.	# of Containers	Analyses Requested											Comments

Sample Condition Upon Receipt at Laboratory: Cooler temperature:
Special Instructions/Comments:

#1 Released by: (Sig)	Date:	#2 Released by: (Sig)	Date:	#3 Released by: (Sig)	Date:
Company Name:	Time:	Company Name:	Time:	Company Name:	Time:
#1 Received by: (Sig)	Date:	#2 Received by: (Sig)	Date:	#3 Received by: (Sig)	Date:
Company Name:	Time:	Company Name:	Time:	Company Name:	Time:

6.7 Data Storage and Retrieval

Hardcopy and electronic data shall be archived in project files and on electronic archive tapes for the duration of the project or a minimum of five years, whichever is longer.

The laboratory shall maintain electronic and hardcopy records sufficient to recreate each analytical event conducted pursuant to the Statement of Work (SOW). The minimum records the laboratory shall keep contain the following: (1) COC forms; (2) initial and continuing calibration records including standards preparation traceable to the original material and lot number; (3) instrument tuning records (as applicable); (3) method blank results; (4) IS results; (5) surrogate spiking records and results (as applicable); (6) spike and spike duplicate records and results; (7) laboratory records; (8) raw data, including instrument printouts, bench work sheets, and/or chromatograms with compound identification and quantitation reports; (9) corrective action reports; (10) other method and project-required QC samples and results; and (11) laboratory-specific written SOPs for each analytical method and QA/QC function in place at the time of analysis of project samples.

7.0 ASSESSMENT AND OVERSIGHT

Technical systems and performance audits shall be performed as independent assessments of sample collection and analysis procedures. Audit results will be used to evaluate the ability of a contractor to: (1) produce data that fulfill the objectives established for the program; (2) comply with the QC criteria; and (3) identify any areas requiring corrective action. The systems audit is a qualitative review of the overall sampling or measurement system, while the performance audit is a quantitative assessment of a measurement system. Audit guidance can be found in the current version of the *HQ AFCEE Technical Services Quality Assurance Program.* Full data validation is also a quantitative check of the analytical process, where all documentation and calculations are evaluated and verified. Data validation is discussed in Section 6.0.

7.1 State/Federal Project Audits

Audits by various state and federal agencies are commonly conducted for the laboratories that will analyze project samples. Audit reports from these agencies shall be reviewed by the prime contractor to determine whether data produced by the analytical contractor fulfill the objectives of the program.

Audit findings shall be transmitted to the prime contractor and to CSSA. The prime contractor shall review the audit findings and provide a written report to CSSA. This report shall include the recommended corrective actions or procedures to correct the deficiencies identified during the state/federal audits(s). The audit results and discussion shall be incorporated into the QA report for each sampling effort.

7.2 Technical Systems Audits

A technical systems audit is an on-site, qualitative review of the sampling or analytical system to ensure that the activity is being performed in compliance with the SAP specifications. Prior to the initiation of a project, the prime contractor shall audit sampling and field procedures, and the analytical laboratories. In addition, a laboratory systems audit may be performed by CSSA if previous audit reports indicate corrective actions are outstanding, a recent audit has not been conducted, or quality concerns have arisen based upon the use of that laboratory for other projects. The laboratory systems audit results will be used to assess the prime contractor's oversight and to review laboratory operations and ensure the technical procedures and documentation are in place and operating to provide data that fulfill the project objectives and to ensure outstanding corrective actions have been addressed.

Critical items for a laboratory or field systems audit include: (1) sample custody procedures; (2) calibration procedures and documentation; (3) completeness of data forms, notebooks, and other reporting requirements; (4) data review procedures; (5) data storage, filing, and record keeping procedures; (6) QC procedures, tolerances, and documentation; (7) operating conditions of facilities and equipment; (8) documentation of training and maintenance activities; (9) systems and operations overview; and (10) security of laboratory automated systems.

Critical items for a sampling systems audit include: (1) calibration procedures and documentation for field equipment; (2) documentation in field logbooks and sampling data sheets; (3) organization and minimization of potential contamination sources while in the field; (4) proper sample collection, storage, and transportation procedures; and (5) compliance with established COC and transfer procedures.

After each on-site audit, a debriefing session will be held for all participants to discuss the preliminary audit results**.** The auditor will then complete the audit evaluation and submit an audit report including observations of the deficiencies and the necessary recommendations for corrective actions (RCAs) to the prime contractor. Compliance with the specifications presented in the SAP will be noted and noncompliance or deviations shall be addressed in writing by the prime contractor to CSSA with corrective actions and a timeframe for implementation of the corrective actions. Follow-up audits will be performed prior to completion of the project to ensure corrective actions have been taken.

7.3 Performance Evaluation Audits

Performance evaluation audits quantitatively assess the data produced by a measurement system. A performance audit involves submitting performance evaluation samples for analysis for each analytical method used in the project. The PE samples should contain analytes of interest for the project, preferably in the anticipated concentrations of field samples. The prime contractor shall submit PE samples once per project. The performance audit answers questions about whether the measurement system is operating within control limits and whether the data produced meet the analytical QA specifications.

