SCS ENGINEERS

Sampling and Analysis Plan

Assessor Parcel Numbers 644-042-02 and -10 Chula Vista, California Grant Number: BF-969433-01

Presented to:

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June 6, 2012
Project Number: 11012021.07

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US EPA Waste Programs WST-5
75 Hawthorne Street
San Francisco, California 94105

Subject: **Sampling and Analysis Plan**

Site: **Assessor Parcel Numbers 644-042-02 and -10**
Chula Vista, California

Dear Ms. Nurre:

SCS Engineers (SCS) is pleased to present this Sampling and Analysis Plan (SAP) for soil investigation at the Site. This SAP was prepared by SCS in general accordance with the Agreement for Professional Services and Assistance (Contract) between SCS and the City of Chula Vista (Client). The Contract was fully executed on November 25, 2008.

If you have any questions regarding this SAP, please do not hesitate to call our office at (858) 571-5500.

Sincerely,

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Acronyms and Abbreviations

ASTM	American Society for Testing and Materials
APN	Assessor parcel number
ARAR	Applicable or relevant and appropriate requirement
BTEX	Benzene, toluene, ethylbenzene, and total xylenes
CERCLA	Comprehensive Environmental Response, Cleanup, and Liability Act
CFR	Code of Federal Regulations
CHHSL	California Human Health Screening Levels
CLP	Contract laboratory program
COCs	Constituents of concern
CWA	Clean Water Act
DQA	Data quality assessment
DQI	Data quality indicators
DQO	Data quality objectives
EPA	U.S. Environmental Protection Agency
ESA	Environmental site assessment
ESL	Environmental Screening Levels
FOCs	Features of concern
FSP	Field sampling plan
GC/MS	Gas chromatography and mass spectrometry
IDW	Investigation-derived waste
LCS	Laboratory control sample
MDL	Method detection limit
MQO	Measurement quality objective
MS/MSD	Matrix spike and matrix spike duplicate
mg/kg	Milligrams per kilogram
µg/kg	Micrograms per kilogram
mg/L	Milligrams per liter
µg/L	Micrograms per liter
PARCCS	Precision, accuracy, representativeness, completeness, comparability, and sensitivity
PCBs	Polychlorinated biphenyls
PE	Performance evaluation
PRG	Preliminary remediation goal
PRQL	Project-required quantitation limit
QA	Quality assurance
QA/QC	Quality assurance/quality control
QAPP	Quality assurance project plan
QC	Quality control
QL	Quantitation limit
RCRA	Resource Conservation and Recovery Act
REC	Recognized environmental condition
RPD	Relative percent difference
%R	Percent recovery
SAP	Sampling and analysis plan (an integrated FSP and QAPP)
SOP	Standard operating procedures
SOW	Statement of work
SVOCs	Semi-volatile organic compounds
UST	Underground storage tank
VOCs	Volatile organic compounds

1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) has been prepared for approval by the United States Environmental Protection Agency (EPA) pursuant to the City of Chula Vista's EPA Community-Wide Brownfield Assessment Grant for Hazardous Substances. The purpose of the proposed soil sampling is to:

- Assess the extent and thickness of the waste.
- Characterize the waste so that the waste characterization information can be incorporated into closure design and planning.
- Assess whether residual pesticides are present in the shallow surface soil at concentrations that might pose risks to construction workers and others.

Pending approval by the EPA, the scope of work (SOW) described in this SAP will be conducted in the third quarter of 2012.

1.1 SITE NAME OR SAMPLING AREA

The area to be sampled will be referred to as the "Site" throughout this SAP.

1.2 SITE NAME OR SAMPLING AREA LOCATION

The Site is an approximately 4.5-acre property that lies on the south bank of the Otay River in Chula Vista, California (Figure 1), and is part of a larger property made up of two parcels. The Site is owned by two parties: the Shinohara Family Trust #2 is the owner of the parcel identified by assessor parcel number (APN) 644-042-10; and the City of Chula Vista is the owner of APN 644-042-02. The Site is known as the Shinohara II Burn Site, where waste from a burn dump was historically deposited. The City owns approximately 1 acre of the Site, and the remainder is owned by the Shinohara Family Trust (Figure 2).

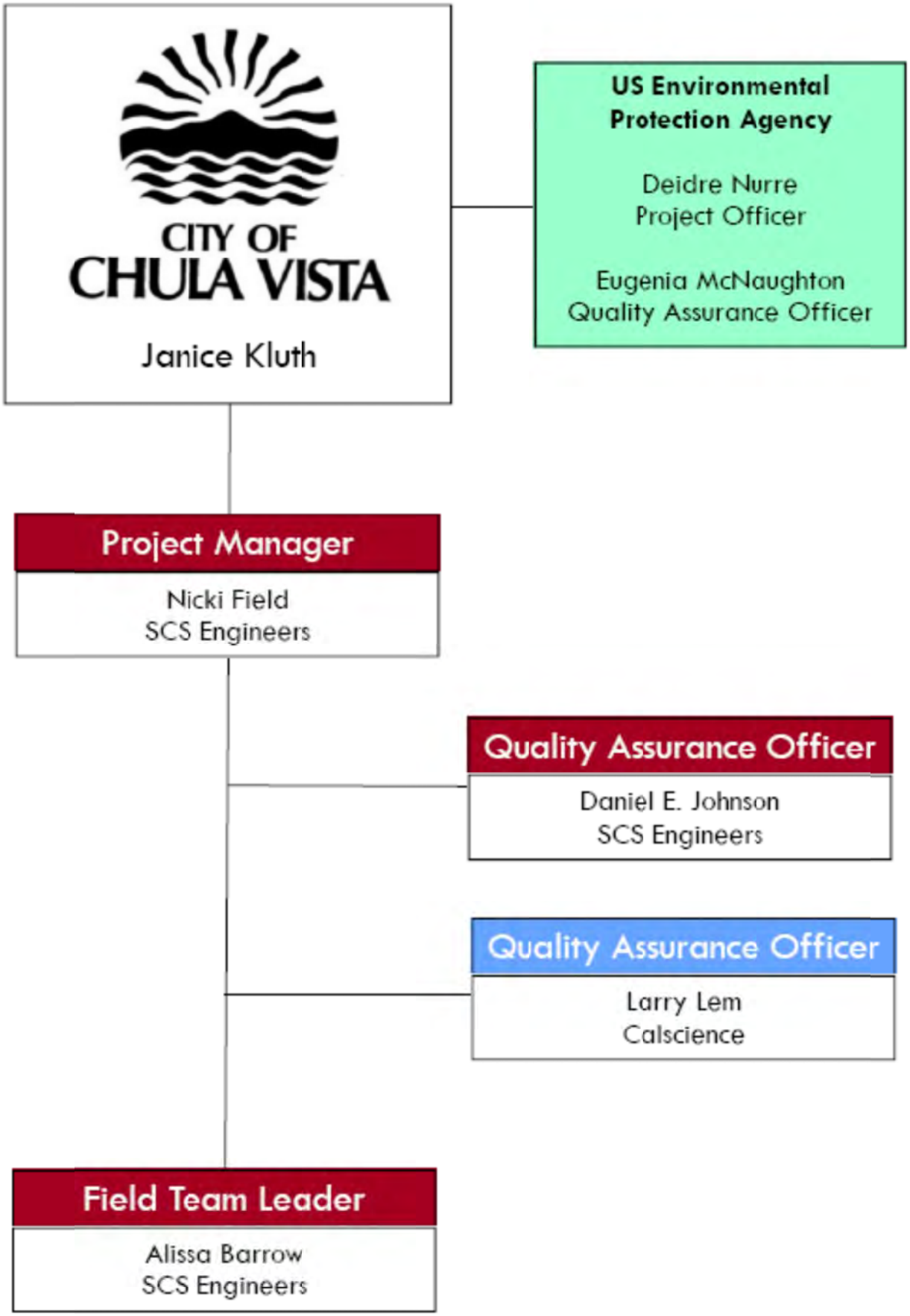
The Site is bounded to the north and east by the Otay River, to the south by an access road associated with an adjacent residential development, and to the west by powerlines.

1.3 RESPONSIBLE AGENCY

The Responsible Agency is the City of Chula Vista located at 276 Fourth Avenue in Chula Vista, California 91910.

1.4 PROJECT ORGANIZATION

Title	Name	Contact Information	Responsibilities
EPA Project Officer	Deirdre Nurre US EPA Waste Programs	415-947-4290 Nurre.Deirdre@epamail.epa.gov	Review and approve QA documents, provide technical assistance
EPA Quality Assurance Officer (QAO)	Eugenia McNaughton, PhD EPA Region 9	415-972-3411 Mcnaughton.Eugenia@epa.gov	Coordinate preparation and review of QA documents, provide technical assistance
Grantee Project Manager	Janice Kluth City of Chula Vista	619-691-5022 JKluth@chulavistaca.gov	Review, authorize, and facilitate implementation of the proposed scope of work
Contractor Project Manager	Nicki Field SCS Engineers	858-571-5500 nfield@scsengineers.com	Prepare SAP and oversee overall project implementation
Contractor QAO	Daniel Johnson SCS Engineers	858-571-5500 djohnson@scsengineers.com	Oversee the implementation of the SAP in a QA role and peer reviewer
Contractor Field Team Leader	Alissa Barrow SCS Engineers	858-571-5500 abarrow@scsengineers.com	Lead field investigation
Laboratory QAO	Larry Lem Calscience Environmental Laboratories, Inc.	714-895-5494 x237 llem@calscience.com	Oversee the implementation of laboratory Quality Systems Manuals for project sample handling, analysis, and data reporting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2.0 BACKGROUND

2.1 SITE OR SAMPLING AREA DESCRIPTION

The Site is an approximately 4.5-acre property that lies on the south bank of the Otay River in Chula Vista, California (Figure 1), and is part of a larger property made up of two parcels. The Site is owned by two parties: the Shinohara Family Trust #2 is the owner of APN 644-042-10; and the City of Chula Vista is the owner of APN 644-042-02. The Site is known as the Shinohara II Burn Site, where waste from a burn dump was historically deposited. The City owns approximately 1 acre of the Site, and the remainder is owned by the Shinohara Family Trust (Figure 2).

The Site is bounded to the north and east by the Otay River, to the south by an access road associated with an adjacent residential development, and to the west by powerlines.

The following table provides information for the Site parcels from the Win2 property information database.

APN	Address	Area	Description
644-042-02	Not reported	11.02 acres total, Site portion approximately 1 acre	Not reported
644-042-10	Not reported	9.11 acres total, Site portion approximately 3.5 acres	Vacant land

The Shinohara II Burn Site portion of the Site is fenced, and signage is posted to discourage trespassing. The burn dump area is covered with soil, though coverage is minimal. Ash waste, broken ceramic, melted glass, and metal are visible on the ground's surface.

2.2 OPERATIONAL HISTORY

The following table presents a summary of the Site ownership and operations/land use for each parcel over time.

Years	Reported Site Owners	Interpreted Site Tenants	Interpreted Site Use
APN 644-042-10			
1928 to 1938	Kimiko Kuratomi	None	Vacant undeveloped land
1938 to 1953	Jimmie H. and Charles T. Shinohara	None	
1953 to 1965	Jimmie H. and Judi Shinohara	None	
1965-1973	Jimmie H. and Judi Shinohara, trustees under Trust dated October 21, 1987	None	

Years	Reported Site Owners	Interpreted Site Tenants	Interpreted Site Use
1978	Jimmie H. and Judi Shinohara, trustees under Trust dated October 21, 1987	None	Appears that fresh fill had recently been imported in aerial photograph from this year
1981 to 1989	Jimmie H. and Judi Shinohara, trustees under Trust dated October 21, 1987	None	Agricultural
2003 to present	Jimmie H. and Judi Shinohara, trustees under Trust dated October 21, 1987	None	Vacant land
APN 644-042-02			
1928 to 1937	John Mosto	None	Vacant undeveloped land
1937 to 1954	Merril L. Nelson and Paul R. Sloan	None	Vacant undeveloped land
1954 to 1963	Elma Nelson	None	Vacant undeveloped land
1964 to 1966	Nelson and Sloan, a co-partnership	None	Mining activities
1970 to 1991	Nelson and Sloan, a co-partnership	None	Vacant land
1967 to 1970	N&S Materials, Inc.	None	Vacant land
1973 to 1983	N&S Materials, Inc.	Unknown	Site use unknown. Structures/ buildings are evident in the southeastern portion of the parcel in the 1973, 1974, 1978, 1980, and 1983 aerials, possible out-buildings in support of agricultural activities to the south.
1991 to present	Chula Vista Redevelopment Agency/City of Chula Vista	None	Vacant land

2.3 PREVIOUS INVESTIGATIONS/REGULATORY INVOLVEMENT

The Site is a known burn site and has an active case (SWIS number 37-CR-0075) with the County of San Diego Solid Waste Local Enforcement Agency (LEA). SCS recently requested and reviewed regulatory agency files for the Site as part of a Phase I Environmental Site Assessment (Phase I).[1] The County of San Diego Department of Environmental Health (DEH)

1 *Phase I Environmental Site Assessment and Regulatory Agency File Review and Document Compilation, Assessor's Parcel Numbers, 644-042-02 and -10, Chula Vista, California*, by SCS Engineers, dated January 23, 2012.

reported that it does not have any files associated with the Site. The LEA and CalRecycle (formerly California Integrated Waste Management Board) both had extensive files for the Site that were reviewed by SCS personnel in October and November 2011. In addition, SCS also reviewed numerous Client-provided documents associated with the Site.

Based on the regulatory records review, the Site was never operated as a burn dump; however, burn ash was brought to the Site as fill material in the late 1970s when the owner of the Shinohara parcel reportedly allowed fill to be imported from various sources to create a level surface. In particular, fill was placed on the southern portion of the Shinohara parcel, which had been approximately 30 feet below the elevation of the rest of the Shinohara property. It was reported that fills materials imported to the Site included burn ash from the former "South Bay Burn Site" (formerly located approximately 0.25 mile southwest of the Site at I-805 and Palm) during the construction of Interstate 805. Approximately 850,000 cubic yards of burn ash-containing soil were reported to have been exported from the South Bay Burn Site in 1978 and used in the vicinity at various construction sites as artificial fill. It is unknown how many cubic yards of this material may have been placed at the Site.

The Site was identified as a burn ash site by the County of San Diego LEA in 1992. Initially, the Site was included in the larger Shinohara Farms property burn ash site, which included the properties to the north and south of the Otay River. In 1998, the site was divided into Shinohara I (located north of the Otay River) and the Site (Shinohara II) (located south of the Otay River).

The LEA has conducted quarterly inspections of the Site since 1998. Burn ash is reported to be present at the surface of the Site, and Site security has been an ongoing issue. The LEA issued an Official Notice on March 1, 2007, which directed the City of Chula Vista (City) to address the following issues to be completed by the stated dates: (1) Site security and signage by May 1, 2007; (2) Cover installation (a minimum 2-foot cap of clean soil) by July 1, 2007; and (3) Drainage and erosion control measures by August 1, 2007.

A limited health risk assessment[2] was conducted for the Site in 2005 during construction of the Dennery Ranch housing development located adjacent to the south and southeast of the Site. In pre-development meetings, the potential for burn ash from the Site impacting future residents of Dennery Ranch was identified as a concern. The limited health risk assessment focused on potential risk to residents at Dennery Ranch. Environ conducted a screening level air dispersion analysis to calculate the maximum lead concentration in potential airborne dust that would not pose a significant health risk to future Dennery Ranch residents.

Environ used data from the Shinohara I Burn Site and the Dennery Ranch to perform the risk assessment because no subsurface investigation had been conducted for the Site, and therefore, no Site-specific data were available. Since the burn ash at Shinohara I, the Dennery Ranch, and the Site were reported to have come from the same source, the South Bay Burn Site, Environ stated that "the lead concentrations found in the burn ash at the Shinohara I and Dennery Ranch sites should be similar to, and representative of, the lead concentrations in the burn ash present at the Shinohara II burn ash site."

2 *Revised Report, Pardee Homes – Dennery Ranch Village 2 and 3 Developments, Off-Site Burn Ash Health Risk Assessment*, by Environ, dated August 8, 2005.

Air dispersion modeling was conducted and found that the maximum allowable lead concentration in total suspended particulates (TSP) (dust) at the Site that would not cause an increase in lead concentrations greater than 133 milligrams per kilogram (mg/kg) at the Dennery Ranch was 7,800 mg/kg.

Based on the available lead data from Shinohara I and the Dennery Ranch, the mean lead concentration present for the Site was estimated to be 925 mg/kg, and the 95 percent upper confidence limit (UCL) was estimated to be 1,132 mg/kg. Environ compared these numbers to the maximum allowable lead concentration in TSP at the Site of 7,800 mg/kg and concluded that burn ash present at Shinohara II Burn Site does not pose significant health risk to future residents of the Dennery Ranch property and that no additional assessments will be necessary. In a response letter[3] dated January 19, 2006, the Department of Toxic Substances Control (DTSC) agreed with the findings in the Environ report, stating that the Site "does not pose a significant health threat to future residents of the proposed DRV (Dennery Ranch Village) 2 and 3 development."

Based on the available data, SCS believes that further waste characterization will confirm the finding of minimal risk and that the Shinohara II Site will be able to be closed under regulation of the San Diego County LEA and the San Diego Regional Water Quality Control Board (RWQCB).

The City and the Shinoharas have taken steps to address other LEA directives, including installing a security fence and signs to restrict public access, as well as installing straw waddles for erosion control. In addition, the City has made repairs to the fence on numerous occasions; however, security continues to be an issue at the Site.

In May 2010, the LEA was notified that Ms. Judy Shinohara, the primary controller of the trust that owns the Shinohara portion of the Site, had passed away in April 2009 and that the three daughters of Ms. Shinohara had disclaimed any interest in the property held by the trust and had declined to act as trustees for the Trust.

A Notice and Order was issued by the LEA on September 14, 2010 which required the submittal of a workplan to complete corrective actions (i.e., installation of cover) and bi-monthly status reports and to either remove all of the waste at the Site or to install a minimum 2-foot thick final cover and initiate a Site maintenance and monitoring program.

To comply with the LEA's directives and as the first step in the process to design and install an appropriate cover and obtain closure for the Site, SCS Engineers (SCS) conducted two preliminary field investigations of the property: one on August 5, 2009, and the other on September 29, 2009.

SCS staff recorded ground elevations and pothole locations by means of a hand-held global positioning system (GPS) survey data collector (Figure 2). Depth of cover was evaluated by

3 *Dennery Ranch Village 2 and 3 Residential Development, Palm Avenue and Dennery Ranch Road, San Diego, San Diego County – Final Border Zone Property Determination,* from DTSC, dated January 19, 2006.

digging with a hand auger until evidence of burn ash or refuse was observed. The findings of these field investigations were described in a report/workplan (March 2012 Report).[4]

The following Site conditions were encountered during the visits:

- "Cover was found to vary from 0 to 12 inches in thickness throughout the Site.
- The western edge of the fill may be eroded by a natural drainage course, which flows northward on the west side of the burn dump towards the river.
- Low points exist on both the Shinohara- and City-owned parcels.
- The slope along the river has no embankment protection.
- There is evidence of protruding waste (e.g., concrete and construction waste) along the river bank, which has cut into the fill in the past.
- The bank varies in height up to 12 feet above the river channel on an approximate slope of 2:1 (horizontal: vertical).
- Limits of visible waste appear to be contained within known landfill limits as defined by the City and Shinohara fence on the south and by drainage courses that delineate the eastern, western, and northern boundaries. The approximate limits of waste, based on the Site reconnaissance and limited hand excavation, are shown on Figure 2."

In the March 2012 Report, SCS made the following preliminary recommendations regarding closure design and construction at the Shinohara II Site:

- "Consolidate, where economically feasible, wastes or portions of wastes located in unsuitable areas (such as river banks and along property lines) in appropriate areas and depths.
- Place a minimum cover of 24 inches of clean compacted soil over the existing refuse and burn ash fill. Areas to receive cover placement will be stripped of all existing vegetation.
- Grade compacted soil cover such that a minimum grade of 3 percent can be achieved.
- Provide engineered storm water runoff collection and conveyance facilities to prevent future ponding of storm water over the burn dump.
- Provide improved drainage channel walls or slope armoring, and scour protection along natural drainage courses, to prevent washout of the landfill from a 100-year, 24-hour storm event.
- Provide erosion control and seeding to prevent future erosion of the final cover.

4 *Site Investigation Report and Workplan for Additional Assessment, Shinohara II Burn Site, Portions of Assessor Parcel Numbers 644-042-02 and -10, Chula Vista, California, SWIS Number: 37-CR-0075*, by SCS Engineers, dated March 30, 2012.

- Provide final cover planting to sustain natural erosion protection compatible with the surrounding biota and to be consistent with the proposed end-use of the property as specified by the City's General Plan and Multiple Species Conservation Program (MSCP).

- Provide a maintenance and inspection plan for the Site during the postclosure period (typically 10 to 30 years, as determined by the regulatory authority)."

SCS based our recommendations for a closure cap on the assumption of minimal risk, which will be assessed during this proposed waste characterization study. The LEA approved the work proposed in the March 2012 Report on April 12, 2012. A copy of the workplan approval letter is included in Appendix A.

In addition to the issues associated with the waste at the Shinohara II Burn Site, the Phase I also identified potential recognized environmental conditions (RECs) associated with historical agricultural and mining/mining reclamation uses at the Site and historical industrial land uses on adjacent properties. SCS concluded in the Phase I that:

"With the possible exceptions below, there is a low likelihood that RECs are present at the Site as a result of the current or historical Site land use or from a known and reported off-site source:

- A review of aerial photographs revealed that undocumented fill was imported to the Site in the 1970's. Based on regulatory file reviews, burn ash material was reportedly imported to the Site from the South Bay Burn Dump Site in the late 1970's. The 1973 historic aerial from the San Diego County Department of Cartographic Services shows construction of Interstate 805 to the west of the Site, as well as stockpiles (interpreted to be related to the 805 construction) located on the Shinohara-owned portion of the Site.

  Evidence of burn ash-containing material (i.e., soil containing fragments of glass, clay, and brick) was observed throughout the majority of the Site. The San Diego County LEA has presented the Site owners with a Notice and Order to remediate the burn ash at the Site. The LEA's directives regarding cover installation have not yet been addressed. However, the City of Chula Vista and the Shinoharas have taken steps to address other LEA directives, including installing a security fence and signs to restrict public access, as well as installing straw wattles for erosion control. In addition, the Client has embarked on the process of properly characterizing the burn ash at the Site, and it is our understanding that the Site will eventually be capped with clean soil in an effort to close the burn ash case.

  SCS is currently in the process of preparing a workplan for waste characterization at the Shinohara II Burn Dump. The workplan includes the following tasks:

  - Exploration for waste limits in which the horizontal extent and thickness of the waste will be assessed by trenching;

  - Topographical survey for the purposes of grading design; and

  - Waste characterization.

Subsequent to the implementation of the workplan, the results of the waste characterization will be used for closure design (including a surface runoff hydrogeology study, river flow analysis, specified soil performance characteristics, preparation of a Construction Quality Control manual, and preparation of engineering drawings and technical specifications for the waste consolidation and capping) and preparation of a Closure/Post-Closure Monitoring and Maintenance Plan.

This Phase I Report, along with the workplan and the scope of work listed above, will be funded with Chula Vista's EPA Brownfields Assessment Grant. SCS understands that Chula Vista will seek additional grant funding from CalRecylce for the eventual remediation and capping of the Shinohara II Burn Dump.

- A review of the aerial photographs revealed that mining activities took place on APN 644-042-10 in 1964 and 1966. Nelson and Sloan owned the parcel until 1991, so it is possible that mining operations continued past 1966. Based on the type of material mined (i.e., gravel), there is a low likelihood that historic mining activities have resulted in a REC at the Site. However, mining reclamation took place on the parcel after the mining operation ceased. The origin or composition of this reclamation material is not known, therefore, SCS is unable to assess the potential that the reclamation material contains CoCs [constituents of concern] and/or has resulted in a REC at the Site. To assess the potential, subsurface assessment activities could be conducted at this portion of the Site.

- A review of aerial photographs revealed that some type of agricultural activity took place at the Site from 1981 to 1989, and in the Site vicinity, possibly prior to 1928 and continuing to circa 1989. The agricultural activity at the Site took place on an area of the parcel that was covered with artificial fill. It is likely that top soil was imported (possibly from Shinohara I, which is interpreted to have had agricultural activity dating back to 1928) and placed over the fill, as artificial fill is typically not suitable for agriculture. The agricultural activity at the Site is not interpreted to have taken place at the time when organochlorine pesticides such as dichlorodiphenyltrichloroethane (DDT), chlordane, and metal-based pesticides, such as lead, copper, and arsenic, were widely used for pest control; however, it is possible that the top soil at the Site, and likely that the soil in the Site vicinity contains such pesticides.

  These classes of pesticides are known to have the potential to remain detectable in the subsurface soil for extended periods of time. Based on the interpreted land use, SCS's experience with agricultural properties, and a review of the available literature, it is our judgment that it is likely that trace concentrations of organochlorine or metal-based pesticides are present in the soil at the Site and Site vicinity as a result of the interpreted agricultural land use. These trace concentrations are likely to be present even after mass grading and earth movement. However, it has generally been our experience that, unless a pesticide mixing, storage, or disposal area was present, concentrations of organochlorine pesticides in the subsurface in general agricultural areas tend to be low. No such areas were reported or are known to have existed at the Site and Site vicinity.

  While there are currently no regulations that stipulate cleanup levels for pesticides in soil, there is a level at which soil could be classified as a hazardous waste based on, for example, a DDT concentration. However, it has been SCS's experience that in order for pesticide-impacted soil to be classified as a hazardous waste, the soil would first need to

be classified as a "waste" (e.g., to be excavated and transported off Site). In addition, it would need to have concentrations of pesticides and/or metals, in excess of regulatory values, such as the total threshold limit concentration (TTLC) or soluble threshold limit concentration (STLC) values.

Based on our experience, there is a moderate likelihood that residual concentrations of organochlorine and metal-based pesticides are present in the shallow surface soil beneath the Site. Assuming the legal and permitted application of these pesticides, and assuming existing Site use remains the same, this common occurrence is, in our experience, unlikely to lead to a health risk or an enforcement action and is, therefore, likely to be considered *de minimis* as defined by ASTM [American Society for Testing and Materials].

For the Shinohara II portion of the Site, soil in this part of the Site will be disturbed during remediation and capping activities conducted to mitigate burn ash; however, because the material to be reconsolidated and capped will require special handling (i.e., dust control during excavation and reconsolidation, use of 40-hour HAZWOPPER-trained contractors, etc.) and precautions due to the presence of CoCs (particularly metals), the presence of pesticides would not require additional efforts and will be assessed during waste characterization.

For the remainder of the Site, because this area is designated to remain undeveloped open space and no excavation or soil export is planned for this area, the potential presence of pesticides at this portion of the Site is considered *de minimis*.

- Portions of the Site vicinity (1870 Main Court and APNs 624-071-01 and 624-060-74) have a history of industrial uses that span approximately 20 years and pre-date the period in which detailed records were maintained and regulations were enforced regarding USTs [underground storage tanks], the use or storage of hazardous materials, and the generation of hazardous waste. Therefore, there is the potential that undocumented features of concern (FOCs) and/or releases exist at three portions of the Site vicinity and may impact the Site.

- Auto dealerships located to the north of the Site (560 and 580 Autopark Drive and 650 Main Street) have been in operation from 1995 to the present. Auto dealerships generally contain auto repair and washing facilities that may include FOCs such as inground hydraulic lifts, solvent parts-cleaning units, and inground wastewater clarifiers. However, 1870 Main Court, APN 624-071-01, and the auto dealerships are located across the Otay River from the Site; therefore, they are unlikely to result in a REC at the Site. The west adjacent parcel (APN 624-060-45) is located adjacent to the burn ash portion of the Site, which, as mentioned above, we understand will be mitigated as part of a Site closure planning process. Therefore, any potentially hazardous materials at the Site associated with industrial land use at the west adjacent parcel (APN 624-060-45) will be managed together with the burn ash material when the Site is remediated.

- SCS recommends continuation of the characterization and closure planning process of the Shinohara II Burn Dump, as outlined above."

2.4 SCOPING MEETING

A meeting between City of Chula Vista personnel and SCS was held on May 2012 at the City offices to discuss the Site, proposed work, and SAP.

2.5 GEOLOGICAL INFORMATION

2.5.1 Geology and Soil Conditions

Based on review of a soil survey[5] for the Site and Site vicinity, soil at the Site was reported to consist of Otay River bed and riverwash deposits. These soils are reported to occur in intermittent stream channels and are typically sandy, gravely, or cobbly. Often, these soils are used as a source of sand and gravel. These soils are excessively drained and rapidly permeable. Many areas are barren. Scattered sycamores and coast live oaks grow along the banks. Sparse shrubs and forbs occur in patches. The designation "gravel pit," which refers to areas where gravel was historically mined from the riverbed, was noted on the map to the east and west of the Site. Based on historical research conducted as part of the Phase I, the Site was also mined for gravel from at least circa 1964 to 1966.

Based on review of a geological map[6] for the Site and Site vicinity, the underlying geology of the Site is reported to consist of alluvium and slopewash (underlies the majority of the Site) and stream terrace deposits (under the northern portion of the Site). The alluvium was reported to consist mostly of poorly consolidated stream deposits of silt, sand, and cobble-sized particles derived from bedrock sources. These deposits were reported to intertongue with Holocene slopewash that commonly mantles the lower valley slopes throughout much of coastal San Diego County. The slopewash was reported to consist of poorly consolidated surficial materials derived chiefly from nearby sources of soil and decomposed bedrock and deposited along the banks of the lower valley slopes by the interaction of gravity and water.

Steam terrace deposits were reported to occur locally as thin veneer along the larger drainage courses. The deposits include unconsolidated sand and gravel derived locally from the sedimentary, igneous, and metamorphic rocks of the area.

A more recent geologic map[7] was also reviewed and included a similar designation of the formation beneath the Site. Young alluvial flood-plain deposits (Holocene and late Pleistocene) were reported to underlie the majority of the Site and were defined as poorly consolidated, poorly sorted, permeable flood-plain deposits of sandy, silty or clay-bearing alluvium. The northern portion of the Site was reported to be underlain by old alluvial flood-plain deposits, undivided (late to middle Pleistocene), described as fluvial sediments deposited on canyon floors

5 *Imperial Beach Quadrangle, Soil Survey, San Diego County, California*, compiled by the United States Department of Agriculture, issued December 1973.

6 *Geology of the National City, Imperial Beach, and Otay Mesa Quadrangles, Southern San Diego Metropolitan Area, California*, by Michael P. Kennedy and Siang S. Tan, 1977, California Division of Mines and Geology.

7 *Geologic Map of the San Diego 30' X 60' Quadrangle, California* by Michael P. Kennedy and Siang S. Tan, 2005, Department of Conservation.

and consisting of moderately well consolidated, poorly sorted, permeable, commonly slightly dissected gravel, sand, silt, and clay-bearing alluvium.

2.5.2 Hydrogeology

Surface water is present in the central portion of the Site within the Otay River floodplain. The elevation of the surface water likely coincides with the approximate elevation of groundwater in the immediate vicinity of the Site. The topographic elevation of the surface water ranges from approximately 88 to 91 feet. Therefore, depth to groundwater would be expected to be relatively shallow along the northern portion of the Site and up to approximately 15 to 18 feet below grade in the higher elevation portions of the Site.

Data regarding groundwater flow direction for the Site were not readily available. However, in SCS's experience, groundwater flow may generally be consistent with stream flow direction. Therefore, the groundwater flow direction at the Site may generally be to the west, consistent with the Otay River flow direction, with a localized component toward the Otay River floodplain (i.e., northwesterly flow along the south side of the river floodplain and southwesterly flow along the north side of the river floodplain).

Please note that many variables influence groundwater depth and flow direction and that the actual depth and flow direction at the Site may be different than presented in this section.

2.5.3 Water Quality Survey

Based on review of the RWQCB's Basin Plan,[8] the Site is located in the Otay Valley Hydrologic Area (910.20) of the Otay Hydrologic Unit (910.00). The groundwater is reported to have existing beneficial uses for industrial purposes and has been exempted for municipal purposes. The surface water is reported to have existing beneficial uses for agriculture, non-contact water recreation, warm freshwater habitat, wildlife habitat, and rare, threatened or endangered species; to have potential beneficial uses for contact water recreation and industrial purposes; and to have been exempted for municipal purposes.

2.5.4 Topography

Based on review of a topographic map[9] for the Site and Site vicinity, the Site was observed to be at an elevation of approximately 100 feet above mean sea level (MSL), and to be mounded in appearance. Based on elevation measurements taken by SCS in August and September 2009, topography at the Site ranges from approximately 92 to 106 feet above MSL.

8 *Comprehensive Water Quality Plan*, RWQCB, originally adopted in 1974, amendments adopted in May 1998.

9 *United States Geological Survey 7.5 Minute Topographic Map, Imperial Beach Quadrangle, California - San Diego County*, 1967, photo-revised 1975.

2.6 IMPACT ON HUMAN HEALTH AND/OR THE ENVIRONMENT

Burn ash and related waste constituents are present in surficial and shallow soil at the Site. There is also the potential that residual concentrations of organochlorine pesticides are present in shallow soil. The vertical extent of burn ash in the soil has not been assessed. No analytical data have been collected from the Site to date. However, data from the adjacent Shinohara I and Dennery Ranch burn sites (interpreted to have the same source of burn ash as the Site, the South Bay Burn Site) have indicated that the primary CoC at the Site is likely to be lead. Other heavy metals are also likely to be present in elevated concentrations.

Based on the available information, there are several potentially complete exposure pathways at the Site in its current condition, and potential receptors include: trespassers on the Site, off-Site residents, Otay Valley Regional Park users, and various ecological receptors associated with the Otay River. The only potential exposure pathway that has been evaluated is lead-bearing dust impacting residents of the adjacent Dennery Ranch. Environ's previously discussed risk assessment indicated there was no significant risk to future Dennery Ranch residents from lead-bearing dust from the Site.

In order to obtain closure for the Site, the City plans to proceed with cover design and installation. Based on our evaluation, there will be only two potentially complete exposure pathways at the Site post-closure: the potential for impacted groundwater migrating to the Otay River and potentially impacting various ecological receptors, and human trespassers (e.g., digging for bottles). Please note these may not be complete exposure pathways, as the depth of burn ash-containing soils is not known and may not be in contact with the water table, and human trespassers can be deterred through security measures.

During the construction of the engineered cap, there will be several potentially complete exposure pathways; therefore, measures, such as various best management practices (BMPs), will be employed to minimize the likelihood of exposing construction workers, nearby off-Site residents, Otay Valley Regional Park users, and ecological receptors to CoCs.

Burn dump sites in California that have minimal environmental and health risks are typically closed by means of a constructed soil cap with a minimum thickness of 2 feet. The proposed cover for a burn dump site is typically selected following a waste characterization study in which constituents of the dump are sampled and analyzed to evaluate levels of health and environmental risk associated with the site. Burn dumps with low levels of environmental risk are usually closed under the authority of the LEA, in conjunction with the local RWQCB.

The work proposed in this SAP typically conforms to the requirements of the DTSC's *Protocol for Burn Dump Site Investigation and Characterization*[10] and is intended to collect sufficient samples to characterize the waste at the Site so that cap design and Site closure can proceed. The results of the proposed investigation will be compared to applicable or relevant and appropriate requirements (ARARs) for the Site, which include waste criteria, such as the TTLCs, STLCs, and Maximum Concentrations of the Contaminant for the Toxicity Characteristic (MCCTC), for metals and pesticides in soil.

10 *Protocol for Burn Dump Site Investigation and Characterization*, by DTSC, dated June 30, 2003.

3.0 PROJECT AND DATA QUALITY OBJECTIVES

3.1 PROJECT TASK AND PROBLEM DEFINITION

There are known deposits of burn ash and metals-bearing fill material at the Site from historical filling operations, and there is also the potential that residual concentrations of pesticides are present in shallow soil from agricultural land uses. Because no analytical data have been collected from the Site to date, SCS has proposed soil sampling to assess the potential CoCs at the Site from agricultural land uses and the stockpiled soil of unknown origins. The purpose of the proposed soil sampling is to:

- Assess the extent and thickness of the waste.
- Characterize the waste so that the waste characterization information can be incorporated into closure design and planning.
- Assess whether residual pesticides are present in the shallow surface soil at concentrations that might pose risks to construction workers and others.

3.2 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) have been established to increase the likelihood that the data collected are analyzed with the appropriate analytical methods and detection limits. Table 3-1 includes the lists of target soil CoCs to be analyzed and their respective laboratory analytical methods, laboratory reporting limits, and action levels. TTLC, STLCs, and MCCTC values were used as screening criteria for metals and suspected pesticides in soil. If one or more of the CoCs are reported to be present at concentrations above their action levels, then recommendations for further action, including possible additional assessment or remediation, will be made.

Table 3-1: Soil Matrix
Total Constituents of Concern, Laboratory Reporting Limits and Action Levels

Total CoC	Analytical Methods	Laboratory Reporting Limits (in mg/kg)	TTLC (in mg/kg)
Antimony	EPA Method 6010B	0.75000	500
Arsenic		0.75000	500
Barium		0.50000	10,000
Beryllium		0.25000	75
Cadmium		0.50000	100
Chromium		0.25000	2,500
Chromium VI	EPA Method 7196A	0.8000	500
Cobalt	EPA Method 6010B	0.25000	8,000
Copper		0.50000	2,500
Lead			1,000
Mercury	EPA Method 7471A	0.08350	20
Molybdenum	EPA Method 6010B	0.25000	3,500

Total CoC	Analytical Methods	Laboratory Reporting Limits (in mg/kg)	TTLC (in mg/kg)
Nickel	EPA Method 6010B	0.25000	2,000
Selenium		0.75000	100
Silver		0.25000	500
Thallium		0.75000	700
Vanadium		0.25000	2,400
Zinc		1.00000	5,000
DDD	EPA Method 8081A	0.005000	1.0
DDE			1.0
DDT			1.0
Aldrin			1.4
Chlordane		0.050000	2.5
Dieldrin		0.005000	8.0
Endrin			0.2
Gamma-BHC (Lindane)			4.0
Heptachlor			4.7
Heptachlor epoxide			NA
Methoxychlor			100
Toxaphene		0.100000	5

Notes:
CoC = constituent of concern
TTLC = Total Threshold Limit Concentration
mg/kg = milligram per kilogram
BHC = benzene hexachloride
NA = not applicable

Table 3-2: Soil Matrix
Soluble Constituents of Concern, Laboratory Reporting Limits and Action Levels

Soluble Metals	Analytical Methods	Laboratory Reporting Limits (in mg/L)	STLC (in mg/L)	MCCTC (in mg/L)
Antimony	WET or TCLP/ EPA Method 6010B	0.15000	15	NA
Arsenic		0.15000	5	5
Barium		0.10000	100	100
Beryllium		0.10000	0.75	NA
Cadmium		0.10000	1	1
Chromium		0.10000	5	5
Chromium VI	WET or TCLP/EPA Method 7196A	0.0200	5	NA
Cobalt	WET or TCLP/ EPA Method 6010B	0.10000	80	NA
Copper		0.10000	25	NA
Lead		0.10000	5	5
Mercury	WET or TCLP/EPA Method 7470A	0.00500	0.2	0.2
Molybdenum	WET or TCLP/ EPA Method 6010B	0.10000	350	NA
Nickel		0.10000	20	NA
Selenium		0.15000	1	1

Soluble Metals	Analytical Methods	Laboratory Reporting Limits (in mg/L)	STLC (in mg/L)	MCCTC (in mg/L)
Silver	WET or TCLP/ EPA Method 6010B	0.05000	5	5
Thallium		0.15000	7	NA
Vanadium		0.10000	24	NA
Zinc		0.10000	250	NA
DDD	WET or TCLP/ EPA Method 8081A	0.001/NA	0.1	NA
DDE		0.001/NA	0.1	NA
DDT		0.001/NA	0.1	NA
Aldrin		0.001/NA	0.14	NA
Chlordane		0.010/0.010	0.25	0.03
Dieldrin		0.001/0.001	0.8	NA
Endrin		0.001/0.001	0.02	0.02
Gamma-BHC (Lindane)		0.001/0.001	0.4	0.4
Heptachlor		0.001/0.001	0.47	0.008
Heptachlor epoxide		0.001/0.001	NA	0.008
Methoxychlor		0.001/0.001	10.0	10
Toxaphene		0.010/0.010	0.5	0.5

Notes:
CoC = constituent of concern
STLC = Soluble Threshold Limit Concentration
MCCTC = Maximum Concentration of Contaminant for Toxicity Characteristic
TCLP = Toxicity Characteristic Leaching Procedure
mg/L = milligram per liter
BHC = benzene hexachloride
NA = not applicable
0.001/NA indicates that first value is reporting limit for Waste Extraction Test (WET) which is compared to STLC, and second value is for Toxicity Characteristic Leaching Procedure (TCLP) which is compared to MCCTC. NA indicates respective analyte not included in WET or TCLP analysis.

3.3 MEASUREMENT QUALITY OBJECTIVES

MQOs are criteria established to assess the viability and usability of data. These are based on both field and laboratory protocols that examine whether the data quality indicators (DQIs) (i.e., precision, accuracy, representativeness, completeness, comparability, and sensitivity [PARCCS]), meet criteria established for various aspects of data gathering, sampling, or analysis activity. In defining the MQOs, the level of uncertainty associated with each measurement is defined.

The values assigned to the quantitative data quality indicators (precision, accuracy, completeness and sensitivity) and statements concerning the qualitative indicators (representativeness and comparability) are based upon the DQOs established in Section 3.2. Project-specific requirements for PARCCS are discussed below. Laboratory standard operating procedures (SOPs) are also included in the Appendix and are referenced below.

3.3.1 Precision

Precision is a quantitative measure of how reproducible a specific set of data may be. Precision is addressed in Section 10.5 and Appendix D of the Calscience Quality Systems Manual that is included in the SAP Appendix. Also, refer to the Calscience SOPs in the SAP Appendix for each

analytical procedure proposed for the specific Matrix Spike and Blank Spike/Laboratory Control Sample percent recovery acceptance limits and acceptable relative percent differences (RPD).

The following laboratory quality control (QC) procedures are to be utilized:

- A method blank will be performed with each analytical batch of samples.
- At least one Matrix Spike (MS) and one Matrix Spike Duplicate (MSD) will be analyzed per analytical batch (if not enough sample is available for a matrix spike, then a Laboratory Control Sample and a Laboratory Control Sample Duplicate will be used as QC samples).
- Surrogates will be run on all organic analysis including spikes and blanks. If surrogate recoveries are outside their specified control limits, corrective action will be implemented as specified in the individual method SOPs.

The following field QC procedures are to be utilized:

- A blind duplicate sample will be collected at a frequency of one blind duplicate for every 10 samples collected for soil analyses. Blind duplicates will be labeled with a unique sample number.
- Field blanks will be collected each day of sampling.

3.3.2 Accuracy

Accuracy is a quantitative measure of how well measurements reflect what is actually in the sample. Accuracy is addressed in Section 9.0 of the Calscience Quality Systems Manual. Also, refer to the Calscience SOPs in the Appendix for each analytical procedure to be performed, including the specific Matrix Spike and Blank Spike/Laboratory Control Sample percent recovery acceptance limits. The laboratory QC procedures to be utilized are discussed in the previous section.

QC samples to assess potential sample contamination from the field activities or laboratory contaminants will be addressed by the use of field blanks to be collected at the Site and a method blank will be analyzed with each batch of samples as discussed in Appendix D of the Calscience Quality Systems Manual. The acceptance of the field blank and the method blank will be set at equal or less than the method reporting limit.

3.3.3 Representativeness

Representativeness is the measure that can be both qualitative and quantitative of how well the sample data reflect the environmental conditions.

Due to the interpreted nature of the waste deposition activities at the Site (waste was reported to have been excavated from the nearby South Bay Burn Site, transported to the Site, placed, and capped), it is anticipated that the waste would have been mixed during excavation and filling activities. In order to collect samples representative of waste, 38 trenches will be advanced at the interpreted edges of the waste, and soil samples will be collected from the exposed waste in 23 of the trench locations. Samples will be collected at alternating depths, at the surface of the waste,

at the approximate center of the waste, and at the deepest extent of the waste, in order to characterize the entire waste horizon.

The trench locations were selected in order to assess the extent of waste and to be approximately evenly distributed around the Site. This lateral and vertical sample distribution should provide sufficient data to be representative of Site conditions in relation to the former burn site and for waste characterization.

The Site was reported to have been used for agriculture from between 1981 and 1989 by the Shinohara family. Details regarding the agricultural activities are not available, but it was reported that the agricultural activities took place on the Shinohara II Burn Site portion of the Site. It is interpreted that top soil must have been placed over this area so that it could be farmed. There are no known or suspected pesticide storage or mixing areas. It is anticipated that pesticides would have been applied in a homogenous manner throughout the areas used for agriculture although mixing (by plowing) would likely have not been conducted (to not mix burn ash and waste with clean top soil).

In order to collect samples representative of Site conditions in shallow soil, surface soil samples will be collected from five locations in the reported location of the former agricultural activities within the waste area. This sample distribution should provide sufficient data to be representative of Site conditions in relation to potential pesticide impacts at the Site.

In order to collect samples representative of the waste for metals, samples will be collected from the teeth of the backhoe and placed directly in laboratory-supplied glass jars and placed in an ice-filled cooler at approximately 4 degrees Centigrade pending delivery to a state-accredited laboratory (Calscience) for analysis. In order to collect soil samples representative of the subsurface for pesticide sampling, shallow soil samples will be collected in stainless steel sleeves with a hand auger and a manual drive-sampler. Upon collection, sample sleeves will be capped with Teflon sheets and end caps, labeled, and placed in an ice-filled cooler. Chain-of-custody procedures will be followed for sample tracking.

3.3.4 Completeness

Completeness is a quantitative measure of the amount of data to be collected that is essential to meet the project objectives. Since there are no available data for the Site, the proposed sampling will need to yield sufficient, useable data to adequately assess the presence, or possible presence, and concentration of CoCs associated with the known Shinohara II Burn Site and potential releases from historical agricultural land uses.

Given the lack of existing or useable data to date for the Site, a completeness of 90 percent will be acceptable. No specific sample locations are considered more critical than any of the others; therefore, if for some reason a proposed location cannot be sampled and/or relocated to an area near the originally proposed locations, remobilization to the Site for additional sampling will not be necessary or required so long as 90 percent of the proposed locations can be sampled.

3.3.5 Comparability

Comparability is a qualitative measurement of how similar project data need to be to those collected from other studies, similar locations within the same study, same sampling locations at

different times of the year, etc. The purpose of the proposed soil sampling is to provide a baseline and to assess the presence, or possible presence, and concentration of CoCs associated with known waste deposits and potential releases from historical agricultural land uses. Since there are no existing, available, and useable data for the Site, the proposed sampling will need to yield sufficient, useable data necessary to meet the sampling objectives. Since all the samples will be collected over the course of a few days by SCS and analyses will be conducted by the same laboratories, via the same analytical procedures, the data will have a very high level of comparability.

3.3.6 Sensitivity

Sensitivity is a quantitative measure of field and/or laboratory methods used to assess if the resulting data are of a quality (low enough quantitation limits) that will be sensitive enough to quantify CoCs at or below regulatory standards or action levels. Sensitivity is addressed in Section 9.4 of the Calscience Quality Systems Manual. Also, refer to the Calscience method sheets for each analytical procedure proposed for the method detection limits and reporting limits.

Comparisons of the laboratory reporting limits and action levels are presented in Tables 3-1 and 3-2. QC samples to be collected and acceptance limits are discussed in the Accuracy and Precision sections.

3.4 DATA REVIEW AND VALIDATION

Pursuant to Appendix D of the Calscience Quality Systems Manual, data processing and validation within the analytical laboratory ensure that the reported results will correctly represent the analyses performed. This function has two primary activities:

- The processing of quality control sample results to demonstrate that analyses are within laboratory prescribed bounds for accuracy, precision, and completeness.
- Sample reduction and validation to demonstrate that numerical computation of data is correct and that it is correctly reported.

Calscience will review sample management, analysis, and reporting pursuant Appendix D of the Calscience Quality Systems Manual in order to meet the DQIs as presented in Section 3.3. Results and variances from the DQIs will be reported with the laboratory reports.

The SCS Project Manager and QA Officer, or their designee, will review staff field notes of field sampling methodologies and laboratory reports for compliance with SCS standard operating procedures. Data validation will be conducted by the QA Officer and will follow EPA Region 9 Evaluation Tier 1B criteria. The data validation will include review of sample holding times, QC results, and duplicate sample and equipment blank results; and cross-checking analytical results against raw data and calculations used in the data set. Reviews will be documented in the project file.

Data that do not meet data quality objectives will be flagged and discussed in the Phase II Site Assessment report. The actions taken to address the data that do not meet DQOs will be presented in the Phase II report and may include the rejection of the data and exclusion from

consideration during the preparation of the assessment report; a description of reasoning why the data may still be used even though it does not meet data quality objectives; or a description of the steps taken to reanalyze samples or return to the Site to resample.

3.5 DATA MANAGEMENT

Calscience will follow the procedures in Sections 10.0 and 11.0 of the Calscience Quality Systems Manual for proper data management from receipt of samples with a properly completed chain of custody to reporting of final laboratory data and quality assurance/quality control data in its final report.

The SCS Project Manager and QA Officer will review staff field notes of field sampling methodologies, chain-of-custody documentation, and laboratory reports for compliance with SCS standard operating procedures. Reviews will be documented in the project file. Transfer of data from laboratory reports to tables, figures, and report text will be reviewed by the SCS technical editor for accuracy and documented on a standard SCS review form. A final review of the Phase II Site Assessment report, including data, will be performed by the Project Manager, QA Officer, and a Professional Geologist or Engineer and documented on a standard SCS review form.

3.6 ASSESSMENT OVERSIGHT

A premobilization meeting will be held prior to fieldwork. During this meeting, the Project Manager and field team will review the SAP, Site Health and Safety Plan, and project objectives. The SCS Project Manager and QA Officer will both supervise field activities and review staff field notes of field sampling methodologies, chain of custody, and laboratory reports for compliance with SCS standard operating procedures. Reviews will be documented in the project file. Transfer of data from laboratory reports to tables, figures, and report text will be reviewed by the SCS technical editor for accuracy and documented on a standard SCS review form. A final review of the assessment report, including data, will be performed by the Project Manager and documented on a standard SCS review form. At any time during the project, the Project Manager or QA Officer has the authority to direct that corrective actions be taken if it is deemed necessary. The QA Officer has and maintains independent authority from the Project Manager to take corrective action, as necessary.

SCS has implemented a Quality Management System (QMS). As part of the QMS, selected completed projects are audited for compliance with SCS procedures, including health and safety procedures and quality control protocols. During the auditing process, the QA Officer or designee randomly selects completed projects for a detailed review for adherence to the QMS. The following is a short description of the QMS.

3.6.1 SCS Quality Management System

SCS has implemented a QMS that conforms to EPA Requirements for Quality Management Plans, EPA QA/R-2, EPA/240/B-01/002, reissued May 2006. The purpose of the QMS is to provide the framework to plan, implement, and assess the effectiveness of quality assurance (QA) and QC operations within SCS. The QMS addresses all aspects of work performed by SCS.

Our overall objective is to provide quality solutions to environmental problems in an ethical manner. Briefly summarized, the QMS requires that SCS staff and subcontractors have the necessary training and skills, use the proper tools, and employ sound procedures to conduct work safely and appropriately. SCS continuously seeks to improve the quality of the services we deliver by checking our work, considering new technologies and work processes, and documenting compliance with the QMS.

In accordance with EPA guidance, the QMS specifies standards and procedures regarding the following quality management plan elements:

1. Quality Policy
2. Organization and Authority
3. Quality System Components
4. Personnel Qualifications and Training
5. Procurement of Items and Services
6. Documents and Records
7. Computer Hardware and Software
8. Planning
9. Implementation
10. Assessment and Response
11. Quality Improvement

4.0 SAMPLING DESIGN AND RATIONALE

The following sections describe the specifics of and rationales for the proposed soil sampling.

4.1 SOIL SAMPLING

The purpose of the proposed soil sampling is to:

- Assess the extent and thickness of the waste.
- Characterize the waste so that the waste characterization information can be incorporated into closure design and planning.
- Assess whether residual pesticides are present in the shallow surface soil at concentrations that might pose risks to construction workers and others.

To accomplish this, SCS will excavate 38 trenches at the edges and interior of the Site, collect soil samples from the waste horizon in 23 trench locations, and collect surface soil samples from the topsoil/cap soil at the surface of the burn site. Trench locations and surface soil samples locations are depicted on Figure 3.

Samples will be sent to Calscience, a state-certified, fixed-based laboratory, and analyzed as follows:

- Title 22 metals with EPA Method 6010B/7471A.
- Total chromium VI with EPA Method 7196.
- For the samples with the highest total lead concentrations, soluble metals with the Waste Extraction Test (WET) and EPA Methods 6010B/7196/7470A.
- For the samples with the highest total lead concentrations, soluble metals with the Toxicity Characteristic Leaching Procedure (TCLP) and EPA Methods 6010B/7196/7470A.
- For the samples with the highest total lead concentrations, soluble lead with the deionized Waste Extraction Test (DI-WET) and EPA Methods 6010B.
- pH with EPA Method 9045.
- Organochlorine pesticides with EPA Method 8081A.
- Arsenic, copper, and lead with EPA Method 6010B.

Table 4-1: Soil Matrix Sampling Design and Rationale

Sampling Location/ID Number	Depth (ft)	CoCs and Analytical Methods	Rationale
T1 through T23	Unknown, dependent on waste thickness in each location	Title 22 Metals (6010B/7471A), chromium VI (7196), pH (9045), soluble lead (WET/TCLP/DI-WET and 6010B)	To characterize the waste
SS1-SS8	0.5	Organochlorine pesticides (8081A), arsenic/copper/lead (6010B)	To assess potential impacts from historical agricultural operations and potential impacts to construction workers

Notes:
ft = feet
CoCs = constituents of concern
WET = Waste Extraction Test
TCLP = Toxicity Characteristic Leaching Procedure
DI-WET = De-ionized WET

5.0 REQUEST FOR ANALYSES

5.1 ANALYSES NARRATIVE

Samples will be sent to Calscience, a state-certified, fixed-based laboratory, for analysis.

5.1.1 Waste Samples

The waste samples (from sample locations T1 through T23) will be analyzed as follows:

- Twenty-three samples for Title 22 metals with EPA Method 6010B/7471A.
- Twenty-three samples for Total chromium VI with EPA Method 7196.
- Three samples (the samples with the highest total lead concentrations) for soluble metals with the WET and EPA Methods 6010B/7196/7470A.
- Three samples (the samples with the highest total lead concentrations) for soluble metals with the TCLP and EPA Methods 6010B/7196/7470A.
- Three samples (the samples with the highest total lead concentrations) for soluble lead with the DI-WET and EPA Methods 6010B.
- Twenty-three samples for pH with EPA Method 9045.

Trenches will be advanced at the interpreted edges of the waste using a backhoe. Trench locations will be recorded by a licensed land surveyor subsequent to the trenching activities by physically staking locations and conducting a field topographic survey.

In all, 38 trenches will be excavated at the interpreted edges and interior of the waste (see Figure 3 for proposed trench and sample locations). Samples will be collected from interpreted burn ash from each trench. Sample depths will alternate between trenches so that samples of burn ash are collected at the surface, the approximate center of the waste horizon, and the deepest extent of waste based on visual observations. Samples will be collected by using the excavator bucket to collect a sample at the desired depth and scooping the sample from the teeth of the bucket directly into laboratory-supplied glass jars.

5.1.2 Surface Samples

The surface soil samples (from sample locations SS1 through SS8) will be analyzed as follows:

- Eight surface samples for organochlorine pesticides with EPA Method 8081A.
- Eight surface soil samples for arsenic, copper, and lead with EPA Method 6010B.

Surface soil sampling will be conducted to assess whether historical agricultural operations resulted in pesticide impacts to the soil overlying the waste. Eight shallow soil samples will be collected from eight locations, evenly distributed across the Site. The samples will be collected with disposable trowels and placed into laboratory-supplied glass jars.

Table 5-1: Soil Matrix Analytical Services

Sample Number	Sample Location	Depth (ft)*	Special Designation	Analytical Methods						
				Title 22 Metals 6010B/ 7471A	Chromium VI 7196	pH 9045	Soluble Metals WET/TCLP/ 6010B/ 7471A/ 7196**	Soluble lead DI-WET/ 6010B**	Pesticides 8081A	Arsenic/ Copper/ Lead 6010B
T1	T1	Shallow	MS/MSD	✓	✓	✓				
T2	T2	Center	***	✓	✓	✓				
T3	T3	Deep		✓	✓	✓				
T4	T4	Shallow		✓	✓	✓				
T5	T5	Center		✓	✓	✓				
T6	T6	Deep		✓	✓	✓				
T7	T7	Shallow		✓	✓	✓				
T8	T8	Center		✓	✓	✓				
T9	T9	Deep		✓	✓	✓				
T10	T10	Shallow		✓	✓	✓				
T11	T11	Center		✓	✓	✓				
T12	T12	Deep	***	✓	✓	✓				
T13	T13	Shallow		✓	✓	✓				
T14	T14	Center		✓	✓	✓				
T15	T15	Deep		✓	✓	✓				
T16	T16	Shallow		✓	✓	✓				
T17	T17	Center		✓	✓	✓				
T18	T18	Deep		✓	✓	✓				
T19	T19	Shallow		✓	✓	✓				
T20	T20	Center		✓	✓	✓				
T21	T21	Deep	MS/MSD	✓	✓	✓				
T22	T22	Shallow		✓	✓	✓				
T23	T23	Center		✓	✓	✓				
SS1	SS1	0.5	***						✓	✓
SS2	SS2	0.5							✓	✓
SS3	SS3	0.5							✓	✓
SS4	SS4	0.5							✓	✓
SS5	SS5	0.5							✓	✓
SS6	SS6	0.5							✓	✓
SS7	SS7	0.5							✓	✓
SS8	SS8	0.5							✓	✓
T24	T24	Center	BD of T2	✓	✓	✓				
T25	T25	Deep	BD of T12	✓	✓	✓				
SS9	SS9	05	BD of SS1						✓	✓
Total Number of Soil Samples, Excluding QC: 31				23	23	23	3	3	8	8
Total Number of Soil Samples, Including QC: 33				28	28	28	3	3	9	9

Notes:
ft = feet
WET = Waste Extraction Test
TCLP = Toxicity Characteristic Leaching Procedure
DI-WET = De-ionized WET
BD = blind duplicate
QC = quality control
* For samples T1 through T23, the depth of sample will be determined by the thickness of the waste. Samples will be collected from the upper third, center third, and lower third of the waste horizon, alternately.

**The three samples with the highest total lead concentrations will be selected for soluble metals analysis with the WET, TCLP, and EPA Methods 6010B/7471A/7196A, and for soluble lead with the DI-WET and EPA 6010B.
*** indicates that a blind duplicate will be collected and analyzed for the respective sample.

In addition, the laboratory will perform the following analyses:

- A method blank will be performed with each analytical batch of samples.
- Surrogates will be run on all organic analysis including spikes and blanks.

A temperature blank will be included with the sample shipment to Calscience to verify temperature upon acceptance of the samples.

Table 5-2: Soil Matrix
Analytical Method, Containers, Preservation, and Holding Times Requirements

Analytical Parameter and/or Field Measurements	Analytical Method Number	Containers (number, type, size/volume)	Preservation Requirements (chemical, temperature, light protection)	Maximum Holding Times
Title 22 Metals (not mercury)	6010B	One 8-ounce glass jar with Teflon lid	None	180 days
Mercury	7471A	One 8-ounce glass jar with Teflon lid	None	28 days
Chromium VI	7196A	One 8-ounce glass jar with Teflon lid	Chill to 4° Celsius	30 days
Soluble Metals	WET/TCLP/6010B	One 8-ounce glass jar with Teflon lid	None	No holding time for extraction, 180 days for analysis
Soluble Mercury	WET/TCLP 7471A	One 8-ounce glass jar with Teflon lid	None	No holding time for extraction, 28 days for analysis
Soluble Chromium VI	WET/TCLP/7196A	One 8-ounce glass jar with Teflon lid	Chill to 4° Celsius	24 hours for extraction, 180 days for analysis
Soluble Lead	DI-WET/6010B	One 8-ounce glass jar with Teflon lid	None	No holding time for extraction, 180 days for analysis
pH	9045	One 8-ounce glass jar with Teflon lid	Chill to 4° Celsius	ASAP, within 24 hours
Pesticides	8081A	One 8-ounce glass jar with Teflon lid	Chill to 4° Celsius	14 days for extraction, 40 days for analysis

Notes:
WET = Waste Extraction Test
TCLP = Toxicity Characteristic Leaching Procedure
DI-WET = De-ionized WET
ASAP = as soon as possible

5.2 ANALYTICAL LABORATORY

Samples will be sent to Calscience, a state-certified, fixed-based laboratory, for analysis.

Copies of the Calscience Quality Systems Manual are included in the Appendix along with copies of the Calscience SOPs for each proposed analytical method. MQOs for work performed by the laboratory can be found in the H&P Quality Systems Manual and the Calscience Quality Systems Manual.

6.0 FIELD METHODS AND PROCEDURES

Additional Site investigation is necessary to confirm the limits and transition of the waste. In addition, the thickness of the waste deposits and the approximate contact with underlying native soils will be assessed in the field by qualified personnel. This will be accomplished by using a backhoe to excavate 38 trenches at the interpreted edges and interior of waste on the Site, and collecting soil samples from 23 of the trenches.

Surface soil sampling will be conducted to assess whether historical agricultural operations resulted in pesticide impacts to the soil overlying the waste. Eight shallow soil samples will be collected from eight locations, evenly distributed across the Site.

Personnel will wear disposable nitrile gloves while collecting samples and handling sample containers. Detailed information about sample containers, preservation, packaging, and shipping are provided in Section 7.0.

6.1 FIELD EQUIPMENT

6.1.1 List of Equipment Needed

The following equipment and supplies will be used at the Site for sample collection:

- Laboratory-supplied glass jars with Teflon-lined lids
- Disposable trowels
- Mini-RAE photo-ionization detector (PID)
- Walking wheel and measuring tapes
- Scale
- Sharpies/pens
- Site plan
- Daily field log forms
- Labels
- Chain-of-Custody forms
- Ice and Cooler
- Paper towels
- Personal protective equipment (PPE), including hard hat, boots, safety glasses, nitrile gloves, and ear plugs
- Sunscreen, sun hat, and water

In case of equipment failure, some supplies (e.g., spare batteries, extra lamps and filters for the PID, etc.) will be kept on hand to attempt fixes in the field.

6.1.2 Calibration of Field Equipment

Initial calibration and maintenance of the PID are performed per the manufacturer's recommendations. SCS will include copies of the calibration and maintenance records in the project field notes in the project file.

The PID will be zeroed to ambient air and recalibrated with isobutylene standard gas at the beginning of each day. Documentation of calibration completed in the field will be included in the field notes for the project in the project file.

6.2 FIELD SCREENING

Given that volatile organic compounds (VOCs) are not usually associated with burn ash, it is not anticipated that VOC-containing vapors will be encountered at the Site; however, as a precautionary measure, a PID will be used to monitor for vapors during excavation activities as part of standard SCS operating procedures. Readings will be taken periodically during excavation activities in the breathing zone of workers.

6.3 SOIL SAMPLING

6.3.1 Waste Sampling

Additional Site investigation is necessary to confirm the limits and transition of the waste, and assess the thickness of the waste deposits and the approximate contact with underlying native soils in the field by qualified personnel. This will be accomplished by using a backhoe to excavate trenches at the interpreted edges of waste on the Site. Trench locations will be recorded by physically staking locations and conducting a field topographic survey by a licensed land surveyor subsequent to the trenching activities.

In all, 38 trenches will be excavated at the interpreted edges and interior of the waste (Figure 3 depicts the proposed trench locations). Waste samples (T1 through T23) will be collected from interpreted burn ash from 23 of the trenches. Sample depths will alternate between trenches so that samples of burn ash are collected at the surface, at the approximate center of the waste horizon, and at the deepest extent of waste based on visual observations. Samples will be collected by using the excavator bucket to collect a sample at the desired depth and scooping the sample from the teeth of the bucket (only soil from the top of the teeth, not in contact with the teeth or bucket will be collected for the sample) directly into laboratory-supplied glass jars with Teflon-lined lids.

Upon sample collection, the glass jars will be tightly shut, and the samples will be labeled. The samples will be placed in sealable plastic bags and placed in an ice-filled cooler pending pick-up by the Calscience courier under chain-of-custody.

Trench and sample locations, shown on Figure 3, were selected based on the interpreted and reported limits of waste.

6.3.2 Surface Sampling

Surface soil sampling will be conducted to assess whether historical agricultural operations resulted in pesticide impacts to the soil overlying the waste. Five shallow soil samples (SS1 through SS5) will be collected from five locations, evenly distributed across the Site. The samples will be collected with disposable trowels and placed into laboratory-supplied glass jars.

Upon sample collection, the glass jars will be tightly shut, and the samples will be labeled. The samples will be placed in sealable plastic bags and placed in an ice-filled cooler pending pick-up by the Calscience courier under chain-of-custody.

Sample locations are shown on Figure 3 and were selected based on DTSC guidelines for pesticide sampling for school sites,[11] which states that samples should be placed at a frequency of approximately 8 samples for sites between 4 and 20 acres in size.

See Section 7.1 for preservation and shipping procedures.

6.4 SEDIMENT SAMPLING

Sediment sampling is not proposed for the Site.

6.5 WATER SAMPLING

6.5.1 Surface Water Sampling

Surface water sampling is not proposed for the Site.

6.5.2 Groundwater Sampling

Groundwater sampling is not proposed for the Site.

6.6 DECONTAMINATION PROCEDURES

Decontamination of sampling equipment must be conducted consistently to assure the quality of samples collected. All equipment that comes into contact with potentially contaminated soil and water will be decontaminated. Disposable equipment intended for one-time use will not be decontaminated but will be packaged for appropriate disposal.

SCS intends to use one-time disposable equipment (plastic trowels) and place samples directly into sample containers. If any equipment, such as a hand auger, is used, decontamination will occur prior to and after each use of a piece of equipment. Any equipment used will be decontaminated according to EPA Region 9 recommended procedures. The following, to be carried out in sequence, is an EPA Region 9 recommended procedure for the decontamination of sampling equipment.

- Non phosphate detergent and tap water wash, using a brush if necessary

11 *Interim Guidance for Sampling Agricultural Fields for School Sites*, DTSC, August 26, 2002.

- Tap water rinse
- Deionized/distilled water rinse (first rinse)
- Deionized/distilled water rinse (second rinse)

Table 6-1: Field and Sampling Equipment

Description of Equipment	Material (if applicable)	Dedicated (Yes/No)
Disposable trowels	NA	Yes
Glass jars	NA	Yes
PID	NA	No

Notes:
PID = photoionization detector
NA = not applicable

7.0 SAMPLE CONTAINERS, PRESERVATION, PACKAGING AND SHIPPING

The number and type of sample containers, volumes, and preservatives are listed in Table 5-2. The containers will be pre-cleaned and will not be rinsed prior to sample collection.

7.1 SOIL SAMPLES

Soil samples will be collected in laboratory-supplied glass jars with Teflon-lined lids. Immediately upon sample collection, the jars will be tightly closed and the samples will be placed in an ice-filled cooler and chilled to approximately 4 degrees Celsius immediately upon collection pending pickup by the Calscience courier.

7.2 PACKAGING AND SHIPPING

Soil samples for pesticide and metals analysis will be placed in an ice-filled, strong-outside shipping container (a plastic cooler) pending pickup by the Calscience courier. The following outlines the packaging procedures that will be followed for low concentration samples.

1. Seal the drain plug of the cooler with fiberglass tape to prevent melting ice from leaking out of the cooler.

2. Line the bottom of the cooler with bubble wrap to prevent breakage during shipment.

3. Check screw caps for tightness and, if not full, mark the sample volume level of liquid samples on the outside of the sample bottles with indelible ink.

4. Secure bottle/container tops with clear tape and custody seal all container tops.

5. Affix sample labels onto the containers.

6. Wrap all glass sample containers in bubble wrap to prevent breakage.

7. Seal all sample containers in heavy duty plastic zip-lock bags. Write the sample numbers on the outside of the plastic bags with indelible ink.

8. Place samples in a sturdy cooler(s) lined with a large plastic trash bag. Enclose the appropriate chain-of-custody forms in a zip-lock plastic bag affixed to the underside of the cooler lid.

9. Fill empty space in the cooler with bubble wrap to prevent movement and breakage during shipment.

10. Double seal ice used to cool samples in two zip-locked plastic bags and place on top and around the samples to chill them to the correct temperature.

8.0 DISPOSAL OF RESIDUAL MATERIALS

In the process of collecting environmental samples, the sampling team will generate different types of potentially contaminated investigation-derived waste (IDW) that may include the following:

- Used PPE
- Incidentals (paper towels, plastic bags)
- Soil from trenches

The EPA's National Contingency Plan (NCP) requires that management of IDW generated during sampling comply with ARARs to the extent practicable. The sampling plan will follow the Office of Emergency and Remedial Response (OERR) Directive 9345.3-02 (May 1991), which provides the guidance for the management of IDW. In addition, other legal and practical considerations that may affect the handling of IDW will be considered.

Used PPE and incidentals will be double bagged and placed in a municipal refuse dumpster. These wastes are not considered hazardous and can be sent to a municipal landfill. Any PPE that is to be disposed of and can still be reused will be rendered inoperable before disposal in the refuse dumpster.

Since the samples will be collected directly from the teeth of the backhoe into sample jars, it is not anticipated that any equipment will be used that will required decontamination. Therefore, no decontamination water will be generated.

Soil excavated from the trenches will be used to backfill the trenches upon completion of sampling and trench logging. Trenches will be backfilled at the end of each day’s activities.

9.0 SAMPLE DOCUMENTATION

9.1 FIELD NOTES

9.1.1 Field Log Forms

Daily field log forms will be used to document field activities. Each day's field log forms will be dated, and the pages will be consecutively numbered. The log forms will be completed in ink and signed by the individual recording the information. Field log forms will be supplemented with detailed trench logs and field-edited site plans.

At a minimum, the following information will be recorded during the collection of each sample:

- Sample location and description
- Site plan showing sample location and measured distances
- Sampler's name(s)
- Date and time of sample collection
- Designation of sample as composite or grab
- Type of sample (waste or surface)
- Type of sampling equipment used
- Field instrument readings and calibration
- Field observations and details related to analysis or integrity of samples (e.g., weather conditions, noticeable odors, colors, etc.)
- Preliminary sample descriptions (e.g., for soils: clay loam, very wet)
- Sample preservation
- Lot numbers of the sample containers, sample identification numbers and any explanatory codes, and chain-of-custody form numbers
- Shipping arrangements (overnight air bill number)
- Name(s) of recipient laboratory(ies)

In addition to the sampling information, the following specific information will also be recorded in the field notes for each day of sampling:

- Team members and their responsibilities
- Time of arrival/entry on Site and time of Site departure
- Other personnel on Site
- Summary of any meetings or discussions with City, contractor, or federal agency personnel
- Deviations from sampling plans, Site safety plans, and SAP procedures
- Changes in personnel and responsibilities with reasons for the changes
- Levels of safety protection
- Calibration readings for any equipment used, along with the equipment model and serial number

9.1.2 Photographs

Photographs will be taken at the sampling locations and at other areas of interest on Site or sampling area. They will serve to verify information entered in the daily field log forms. For each photograph taken, the following information will be written in the daily field log forms:

- Time, date, location, and weather conditions
- Description of the subject photographed
- Name of person taking the photograph

9.2 SAMPLE LABELING

All samples collected will be labeled in a clear and precise way for proper identification in the field and for tracking in the laboratory. The samples will have pre-assigned, identifiable, and unique numbers. At a minimum, the sample labels will contain the following information: station location, date of collection, analytical parameter(s), and method of preservation, if any.

9.3 SAMPLE CHAIN-OF-CUSTODY FORMS AND CUSTODY SEALS

All sample shipments for analyses will be accompanied by a chain-of-custody record. Chain-of-custody records will be completed and provided to the laboratories along with the samples each day. If multiple coolers are sent to a single laboratory on a single day, form(s) will be completed and sent with the samples for each cooler.

The chain-of-custody form will identify the contents of each shipment and maintain the custodial integrity of the samples. Generally, a sample is considered to be in someone's custody if it is either in someone’s physical possession, in someone's view, locked up, or kept in a secured area that is restricted to authorized personnel. Until the samples are provided to the laboratory, the custody of the samples will be the responsibility SCS. The sampling team leader or designee will sign the chain-of-custody form in the “relinquished by” box and note date and time.

A self-adhesive custody seal will be placed across the lid of each sample. The shipping containers in which samples are stored (usually a picnic cooler or ice chest) will be sealed with self-adhesive custody seals any time they are not in someone’s possession or view before shipping. All custody seals will be signed and dated.

10.0 QUALITY CONTROL

10.1 FIELD QUALITY CONTROL SAMPLES

10.1.1 Assessment of Field Contamination (Blanks)

10.1.1.1 Equipment Blanks

Equipment rinsate blanks will not be collected since it is anticipated that only single-use disposable equipment will be used.

10.1.1.2 Field Blanks

Field blanks will be collected to evaluate field sampling and ambient sources of cross-contamination. Field blanks will be collected by pouring organic-free (for organics) or deionized water (for inorganics) into sample containers. One field blank will be collected each day. The field blanks will be analyzed for Title 22 metals, Chromium VI, pH, and organochlorine pesticides.

The field blanks will be preserved, packaged, and sealed in the manner described for the environmental samples. A separate sample number and station number will be assigned to each sample, and it will be submitted blind to the laboratory.

10.1.1.3 Trip Blanks

Trip blanks will be not be collected since field blanks will be collected.

10.1.1.4 Temperature Blanks

For each cooler that is shipped or transported to an analytical laboratory a 40 milliliter (ml) volatile organic analysis (VOA) vial will be included and marked "temperature blank." This blank will be used by the sample custodian to check the temperature of samples upon receipt.

10.1.1.5 Assessment of Field Variability (Field Duplicate or Co-located Samples)

Duplicate soil samples will be collected at sample locations T2 (T24), T12 (T25), and SS1 (SS9). As no assessment data or previous sampling locations are available for the Site, these locations have been selected based on geographic distribution, anticipated order of collection, and/or requirements for the number of duplicate samples collected. Soil samples will be collected in glass jars with Teflon-lined lids, and the jars will be tightly closed.

Duplicate samples will be preserved, packaged, and sealed in the same manner as other samples of the same matrix. A separate sample number and station number will be assigned to each duplicate, and it will be submitted blind to the laboratory.

10.2 BACKGROUND SAMPLES

No background samples will be collected as part of this assessment.

10.3 FIELD SCREENING, INCLUDING CONFIRMATION SAMPLES, AND SPLIT SAMPLES

10.3.1 Field Screening Samples

Not applicable

10.3.2 Confirmation Samples (Field Screening)

Not applicable

10.4 LABORATORY QUALITY CONTROL SAMPLES

A routinely collected soil sample (a full 8-ounce glass jar with Teflon-lined lid) contains sufficient volume for both routine sample analysis and additional laboratory QC analyses. Therefore, a separate soil sample for laboratory QC purposes will not be collected.

At a minimum, one laboratory QC sample is required per 14 days or one per 20 samples (including blanks and duplicates), whichever is greater. If the sample event lasts longer than 14 days or involves collection of more than 20 samples per matrix, additional QC samples will be designated.

For this sampling event, samples collected from sampling locations T1 and T21 will be the designated laboratory QC samples.

As no assessment data or previous sampling locations are available for the Site, these sample locations have been selected based on geographic distribution, anticipated order of collection, and/or requirements for the number of duplicate samples collected.

11.0 FIELD VARIANCES

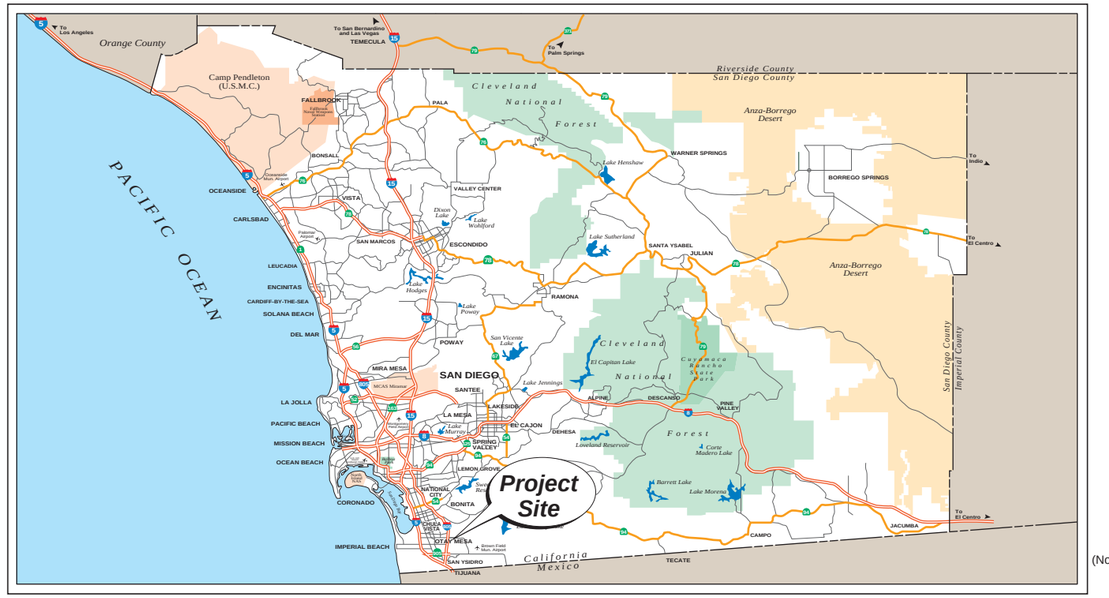
As conditions in the field may vary, it may become necessary to implement minor modifications to sampling procedures, methods, and activities as presented in this SAP. When appropriate, the QA Office will be notified, and a verbal approval will be obtained before implementing changes. Modifications to the approved SAP will be documented in the sampling project report.

If obvious contamination, other than what is anticipated, is encountered (based on PID readings, staining, odors, etc.) in the trenches, additional samples may be collected, and additional analyses may be conducted, if feasible.

12.0 FIELD HEALTH AND SAFETY PROCEDURES

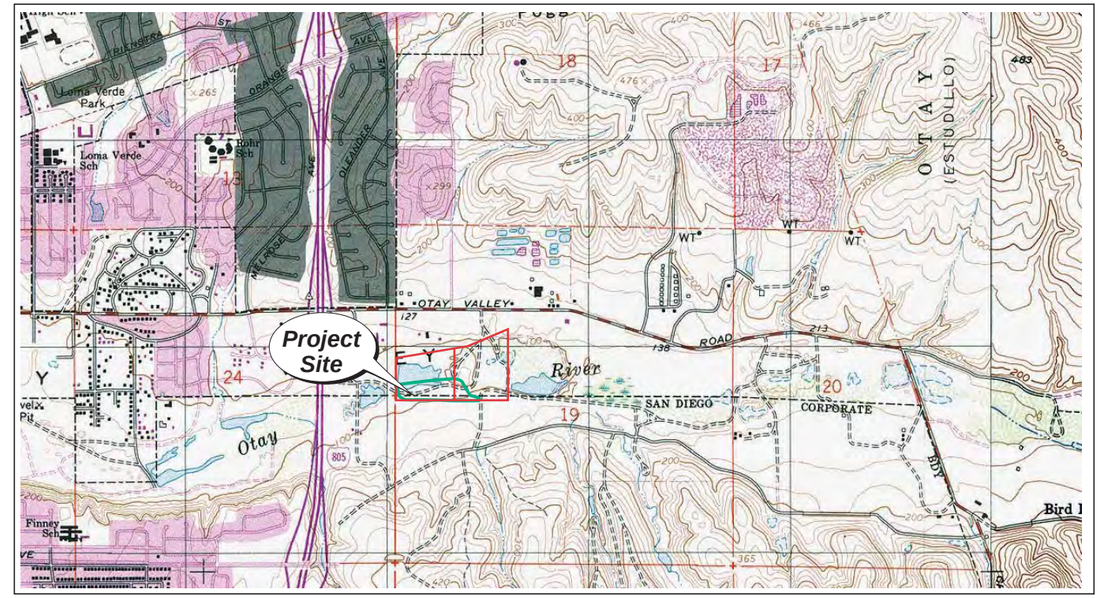
A health and safety plan for work conducted at the Site and workers within the “exclusion zone” is required pursuant to the regulations found in 29 Code of Federal Regulations (CFR) Part 1910.120 and California Code of Regulations (CCR), Title 8, Section 5192. Therefore, a health and safety plan will be prepared for the proposed work scope and will outline the potential chemical and physical hazards that may be encountered during the sampling activities. The appropriate personal protective equipment and emergency response procedures for the anticipated Site-specific chemical and physical hazards will be detailed in this plan including the route to the nearest hospital emergency room. SCS and contracted personnel involved with the proposed field work will be required to read and sign this document in order to encourage proper health and safety practices.

FIGURES



North (Not to scale)

REGIONAL SITE LOCATION



North

Reference:
U.S.G.S. 7.5 Minute Quadrangle map
Imperial Beach, California - 1977. Photo revised 1982.

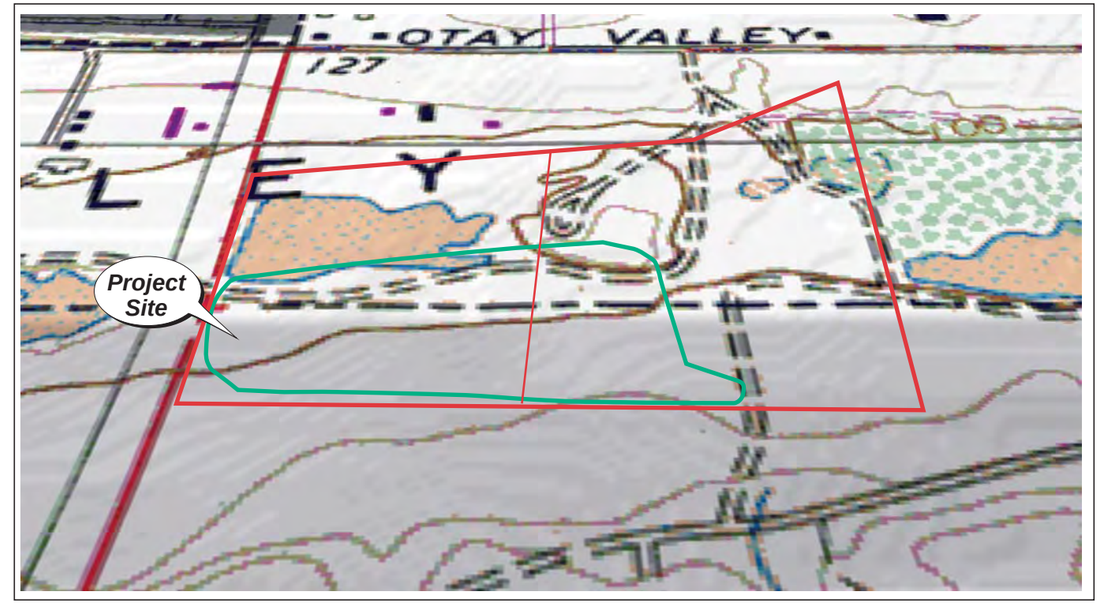
2-DIMENSIONAL SITE LOCATION

0 1,000 2,000 3,000
Approximate Graphic Scale in Feet

North (Not to scale)

Reference:
Google Earth Aerial Photograph
Chula Vista, California - December 2008

SITE AERIAL PHOTOGRAPH



North (Not to scale)

Reference:
U.S.G.S. 7.5 Minute Quadrangle map
Imperial Beach, California - 1977. Photo revised 1982.

3-DIMENSIONAL SITE LOCATION

Approximate parcel boundary

Approximate Site boundary

Disclaimer: This figure is based on available data. Actual conditions may differ. All locations and dimensions are approximate.

SCS ENGINEERS
Environmental Consultants
8799 Balboa Avenue, Suite 290
San Diego, California 92123

4-WAY SITE LOCATION MAP
City of Chula Vista
Shinohara II Burn Ash Site
Chula Vista, California

Project No.: 11012021.07

Figure 1

Date Drafted: 3/28/12

EXPLANATION

Approximate parcel boundary

Approximate Site boundary

Approximate Otay River boundary

Disclaimer: This figure is based on available data. Actual conditions may differ. All locations and dimensions are approximate.

North
Not to Scale

SCS ENGINEERS
Environmental Consultants
8799 Balboa Avenue, Suite 290
San Diego, California 92123

SITE AND SITE VICINITY PLAN
City of Chula Vista
Shinohara II Burn Site
Chula Vista, California

Project No.: 11012021.07

Figure 2

Date Drafted: 6/6/12

BOUNDARIES

APPROXIMATE PROPERTY LINE

APPROXIMATE RIVER LIMIT

ESTIMATED EDGE OF REFUSE

SURVEY

HAND EXCAVATION LOCATION
(COVER THICKNESS IN INCHES)

PROPOSED TRENCH LOCATION

TOPOGRAPHY CONTOURS

WHITE NUMBERS INDICATE ELEVATION IN FEET ABOVE MEAN SEA LEVEL

PROPOSED TRENCH SAMPLING LOCATION

PROPOSED SHALLOW SOIL SAMPLING LOCATION

0 50 100 150
Approximate Graphic Scale in Feet

North

Disclaimer: This figure is based on available data. Actual conditions may differ. All locations and dimensions are approximate.

SCS ENGINEERS
Environmental Consultants
8799 Balboa Avenue, Suite 290
San Diego, California 92123

PROPOSED SAMPLING LOCATIONS
City of Chula Vista
Shinohara II Burn Site
Chula Vista, California

Project No.: 11012021.07

Figure 3

Date Drafted: 6/6/12

APPENDIX A
LEA Workplan Approval Letter

NF

County of San Diego

JACK MILLER
DIRECTOR

SOLID WASTE LOCAL ENFORCEMENT AGENCY
5500 OVERLAND AVENUE, SUITE 110 SAN DIEGO, CA 92123
(858) 694-2888 FAX (858) 495-5004
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www.sdcdeh.org

ELIZABETH POZZEBON
ASSISTANT DIRECTOR

RECEIVED

APR 19 2012

SCS Engineers
San Diego Office

April 12, 2012

Ms. Carla Blackmar
Mr. Eric Crockett
City of Chula Vista
Development Services Department
276 Fourth Avenue
Chula Vista, CA 91910

SITE INVESTIGATION REPORT AND WORKPLAN FOR ADDITIONAL ASSESSMENT
SHINOHARA II BURN SITE
CHULA VISTA, CA
SWIS # 37-CR-0075

Dear Ms. Blackmar and Mr. Crockett:

The County of San Diego Solid Waste Local Enforcement Agency (LEA) has received and reviewed the *Site Investigation Report and Workplan for Additional Assessment – Shinohara II Burn Site*, prepared by SCS Engineers and dated March 30, 2012.

The workplan proposes the completion of up to 23 exploratory trenches throughout the site to evaluate the lateral and vertical extent of burn ash. One analytical sample will be collected for laboratory analysis from each trench, at varying depths. Soil samples will be analyzed for total metals, and the three samples with the highest lead concentrations will also be analyzed for soluble metals using a variety of test methods.

The work plan is approved.

Please notify the LEA a minimum of 48-hours prior to the beginning of the investigation work and provide the LEA with a written report at the completion of the investigation. Please notify the LEA of any long term delays in implementing the project.

If you have any questions, please call me at (858) 694-2801.

Sincerely,

MELSISSA PORTER, Environmental Health Specialist
Local Enforcement Agency

cc: Nicki Field, SCS Engineers
Rodney Lorang, County Counsel
LEA file

APPENDIX B
Calscience Quality Systems Manual and Supporting Documents

UNCONTROLLED COPY

QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL ANALYTICAL SERVICES

Calscience Environmental Laboratories, Inc.

Version 5.4
June 2011

Prepared By

Calscience Environmental Laboratories, Inc.
7440 Lincoln Way
Garden Grove, CA 92841-1427
Telephone: (714) 895-5494
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Based On
National Environmental Laboratory Accreditation Program (NELAP)
The NELAC Institute
Chapter 5 (Quality Systems)
NELAC Standard Effective July 01, 2010

Larry Lem
QUALITY ASSURANCE MANAGER

Linda Sharpenberg
TECHNICAL SERVICES MANAGER

Virginia Huang
TECHNICAL DIRECTOR

Steven L. Lane
LABORATORY DIRECTOR

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PREFACE TO THE QUALITY SYSTEMS MANUAL

Purpose

The purpose of this document is to provide implementation guidance on the establishment and management of quality systems for Calscience Environmental Laboratories, Inc and is based on the National Environmental Laboratory Accreditation Conference"s (NELAC) Quality System requirements.

Background

To be accredited under the National Environmental Laboratory Accreditation Program (NELAP), laboratories shall have a comprehensive quality system in place, the requirements for which are outlined in NELAP Chapter 5 (Quality Systems).

Project Specific Requirements

Project-specific requirements or regulations may supersede requirements contained in this manual. The laboratory bears the responsibility for meeting all **State requirements**. Nothing in this document relieves the laboratory from complying with contract requirements, or with **Federal, State, and/or local regulations**.

Results and Benefits

- **Standardization of Processes** – Because this manual provides the laboratory with a comprehensive set of requirements that meet the needs of many clients, as well as the NELAP, the laboratory may use it to create a standardized quality system. Ultimately, this standardization saves laboratory resources by establishing one set of consistent requirements for all environmental work. Primarily, the laboratory bears the responsibility for meeting all State requirements as outlined in their respective certification programs.

- **Deterrence of Improper, Unethical, or Illegal Actions** – Improper, unethical, or illegal activities committed by only a few laboratories have implications throughout the industry, with negative impacts on all laboratories. This manual establishes a minimum threshold program for all laboratories to use to deter and detect improper, unethical, or illegal actions.

- **Foundations for the Future** – A standardized approach to quality systems, shared by laboratories and the NELAP, paves the way for the standardization of other processes. For example, this manual might serve as a platform for a standardized strategy for Performance Based Measurement System (PBMS) implementation.

Document Format

This Calscience Environmental Laboratories, Inc. (Calscience) *Quality Systems Manual* (QSM) is designed to implement NELAP Chapter 5 (Quality Systems) and the NELAP Chapter 5 document serves as the primary text for this implementation manual. This Calscience QMS is also a complement to NELAP chapter 5. The section numbering has been changed from that of NELAP Chapter 5 as the manual is meant to be a stand-alone document. The number 5 has been eliminated from all section and subsection headings. However, second-level numbering has been retained to ensure maintenance of a parallel organization to the NELAC Quality Systems requirements. For instance, Section 5.4.2 in NELAP Chapter 5 (referencing Chapter 5 of the NELAC standards) is equivalent to Section 4.2 in this manual. In addition, there is one set of NELAC appendices.

ACROYNM LIST

°C: Degrees Celsius
ANSI/ASQC: American National Standards Institute/American Society for Quality Control
ASTM: American Society for Testing and Materials
CAS: Chemical Abstract Service
CCV: Continuing calibration verification
CFR: Code of Federal Regulations
CLP: Contract Laboratory Program
COC: Chain of custody
CV: Coefficient of variation
DO: Dissolved oxygen
DOC: Demonstration of capability
DQOs: Data quality objectives
EPA: Environmental Protection Agency
g/L: Grams per liter
GC/MS: Gas chromatography/mass spectrometry
ICP-MS: Inductively coupled plasma-mass spectrometer
ICV: Initial calibration verification
ID: Identifier
ISO/IEC: International Standards Organization/International Electrotechnical Commission
LCS: Laboratory control sample
LCSD: Laboratory control sample duplicate
LQMP: Laboratory Quality Management Plan
MDL: Method detection limit
mg/kg: Milligrams per kilogram
MS: Matrix spike
MSD: Matrix spike duplicate
NELAC: National Environmental Laboratory Accreditation Conference
NELAP: National Environmental Laboratory Accreditation Program
NIST: National Institute of Standards and Technology
OSHA: Occupational Safety and Health Administration
PBMS: Performance Based Measurement System
PC: Personal computer
PCBs: Polychlorinated biphenyls
PT: Proficiency testing
QA: Quality assurance
QAD: Quality Assurance Division (EPA)
QAMS: Quality Assurance Management Section
QAPP: Quality Assurance Project Plan
QSM: Quality Systems Manual
QC: Quality control
RL: Reporting limit
RPD: Relative percent difference
RSD: Relative standard deviation
SD: Serial dilutions
SOP: Standard operating procedure
TSS: Total suspended solids
UV: Ultraviolet
VOC: Volatile organic compound

QUALITY SYSTEMS

Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures that are delineated in a Quality Systems Manual (QSM) and followed to ensure and document the quality of the analytical data. Calscience, accredited under the National Environmental Accreditation Program (NELAP), assures implementation of all QA policies and the applicable QC procedures specified in this Manual. The QA policies, which establish essential QC procedures, are applicable to all areas of Calscience, regardless of size and complexity.

The intent of this document is to provide sufficient detail about quality management requirements so that all accrediting authorities evaluate laboratories consistently and uniformly.

The National Environmental Laboratory Accreditation Institute (TNI) is committed to the use of Performance Based Measurement Systems (PBMS) in environmental testing and provides the foundation for PBMS implementation in these standards. While this standard may not currently satisfy all the anticipated needs of PBMS, NELAC will address future needs within the context of State statutory and regulatory requirements and the finalized EPA implementation plans for PBMS.

Chapter 5 is organized according to the structure of ISO/IEC 17025, 2005. Where deemed necessary, specific areas within this Chapter may contain more information than specified by ISO/IEC 17025.

All items identified in this QSM shall be available for on-site inspection or data audit.

1.0 SCOPE

a) This QSM sets the general requirements that Calscience must successfully demonstrate to be recognized as competent to perform specific environmental tests.

b) This QSM includes additional requirements and information for assessing competence or for determining compliance by the organization or accrediting authority that grants approval.

   If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory demonstrates that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed.

c) Calscience uses this QSM in the development and implementation of its quality systems. Accreditation authorities use this NELAC based standard to assess the competence of environmental laboratories.

2.0 REFERENCES

See Appendix A.

3.0 DEFINITIONS

The relevant definitions from ISO/IEC Guide 2, ANSI/ASQC E-4, 1994, the EPA “Glossary of Quality Assurance Terms and Acronyms,” and the *International vocabulary of basic and general terms in metrology (VIM*) are applicable. The most relevant is quoted in Appendix A, Glossary, of Chapter 1 of NELAC, together with further definitions applicable for the purposes of this Standard.