PE samples should be obtained from commercial vendors and must have statistically established acceptance criteria for all analytes. Generally, PE samples supplied by commercial vendors for laboratory certification in the Water Pollution (WP) and Water Supply (WS) programs are acceptable. However, some of the PE samples may have to be diluted by the vendor prior to shipping to the laboratory for analysis.

CSSA may submit double blind PE samples as part of a field-sampling event. The prime contractor will send these samples to the laboratory as unknown samples. The prime contractor shall submit the results of the PE samples and the associated calibration and quality control data to CSSA. The results will be evaluated by an independent contractor and shall be communicated to CSSA.

The critical elements for review of PE results include: (1) correct identification and quantitation of the PE sample analytes; (2) accurate and complete reporting of the results; and (3) measurement system operation within established control limits for precision and accuracy.

The prime contractor shall evaluate the results of the PE samples as soon as they are received from the laboratory. The concentrations reported for the PE samples shall be compared to the known or expected concentrations spiked in the samples. The percent recovery shall be calculated and the results compared to the accuracy criteria provided by the commercial vendor.

If the accuracy criteria are not met, the laboratory shall investigate the cause of the failure and submit a corrective action report to the prime contractor. The prime contractor shall communicate the findings to the Environmental Officer of CSSA immediately after evaluation.

7.4 Magnetic Tape Audits

Magnetic tape audits involve the examination of the electronic media used by the analytical laboratory and by the prime contractor to collect, analyze, report, and store data. These audits are used to assess the authenticity of the data generated, and assess the implementation of good automated laboratory practices. CSSA may perform magnetic tape audits of the laboratories or of the prime contractors when warranted by project PE results, on-site audit results, or by other state/federal investigations.

7.5 Performance Evaluation Sample Programs

All laboratories shall participate in the WS and WP studies' PE programs or equivalent programs required for state certifications. Satisfactory performance in these PE programs also demonstrates proficiency in methods used to analyze CSSA samples. The laboratory shall document the corrective actions to unacceptable PE results to demonstrate resolution of the problems.

8.0 PREVENTIVE MAINTENANCE

A preventive maintenance program shall be in place to promote the timely and effective completion of a measurement effort. The preventive maintenance program is designed to minimize the downtime of crucial sampling and/or analytical equipment due to unexpected component failure. In implementing this program, efforts are focused in three primary areas: (1) establishment of maintenance responsibilities; (2) establishment of maintenance schedules for major and/or critical instrumentation and apparatus; and (3) establishment of an adequate inventory of critical spare parts and equipment.

8.1 Maintenance Responsibilities

The respective facility managers assume maintenance responsibilities for equipment and instruments. The managers then establish maintenance procedures and schedules for each major equipment item. This responsibility may be delegated to laboratory personnel, although the managers retain responsibility for ensuring adherence to the prescribed protocols.

8.2 Maintenance Schedules

The effectiveness of any maintenance program depends to a large extent on adherence to specific maintenance schedules for each major equipment item. Other maintenance activities are conducted as needed. Manufacturers' recommendations provide the primary basis for the established maintenance schedules, and manufacturers' service contracts provide primary maintenance for many major instruments (e.g., GC/MS instruments, AA spectrometers, and analytical balances).

8.3 Spare Parts

Along with a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment downtime. The inventory includes those parts (and supplies) that are subject to frequent failure, have limited useful lifetimes, or cannot be obtained in a timely manner should failure occur.

Field sampling task leaders and the respective laboratory managers are responsible for maintaining an adequate inventory of spare parts. In addition to spare parts and supply inventories, the contractor shall maintain an in-house source of backup equipment and instrumentation.

8.4 Maintenance Records

Maintenance and repair of major field and laboratory equipment shall be recorded in field or laboratory logbooks. These records shall document the serial numbers of the equipment, the person performing the maintenance or repairs, the date of the repair, the procedures used during the repair, and proof of successful repair prior to the use of the equipment.

9.0 CORRECTIVE ACTION

Anytime an error, deficiency, or deviation from specified criteria occurs in the field or the laboratory, it is defined as an out-of-control or non-conformance event. The contractor or the subcontractor must take necessary actions to resolve these events and bring the system back into control. These actions are defined as corrective actions.

Corrective actions must be carried out whenever acceptance criteria are exceeded in laboratory activities. For all the laboratory analytical methods, the acceptance criteria for calibration, quality control and reporting limits are provided in respective tables in Section 4.0 of this QAPP. If acceptance criteria are not met, a corrective action is warranted.

Some examples of laboratory non-compliance are:

- Missing holding times
- Uncorrected initial calibration or calibration verification events
- Uncorrected recoveries of laboratory control samples, surrogates, internal standards, and other quality control parameters.