4.0 ORGANIZATION AND MANAGEMENT

4.1 Legal Definition of Laboratory

Calscience is legally definable as evidenced by its business license, and current California Department of Health Services Environmental Laboratory Accreditation Program (CADHS ELAP) certificate. It is organized and operates in such a way that its facilities meet the requirements of the Standard. See the graphical presentations of the Organization and QA responsibility in Figures 1 and 2, respectively.

4.2 Organization

Calscience:

a) Has a managerial staff with the authority and resources necessary to discharge their duties;

b) Has processes to ensure that its personnel are free from any commercial, financial and other undue pressure that adversely affect the quality of their work;

c) Is organized in such a way that confidence in its independence of judgment and integrity is maintained at all times;

d) Specifies and documents the responsibility, authority, and interrelationship of all personnel who manage, perform or verify work affecting the quality of calibrations and tests;

   Such documentation includes:

   1) A clear description of the lines of responsibility in the laboratory, and is proportioned such that adequate supervision is ensured, and

   2) Job descriptions for all positions.

e) Provides supervision by persons familiar with the calibration or test methods and procedures, the objective of the calibration or test, and the assessment of the results.

   The ratio of supervisory to non-supervisory personnel ensures adequate supervision and adherence to laboratory procedures and accepted techniques.

f) Has a technical director who has overall responsibility for the technical operation of Calscience.

   The technical director certifies that personnel who perform the tests for which the laboratory is accredited have the appropriate educational and/or technical background. Such certification is documented.

   The technical director meets the requirements specified in the Accreditation Process. (See NELAC Section 4.1.1.1.)

g) Has a quality assurance manager who has responsibility for the quality system and its implementation.

   The quality assurance officer has direct access to the technical director and to the highest level of management at which decisions are made regarding laboratory policy or resources.

   The quality assurance manager (and/or his/her designees):

   1) Serves as the focal point for QA/QC activities, and is responsible for the oversight and/or review of quality control data;

2) Has functions independent from laboratory operations for which she/he has quality assurance oversight;

3) Is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence;

4) Has documented training and/or experience in QA/QC procedures and is knowledgeable in the quality system, as defined under NELAC;

5) Has a general knowledge of the analytical test methods for which data review is performed;

6) Arranges for and conduct internal audits as per Calscience QSM section 5.3 annually; and

7) Notifies Calscience management of deficiencies in the quality system and monitors corrective action.

h) Nominates, by way of the "Alternates List," deputies in case of absence of the technical director and/or the quality assurance officer;

i) Calscience makes every effort to ensure the protection of its clients' information as confidential and proprietary.

ii) Calscience is sensitive to the fact that much of the analytical work performed for clientele may be subject to litigatory processes. Calscience, therefore, holds all information in strict confidence with laboratory release only to the client.
iii) Information released to entities other than the client is performed only upon written request from the client.
iv) Due to the investigative nature of most site assessments, analytical information may become available to regulatory agencies or other evaluating entities during site assessment of the laboratory for the specific purpose of attaining laboratory certifications, accreditations, or evaluation of laboratory qualification for future work. During these occurrences, the laboratory will make every effort to maintain the confidence of client specific information.

j) For purposes of qualifying for and maintaining accreditation, participates in a proficiency test program as outlined in Chapter 2 of NELAC. Results of Calscience"s performance in rounds of proficiency testing are available by request.

5.0 QUALITY SYSTEM – ESTABLISHMENT, AUDITS, ESSENTIAL QUALITY CONTROLS, AND DATA VERIFICATION

5.1 Establishment

Calscience establishes and maintains quality systems based on the required elements contained in this Manual and appropriate to the type, range and volume of environmental testing activities it undertakes.

a) The elements of this quality system are documented in this quality manual.

b) The quality documentation is available for use by all laboratory personnel.

c) The laboratory defines and documents its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.

d) The laboratory management ensures that these policies and objectives are documented in the quality manual and are communicated to, understood and implemented by all laboratory personnel concerned.

i. All staff members are issued a copy of the quality manual at the commencement of work at Calscience. Employees read and endorse the following statement when they receive their quality manual: "By signature below, I acknowledge that I have received a copy of Revision [number] of Calscience"s Quality Assurance Program Manual dated [effective date of the subject manual]. Furthermore, I agree to read and abide by the policies contained therein."

ii. A controlled copy of the quality manual is also available at the designated data reduction areas.

e) The quality manual is maintained current under the responsibility of the quality assurance officer. This manual is reviewed on an annual basis or more frequently, and revised as necessary. The review process begins in January of each year, and concludes on/before March of the same year. Where no revision is required, the manual is reissued in its entirety and review is scheduled for January of the following year.

5.2 Quality Systems Manual (QSM) Elements

This quality systems manual and related quality documentation state Calscience's policies and operational procedures established in order to meet the requirements of this Standard.

This Manual lists on the title page: a document title; the laboratory's full name and address; the name, address, and telephone number of individuals responsible for the laboratory; the name of the quality assurance manager; the identification of all major organizational units that are covered by this quality manual and the effective date of the version.

This quality manual and related quality documentation also contains:

a) A quality ***policy statement***, including objectives and commitments, by top management;

i. Calscience Environmental Laboratories, Inc. (Calscience) is committed to providing the highest quality environmental analytical services available. To ensure the production of scientifically sound, legally defensible data of known and proven quality, an extensive Quality Assurance program has been developed and implemented. This document, Calscience"s Quality Systems Manual for Environmental Analytical Services, presents an overview of the essential elements of our Quality Assurance program. Calscience has modeled this systems manual after EPA guidelines as outlined in "Guidance for Quality Assurance Project Plans (EPA QA/G-5)", Office of Monitoring Systems and Quality Assurance, Office of Research and Development, U.S. EPA, EPA/240-R-02/009 December 2002. Calscience"s QA Program is closely monitored at the Corporate, Divisional, and Group levels, and relies on clearly defined objectives, well-documented procedures, a comprehensive quality assurance/quality control system, and management support for its effectiveness.

ii. This QA Program Systems Manual is designed to control and monitor the quality of data generated at Calscience. The essential elements described herein are geared toward generating data that is in compliance with federal regulatory requirements specified under the Clean Water Act, the Safe Drinking Water Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response, Compensation, and Liability Act, and applicable amendments, and state and DoD/DoE equivalents. Although the quality control requirements of these various programs are not completely consistent, each of the programs base data quality judgments on the following three types of information, the operational elements of each being described elsewhere in this manual.

⇨ Data which indicates the overall qualifications of the laboratory to perform environmental analyses;
⇨ Data which measures the laboratory"s daily performance using a specific method; and
⇨ Data which measures the effect of a specific matrix on the performance of a method.

iii. It is important to note that the QA guidelines presented herein will always apply unless adherence to specific Quality Assurance Project Plans (QAPPs) or client and/or regulatory agency specific requirements are directed. In these cases, the elements contained within specified direction or documentation shall supersede that contained herein.

iv. This manual is a living document subject to periodic modifications to comply with regulatory changes and technological advancements. All previous versions of this document are obsolete. Users are urged to contact Calscience to verify the current revision of this document.

b) The organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;

See Figure 1 Organizational Chart, and Figure 2 QA Responsibility Chart.

c) The relationship between management, technical operations, support services and the quality system;

d) Procedures to ensure that all records required under the NELAP are retained, as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force;

i. Ensuring a high quality work product in the environmental laboratory not only requires adherence to the quality issues discussed in the previous sections, but also requires the ability to effectively archive, restore, and protect the records that are generated.

ii. Procedures are in place to ensure that all records are retained. In addition, a documentation control system is employed to clearly indicate the time period during which a standard operating procedure, manual, or document was in force. These procedures are outlined in the laboratory standard operating procedure SOP-T002.

iii. All laboratory logbooks, instrument response printouts, completed analytical reports, chain-of-custodies, and laboratory support documentation are stored for a minimum of five years. Project specific data are stored in sequentially numbered project files and include copies of the applicable laboratory logbooks, instrument response printouts, completed analytical reports, chain-of-custodies, and any other pertinent supporting documentation.

iv. When complete, the project specific data are high speed optically scanned and transformed into digital CD media. Additional copies of these records are created at the time of scanning and are stored off-site for protection of the data. These records are stored for a minimum of five years.

v. Access to all systems is limited by use of log-in and password protection and is maintained by the system administrator.

vi. There are four forms of electronic data that are generated in the laboratory. Refer to Table 1 – Data Archiving Schedule on page 15 for a synopsis of general data archiving schedules.

vii. All electronic records are stored for a minimum of five years.

FIGURE 1: ORGANIZATIONAL CHART

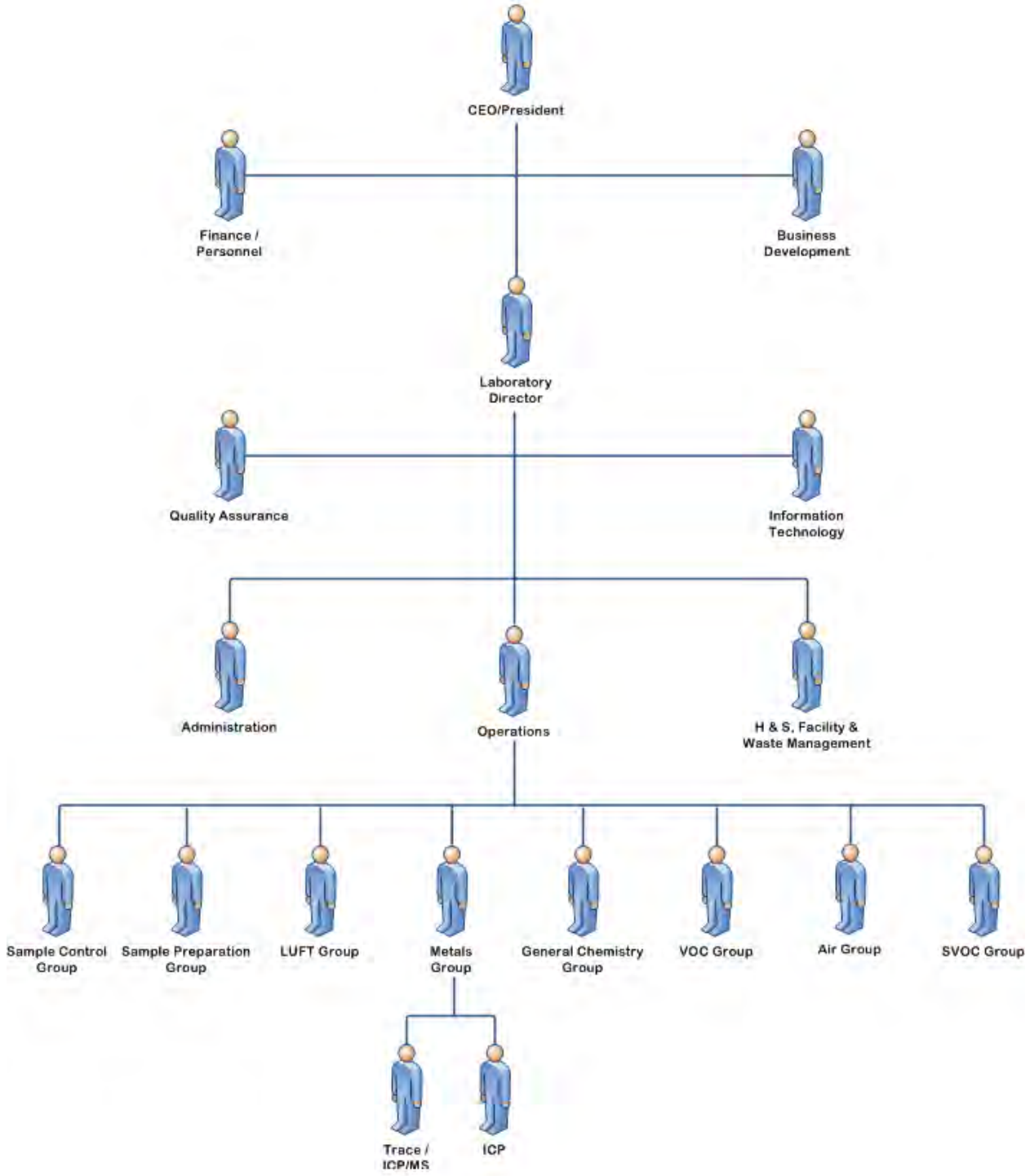

FIGURE 2: RESPONSIBILITY CHART

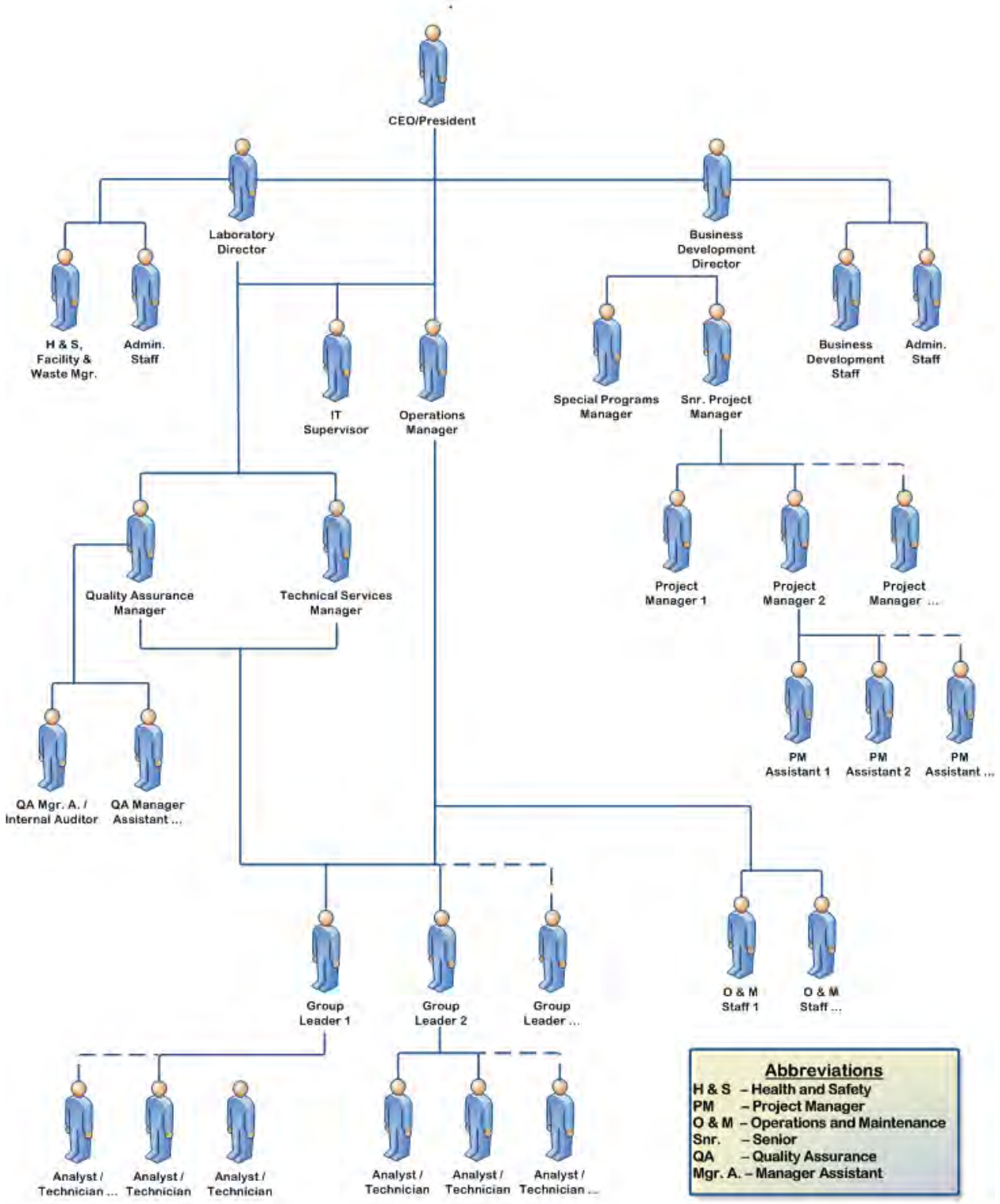

TABLE 1 – DATA ARCHIVING SCHEDULE

LIMS Database

Backup frequency:	Daily
Backup media:	Hard Disk
Backup software:	MS SQL Server Backup
Backup versions kept:	Ten previous versions
Onsite copy:	Redundancy by using mirrored hard drive

Instrument Data

Backup frequency:	Weekly
Backup media:	Quantum 4000 DLT Raid Tape and DLT Tape
Backup software:	Computer Associates ArcServIT
Backup versions kept:	All versions
Offsite copy:	One

Manual Data

Backup frequency:	Weekly
Backup media:	Quantum 4000 DLT Raid Tape and DLT Tape
Backup software:	Computer Associates ArcServIT
Backup versions kept:	All versions
Offsite copy:	One

Hard Copy Data

Backup media:	Digital CD
Backup software:	Xerox Pagis
Backup versions kept:	All versions
Offsite copy:	One

e) Job Descriptions, Roles and Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to their job function and the quality program as a whole.

The responsibility for quality lies with every employee at Calscience. As such, all employees have access to the Quality Assurance Manual and are responsible for knowing the content of this manual and upholding the standards therein. Each employee is expected to conduct themselves in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory"s SOPs.

The following descriptions define the primary roles and their relationship to the Quality Assurance Program.

Laboratory Director:

Calscience's Laboratory Director, through its President, is the final authority on all issues dealing with data quality and has the authority to require that procedures be amended or discontinued, or analytical results voided or repeated. He or she also has the authority to suspend or terminate employees on the grounds of non-compliance with QA/QC procedures. In addition, the Laboratory Director:

- ⇨ Ensures that Calscience remains current with all regulations which affect operations and disseminate all such changes in regulatory requirements to the QA Manager, Technical Director, and Group Leaders;

- ⇨ Provides one or more Technical Directors for the appropriate fields of testing. The name(s) of the Technical Director are included in the national database. (The Laboratory Director may also act in the Technical Director capacity.) If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director will designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 35 consecutive calendar days, the primary accrediting authority will be notified in writing;
- ⇨ Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented;
- ⇨ Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work;
- ⇨ Oversees the development and implementation of the QA Program which assures that all data generated will be scientifically sound, legally defensible, and of known quality;
- ⇨ In conjunction with the QA Manager, conducts annual reviews of the QA Program;
- ⇨ Oversees the implementation of new and revised QA procedures to improve data quality;
- ⇨ Ensures that appropriate corrective actions are taken to address analyses Identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director;
- ⇨ Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to;
- ⇨ Oversees all laboratory accreditation efforts; and
- ⇨ Oversees in-house training on quality assurance and control.

Operations Manager:

The Operations Manager manages and directs the analytical production sections of the laboratory. He or she reports directly to the Laboratory Director and assists in determining the most efficient instrument utilization. More specifically, they:

- ⇨ Evaluate the level of internal/external non-conformances for all departments;
- ⇨ Continuously evaluate production capacity and improves capacity utilization;
- ⇨ Continuously evaluate turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments;
- ⇨ Develop and improve the training of all analysts in cooperation with the Laboratory Director, QA Manager and Group Leaders, and in compliance with regulatory requirements;
- ⇨ Ensure that scheduled instrument maintenance is completed;
- ⇨ Are responsible for efficient utilization of supplies;
- ⇨ Constantly monitor and modify the processing of samples through the departments; and
- ⇨ Maintain sufficient personnel, equipment and supplies to achieve production goals.

Quality Assurance Manager:

The QA Manager has full authority through the Laboratory Director in matters dealing within the laboratory. The QA Manager can make recommendations to the Laboratory Director regarding the suspension or termination of employees on the grounds of non-compliance with QA/QC procedures. An alternate QA Manager is always assigned. In the absence of the primary designate, the alternate will act in the QA Manager"s capacity with the full authority of the position as allowed by Calscience governing documents. In addition, the QA Manager performs the following:

- ⇨ Maintains and updates the QAM on an annual basis;
- ⇨ Implements Calscience's QA Program;

- ⇨ Monitors the QA Program within the laboratory to ensure complete compliance with its objectives, QC procedures, holding times, and compliance with client or project specific data quality objectives;
- ⇨ Distributes performance evaluation (PE) samples on a routine basis to ensure the production of data that meets the objectives of its QA Program;
- ⇨ Maintains all SOPs used at Calscience;
- ⇨ Maintains records and archives of all PE results, audit comments, and customer inquiries concerning the QA program;
- ⇨ Performs statistical analyses of QC data and establish controls that accurately reflect the performance of the laboratory;
- ⇨ Conducts periodic performance and system audits to ensure compliance with the elements of Calscience"s QA Program;
- ⇨ Prescribes and monitors corrective action;
- ⇨ Serves as in-house client representative on all project inquiries involving data quality issues;
- ⇨ Coordinates data review process to ensure that thorough reviews are conducted on all project files;
- ⇨ Develops revisions to existing SOPs;
- ⇨ Reports the status of in-house QA/QC to the Laboratory Director;
- ⇨ Maintains records and archives of all QA/QC data including but not limited to method detection limit (MDL) studies, accuracy and precision control charts, and completed log books; and
- ⇨ Conducts and/or otherwise ensures that an adequate level of QA/QC training is conducted within the laboratory.

Quality Assurance Assistant:

The QA Assistant reports to the QA Manager and performs the following functions:

- ⇨ Assists the QA Manager and lab staff with internal audits, corrective action review and overall implementation of the QA program;
- ⇨ Generates and reviews, in conjunction with the QA Manager, Control Charts and Method Detection Limit (MDL) studies;
- ⇨ Reviews and revises SOPs as needed;
- ⇨ Distributes new SOPs to all applicable lab areas.

Business Development Director:

The Business Development Director reports to the Laboratory Director and serves as the interface between the laboratory"s technical departments and the laboratory"s clients. The staff consists of the Project Management team, Business Development team and satellite office Operations Managers. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- ⇨ Technical training and growth of the Project Management team;
- ⇨ Business liaison for the Project Management team;
- ⇨ Human resource management of the Project Management team;
- ⇨ Responsible for the review and negotiation of client contracts and terms and conditions;
- ⇨ Responsible for establishing standard fee schedules for the laboratory;
- ⇨ Responsible for preparation of proposals and quotes for clients and client prospects;
- ⇨ Accountable for response to client inquiries concerning sample status;
- ⇨ Responsible for assistance to clients regarding the resolution of problems concerning Chains-of-Custody;
- ⇨ Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory;
- ⇨ Notifying the department managers of incoming projects and sample delivery schedules;

- ⇨ Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates;
- ⇨ Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff;
- ⇨ Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness; and
- ⇨ Ensure that all non-conformance conditions are reported to the QA Manager, Operations Manager, and/or Laboratory Director via the Corrective Action process.

Technical Directors:

The Technical Director(s) report directly to the Laboratory Director. They are accountable for all analyses and analysts within their scope of responsibility. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts.

Specific responsibilities include, but are not limited to:

- ⇨ Coordinating, writing, and reviewing test methods and SOPs, with regard to quality, integrity, regulatory requirements and efficient production techniques;
- ⇨ Reviewing and approving, with input from the QA Manager and Business Development Manager, business proposals, in accordance with established procedures for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory"s capability and resources, the client"s expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding his requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved and requested by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented;
- ⇨ Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process and providing technical and troubleshooting expertise on routine and unusual or complex problems;
- ⇨ Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis; and
- ⇨ Coordinates audit responses with supervisors and QA Manager.

Group Leaders:

The Group Leaders report directly to the Operations Manager. They have the authority to accept or reject data based on pre-defined QC criteria. In addition, with the approval of the QA Manager, the Group Leaders may accept data that falls outside of normal QC limits if, in his or her professional judgment, there are technical justifications for the acceptance of such data. The circumstances must be well documented and any need for corrective action identified must be defined and initiated. The authority of the Group Leaders in QC related matters results directly from the QA Manager. The Group Leaders also

- ⇨ Actively support the implementation of Calscience's QA Program;
- ⇨ Ensure that their employees are in full compliance with Calscience's QA Program;
- ⇨ Maintain accurate SOPs (by reviewing and implementing updates) and enforce routine compliance with SOPs;
- ⇨ Conduct technical training of new staff and when modifications are made to existing procedures;
- ⇨ Maintain a work environment which emphasizes the importance of data quality;
- ⇨ Ensure all logbooks are current, reviewed and properly labeled or archived;

- ⇨ Ensure that all non-conformance conditions are reported to the QA Manager, Operations Manager, and/or Laboratory Director via Corrective Action reports;
- ⇨ Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Director, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, nonconformance issues, and the timely and accurate completion of performance evaluation samples and MDLs, for his/her department;.
- ⇨ Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and using appropriate documentation techniques;.
- ⇨ Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He or she is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments;
- ⇨ Provide written responses to external and internal audit issues; and
- ⇨ Provide support to all levels of Calscience Management.

Sample Control Group Leader:

The Sample Control Group Leader reports to the Operations Manager. The responsibilities are outlined below:

- ⇨ Direct the receipt, handling, labeling and proper storage of samples in compliance with laboratory procedures and policies;
- ⇨ Oversee the training of Sample Control Technicians regarding the above items;
- ⇨ Direct the logging of incoming samples into the LIMS and ensure the verification of data entry from login;
- ⇨ Oversee all sample courier operations;
- ⇨ Acts as a liaison between Project Managers and Analytical departments in respect to handling rush orders and resolving inconsistencies and problems with chain-of-custody forms, and routing of subcontracted analyses; and
- ⇨ Oversees the handling of samples in accordance with the Waste Disposal SOP, the Hazardous Waste Contingency Plan in the Chemical Hygiene/Safety Manual, and the U. S. Department of Agriculture requirements.

Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- ⇨ Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, the Data Integrity Policy, and project-specific QA plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- ⇨ Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on work sheets, bench sheets, preparation logbook, and/or a Non-Conformance report;
- ⇨ Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to the Group Leader and/or the QA Manager;
- ⇨ Perform 100% review of the data generated prior to entering and submitting for secondary level review; and
- ⇨ Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

Laboratory Technicians:

- ⇨ Prepare samples for analysis by weighing, extracting or digesting, filtering, or concentrating samples; and
- ⇨ Prepare method specific QC Samples with each preparation batch. All personnel must adhere to all QC procedures specified in the analytical method and in accordance to procedures or policies and are responsible for the full documentation of these procedures.

Project Manager:

The Project Manager normally reports to the Senior Project Manager and/or Business Development Director. Typical responsibilities include:

- ⇨ Serving as the laboratories‟ primary point of contact for assigned clients;
- ⇨ Working with laboratory chemists to resolve questions on data;
- ⇨ Scheduling of courier deliveries and pick-ups;
- ⇨ Tracking the progress of all laboratory production efforts;
- ⇨ Advising clients of any scheduling conflicts, possible delays, or other problems which may arise;
- ⇨ Resolving any questions or issues that clients may have with regard to our services, especially our reports;
- ⇨ Preparation of bottle kits for use by clients in their sampling efforts (as necessary);
- ⇨ Reviewing of reports/EDDs (Electronic Data Deliverables) as necessary prior to release;
- ⇨ Invoice preparation and review prior to release to client;
- ⇨ Serving as back-up contact person for other Project Managers in the event of his/her absence;
- ⇨ Coordination of all subcontracting efforts for projects assigned;
- ⇨ Preparation and implementation of program QAPPs (Quality Assurance Project Plans), if needed;
- ⇨ Preparation of project Case Narratives, as needed; and
- ⇨ Assembly of full data packages in accordance with company or client protocol, as needed.

Project Management Assistant:

The Project Management Assistant normally receives direction from the Project Manager(s) for which he/she is assigned. Typical responsibilities include:

- ⇨ Working with laboratory chemists to resolve questions on data;
- ⇨ Scheduling of courier deliveries and pick-ups;
- ⇨ Tracking the progress of all laboratory production efforts;
- ⇨ Advising clients of any scheduling conflicts, possible delays, or other problems which may arise;
- ⇨ Resolving any questions or issues that clients may have with regard to our services, especially our reports;
- ⇨ Preparation of bottle kits for use by clients in their sampling efforts;
- ⇨ Reviewing of reports/EDDs (Electronic Data Deliverables) prior to release;
- ⇨ Invoice preparation and review prior to release to client;
- ⇨ Serving as back-up contact person for the project managers in the event of his/her absence;
- ⇨ Coordination of all subcontracting efforts for projects assigned; and
- ⇨ Preparation and implementation of program QAPPs (Quality Assurance Project Plans), if needed.
- ⇨ As part of the administrative staff, this person may also be required to answer phones, do occasional filing, mailing, etc.

Environmental Health & Safety Manager:

The Health and Safety Manager reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. The EHS Manager is responsible for:

- ⇨ Conducting ongoing, necessary safety training and conducting new employee safety orientations;
- ⇨ Assisting in developing and maintaining the Chemical Hygiene/Safety Manual;
- ⇨ Oversees the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed; and
- ⇨ Completes accident reports, follows up on root causes and defines corrective actions.

Hazardous Waste Coordinator:

The Hazardous Waste Coordinator reports directly to the Environmental Health & Safety Manager. The duties of the HWC consist of:

- ⇨ Staying current with the hazardous waste regulations and continuing training on hazardous waste issues;
- ⇨ Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste;
- ⇨ Supervise the recording of the transfer of samples from refrigerated conditions to ambient conditions [in the sample disposal log sheets (SDLS)];
- ⇨ Check the records in SDLS against the logbook (LIMS) records;
- ⇨ Coordinate the collection of waste throughout the laboratory that will be disposed of through “Lab Packs”;
- ⇨ Coordinate and supervise Hazardous Waste Technician(s);
- ⇨ Dispose of solid waste to an assigned Tote;
- ⇨ Supervise the recording and disposal of acid and soil with methylene chloride extracts into appropriate drums;.
- ⇨ Prepare and discharge treated wastewater to the sewer system;
- ⇨ Maintain Uniform Hazardous Waste Manifest files;
- ⇨ Prepare weekly sample disposal schedules;
- ⇨ Coordinate and schedule waste pick-up;
- ⇨ Check all waste containers for appropriate labels; and
- ⇨ Maintain safe housekeeping and practices.

Education and Experience

Calscience makes every effort to hire analytical staff that posses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions are made based upon experience and an individual"s ability to learn as there are many in the industry that are more than competent, experts perhaps, who have not earned a college degree.

Selection of qualified individuals for employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Experience and specialized training may be accepted in lieu of a college degree (basic lab skills such as using a balance, aseptic or quantitation techniques, etc. are also considered).

Included in Section 5.2 (e) of this Quality Assurance Manual are the basic job titles and personnel responsibilities for anyone who manages, performs or verifies work affecting the quality of the environmental testing that the laboratory performs. Minimum education and training requirements are summarized in the following table:

When an analyst does not meet these minimum requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Group Leader, and are considered an analyst in training. The person supervising an analyst in training is directly accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

f) Identification of the laboratory's approved signatories; at a minimum, the title page of the quality manual has the signed and dated concurrence (with appropriate titles) of all responsible parties including the QA manager, technical director, and the laboratory director;

Job Type	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), Titrimetric and Gravimetric Analyses,	H.S. Diploma or GED	On the job training
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college with at least 1 year of college chemistry, or	2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pest, PCB, Herb, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry, or	5 years of prior analytical experience is required
Spectra Interpretation	A college degree in an applied science or 2 years of college Chemistry, and	2 years relevant experience, or 5 years of prior analytical experience is required
Group Leaders – Advanced Instrumentation	Bachelors Degree in an applied science with 16 semester hours in chemistry. An advanced (MS, PhD.) degree may substitute for one year of experience, and	2 years experience in the analytical technique for environmental analysis of representative analytes for which they will oversee
Group Leaders – Wet Chemistry (Basic Skills)	Associates degree in an applied science or 2 years of college with 16 semester hours in Chemistry, and	2 years relevant experience

g) The laboratory's procedures for achieving traceability of measurements;

h) A list of all test methods under which the laboratory performs its accredited testing may be found in the Index of Standard Operating Procedures, a separate document.

i) Mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;

j) Reference to the calibration and/or verification test procedures used;
Calibration procedures and verification of acceptability for each set of required calibrations are defined in Section 13 (Calibration) and Section 12 (Quality Control) of each standard operating procedure.

k) Procedures for handling samples received;

The generation of quality analytical data begins with the collection of the sample and, therefore, the integrity of the sample collection process is of importance to Calscience. Samples must be collected in such a way that foreign material is not introduced into the samples and that analytes of interest do not escape from the samples or degrade prior to their analysis. To ensure sample integrity and representativeness, the following items must be considered:

- ⇨ Samples must be collected in appropriate containers. In general, glass containers are used for organic analytes and polyethylene for inorganic/metal analytes;
- ⇨ Only new sample containers which are certified and documented clean in accordance with U.S. EPA OSWER Directive No. 9240.0-0.05 specifications shall be provided by Calscience for sample collection;
- ⇨ Certain extremely hazardous samples or samples that have the potential to become extremely hazardous will not be accepted. These include (but are not limited to)

    1. Radioactive samples that exceed background levels
    2. Biohazardous samples (medical wastes, body fluids, etc.)
    3. Explosive samples (Flash or gunpowder, ammunition, flares, etc.)
    4. Neurological or other toxic agents (Sarin, Anthrax, Ricin, etc.)

Calscience's chain-of-custody document is used to forward samples from the client to the laboratory. As the basic elements of most all chain-of-custody (COC)documents are similar, clientele may choose to use their own chain-of-custody document to forward samples to Calscience.

Any discrepancies in the COC must be documented on the Sample Receipt Form and resolved prior to analysis of samples. Further guidance may be found in SOP T100 “Sample Receipt and Log-In Procedures”.

Upon receipt by Calscience, samples proceed through an orderly processing sequence designed to ensure continuous integrity of both the sample and its documentation from sample receipt through its analysis and beyond.

All coolers that are received by the Sample Control Group undergo a preliminary examination in accordance with Part A of the Sample Receipt Form. Specifically, each sample is carefully examined for label identification, proper container (type and volume), chemical preservation when applicable, container condition, and chain-of-custody documentation consistency with sample labels. Discrepancies are noted on the Sample Receipt Form, the chain-of-custody and, if possible, discussed with the client prior to his or her departure. If this is not possible, the discrepancies are communicated to the client for resolution prior to the completion of the log-in process. The temperature of the cooler is measured and, with other observations, is recorded in Part B of the Sample Receipt Form. Additional comments are recorded in Part C of the Sample Receipt Form.

During the log-in process each sample is assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS), which stores all essential project information. Calscience maintains multiple security levels of access into LIMS to prevent unauthorized tampering/release of sample and project information.

Once all analyses for a sample have been completed and the sample container is returned to Sample Control, it shall remain in refrigerated storage for a period not less than 14 days following sample receipt unless the client requests return/forwarding of the sample. Following the 14-day refrigerated storage period, the samples are placed into ambient storage for another period not less than 14 days after which the samples are bulked into drums for later disposal.

Extended storage may be requested at prevailing per sample rates.

l) Reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;

A list of major equipment is kept up-to-date on the List of Major Assets, reference APPENDIX G. This, as well as a list of reference measurement standards and their certificates of calibration, is maintained by the QA Manager or the respective departments. In general, all calibrations and references should be traceable to NIST

m) Reference to procedures for calibration, verification and maintenance of equipment; Laboratory SOPs (T043 and T066) are available to staff for calibration, verification and maintenance of equipment. In general,

n) Reference to verification practices which may include interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;

Instrument calibration is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity such that required reporting limits can be met. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. The manufacturer"s guidelines, the analytical method, and/or the requirements of special contracts determine the frequency of calibration and the concentration of calibration standards, whichever is most applicable. The following are very general guidelines and are not meant to be all-inclusive. Detailed calibration procedures are specified in the SOP for each method performed.

Gas Chromatography/Mass Spectroscopy (GC/MS): Each day prior to analysis of samples, all GC/MS instruments are tuned with 4-bromofluorobenzene (BFB) for VOCs and decafluorotriphenylphosphine (DFTPP) for SVOCs in accordance with the tuning criteria specified in the applicable methods. Samples are not analyzed until the method-specific tuning requirements have been met.

After the tuning criteria are met, the instrument is then calibrated for all target analytes and an initial multipoint calibration curve established. The calibration curve is then validated by the analysis of a second source standard, referred to as the initial calibration verification (ICV). Alternatively, the previous calibration curve may be used if validated by a continuing calibration verification (CCV) standard. All target analytes are represented in the calibration and certain key target analytes referred to as system performance calibration compounds (SPCCs) and calibration check compounds (CCCs) are used for curve acceptance determination. For the initial calibration to be deemed acceptable, the SPCCs and CCCs must meet established acceptance criteria and must be re-evaluated and meet the acceptance criteria, at a minimum, every twelve (12) hours thereafter.

Non-GC/MS Chromatography: The field of chromatography involves a variety of instrumentation and detectors. While calibration standards and control criteria vary depending upon the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply uniformly. Each chromatographic system is calibrated prior to sample analysis. An initial multipoint calibration curve is generated using all target analytes. All target analytes must meet the acceptance criteria for the calibration to be deemed acceptable. The calibration curve is then validated by the analysis of a second source standard, referred to as the initial calibration verification (ICV). The continued validity of the initial multipoint calibration is verified every 12 hours using continuing calibration verification (CCV) standard containing all target analytes. If the CCV fails to meet the acceptance criteria, the system is re-calibrated and all samples analyzed since the last acceptable CCV must be re-analyzed.

Inductively Coupled Plasma Emission Spectroscopy: Initial calibration consists of a calibration blank (CB) plus one calibration standard. The calibration is verified by the re-analysis of the standard and initial calibration verification (ICV) standard. If the standard and the ICV fail to meet the acceptance criteria, the initial calibration is considered invalid and is re-performed.

Continuing calibration verification (CCV) consists of a mid-concentration standard plus a calibration blank (CB) analyzed every 10 samples and at the end of the sequence. If the CCV and/or CB fail to meet the acceptance criteria, the instrument must be re-calibrated and all samples analyzed since the previous acceptable CCV and/or CB must be re-analyzed.

ICP/MS Spectroscopy: Each day prior to the analysis of samples, all ICP/MS instruments undergo mass calibration and resolution checks prior to initial calibration. Initial calibration consists of a calibration blank (CB) and at least one calibration standard. The calibration is verified by the re-analysis of the standard and initial calibration verification (ICV) standards. If the standard and the ICV fail to meet the acceptance criteria, the initial calibration is considered invalid and is re-performed.

Continuing calibration verification (CCV) consists of a mid-concentration standard plus a calibration blank (CB) analyzed every 10 samples and at the end of the sequence. If the CCV and/or CB fail to meet the acceptance criteria, the instrument must be re-calibrated and all samples analyzed since the previous acceptable CCV and/or CB must be re-analyzed.

Cold Vapor Atomic Absorption Spectroscopy: Initial calibration consists of a calibration blank plus a series of at least 5 standards. The calibration curve is then validated by the analysis of a second source standard, referred to as the initial calibration verification (ICV). Continuing calibration verification (CCV) consists of midpoint calibration standard plus a continuing calibration blank (CCB) analyzed every 10 samples and at the end of the sequence. If the CCV and/or CCB fail to meet the acceptance criteria, the instrument must be re-calibrated and all samples analyzed since the previous acceptable CCV and/or CCB must be re-analyzed. If the calibration blanks contain target analyte concentrations exceeding the acceptance limits, the cause must be determined and corrected.

Flame and Graphite Furnace Atomic Absorption Spectroscopy: Initial calibration consists of a calibration blank plus a low, medium, and high calibration standard. Continuing calibration verification (CCV) consists of midpoint calibration standard plus a continuing calibration blank (CCB) analyzed every 10 samples and at the end of the sequence. If the CCV and/or CCB fail to meet the acceptance criteria, the instrument must be re-calibrated and all samples analyzed since the previous acceptable CCV and/or CCB must be re-analyzed. If the calibration blanks contain target analyte concentrations exceeding the acceptance limits, the cause must be determined and corrected.

General Inorganic Analyses: General inorganic (non-metal) analyses involve a variety of instrumental and wet chemistry techniques. While calibration procedures vary depending on the type of instrumentation and methodology, the general principles of calibration apply universally. Each system or method is initially calibrated using standards prior to analyses being conducted with continual verification that the calibration remains acceptable throughout analytical processing. If continual calibration verification fails to meet the acceptance criteria, the instrument must be re-calibrated and all samples analyzed since the previous acceptable CCV must be re-analyzed.

o) Procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;

These procedures may be found in SOP-T015 (Correction/Prevention of Errors in Test Records) and SOP-T022 (Corrective/Preventive Actions).

p) The laboratory management arrangements for permitting exceptions and departures from documented policies and procedures or from standard specifications;

Calscience"s SOPs are in substantial conformity with their corresponding published method references. Departure from approved SOPs shall be approved if necessary or appropriate due to the nature or composition of the sample or otherwise based on the reasonable judgment of Calscience"s Laboratory Director, Technical Director, or QA Manager.

Departures shall be made on a case-by-case basis consistent with recognized standards of the industry. In no case shall departures be approved without written communication between Calscience and the affected client.

q) Procedures for dealing with complaints;

Procedures for dealing with complaints may be found in SOP-T018, Handling of Inquiries and Complaints.

r) Procedures for protecting confidentiality (including national security concerns) and proprietary rights;

Calscience is sensitive to the fact that much of the analytical work performed for clientele may be subject to litigatory processes. Calscience, therefore, holds all information in strict confidence with laboratory release only to the client or designee. Information released to entities other than the client is performed only upon written, facsimile or e-mail request from the client.

Due to the investigative nature of most site assessments, analytical information may become available to regulatory agencies or other evaluating entities during site assessment of the laboratory for the specific purpose of attaining laboratory certifications, accreditations, or evaluation of laboratory qualification for future work. During these occurrences, the laboratory will make its best effort to maintain the confidence of client specific information.

s) Procedures for audits and data review;

Calscience participates in a wide variety of system and performance audits conducted by numerous federal and state agencies, as well as through its major clientele. These audits are conducted to verify that analytical data produced conforms to industry standards on a routine basis.

A System Audit is a qualitative evaluation of the measurement systems utilized at Calscience, specifically, that Calscience has, in place, the necessary facilities, staff, procedures, equipment, and instrumentation to generate acceptable data. This type of audit typically involves an on-site inspection of the laboratory facility, operations, and interview of personnel by the auditing agency.

A Performance Audit verifies the ability of Calscience to correctly identify and quantitate compounds in blind check samples. This type of audit normally is conducted by the auditing agency through laboratory participation in round robin Performance Evaluation (PE) programs. Examples of current PE program involvement include those offered by commercial suppliers like ERA (WS/WP/SOIL and DMR-QA), or other inter-laboratory studies not required for certification but done to ensure laboratory performance, as well as programs administered by major industry.

Outliers in required PE samples will be investigated and corrective actions documented using the Corrective/Preventive Action Record.

Should the result of any audit detect a significant error, which has been identified to adversely affect released data, the situation shall be thoroughly investigated. Corrective measures shall be enacted to include system re-evaluation, the determined affect on released data and client notification, as necessary. These measures shall be documented using the Corrective/Preventive Action Record.

t) Processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training;

Quality control begins prior to sample(s) receipt at the laboratory. The selection of well qualified personnel, based upon education and/or experience is the first step in successful laboratory management. A thorough screening of job applicants and selection of the best candidate to fulfill a well-defined need is as important an aspect of a successful QA/QC program as a careful review of analytical data.

Employee training and approval procedures used at Calscience are specified in SOP-T010, "Employee Training", and includes but is not limited to the following:

- ⇨ A thorough understanding of the applicable regulatory method and Calscience SOP;
- ⇨ A review of Calscience's QA Program Manual and thorough understanding of the specifics contained therein that are directly related to the analysis to be performed;
- ⇨ Instruction by the applicable Group Leader on all aspects of the analytical procedure;
- ⇨ Performance of analyses under supervision of experienced laboratory personnel, which shall include analysis of blind QC check samples, when deemed appropriate;
- ⇨ Participation in in-house seminars on analytical methodologies and procedures;
- ⇨ Participation in job related seminars outside of the laboratory; and
- ⇨ Participation in conventions and meetings, i.e., ACS, etc.

u) Ethics policy statement developed by the laboratory and processes/procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical, or illegal actions;

A vital part of Calscience Environmental Laboratories" analytical laboratory services is their Laboratory Ethics Training Program. An effective program starts with an Ethics Policy Statement that is supported by all staff, and is reinforced with initial and ongoing ethics training.

"It shall be the policy of Calscience to conduct all business with integrity and in an ethical manner. It is a basic and expected responsibility of each staff member and manager to hold to the highest ethical standard of professional conduct in the performance of all duties."

A proactive ethics training program is the most effective means of deterring and detecting improper, unethical, or illegal actions in the laboratory. There are six facets to the program: (1) clearly define improper, unethical, and illegal actions; (2) outline elements of prevention and detection programs for improper, unethical, or illegal actions; and (3) identify examples of inappropriate (i.e., potentially fraudulent) laboratory practices; (4) Annual Ethics and Data Integrity Training to be documented and maintained in the personnel file of each employee., (5) Documented training on new revisions of the Quality Systems Manual (QSM) and for new employees as needed. (6) Signed Ethics and Data Integrity Agreement (to be completed for new employees and annually thereafter)

<u>Definition of Improper, Unethical, and Illegal Actions</u>

Improper actions are defined as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional.

Unethical or illegal actions are defined as the deliberate falsification of analytical or quality assurance results, where failed method or contractual requirements are made to appear acceptable.

Prevention of laboratory improper, unethical, or illegal actions begins with a zero-tolerance philosophy established by management. Improper, unethical, or illegal actions are detected through the implementation of oversight protocols.

<u>Prevention and Detection Program for Improper, Unethical, or Illegal Actions</u>

Calscience management has implemented a variety of proactive measures to promote prevention and detection of improper, unethical, or illegal activities. The following components constitute the basic program:

- ⇨ Data Integrity Standard Operating Procedure (SOP) T065
- ⇨ Data Integrity Documentation Procedures
- ⇨ An Ethics and Data Integrity Agreement that is read and signed by all personnel;

- ⇨ Initial and annual ethics training;
- ⇨ Internal audits;
- ⇨ Inclusion of anti-fraud language in subcontracts;
- ⇨ Analyst notation and sign-off on manual integration changes to data;
- ⇨ Active use of electronic audit functions when they are available in the instrument software; and
- ⇨ A "no-fault" policy that encourages laboratory personnel to come forward and report fraudulent activities.

A proactive, "beyond the basics" approach to the prevention of improper, unethical, or illegal actions are a necessary part of laboratory management. As such, in addition to the requirements above, Calscience has a designated ombudsman (data integrity officer) to whom laboratory personnel can report improper, unethical, or illegal practices, or provide routine communication of training, lectures, and changes in policy intended to reduce improper, unethical, or illegal actions.

Examples of Improper, Unethical, or Illegal Practices

Documentation that clearly shows how all analytical values were obtained are maintained by Calscience and supplied to the data user as needed. To avoid miscommunication, Calscience clearly documents all errors, mistakes, and basis for manual integrations within the project file and case narrative as applicable. Notification is also made to the appropriate supervisor so that appropriate corrective actions can be initiated. Gross deviations from specified procedures are investigated for potential improper, unethical, or illegal actions, and findings of fraud are fully investigated by senior management. Examples of improper, unethical, or illegal practices are identified below:

- ⇨ Improper use of manual integrations to meet calibration or method QC criteria (for example, peak shaving or peak enhancement are considered improper, unethical, or illegal actions if performed solely to meet QC requirements);
- ⇨ Intentional misrepresentation of the date or time of analysis (for example, intentionally resetting a computer system"s or instrument"s date and/or time to make it appear that a time/date requirement was met);
- ⇨ Falsification of results to meet method requirements;
- ⇨ Reporting of results without analyses to support (i.e., dry-labbing);
- ⇨ Selective exclusion of data to meet QC criteria (for example, initial calibration points dropped without technical or statistical justification);
- ⇨ Misrepresentation of laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported, or QC control limits presented within QAPP that are not indicative of historical laboratory performance or used for batch control;
- ⇨ Notation of matrix inference as basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices (for example, method blanks or laboratory control samples);
- ⇨ Unwarranted manipulation of computer software (for example, improper background subtraction to meet ion abundance criteria for GC/MS tuning, chromatographic baseline manipulations);
- ⇨ Improper alteration of analytical conditions (for example, modifying EM voltage, changing GC temperature program to shorter analytical run time) from standard analysis to sample analysis;
- ⇨ Misrepresentation of QC samples (for example, adding surrogates after sample extraction, omitting sample preparation steps for QC samples, over- or under-spiking); and
- ⇨ Reporting of results from the analysis of one sample for those of another.

v) Reference to procedures for reporting analytical results;

Standard operating procedures pertaining to the reporting of results are available to all laboratory personnel. They are: SOP-T009, Significant Figures, Rounding, and Reporting of Results; SOP-T025, Reporting of Tentatively Identified Compounds (TICs); and T-026, Reporting of Data Qualifiers.

All analytical data generated within Calscience is thoroughly checked for accuracy and completeness. The data validation process consists of data generation, reduction, and four levels of review as described below.

The analyst generating the analytical data has the primary responsibility for its correctness and completeness. All data is generated and reduced following protocols specified in the appropriate SOPs. Each analyst reviews the quality of his or her work based upon an established set of guidelines specified in the SOPs or as specified by project requirements. The analyst reviews the data package to ensure that:

- ⇨ Holding times have not been exceeded;
- ⇨ Sample preparation information is correct and complete;
- ⇨ Analysis information is correct and complete;
- ⇨ The appropriate procedures were employed;
- ⇨ Analytical results are correct and complete;
- ⇨ All associated QC is within established control limits and, if not, out-of-control forms are completed thoroughly explaining the cause and corrective action taken;
- ⇨ Any special sample preparation and analytical requirements have been met; and
- ⇨ Documentation is complete, i.e., all anomalies in the preparation and analysis have been documented; out-of-control forms, if required, are complete, etc.

The data reduction and validation steps are documented, signed, and dated by the analyst on the QC Review coversheet accompanying each data package. This initial review step, performed by the analyst, is designated as primary review. The analyst then forwards the data package to his or her Group Leader, or designated data reviewer, who performs a secondary review. Secondary reviews consist of an independent check equivalent to that of the primary review and are designed to ensure that:

- ⇨ Calibration data is scientifically sound, appropriate to the method, and completely documented;
- ⇨ QC data is within established guidelines or reported with appropriate clarification/qualification;
- ⇨ Qualitative identification of sample components is correct;
- ⇨ Quantitative results are correct;
- ⇨ Documentation is complete and any anomalies properly addressed and documented;
- ⇨ The data is ready for incorporation into the final report package; and
- ⇨ The data package is complete and ready for archiving.

A significant component of the secondary review is the documentation of any errors that have been identified and corrected during the review process. Calscience believes that the data package that is submitted for a secondary review should be free from errors. Errors that are discovered are documented and formally transmitted to the appropriate Group Leader. The cause of the errors are then addressed by additional training or clarification of procedures (SOP revisions) to ensure that similar errors do not recur and high quality data will be generated.

Signature of Data Reviewer and the date of review document the completion of secondary reviews on the QC Review coversheet. These constitute approval for data release and generation of analytical report.

During both of the QC review processes, 100% of the raw data associated with the entire project is available to the reviewer. Data packages are checked back to the raw data as deemed necessary by the reviewer.

Following draft report generation, the report is reviewed by the Project Manager to ensure that the data set and quality control data is complete and meets the specific requirements of the project. When available, the data is also evaluated against historical site information. Once all requested analytical work has been verified as complete, a final report is generated and signed by the Project Manager.

Following approval for release by the Project Manager, the Quality Assurance Manager or Designee to ensure that the analytical and quality control data is correct performs a final review. The Quality Assurance Manager may review 10% of the project files back to the raw data as an additional check.

A variety of reporting formats, from Portable Document File (PDF), normal typed reports to computerized data tables to complex reports discussing regulatory issues are available. In general, Calscience reports contain the following information.

Analytical Data

Analytical data is reported by sample identification (both client and laboratory) and test. Pertinent information including date(s) sampled, received, prepared, and analyzed; any required data qualifiers are included on each results page. The reporting limit for each method analyte is also listed. Additional data may include Method Detection Limits (MDLs).

QC Data

A QC Summary is provided with each final report. Unless otherwise specified in a QAPP or requested by the client, QC Summaries include results for method blanks, matrix spikes, matrix spike duplicates, and surrogate spikes. Laboratory control sample and method blank surrogates are routinely included if matrix interference results in a QC outlier. The effective control limits for the reported QC values are also provided on the QC Summary as well as explanations for any QC outliers. Case Narratives may be included as appropriate.

As required for the project, data reports from “results only” through “full CLP-like” will be generated and provided. Included in this range are reports for the major DoD programs including NFESC, AFCEE, and USACE.

Methodology

References for the preparative and analytical methodology employed is included on all preliminary or final analytical reports.

Signatory

Final reports are ready for release to the client following review and approval by the Project Manager, as evidenced by his/her signature on the final report cover page.

Preliminary Data

Upon client request, preliminary data shall be released prior to completion of a full QC review. Preliminary data is subject to change pending QC review and, therefore, shall be clearly marked as “Preliminary”. This qualification is provided as notification to the client that the data review process has not been completed yet and that the data is subject to possible modification resulting therefrom.

Revised Data

Analytical reports that have been revised for any reason from the original sent report shall be noted as being revised with a report note, case narrative or indication as to the revision.

Formatting

At a minimum, an analytical report shall consist of the Report Cover Page, Analytical Results, QA/QC Data (Default), Footnotes/Comments Page, Sample Receipt Form and COC. Paginated reports shall be employed for all reports unless used for non-NELAP analysis.

w) A Table of Contents and applicable lists of references and glossaries, and appendices.

5.3 Audits

5.3.1 Internal Audits

The laboratory arranges streamlined quarterly and comprehensive annual internal audits to verify that its operations continue to comply with the requirements of the laboratory"s said quality system. The quality assurance officer or the Quality Assurance Assistant plans and organizes audits as required by a predetermined schedule and requested by management. The Quality Assurance Assistant, independent of the activity to be audited, will carry out such audits following the procedures noted in SOP T028, Internal Audit Procedures. Personnel do not audit their own activities except when it can be demonstrated that an effective audit will be carried out. Where the audit findings cast doubt on the correctness or validity of the laboratory's calibrations or test results, the laboratory takes immediate corrective action and immediately notifies, in writing, any client whose work was involved.

Any outside audit findings will also be included in the Internal Audits.

5.3.2 Management Review

Calscience management conducts an annual review of its quality system and its testing and calibration activities to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. This review takes account of reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of inter-laboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions, and other relevant factors. The laboratory shall have a procedure for review by management, and maintain records of review findings and actions. Reference section 18.1 of this QSM and SOP T030 for more detailed descriptions.

5.3.3 Audit Review

All audit and review findings and any corrective actions that arise from them are documented. The laboratory management ensures that these actions are discharged within the agreed time frame as indicated in the quality manual and/or SOPs.

5.3.4 Performance Audits

In addition to periodic audits, the laboratory ensures the quality of results provided to clients by implementing checks to monitor the quality of the laboratory"s analytical activities. Examples of such checks are:

a) Internal quality control procedures using statistical techniques (see Section 5.4 below);

b) Participation in proficiency testing or other interlaboratory comparisons;

c) Use of certified reference materials and/or in-house quality control using secondary reference materials as specified in Calscience QSM Section 5.4;

d) Replicate testing using the same or different test methods;

e) Re-testing of retained samples;

    e) Correlation of results for different but related analysis of a sample (for example, total phosphorus should be greater than or equal to orthophosphate).

5.3.5 Corrective / Preventive Actions

a) In addition to providing acceptance criteria and specific protocols for corrective/preventive actions in SOP-T022, the laboratory implements general procedures to be followed to determine when departures from documented policies, procedures and quality control have occurred. These procedures include but are not limited to the following:

   1) Identify the individual(s) responsible for assessing each QC data type;

   2) Identify the individual(s) responsible for initiating and/or recommending corrective/preventive actions;

   3) Define how the analyst shall treat a data set if the associated QC measurements are unacceptable;

   4) Specify how out-of-control situations and subsequent corrective actions are to be documented; and

   5) Specify procedures for management (including the QA officer) to review corrective/preventive action reports.

b) To the extent possible, sample results are reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data are to be reported, all samples associated with the failed quality control measure are reported with the appropriate data qualifier(s).

5.4 Essential Quality Control Procedures

These general quality control principles apply, where applicable, to all testing at Calscience. The manner in which each is implemented is dependent on the types of tests performed by the laboratory and is further described in Appendix D and in SOP-T020, Internal Quality Control Checks. The standards for any given test type assures that the applicable principles are addressed:

a) All laboratories have detailed written protocols in place to monitor the following quality controls:

   1) Positive and negative controls (blanks, spikes, reference toxicants, etc.) to monitor tests;

   2) Tests to define the variability and/or repeatability of the laboratory results such as replicates;

   3) Measures to assure the accuracy of the test method including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

   4) Measures to evaluate test method capability, such as detection limits and quantitation limits or range of applicability such as linearity;

   5) Selection of appropriate formulae to reduce raw data to final results such as regression analysis, comparison to internal/external standard calculations, and statistical analyses;

   6) Selection and use of reagents and standards of appropriate quality;

   7) Measures to assure the selectivity of the test for its intended purpose; and

   8) Measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the test method, such as temperature, humidity, light or specific instrument conditions.

b) All quality control measures are assessed and evaluated on an on-going basis, and quality control acceptance criteria are used to determine the usability of the data. (See Appendix D.)

c) The laboratory has procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist. (See Calscience QSM Section 11.2, Sample Acceptance Policy.)

d) The quality control protocols specified in the method manual (Calscience QSM Section 10.1.2) is followed. Calscience ensures that the essential standards outlined in NELAC 5, Appendix D, or mandated methods or regulations (whichever are more stringent) are incorporated into the method manuals. When it is not apparent which is more stringent the QC in the mandated method or regulations is to be followed.

   The essential quality control measures for testing are found in Appendix D.

6.0 PERSONNEL

6.1 General Requirements for Laboratory Staff

Calscience"s testing departments have a sufficient level of personnel with the necessary education, training, technical knowledge and experience to perform the assigned functions.

All personnel are responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function. Each technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function and a general knowledge of laboratory operations, test methods, quality assurance/quality control procedures and records management.

6.2 Laboratory Management Responsibilities

In addition to Calscience QSM Section 4.2.d, the laboratory management:

a) Defines the minimum level of qualification, experience and skills necessary for all positions in the laboratory. In addition to education and/or experience, basic laboratory skills such as using a balance and quantitative techniques, are considered.

b) Ensures that all technical laboratory staff members demonstrate capability in the activities for which they are responsible. Such demonstration is documented (See Appendix C). Note: In departments with specialized “work cells” (a well-defined group of analysts that together perform the method analysis), the group as a unit meets the above criteria and this demonstration is fully documented.

c) Ensures that the training of each member of the technical staff is kept up-to-date (on-going) by the following:

   1) Keeping evidence on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation that relates to his/her job responsibilities.

   2) Documenting training courses or workshops on specific equipment, analytical techniques, or laboratory procedures.

   3) Documenting employee attendance at training courses on ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions. Keeping on file evidence that demonstrates that each employee has read, acknowledges, and understands their personal ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions.

4) Maintains up-to-date analyst training records that contain a certification that technical personnel have read, understood and agreed to perform the most recent version of the test method (the approved method or SOP as defined by the laboratory document control system, Calscience QSM Section 5.2.d) and documentation of continued proficiency by at least one of the following once per year:

   i. Acceptable performance of a blind sample (single blind to the analyst);

   ii. Another demonstration of capability;

   iii. Successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624, or 5035/8260) would only require documentation for one of the test methods;

   iv. At least four consecutive laboratory control samples with acceptable levels of precision and accuracy;

   v. If i-iv cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.

d) Documents all analytical and operational activities of the laboratory;

e) Supervises all personnel employed by the laboratory;

f) Ensures that all sample acceptance criteria (Calscience QSM Section 11.0) are verified and that samples are logged into the sample tracking system and properly labeled and stored.

g) Documents the quality of all data reported by the laboratory.

h) Develops a proactive program for the prevention and detection of improper, unethical, or illegal actions. Components of this program could include: internal proficiency testing (single and double blind); post-analysis electronic and magnetic tape audits; effective reward program to improve employee vigilance and co-monitoring; and separate SOPs identifying appropriate and inappropriate laboratory and instrument manipulation practices.

6.2.1 Ownership Transfer / Out of Business

a) In the event that the laboratory transfers ownership or goes out of business, Calscience Environmental Laboratories (CEL) will ensure that the records are maintained or transferred according to client instruction.

b) Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives will be clearly established. In cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records will be followed.

c) In the event that the laboratory goes out of business, all records will revert to the control of the client or regulatory agency, as applicable. As much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

6.3 Records

Records on the relevant qualifications, training, skills and experience of the technical personnel are maintained by the laboratory (see Calscience QSM Section 6.2.c), including records on demonstrated proficiency for each laboratory test method, such as the criteria outlined in Calscience QSM Section 10.5 for chemical testing.

7.0 PHYSICAL FACILITIES – ACCOMMODATION AND ENVIRONMENT

7.1 Environment

a) Laboratory accommodations, test areas, energy sources, lighting, heating and ventilation are such that they facilitate proper performance of tests.

b) The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of the measurements. Particular care shall be taken when such activities are undertaken at sites other than the permanent laboratory premises.

c) The laboratory shall provide for the effective monitoring, control and recording of environmental conditions as appropriate. Such environmental conditions may include biological sterility, dust, electromagnetic interference, humidity, main voltage, temperature, and sound and vibration levels.

d) In instances where monitoring or control of any of the above-mentioned items is specified in a test method or by regulation, the laboratory meets and documents adherence to the laboratory facility requirements.

7.2 Work Areas

a) There is effective separation between neighboring areas when the activities therein are incompatible including volatile organic chemicals handling areas.

b) Access to and use of all areas affecting the quality of these activities are defined and controlled.

c) Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality.

d) Workspaces are available to ensure an unencumbered work area. Work areas include:

   1) Access and entryways to the laboratory;

   2) Sample receipt areas;

   3) Sample storage areas;

   4) Chemical and waste storage areas; and

   5) Data handling and storage areas.

8.0 EQUIPMENT AND REFERENCE MATERIALS

a) Calscience is furnished with all items of equipment (including reference materials) required for the correct performance of tests for which accreditation is maintained. Note that Calscience does not use equipment outside its permanent control.

b) All equipment is properly maintained, inspected, and cleaned. Maintenance procedures are documented.

c) Any equipment item that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown by verification or otherwise to be defective, is taken out of service, clearly identified and wherever possible stored at a specified place until it has been repaired and shown by calibration, verification or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous calibrations or tests.

d) When appropriate, each item of equipment, including reference materials, is labeled, marked, or otherwise identified to indicate its calibration status.

e) Records are maintained of each major item of equipment and all reference materials significant to the tests performed. These records include documentation on all routine and non-routine maintenance activities in assigned log books and reference material verifications.

The records include:

1) The name of the item of equipment;

2) The manufacturer's name, type identification, and serial number or other unique identification;

3) Date received and date placed in service (if available);

4) Current location, where appropriate;

5) If available, condition when received (e.g., new, used, reconditioned);

6) Copy of the manufacturer's instructions, where available;

7) Dates and results of calibrations and/or verifications and date of the next calibration and/or verification;

8) Details of maintenance carried out to date and planned for the future; and

9) History of any damage, malfunction, modification or repair.

9.0 MEASUREMENT TRACEABILITY AND CALIBRATION

9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before being put into service and on a continuing basis. The laboratory has an established program for the calibration and verification of its measuring and test equipment. This includes balances, thermometers and control standards.

9.2 Traceability of Calibration

a) The overall program of calibration and/or verification and validation of equipment is designed and operated so as to ensure that measurements made by the laboratory are traceable to national standards of measurement.

b) Calibration certificates indicate the traceability to national standards of measurement and provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory maintains records of all such certification in the QA office.

c) Where traceability to national standards of measurement is not applicable, the laboratory provides satisfactory evidence of correlation of results, for example, by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis.

9.3 Reference Standards

a) Reference standards of measurement held by the laboratory (such as Class S or equivalent weights, or traceable thermometers) are used for calibration only and for no other purpose, unless it can be demonstrated that their performance as reference standards has not been invalidated. A body that can provide traceability calibrates reference standards of measurement. Where possible, this traceability is to a national standard of measurement.

b) There is a program of calibration and verification for reference standards.

   i. Two weeks prior to their date of calibration expiration, individual thermometers are removed from service and replaced by newly calibrated units from the supplier.

   ii. Calscience keeps two sets of Class S weights on hand for use in the laboratory. One set is used for daily calibration checks, and the second set is kept for back up use should the first set be damaged, lost or otherwise compromised. The second set of weights is also place in service when the daily use set is shipped off site for recalibration.

   iii. Analytical balances are serviced and calibrated on a routine, annual schedule.

c) Where relevant, reference standards and measuring and testing equipment are subjected to in-service checks between calibrations and verifications. Reference materials are traceable. Where possible, traceability is to national or international standards of measurement, or to national or international standard reference materials.

d) NIST-Traceable Weights and Thermometers

   i. Reference standards of measurement shall be used for the purposes of calibration only. NIST-traceable thermometers and NIST-traceable weights shall not be used for routine testing. If NIST traceable reference sources are used for routine testing they shall not be used for calibration purposes unless it can be shown that their performance as reference standards would not be invalidated.

   ii. For NIST-traceable weights and thermometers, Calscience requires that all calibrations be conducted by a calibration laboratory accredited by A2LA or other recognized, ISO9001-compliant laboratory. The calibration certificate or report supplied by the calibration laboratory must contain a traceability statement, the conditions under which the calibrations were made, a compliance statement with an identified metrological specification and the pertinent clauses when applicable, and a clearly identified record of the quantities and functional test results before and after re-calibration. The certificate and scope of accreditation is kept on file at the laboratory.

   iii. If significant amendments are made to a calibration certificate, it must have its own unique report identifier and must reference the one it is replacing. The piece of equipment must be identified in the amended report using its unique serial number or other laboratory defined identifier. The amended report is maintained with the original calibration report.

   iv. Laboratory balances are recalibrated annually by an external, certified vendor. This service is documented on each balance with a signed and dated certification sticker. All mercury thermometers are calibrated annually against a NIST-traceable reference thermometer. Equipment that does not meet acceptance criteria is removed from service and repaired or replaced. Calibration reports are maintained by the Technical Manager or the QA Manager.

   v. Balance calibrations and temperature readings of ovens, refrigerators, and incubators are checked on each day of use. Min/Max thermometers are used for refrigerators and freezers to continually monitor temperature performance.

e) Traceable Reference Standards and Materials

i. Reference standards and materials are traceable to certified reference materials, where available. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP (National Voluntary Lab Accreditation Program) or other recognized vendor, and come with a Certificate of Analysis that documents the purity of the standard and expiration date, if assigned. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis against a known reference.

ii. Analytical reagents must be at a minimum the purity required by or stated in the test method. Commercial materials that are purchased for the preparation of calibration, verification or spiking solutions, are usually accompanied by an assay certificate or the purity is noted on the label. If the purity is ≥96%, the weight provided by the vendor may be used without correction. If the purity is <96%, a correction will be made to solution concentrations prepared from that material.

iii. The receipt of all reference standards and materials, including received date and expiration date, is documented by the laboratory at the time of receipt, in chemical receiving logbooks. All documentation received with the reference standard or material (Certificate of Analysis or Purity Certificates) is retained by the laboratory. To prevent contamination and/or deterioration in quality, all standards and materials are handled and stored according to the method or manufacturer"s requirements.

iv. Preparation of standard or reference materials are documented in Standard Preparation Logbooks maintained in each department. These records show the traceability to the purchased standards or materials, and include the method of preparation, date of preparation, expiration date, and preparer"s initials, at a minimum. Reference standards are assigned a unique identifier and are then labeled with the identifier and expiration date. Refer to Calscience SOP, T003, Standards and Reagents Login, Preparation, Storage and Disposal, for additional information.

v. All standards, reference, primary and working, whether purchased from a commercial vendor or prepared by the laboratory, must be checked regularly to ensure that the variability of the standard from the „true" value does not exceed method requirements. Calibration standards are checked by comparison with a standard from a second source, usually another manufacturer and vendor. In cases where a second manufacturer is not available, a different lot, with vendor certification, may be used as a second source.

vi. Quality control (QC) criteria for primary and second source standards are defined in laboratory SOPs. The Reagent and Chemicals SOP, T107, gives a general overview of the requirements with the determinative SOPs for each process further defining the QC acceptance criteria. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS/LCSD (where there is no sample preparation) is used as the second source verification of a primary calibration source.

9.4 Calibration

Calibration requirements are divided into two parts: (1) requirements for analytical support equipment, and (2) requirements for instrument calibration. In addition, the requirements for instrument calibration are divided into initial calibration and second source or initial calibration verification, and continuing calibration verification.

9.4.1 Support Equipment

These standards apply to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, thermometers, and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume.

a) All support equipment is maintained in proper working order. The records of all repair and maintenance activities, including service calls is kept.

b) All support equipment is calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration are within the specifications required of the application for which this equipment is used or:

   1) The item is removed from service until repaired; or

   2) The laboratory maintains records of established correction factors to correct all measurements.

c) Raw data records are retained to document equipment performance.

d) Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths are checked in the expected use range, with NIST traceable calibrated references. The acceptability for use or continued use is according to the needs of the analysis or application for which the equipment is being used.

e) Mechanical volumetric dispensing devices including burettes (except Class A glassware) are checked for accuracy on at least a quarterly use basis. Glass microliter syringes are to be considered Class A glassware, and come with a certificate from the manufacturer attesting to established accuracy or the accuracy is initially demonstrated and documented by the laboratory.

9.4.2 Instrument Calibration

This manual specifies the essential elements that define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data are of known quality and be appropriate for a given regulation or decision. This manual does not specify detailed procedural steps ("how to") for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory demonstrates that such requirements are met. If it is not apparent which standard is more stringent, then the requirements of the regulation or mandated test method are to be followed.

Note: In the following sections, initial instrument calibration is directly used for quantitation and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration.

9.4.2.1 Initial Instrument Calibrations

The following items are essential elements of initial instrument calibration:

a) The details of the initial instrument calibration procedures including calculations, integrations, acceptance criteria and associated statistics are included or referenced in the test method SOP. When initial instrument calibration procedures are referenced in the test method, the referenced material is retained by the laboratory and is available for review.

b) Sufficient raw data records are retained to permit reconstruction of the initial instrument calibration, e.g., calibration date, test method, instrument, analysis date, each analyte name, analyst"s initials or signature; concentration and response, calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to concentration.

c) Sample results are quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification unless specifically stated in a mandated test method.

d) All initial instrument calibrations is verified with a standard obtained from a second manufacturer or lot. Traceability shall be to a national standard, when available.

e) Criteria for the acceptance of an initial instrument calibration is established, e.g., correlation coefficient or relative percent difference. The criteria used is appropriate to the calibration technique employed.

f) Results of samples not bracketed by initial calibration standards (within calibration range) are reported as having less certainty, e.g., defined qualifiers or flags or explained in the case narrative. As determined by the method, the lowest calibration standard is at or above the method detection limit and at or below the reporting limit.

g) If the initial instrument calibration results are outside established acceptance criteria, corrective actions are performed. Data associated with an unacceptable initial instrument calibration is not reported.

h) Calibration standards include concentrations at or below the regulatory limit/decision level, if the laboratory knows these limits/levels, unless these concentrations are below the laboratory"s demonstrated detection limits (See Calscience QSM Section Appendix D.1.5 Detection Limits).

i) If a reference or mandated method does not specify the number of calibration standards, the minimum number is two, not including blanks or a zero standard. The laboratory"s standard operating procedure defines the number of points for establishing the initial instrument calibration.

9.4.2.2 Continuing Instrument Calibration Verification

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

a) The details of the continuing calibration procedure, calculations and associated statistics must be included or referenced in the test method SOP.

b) A continuing calibration verification standard must be analyzed at the beginning and end of each analytical batch, and where required by method or project, at a specific frequency, every 10 or 20 samples or 12 hours, within the batch. The concentrations of the calibration verification shall be varied within the established calibration range. If an internal standard is used, only one continuing calibration verification standard must be analyzed, prior to sample or QC analysis, per analytical batch.

c) Sufficient raw data records must be retained to permit reconstruction of the continuing calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations. Continuing calibration verification records must explicitly connect the continuing calibration verification data to the initial calibration.

d) Criteria for the acceptance of a continuing calibration verification must be established, e.g., relative percent difference.

e) If the continuing calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second (consecutive and immediate) calibration verification within acceptance criteria, then the laboratory shall demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new instrument calibration must be performed. If the laboratory has not demonstrated acceptable performance, sample analyses shall not occur until a new initial calibration curve is established and verified.

As an exception, sample data associated with an unacceptable continuing calibration verification may be reported as qualified data under the following special conditions:

i. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification are reanalyzed after a new calibration curve has been established, evaluated and accepted.

ii. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification are reanalyzed after a new calibration curve has been established, evaluated and accepted.

10.0 TEST METHODS AND STANDARD OPERATING PROCEDURES

10.1 Methods Documentation

a) The laboratory has documented instructions on the use and operation of all relevant equipment, on the handling and preparation of samples and for calibration and/or testing, where the absence of such instructions could jeopardize the calibrations or tests.

b) All instructions, standards, manuals, and reference data relevant to the work of the laboratory are maintained up-to-date and be readily available to the staff.

10.1.1 Standard Operating Procedures (SOPs) Administrative

Calscience maintains standard operating procedures that accurately reflect all phases of current laboratory activities such as instrument operation, assessing data integrity, corrective actions, handling customer complaints, reporting of test results, etc.

a) These documents, for example, may be equipment manuals provided by the manufacturer or internally written documents.

b) The test methods may be copies of published methods as long as any changes or selected options in the methods are documented and included in the SOP (See 10.1.2.)

c) Copies of all SOPs are accessible to all personnel.

d) The SOPs are organized.

e) Each SOP clearly indicates the effective date of the document, the revision number and the signatures of the approving authorities.

10.1.2 Standard Operating Procedures (SOPs) Analytical

a) The laboratory has and maintains SOPs for each accredited analyte or test method.

b) This SOP may consist of copies of published or referenced test methods or standard operating procedures that have been written by the laboratory. In cases where modifications to the published method have been made by the laboratory or where the referenced test method is ambiguous or provides insufficient detail, these changes or clarifications are clearly described. Each test method includes or references where applicable:

1) Identification of the test method;
2) Applicable matrix or matrices;
3) Detection limit;
4) Scope and application, including components to be analyzed;
5) Summary of the test method;
6) Definitions;
7) Interferences;
8) Safety;
9) Equipment and supplies;
10) Reagents and standards;
11) Sample collection, preservation, shipment, and storage;
12) Quality control;
13) Calibration and standardization;
14) Procedure;
15) Calculations;
16) Method performance;
17) Pollution prevention;
18) Data assessment and acceptance criteria for quality control measures;
19) Corrective actions for out-of-control data;
20) Contingencies for handling out-of-control or unacceptable data;
21) Waste management;
22) References; and
23) Any tables, diagrams, flowcharts, and validation data.

Laboratory procedures other than preparative or analytical procedure may use a shortened format as outlined in SOP T001.

10.2 Exceptionally Permitting Departures from Documented Policies / Procedures

a) If it is necessary to depart from a documented procedure or policy due to circumstances outside of Calscience"s control or due to conditions encountered while preparing or analyzing a sample, the following will be documented.

   1) The nature of the exception
   2) How the data or procedure may be impacted
   3) Any Corrective Action that may be needed.
   4) Any approval from a client that may be required.
   5) Approval by management to report or proceed with the exception.
   6) A Case Narrative with the Final Report explaining the exception.

10.3 Test Methods

The laboratory uses appropriate test methods and procedures for all tests and related activities within its responsibility (including, as applicable, sample collection, sample handling, transport and storage, sample preparation and sample analysis). The method and procedures shall be consistent with the accuracy required, and with any standard specifications relevant to the calibrations or tests concerned.

a) When the use of specific test methods for a sample analysis is mandated or requested, only those methods are used.

b) Where test methods are employed that are not required, as in the Performance Based Measurement System approach, the methods are fully documented and validated (see Calscience QSM Section 10.1.2 and Appendix C), and are available to the client and other recipients of the relevant reports.

10.4 Test Method Assessment

The laboratory will periodically conduct a Test Method Assessment (TMA) on the analytical methods in use. These TMAs will be conducted under the guidance of SOP T029. The purpose is to evaluate the compliance between bench performances of the method versus the current CEL Standard Operating Procedure versus the promulgated or published method. Discrepancies will need to be addressed and resolved. Note that some methods are totally prescriptive while others may contain prescriptive aspects, and still others are performance based. In many cases, modifications to the published method may be required due to circumstances outside the laboratories" control.

10.5 Demonstration of Capability

a) Prior to acceptance and institution of any test method, satisfactory demonstration of method capability is required. (See Calscience QSM Section Appendix C and 6.2.b.) This demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (sample of a matrix is which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., water, solids and air. In addition, for analytes that do not lend themselves to spiking, the demonstration of capability may be performed using quality control samples.

b) Continuing demonstration of method performance, as per the quality control requirements in Appendix D (such as laboratory control samples) is required.

c) In cases where Calscience analyzes samples using a test method that has been in use by the laboratory before July 1999, and there have been no significant changes in instrument type, personnel or test method, the continuing demonstration of method performance and the analyst"s documentation of continued proficiency shall be acceptable. The laboratory shall have records on file to demonstrate that an initial demonstration of capability is not required.

d) In all cases, the appropriate forms, such as the Certification Statement (Appendix C), is completed and retained by the laboratory to be made available upon request. The laboratory retains all associated supporting data necessary to reproduce the analytical results summarized in the Certification Statement. (See Appendix C for an example of a Certification Statement.)

e) Demonstration of capability is completed each time there is a significant change in instrument type, personnel, or test method.

f) In departments with specialized "work cell(s)" (a group consisting of analysts with specifically defined tasks that together perform the test method), the group as a unit must meet the above criteria and this demonstration of capability is fully documented.

g) When a work cell is employed, and the members of the cell change, the new employee(s) must work with an experienced analyst in that area of the work cell where they are employed. This new work cell must demonstrate acceptable performance through acceptable continuing performance checks (appropriate sections of Appendix D, such as laboratory control samples). Such performance is documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. In addition, if the entire work cell is changed or replaced, the new work cell must perform the demonstration of capability (Appendix C).

h) Performance of the work cell is linked to the training records of the individual members of the work cell (See Calscience QSM Section 6.2).

10.6 Sample Aliquots

Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use documented procedures and appropriate techniques to obtain representative subsamples. Reference SOP M230, Homogenization and Compositing of Solid, Soil and Sediment Samples for further guidance.

10.7 Data Verification

Calculations and data transfers are subject to appropriate checks.

a) The laboratory has Standard Operating Procedures that ensure that the reported data are free from transcription and calculation errors.

b) The laboratory has Standard Operating Procedures that ensure that all quality control measures are reviewed, and evaluated before data are reported. Refer to SOPs T020, internal Quality Control Checks and T062, Project Management and Analytical Report Review

c) The laboratory has Standard Operating Procedures that address manual calculations including manual integrations. Refer to SOPs T065, Data Integrity and T023, Peak Integration Procedures.

10.8 Documentation and Labeling of Standards and Reagents

Documented procedures exist for the purchase, receipt and storage of consumable materials used for the technical operations of the laboratory.

a) The laboratory retains records for all standards, reagents and media including the manufacturer/vendor, the manufacturer"s Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material is not used, unless the laboratory verifies its suitability for testing use.

b) Original containers (such as those provided by the manufacturer or vendor) are labeled with an expiration date.

c) Records are maintained on reagent and standard preparation. These records indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.

d) All containers of prepared reagents and standards bear a unique identifier and expiration date and are linked to the documentation requirements in Calscience QSM Section 10.8.c above.

10.9 Computers and Electronic Data Related Requirements

Where computers, automated equipment, or microprocessors are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data, Calscience ensures that:

a) All requirements of the NELAC Standard (i.e., Chapter 5 of NELAC) are met;

b) Computer software is tested and documented to be adequate for use, e.g., internal audits, personnel training, focus point of QA and QC;

c) Procedures are established and implemented for protecting the integrity of data. Such procedures include, but are not limited to, integrity of data entry or capture, data storage, data transmission and data processing;

d) Computer and automated equipment are maintained to ensure proper functioning and provided with the environmental and operating conditions necessary to maintain the integrity of calibration and test data; and,

e) It establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.

11.0 SAMPLE HANDLING, SAMPLE ACCEPTANCE POLICY AND SAMPLE RECEIPT

While Calscience does not have control of field sampling activities, the following are essential to ensure the validity of the laboratory"s data.

11.1 Sample Tracking

a) The laboratory has a documented system for uniquely identifying the items to be tested, to ensure that there can be no confusion regarding the identity of such items at any time. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates. The laboratory assigns a unique identification (ID) code to each sample container received in the laboratory. (The use of container shape, size, or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.)

b) This laboratory code is maintained as an unequivocal link with the unique field ID code assigned each container.

c) The laboratory ID code is placed on the sample container as a durable label.

d) The laboratory ID code is entered into the laboratory records (see Calscience QSM Section 11.3.d) and is the link that associates the sample with related laboratory activities such as sample preparation or calibration.

e) In cases where the sample collector and analyst are the same individual or the laboratory pre-assigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

11.2 Sample Acceptance Policy

The laboratory has a written sample acceptance policy that clearly outlines the circumstances under which samples are accepted or rejected. Data from any samples that do not meet the following criteria are flagged in an unambiguous manner, and the nature of the variation is clearly defined. The sample acceptance policy is available to sample collection personnel and includes, but is not limited to, the following areas of concern:

a) Proper, full, and complete documentation, that includes sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;

b) Proper sample labeling that includes a unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

c) Use of appropriate sample containers;

d) Adherence to specified holding times;

e) Adequate sample volume. Sufficient sample volume must be available to perform the necessary tests; and,

f) Procedures to be used when samples show signs of damage, contamination or inadequate preservation.

g) Samples are NOT accepted if classified as extremely hazardous, reference section 5.2 k for examples.

11.3 Sample Receipt Protocols

a) Upon receipt, the condition of the sample, including any abnormalities or departures from standard condition as prescribed in the relevant test method, is recorded. All items specified in Calscience QSM Section 11.2 above are checked.

   1) All samples that require cold temperature preservation are considered acceptable if the arrival temperature is within 2°C of the required temperature or the method-specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

   2) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or free chlorine, prior to or during sample preparation or analysis.

      With the exception of residual chlorine measurements in aquatic toxicity samples, certain measurements, such a pH, are performed and recorded just prior to analysis.

b) The results of all checks are recorded on Sample Receipt and, as needed, Sample Anomaly forms.

c) When there is any doubt as to the item's suitability for testing, when the sample does not conform to the description provided, and when the test required is not fully specified, the laboratory makes every attempt to consult the client for further instruction before proceeding. The laboratory establishes whether the sample has received all necessary preparation, or whether sample preparation has yet to be performed. If the sample does not meet the sample receipt acceptance criteria listed in this standard, the laboratory:

   1) Retains correspondence and/or records of conversations concerning the final disposition of rejected samples; or

   2) Fully documents any decision to commence with the analysis of samples not meeting acceptance criteria.

      i. The condition of these samples is, at a minimum, noted on the chain of custody record or transmittal form, and laboratory receipt documents.

      ii. The analysis data is/are appropriately "qualified" on the final report.

d) The laboratory utilizes a permanent chronological record such as a logbook or electronic database to document receipt of all sample containers.

   1) This sample receipt log records the following:

      i. Client/Project Name;

      ii. Date and time of laboratory receipt;

      iii. Unique laboratory ID code (see Calscience QSM Section 11.1); and

      iv. Signature or initials of the person making the entries.

2) During the login process, the following information is linked to the log record or included as a part of the log. If such information is recorded/documented elsewhere, that document becomes part of the laboratory's permanent records, easily retrievable upon request, and readily available to individuals who will process the sample. Note: The placement of the laboratory ID number on the sample container is not considered a permanent record.

   i. The field ID code that identifies each container is linked to the laboratory ID code in the sample receipt log.

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

   iii. The requested analyses (including applicable approved test method numbers) are linked to the laboratory ID code.

   iv. Any comments resulting from inspection for sample rejection are linked to the laboratory ID code.

e) All documentation (i.e., memos or transmittal forms) that are conveyed to the laboratory by the sample submitter is retained.

f) A complete chain of custody record form is maintained.

11.4 Storage Conditions

The laboratory has documented procedures and appropriate facilities to avoid deterioration, contamination, and damage to the sample during storage, handling, preparation, and testing; any relevant instructions provided with the item are followed. Where items must be stored or conditioned under specific environmental conditions, these conditions are maintained, monitored, and recorded.

a) Samples are stored according to the conditions specified by preservation protocols:

   1) Samples that require thermal preservation are stored under refrigeration at +/-2° of the specified preservation temperature unless method-specified criteria exist. For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C is acceptable.

   2) Samples are stored away from all standards, reagents, food, and other potentially contaminating sources. Samples are stored in such a manner to prevent cross contamination.

b) Sample fractions, extracts, leachates, and other sample preparation products are stored according to Calscience QSM Section 11.4.a above or according to specifications in the test method.

c) When a sample or portion of a sample needs to be held secure (for example, for reasons of record, safety or value, or to enable check calibrations or tests to be performed later), the laboratory has storage and security arrangements that protect the condition and integrity of the secured items or portions concerned.

11.5 Sample Disposal

The laboratory has standard operating procedures for the disposal of samples, digestates, leachates and extracts or other sample preparation products. Refer to SOP T005, Disposal of Laboratory Samples and Wastes.

12.0 RECORDS

The laboratory maintains a record system to suit its particular circumstances and comply with any applicable regulations. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the test report for a minimum of five years.

There are two levels of sample handling: 1) sample tracking and 2) legal chain of custody protocols that are used for evidentiary or legal purposes. All essential requirements for sample tracking (e.g., chain of custody form) are outlined in Calscience QSM Sections 12.1, 12.2 and 12.3. Calscience details the Legal/Evidentiary and Internal Chain of Custody procedures in SOP T100, Sample Receipt and Log-In Procedures.

12.1 Record Keeping System and Design

The Calscience record keeping system allows historical reconstruction of all laboratory activities that produced the analytical data. The history of the sample is readily understood through the documentation. This includes inter-laboratory transfers of samples and/or extracts.

a) The records include the identity of personnel involved in sampling, sample receipt, preparation, calibration or testing.

b) All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification, are documented.

c) The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes, e.g., set format for naming electronic files.

d) All changes to records are signed or initialed by responsible staff. The reason for the signature or initials is clearly indicated in the records such as “sampled by,” “prepared by,” or “reviewed by.”

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

f) Entries in records are not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record-keeping errors are made by one line marked through the error. The individual making the correction signs (or initials) and dates the correction. These criteria also apply to electronically maintained records.

g) Refer to 10.9 for Computer and Electronic Data.

12.2 Records Management and Storage

a) All records (including those pertaining to calibration and test equipment), certificates and reports are safely stored, and held secure and in confidence to the client. NELAP-related records are available to the accrediting authority.

b) All records, including those specified in Calscience QSM Section 12.3, are retained for a minimum of five years from generation of the last entry in the records. The laboratory maintains all information necessary for the historical reconstruction of data. Records stored only on electronic media are supported by the hardware and software necessary for their retrieval.

c) Records that are stored or generated by computers or personal computers have hard copy or write-protected backup copies.

d) The laboratory has an established record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation storage and reporting.

e) Access to archived information is documented with an access log. These records are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.

f) The laboratory has a plan to ensure that the records are maintained or transferred according to the clients" instructions (see 4.1.8.e of NELAC) in the event of Laboratory Transfer of Ownership, Going out of Business or Bankruptcy. In all cases, appropriate regulatory and state legal requirements concerning laboratory records will be followed. Reference QSM Section 6.2.1 and SOP T-002, Document Control, for detailed policies and procedures for handling of client records and data in these situations.

12.3 Laboratory Sample Tracking

12.3.1 Sample Handling

A record of all procedures to which a sample is subjected while in Calscience"s possession is maintained. These include but are not limited to all records pertaining to:

a) Sample preservation, including appropriateness of sample container and compliance with holding time requirement;

b) Sample identification, receipt, acceptance or rejection, and log-in;

c) Sample storage and tracking, including shipping receipts, sample transmittal forms (chain of custody form); and

d) Documentation procedures for the receipt and retention of test items, including all provisions necessary to protect the integrity of samples.

12.3.2 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following is retained:

a) All original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);

b) A written description or reference to the specific test method used, which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;

c) Copies of final reports;

d) Archived standard operating procedures;

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

f) All corrective/preventive action reports, audits and audit responses;

g) Proficiency test results and raw data; and,

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

12.3.3 Analytical Records

The essential information associated with analyses, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

a) Laboratory sample ID code;

b) Date of analysis and time of analysis if the method-specified holding time is 72 hours or less, or when time critical steps are included in the analysis, e.g., extractions, and incubations;

c) Instrument identification and instrument operating conditions/parameters (or reference to such data);

d) Analysis type;

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

f) Analyst's or operator's initials/signature or chemist ID number;

g) Sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

h) Sample analysis;

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

j) Calibration criteria, frequency and acceptance criteria;

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

l) Quality control protocols and assessment;

m) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and,

n) Method performance criteria including expected quality control requirements.

12.3.4 Administrative Records

The following are maintained:

a) Personnel qualifications, experience and training records;

b) Ethics Statements;

c) Records of demonstration of capability for each analyst; and

d) A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

13.0 LABORATORY REPORT FORMAT AND CONTENTS

The results of each test, or series of tests carried out by the laboratory must be reported accurately, clearly, unambiguously and objectively. The results normally reported in a test report and include all the information necessary for the interpretation of the test results and all information required by the method used. Some regulatory reporting requirements or formats, such as monthly operating reports may not require all items listed below, however, Calscience will provide all the required information to their client for use in preparing such regulatory reports.

a) Except as discussed in 13.b, each report to an outside client includes at least the following information (those prefaced with “where relevant” are not mandatory):

   1) A title, e.g., "Analytical Report," or "Test Certificate," "Certificate of Results" or "Laboratory Results”;

   2) Name and address of laboratory, and location where the test was carried out if different from the address of the laboratory and phone number with name of contact person for questions;

   3) Unique identification of the certificate or report (such as serial number) and of each page, and the total number of pages;

      This requirement may be presented in several ways:

      i. The total number of pages may be listed on the first page of the report as long as the subsequent pages are identified by the unique report identification and consecutive numbers, or

      ii. Each page is identified with the unique report identification, the pages are identified as a number of the total report pages (example: 3 of 10, or 1 of 20).

      Other methods of identifying the pages in the report may be acceptable as long as it is clear to the reader that discrete pages are associated with a specific report, and that the report contains a specified number of pages.

   4) Name and address of client, where appropriate and project name if applicable;

   5) Description and unambiguous identification of the tested sample including the client identification code;

   6) Identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature;

   7) Date of receipt of sample, date and time of sample collection, date(s) of performance test, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 72 hours;

   8) Identification of the test method used, or unambiguous description of any nonstandard method used;

   9) If the laboratory collected the sample, reference to sampling procedure;

   10) Any deviations from (such as failed quality control), additions to or exclusions from the test method (such as environmental conditions), and any nonstandard conditions that may have affected the quality of results, and including the use and definitions of data qualifiers.

11) Measurements, examinations and derived results, supported by tables, graphs, sketches, and photographs as appropriate, and any failures identified; identify whether data are calculated on a dry weight or wet weight basis; identify the reporting units such as µg/l or mg/kg;

12) When required, a statement of the estimated uncertainty of the test results;

13) A signature and title, or an equivalent electronic identification of the person(s) accepting responsibility for the content of the certificate or report (however produced), and date of issue;

14) At the Calscience"s discretion, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory;

15) At the Calscience"s discretion, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory;

16) Clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc.; and

17) Clear identification of numerical results with values outside of quantitation limits.

b) Where the certificate or report contains results of tests performed by subcontractors, these results are clearly identified by subcontractor name or applicable accreditation number and the entirety of the subcontract report is included with the final CEL report.

c) After issuance of the report, the laboratory report remains unchanged. Material amendments to a calibration certificate, test report or test certificate after issue may be made only in the form of a further document, or data transfer, including the statement "Supplement to Test Report or Test Certificate, serial number . . . [or as otherwise identified]", or equivalent form of wording. Such amendments meet all the relevant requirements of the NELAC Standard.

d) Calscience notifies clients promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any calibration certificate, test report or test certificate or amendment to a report or certificate.

e) The laboratory will, where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, follow documented procedures that ensure that the requirements of this Standard are met and that confidentiality is preserved.

f) Calscience will certify that all its NELAC-certified test results reported meet all requirements of NELAC or provide reasons and/or justification if they do not.

14.0 SUBCONTRACTING ANALYTICAL SAMPLES

When CEL subcontracts work whether because of unforeseen circumstances (e.g. workload, need for further expertise or temporary incapacity) or on a continuing basis (e.g. through client direction, contractual arrangement or permanent subcontracting), this work shall be placed with a laboratory accredited under NELAP, or other appropriate certification, for the tests to be performed or with a laboratory that meets applicable statutory and requirements for performing the tests and submitting the results of tests performed. All subcontracted work shall be referenced and so noted in the final CEL analytical report.

Subcontract laboratories will provide or make available, current copies of the following documents prior to CEL submitting samples. This information will be updated annually or on an as needed basis.

a) Laboratory accreditations / certifications

b) Upon request, any Proficiency Testing (PT) or Performance Evaluation (PE) results relevant to the subcontracted samples.

c) Insurance Certificates

d) Quality Assurance Manual

e) Subcontract laboratories will also submit statements affirming that CEL will be notified if any of the following occur.

- There is a change or loss in accreditation for the applicable analysis.
- Most recent PT or PE study results for the applicable analysis are unacceptable *AND* are not able to be addressed via Corrective Action.
- There is a need to subcontract CEL project samples. Prior CEL approval is required in writing for subcontracting samples.

f) The client project requirements will be used to evaluate the subcontract laboratories and to determine their acceptability. Approval by either: the QA Manager, Laboratory Director or Client Services Director (or designee) is required.

g) A master list of approved laboratories will be created and distributed to Sample Control and all Project Managers. All subcontracting must utilize a laboratory from this list.