The laboratory director or the designated representative shall communicate all out-of-control and/or non-compliance events immediately to the project manager (if corrective actions taken by the laboratory proved ineffective or if no corrective actions were taken). The project manager shall review each event with the respective field and laboratory staff and exercise professional judgment in recommending a course of action. CSSA and AFCEE must approve the recommended actions. In the absence of the AFCEE team chief, the designated CSSA chemist will discuss the event/issue and generate response for AFCEE team chief's approval. The approval scheme is presented in Figure 9.0

During field operations, all activities must be carried out according to the approved field-sampling plan. If deviations from the approved plan are noted, the prime contractor must redo the activity according to requirements.

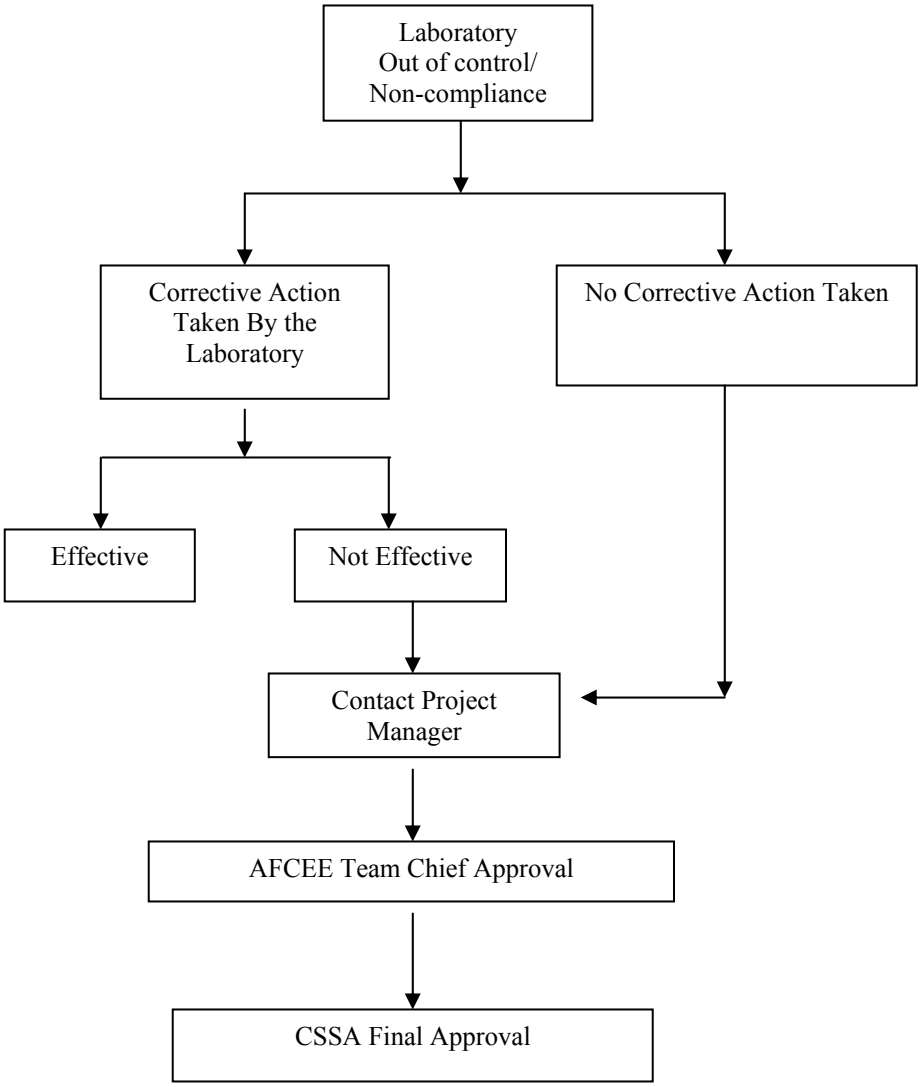

Figure 9.0 Corrective Action Approval Scheme

9.1 Corrective Action System

CSSA requires the prime contractor to have a Corrective Action System (CAS) to describe the resolution of field and laboratory out-of-control events associated with a particular project. The CAS should include the following elements:

- The ability to identify random and/or systematic out-of-control events
- The procedures and responsible parties for carrying out corrective actions
- Identifying the responsible parties for approving corrective actions
- Establishing proof that corrective actions were effective and the affected parameters are back in control
- Methods to document each corrective action using a Corrective Action Report (see Section 9.2)

9.2 Corrective Action Report

Problems requiring corrective action in the laboratory shall be documented by the use of a Corrective Action Report. The QA coordinator or any other laboratory member can initiate the corrective action request in the event QC results exceed acceptance limits, or upon identification of some other laboratory problem. Corrective actions can include reanalysis of the sample(s) affected, resampling and analysis, or a change in procedures, depending upon the severity of the problem.

Corrective action reports should include what went wrong in the analytical system; how it was corrected; and after corrective action, proofs that the system was back in control. The laboratory section supervisor and the QA officer must approve corrective action reports. All corrective action reports should be submitted, as part of the data package, to the prime contractor and to CSSA. The content of the corrective actions should be covered in the case narrative prepared by the laboratory and sent to the prime contractor.