The procedure for subcontracting samples will follow these guidelines:

a) CEL will advise its client via written, facsimile or e-mail notification of its intention to subcontract any portion of the testing to another party in cases when unforeseen circumstances occur. CEL shall gain approval by the client in writing, facsimile or via e-mail response.

b) CEL may subcontract samples on a continuing basis without written, facsimile or e-mail notification under the following (but not limited to) cases:

- Standing Client direction or instruction
- Contractual specification or requirement
- Project historical precedent

c) A separate Chain of Custody will be created specifically for the subcontracted sample(s). This (or a copy) will be included with the full and complete subcontract report in the final CEL analytical report.

d) CEL shall retain records demonstrating that the above requirements have been met.

e) If the samples to be subcontracted are submitted to CEL under special regulatory, agency or governmental accreditation, Example: Department of Defense, that have more comprehensive or differing quality criteria, Example: DoD QSM for Environmental Laboratories Version 4.2 October 2010, then the subcontract laboratory MUST have certification for the subcontracted analysis from the same entity and MUST have undergone similar assessment as the primary laboratory for the subcontracted component. Written authorization from the client or authorizing body must be obtained prior to usage of each subcontract laboratory.

15.0 OUTSIDE SUPPORT SERVICES AND SUPPLIES

Calscience does not procure outside services and supplies, other than those referred to in this Manual.

16.0 INQUIRIES AND COMPLAINTS

Calscience SOP-T018 addresses the policies and procedures for the resolution of inquiries and complaints received from clients or other parties about the laboratory's activities. Where an inquiry or complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the requirements of this manual or otherwise concerning the quality of the laboratory's calibrations or tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited in accordance with NELAC Section 5.3.1. Records of the complaint and subsequent actions are maintained and are available for audits.

17.0 REVIEW OF WORK REQUESTS, CONTRACTS AND TENDERS

Calscience Environmental Laboratories (CEL) has established procedures for the review of work requests contracts and tenders. Projects, proposals and contracts are reviewed for adequately defined requirements and the ability of CEL to meet those requirements. A thorough review of all technical and quality control requirements contained in these requests is performed to ensure a project"s success. The appropriateness of requested methods, and the lab"s capability to perform them must be established. A review of the laboratory"s capability to analyze non-routine analytes is also part of this review process. Additionally, alternate test methods that are capable of meeting the clients" requirements may be proposed by the lab.

All projects, proposals and contracts are reviewed for the client"s requirements in terms of compound lists, test methodology requested, detection and reporting levels, and quality control limits. During the review process, the laboratory determines whether it has the necessary physical, personnel and information resources to meet the project requirements, and if the personnel have the expertise needed to perform the required testing. Each proposal is also checked for its impact on the overall capacity of the laboratory. The proposed turnaround time will be checked for feasibility. Electronic or hard copy deliverable requirements are evaluated against the laboratory"s ability to produce such documentation.

This review process ensures that the laboratory"s test methods are suitable to achieve regulatory and/or client requirements and that the laboratory holds the appropriate certifications to perform the work. In the event that the use of a subcontract laboratory is needed, also confirming that they meet all project requirements and maintain the appropriate certifications for the proposed subcontract analyses. If the laboratory cannot provide all services and therefore intends to use the services of a subcontract laboratory, this will be documented and discussed with the client prior to project or contract approval.

Following the review process, the laboratory informs the client of the results of the review and notes any potential conflict, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client"s requirements and the capability of the laboratory to meet those requirements is resolved in writing before acceptance of the project or contract. It is necessary that the project requirements or contract be acceptable to both the client and the laboratory prior to the start of the work. The review process is repeated when there are amendments to the original contract by the client.

All contracts, Quality Assurance Project Plans (QAPPs), Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

Review Personnel

Depending upon the scope of a project or contract, one or more key persons may review and accept work on behalf of the laboratory. For routine projects, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has the necessary certifications, that it can meet the clients" data quality, reporting and turn-around time requirements.

For new, complex or large projects, the proposed project proposal or contract is given to the Business Development Director for an initial review that encompasses all facets of the operation. The scope of work is then distributed to the following personnel, as needed based on scope of contract, to evaluate all of the project related requirements:

- Laboratory Director
- Operations Manager
- Technical Services Manager
- Special Projects Manager
- Quality Assurance Manager
- Group Leaders
- Project Manager(s)

Appropriate records are maintained for every contract or work request. Copies of the agreed-upon contract will be distributed to key personnel as needed and the signed copies maintained by the Business Development Director and/or Laboratory Director.

Project Kick-off and Status Meetings

For routine project work, project managers ensure that specific technical and QC requirements are effectively evaluated and communicated to laboratory personnel through the use of the LIMS system: special requirements section of the chemist"s worksheet.

Prior to work on a new or complex project, project managers or key personnel will hold meetings with operations personnel to discuss schedules and any unique aspects of the project. Items discussed include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, and any other special requirements.

Project requirements are given to the laboratory staff during project kick-off meetings or the daily status meetings. Information disseminated during these meetings provides direction to the laboratory staff in order to maximize production, maintain high quality and ensure client satisfaction.

During the project, changes to the scope of work may occur due to client, sampling or regulatory reasons. If these changes impact the laboratory"s role in the project (use of a non-standard method or modification of a method to comply with revised requirements) then the changes need to be discussed with and agreed upon with the client prior to continuing with the work. These changes must be documented prior to implementation and communicated to the laboratory staff during a status or project specific meeting. Documentation of the modification is made in the analytical report narrative.

And at all times, records of all pertinent discussions with a client relating to the project or contract are documented and maintained as a part of the project record.

18.0 MANAGEMENT REVIEW, MANAGEMENT OF CHANGE AND CONTINUOUS IMPROVEMENT

18.1 Management Review

A comprehensive Management Review of the entire CEL Quality System will be conducted by the Laboratory Director on an annual basis. The SOP T-030 may be consulted for detailed guidance. All major stakeholders will be given an opportunity to provide comment or input for the review. These will include:

- Laboratory Director
- Business Development Director
- Operations Manager
- Technical Manager
- Senior Client Manager
- Other Operational / Project Management personnel as appropriate.
- Clients

The purpose and goal of the Management Review will identify weaknesses, areas requiring more resources or oversight, opportunities for continuous improvement and follow up on previous recommendations.

The final completed review is part of the NELAP laboratory documentation requirements and may be submitted to CEL authorized auditing agencies or clients upon request.

18.2 Management of Change

Whenever a change is made in a controlled environment (not just production) the laboratory is put at risk. However, one needs to constantly make changes to keep pace with business / regulatory requirements. The challenge to the laboratory is to minimize the risk and impact of that change.

An organization must have an operating process in place for which an evaluation has been conducted, and that allows proper lead times and approvals to ensure that the laboratory is unaffected when changes are made. But to successfully implement a change, one also needs to have a comprehensive understanding of the infrastructure that supports the services to determine the overall impact. The Management of Change process will facilitate, as referenced in SOP T030, this evaluation.

The Management of Change process will track and implement the following types of changes:

a) Permanent Change: – A change that is considered long term and durable. Any change which is not categorized as a Temporary Change.

b) Temporary Change: – A change which has a defined lifetime and which will be removed before a defined date (usually no more than six months). All temporary changes must have a specified removal date that is documented on the approved MOC form.

c) Emergency Change: – An emergency change path that allows the change to be implemented and commissioned immediately in order to address an immediate safety, operational, health, environmental, or product quality situations.

The functional categories that will be managed include:

a) Laboratory Facility Acquisition
b) Laboratory Instrument Acquisition
c) Analytical Method Development and Validation

d) Laboratory Operations Process Change
e) Department Relocation
f) Activation of Analytical Method
g) Waste Stabilization and Disposition
h) Human Resources
i) Information Technology
j) Safety and Security

18.3 Continuous Improvement

In order for CEL to be proactive and a leader in the industry, the entire CEL Quality system is designed to ensure the production of scientifically sound, legally defensible data of known and proven quality. The addition of the Management Review and Management of Change processes enhances CEL"s ability to foster continuous improvement.

Continuous improvement is an ongoing effort to improve data integrity, services or processes. These efforts can seek "incremental" improvement over time or "breakthrough" improvement all at once. All staff at CEL participates in continuous improvement, from the Laboratory Director down to the beginning technician, as well as external stakeholders when applicable.

The following procedures / inputs have direct involvement in the continuous improvement process:

a) External Audits (Regulatory and Client Based)
b) Internal Audits
c) Corrective / Preventive Actions
d) Statistical Quality Control (SQC) Monitoring
e) Proficiency Testing Performance
f) Client Feedback – Complaints and Commendations
g) Management Review
h) Management of Change

The Management of Change process will guide and document the major improvements. The Corrective / Preventive Action procedure will enable and record the more incremental changes.

The principal elements are commitment to quality, focused effort, involvement of all employees, willingness to change, and communication.

NELAC APPENDICES

APPENDIX A - REFERENCES

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APPENDIX B - GLOSSARY

The following definitions are used in the text of Quality Systems. In writing this document, the following hierarchy of definition references was used: ISO 8402, ANSI/ASQC E-4, EPA"s Quality Assurance Division Glossary of Terms, and finally definitions developed by NELAC. The source of each definition, unless otherwise identified, is the Quality Systems Committee.

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority: The Territorial, State, or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation. (NELAC) [1.5.2.3]

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analysis Duplicate: The second measurement of the target analyte(s) performed on a single sample or sample preparation.

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Analytical Reagent (AR) Grade: Designation for the high purity of certain chemical reagents and solvents given by the American Chemical Society. (Quality Systems)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

Audit: A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch: Environmental samples, which are prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample: A sub-sample for analysis with a composition known to the submitter. The analyst/ laboratory may know the identity of the sample but not its composition. It is used to test the analyst"s or laboratory"s proficiency in the execution of the measurement process. (NELAC)

Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method: A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard: A substance or reference material used to calibrate an instrument. (QAMS)

Certified Reference Material (CRM): A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)

Chain of Custody Form: A record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. (NELAC)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions compromised samples are not analyzed. If emergency situations require analysis, the results must be appropriately qualified. (NELAC)

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

- Second column confirmation;
- Alternate wavelength;
- Derivatization;
- Mass spectral interpretation;
- Alternative detectors; or
- Additional cleanup procedures. (NELAC)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ ASQC E4-1994)

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)

Desorption Efficiency: The mass of target analyte recovered from sampling media, usually a sorbent tube, divided by the mass of target analyte spiked on to the sampling media expressed as a percentage. Sample target analyte masses are usually adjusted for the desorption efficiency. (NELAC)

Detection Limit: The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA- QAD)

Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR Part 136)

Inspection: An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ ASQC E4-1994)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method. (NELAC)

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Laboratory: A body that calibrates and/or tests. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC)

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank. See also Method Detection Limit, Detection Limit, and Quantitation Limit. (Analytical Chemistry, 55, p. 2217, December 1983, modified)

Manager (however named): The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

- Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

- Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- Non-aqueous Liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.
- Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.
- Air: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)

May: Denotes permitted action, but not required action. (NELAC)

Media: Material that supports the growth of a microbiological culture.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B)

Must: Denotes a requirement that must be met. (Random House College Dictionary)

National Accreditation Database: The publicly accessible database listing the accreditation status of all laboratories participating in NELAP. (NELAC)

National Environmental Laboratory Accreditation Conference (NELAC): A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP): The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Objective Evidence: Any documented statement of fact, other information, or record, either quantitative or qualitative, pertaining to the quality of an item or activity, based on observations, measures, or tests that can be verified. (ASQC)

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS): A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing: A means of evaluating a laboratory"s performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, and analysis) which must be strictly followed. (EPA- QAD)

Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method. (NELAC)

Quality Assurance: An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance (Project) Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

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. (QAMS)

Quality Control Sample: An uncontaminated sample matrix with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ ASQC E-41994)

Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence. (NELAC)

Range: The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)

Reference Material: A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30- 2.1)

Reference Method: A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)

Reference Toxicant: The toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory"s ability to perform the test correctly and obtain consistent results (see Chapter 5, Appendix D, Section 2.1.f). (NELAC)

Replicate Analyses: The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement: Denotes a mandatory specification; often designated by the term "shall". (NELAC)

Sampling Media: Material used to collect and concentrate the target analytes(s) during air sampling such as solid sorbents, filters, or impinger solutions.

Selectivity: (Analytical chemistry) The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Spike: A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM): A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named): The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director: Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method: An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Testing Laboratory: Laboratory that performs tests. (ISO/ IEC Guide 2 - 12.4)

Test Sensitivity/Power: The minimum significant difference (MSD) between the control and test concentration that is statistically significant. It is dependent on the number of replicates per concentration, the selected significance level, and the type of statistical analysis (see Chapter 5, Appendix D, Section 2.4.a). (NELAC)

Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM - 6.12)

Validation: The process of substantiating specified performance criteria. (EPA- QAD)

Verification: Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

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APPENDIX C - DEMONSTRATION OF CAPABILITY

C.1 PROCEDURE FOR DEMONSTRATION OF CAPABILITY

A demonstration of capability (DOC) must be made prior to using any test method, and at any time there is a change in instrument type, personnel or test method. (See NELAC 10.2.1.)

Note: Where tests are performed by specialized "work cells" (a well-defined group of analysts that together perform the method analysis), the work cell as a unit meets the above criteria and this demonstration is fully documented.

In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., water, solids and air. However, before any results are reported using this method, actual sample spike results may be used to meet this standard, i.e., at least four consecutive matrix spikes within the last twelve months. In addition, for analytes that do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples.

All demonstrations shall be documented through the use of the form in this appendix.

The following steps, which are adapted from the EPA test methods published in 40 CFR Part 136, Appendix A, are performed if required by mandatory test method or regulation. Note: For analytes for which spiking is not an option and for which quality control samples are not readily available, the 40 CFR approach is one way to perform this demonstration. The laboratory documents that other approaches to DOC are adequate, and this is documented in the laboratory"s Quality Manual.

a) A quality control sample is obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The analyte(s) is diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.

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

d) Using all of the results, the mean recovery ($\overline{X}$) is calculated in the appropriate reporting units (such as µg/L) and the standard deviations of the population sample (n-1) (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory will assess performance against established and documented criteria.

e) Compare the information from (d) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are no established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to 1) or 2) below.

1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.

2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

C.2 CERTIFICATION STATEMENT

The following certification statement shall be used to document the completion of each demonstration of capability. A copy of the certification statement shall be retained in the personnel records of each affected employee (see Calscience QSM Section 6.3 and 12.3.4.b.).

Demonstration of Capability
Certification Statement

Date: Page __of __
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

Matrix: _____
(examples: laboratory pure water, soil, air, solid, biological tissue)

Method number, SOP#, Rev #, and Analyte, or Class of Analytes or Measured Parameters:
_____ (examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

_____	_____	_____
Technical Director"s Name and Title	Signature	Date
_____	_____	_____
Quality Assurance Officer"s Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

(*Note: Form may be modified so long as the essential items are included in the updated form)*

APPENDIX D - ESSENTIAL QUALITY CONTROL REQUIREMENTS

The quality control protocols specified by the laboratory"s method manual (10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals.

All quality control measures shall be assessed and evaluated on an ongoing basis and quality control acceptance criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

The requirements from the body of Chapter 5, e.g., Section 5.4, apply to all types of testing. The specific manner in which they are implemented is detailed in each of the sections of this Appendix, i.e., chemical testing.

D.1 CHEMICAL TESTING

D.1.1 Positive and Negative Controls

a) Negative Controls

1) Method Blanks - Shall be performed at a frequency of one per preparation batch of samples per matrix type. The results of this analysis shall be one of the QC measures to be used to assess the batch. The source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem if

   i) the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch or

   ii) the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

Any sample associated with the contaminated blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

b) Positive Controls

1) Laboratory Control Sample (LCS) - (QC Check Samples) Shall be analyzed at a minimum of 1 per preparation batch of 20 or less samples per matrix type, except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to assess the batch. NOTE: The matrix spike (see 2 below) may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS.

2) Matrix Spikes (MS) - Shall be performed at a frequency of one out of every 20 samples per matrix type prepared over time, except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike.

3) Surrogates - Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

4) If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene, and PCBs in Method 608), the test method has an extremely long list of components or components that are incompatible, a representative number (minimum of 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit-specified analytes, and other client-requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

D.1.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates - Shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document its procedure to select the use of appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate.

D.1.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Analytical Capability - (Section 10.5) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

b) Calibration - Calibration protocols specified in Section 9.4 shall be followed.

c) Proficiency Test Samples - The results of such analyses (4.2.j or 5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.1.4 Analytical Measurement Uncertainty Estimation

Uncertainty is “a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand” (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. For environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to have a Gaussian distribution, and be reducible by increasing the total number of measurements.

Knowledge of the uncertainty of a measurement provides additional confidence in the validity of a result as its value accounts for all the factors which could possibly affect the result. Certain test methods will specify limits to the values of sources of uncertainty of measurement (EPA 500 series methods, etc.) and will specify the form of presentation of calculated results. When the method makes these stipulations, there is no need to provide a mechanism for calculating the uncertainty. Where this information is not provided within a method or other regulatory device, the uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte and is the CEL standard for estimating analytical data uncertainty.

D.1.4.1 Using the Laboratory Control Sample (LCS) to Estimating Analytical Uncertainty

a) The LCS limits can be used to assess the performance of the measurement system since they take into consideration all of the laboratory-related variables associated with a given test over time. The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

- To calculate the uncertainty of a result, multiply the analytical result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the analytical result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. Example: If the reported result is 1.0 mg/l, and the LCS percent recovery range is 75 to 125%. The uncertainty range would be 0.75 to 1.25 mg/l, which could also be written as 1.0 +/- 0.25 mg/l.

D.1.4.2 Additional Components to Estimating Analytical Uncertainty

When estimating analytical measurement uncertainty, all significant components of uncertainty must be identified and quantified. Components that affect analytical measurement uncertainty include sampling, handling, transport, storage, preparation and testing. A typical environmental laboratory will have the greatest contribution to uncertainty in the storage, preparation and testing portion of the analytical train, hence the estimation can be limited to those three areas, assuming all other factors are within recommended guidelines for sample size, container type, preservation (chemical, temperature, temporal) and handling/transport. If the latter are *NOT* within guidelines then these additional estimations of variability must be accounted for, and may supersede the laboratory contribution to uncertainty.

Definitive references and procedural manuals for calculating Analytical Measurement Uncertainty are listed below. Note that there are different theories on the “best” way to estimate uncertainty, it is up to the end user to determine that which best meets their project needs.

a) “Environmental Analytical Measurement Uncertainty Estimation – Nested Hierarchical Approach”, William Ingersoll, Defense Technical Information Center # ADA396946, 2001

b) “Quantifying Uncertainty in Analytical Measurement”, EuraChem / CITAC Guide CG 4, Second Edition, QUAM 2000.1

c) “Quantifying Measurement Uncertainty in Analytical Chemistry – A Simplified Practical Approach”, Thomas W. Vetter, National Institute of Standards and Technology

The process in general involves the following steps:

1. Specify the Measurand – Write down a clear statement of what is being measured, including the relationship between the measurand and the input quantities, i.e., measured quantities, constants, calibration standard values, etc.

2. Identify uncertainty sources – This will include sources that contribute to the uncertainty on the parameters in the relationships identified in step 1, but may include other sources and must include sources arising from chemical assumptions.

3. Quantify uncertainty components – Measure or estimate the size of the uncertainty component associated with each potential source of uncertainty identified. It is often possible to estimate or determine a single contribution to uncertainty from the aggregate of multiple sources.

4. Calculate combined uncertainty – The information obtained in step 3 will consist of a number of quantified contributions to overall uncertainty, whether associated with individual sources or with the combined effects of several sources.

The process outlined above relates to the measurement of uncertainty for the preparative / analytical laboratory procedure. However, there are uncertainty contributions from other factors outside the preparative / analytical procedure. These can be controlled to a great extent by specifying uniform and standardized training or conditions.

Examples:

Human Factors

a) All personnel at Calscience undergo documented training in the method and / or instrument used. Minimum levels of education or experience are required.

b) Initial and continuing Demonstrations of Capability (DOC) must be performed and documented prior to and in continuance of analytical work related to their areas of responsibilities.

c) Blind Proficiency Testing samples are analyzed twice a year to gauge each department, matrix and method.

d) Data Integrity and Ethics Training are provided to new employees and on an annual basis to all employees.

Accommodation and Environmental Conditions

a) Calscience has standardized operating procedures for transport, storage and tracking of samples, extracts and digests through out the laboratory. All incoming orders are logged into a Laboratory Information System that assigns a specific identifier code to each work order, sample container and analytical result.

b) The sample control areas are secured with restricted access using card key portals. Internal chain of custody is available if the project requires.

c) The laboratory has over 35,000 sq ft of laboratory space with temperature controlled and air positive or negative environmental controls.

d) Regular safety inspections are performed to identify potentially hazardous conditions and to ensure general cleanliness.

Environmental Test Methods and Method Validation

a) All methods in use have Standard Operating Procedures (SOPs) based upon published methods from the EPA, ASTM, Standard Methods or other established body. These are controlled documents assigned to each department. An annual review is performed.

b) Each method has internal and external quality control criteria for preparative efficiency, instrument performance, calibration, continuing method performance and possible matrix effects as appropriate.

c) Ongoing Proficiency Testing program.

Equipment and Instrumentation

a) Each instrument in use has performance parameters that must be evaluated to specific standards based on the established method prior to any analytical use.

b) Routine and preventative maintenance is performed to maintain optimum operational performance.

c) Complex instrument systems are covered under manufacturer service contracts as appropriate.

Measurement Traceability

a) Every reagent used must meet the indicated purity and fitness for usage as referenced in the method SOPs.

b) All calibration standards are certified by the manufacturer to meet or exceed purity levels as recorded in the accompanying Certificate of Traceability to NIST or other standards verification.

c) Each reagent, standard or working standard is recorded, assigned a tracking identifier. This is referenced in the analytical log book as needed to assure traceability to the original source.

d) All Balances, Dispensers, Pipettors, Refrigerators, Freezers and Thermometers are checked on a daily or other routine basis to specified tolerances.

D.1.5 Detection Limits

The laboratory shall utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. Detection limits shall be determined by the protocol in the mandated test method or applicable regulation, e.g., Method Detection Limit (MDL). If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method. Refer to SOP T006, Determination of Detection Limits.

a) A detection limit study is not required for any component for which spiking solutions or quality control samples are not available such as temperature.

b) The detection limit shall be initially determined for the compounds of interest in each test method in a matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the detection limit must be determined in the matrix of interest (see definition of matrix).

c) Detection limits must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

d) All samples processing steps of the analytical method shall be included in the determination of the detection limit.

e) All procedures used must be documented. Documentation must include the matrix type. All supporting data must be retained.

f) The laboratory must have established procedures to relate detection limits with quantitation limits.

g) The test method"s quantitation limits must be established and must be above the detection limits.

D.1.6 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.1.7 Quality of Standards and Reagents

a) The source of standards shall comply with 9.3.

b) Reagent Quality, Water Quality and Checks:

   1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. The

labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.

2) Water - The quality of water sources shall be monitored and documented and shall meet method specified requirements.

3) The laboratory will verify the concentration of titrants in accordance with written laboratory procedures.

D.1.8 Selectivity

a) Absolute retention time and relative retention time aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column to separate constituents. The laboratory shall develop and document acceptance criteria for retention time windows.

b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

c) The laboratory shall document acceptance criteria for mass spectral tuning.

D.1.9 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of the test method.

Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

D.1.10 Method Validation – Modified Procedures, Non-Standard Methods, Additional Analytes

Often times, modifications to published methods are promulgated to allow the laboratory flexibility, increased productivity and, in some cases, it allows for better hazardous waste management, all while maintaining the quality of the data generated. But, this cannot be done without following standard method validation procedures to guarantee that the results achieved from the modified version are equal to or greater than the actual published or routinely accepted method.

Validation procedures are done to make sure that the sensitivity and selectivity of the process is appropriate for the method or analytes chosen. Interference checks are performed to show that the changes or additions will not contribute interferences to previous analytes or on-going processes. Accuracy and precision requirements are established, or previously defined, and used to demonstrate the capability of an analyst to perform the method, initially and on-going.

In the event that a non-standard method (significantly modified or newly-developed) is needed to meet client requirements, the method specifications and how they impact the project requirements must be relayed to the client for approval prior to beginning work on project samples. The client must understand the limits of the method, why it was developed and when it will be used on their project samples, and they must agree to its use.

Any significantly modified or newly-developed method (including the addition of analytes to established procedures) must be fully defined in a Standard Operating Procedure. The validation must be performed by qualified personnel, using appropriate reagents, standards and equipment/instrumentation and that process must be documented. The following items must be performed (as applicable to the method) and the completed documentation with all raw data provided to the Operations Manager and QA Manager for review prior to granting approval for use. A new method cannot be put into production without Operations and QA approval. For situations where NELAP approval is being sought, the method cannot be used for client samples until the certification has been received from the State, unless approval is given by the client.

D.1.10.1 Significant Modification / New Method / Additional Analyte Documentation:

Prior to the acceptance of client samples for analysis, the following documentation, as applicable to the type of modification or method status, must be provided to both Operations and QA for review and approval.

1. Approved Standard Operating Procedure for Analytical or Preparation Processes. Include all related raw data for the SOP revision with the draft version.

    a) Modification of existing method: - Revised SOP with modifications clearly spelled out:

    b) New Method: - New SOP in NELAC format – QA will assign SOP number

    c) Additional Analytes: - Revised SOP with modifications clearly spelled out:

2. Method Detection Limit (MDL) Study: Compliant with 40CFR, Part 136.

    a) Include summary form and all raw data for the review

3. MDL Verification Standard spiked at 1-4x the MDL, or the level specified by the specific program or contract. Example: 1-2x the MDL, reference specific program requirements.

    b) Recovery within 30 -150%, or a minimum response distinguishable from the established instrument noise level.

4. Reporting Limit Verification (when an MDL verification is not performed)

    a) For analytical methods, reprocess the low calibration standard as percent recovery – recovery between 50% and 150% is acceptable.

    b) For extraction methods, or where required by project or program, spike a blank matrix at the reporting limit and process through all steps of the procedure. Note the spike level and percent recoveries. Method defined control limits are used for recovery evaluation, or default recoveries between 40% and 160% if method defined limits are not available.

5. Tuning Check (as applicable to the method)

6. Degradation Check (as applicable to the method)

7. A Valid Initial Calibration and Verification

    a) Minimum of 5 sequential points, unless otherwise stated in the method or in-house SOP.

    b) Low calibration standard at or below the Reporting/Quantitation Limit.

    c) Initial Calibration Verification Standard

8. Retention Time Window Study

9. Second Column Confirmation for all analytes (as applicable to the method)

10. Inter-element Correction (as applicable to the method)

11. Linear Range Study (as applicable to the method)

12. GCMS Spectral Profile(s) (as applicable to the method)

13. Interference Check – Method Blank

    a) Analysis of a blank matrix that has gone through all related steps, preparation and /or analysis, as applicable.

14. Acceptable PT Sample required for all new analytes where NELAP accreditation is being sought.

    a) At least one PT sample (preferably two) required for all new methods

    b) Where a PT sample is not available, or accreditation is not needed, accuracy can be measured through the use of a second source standard.

15. For California ELAP or NELAP, process a real world sample for MS and MSD. The sample does not have to contain any target analytes but recoveries for surrogates, internal standards and spikes must be within lab or method defined criteria.

    a) Use Tap Water for drinking water only methods, tap or other clean water source for ground, surface, etc. methods

    b) Local Soil sample for SW-846 methods (if applying for soil or soil/water)

16. Initial Demonstration of Capability (IDOC) per analyst

    a) 4 LCS for each matrix, spiked with all associated new analytes – most acceptance criteria are in the methods, if none, use an initial recovery range of 40-160% and an RPD of 30%.

    b) Non-Standard methods – Follow the procedure in the 2003 NELAC Standards, Chapter 5 appendix C.3.3 (b).

17. Certification / Approval from Regulatory Agency where available.

APPENDIX E – LIST OF ACCREDITED METHODS

E.1 California State Department of Health NELAP Certificate 03220CA

a) View at: http://www.calscience.com/PDF/NELAP1.pdf

California State Department of Health NELAP Fields of Accreditation

a) View at: http://www.calscience.com/PDF/NELAP2.pdf

E.2 New York State Department of Health NELAP Certificate 11896 and Fields of Accreditation

a) View at: http://www.calscience.com/PDF/New_York.pdf

E.3 United States Department of Defense ELAP Certificate L10-41 and Fields of Accreditation

a) View at: http://www.calscience.com/PDF/DoD_ELAP.pdf

APPENDIX F – SPECIAL PROGRAM REQUIREMENTS

F.1 United States Department of Defense Environmental Laboratory Accreditation Program

1. Calscience Environmental Laboratories, Inc. (CEL) participates and is accredited in the United States Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP).

2. The DoD ELAP will provide a means for laboratories to demonstrate conformance to the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM) as authorized by DoD Instruction 4715.15, Environmental Quality Systems, December 2006 and as required by the DoD Policy and Guidelines for Acquisitions Involving Environmental Sampling or Testing, December, 2007. The DoD QSM is based on the National Environmental Laboratory Accreditation Conference (NELAC) Quality Systems standard (Chapter 5), which provides guidelines for implementing the international standard, ISO/IEC 17025, General Requirements for the Competence of Testing and Calibration Laboratories.

3. The DoD ELAP will apply to environmental programs / projects at DoD operations, activities, and installations, including Government-owned, contractor-operated facilities and formerly used defense sites, where testing is being performed in support of environmental restoration programs. The program will apply to all laboratories, including permanent, temporary, or mobile facilities, that generate definitive data, regardless of their size, volume of business, or field of accreditation; the collection of screening data will be governed by project specific requirements.

4. The current DoD Quality Systems Manual for Environmental Laboratories is Version 4.2, dated October 25, 2010.

5. The CEL Management will provide sufficient training, resources and other measures to ensure compliance with the DoD QSM as appropriate. (including but not limited to):

   a. Specific Standard Operating Procedures (SOPs) and / or Appendicles
   b. DoD compliant Laboratory Information Management System (LIMS) analytical test codes
   c. Specialized technician and chemist training
   d. Enhanced Quality Assurance (QA) oversight
   e. Project specific instruments
   f. Assigned Project Management personnel
   g. Quality Assurance Project Plans (QAPP)
   h. DoD analytical data reporting qualifiers
   i. Calibration and reference materials that meet DoD requirements.

APPENDIX G – LISTING OF MAJOR ANALYTICAL INSTRUMENTATION

GC/MS SYSTEMS

Designation	Manufacturer/Model	Serial Number	Acquired	Department
GC/MS-H	HP 6890/5973		1997	Semivolatiles
GC/MS-I	HP 6890/5973		1998	Summa Canister QC
GC/MS-J	HP 6890/5973		1998	Summa Canister QC
GC/MS-K	HP 6890	US00024158	1998	Air
	HP5973	US82311263	1998	
	Entech 7100A	0063		
	Entech 7016CA	00142		
GC/MS-L	HP 6890	US00023714	1998	Volatiles
	Agilent 5973	US82311287		
	Tekmar	US09163001		
GC/MS-M	HP 6890	US00028876	1999	Volatiles
	HP 5973	US9192601	1999	
	Tekmar 3100	US01313004		
	Varian Archon	15287		
GC/MS-N	HP 6890/5973		1999	Summa Canister QC
GC/MS-O	Agilent 6890N	US00034260	2000	Volatiles
	Agilent 5973	US94240048	2000	
	Tekmar 3100	US02261003		
	Varian Archon	13863		
GC/MS-P	HP 6890/5973		2000	Semivolatiles
GC/MS-Q	Agilent 6890	US00037519	2000	Volatiles
	Agilent 5973	US03340458	2000	
	Tekmar 3100	00242002		
	Varian Archon	13386		
GC/MS-R	Agilent 6890	US00037782	2000	Volatiles
	Agilent 5973	US03340489	2000	
	Tekmar 3100	00277002		
	Varian Archon	14040		
GC/MS-S	Agilent 6890	US00030897	2000	Volatiles
	Agilent 5973	US03340414	2000	
	Tekmar 3100	US01317008		
	Varian Archon	13431		
GC/MS-T	Agilent 6890	US00039185	2000	Volatiles
	Agilent 5973	US03940628	2000	
	Tekmar 3100	US01362002		
	Varian Archon	15301		

GC/MS-U	Agilent 6890	US00036171	2001	Volatiles
	Agilent 5973	US02450134	2001	
	Tekmar 3100	US02203002		
	Varian Archon	MS0903W013		
GC/MS-V	Agilent 6890	US00036172		Air
	Agilent 5973	US02450131		
	Entech 7100A	1092		
	Entech 7016CA	1041		
GC/MS-W	Agilent 6890	US00036170	2001	Volatiles
	Agilent 5973	US02450128	2001	
	Tekmar	US01362003		
	Varian Archon	13573		
GC/MS-X	Agilent 6890N	US10203064	2002	Volatiles
	Agilent 5973	US10462129	2002	
	Tekmar 3100	00305010		
	Varian Archon	13616		
GC/MS-Y	Agilent 6890/5973		2002	Semivolatiles
GC/MS-Z	Agilent 6890N	US10225110	2002	Volatiles
	Agilent 5973	US21842958	2002	
	Tekmar 3100	US02213007		
	Varian Archon	13764		
GC/MS-AA	Agilent 6890N	US10225149	2002	Air
	Agilent 5973	US21843250	2002	
	Entech 7100A	1034		
	Entech 7016CA	1183		
	Entech 7016CA	1212		
GC/MS-BB	Agilent 6890N	US1023004	2002	Volatiles
	Agilent 5973	US21843288	2002	
	Tekmar 3100	US02249001		
	Varian Archon	15208		
GC/MS-CC	Agilent 6890N	US10233039	2002	Volatiles
	Agilent 5973	US21843272	2002	
	Tekmar	US05024008		
	Varian Archon	15278		
GC/MS-DD	Agilent 6890N	US10239018	2002	Air
	Agilent 5973	US21843913	2002	
	Entech 7100A	1384		
	Entech 7016CA	1018		
	Entech 7016CA	1187		

GC/MS-EE	Agilent 6890N	US10248096	2003	Volatiles
	Agilent 5973	US21844395	2003	
	Tekmar 3100	US01317009		
	Varian Archon	MS092W026		
GC/MS-FF	Agilent 6890N	US10247091	2003	Volatiles
	Agilent 5973	US21844222	2003	
	Tekmar 3100	US02331005		
	Varian Archon	15077		
GC/MS-GG	Agilent 6890/5973		2003	Semivolatiles
GC/MS-HH	Agilent 6890N	CN10337015	2003	Air
	Agilent 5973	US30945837	2003	
	Entech 7100A	1081		
	Entech 7016CA	1012		
	Entech 7016CA	1038		
GC/MS-II	Agilent 6890	CN10517039	2005	Air
	Agilent 5973	US44647341	2005	
	Entech 7100A	1458		
	Entech 7016CA	1098		
	Entech 7016CA	1225		
GC/MS-JJ	Agilent 6890N	CN10547073	2005	Volatiles
	Agilent 5973	US53941344	2005	
	Tekmar	US05291003		
	Varian Archon	14529		
GC/MS-KK	Agilent 6890	CN10545117	2005	Air
	Agilent 5973	US53941343	2005	
	Entech 7100A	1221		
	Entech 7016CA	1207		
	Entech 7016CA	1210		
GC/MS-LL	Agilent 6890N	CN10651084	2007	Volatiles
	Agilent 5975B	US63214670	2007	
	Tekmar 3100	00088011		
	Varian Archon	15078		
GC/MS-MM	Agilent 6890/5975		2007	Semivolatiles
GC/MS-NN	Agilent 7890A	CN10717056	2007	Air
	Agilent 5975C	US71215995	2007	
	Entech 7100A	1432		
	Entech 7016CA	1211		
	Entech 7016CA	1213		

GC/MS-OO	Agilent 7890A	CN10745139	2007	Volatiles
	Agilent 5975C	US73317841	2007	
	Tekmar Stratum	US07277008		
	Tekmar SOLATek	US09049003		
GC/MS-PP	Agilent 7890A	CN10744086	2007	Volatiles
	Agilent 5975C	US73317584	2007	
	Tekmar Stratum	US07277012		
	Tekmar SOLATek	US09051008		
GC/MS-QQ	Agilent 7890A	CN10742034	2007	Volatiles
	Agilent 5975C	US71216778	2007	
	Tekmar Stratum	US07277018		
	Tekmar SOLATek	US08032004		
GC/MS-RR	Agilent 7890A	CN10730015	2007	Volatiles
	Agilent 5975C	US73317844		
	Tekmar Stratum	US08032004		
	Tekmar SOLATek	US08032006		
GC/MS-SS	Agilent 7890/5975		2007	Semivolatiles
GC/MS-TT	Agilent 7890/5975		2007	Semivolatiles
GC/MS-UU	Agilent 7890A	CN10805004	2007	Volatiles
	Agilent 5975C	US71215984	2007	
	Tekmar Stratum	US08087006		
	Tekmar SOLATek	US08092001		
GC/MS-VV	Agilent 7890A	CN10805094	2007	Volatiles
	Agilent C5975	US80118376	2007	
	Tekmar Stratum	US08283015		
	Tekmar SOLATek	US09050003		
GC/MS-WW	Agilent 7890A	CN10803015	2007	Volatiles
	Agilent 5975C	US80118375	2007	
	Tekmar Stratum	US08283014		
	Tekmar SOLATek	US09040001		
GC/MS-XX	Agilent 7890A	CN10815050	2008	Volatiles
	Agilent 5975C	US80828968	2008	
	Tekmar 3100	US02233006		
	Varian Archon	15273		
GC/MS-YY	Agilent 7890A	CN10814115	2008	Air
	Agilent C5975	US80828967	2008	
	Entech 7100A	1431		
	Entech 7016CA	1208		
	Entech 7016CA	1214		

GC/MS-ZZ	Agilent 7890A	CN10814050	2008	Air
	Agilent 5975C	US80828953	2008	
	Entech 7100A	1045		
	Entech 7016CA	1209		
	Entech 7016CA	1184		
GC/MS-AAA	Agilent 7890/5975		2008	Semivolatiles
GC/MS-BBB	Agilent 7890/5975		2009	Semivolatiles
GC/MS-CCC	Agilent 7890/5975		2009	Semivolatiles

GC SYSTEMS

Designation	Manufacturer/Model	Serial Number	Acquired	Department
GC-0	HP 5890 Series II Detector(s): ECD/ECD		1990	Semivolatiles
GC-1	HP 5890 Series II Detector(s): PID/FID	3310A48771	1987	LUFT-GRO
	OI 4560	M049460695		
	Varian Archon	12980		
GC-3	HP 5890 Detector(s): FID		1988	LUFT-DRO
GC-4	HP 5890 Detector(s): PID/FID	2750A17251	1989	LUFT-GRO
	OI 4560	B239040		
	Varian Archon	13142		
GC-5	HP 5890A Detector(s): PID/FID	2916A22147	1989	LUFT-GRO
	OI Eclipse 4660	D810466201P		
	Varian Archon	13869		
GC-6	HP 5890 Series II Detector(s): FID		1990	LUFT-DRO
GC-8	HP 5890 Series II PID/FID	3033A31219	1990	LUFT-GRO
	Tekmar 3100	US02249004		
	Tekmar 2016	US91420421		
	Tekmar 2032	95224040		
GC-9	HP 5890 Series II Detector(s): FID/FID		1991	Semivolatiles
GC-11	HP 5890 Series II Detector(s): PID/FID	3118A35205	1991	LUFT-GRO
	Tekmar 3000	94104007		
	Varian Archon	14697		
GC-12	HP 5890 Series II Detector(s): FID/TCD		1991	Semivolatiles

GC-13	HP 5890 Series II FID/TCD	3033A32929	1990	Air
GC-14	HP 5890 Series II Detector(s): FPD	3126A36770	1991	Air
GC-15	HP 5890 Series II Detector(s): FID		1991	LUFT-DRO
GC-16	HP 5890 Series II Detector(s):ECD/ECD		1992	Semivolatiles
GC-18	HP 5890 Series II Detector(s): PID/FID		1992	LUFT-GRO
	EST ENCON	512080906		
	Varian Archon	15307		
GC-19	HP 5890 Detector(s): FID	2750A17344	1988	Air
GC-21	HP 5890 Series II Detector(s): PID/FID	3336A51475	1994	LUFT-GRO
	OI 4560	H416460182		
	HP35900E	3350A01927		
	Varian Archon	MS0902W025		
GC-22	HP 5890 Series II+ Detector(s): PID/FID	3336A61360	1994	LUFT-GRO
	OI Eclipse 4660	D815466810P		
	Varian Archon	14699		
GC-24	HP 5890 Series II+ Detector(s): PID/FID	3336A53949	1994	LUFT-GRO
	Tekmar 3000	98194007		
	Varian Archon	13864		
GC-25	HP 5890 Series II+ Detector(s): PID/FID	2921A23805	1994	LUFT-GRO
	Tekmar 3100	314009		
	Varian Archon	13478		
GC-26	HP 6890 Detector(s): NPD/NPD		1995	Semivolatiles
GC-27	HP 6890 Detector(s): FID/FID		1998	LUFT-DRO
GC-29	HP 5890 Series II Detector(s): PID/FID	3310A47430	2000	LUFT-GRO
	Tekmar 3100	US02249008		
	Tekmar 2016	US90120414		
	Tekmar 2032	95124003		
GC-31	HP 6890 Detector(s): ECD/ECD		2000	Semivolatiles

GC-33	HP 5890 Series II Detector(s): FID	2908A21573	2000	Air
GC-34	HP 5890 Series II Detector(s): FID	3033A32699	2000	Air
GC-35	Agilent 6890N Detector(s): NPD/NPD		2002	Semivolatiles
GC-36	Agilent 6890N Detector(s): FID/TCD	us10346058	2004	Air
GC-37	Agilent 6890N Detector(s): ECD/ECD		2004	Semivolatiles
GC-38	HP 5890 Series II Detector(s): FID	3029A30188	1995	Air
GC-39	HP 5890 Series II Detector(s): FID	3336A58721	1995	Air
GC-40	Agilent 7890N Detector(s): ECD/ECD		2007	Semivolatiles
GC-41	Agilent 7890N Detector(s): ECD/ECD		2007	Semivolatiles
GC-42	Agilent 6890N Detector(s): PID/FID	CN10647056	2007	LUFT-GRO
	Tekmar 3100	US01274007		
	Varian Archon	14370		
GC-43	Agilent 6890N Detector(s): FID		2007	LUFT-DRO
GC-44	Agilent 6890N Detector(s): FID/FID		2007	Semivolatiles
GC-45	Agilent 7890A Detector(s): FID/FID		2007	LUFT-DRO
GC-46	Agilent 7890A Detector(s): FID/FID		2007	LUFT-DRO
GC-47	Agilent 7890A Detector(s): FID/FID		2008	LUFT-DRO
GC-48	Agilent 7890A Detector(s): FID/FID		2008	LUFT-DRO
GC-49	Agilent 7890A Detector(s): FID/FID		2008	LUFT-DRO
GC-50	Agilent 7890A Detector(s): FID/FID		2008	LUFT-DRO
GC-51	Agilent 7890A Detector(s): ECD/ECD		2008	Semivolatiles
GC-52	Agilent 7890N Detector(s): FID	CN10824005	2008	Air
GC-53	Agilent 6890N Detector(s): FID	US00002691	2000	Air

GC-54	Agilent 7890A Detector(s): FPD	US10840051	2008	Air
GC-55	Agilent 7890N Detector(s): TCD	CN10844112	2008	Air
GC-56	Agilent 7890N Detector(s): FID	CN10847124	2009	LUFT-GRO
	OI Eclipse	D647466449P		
	Varian Archon	15139		
GC-57	Agilent 7890N Detector(s): ECD/ECD	CN10847113	2009	LUFT-GRO
	OI Eclipse	D81466987P		
	Varian Archon	15140		
GC-58	Agilent 7890N		2009	Semivolatiles
GC-59	Agilent 7890N Detector(s): FID	CN10041127	2009	Air
SPARE EQP.	OI Eclipse 4660	D709466195P		LUFT-GRO
	Varian Archon	13874		

Inductively Coupled Plasma Spectrophotometers (ICP)

Designation	Manufacturer/Model	Serial Number	Acquired	Department
ICP-6	PE Optima 5300 DV		2006	Metals
ICP-7	PE Optima 7300 DV		2008	Metals

Inductively Coupled Plasma/Mass Spectrometers (ICP/MS)

Designation	Manufacturer/Model	Serial Number	Acquired	Department
ICP/MS-3	PE ELAN DRC-e		2009	Metals
ICP/MS-4	PE ELAN DRC-e		2009	Metals

Flame Atomic Absorption Spectrometers (FAA)

Designation	Manufacturer/Model	Serial Number	Acquired	Department
FAA-2	PE AA300		1999	Metals

Mercury Analyzers

Designation	Manufacturer/Model	Serial Number	Acquired	Department
HG-4	PE FIMS-400		2005	Metals
HG-5	PE FIMS-400		2005	Metals

High Performance Liquid Chromatographs (HPLC)

Designation	Manufacturer/Model	Serial Number	Acquired	Department
HPLC-5	Agilent 1100 HPLC Detector(s): UV		2001	Semivolatiles
HPLC-6	Agilent 1100 HPLC Detector(s): UV		2001	Semivolatiles
HPLC-7	Agilent 1100 HPLC Detector(s): UV		2004	Semivolatiles
HPLC-8	Agilent 1100 HPLC Detector(s): UV		2007	Semivolatiles

Liquid Chromatography/Mass Spectrometry (LC/MS/MS)

Designation	Manufacturer/Model	Serial Number	Acquired	Department
LC/MS/MS-1	Varian 1200L Triple Quad Detector(s): MS		2005	Inorganics

WET Chemistry Instrumentation

Designation	Manufacturer/Model	Serial Number	Acquired	Department
UV-2	HP 8453 Detector(s): Diode Array		1999	Inorganics
VIS-1	Milton Roy Spectronic 20 Detector(s): VIS		1992	Inorganics
TOC-2	OI Model 1010 Detector(s): IR		2000	Inorganics
TOC-3	OI Model 1020 Detector(s): IR		2003	Inorganics
TOC-4	OI Soil Module Detector(s): IR		2003	Inorganics
TOC-5	OI Soil Module Detector(s): IR		2007	Inorganics
IC-2	Dionex DX-100 Detector(s): Conductivity		1996	Inorganics (Anions)
IC-3	Dionex DX-500 Detector(s): Absorbance		2000	Inorganics (Cr(VI))
IC-5	Dionex DX-600 Detector(s): Absorbance		2001	Inorganics (Cr(VI))
IC-6	Dionex DX-320 Detector(s): Conductivity		2002	Inorganics (Perchlorate)

IC-7	Dionex ICS-1000 Detector(s): Conductivity		2003	Inorganics (Anions)
IC-8	Dionex ICS-2000 Detector(s): Conductivity		2004	Inorganics (Perchlorate)
IC-9	Dionex ICS-1000 Detector(s): Conductivity		2008	Inorganics (Anions)
IC-10	Dionex ICS-1000 Detector(s): Conductivity		2008	Inorganics (Anions)
IC-11	Dionex ICS-3000 Detector(s): Absorbance		2009	Inorganics (Cr(VI))
IC-12	Dionex ICS-3000 Detector(s): Absorbance		2009	Inorganics (Cr(VI))
FA1	OI 3360 Flow Analyzer Detector(s): UV		2007	Inorganics
UV-2	HP 8453 Detector(s): UV		1999	Inorganics
UV-3	Thermo Detector(s): UV		2007	Inorganics
UV-4	Thermo Detector(s): UV		2007	Inorganics
UV-5	Thermo Detector(s): UV		2007	Inorganics
UV-6	Bekman-Coulter Detector(s): Diode Array		2000	Inorganics
UV-7	Agilent 8453 Detector(s): Diode Array		2008	Inorganics

Automated Extractors

Designation	Manufacturer/Model	Serial Number	Acquired	Department
ASE-1	Dionex ASE-200		1999	Extractions
ASE-2	Dionex ASE-200		1999	Extractions
ASE-3	Dionex ASE-300		2002	Extractions
ASE-4	Dionex ASE-300		2002	Extractions
ASE-5	Dionex ASE-200		2007	Extractions
ASE-6	Dionex ASE-200		2007	Extractions

Miscellaneous

Designation	Manufacturer/Model	Serial Number	Acquired	Department
Gas Mixer	Environics Series 2000	1490		Air
Gas Mixer	Environics Series 2000	4618	2009	Air

END OF DOCUMENT

Title : **INTERNAL QUALITY CONTROL CHECKS**

Document No. : SOP-T020
Revision No. : 2.4
Supersedes : 2.3

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Revision 2.4 changes are noted in bold italicized typeface and preceded by a "►" marker.

APPROVED FOR RELEASE BY: _____ 03/28/11
MANAGEMENT **DATE**

_____ 03-28-11
QA DEPARTMENT **DATE**

ANNUAL SOP REVIEW

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

1. PURPOSE

1.1. The laboratory must, on an ongoing basis, demonstrate through the analysis of quality control (QC) performance based measurement systems (PBMS) that the operation of the analytical system is in control.

1.2. This document is provided to clarify and standardize the performance and evaluation of these QC measurements or checks. The types of checks that are addressed are initial calibrations, initial and continuing calibrations, method blanks, laboratory control samples, matrix spikes, unspiked duplicate samples, surrogates and control limits.

1.3. These QC Checks are a subset of Quality Control Procedures instituted for the entire laboratory and outlined in the current "Quality Systems Manual".

2. POLICIES

2.1. Different regulatory programs have different requirements to monitor the appropriateness and performance of the analytical process. The guidance provided in this document is intended to establish an operational standard for the laboratory, but shall be subordinate to specific requirements in the regulatory methods or current NELAC accreditation standard. At a minimum, quality control checks shall be prepared and analyzed at the frequency to meet the regulatory requirements of the method or current NELAC accreditation standard.

2.2. This policy for quality control checks may also be superseded by project or client-specific requirements that are equal or more stringent. Requests for changes to this policy should be received in writing from the client and filed with the project report data or can be noted in a Quality Assurance Project Plan (QAPP) that will be developed and referenced prior to log-in. Deviations to standard procedures should be summarized in the case narrative on the final laboratory report.

3. COMMENTS

3.1. DEFINITION OF TERMS

3.1.1. ENVIRONMENTAL SAMPLE: A portion of soil, water, air or other matrix submitted to Calscience for qualitative and/or quantitative analysis. Routinely, these are billable samples submitted by clients under a chain of custody (CoC) record for analysis by the analytical method noted on the CoC record.

3.1.2. ANALYTICAL SYSTEM: The complete analytical process conducted on environmental samples. This includes sample storage, preparation procedures/apparatus, analytical procedures/instrumentation, and data processing/result generation.

3.1.3. QUALITY CONTROL MEASUREMENTS OR CHECKS: Internal and external processes performed to monitor various steps throughout the sampling, transfer, preparation and analysis of environmental samples.

External QC checks typically involve the use of trip, equipment or rinsate, and temperature blanks to monitor contamination/degradation originating from the collection and transportation of environmental samples. Internal QC checks involve the use of method blanks, matrix spike and laboratory control samples, unspiked duplicates, and surrogate spike analyses to monitor matrix effects and contamination/ degradation originating from the laboratory.

3.1.4. PREPARATION BATCH: All environmental and QC check samples of similar matrix prepared at the same time using the same preparation procedure, typically 20 or less in number prepared within the batch window.

3.1.5. ANALYTICAL BATCH: All environmental and QC check samples of similar matrix prepared and analyzed referencing the same QC sets. Note: Some samples may reference the same MS/MSD data, but different MB and LCS data, typically 20 or less in number analyzed within the batch window.

3.2. Other types of QC checks related to specific analytical technologies, such as post-digestion spikes (PDS), interference check samples (ICS), and serial dilutions for trace metals analysis, are performed as required by the regulatory method. Due to their specificity, they are beyond the scope of this document and shall be addressed in the individual analytical SOPs.

3.3. A Laboratory Information Management System (LIMS) program called "Statistical Quality Control" (SQC) is an additional layer of QC checks that is available for evaluation of ongoing QC processes.

4. HAZARDS AND PRECAUTIONS

4.1. None.

5. APPARATUS AND MATERIALS

5.1. None.

6. REAGENTS AND STANDARDS

6.1. None.

7. ►PROCEDURE

7.1. CALIBRATION STANDARDS

7.1.1. For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the appropriate determinative SOP or mandated method. ***Additional standards may be prepared to extend the calibration range of the instrument.***

7.1.2. If a reference or mandated method does not specify the number of calibration standards, usually five, the minimum number is three, not including blanks or a zero standard.

7.1.2.1. ***In the event that a determinative SOP specifies the number of standards for use in the calibration, approval to deviate or depart from this procedure must be granted by Operations <u>and</u> QA, and this approval documented, if the total number of standards used will be less than the number stated in the SOP. In all cases, the minimum number must be three, not including blanks or a zero standard, unless the SOP or published method specifies a lesser amount. And, in all cases the lowest standard must support the reporting limit, or the reporting limit must be elevated to the level of the next valid standard.***

7.1.2.1.1. ***Before approval is granted, Operations and QA must be aware of any potential conflicts with client or project specific calibration requirements and they must take into account any potential impacts on the data created by the deviation. Reasons for permitting the departure from standard procedures must be documented and the documentation maintained by QA.***

7.1.2.1.2. ***Additionally, the client must be informed of the permitted departure and any potential impacts on the data must be noted in a case narrative.***

7.1.2.2. ***The addition of standards beyond the number stated in the SOP can be done without requesting a deviation. The additional levels must be documented in the run sequence. If the addition of the standards will be part of the routine process, the SOP must be revised to include this information. At no time can the addition of standards for the sole purpose of extending the calibration range be used to report data beyond the linear range of the instrument. The calibration curve must meet the correlation coefficient or %RSD requirements.***

7.1.2.3. ***If defined in an SOP, standard concentrations may be different from those stated in the SOP as long as the lowest calibration standard is at or below the method/analyte reporting limit. Samples with results above the upper concentration range of the curve must be diluted and reanalyzed to bring the values within the mid to upper range of the active calibration curve. The concentrations should be noted on the run sequence.***

7.1.3. The lowest concentration standard must be at or below the stated reporting limit for the method based on the final volume of extract (sample). The highest standard normally defines the upper portion of the detector's linear range.

7.1.4. Due to instrument and analyte responses, it may be useful to prepare six or maybe even seven standards for calibration. The lower five standards could be used for most analytes and the upper five for analytes that have poor instrument response. In situations such as these, the reporting limit or linear range will need to be adjusted to account for the standards used in generating the calibration.

7.1.4.1. The analysis of six or more standards also allows for the generation of a least squares regression (quadratic curve) for those analytes that do not have a linear response.

7.2. INITIAL CALIBRATION

7.2.1. At a minimum, analytical systems shall be calibrated once a year independent of whether the analytical system is still deemed to be in control.

7.2.2. Establish operating parameters based upon method and determinative SOP guidance. Select appropriate columns, injection volumes, detectors, temperatures, temperature programming and flow rates to optimize chromatographic resolution. Once the operating parameters have been established according to the determinative SOP, each instrument may then be calibrated for sample analysis.

7.2.3. Before initial calibration, make sure the system is 'clean' (no results or contamination greater than the reporting limit or ½ the reporting limit depending on the project) by injecting a solvent blank into the system. If the system is not clean, inject another blank. If contamination is still an issue, perform system maintenance to eliminate the source of the problem.

7.2.3.1. Methods that use Mass Spectral determination (GC/MS, ICP/MS, LC/MS, etc) will need to be tuned prior to the analysis of the initial calibration standards. Methods may use internal or external calibration techniques. Please refer to the appropriate determinative SOP for additional guidance on tuning and calibrations.

7.2.4. Once it is determined that the system is ready for calibration, inject the calibration standards sequentially into the instrument, preferably beginning with the lowest standard first and working up to the highest.

7.2.4.1. Do not inject multiple standards of the same calibration level. The process of injecting multiple standards of the same value and then picking the "best" standard is considered "cherry picking' and is not considered to be good laboratory practice. A properly maintained system operating at its most optimal levels should not be difficult to calibrate.

7.2.5. Policies regarding the use of calibration standard results for creating the curve are:

- It is never appropriate to remove points from a calibration curve unless they are the highest or the lowest point and the reporting limit and/or linear range is adjusted accordingly. ***For a curve requiring a minimum of 5 points, 5 points must remain in the curve (unless otherwise stated in the method) for it to be considered valid. For a curve requiring a minimum of 3 points, 3 points must remain in the curve for it to be considered valid (unless otherwise stated in the method). Refer to section 7.1.2. for information on limited permission to depart from this policy.***
- A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting level must be elevated to be the lowest calibration standard used for calibration.
- The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
- Mid-calibration standards may not be excluded unless an obvious reason is found. In the instance where an analyst can clearly document the reason for removing a middle point (not the highest or lowest) from the curve (e.g., bad injection, broken vial, no injection, etc.), a single point may be reanalyzed once and the reanalysis used for the calibration. The failed standard should be re-run and inserted into the initial calibration. This may only happen if the re-analysis of the point occurs before any QC or client samples are analyzed or before the analytical shift or tune cycle (12 or 24 hours) has ended. If not useful, or if anything else is run on the instrument, recalibration is required.

7.2.6. Once the calibration standards have been analyzed and the curve has been generated, carefully review it for compliance with the associated SOP, ***published method or QAPP*** criteria (%RSD, correlation coefficient, etc.), ***as applicable.*** If there is a problem with the calibration using average response factors, utilize a linear regression model (1st order), or quadratic (2nd order) if sufficient standards (≥ 6) were analyzed.

7.2.6.1. For linear models, a correlation coefficient (r) of at least 0.995 is required before beginning sample analysis. ***Project specific criteria may only require a correlation coefficient of ≥ 0.990. Refer directly to the project specific QAPP for calibration acceptance criteria, if available. Otherwise, the current default acceptance criterion is ≥ 0.995.***

7.2.6.2. Instead of a linear curve model, a second order curve (quadratic) may be used as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then

quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (r^2) for the quadratic curve must be at least 0.990 for it to be considered acceptable.

7.2.6.2.1. Some limitations on the use of Quadratic Curve fits;

- Care MUST be exercised to assure that the results are real, positive, and fit the range of the initial calibration.
- They may not be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).
- They may not be used to compensate for a saturated detector. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve, leaving at least five, and try a 1st order fit or average RF.

7.3. INITIAL CALIBRATION VERIFICATION (ICV)

7.3.1. Also known as the Second Source Standard because it is acquired from a source other than that used to make the initial calibration standards.

7.3.2. In general, each new initial calibration must be verified using a second-source standard (ICV) that contains all of the analytes of interest for the specific method being analyzed. The ICV is normally prepared at a level that represents the mid-point of the calibration curve and is analyzed immediately after the calibration curve has been analyzed and before the analysis of samples or batch QC begins.

7.3.3. The initial calibration is deemed valid if the percent difference (or drift) for the ICV is within the criteria specified in the appropriate determinative SOP.

7.3.3.1. As a general rule, acceptance limits for calibration verifications (%D) are; GCMS: ± 20%, GC and HPLC: ± 15%, Inorganics: ± 10 or 15%. Actual methods may have wider or tighter limits.

7.3.3.2. EPA 8260 and EPA TO-15 may use a second source standard for the LCS. In this case, as long as the 'LCS' is analyzed immediately following the calibration standards, it is treated as an ICV and must be verified against the ICV criteria specified in the ***SOP or published*** method.

7.3.3.3. ***If there is no method or SOP criteria available, compare the results to the historical control limits for the LCS/LCSD.***

7.3.4. If the ICV does not validate the initial calibration then:

1) Reanalyze the ICV once, or
2) Reprepare or acquire new standard and analyze once,
3) Evaluate instrument conditions, and
4) Regenerate a new ICAL.

7.3.5. If the initial calibration has been verified by the ICV then the analysis of samples may proceed.

7.4. CONTINUING CALIBRATION VERIFICATION (CCV)

7.4.1. Usually made from the same source as the calibration standards but may be from a second or even third source.

7.4.2. Once established, every initial calibration must be verified periodically by the analysis of a continuing calibration verification standard (CCV).

7.4.3. In general, the initial calibration must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. The analytical shift begins with the injection of the CCV (or the tuning standard in MS methods). A CCV must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the minimum, the end of each analytical batch.

7.4.3.1. Some methods or client/project directives may have more frequent CCV requirements; opening CCV, closing CCV and bracketing CCVs every 10 or 20 samples, and most Inorganic methods require the CCV to be analyzed after ever 10 samples.

7.4.4. It is recommended that only one CCV be analyzed at a time. The process of routinely analyzing 2 CCV's in a row creates the potential for confusion regarding acceptance of the sequence. If both pass, or both fail, then the acceptability or the corrective action needed is clear. But, if one passes and one fails then the analyst is left to decide the validity of the associated data.

7.4.4.1. If the initial CCV passes but the second CCV fails then any data previous to the initial CCV has been successfully bracketed, but any data following the failed CCV has not been, (the 'opening' CCV has failed). Corrective action for the later samples would need to be effected (possibly involving recalibration) and the samples re-analyzed. The opposite may be true for an initial CCV that failed but the second CCV passed.

7.4.5. The initial calibration is deemed valid if the percent difference (or drift) for the CCV is within the criteria specified in the appropriate determinative SOP.

7.4.5.1. As a general rule the acceptance limits for calibration verifications (%D) are; GCMS: ± 20%, GC and HPLC: ± 15%, Inorganics: ± 10 or 15%. Actual methods may have wider or tighter limits.

7.4.6. If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the initial calibration to quantitate sample results.

7.4.6.1. Unless otherwise stated in a specific method, sample results are always quantitated from the ICAL and not the CCV.

7.4.7. If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second consecutive calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications (one below the mid-point of the curve), or a new initial calibration must be performed.

7.4.7.1. A second CCV may be analyzed following a failing CCV if no samples / QC have been analyzed and the analytical tune or batch period has not expired. The reason for the analysis of the additional CCV must be documented in the run log (i.e., note why the initial CCV failed and what corrective action was taken).

7.4.8. However, sample data associated with an 'unacceptable' CCV may be reported as qualified data under the following special conditions:

- When the acceptance criteria for the CCV are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the CCV are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

7.5. METHOD BLANK (MB)

7.5.1. Method blanks, also known as analytical, reagent or preparation blanks, are analyzed to determine the existence and magnitude of background interference or contamination resulting from the analytical system which might lead to the reporting of elevated concentration levels, including false positive data.

7.5.2. The method blank must contain all the reagents and in the same volumes as used in the processing of environmental samples and must be carried through the same preparation procedures using the same type lab ware and apparatus. This includes any cleanup steps that associated samples may be subjected to.

7.5.3. Unless specified otherwise in the analytical method or by project-specific requirements, one (1) method blank, at a minimum, must be processed and analyzed for each group of twenty (20) or fewer samples of similar matrix.

7.5.4. Concentration of target analytes in the method blank should be less than their respective reporting limits. If the concentration is equal to or exceeds this limit, the source of contamination must be investigated and if possible, eliminated. Some common laboratory solvents and metals are difficult to eliminate from the analytical system. Project specific concentrations in the method blanks may call for a lower standard of detection, i.e., must be ≤ ½ Reporting Limit (RL).

7.5.5. It is not required to run the method blank on all instruments or systems analyzing samples, but the analyst must demonstrate (through a procedure or other blank) that each instrument is void of contamination prior to analysis each day of use. However, unless specifically exempted by the method, a method blank must be analyzed with every analysis. Certain methods are precluded from this provision, i.e., pH and Specific Conductance.

7.5.6. When target analytes are found in the method blank, their levels must be evaluated to determine their effect on the analysis of environmental samples.

7.5.7. Sample results must <u>not</u> be corrected by subtracting the method blank value.

7.5.8. For organic analyses, the following shall apply:

7.5.8.1. If no target analytes are found in the method blank, report sample and method blank data with no qualifications.

7.5.8.2. If target analytes are found in the method blank, but not in the associated samples, report sample and method blank data with no qualifications. The analytical system should be monitored, but no further corrective action is required.

7.5.8.3. If a target analyte is found in the method blank and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of environmental samples. Report the sample data with a "B" qualification footnote indicating that the analyte was present in the corresponding method blank. Determine and eliminate the source of contamination. Depending upon the effect on the data quality objectives (DQOs) of the project, samples may require re-preparation and reanalysis or sample data may be reported with appropriate qualification indicating that the analyte was present in the corresponding method blank. Professional judgement should be used to determine if the data should be qualified or rejected and samples re-prepared and reanalyzed.

7.5.9. For inorganic analyses, where reporting limits are usually near the Instrument Detection Limit (IDL), the following shall apply:

7.5.9.1. If no target analytes are found in the method blank, report sample and method blank data with no qualifications. No corrective action is required.

7.5.9.2. If a target analyte is found in the method blank and the analyte concentration in all samples associated with the method blank is below the RL, report sample and method blank data with no qualifications. The analytical system should be monitored, but no further corrective action is required.

7.5.9.3. If a target analyte is found in the method blank between the reporting limit and two (2) times the reporting limit, the analyte in the associated samples are reported with qualification to indicate contamination was present in the corresponding method blank. No further corrective action is required.

7.5.9.4. A method blank with a target analyte above two (2) times the reporting limit is unacceptable unless the lowest concentration of the analyte in question in the associated samples is at least 10 times the concentration in the method blank. In this case, the analyte in the associated samples is reported with appropriate qualification to indicate contamination was present in the corresponding method blank at less than or equal to ten (10) times the concentration found in the samples. Report the sample data with a "B" qualifier indicating that the analyte was present in the corresponding method blank. No further corrective action is required.

7.5.9.5. If a target analyte is: a) found in the method blank above two (2) times the RL, and b) found in the corresponding samples below ten (10) times MB level, the background level of this target analyte must be evaluated to determine its effect on the analysis of environmental samples. Determine and eliminate the source of contamination. Depending upon the effect on the data quality objectives (DQOs) of the project, samples may require repreparation and reanalysis or sample data may be reported with appropriate qualification indicating that the analyte was present in the corresponding method blank. Professional judgement should be used to determine if the data should be qualified or rejected and samples reprepared and reanalyzed. If reported, report the sample data with a "B" qualification footnote indicating that the analyte was present in the corresponding method blank.

7.5.10. Drinking water samples (or samples analyzed by drinking water methodologies) will not be analyzed if the method blank contains regulated analytes above the reportable limits. In this case, the analytical system is

considered out of control and, as such, must be corrected prior to proceeding with sample analyses.

7.6. LABORATORY CONTROL SAMPLES (LCS)

7.6.1. Laboratory control samples (LCS/LCSD) are analyzed at a minimum of one pair per batch to assess the overall performance of the analytical system, including all preparation and analysis steps. Results for the LCS/LCSD are compared to established criteria and if found to be outside of these criteria, indicates that the analytical system is out of control.

7.6.1.1. As the acceptability of the MS/MSD data cannot be determined at time of batch preparation, the LCS/LCSD shall be prepared, analyzed, and evaluated with each batch regardless of need.

7.6.2. The LCS/LCSD are prepared identical to the MS/MSD, but employ a blank matrix (reagent water for aqueous, clean sand for solids) to preclude any interference from environmental samples.

7.6.3. The LCS/LCSD are identically fortified with known quantities of representative target analyte(s) and processed along with environmental samples. The spike compounds are prepared or obtained from sources independent of the calibration standards except in cases where an independent source Initial Calibration Verification (ICV) is performed daily. The spike concentration shall be near the midpoint calibration range. Unless project or method requirements specify otherwise, the LCS shall be prepared using the same spike solution used for the MS. Spiking shall occur before sample preparation.

7.6.4. The LCS spike compound list is identical to that of the MS compound list.

7.6.5. The laboratory control samples must contain all the reagents and in the same volumes as used in the processing of environmental samples and must be carried through the same sample preparation procedures using the same labware and apparatus.

7.6.6. Organic methods typically require the reporting of laboratory control samples whether the MS/MSD data are outside control limits or not. In instances where the MS/MSD data is non-compliant, analysis of additional fortified samples in the form of Laboratory Control Sample Duplicates (LCSD) are required to properly evaluate the performance of the analytical system and validity of the resultant data. Typically, inorganic methods, such as trace metals analysis, require a LCS in conjunction with other QC checks, e.g., MS and Matrix Spike Duplicate (MSD) sample. Unless specified otherwise in the analytical method or by project-specific requirements, the following frequency shall apply.

7.6.7. LCS/LCSD FOR UNACCEPTABLE MS/MSD

7.6.7.1. If a LCS/LCSD pair is analyzed due to an unacceptable MS/MSD set, the LCS/LCSD shall be prepared at the same frequency as that for the MS/MSD. That is, at a minimum, one (1) laboratory control sample and laboratory control sample

duplicate (LCS/LCSD) must be analyzed for each group of twenty (20) or fewer samples.

7.6.7.2. The analytical or preparatory batch shall not extend past one 24-hour period from the start of batch. Certain methods may require a shorter time period, e.g., 12 hours. In all cases, the method requirements shall take precedence.

7.6.7.3. It is not required to run the LCS/LCSD set on all instruments analyzing samples, but it is recommended that the set should be analyzed together on the same instrument. The analytical group may elect to analyze the LCS/LCSD to maximize use of instrument time, but the LCS/LCSD data may be reported at the discretion of the project manager or per QAPP guidelines.

7.6.7.4. Accuracy information is obtained by determining the recovery of the fortified analyte(s) in each duplicate spike following sample analysis and is expressed in percent recovery (%REC). The %REC must be evaluated against the method-specified control limits. When control limits are not specified in the regulatory method, limits may be statistically generated from in-house historic analytical data or adopted from similar methods.

$$\%REC = \frac{\text{spiked sample result}}{\text{spike added}} \times 100$$

7.6.7.5. Precision information is obtained by determining the relative percent difference (RPD) between duplicate spike measurements. The RPD must be evaluated against the method-specified control limits. When control limits are not specified in the regulatory method, limits may be statistically generated from in-house historic analytical data or adopted from similar methods.

$$RPD = \frac{|\text{LCS result} - \text{LCSD result}|}{(\text{LCS result} + \text{LCSD result}) \div 2} \times 100$$

7.6.7.6. When the %REC and RPD of the LCS/LCSD are at or within the established control limits, the analytical system is deemed to be compliant with the accuracy and precision requirements of the method. The LCS/LCSD data shall be reported in addition to the MS/MSD data with the corresponding environmental samples. No other fortified check samples are required for the analytical batch.

7.6.7.7. If the %REC and/or RPD of the LCS/LCSD are not within the established control limits, the analytical sequence should be terminated. Given the absence of matrix interferences from environmental samples, unacceptable LCS/LCSD data is

indicative of a problem with the analytical system. The source(s) of system error must be determined and eliminated prior to recalibration and restart of sample analysis.

7.7. MATRIX SPIKES (MS)

7.7.1. Matrix spikes are analyzed, as required, to provide information about the effect of each sample matrix on the accuracy and precision of the sample preparation procedures and measurement method.

7.7.2. Matrix spikes are aliquots of environmental samples identically fortified with known quantities of representative target analyte(s) and processed along with environmental samples. The spike compounds are prepared or obtained from sources independent of the calibration standards. The spike concentration shall be near the midpoint calibration range. Spiking shall occur before sample preparation.

7.7.3. When possible, the sample for spike shall not contain the target analyte (or other spike interferences) above 4 times the spike level. That is, the sample spike should be at least ¼ the background level found in the sample for spike. If this condition is not met, the resultant %REC & RPD measurements may not correctly reflect the true accuracy and precision of the analytical system. If this situation arises, the QC data should be qualified as such.

7.7.4. SPIKING COMPOUNDS

7.7.4.1. For Non-Drinking Water (Hazardous Waste) Samples

7.7.4.1.1. Spiked samples shall contain a minimum of six (6) representative or ten percent (10%) of the target analytes, whichever is greater. If there are less than six analytes in the method, all target analytes must be used.

7.7.4.1.2. The spike compounds selected should span the range and categories of method analytes. The analytes chosen for spiking should range from early to late eluters and should, if possible, cover the general types of method analytes, e.g., for EPA Method 8260, chlorinated, aromatic, and ketone solvents.

7.7.4.2. For Drinking Water Samples, samples must be spiked with a minimum of all regulated analytes in the applicable method.

7.7.4.3. For single analyte analysis such as those typical of fuels analyses, the spiking compound will be the standard used for the analysis (e.g., gasoline, diesel, etc.).

7.7.5. The matrix spike must contain all the reagents and in the same volumes as used in the processing of environmental samples and must be carried

through the same sample preparation procedures using the same type labware and apparatus.

7.7.6. For certain methods, such as pH and Conductivity, matrix spikes are not required. Typically, instrumental organic and inorganic methods require both a MS and a MSD, at a minimum, per analytical batch. Unless specified otherwise in the analytical method or by project-specific requirements, the following frequency shall apply.

7.7.6.1. For instrumental organic and inorganic methods requiring matrix spikes, at a minimum, one (1) matrix spike and matrix spike duplicate (MS/MSD) must be processed and analyzed for each group of twenty (20) or fewer samples of similar matrix.

7.7.6.2. The analytical or preparatory batch shall <u>not</u> extend past one 24-hour period from the start of batch. Certain methods may require a shorter time period, e.g., 12 hours. In all cases, the method requirements shall take precedence.

7.7.7. It is recommended that the MS/MSD set should be analyzed together on the same instrument.

7.7.8. Accuracy information is obtained by determining the recovery of the fortified analyte(s) in each duplicate spike following sample analysis and is expressed in percent recovery (%REC). The %REC must be evaluated against the method-specified control limits. When control limits are not specified in the regulatory method, limits may be statistically generated from in-house historic analytical data or adopted from similar methods.

$$\%\text{REC} = \frac{\text{spiked sample result} - \text{sample result}}{\text{spike added}} \times 100$$

7.7.9. Precision information is obtained by determining the relative percent difference (RPD) between duplicate spike measurements. The RPD must be evaluated against the method-specified control limits. When control limits are not specified in the regulatory method, limits may be statistically generated from in-house historic analytical data or adopted from similar methods.

$$\text{RPD} = \frac{|\text{MS result} - \text{MSD result}|}{(\text{MS result} + \text{MSD result}) \div 2} \times 100$$

7.7.10. When the %REC and RPD of the MS/MSD are at or within the established control limits, the analytical system is deemed to be compliant with the accuracy and precision requirements of the method for this matrix. The MS/MSD data shall be reported with the corresponding environmental samples along with the LCS. Additionally, if an LCS/LCSD pair was analyzed the LCS and LCSD should be reported.

7.7.11. If the %REC and/or RPD of the MS/MSD are not within the established control limits, the analytical system performance shall be suspect.

7.7.12. Out of control %REC values are typically caused by matrix effects or poor instrument performance/technique. Out of control RPD values are typically caused by sample inhomogeneity or poor instrument performance/ technique. To properly evaluate the performance of the analytical system in these situations, laboratory control samples (LCS/LCSD) must be analyzed and evaluated to determine if this condition is due to matrix interference. Inorganic metals should report one LCS if the MS/MSD % recoveries are out of control or both LCS/LCSD if MS/MSD % RPD is outside control limits.

7.7.13. If the LCS/LCSD data are acceptable, the data for all environmental samples associated with the MS/MSD are reported with qualification to indicate matrix interference in the case narrative. If the LCS/LCSD results are not acceptable, thus indicating a problem with the analytical system, all samples associated with the analytical batch shall be re-prepared and reanalyzed if sufficient sample and holding times allow. Otherwise, qualify all data and discuss in the case narrative.

7.8. UNSPIKED DUPLICATE SAMPLE

7.8.1. Some regulatory methods specify the analysis of unspiked duplicate samples, also known as replicates. Because this measurement monitors method precision, typically, only a single spike (MS or LCS) is required to measure method accuracy.

7.8.2. A lab-wide requirement for unspiked duplicate sample analysis does not exist. They shall be performed only when required by the regulatory method or as specified by project requirements, based upon the following calculation.

$$\text{RPD} = \frac{|\text{Sample result} - \text{Duplicate result}|}{(\text{Sample result} + \text{Duplicate result}) \div 2} \times 100$$

7.8.3. Samples identified as field blanks (equipment, rinsate, or field) should not be used for unspiked duplicate analysis as the anticipated ND values may not provide a meaningful measurement of method precision.

7.8.4. In situations where all samples in the analytical batch contain little or no detectable target analytes, the RPD policy for the handling and calculation of non-detect sample duplicates should be exercised. The policy is as follows:

- If the target is detected in both duplicate runs, the RPD between the duplicate runs shall be calculated and reported.
- If the target analyte is not detected (ND) in both duplicate runs, the RPD shall not be calculated and "N/A" shall be reported.

- If the target analyte is detected on one run and ND in the other, calculate and report the RPD using the reporting limit as the concentration of the ND run.

7.9. SURROGATE SPIKE ANALYSIS

7.9.1. Surrogates are added to samples to determine, primarily, the effect of the matrix on the accuracy of analysis of each individual sample. Information on laboratory performance of each sample may also be obtained from analysis of surrogate spikes.

7.9.2. Surrogates are organic compounds which are similar to the analytes of interest in analytical behavior, but are not normally found in environmental samples. The surrogate(s) should not interfere with or be subject to interference from target analytes.

7.9.3. When use of a surrogate(s) is specified in the regulatory method and analytical SOP, they shall be added to all environmental and QC check samples. The surrogate(s) are to be added to each sample prior to sample preparation.

7.9.4. The surrogate(s) shall be evaluated against its respective method-specified control limits by determining the percent recovery (%REC). When control limits are not specified in the regulatory method, limits may be statistically generated from in-house historic analytical data or adopted from similar methods.

$$\%REC = \frac{\text{surrogate result}}{\text{surrogate spike added}} \times 100$$

7.9.5. If the surrogate(s) recovery falls within acceptance criteria, report surrogate(s) and sample data with no qualifications.

7.9.6. If the surrogate(s) recovery falls outside acceptance criteria, evaluation is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high analyte concentrations or a problem may have occurred in sample preparation. This data alone cannot be used to evaluate the precision and accuracy of individual sample analyses. However, when exercising professional judgement, this data should be used in conjunction with other available QC information.

7.9.7. The following corrective actions must be taken if the surrogate(s) recovery is outside the method-specified recovery criteria.

7.9.7.1. Check raw data to ensure that there are no calculation or transcription errors. Check that surrogate spike recoveries were calculated correctly.

7.9.7.2. Check internal standard and surrogate spiking solutions for degradation and contamination.

7.9.7.3. If the nonconformance is due to poor instrument performance or if the above corrective actions fail to reveal the cause of the noncompliant surrogate recovery, the same extract may be reanalyzed if it has been properly stored and remains intact. Although, advantageous for most semivolatile methods, reanalysis of the extract is not applicable to volatile methods unless the sample had undergone solvent extraction (e.g., due to high analyte concentration).

7.9.7.4. By itself, a surrogate recovery outside acceptance criteria does not invalidate the run. If the above corrective actions fail to correct the problem, evaluate all other QC parameters to determine if the analytical system is out of control.

7.9.7.5. If the analytical process is judged to be out of control, the cause of the error must be determined and corrected before sample analysis proceeds. All affected samples should be reextracted and reanalyzed. Recalibration of the instrument may be necessary.

7.10. CALCULATION OF IN-HOUSE CONTROL LIMITS

7.10.1. The %REC and RPD data from spiked and unspiked sample analyses are compared to their respective control limits to identify out-of-control and potential out-of-control situations.

7.10.2. When control limits are not specified in the regulatory method, the laboratory may choose to generate its own limits or adopt limits from similar methods. In no case are control limits allowed to exceed maximums which may be set by the various methods.

7.10.3. In addition to environmental sample data, Calscience's Laboratory Information Management System (LIMS) collects and stores, pertinent QC data. Statistical analysis of this data from the actual instrumentation, processes, and analysts performing the method generate the most representative limits for statistical control of that particular operation. Use of the laboratory's historic QC data is the preferred method of establishing control limits when they are not designated in the regulatory method.

7.10.4. Laboratory-generated (in-house) control limits for acceptance and warning levels are established by statistical analysis of a pool of historic QC data generated at Calscience.

7.10.4.1. The acceptance limits represent the range in which the analytical system is deemed to be in statistical control. Under a normal distribution curve, the probability of a single measurement lying in the range is 99.74%. Acceptance limits for accuracy (%REC) are based upon three standard deviation (± 3S) units about the mean. Acceptance limits for precision (RPD) are based upon zero (no difference) and three standard deviation (3S) units above zero.

7.10.4.2. The warning limits represent the range in which the probability of a single measurement lying in the range is 95.44%. Measurements outside this range indicate an increased potential for the analytical system to go out of control and should prompt the analyst to more closely monitor the process. Procedures for the determination of warning limits are identical to those for acceptance limits, except 2S is used instead of 3S.

7.10.5. Generation of in-house control limits shall be coordinated by the QA Department and determined as follows:

7.10.5.1. For a given method and matrix, representative data points are pooled for each control parameter (e.g., MS data shall be pooled with MSD data, but separate from LCS/LCSD data).

7.10.5.2. The pool shall consist of only values that vary due to random error. Random errors, also known as indeterminate errors, are unavoidable random variations in measurements that occur when the analytical system, including the analyst, is operating correctly.

7.10.5.3. Blunders (mistakes that occur on occasion and produce erroneous results) are not included in the pool. Values below 20% and above 180% recovery normally result from an incorrectly operating instrument or analyst error and shall be excluded from the data pool for %REC measurements.

7.10.5.3.1. Exceptions: Some poorly performing analytes may be allowed wider ranges, i.e., 10% to 190% when control limits are established. Acceptable recovery limits should not be set at less than 10% for any analyte.

7.10.5.4. If thirty (30) data points meeting the above criteria are not available, the laboratory may elect to perform statistical analysis on a smaller sample population (not less than 20).

7.10.5.5. From the pool of QC data, calculate arithmetic mean ($\bar{x}$) and standard deviation (S):

$$\bar{x} = \frac{x_1 + x_2 + x_3 + \cdots + x_n}{n}$$

$$S = \sqrt{\frac{\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2 \Big/ n}{n-1}}$$

where a concentration x_i is measured n times.

Note: When the Standard Deviation is calculated using a calculator, spreadsheet or other automated device, it must be QA verified that the calculation is performed as "sample-based", not "population-based".

7.10.5.6. The new control limits shall be based upon ±3 standard deviations units about the arithmetic mean.

Upper control limit (UCL) = $\bar{x}$ + 3S
Lower control limit (LCL) = $\bar{x}$ − 3S

7.10.6. Control limits require periodic updates to accurately redefine statistically acceptable operation of an analytical system over a given period. The frequency of updates is dependent upon the stability of the analytical system, which varies with instrumentation and technique employed.

7.10.7. The NELAC requirements (2003 Standard, Section D.1.1.2.1 Item e) allow the usage of Marginal Exceedance control limits for those analyses with multiple reporting analytes.

7.10.7.1. The NELAC standards state that if a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control therefore corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.

- > 90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit.
- 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit.
- 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit.
- 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit.
- 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit.

7.10.7.2. The ME must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systemic problem. The same analyte exceeding the LCS control limit 2 out of 3 consecutive LCS is indicative of non-random behavior.

7.10.7.3. The number of allowable marginal exceedance is based on the number of analytes in the LCS. If there is any analyte that exceed the LCS control limits, it does not necessary mean the LCS fails. The NELAC standard states if the number of

analytes fails LCS control limits but within the ME limits, it is acceptable. The rules of the number of allowable ME is as follows:

7.10.7.4. The ME must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systemic problem. The same analyte exceeding the LCS control limit 2 out of 3 consecutive LCS is indicative of non-random behavior.

7.10.7.5. For DoD projects, there is no allowance for project-specific ***analytes*** of concern to exceed the LCS ***and/or LCSD*** control limits, even marginally. ***Where target analytes are defined by the project, those analytes must fall within the LCS/LCSD criteria.***

7.10.8. The control limits calculated via historical data are subject to over-ride to arbitrary default limits, either on the lower control limit (LCL) or upper Control limit (UCL) if the calculated limits fall out side of these nominal ranges:

7.10.8.1. Surrogates:

- Minimum Percent Recoveries Range @ 80–120%
- Maximum Percent Recoveries Range @ 20–180%

7.10.8.2. Matrix Spikes:

- Minimum Percent Recoveries Range @ 75–125%
- Maximum Percent Recoveries Range @ 20–180%
- Minimum Reported Percent Difference @ 15%
- Maximum Reported Percent Difference @ 40%
- Exceptions: Some poorly performing analytes may be allowed wider ranges, i.e., 10–190% when control limits are over-ridden. These are typically semi-volatile extractable compounds. At no time should the LCL be set at less than 10%.

7.10.8.3. Laboratory Control Samples:

- Minimum Percent Recoveries Range @ 80–120%
- Maximum Percent Recoveries Range @ 50–150%
- Minimum Reported Percent Difference @ 10%
- Maximum Reported Percent Difference @ 30%

7.11. STATISTICAL QUALITY CONTROL (SQC) PROGRAM

7.11.1. A Laboratory Information Management System (LIMS) program called "Statistical Quality Control" (SQC) is an additional layer of QC checks that is used.

7.11.2. It applies a modified version of the "Western Electric Company Rules for Control Charts (WECO)" in an automatic scan of entered analytical data for Method Blanks, Laboratory Control ***and Matrix Spike*** Percent Recoveries and Continuing Calibration Verifications (CCVs).

7.11.3. An e-mail notification is sent to the appropriate party(s) for investigation if an activated rule is violated. The system allows real time monitoring to allow quick intervention by the chemist or group leader prior to an analytical process becoming out of control. See example Appendix 9.1.

7.11.3.1. ***Upon receipt of the e-mail, the notified party can click on a link at the bottom which will take them directly to the QC chart of concern. There, both the flagged data and associated control chart can be viewed.***

7.11.4. Note: Not all QC checks flagged by the SQC program warrant corrective action. Examples may include ME LCS outliers, MS outliers, etc. However, repeated outliers of a similar type may indicate that the current control limits need revision.

7.11.5. The available rules are:

- Rule 1: Any point above average + 3 sigma
- Rule 2: Two out of the last three points above average + 2 sigma
- Rule 3: Four out of the last five points above average + 1 sigma
- Rule 4: Eight consecutive points above average
- Rule 5: Eight consecutive points below average
- Rule 6: Four out of the last five points below average − 1 sigma
- Rule 7: Two out of the last three points below average − 2 sigma
- Rule 8: Any point below average − 3 sigma
- Rule 9: Any point of concentration value above ½ RL for MB (check for client specific failure)
- Rule 10: Any point of concentration value above RL for MB
- Rule 11: Six consecutive points in a downward trend
- Rule 12: Six consecutive points in a upward trend
- Rule 13: Fourteen points in a row alternating up and down
- Rule 14: Eight consecutive points above average + 2 sigma
- Rule 15: Eight consecutive points below average − 2 sigma

7.11.6. The current activated rules are:

- ***Rule 4: Eight consecutive points above average + 1 sigma***
- ***Rule 5: Eight consecutive points below average − 1 sigma***
- ***Rule 9: Any point of concentration value above ½ RL for MB***

- ***Rule 11: Six consecutive points in a downward trend***
- ***Rule 12: Six consecutive points in a upward trend***
- ***Rule 14: Eight consecutive points above average + 2 sigma***
- ***Rule 15: Eight consecutive points below average – 2 sigma***

7.11.7. ***The rules have different meanings for how the control data should be interpreted, and in general, are as follows:***

<u>Rules 4 and 5:</u> Systems operating in control would tend to distribute points symmetrically around the mean. Therefore, if this pattern is identified then it is assumed that the 'process average' has changed due to potential shifts in the process itself and a prolonged bias exists. Successive analyses with less-than-average variability are worth investigating to possibly determine ways to decrease the variability in the process.

If, after investigation it appears that the process is sound, run new control limits to account for the adjustment, up or down, in the mean. A minimum number of points (20) must be available before running new control limits. It is suggested that at least 40 or 50 points be available.

<u>Rule 9:</u> This rule is used for monitoring the potential contamination of the process or system. The acceptance criteria are only applied to samples that are required to meet this project defined criteria. But, it is a good tool for monitoring the entire process.

If low level contamination is noted on a regular basis, then investigation into the cause, and corrective action to remove the contamination, should be initiated.

<u>Rules 11 and 12:</u> This indicates that there is a trend or drift in the 'process average' possibly caused by standard degradation, an increase or decrease in skill level for production-related individuals (manual extraction, standard preparation, etc.) or increased or decreased instrument performance.

From this we may need to initiate further training, prepare new reagents or standards or perform instrument maintenance.

<u>Rules 14 and 15:</u> Systems operating in control would tend to distribute points symmetrically around the mean. Therefore, if this pattern is identified then it is assumed that the 'process average' has changed dramatically due to potential shifts in the process itself and a significant prolonged bias exists. Successive analyses with less-than-average variability are worth investigating to possibly determine ways to decrease the variability in the process.

If, after investigation it appears that the process is sound, run new control limits to account for the adjustment, up or down, in the mean.

A minimum number of points (20) must be available before running new control limits. It is suggested that at least 40 or 50 points be available.

7.11.8. To access the program directly through LIMS, enter the SQC (QC Rule Check) program under "Launcher". When in the data program, enter the following information based upon your e-mail notification:

- Work Order Number
- Sample Number
- Instrument Identification
- Test Code

Go to "Special" in the header of the data entry page and then go down to where the QC Rule Checker tab is located. Click on the link.

7.11.9. Press show results button and perform investigation. The "Rules" Column" may show one of three color-coded boxes for each evaluated parameter. Red for Failure, Yellow for Warning and Green for Pass. Clicking the selected box will choose increasingly descriptive and detailed information on the data points being evaluated. Effect corrective actions if needed or as appropriate. See example Appendix 9.2.

8. ►RESPONSIBILITIES

8.1. The Project Manager must ascertain the data quality objectives (DQOs) to determine the QA/QC requirements specific to each project. Full knowledge of analytical and deliverable requirements is necessary to properly determine required resources and to properly bid the work.

8.2. Should the project specifications require the laboratory to deviate from standard procedures, the Project Manager(s) must effectively communicate the deviations to the appropriate laboratory personnel, or establish a Quality Assurance Project Plan (QAPP). It is highly recommended that communications of this sort be in written form.

8.3. All personnel involved in the generation, review, and approval of laboratory data must be apprised and act in accordance with the QC requirements of the project at hand.

8.4. The QA department is responsible for generating and updating the various laboratory-established control limits.

8.5. ***The Group Leaders and Data reviewers are responsible for reviewing the QC Rule Checker program and the associated details with outlier events. Where it is reasonably suspected that a QC rule violation is occurring due to a process change, Operations and QA must be notified. If it is instrument related, notify the appropriate operations personnel and perform the required maintenance to bring the system back into control.***

9. APPENDICES

9.1. Example of SQC e-mail.

9.2. Examples of SQC LIMS screen.

Appendix 9.1 – Example of SQC E-mail

From: QCRuleChecker@calscience.com [mailto:QCRuleChecker@calscience.com]
Sent: Friday, March 25, 2011 1:01 PM
To: Larry Lem; ankit@paccommtech.com; Xue-Wen Xu
Subject: Warning QC: LCSDME/

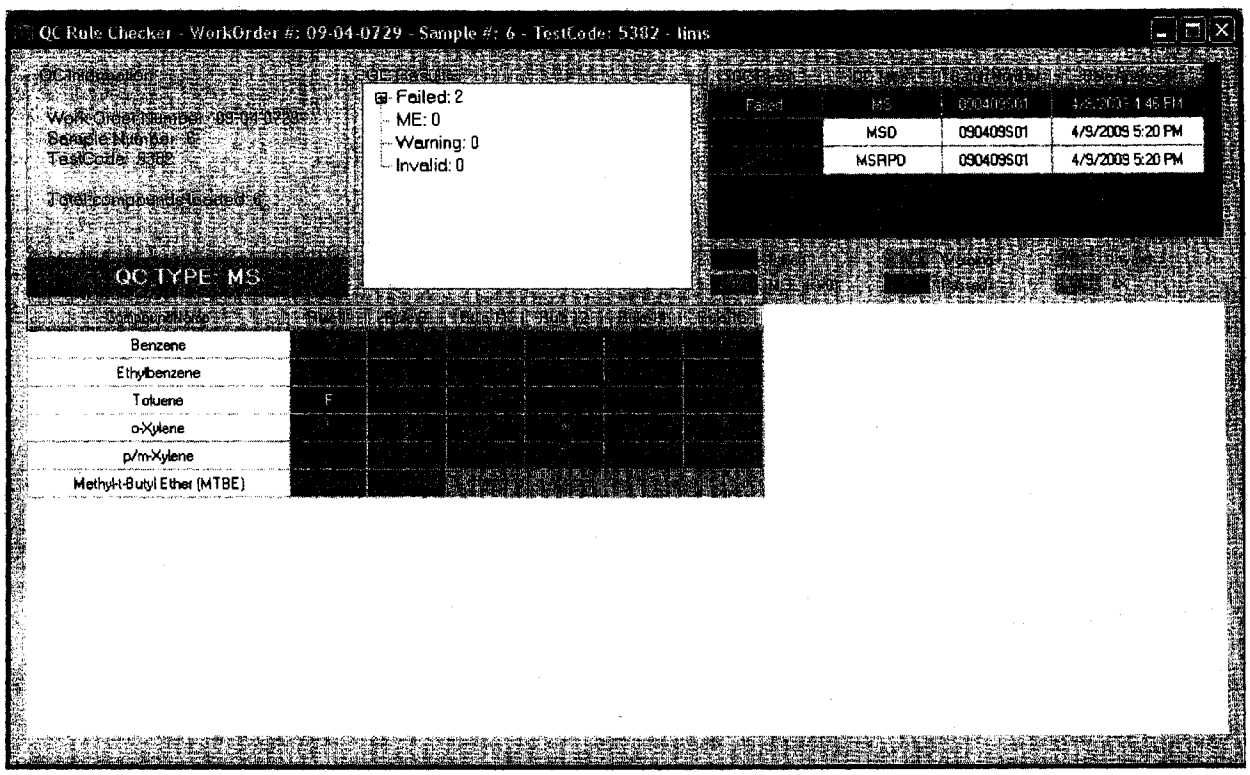
Work Order #: 097-01-003
Sample #: 11514
Testcode: 6010B_W_TTLC

QC TYPE: LCSDME
Result: Warning
Failed Compounds -
Cobalt - Rule 4
Nickel - Rule 4

Rule - Rule Description
4 - Eight consecutive points above average + 1 sigma

Click Here to run QC Rule Checker program for this validation.

Appendix 9.2 – Examples SQC LIMS Screen



Appendix 9.2 – Examples of SQC LIMS Screen (Cont.)

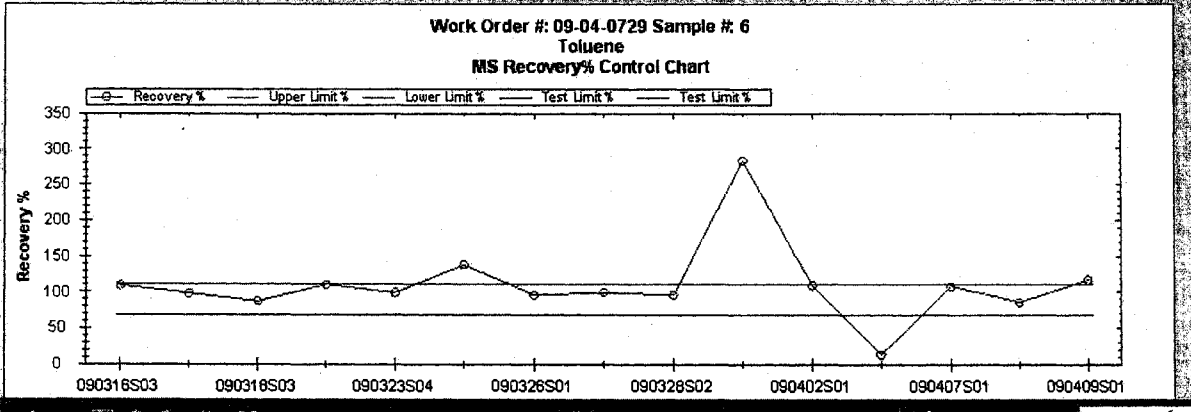
[QC Type: MS] [Compound: Toluene] [Rule #: 1] - lims

QC TYPE: MS
Failed

Sample Num	Batch Num	Date Analyzed	Conc.	Recovery	Analyst	Approved By
6	090409S01	4/9/2009 4:46 PM	23.533	117.13	Dung Do	
2	090408S01	4/8/2009 2:24 PM	17.4	86.37	Dung Do	
1	090407S01	4/7/2009 4:20 PM	21.873	108.475	Suketu Parikh	Ilsun Kwak
1	090403S02	4/4/2009 2:15 AM	10.545	13.73	Dung Do	Ilsun Kwak
3	090402S01	4/2/2009 3:01 PM	22.139	109.205	Dung Do	Ilsun Kwak
2	090401S01	4/1/2009 5:53 PM	60.608	283.307	Dung Do	Ilsun Kwak
1	090328S02	3/29/2009 7:13 AM	21.935	96.385	Dung Do	Ilsun Kwak
2	090326S02	3/26/2009 9:31 PM	20.137	99.775	Dung Do	Ilsun Kwak
2	090326S01	3/26/2009 2:10 PM	19.281	95.825	Dung Do	Ilsun Kwak
2	090325S01	3/25/2009 1:28 PM	55.044	137.77	Dung Do	Ilsun Kwak
1	090323S04	3/25/2009 4:24 AM	23.42	99.17	Dung Do	Ilsun Kwak

Compound Detail
Compound SD: 7%
AVG: 89%
Spike: 20
Control Limit: 68-110%

Rule Description
Rule Number 1
Any point above average + 3 sigma
Test Limit: 110%



start | Microsof... | QCRuleChe... | QC Rule Ch... | [QC Type: ... | Document1 ... | copernic

Title : **PROJECT MANAGEMENT AND ANALYTICAL REPORT REVIEW**

Document No. : SOP-T062
Revision No. : 1.1
Supersedes : 1.0

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Revision 1.1 changes are noted in bold italicized typeface and preceded by a "►" marker.

APPROVED FOR RELEASE BY: _____ 12/7/9
MANAGEMENT **DATE**

_____ 12/07/09
QA DEPARTMENT **DATE**

ANNUAL SOP REVIEW

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

1. PURPOSE

1.1. This document presents the procedures for Project Management including project development, log-in, invoicing and the review of analytical reports prior to client release via paper hardcopy or electronic media.

1.2. Procedures for preparation of electronic data deliverables (EDDs), i.e., numeric data via e-mail/disk, Portable Document File (PDF) or other electronic media may be referenced at SOP-T040, Electronic Data Deliverables.

1.3. Procedures for submission of EDDs can be found in SOP-T012, Transmission of Test Records.

2. POLICIES

2.1. Project Management is a systemic process of understanding a client's requirements, developing guidelines for implementation and adherence of those requirements by the laboratory, review of final reports to ensure those requirements have been met, and any follow up work based upon client feedback after receipt of the analytical report.

2.2. Project Management entails (but is not limited to) project inquiries, project development, certifications, quotations, regulatory guidance, analytical advisement, sample containers, holding times, shipping, subcontracting, sample receipt, work order log-in, QA/QC issues and final report review.

2.3. Typically, only assigned Project Managers (PMs), either permanent or temporary, may sign a finalized analytical report. Assistant Project Managers (APMs) with appropriate training and experience may also do so as approved by the Client Manager and Client Services Director.

2.4. Analytical reports will be released to the client only after a thorough review of all raw and final data is performed. The Report Review Checklist, Laboratory Reporting Requirements and Quality Assurance Project Plans (QAPPs) are tools to assist in this review.

2.5. All analytical report submissions to the client will be documented on the work order Project Status Sheet.

3. COMMENTS

3.1. None.

4. HAZARDS AND PRECAUTIONS

4.1. None.

5. APPARATUS AND MATERIALS

5.1. None.

6. REAGENTS AND MATERIALS

6.1. None.

7. DEFINITIONS

7.1. Analytical Report: A compilation of test results that have been reviewed and signed by the reviewer, either in ink or via electronic signature, as final and which will subsequently be conveyed to the client.

7.2. Case Narrative: A comprehensive summary and overview of all items pertinent to the receipt, condition and analytical processes for environmental samples that assist a reviewer of the analytical report to judge the integrity and validity of the analytical results.

7.3. ►Laboratory Reporting Requirements: A generalized listing of requirements for Level I, II, III and IV data deliverable packages, reference Appendix ***A***. The Level II package is the default.

7.4. Project Status Sheet: A sheet attached to the front of a specific work order file that details the work order number, project manager, due dates(s), specific analysis, tracking of subcontracted analysis, analysis completion dates, change orders and any and all transmittals of reports, results and invoices to the clients.

7.5. Quality Assurance Project Plan (QAPP): Projects that have specialized, extensive QA programs or other project requirements that differ from the normal CEL QA package have a specific laboratory directive or guide known as Quality Assurance Project Plan (QAPP). This is written and implemented by the Project Manager. These QAPPs are cataloged and are referenced on the Work Order log-in as appropriate.

7.6. ►Quotation/Project Checklist: Streamlined checklist ensuring that all pertinent areas have been accounted for, reference Appendix ***B***.

7.7. ►Quotation/Project Questionnaire: A comprehensive and detailed expansion of the Quotation/Project Checklist that is recommended for less experienced Project Managers or Assistant Project Managers, reference Appendix ***C***.

7.8. ►Report Review Checklist: The checklist used by the Project Manager or Assistant Project Manager to perform step-by-step review of all facets of the analytical report and data package(s), reference Appendix ***D***.

7.9. Test Results: Hardcopy or electronic information related to or resulting from the performance of analytical tests. This includes instrument output, datasheets, logbooks, forms, analytical reports, project records and all other records that require traceability.

7.10. Work Order File: A file that holds all associated data for that particular Work Order and is fronted with the Project Status Sheet. These are color coded to the Project Manager to aid in quick identification.

8. PROCEDURE

8.1. Project Development

8.1.1. An initial consultation with the client using the Quotation/Project Checklist and Questionnaire enables the Project Manager to assess the scope of the proposed project and to evaluate the ability of the laboratory to meet the analytical requirements. It is HIGHLY recommended that a project be fully understood and delineated prior to receipt of samples. Once the project parameters are prepared and conveyed to laboratory staff, i.e., via work notes, QAPPs, etc., the ease in project management, evaluation and review of the final report is significantly enhanced.

8.2. Work Order Log-in

8.2.1. When samples are received at the laboratory they undergo an inspection to check that all containers, preservatives are as appropriate for the requested analysis and that the sample identifications, dates and other chain of custody information matches the samples. Reference SOP T100 for a more detailed narrative of the sample receipt process. Discrepancies should be resolved as expeditiously as possible so that accurate log-in may proceed.

8.2.2. The sample receipt log-in will produce a Project Work Sheet that will detail the exact set of analysis being performed on each sample(s). It is imperative that the Project Manager or Assistant Project Manager review the Project Work Sheet and revise any log-in errors so that the project samples are analyzed correctly in the requested time frame and to obviate misdirected laboratory time and effort.

8.3. Ongoing Project Management

8.3.1. Large or complicated projects require continued assessment during the analytical process to ensure that QA/QC issues are addressed as they develop. Examples where this may be necessary include: Sample matrix effects on reporting levels, QA/QC parameters that are missing, incomplete or marginal or questions of interpretation in client requirements.

8.3.2. The Laboratory Information Management System (LIMS) has a data reporting program that allows real time viewing of completed work and is useful for gauging the progress of analytical work as the report due date nears. This program is also used to process the final report.

8.4. Evaluation of Completeness

8.4.1. ►Analytical reports are a compilation of practices, procedures and employee work product that are designed to provide a final report that meets the client's requirements. It is incumbent for the PM or APM to

efficiently review all data packages so that any remedial analysis, if so needed, can be performed within analytical holding time. Proper ongoing project management will be helpful in precluding any "surprises" as the report comes due. The Report Review Checklist (Appendix ***D***) is an invaluable aid as a template for covering all facets in the review process.

8.5. Final Reporting

8.5.1. ►Assuming all data packages are complete and the data passes the review checklist, the PM/APM may begin assembling the final report package using the previously mentioned LIMS program. Certain analytical formats may allow for an all electronic package, i.e., Portable Document File (PDF), others may require extensive paper hardcopy that requires additional data files from the respective departments, i.e., Level III raw data. If a Level III package is required, the associated checklist (Appendix ***E***) is available to ensure completeness.

8.5.2. ►Projects may require a Case Narrative. This is a comprehensive summary and overview of the analytical process from the point of sample receipt to final reporting. The items covered may include (but not limited to):

8.5.2.1. Sample Receipt: Temperature, condition, any anomalies in samples, etc.

8.5.2.2. Chain of Custody Review: Discrepancies between samples and COC, inconsistencies between tests requested and sample volumes/types received.

8.5.2.3. Change Orders: Any changes in scope of work after receipt of samples.

8.5.2.4. Analytical Process Review: Sample preparation, analytical QA/QC outliers, manual integrations, any other items that are relevant to the validity and integrity of the analytical processes.

8.5.3. Once the final report is assembled and ready to report, note any special instructions or QAPP guidance for reporting, i.e., Electronic Data Deliverables (EDD), other report recipients, etc.

8.5.4. Reference SOP-T040, Electronic Data Deliverables or SOP-T012, Transmission of Test Records for additional guidance.

8.6. Invoicing

8.6.1. ►Many clients or projects are priced individually; hence, it is important to invoice the analytical work using the agreed contracted costs. The Laboratory Information Management System (LIMS) has a specialized program to allow client specific or project specific pricing, based on test codes used and a fixed price assigned or a specified percentage from list price. Additional features allow variable RUSH surcharges, miscellaneous item costs (EDDs, Level III Packages) and supplies (Tedlar™ bags, En Core® sampling devices, ***Terra Core® sampling devices***, etc.).

9. RESPONSIBILITIES

9.1. All Project Managers and Assistant Project Managers are responsible for adhering to the policy and procedures provided in this document.

9.2. The Client Services Director shall ensure that the respective staff understand the requirements and execute the procedure outlined in this document.

10. REFERENCES

10.1. ►***None.***

11. ►APPENDICES

11.1. ***Appendix A: Laboratory Reporting Requirements.***

11.2. ***Appendix B: Quotation/Project Checklist.***

11.3. ***Appendix C: Quotation/Project Questionnaire.***

11.4. ***Appendix D: Report Review Checklist.***

11.5. ***Appendix E: Standard Level III Deliverable Checklists.***

Appendix A

LABORATORY REPORTING REQUIREMENTS

Calscience Environmental Laboratories, Inc.

Appendix A

Laboratory Reporting Requirements

Unless stated otherwise by project requirements, a Level II deliverables package shall be provided by default.

Level I

General Information:

1) Narrative or analytical summary.
   a) Condition of samples, as received.
   b) Summary of method deviations, if any.
   c) QC or sample problems (and corrective action) not already addressed within the report, if any.
2) Date of sample collection.
3) Date of sample receipt.
4) Date of sample preparation/extraction, if applicable.
5) Date of analysis.
6) Methods of analysis.
7) Parameters of interest.
8) Sample results (with qualifiers, as appropriate).
9) Results of field (trip, equipment, rinsate) blanks, if provided.
10) Reporting limits.
11) Original chain of custody (copy to remain in project file).

Inorganic Analyses:

1) Results of method blanks (MBs), as applicable.
2) Results of matrix spike/matrix spike duplicate (MS/MSD) analyses, as applicable.
   a) Parameters of interest.
   b) Percent recovery, control limits.
   c) RPD, control limits.
   d) Qualifiers, as appropriate.
3) If method employs analysis of duplicate samples in lieu of MS/MSD, the RPD of the sample/sample duplicate shall be reported as the measurement of precision.

Organic Analyses:

1) Results of method blanks (MBs).
2) Results of matrix spike/matrix spike duplicate (MS/MSD) analyses.
   a) Parameters of interest.
   b) Percent recovery, control limits.
   c) RPD, control limits.
   d) Qualifiers, as appropriate.

Appendix A

Laboratory Reporting Requirements (Cont.)

Level II

All requirements specified for Level I plus the following where applicable:

Inorganic Analyses:

1) If MS/MSD out of control, also report results of laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses.
   a) Parameters of interest.
   b) Percent recovery, control limits.
   c) RPD, control limits.
   d) Qualifiers, as appropriate.

Organic Analyses:

1) If MS/MSD out of control, also report results of laboratory control sample/laboratory control sample duplicate (LCS/LCSD) analyses.
   a) Parameters of interest.
   b) Percent recovery, control limits.
   c) RPD, control limits.
   d) Qualifiers, as appropriate.
2) Results of surrogates, as applicable.
   a) Parameters of interest.
   b) Percent recovery, control limits.
   c) Qualifiers, as appropriate.

Appendix A

Laboratory Reporting Requirements (Cont.)

Level III

All requirements specified for Level II plus the following where applicable:

General Information:

1) Client/Laboratory sample ID cross reference list.

Inorganic Analyses:

1) Preparation and run logs.
2) Initial calibration curve (ICAL) standards.
   a) Concentrations of standards.
   b) Results of standards.
   c) Acceptability (RSD or correlation coefficient).
   d) Acceptance range.
3) Result of initial calibration blank (ICB).
   a) Result of blank.
   b) Acceptability.
4) Result of initial calibration verification (ICV) standard.
   a) Concentration of standard.
   b) Result of standard.
   c) Acceptability (percent recovery or difference).
   d) Acceptance range.
5) Results of continuing calibration blanks (CCBs).
   a) Results of blanks.
   b) Acceptability.
6) Results of continuing calibration verification (CCV) standards.
   a) Concentrations of standards.
   b) Results of standards.
   c) Acceptability (percent recoveries or differences).
   d) Acceptance range.
7) Results of interference check samples (ICS), where applicable.
   a) Concentration of elements.
   b) Results of elements.
   c) Acceptability (percent recoveries).
   d) Acceptance range.
8) Results of dilution check samples, where applicable.
   a) Results of samples.
   b) Expected values.
   c) Acceptability (percent recoveries).
   d) Acceptance range.
9) Results of post-digestion spike (PDS) analyses, where applicable.
   a) Results of analyses.
   b) Expected values.
   c) Acceptability (percent recoveries).
   d) Acceptance range.

Appendix A

Laboratory Reporting Requirements (Cont.)

Level III (Cont.)

All requirements specified for Level II plus the following where applicable:

Organic Analyses:

1) Preparation and run logs.
2) Initial calibration curve (ICAL) standards.
   a) Concentrations of standards.
   b) Results of standards.
   c) Acceptability (RSD or correlation coefficient).
   d) Acceptance range.
3) Result of initial calibration blank (ICB).
   a) Result of blank.
   b) Acceptability.
4) Result of initial calibration verification (ICV) standard.
   a) Concentration of standard.
   b) Result of standard.
   c) Acceptability (percent difference or drift).
   d) Acceptance range.
5) Results of continuing calibration blanks (CCBs).
   a) Results of blanks.
   b) Acceptability.
6) Results of continuing calibration verification (CCV) standards.
   a) Concentrations of standards.
   b) Results of standards.
   c) Acceptability (percent differences or drifts).
   d) Acceptance range.
7) Sample chromatograms for chromatographic analyses.
8) Results of spectral tuning with BFB or DFTPP for GC/MS analyses.
9) Results of system performance check compounds (SPCC) and calibration check compounds (CCC) for GC/MS analyses.
10) Internal standard summaries.
11) Results of tentatively identified compounds (TICs), if requested.

Appendix A

Laboratory Reporting Requirements (Cont.)

Level IV

Level IV is Contract Laboratory Program (CLP) protocol and deliverables.

Refer to applicable CLP Statement of Work (SOW). This includes CLP detection limits, control limits and target analyte lists (TALs). SOW must be specified during project set-up.

If CLP deliverables are not specified, Level IV shall be interpreted to be "CLP-like" deliverables. These shall include all of the data specified for Level III, with the addition of all applicable supporting raw data and quality control data.

Appendix B

QUOTATION/PROJECT CHECKLIST

Calscience Environmental Laboratories, Inc.

Appendix B

Calscience Environmental Laboratories, Inc.

Quotation/Project Checklist

1. Any special **certifications** required for this project? _____
2. **Project I.D**/Site/Number? _____
3. **Target analytes & methods** defined? _____
4. **Normal Reporting Limits** faxed to client or otherwise defined by client? _____
   Any unusual reporting units? _____
5. **Turnaround Time** defined? Ensure that client understands what our definition of TAT is. _____
   For RUSH TAT, what is due exactly when? _____
6. **QA/QC reporting** discussed? _____
7. **Are** the use of **flags acceptable**? _____
   Our standard procedure is to dilute so that the highest concentration target analyte is within linear range. This may cause certain target analytes to become ND at elevated reporting limits. If this is not acceptable, address cost consequences of multiple analytical runs.
8. **Are EDDs required** and defined? _____
   When are they due? _____
9. Is there any **historical data**? _____
   What are the expected concentrations of the samples? _____
10. Any **special reporting format**, e.g. RWQCB? _____
11. **Who will pay** our invoice? If 3rd party, obtain written acceptance by 3rd party. _____
    Credit approval? _____
12. Are **sample containers** required to be provided? _____
    Preserved? _____
13. Are field blanks or **trip blanks** needed? _____
14. Do samples need to be **stored past** our normal **60 days**? _____
    Discuss costs? _____

For all projects, communicate all information to the laboratory via Work Notes or QAPP and verbally via a kickoff meeting.

12/11/09 Revision

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Appendix C

QUOTATION/PROJECT QUESTIONNAIRE

Calscience Environmental Laboratories, Inc.

Appendix C

Calscience Environmental Laboratories, Inc.

Quotation/Project Questionnaire

1. What is the project identification and when is the project expected to commence?
2. What are the methodologies and target analyte lists that will be required?
3. Discuss our normal reporting limits with the client (fax or e-mail a listing) and ensure that they will meet their requirements. If not, determine what their requirements are and if we can reasonably meet them. This information will need to be communicated to the laboratory in written form.
4. How many sample analyses for each sample or matrix will be required?
5. Determine what the required reporting units are? If these differ from our standard, obtain QA approval and communicate it to the laboratory in written form.
6. Determine what the Turn-Around-Time (TAT) will be required. Ensure that the client understands what our definition of TAT is and ascertain if this will be acceptable to them. If not determine their needs and communicate it to the laboratory in written form. Especially for rush TAT, ensure that we clearly understand what is required at what times.
7. Discuss our standard quality control measures and determine if the client has any requirements beyond the normal. If so, these again need to be communicated to the laboratory in written form.
8. Is the use of flags requested or acceptable? If not, our standard procedure is appropriately dilute so that the highest concentration target analyte is within linear range. Note that this may cause our target analysis to become Not Detected (ND) at elevated reporting limits. If this is not acceptable, then address the cost consequences of multiple analytical runs.
9. With regard to TAT, address the cost consequences of client holds and releases with very little holding time remaining. Additionally, ensure that the clients understand that, in this scenario, there is a chance (resulting from capacity issues) that we may be unable to analyze samples released with little or no holding time remaining.
10. Are Electronic Data Deliverables (EDDs) required? If so, what is the desired format? When will they be required?
11. What are the expected concentrations of the samples? Is historical data available?
12. Does the client have any special reporting requirements, e.g., RWQCB or is our normal analytical reporting format acceptable?
13. Who will be responsible for the payment of invoices? If it is a third party other that the samplers or consultant receiving the analytical report, obtain written authorization and acceptance from the third party.
14. Are there any special or state certifications required for the project?
15. Are preserved containers required? If so when and where does the client want them delivered?
16. Are field or trip blanks required? If so, for what analysis?
17. Any subcontracted analysis? Determine that the proposed subcontract laboratory meets all client requirements. Need client approval in writing.
18. Local projects may require coordination with Calscience couriers. Determine the availability and scheduling for ongoing projects.
19. Shipping issues. Out of state or long distance projects may require coordination with shipping schedules. For example, short hold time samples sent Friday for Saturday delivery. Other shipping issues include costs and methods of shipment (e.g., FEDEX, priority overnight, overnight, two-day, GSO, other).

12/11/09 Revision

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Appendix D

REPORT REVIEW CHECKLIST

Calscience Environmental Laboratories, Inc.

Appendix D

Calscience Environmental Laboratories, Inc.

Report Review Checklist

Work Order No: _____

Client: _____ Contact: _____

PROJECT MANAGER REVIEW

A. Project Objectives	Completed
1 Is there a QAPP or sampling and Analysis Plan (SAP) that applies to this Work Order? If so, ensure data set complies with project requirements.	

B. Completeness Check	Completed
2 Are all data packets present for each test requested on the COC form?	
3 Are the cover sheets for each data packet completed with the Group Leader initials/dates?	
4 Is the COC form and Sample Receipt form present and completed/signed?	

C. Data Review	Completed
5 Review cover page. Verify that the following is correct: Client, client contact, Project ID, date samples received.	
6 Reconcile all sample IDs versus COC form.	
7 Verify all testing methods were performed in accordance with the COC or QAPP.	
8 Check sampling dates and sample received dates for each sample versus COC.	
9 For each test method, check sample prep/analysis dates and batch ID nos.	
10 For each test method, check data, reporting limits, reporting units, and dilution values.	
11 Check Method Blanks. Ensure Method Blank is below 1/2 the RL value for DoD (Navy), EM or other projects as required.	
12 Check for J qualifiers and MDL values if required.	
13 Check for other data and QC qualifiers.	
14 Check surrogate recoveries.	
15 Is QA/QC complete and performed at the required frequency?	
16 Check matrix spike (MS/MSD) and laboratory control sample (LCS/LCSD) recoveries.	
17 Process and develop Case Narrative (if required).	

Comments:

_____

_____

_____

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Appendix D

Calscience Environmental Laboratories, Inc.

Report Review Checklist

PROJECT MANAGER REVIEW

D. Does Data Make Sense? — Completed

18 Compare data set with historical data, if any is available. _____

19 Evaluate if entire data set makes sense. _____
For example, compare TPH(g) data with BTEX/MTBE data, compare Specific Conductance data with Total Dissolved Solids (TDS) data, etc.

20 Compare project samples. _____
For example, if samples include a treatment system inlet and system outlet, see if the inlet data exceeds the outlet data.

E. Data Deliverables — Completed

21 What level of data package is required? _____

22 Is a narrative required or necessary? _____

23 Deliver data and note method and date/time on file cover page. _____

Completed by: _____ (Reviewer) **on** _____ (Date)

QA MANAGER REVIEW

F. Supplemental Review — Completed

24 Project Manager review complete. _____

25 Case Narrative complete. _____

26 Supplemental review performed (10% frequency). _____

Completed by: _____ (Reviewer) **on** _____ (Date)

Comments:

_____

_____

_____

_____

_____

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Appendix D

Calscience Environmental Laboratories, Inc.

Report Review Checklist

Work Order No: _____

Client: _____ Contact: _____

QA MANAGER REVIEW – HONEYWELL PROJECT

Tertiary Review by QA Manager or Designee	OK	Not OK
1 Qualitative identification done correctly?		
2 Quantitative accuracy within reporting guidelines?		
3 Calibration (all passing, were done at required frequency, etc.).		
4 QC Samples (required samples performed and are within reporting guidelines).		
5 Method Blank QC criteria.		
6 Adherence to method and process SOPs.		
7 Accuracy of final client reporting forms.		
8 Manual integrations (100% secondary review of manual integrations is required).		
9 Completeness of data package verified.		
10 Special requirements/instructions met?		

Data Review / Verification Checklist
Complete and submit with 10% of data packages

Completed by: _____ (Reviewer) **on** _____ (Date)

Comments:

_____

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Appendix E

STANDARD LEVEL III DELIVERABLE CHECKLISTS

Calscience Environmental Laboratories, Inc.

Appendix E

Standard Level III Deliverable Checklist

GC/MS DATA:

1. **Summary Forms Required:**

- ☐ Sample Result(s)
- ☐ QC - Method Blank, LCS (and LSCD, if analyzed) and MS/MSD for all associated batches
- ☐ Surrogates, including the QC (LCS/D and MS/D)
- ☐ ICAL Summary (RRF / %RSD, or r) for each applicable ICAL, by instrument
- ☐ CCV Summary for all associated CCV's, by instrument and date

2. **ICAL Raw Data Required:**

- ☐ Run log(s) – by instrument and date
- ☐ RF report from the data system, linear/quadratic curve report with 'r', if used
- ☐ ICV Evaluation Report from the data system
- ☐ Raw Data for all calibration levels, from lowest to highest point
- ☐ Raw Data for the ICV standard

3. **Sample & QC Raw Data Required - in run log order, by instrument and date:**

- ☐ Run Log, by instrument and date
- ☐ BFB or DFTPP Tuning Reports from the data system (as applicable)
- ☐ CCV data, include evaluation reports from the data system
- ☐ Method Blank(s)
- ☐ LCS (and LSCD, if analyzed)
- ☐ MS / MSD for all associated batches – include parent sample data
- ☐ Sample data
- ☐ Spectra for all positive hits (including "J" flags, if applicable) following each associated sample quant report and chromatogram (if applicable)

4. **Sample Preparation Logs (if applicable)**

_____	_____	_____
Compiled by - Print name	**Initial**	**Date**

Appendix E

Standard Level III Deliverable Checklist

GC / HPLC / IC DATA:

1. **Summary Forms Required:**

- ☐ Sample Result(s)
- ☐ QC - Method Blank, LCS (LSCD, if analyzed) and MS/MSD for all associated batches
- ☐ Surrogates, including the QC samples (LCS/D and MS/D), if applicable
- ☐ ICAL Summary (RRF / %RSD, or r) or spreadsheet, for each applicable ICAL, by instrument and date
- ☐ ICV Summary for each applicable ICAL, by instrument and date
- ☐ CCV Summary for all associated CCV's, by instrument and date

2. **ICAL Raw Data Required:**

- ☐ Run log(s) – by instrument and date
- ☐ RF report from the data system or linear/quadratic curve report with 'r', if used
- ☐ ICV % D or % Rec. – (hand calculated on ICAL report, summary form or include spreadsheet, if needed)
- ☐ Raw Data for all calibration levels, from lowest to highest point
- ☐ Raw Data for the ICV standard or ICV/ICB as applicable to the method
- ☐ DDT / Endrin Breakdown Standard (8081 only)

3. **Sample & QC Raw Data Required - in run log order, by instrument and date:**

- ☐ Run Log
- ☐ DDT / Endrin Breakdown Standard (8081 only)
- ☐ CCV data or CCV/CCB, as applicable to the method
- ☐ Method Blank for all associated batches
- ☐ LCS (and LSCD, if analyzed) for all associated batches
- ☐ Sample data (primary and confirmation column/detector where applicable)
- ☐ MS / MSD for all associated batches – include parent sample data behind the MS/MSD data

4. **Sample Preparation Logs (if applicable)**

_____	_____	_____
Compiled by - Print name	**Initial**	**Date**

Appendix E

Standard Level III Deliverable Checklist

WET CHEMISTRY DATA:

1. **Summary Forms Required:**

- ☐ Sample Result(s)
- ☐ QC - Method Blank, LCS (LSCD, if analyzed), Sample duplicate or MS/MSD for all associated batches

2. **Sample & QC Raw Data Required - in run log order, by instrument and date:**

- ☐ Run Log
- ☐ ICAL data, as applicable

  Includes balance calibration verification data, pH meter calibration data, etc.
- ☐ Sample data – raw data / data reporting forms
- ☐ CCV data or CCV/CCB, as applicable to the method
- ☐ Method Blank data for all associated batches
- ☐ Sample Duplicate data, as applicable to the method
- ☐ LCS (and LSCD, if analyzed) for all associated batches, as applicable
- ☐ MS / MSD for all associated batches – include parent sample data, as applicable

3. **Sample Preparation Logs (if applicable)**

_____	_____	_____
Compiled by - Print name	**Initial**	**Date**

Appendix E

Standard Level III Deliverable Checklist

METALS DATA:

1. Summary Forms Required:

- ☐ Sample Result(s)
- ☐ QC - Method Blank, LCS (LSCD, if analyzed) and MS/MSD for all associated batches
- ☐ Serial Dilution, PDS/PDSD and MSA (if analyzed)
- ☐ ICAL Summary or spreadsheet (% rec. for ICP or correlation coefficient, r for CVAA) for each ICAL *
- ☐ ICV Summary or spreadsheet (% D or % Rec., as applicable) *
- ☐ CCV Summary for all associated CCV's *

2. Sample & QC Raw Data Required - in run log order, by instrument and date:

- ☐ Run Log
- ☐ ICAL data – include CRDL data as applicable
- ☐ ICV / ICB data
- ☐ ICSA / ICSAB Data (as applicable to the method)
- ☐ Sample data
- ☐ CCV / CCB data
- ☐ Method Blank data for all associated batches
- ☐ LCS (and LSCD, if analyzed) for all associated batches, as applicable
- ☐ MS / MSD for all associated batches – include parent sample data, as applicable
- ☐ Tuning and performance check data for ICP/MS, as applicable

3. Sample Preparation Logs

- ☐ Preparation Logs for metals and mercury, as applicable

_____	_____	_____
Compiled by - Print name	**Initial**	**Date**

* Summaries may not currently be available for the ICAL's, ICV's or CCV's.

Title : **SAMPLE RECEIPT AND LOG-IN PROCEDURES**

Document No. : SOP-T100
Revision No. : 8.3
Supersedes : 8.2

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Revision 8.3 changes are noted in bold italicized typeface and preceded by a "►" marker.

APPROVED FOR RELEASE BY: _____ 02/24/11
MANAGEMENT **DATE**

_____ 02-24-11
QA DEPARTMENT **DATE**

ANNUAL SOP REVIEW

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

1. PURPOSE

1.1. The proper transportation, receipt, log-in, and handling protection, storage, retention and/or disposal of samples post receipt is of paramount importance for obtaining accurate analytical data as well as providing defensible data in litigation, if such ever becomes necessary, and to ensure compliance with the applicable NELAC standard.

1.2. This SOP describes the system for the proper transportation and receipt of samples, their log-in to the Laboratory Information Management System (LIMS), and the disposition of samples to their final disposal or return to client.

2. ►POLICIES

2.1. All samples submitted to Calscience Environmental Laboratories (CEL) are received in Sample Control, the central sample receiving area. Samples are received only by properly trained personnel.

2.2. CEL maintains couriers who are similarly trained as the in-house receipt personnel. They will accept and inspect samples at field pickup locations or the client's office or place of business. They will then transport these samples under custody and proper storage conditions to the laboratory. Additional criteria and guidance for couriers can be found in Appendix ***11.5*** "Courier Procedures".

2.3. The Calscience laboratory building is a secure facility. That is, access is restricted to Calscience personnel or non-Calscience personnel who are escorted by an employee. All entrances into the facility are secured against unapproved entry by Card Key lock, by continuous video monitoring with digital recording and by visual monitoring of access points by personnel. Sample Control is a controlled area within the building where access is restricted to specific personnel.

2.4. Sample Control personnel must be aware of the possible types of contaminants to which they may be exposed in the execution of their duties. Proper attire including gloves, lab coats and safety glasses, at a minimum is required for Sample Control personnel.

3. COMMENTS

3.1. None.

4. HAZARDS AND PRECAUTIONS

4.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

4.2. Exposure to potential hazards should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Calscience's Health, Safety, and Respiratory Protection Manual. In

general, protective eye wear (safety glasses or goggles) and protective apparel (lab coats) and gloves are required to be worn when handling chemicals or samples.

4.3. All samples should be treated as though hazardous unless clearly noted otherwise. Sample coolers should be opened in the roll-in hood and evaluated for hazard prior to lining up on the work benches.

4.3.1. Sample receiving personnel are to wear appropriate gloves whenever handling client samples.

4.4. Do not point the beam from the IR gun at any person, their face or eyes especially, as the beam can cause physical damage.

4.5. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials; chemicals or samples:

4.5.1. A NIOSH approved air-purifying respirator with cartridges appropriate for the chemical handled.

4.5.2. Extended length protective gloves.

4.5.3. Face shield.

4.5.4. Full-length laboratory apron.

4.6. Material Safety Data Sheets (MSDS's) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

5. SAMPLE PRESERVATION

5.1. All sample containers provided to our clientele for sample collection shall include the proper type and amount of chemical preservative, where applicable. Please reference SOP-T064 for Sample Container Preparation.

6. ►SAMPLE RECEIPT AND HANDLING

6.1. Samples are received by the laboratory in the Sample Receiving Department. After receipt, a complete review and reconciliation of the samples and receiving documents is done prior to releasing the samples for analysis.

6.1.1. In general, the samples are considered to be 'received by the laboratory' when the COC and samples are relinquished by the client and the COC has been signed by both parties. After receipt, custody seals are examined and the temperature of the sample/cooler is checked for compliance. Sample containers are removed from their coolers and lined up on a bench. Once lined up they are checked for any broken/leaking or compromised, or otherwise anomalous events related to the containers, volume, preservative, and for any discrepancies with the information the sample labels versus the COC.

6.1.2. After reconciliation with the COC the samples are logged in, labeled and placed in the walk in coolers for storage prior to analysis. Any anomalies or discrepancies are noted and the PM and client are contacted for further instruction.

6.2. Work Order Numbers and Sample Labels

6.2.1. Prior to the assessment of containers, an internal Work Order (WO) number is assigned to the project/samples. This number is recorded on the Chain of Custody (COC) in the upper right portion of the document and on all associated project-related paperwork.

6.2.1.1. Sheets of pre-printed WO numbers for the current month are maintained in sample receiving. When a COC is received a copy is made and the next available pre-printed number/label is applied to the COC copy. The WO number is then written on the original COC, in the boxes provided or in the upper right hand corner if it is a client-specific COC.

6.2.1.1.1. The copy of the COC with the label is then filed in a three ring binder maintained in the sample receiving office. All copies of COC's are maintained in the binder, and when the binder is full the copies are sent to QA for archiving.

6.2.2. Along with the COC, all shipping documentation (FedEx, GSO, or other shipping label/information) is maintained with the project paperwork. The associated WO number is also recorded on these documents.

6.2.2.1. In the event that the shipping documentation is an odd size, torn, wet or otherwise problematic, make a copy of the document(s) and maintain them with the project paperwork.

6.2.3. Once the WO number has been assigned the sample labels can be printed. The number of labels is based upon the total number of samples noted on the COC as well as the total number of containers received.

6.2.3.1. Container labels are generated for each sample and container based upon the WO number of the project and then a series of letters (A, B, C, etc.).

Example: If three (3) VOA vials are received for sample number one on WO 10-01-1000, then the container labels would read as follows: container one: 10-01-1000-1A; container two: 10-01-1000-1B, etc.

6.2.3.2. Attach the labels to the original COC with a paper clip and place in the box near the receiving benches. Once the samples have been reconciled against the COC they will be labeled.

6.3. Receiving Procedure

6.3.1. The receiving and log-in process begins with the examination of the shipping cooler/containers and completion of a Sample Receipt Form and

Sample Anomaly Form, if needed (see Appendix 11.1). The Sample Receipt form is comprised of the following sections:

- Temperature
- Custody Seals Intact
- Sample Condition
- Container Type

6.3.1.1. The Sample Receipt Form is started by putting the date of receipt, client name and work order number on the top of the form in the spaces provided. One form is filled out per cooler received even if multiple coolers are received for one COC.

6.3.1.2. In addition to the Sample Receipt Form, a Sample Anomaly Form is also used to document any anomalies or discrepancies noted when samples are received.

6.3.1.3. Items noted on this form include issues related to sample ID, date and time discrepancies, compromised container problems, insufficient sample volume, holding time violations, preservation issues, volatile headspace issues and COC discrepancies.

6.3.1.4. If a Sample Anomaly Form is required, be sure to write the work order number in the upper right corner, in the boxes provided. Keep this form with the Sample Receipt Form.

6.3.2. Chain of Custody and Sample Receipt:

6.3.2.1. Samples are received at the laboratory by sample receiving technicians trained in the procedures contained in this SOP.

6.3.2.2. Upon receipt, the sample receiving technician checks the COC to make sure all necessary information has been completed: ID's, dates, times, tests, project info, client info, sampler info, etc. If not complete, the technician has the client fill in any missing information.

6.3.2.3. Once the COC is complete, the client relinquishes the COC and associated samples to the laboratory by signing the COC, including current date and time, in the "relinquished by" section. After the client has signed the receiving technician also signs, dates and times the COC in the "received by" section. A copy of the COC is then given to the client.

6.3.2.3.1. If the COC used is the 'triplicate' type then provide the two (2) bottom copies (terms and conditions are on the back of these pages) to the client and keep the original. If the COC is a single page, make a copy for the client and maintain the original.

6.3.3. Custody Seals

6.3.3.1. Often sample coolers are received from commercial carriers and as such arrive sealed with tape. And, often, depending upon the project, the coolers and/or samples will have custody seals. If the samples arrive in a sealed cooler, check all around the outside, near the top of the cooler for a custody seal.

6.3.3.1.1. If there is a custody seal on the cooler, examine it to make sure that the seal has not been compromised (cut or torn) indicating that the cooler may have been opened in the field or during transit.

6.3.3.1.2. If there is no custody seal on the cooler there may be seals on the individual sample containers. If there are custody seals on the containers, examine them to make sure that they have not been broken, indicating that the sample may have been compromised.

6.3.3.1.3. If there are seals, either on the cooler or the sample containers, and there is <u>no</u> evidence of compromise, check the 'cooler' or 'sample' box on the receipt form as applicable. If the seal(s) appear compromised, check the 'No" box as applicable and notify the project manager. If there are no seals present on the cooler or containers, check the "not present" or "N/A" box as applicable.

6.3.3.1.4. Write your initials on the form in the space provided.

6.3.4. Temperature

6.3.4.1. The temperature of the samples upon receipt is of critical importance to data quality and its defense. The temperature acts as a preservation technique, helping to limit biological degradation. Almost all analyses, regulatory agencies, etc. require samples to be maintained within a range of 0.0°C to ≤ 6.0°C.

6.3.4.2. Since determination of the actual temperature of each sample container in a cooler is not practical an assumption is made. Specifically, that the temperature of each sample in a cooler is the same as the temperature of a representative container (temperature blank or actual sample) in the same cooler.

6.3.4.3. Upon receipt, the sample control representative will monitor the temperature of a temperature blank, or representative sample if no blank is provided, by pointing a calibrated IR gun, held approximately 1" from the container, at the sample label and pulling the trigger. The temperature will appear on the display screen on the back of the IR gun.

6.3.4.3.1. Prior to using the IR gun, the technician should check the calibration label on the side of the gun to make sure the calibration due date has not expired. They should also check the battery condition by aiming the IR gun at the wall, pulling the trigger and then looking at the lower left portion of the display screen. If an icon in the shape of a battery appears the 9v battery needs to be replaced. Replace the battery prior to proceeding with temperature measurement.

Note: If at any time, the IR gun fails to operate properly or the battery is low and cannot be replaced, notify the supervisor, remove the IR gun from service and contact QA for the calibrated backup IR gun. Note the IR gun ID and new correction factor on the sample receipt form and continue with the process.

6.3.4.4. Record the temperature on the sample receipt form, apply the listed correction factor and then record the final corrected temperature. Check the "blank" or "sample" box as applicable and write your initials on the form in the space provided.

6.3.4.5. The acceptance criteria for sample receipt temperatures is 0.0°C to ≤ 6.0°C, but not frozen. If the measured temperature is > 6.0°C or if any of the samples are frozen, the temperature shall be recorded and the Project Manager notified for possible corrective action by the client.

6.3.4.6. If the temperature is within guidelines, sample inspection proceeds as normal. If it is out of compliance and there is no temperature blank, a second sample from a different section of the cooler is selected to confirm the temperature.

6.3.4.6.1. If the temperature is also outside criteria for the second sample measured, record the initial temperature on the form and check the 'sample outside temperature criteria' box on the receipt form.

6.3.4.6.2. If the temperature of the second container is within criteria write that temperature on the receipt form and proceed with measuring the temperature of all other containers in the cooler.

6.3.4.6.3. For all containers for which the temperature is outside criteria, record the sample ID's and containers types/analyses on the anomaly form <u>and</u> check the 'sample outside temperature criteria'

box on the form. Notify the PM of the temperature outliers and proceed with the receiving process.

6.3.4.7. In certain circumstances the 0.0°C to ≤ 6.0°C temperature requirement may not apply to the samples.

6.3.4.7.1. These instances are few but ***will include samples in tedlar bags or summa canisters for air analyses, or filter samples***. In these cases check the applicable box in the "Ambient Temperature" section of the receipt form.

6.3.4.7.2. Another example is of those samples that are received on the same day that they are collected in the field. As long as the samples are received by the laboratory with evidence that the cooling process has begun, such as arrival on ice, then the samples are considered acceptable. Record the temperature on the receipt form <u>and</u> check the "samples outside temperature criteria but received on ice/chilled on same day of sampling" box on the receipt form.

6.3.4.8. In all cases, out of compliance temperatures will be conveyed to the PM and then the client for guidance to proceed/not proceed with analysis.

6.3.4.9. The process by which samples are labeled and logged in requires that they be out of refrigerated storage for a period of time. Specifically, the samples must be removed from the cooler, wiped/allowed to dry, and arranged for labeling. This process must be completed as expeditiously as possible to ensure that sample temperatures are not allowed to exceed limits. The following may help ensure that sample temperature limits are not exceeded.

6.3.4.9.1. Do not remove the samples from the cooler until personnel are ready to log-in the project.

6.3.4.9.2. If it appears that the temperature limit will be exceeded, suspend the log-in process, place the samples in refrigerated storage until thermal equilibration is achieved, then remove and continue the log-in process.

6.3.4.9.3. Locate the temperature blank adjacent to the associated samples during the log-in process and occasionally monitor its temperature.

6.3.4.9.3.1. If it appears that the temperature limit will be exceeded, suspend the log-in process, place the samples in

refrigerated storage until thermal equilibration is achieved, then remove and continue the log-in process.

6.3.5. Sample Condition

6.3.5.1. Once the samples have been received, the COC signed and the temperature and custody seals checked the samples are ready for assessment, labeling and log-in. Pull the original COC, sample receipt form and sample labels out of the box and begin the labeling process.

6.3.5.2. The sample containers are removed from the cooler(s) and are lined up on the sample receiving benches. If the containers are shipped within bubble or resealable bags, remove the containers and dispose of the bags.

6.3.5.2.1. If the samples are contained in bags, be sure to look for sample information that may be written on the bags. Often times, a sample collector will write pertinent information on the sample bag and not on the sample labels as should happen. In these instances, the bag must be retained until the individual containers are labeled and a note must be made on an anomaly form. Be sure to leave the associated samples in their bags to eliminate confusion prior to and during labeling.

6.3.5.3. VOA vials are to be placed inside the VOA racks and all other containers can be placed directly on the sample bench, lined up in the same order as the COC. Once the containers are lined up the following checks are made and the sample receipt form is completed.

- COC document(s) received with samples
- COC document(s) received complete
- Samplers name indicated on COC
- Sample container label(s) consistent with COC
- Sample container(s) intact and in good condition
- Correct containers and volume for analyses requested
- Analysis received within holding time
- ***pH/Residual Chlorine/Dissolved Sulfide received within 24 hours***
- Proper preservation noted on COC or sample container
- Volatile analysis container(s) free of headspace*

- Tedlar™ bag(s) free of condensation

NOTE: If any of the above items fail the criteria check, the sample control technician is to complete a sample anomaly form and inform the assigned project manager for rectification.

6.3.5.4. COC Documents Received with Samples.

6.3.5.4.1. Samples should always have an accompanying COC. In the instance that a COC is not provided, leave the samples in their cooler and place the entire cooler in the walk in. Identify/label the cooler as being on hold and contact the PM for instruction. Check "No" on the receipt form and put the paperwork aside with the samples.

6.3.5.5. COC Documents Received Complete

6.3.5.5.1. The COC should be completed in full by the client prior to relinquishing the samples to the laboratory, but often times some of the pertinent information such as the collection date and time, sample matrix, number of containers and analysis needed is missing, or the COC was not relinquished or not relinquished properly.

6.3.5.5.2. If the client drops off the samples then the receiving technician is to make sure the COC is completed prior to signing the "received by" section of the COC.

6.3.5.5.2.1. If the individual dropping off the samples does not know what is needed to complete the COC properly (in essence they are just acting as a courier), have them contact their PM at their office for further information. If this does not rectify the problem call the project PM and have them contact the client.

6.3.5.5.3. The majority of samples are shipped to the laboratory via a commercial carrier. Because of this, it is impossible to make sure the COC is complete prior to receipt.

6.3.5.5.4. If the COC is missing information for one or more of the following items: collection date/time, matrix and/or # of containers, no analysis listed, not relinquished with signature, date or time, proceed with the receiving process but notify the PM about

the COC. They will need to contact the client regarding missing information. Check the "No' box and applicable description box(es) on the receipt form.

6.3.5.5.4.1. If the collection date/time, matrix and/or # of containers is missing and the sample labels contain the missing information, make a copy of the original COC, with WO number assigned, stamp it as being a "copy" and fill in the missing information on the copy based upon the sample label(s).

6.3.5.5.4.2. Maintain the copy of the COC with the original COC as the information noted on the copy will be used during the log-in process.

6.3.5.5.4.3. If you cannot proceed until COC clarification has been completed (no tests or sample ID's listed), be sure to maintain all samples at the appropriate temperature by either leaving them packed in ice within the cooler they arrived in, or by placing the entire cooler in the walk-in refrigerator.

6.3.5.6. Samplers Name Indicated on COC

6.3.5.6.1. Most COC's have a space on them that identifies who the sampler was for the associated samples. Review the COC for this information. It is usually near the top of the COC but may also be incorporated into the relinquished by section of the COC.

6.3.5.6.2. If there is a space for the name and it has been filled in, check the "Yes" box. If it is not completed, check the "No" box. If there is no space on the COC for this information, check the "N/A" box.

6.3.5.7. Sample Container Label(s) Consistent with COC

6.3.5.7.1. Once the COC review is done and the containers have been lined up on the receiving benches, it is time to reconcile the information on the COC against the actual sample labels.

6.3.5.7.2. In the event that the COC is missing information please refer to Section 6.3.4.5.4 for further information.

6.3.5.7.3. If the information on the COC matches the sample labels, check the "Yes" box on the receipt form. If the information does not match the sample label(s) check the "No" box and start a Sample Anomaly Form.

6.3.5.7.3.1. On the anomaly form, check the "sample labels do not match the COC" box and then check one or more of the boxes below that which describe the discrepancy.

6.3.5.7.3.2. In the comments section of the anomaly form list the sample numbers of the affected samples and make any additional comments related to the issue that will help the PM and client understand the discrepancy.

6.3.5.8. Sample Container(s) Intact and in Good Condition

6.3.5.8.1. Evaluate the sample containers for possible compromise, broken, leaking, lids cracked, or the lack of sample label or other identifying information on the container. (Often, field personnel will write sample ID information on the container lid and will not apply an actual label to the container.)

6.3.5.8.2. The laboratory also receives Tedlar bags and Summa canisters for air analyses. These must also be evaluated upon receipt for any signs of leaking or damage.

6.3.5.8.2.1. To check a Tedlar Bag, place the entire bag in the bin of water on the bench behind the receipt window. Hold the bag under the water and check for bubbles, which would indicate a leak. Often these bubbles will appear around the valve of the bag.

6.3.5.8.2.2. If the bag is leaking, ask the client, if they are in the receiving area, or contact the PM who in turn will contact the client, about transferring the sample into a new Tedlar bag.

6.3.5.8.2.3. If transferring is acceptable, notify the Sample Control Supervisor and they will initiate the transfer process. Check the appropriate box on the anomaly to indicate that sample transfer was done.

6.3.5.8.2.4. If there are no leaks, stamp the label of the bag with the "Leak Checked" stamp, initial, date and return to the box or bag in which they arrived and set aside for delivery to the Air lab.

6.3.5.8.2.5. Tedlar bags may also have very little volume upon receipt, basically flat, and may not have enough volume for analysis.

6.3.5.8.2.6. Summa canisters are received, checked in using a barcode system, and sent to the Air department for evaluation and analysis. Please refer to Appendix ***11.6*** for instructions on how the barcode system operates.

6.3.5.8.3. If the containers are all intact and all are labeled with the required information, check the "yes" box. If any problems are identified, check the "No" box and start a sample anomaly form.

6.3.5.8.3.1. On the anomaly form, check the "sample containers compromised" box and then check one or more of the boxes below that which describe the discrepancy.

6.3.5.8.3.2. In the comments section of the anomaly form list the sample numbers and tests of the affected samples and make any additional comments related to the issue that will help the PM and client understand the discrepancy.

6.3.5.8.3.3. If a Tedlar bag is flat, low in volume, or compromised/leaking, check the "Air sample containers compromised" box and then the applicable descriptive box below that.

6.3.5.9. Correct Containers and Volume for Analyses Requested

6.3.5.9.1. Evaluate the samples for appropriate containers and sample volume based upon the tests requested on the COC. Refer to the sample container and holding time table in Appendix 11.2. Tables related to containers, preservatives, volumes and holding times are also posted in the sample receiving area for reference.

6.3.5.9.2. If containers and volumes are sufficient for the analyses requested, check the "Yes" box on the receipt form.

6.3.5.9.3. If an inappropriate container or insufficient sample volume is received for a sample, check the "No" box on the receipt form and start an anomaly form.

6.3.5.9.3.1. On the anomaly form, check the "improper container/preservative used" box or the "insufficient quantities for analysis" box as applicable.

6.3.5.9.3.2. In the comments section of the anomaly form list the sample numbers and tests of the affected samples and make any additional comments related to the issue that will help the PM and client understand the discrepancy.

6.3.5.10. Analysis Received Within Holding Time

6.3.5.10.1. When reviewing the COC and sample label information for the analyses requested, be sure to look at the collection date and time for each sample. Several tests have what is considered to be a "short" holding time (anything less than 72 hours).

6.3.5.10.2. Refer to the sample container and holding time table in Appendix 11.2. Tables related to containers, preservatives, volumes and holding times are also posted in the sample receiving area for reference. Short hold samples are also noted on a separate table, specific to the short hold methods.

6.3.5.10.3. Tests such as BOD, nitrate, nitrite and o-phosphate by 300.0, chrome VI in water, EPA 5035-Encores®,

and many others must be handled quickly in order for the laboratory to meet the holding time.

6.3.5.10.3.1. Once reconciled against the COC and labeled, short hold samples can be given to the labs for analysis. Make a copy of the COC and highlight the short hold tests. Take the copy of the COC to the related department (group leader or designee) to let them know we have received the samples.

6.3.5.10.3.2. Once labeled, place the short hold containers on the cart marked "Short Hold and Rush Samples" in the receiving area. Sometimes, if there are just one or two containers, and they have been labeled, they can be taken to the department along with a copy of the COC in order to speed things along.

6.3.5.10.4. If all the samples are received within holding time check the "Yes" box on the receipt form.

6.3.5.10.5. If there are holding time violations, check the "No" box on the receipt form. Start a sample anomaly form and check the "Holding time expired" box. In the comments section of the anomaly form list the sample numbers and tests of the affected samples and make any additional comments related to the issue that will help the PM and client understand the discrepancy.

6.3.5.11. ***pH/Residual Chlorine/Dissolved Sulfide Received within 24 Hours***

6.3.5.11.1. ***Certain tests are, by method, to be performed in the field at the time of collection. These tests are noted in the holding time tables as needing to be analyzed "ASAP" or within 15 minutes from collection. Because this is often not possible for the client, the laboratory considers the samples to be acceptable if received within 24 hours from the date and time of collection.***

6.3.5.11.2. ***These samples will be handled as other short hold tests. Refer to section 6.3.5.10.3 through 6.3.5.10.5 for handling information.***

6.3.5.12. Proper Preservation Noted on COC or Sample Container

6.3.5.12.1. Check all aqueous containers for a sticker, preservative label or notation on the sample label that indicates that the sample container has been properly preserved for the analysis requested. Also review the COC for preservation information.

6.3.5.12.1.1. Refer to the sample container and holding time table in Appendix 11.2. Tables related to containers, preservatives, volumes and holding times are also posted in the receiving area for reference.

Note: Sample Control personnel are required to note that the proper preservation is documented as "present" but shall not breach sample integrity to verify its presence. Verification of required preservation is accomplished at the bench level during analytical processing.

6.3.5.12.2. If evidence of preservation is available, and it is the proper preservative for the analysis requested, check the "Yes" box on the receipt form.

6.3.5.12.3. If soil samples are all that were received, check the "N/A" box on the receipt form. Soil or solid samples are not 'preserved' except by temperature. If the temperature was within criteria then the soil/solid samples are preserved correctly.

6.3.5.12.4. If there is no evidence of preservation and the analysis requires a preserved container, check the "No" box of the receipt form and start an anomaly form. Check the "No preservative noted on the COC or label" box on the anomaly form and list the sample ID's and tests in the comments section of the form, include any additional comments that will help the PM and client understand the discrepancy.

6.3.5.12.4.1. There are analyses that may or may not need preservation depending on what the sample is being analyzed for. Examples of this include the metals analyses. Aqueous metals containers can be preserved when "Total Metals" is being analyzed and

will be unpreserved when "Dissolved Metals" is being analyzed. In both cases the preservation is correct.

6.3.5.12.4.2. Another example is aqueous VOC analyses (EPA 8260, RSK-175 and EPA 8015-GRO) that may be collected in unpreserved vials. In these cases, the unpreserved vials are acceptable. Check the "Yes" box on the receipt form and also the "Unpreserved vials received for volatiles analysis" box. This is done to assist selection of the proper testcode during the log-in process.

6.3.5.12.4.3. Also, confirm the holding time requirements for unpreserved analyses, there is a difference, usually 14 days for preserved versus 7 days for unpreserved.

6.3.5.12.4.4. If unpreserved vials are received they are placed in small resealable bags, by sample. A group of sample bags is then placed inside a larger 4 mil resealable bag, by test. Write the WO# on the outside of the large bag using a red Sharpie and identify the department: i.e.: VOA, LUFT G, or RSK.

6.3.5.13. Volatile Analysis Container(s) Free of Headspace

6.3.5.13.1. Containers for volatiles analyses are required to have <u>no</u> headspace upon receipt. Headspace is defined as anything larger than a "Macro*" bubble.

* Note: The presence of a macro bubble in a sample vial generally indicates either improper sampling technique or a source of gas evolution within the sample. The latter case is usually accompanied by a buildup of pressure within the vial. (e.g., carbonate containing samples preserved with acid).

The EPA has conducted studies that indicate "pea-sized bubbles" (i.e., bubbles not exceeding ¼ inch or 6mm in diameter) did not adversely affect volatiles data. These bubbles were generally encountered in wastewater samples, which are

more susceptible to variations in gas solubility than are groundwater samples.

6.3.5.13.2. To check for headspace, invert the containers for volatiles analyses (usually the VOA vials) and look for bubbles. If there is a bubble and it is larger than 6mm (slightly larger than the end of a "Papermate®" pen) or there is a gap between the liquid level and the bottom of the vial, check the "No" box on the receipt form. Start an anomaly form and list the sample #, container ID (A, B, C, etc) and the total number of vials received for that sample and test in the "Headspace" section of the form.

6.3.5.13.3. Use a red Sharpie and mark an "X" on the sample label of all VOA vials that have headspace. This is done to differentiate the acceptable VOA samples from those with headspace.

6.3.5.13.3.1. Sometimes all VOA's for a sample, or even a project, will have headspace. It is up to the client to decide whether the laboratory will proceed with analysis on these samples/containers.

6.3.5.14. Tedlar™ bag(s) Free of Condensation

6.3.5.14.1. Check Tedlar bags for any evidence of condensation inside the bag. If none is present check the "Yes" box on the receipt form. If there is evidence of condensation/liquid inside the bag, check the "No" box.

6.3.6. Container Type

6.3.6.1. For each type of container received per sample check the appropriate box. In the event that a container is received that is not listed, check the blank box and write in the container type.

6.3.6.1.1. Samples may also be received in plastic bags or other containment devices. In these instances, confirm with the PM that the container is acceptable for the project or test. If not, start an anomaly form.

6.3.6.2. If Trip Blank vials are received, the laboratory prepared them and the Calscience Trip Blank Lot # is visible, check the Trip Blank Lot # box and write in the Lot # on the form.

6.4. Sample Labeling

6.4.1. After the evaluation is complete, the samples are labeled using the pre-printed labels generated in Section 6.2.3.

6.4.1.1. In the event that there are labels left over, or there are not enough labels, re-check the samples and containers against the COC. If no discrepancies are noted, dispose of the additional labels or print extras as needed. If a discrepancy is found, start an anomaly form.

6.5. Completion of the Receiving Process

6.5.1. After the evaluation is completed, the sample containers have all been labeled and the paperwork filled out, initial the "Checked by" box on the receipt form. Initial and date the anomaly form if one was completed, and request a secondary review of the samples, COC and labeling.

6.5.2. The secondary reviewer is to check the sample ID's, labels, etc against the COC. If all is correct, the reviewer will sign the receipt form in the "Reviewed by" section.

6.5.3. The original copy of the COC, receipt form, anomaly form (if used) and any shipping paperwork (if available) is scanned into the LIMS system. Whoever scans the documents must initial the receipt form in the "Scanned by" section prior to scanning the documents.

6.5.4. After scanning, all original paperwork is placed in the log-in box in the log-in room where the information will be entered into the LIMS system.

6.5.5. Sample containers are then placed in appropriate storage.

6.5.5.1. VOA vials are placed in small resealable bags, by sample. A group of sample bags is then placed inside a larger 4 mil resealable bag, by test. Write the WO# on the outside of the large bag with a black Sharpie and identify the analytical department: i.e.: VOA, LUFT G, or RSK.

6.5.5.2. All other containers are placed in storage crates or in transfer coolers if being transferred to the Lampson facility. The bags, crates and coolers are placed in appropriate storage or sent immediately for analysis if short holds/quick turns.

6.6. Sample Storage

6.6.1. Calscience has eleven separate cold-temperature sample storage areas located within Sample Control and the Lampson building.

- Refrigerators for non-volatile solid waste and waste water samples (two)
- Refrigerator for water samples for VOC analyses (three)
- Refrigerator for solid samples for VOC analyses (one)
- Refrigerator for high concentration VOC samples (one)
- Freezers for sediments/marine tissue samples (four)

6.6.1.1. Each cold-temperature sample storage area is specifically designated only for its sample type. Samples are never placed in a refrigerated area not designated for its sample type. Air samples in either Summa® Canisters or Tedlar® Bags are stored at ambient temperature until they are transferred to the Air lab in the Lampson facility.

6.6.2. Refrigerators are monitored for volatile analyte contamination by reagent water holding blanks that are tested and reviewed every two weeks. Refer to Section 7 for further information on holding blanks.

6.6.3. Refrigerators and freezers are monitored for temperature variations using Min/Max calibrated electronic thermometers. During working days the temperatures are noted each day on temperature sheets affixed to each refrigerator or freezer.

6.6.3.1. On days where no personnel are present, the electronic memory is used to monitor the temperature. The minimum and maximum temperatures are noted on the next working day.

6.6.3.2. Refer to SOP-T043, Support Equipment: Calibration, Verification, Monitoring for further information on routine temperature monitoring procedures.

6.6.4. Samples that the client indicates to have high VOCs will be tagged at sample receipt with blue tape and stored in the appropriate refrigerator.

6.6.5. Samples analyzed which are determined to have high VOCs will be tagged and stored in the appropriate refrigerator.

6.6.6. Occasionally solid (soil, paint chip, concrete, metal fines, etc.) samples are submitted to the laboratory in small resealable bags. Because of their size these may be difficult to locate in the storage refrigerators. As such, the bags of samples weighing less than approximately 200 grams must be discretely identified with the laboratory sample identification. All similar samples from the subject work order must then be placed, sample in bag, into a jar labeled with the work order, and stored in the appropriate refrigeration unit.

6.7. Special Sample Requirements

6.7.1. Internal Chain of Custody (Laboratory)

6.7.1.1. An internal COC is required when samples are being transferred to the Lampson building. Samples are couriered to the facility in a Calscience vehicle by a Calscience employee trained in the procedure. These transfers take place every hour, or more frequently if rush samples are involved.

6.7.1.1.1. There are three Internal COC logbooks, designated as Solid Aliquot, Water Aliquot and Air Samples. (As with all logbooks, these must be reviewed at least monthly by a sample control representative

and that review is to be documented in the front of the logbook.)

6.7.1.2. Samples that must be transferred to the Lampson facility are, after assessment and log-in, placed on ice in coolers and held in the walk-in until ready for transport. A temperature blank is added to the cooler prior to transport for monitoring the temperature when the samples arrive at the Lampson facility.

6.7.1.3. When ready for transport the Workorder numbers/sample ID's are recorded in the associated logbook. Also recorded are the initials of the transferor and the date and time that the sample transfer is completed.

6.7.1.4. When received at the secondary facility, the receiver of the samples will sign for the samples and the temperature will be checked using a calibrated IR gun. This temperature is recorded in the ICOC logbook for all associated samples being transferred.

6.7.1.4.1. The temperature is monitored by measuring the temperature blank with a calibrated IR gun. Aim the IR gun at the temperature blank label, holding the gun approximately 1" from the container, and pull the trigger.

6.7.2. Internal Chain of Custody (Client)

6.7.2.1. A Project-Specific Internal Chain of Custody (ICOC) is used when a client's Quality Assurance Plan requires it. The ICOC is maintained in a QA issued binder and is used by the sample custodian and the analysts to track all activities related to the sample and its movement throughout the laboratory.

6.7.2.2. Upon log-in, a special label noting "ICOC Required" is affixed to the sample container.

6.7.2.3. When a container labeled with "ICOC Required" is retrieved from the sample cooler, the analyst records the container type, initials and date in the ICOC logbook. If the entire sample is to be used in the analysis, the analyst documents this on the ICOC. If there is sample remaining after analysis, it is placed back in the refrigerator, and the Analyst signs the sample back in. When the remaining sample is disposed of, the Sample Disposal Technician documents this on the ICOC.

6.7.2.4. The original ICOC is maintained in the QA issued binder and archived when all pages in the binder have been filled in. A copy of the related ICOC is made for the project file.

6.7.3. ***Legal / Evidentiary Chain of Custody***

6.7.3.1. ***If samples are identified for legal/evidentiary purposes on the COC, the sample receiving personnel will initiate the Internal Chain of Custody (ICOC) procedures noted in Section 6.7.2.***

6.7.4. Client-Specific Inter-Facility Transport Coolers

6.7.4.1. In order to limit the chance for cross-contamination between differing client samples, a client specific cooler is designated for sample transfer between buildings.

6.7.4.2. The coolers are labeled with the specific client name and contain ice and a temperature blank. Only client specific samples shall be placed in these coolers and only right before transfer to the other building.

6.7.4.3. When received at the secondary facility, the receiver of the samples will sign for the samples and check the temperature using a calibrated IR gun. This temperature is recorded in the ICOC logbook for all associated samples being transferred.

6.7.5. Samples Imported from Outside the Continental United States

6.7.5.1. Under the current PERMIT TO IMPORT SOIL from the United States Department of Agriculture (USDA) The following conditions/ procedures must be followed:

6.7.5.1.1. All shipments must have the "Soil Samples - Restricted Entry" label on the cooler/package.

6.7.5.1.2. A copy of the Calscience USDA Permit to Import Soil shall be included with the shipping papers or bill of lading.

6.7.5.1.3. Samples will be labeled with special stickers upon receipt in Sample Control to show restricted status / specialized disposal.

6.7.5.1.4. All samples must be sterilized and recorded as being treated prior to disposal.

6.7.5.1.5. No further distribution or sub-contracting of samples is allowed. (Have client send directly to sub lab using sub lab's permit to Import Soil.)

6.7.5.1.6. Upon receipt of samples, the Orange County Agricultural Commissioner's Office (OCACO) shall be notified.

6.7.5.1.7. Sample Control will submit copies of the COC's to the QA department to be faxed to the OCACO.

6.8. ***Sample Cooler Cleaning and Preparation***

6.8.1. ***Following the sample receiving process, inspect the cooler, inside and out, for any evidence of spills or staining which might indicate that the cooler has become contaminated and may no longer be suitable for use. Coolers that show evidence of contamination should be washed prior to continued use for bottleware or field samples.***

6.8.1.1. ***For coolers where contamination is clearly evident and cleaning the cooler will not reduce the potential for future contamination of clean bottleware and/or other field samples, notify the sample control supervisor and remove the cooler from service.***

6.8.1.2. ***If the cooler can be cleaned and all traces of contamination removed, the cooler may be placed back into service. The sample control supervisor or bottle preparation group will make this decision.***

6.8.2. ***Using a phosphate-free soap such as Alconox and a sturdy brush or scrubbing-type sponge, thoroughly clean the inside of the cooler. If needed, scrub the outside of the cooler as well.***

6.8.3. ***Follow with sufficient water to rinse away all traces of the soap.***

6.8.4. ***Dry the cooler with paper towels or allow to air dry.***

6.8.4.1. ***If, after cleaning the cooler, it remains unusable due to evident contamination, remove the cooler from service.***

6.9. ***Multiphasic Sample Handling***

6.9.1. ***Samples that have two or more layers are called biphasic or multiphasic. Depending upon client or project specific requirements, all phases may be analyzed or just a single phase.***

6.9.2. ***In the event that a multiphasic sample is received, notify the PM for log-in and additional handling instructions.***

6.9.3. ***Independent of whether one, or all phases, will be analyzed, sample control technicians will not separate the phases. This process will be carried out by the appropriate laboratory department based upon the handling instructions given by the PM. Refer directly to the appropriate extraction or analytical SOP for further information.***

7. HOLDING BLANKS

7.1. Bi-weekly monitoring of all refrigerators that hold samples is done through the use of holding blanks. These blanks are prepared by sample control personnel as follows:

7.1.1. For each refrigerator used to store analytical samples, two HCl preserved VOA vials are filled completely, no headspace, with NANOpure water and capped. Once capped they are placed in a VOA holder and then put inside the refrigerators that are to be monitored.

7.1.1.1. The refrigerators used for the monitoring of samples are: SC01, SC02, SC05, FG12 in the Lincoln facility and OFG1, OFG2, and OFG3 in the Lampson facility.

7.1.2. The prepared vials are labeled with the associated refrigerator ID, date and time the holding blanks were placed in the refrigerator, the analysis required (8260), and the preservative. A space is left on the label to record the date and time that the holding blanks are removed from the refrigerator and sent for log in and analysis.

7.1.2.1. Two additional vials are prepared at the same time as the holding blank vials. These two vials are treated as source blanks and are stored in the source blank refrigerator used for trip blanks and located in the bottle prep area.

7.1.3. Every 14 days the existing holding blanks are removed from the refrigerators and are replaced with a new set of holding blanks. Once removed, the date and time of removal is recorded on the vial labels.

7.1.4. A COC is completed that lists all of the holding blank sets by refrigerator ID, date and time removed, matrix, number of vials per set and the test needed. The client noted on the COC is Calscience and the QA department acts as the PM for the final report.

7.1.5. Holding Blanks are analyzed for the full list of VOC's by EPA 8260 and the results are reported down to the MDL. If any hits are noted in the initial run, the lab is to analyze the second vial. If the initial hit(s) is confirmed at or above the Reporting Level (RL) the source blank vial is then analyzed, if positives are noted over several refrigerators.

7.1.5.1. If a hit at or above the RL is confirmed as coming from within the refrigerator and not the source water and/or vials, all samples analyzed within the period that the holding blanks were placed in the refrigerator are to be reviewed for the positive analyte(s). If the results have not been sent to the client, the client is contacted by the PM, any associated positive results are flagged and the situation is addressed in the report narrative.

7.1.5.2. If the report has already been issued, the client is informed of the contamination in the refrigerator and the possibility that their results may be impacted. The client is then advised to submit another sample for analysis. If re-sampling and analysis cannot be done, a revised report flagging the data and addressing the issue in the narrative is sent to the client.

7.1.6. The current holding blanks in the contaminated refrigerator are immediately analyzed to check if the contamination is still present.

7.1.7. If contamination is found above the RL a second time, the refrigerator will be taken out of service, if possible, and no additional samples will be placed in the fridge until it is confirmed to be contaminate-free.

7.1.7.1. Samples in the refrigerator may be moved to other refrigerated storage, disposed of if sufficient time has passed, or returned to the client.

7.1.8. The contaminated refrigerator, if free standing (FG12), is cleaned out with Alconox and then allowed to air dry. A vapor suppressor (activated carbon) is also placed in the refrigerator to add in the decontamination process.

7.1.8.1. After the refrigerator has been cleaned and dried, 2 sets of holding blanks are placed in the fridge to check for any residual contamination. After 24 hours the first vial is analyzed, if it comes back clean, no additional analysis is required. If positive above the MDL, the second vial is analyzed. If the both vials come back with indications of further contamination, the entire cleaning process is repeated.

7.1.9. Walk-in refrigerators may be subject to other decontamination procedures as necessary.

7.1.10. Once the refrigerator is deemed to be contaminate-free, it may be put back into service.

8. PROJECT FILE GENERATION

8.1. Upon Calscience's acceptance of samples, a project file is generated as follows:

8.1.1. Using the pre-assigned unique work order number in YY-MM-NNNN format, enter project and sample data into the LIMS log-in program.

8.1.1.1. In the YY-MM-NNNN work order number format YY is the year, e.g., "07" = 2007, MM is the month, e.g., "06" = June, and NNNN is a four digit sequential number which represents the number of projects received in a month to date, e.g., "0125" represents the 125th project received in a particular month.

8.1.1.2. The work order number will be retained throughout the life of the sample in the laboratory. This identifier is used for all samples, sub-samples and subsequent extracts and/or digestates.

8.1.2. Once all information has been entered into the LIMS system a series of information sheets are printed and these include a Project Status Sheet, Project Work Sheet, Sample Summary Report, and a Chemist Work Sheet for each designated department, as needed.

8.1.3. Build the project file using a folder color specific to the applicable Project Manager.

8.1.3.1. Into the project file, place the original COC, Sample Receipt form, Sample Anomaly Form (if used), any shipping documentation received with the samples, a copy of the Project Work Sheet, Sample Summary Sheet, and Sample Receipt Form.

8.1.4. A copy of the Project Status Sheet is attached to the front of the project folder and is completed at log-in.

8.1.5. For all rush and short holding time issues, a copy of the COC is distributed to the appropriate group; otherwise, the Chemist Worksheet is printed automatically to each group's network printer. The Chemist Worksheets are transmitted to the group's printer at the time the work order is committed to the LIMS.

8.1.6. The project file folder is routed to the assigned Project Manager for review. Any changes must be noted via e-mail to the e-mail box "Change Orders".

8.1.6.1. Project Managers or their assistants are to review the log-in for accuracy based upon client and/or project specific-needs. This review is documented by initialing the upper right hand corner of the Project Work Sheet.

8.1.7. It is important to note that "ANALYTICAL TESTING, THE GENERATION OF ANALYTICAL REPORTS, AND SAMPLE DISPOSAL RECORDS, AS WELL AS MANY OTHER AUTOMATED FUNCTIONS RESULT FROM DATA ENTERED DURING THE LOG-IN PROCESS. IT MUST BE CORRECT".

8.1.8. Disposal of samples is covered under SOP-T005 and SOP-T061.

9. RESPONSIBILITIES

9.1. The Operations Manager maintains overall responsibility for sample receipt / log-in operations at Calscience.

9.2. The Sample Control Group Leader is responsible for the day-to-day operations relating to sample receipt, log-in, and related operations. The Sample Control Group Leader reports directly to the Operations Manager.

10. REFERENCES

10.1. ►*Organic Analytes*, ***Test Methods for Evaluating Solid Waste*** (SW-846), Third Edition, Volume 1, Chapter Four, ***USEPA, Revision 4, February 2007***.

11. ►APPENDICES

11.1. Sample Receipt Form / Sample Anomaly Form.

11.2. Sample Preservation and Holding Time Guide.

11.3. ***Miscellaneous Methods and Subcontract Information.***

11.4. Sample Entry into LIMS Procedure.

11.5. Courier Procedure.

11.6. Summa Canister Check-In Procedure.

11.7. Northern California Remote Sample Shipping Procedure.

11.8. ***Example of GSO Shipping Label.***

11.9. ***GSO Sacramento Distribution Center Local Area Map.***

11.10. ***GSO Oakland Distribution Center Local Area Map.***

11.11. ***GSO San Jose Distribution Center Local Area Map.***

Appendix 11.1

SAMPLE RECEIPT FORM / SAMPLE ANOMALY FORM

Calscience Environmental Laboratories, Inc.

WORK ORDER #: ☐☐-☐☐-☐☐☐☐

SAMPLE RECEIPT FORM

Cooler ___ of ___

CLIENT: _____ DATE: ___/___/___

TEMPERATURE: **Thermometer ID: SC1 (Criteria: 0.0°C – 6.0°C, not frozen)**

Temperature _____.___ °C + 0.5°C (CF) = _____.___ °C ☐ **Blank** ☐ **Sample**

☐ Sample(s) outside temperature criteria (PM/APM contacted by: _____).

☐ Sample(s) outside temperature criteria but received on ice/chilled on same day of sampling.

☐ **Received at ambient temperature, placed on ice for transport by Courier.**

Ambient Temperature: ☐ Air ☐ Filter **Initial:** _____

CUSTODY SEALS INTACT:

☐ Cooler ☐_____ ☐ No (Not Intact) ☐ Not Present ☐ N/A **Initial:** _____

☐ Sample ☐_____ ☐ No (Not Intact) ☐ Not Present **Initial:** _____

SAMPLE CONDITION:	Yes	No	N/A
Chain-Of-Custody (COC) document(s) received with samples	☐	☐	☐
COC document(s) received complete	☐	☐	☐
☐ Collection date/time, matrix, and/or # of containers logged in based on sample labels.			
☐ No analysis requested. ☐ Not relinquished. ☐ No date/time relinquished.			
Sampler's name indicated on COC	☐	☐	☐
Sample container label(s) consistent with COC	☐	☐	☐
Sample container(s) intact and good condition	☐	☐	☐
Proper containers and sufficient volume for analyses requested	☐	☐	☐
Analyses received within holding time	☐	☐	☐
pH / Residual Chlorine / Dissolved Sulfide received within 24 hours	☐	☐	☐
Proper preservation noted on COC or sample container	☐	☐	☐
☐ Unpreserved vials received for Volatiles analysis			
Volatile analysis container(s) free of headspace	☐	☐	☐
Tedlar bag(s) free of condensation	☐	☐	☐

CONTAINER TYPE:

Solid: ☐4ozCGJ ☐8ozCGJ ☐16ozCGJ ☐Sleeve (_____) ☐EnCores® ☐TerraCores® ☐_____

Water: ☐VOA ☐VOAh ☐VOAna_2 ☐125AGB ☐125AGBh ☐125AGBp ☐1AGB ☐1AGBna_2 ☐1AGBs ☐500AGB ☐500AGJ ☐500AGJs ☐250AGB ☐250CGB ☐250CGBs ☐1PB ☐500PB ☐500PBna ☐250PB ☐250PBn ☐125PB ☐125PBznna ☐100PJ ☐100PJna_2 ☐_____ ☐_____ ☐_____

Air: ☐Tedlar® ☐Summa® **Other:** ☐_____ **Trip Blank Lot#:**_____ **Labeled/Checked by:** _____

Container: **C:** Clear **A:** Amber **P:** Plastic **G:** Glass **J:** Jar **B:** Bottle **Z:** Ziploc/Resealable Bag **E:** Envelope **Reviewed by:** _____

Preservative: **h:** HCL **n:** HNO_3 **na_2:** $Na_2S_2O_3$ **na:** NaOH **p:** H_3PO_4 **s:** H_2SO_4 **znna:** $ZnAc_2$+NaOH **f:** Field-filtered **Scanned by:** _____

SOP T100_090 (09/13/10)

Calscience Environmental Laboratories, Inc.

WORK ORDER #: ☐☐-☐☐-☐☐☐☐

SAMPLE ANOMALY FORM

SAMPLES - CONTAINERS & LABELS: **Comments:**

- ☐ **Sample(s)/Container(s) NOT RECEIVED but listed on COC** _____
- ☐ **Sample(s)/Container(s) received but NOT LISTED on COC** _____
- ☐ **Holding time expired** – list sample ID(s) and test _____
- ☐ **Insufficient quantities for analysis** – list test _____
- ☐ **Improper container(s) used** – list test _____
- ☐ **Improper preservative used** – list test _____
- ☐ **No preservative noted on COC or label** – list test & notify lab _____
- ☐ **Sample labels illegible** – note test/container type _____
- ☐ **Sample label(s) do not match COC** – Note in comments _____
  - ☐ **Sample ID** _____
  - ☐ **Date and/or Time Collected** _____
  - ☐ **Project Information** _____
  - ☐ **# of Container(s)** _____
  - ☐ **Analysis** _____
- ☐ **Sample container(s) compromised** – Note in comments _____
  - ☐ **Water present in sample container** _____
  - ☐ **Broken** _____
- ☐ **Sample container(s) not labeled** _____
- ☐ **Air sample container(s) compromised** – Note in comments _____
  - ☐ **Flat** _____
  - ☐ **Very low in volume** _____
  - ☐ **Leaking (Not transferred - duplicate bag submitted)** _____
  - ☐ **Leaking (transferred into Calscience Tedlar® Bag*)** _____
  - ☐ **Leaking (transferred into Client's Tedlar® Bag*)** _____
- ☐ **Other:** _____ _____

HEADSPACE – Containers with Bubble > 6mm or ¼ inch:

Sample #	Container ID(s)	# of Vials Received	Sample #	Container ID(s)	# of Vials Received	Sample #	Container ID(s)	# of Cont. received	Analysis

Comments: _____

*Transferred at Client's request.

Initial / Date: _____ / / ____

SOP T100_090 (09/17/10)

Appendix 11.2

SAMPLE PRESERVATION AND HOLDING TIME GUIDE

Holding Times and Containers for Water/Aqueous Samples					
Tests	Method	Holding Time (days)	Min Volume (ml)	Container	Preservation
1,2,3-TCP	SRL-524M-TCP	14	40	VOA	HCl / No Headspace
1,4-Dioxane	GC/MS Isotope Dilution	7	1000	AGB	$Na_2S_2O_3$ or None
1,4-Dioxane	SRL-524M-TCP	14	40	VOA	HCl / No Headspace
Alkalinity (Total)	SM 2320 B	14	50	PB/GB	None
Alkalinity (Bicarbonate, HCO_3)	SM 2320 B	14			None
Alkalinity (Carbonate, CO_3)	SM 2320 B	14			None
Alkalinity (Hydroxide, OH)	SM 2320 B	14			None
Ammonia-Nitrogen (NH_3)	SM 4500 NH3 B/E / 350.1	28	500	GB	H_2SO_4
Bicarbonate (HCO_3)	SM 2320 B	14	50	PB/GB	None
Bioassay (96-Hr Aq Tox, Haz Waste)	Dept. Fish & Game	7	50	PB/GB	None
Bioassay (96-Hr Aq Tox, Effluent)	EPA 600/4-85/013	36 hours	5 gallons	LDPE	None
BTEX/MTBE	8021B	14	40	VOA	HCl / No Headspace
Biochemical Oxygen Demand (BOD)	SM 5210 B	48 hours	300	GB/PB	None
BNAs (Base, Neutral, and Acids)	EPA 8270C/625	7	1000	AGB	$Na_2S_2O_3$ or None
Bromide (Br)	EPA 300.0	28	20	PB/GB	None
Carbon Dioxide or CO_2 (RSK 175)	RSK 175 (M)	7	40	VOA	None / No Headspace
Carbon Dioxide or CO_2 (SM 4500)	SM 4500 CO2D	24 hours	100	AGB	None / No Headspace
Carbonate (CO_3)	SM 2320 B	14	50	PB/GB	None
Chemical Oxygen Demand (COD)	SM 5220 D	28	20	GB	H_2SO_4
Chloride (Cl)	EPA 300.0/SM 4500 Cl	28	50	PB/GB	None
Chlorine, Total Residual (TRC)	SM 4500 Cl	ASAP (24 hours)	100	PB/GB	None
Chromium VI (Cr-6 / Hexavalent Chromium)	EPA 7196A/7199/218.6	24 hours	50	PB/GB	None
CO_2 (Carbon Dioxide)	SM 4500 CO2D	24 hours	100	AGB	None / No Headspace
CO_3 (Carbonate)	SM 2320 B	14	50	PB/GB	None
Cr-6 (Chromium VI / Hexavalent Chromium)	EPA 7196A/7199/218.6	24 hours	100	PB/GB	None
Cyanide, Amenable	SM 4500 CN G	14	200	PB/GB	NaOH
Cyanide, Total	SM4500 CN E	14	200	PB/GB	NaOH
Dissolved Oxygen (DO)	SM 4500 O G	24 hours	300	AGB	None / No Headspace
Dissolved Organic Carbon (DOC) (Lab Filtered)	SM 5310 D	24 hours	40	PB/GB	None
Dissolved Organic Carbon (DOC) (Field Filtered)	SM 5310 D	28	40	GB/VOA	H_2SO_4 or HCL
DRO (Diesel Range Organics)	EPA 8015B	7	500	AGB	None
EDB/DBCP	EPA 504.1	14	40	VOA	$Na_2S_2O_3$ / No Headspace
Ethanol/Methanol	EPA 8015B	7	40	VOA	None
Ferrous Iron (Ferrous Fe or Fe^{2+})	SM 3500 Fe B	24 hours	50	GB	None / No Headspace
Fluoride (Fl)	SM 4500 F C	28	100	PB/GB	None
Formaldehyde	ASTM D6303-98	24 hours	150	AGB	None
Hardness, Total	SM 2340 C/ SM 3500 Ca B	180	100	PB/GB	HNO_3
HCO_3 (Bicarbonate)	SM 2320 B	14	50	PB/GB	None
Herbicides, Chlorinated	EPA 8151A	7	1000	AGB	$Na_2S_2O_3$ or None
Hexane Ext. Material (HEM/SGT-HEM)	EPA 1664A	28	1000	GB	H_2SO_4 or HCL
Hexavalent Chromium (Hex Cr/Cr-6)	EPA 7196A / 7199 / 218.6	24 hours	50	PB/GB	None
Hydrazine	ASTM D1385	24 hours	100	AGB	None
Hydrogen Sulfide (H_2S)	HACH	24 hours	250	PB/GB	None / No Headspace
Hydroxide (OH)	SM 2320 B	14	50	PB/GB	None
Ignitability (Flashpoint)	EPA 1010A	14	150	PB/GB	None
Iodide	SM 4500 ID-M	24 hours	150	PB/GB	None
Mercaptans	LACSD 258	48 hours	50	PB/GB	None
Metals					
Organic Lead	DHS LUFT	7	100	GB/PB	None
Mercury, Total	EPA 7470A / 245.1	28	100	PB/GB	HNO_3
ICP Metals, Total	EPA 6010B / 200.7	180	100	PB/GB	HNO_3
ICP/MS Metals, Total	EPA 6020 / 200.8	180	100	PB/GB	HNO_3
Mercury, Dissolved (Lab Filtered)	EPA 7470A / 245.1	24 hours	100	PB/GB	None
ICP Metals, Dissolved (Lab Filtered)	EPA 6010B / 200.7	24 hours	100	PB/GB	None
ICP/MS Metals, Dissolved (Lab Filtered)	EPA 6020 / 200.8	24 hours	100	PB/GB	None
Mercury, Dissolved (Field Filtered)	EPA 7470A / 245.1	28	100	PB/GB	HNO_3
ICP Metals, Dissolved (Field Filtered)	EPA 6010B / 200.7	180	100	PB/GB	HNO_3
ICP/MS Metals, Dissolved (Field Filtered)	EPA 6020 / 200.8	180	100	PB/GB	HNO_3

Holding Times and Containers for Water/Aqueous Samples					
Tests	Method	Holding Time (days)	Min Volume (ml)	Container	Preservation
Methane in Water	RSK 175 (M)	14	40	VOA	HCl / No Headspace
Methanol	EPA 8015B	7	40	VOA	None
NDMA	EPA 1625C (M)	7	1000	AGB	$Na_2S_2O_3$ or None
Nitrate (NO_3)	EPA 300.0/SM 4500 NO3	48 hours	50	PB/GB	None
Nitrate (NO_3)	SM 4500 NO3	28	50	PB/GB	H_2SO_4
Nitrite (NO_2)	EPA 300.0/SM 4500 NO2	48 hours	50	PB/GB	None
Nitrite (NO_2)	SM 4500 NO2	28	50	PB/GB	H_2SO_4
Oil and Grease	SM 5520 B	28	1000	GB	H_2SO_4 or HCL
TriButyl Tin (TBT) or Organo Tin	Krone et. al.	7	1000	AGB	$Na_2S_2O_3$ or None
ORP (Redox Potential)	ASTM D-1498	24 hours	50	PB/GB	None
PAHs (Polynuclear Aromatic Hydrocarbons)	EPA 8310	7	1000	AGB	$Na_2S_2O_3$ or None
PCBs (PolyChlorinated Biphenyls)	EPA 8082/608	7	1000	AGB	$Na_2S_2O_3$ or None
Perchlorate	EPA 314.0	28	50	PB/GB	None
Perchlorate	EPA 331.0 (M)	28	50	PB/GB	Sterilized / with Headspace
Permanganate	Titrimetric	24 hours	100	AGB	None
Pesticides, Organochlorine	EPA 8081A/608	7	1000	AGB	$Na_2S_2O_3$ or None
Pesticides, Organophosphorus	EPA 8141A	7	1000	AGB	$Na_2S_2O_3$ or None
pH	SM4500 H+B	ASAP (24 hours)	50	PB/GB	None
Phenolics, Total	EPA 420.1	28	200	AGB	H_2SO_4
Phenols	EPA 8270C	7	1000	AGB	$Na_2S_2O_3$ or None
Phosphate, Ortho-	SM4500 P B/E / 300.0 / 365.1	48 hours	50	PB/GB	None
Phosphate, Total or Dissolved	SM 4500 P B/E	28	50	GB	None
Phosphorus, Total	SM 4500 P B/E	28	100	GB	H_2SO_4
PNA (PolyNuclear Aromatic Hydrocarbons)	EPA 8310	7	1000	AGB	$Na_2S_2O_3$ or None
Purgeable Halocarbons/Aromatics(8021B list)	EPA 8260B	14	40	VOA	HCl / No Headspace
Redox Potential (ORP)	ASTM D-1498	24 hours	50	PB/GB	None
RSK 175 (Methane,Ethane,Ethene)	RSK 175 (M)	14	40	VOA	HCl / No Headspace
RSK 175 (Carbon Dioxide, CO_2)	RSK 175 (M)	7	40	VOA	None / No Headspace
Salinity	SM2520B	28	50	PB/GB	None
Semi-Volatiles (SVOC/SVOA/N)	EPA 8270C/625	7	1000	AGB	$Na_2S_2O_3$ or None
Settleable Solids (SS)	SM 2540 F	48 hours	1000	PB/GB	None
Specific Conductance	SM 2510 B	28	50	PB/GB	None
Specific Conductance	EPA 120.0	24 hours	50	PB/GB	None
Sulfate	EPA 300.0/ASTM D516-02	28	50	PB/GB	None
Sulfide, Soluble/Dissolved	SM 4500 S2 D	ASAP (24 hours)	50	PB/GB	None
Sulfide, Total	SM 4500 S2 D	7	50	PB/GB	$ZnAc_2$ & NaOH
Sulfite (SO_3)	SM 4500 SO3B	24	100	PB/GB	EDTA
Surfactants (MBAS)	SM 5540 C	48 hours	200	PB/GB	None
SVOCs (Semi-Volatiles / BNAs)	EPA 8270C/625	7	1000	AGB	$Na_2S_2O_3$ or None
Thiosulfate (S_2O_3)	LACSD 253A	48 hours	200	500PB	None
TKN (Total Kjeldahl Nitrogen)	SM 4500-N Org B / 351.2	28	500	AGB	H_2SO_4
Total Organic Carbon (TOC)	SM 5310 D	28	150/40	GB/VOA	H_2SO_4 or HCL
Total Nitrogen (TN=TKN + NO3 + NO2)	Total Inorganic Nitrogen (TIN=NH3 + NO3 + NO2)		Total Organic Nitrogen (TON=TKN-NH3)		
Total Dissolved Solids (TDS)	SM 2540 C	7	1000	PB/GB	None
Total Suspended Solids (TSS)	SM 2540 D	7	1000	PB/GB	None
Total Solids (TS)	SM 2540 B	7	200	PB/GB	None
TPH(g) (Gas)	EPA 8015B GRO	14	40	VOA	HCl / No Headspace
TPH(d) / TPH-Extractable (TPH-Diesel)	EPA 8015B DRO	7	500	AGB	None
TPH-Purgeable / TVPH (TPH-Gas)	EPA 8015B GRO	14	40	VOA	HCl / No Headspace
TPH-CC (Carbon Chain/Extended Range)	EPA 8015B (M)	7	500	AGB	None
TRC (Chlorine, Total Residual)	SM 4500 Cl	24 hours	100	PB/GB	None
TRPH (Total Recoverable Petroleum Hydrocarbon)	EPA 418.1	28	500	AGB	H_2SO_4 or HCL
TriButyl Tin (TBT) or Organo Tin	Krone et. al.	7	1000	AGB	$Na_2S_2O_3$ or None
Turbidity	SM 2130 B	48 hours	100	PB/GB	None
VOCs	EPA 8260B/624	14	40	VOA	HCl / No Headspace
VOCs (drinking water)	EPA 524.2	14	40	VOA	Ascor Acid/HCL/No Headspace
Volatile Fatty Acids (Organic Acids / VFA)	HPLC/UV	28	125	AGB	H_3PO_4
Volatile Solids (VS)	EPA 160.4	7	200	PB/GB	None
Volatile Suspended Solids (VSS)	SM 2540 D/EPA 160.4	7	200	PB/GB	None

Holding Times and Container-Type for Solid Samples					
Tests	***Method***	***Holding Time (days)***	***Minimum Mass (g)***	***Container***	***Preservation***
Alkalinity	SM 2320 B	14	20	G/P	4°C
Biochemical Oxygen Demand (BOD)	SM 5210 B(M)	14	30	G/P	4°C
Bromide	EPA 300.0(M)	28	10	G/P	4°C
Chemical Oxygen Demand (COD)	SM 5220 D(M)	28	10	G/P	4°C
Chloride	EPA 300.0 (M)	28	10	G/P	4°C
Chromium VI (Hexavalent Chromium)	EPA 7196A/3060A	30	10	G/P	4°C
Chromium VI (Hexavalent Chromium)	EPA 7199/3060A	30	10	G/P	4°C
Cyanide, Amenable	EPA 9010C/9014	14	20	G/P	4°C
Cyanide, Reactive	SW 846 Ch. 7	14	20	G/P	4°C
Cyanide, Total	EPA 9010C/9014	14	10	G/P	4°C
Ferrous Iron	SM 3500-Fe B(M)	ASAP (24 hours)	10	G/P	4°C
Fluoride	SM 4500-F^- C(M)	28	20	G/P	4°C
Hexane Ext. Material (HEM/SGT-HEM)	EPA 1664A(M)	28	30	G/P	4°C
Ignitability (Flashpoint)	EPA 1010A(M)	14	100	G/P	4°C
Moisture Content	ASTM D2216	10	20	G/P	4°C
Nitrogen, Ammonia	SM 4500-HN_3 B/C(M)	28	10	G/P	4°C
Nitrogen, Nitrate	EPA 300.0(M)	7	10	G/P	4°C
Nitrogen, Nitrite	EPA 300.0(M)	7	10	G/P	4°C
Nitrogen, Nitrate+Nitrite (NO_3+NO_2)	SM 4500-NO_3 E(M) / SM 4500-NO_2 B(M)	7	10	G/P	4°C
Nitrogen, Organic	SM 4500-NH_3/4500-N_{org} B	28	10	G/P	4°C
Nitrogen, Total Kjeldahl (TKN)	SM 4500-N_{org} B(M)	28	10	G/P	4°C
Nitrogen, Total	TKN / NO_3 + NO_2	14	30	G/P	4°C
Oil and Grease	SM 5520 B(M)	28	30	G/P	4°C
Perchlorate	EPA 314.0(M)/6850(M)	28	20	G/P	4°C
pH	EPA 9045D	ASAP (24 hours)	20	G/P	4°C
Phenolics, Total	EPA 9065	28	20	G/P	4°C
Phosphate, Ortho	EPA 300.0(M)	14	10	G/P	4°C
Phosphate, Total	SM 4500-P B/E(M)	28	20	G/P	4°C
Phosphorus, Total	SM 4500-P B/E(M)	28	20	G/P	4°C
Specific Conductance	EPA 9050A	28	20	G/P	4°C
Sulfate	EPA 300.0(M)/9038	28	20	G/P	4°C
Sulfide, Reactive	SW 846 Ch. 7	7	20	G/P	4°C
Sulfide, Total	SM 4500-S^{2-} D	7	20	G/P	4°C
Surfactants (MBAS)	SM 5540 C(M)	48 hours	20	G/P	4°C
Total Organic Carbon (TOC)	EPA 9060A	28	2	G/P	4°C
96-Hour Aquatic Toxicity, Haz Waste	CA Dept. Fish & Game	7	100	G/P	4°C
Metals	***Method***	***Holding Time (days)***	***Minimum Mass (g)***	***Container***	***Preservation***
ICP Metals	EPA 6010B	180	2	G/P	None
ICP/MS Metals	EPA 6020	180	2	G/P	None
Mercury	EPA 7471A	28	1	G/P	None
Organic Lead	DHS LUFT	14	10	G/P	None
Semi-Volatile Organics	***Method***	***Holding Time (days)***	***Minimum Mass (g)***	***Container***	***Preservation***
Methanol / Ethanol	EPA 8015B	14	50	G	4°C
PCBs	EPA 8082	14	20	G	None
Pesticides, Organochlorine	EPA 8081A	14	20	G	4°C
Pesticides, Organophosphorus	EPA 8141B	7	20	G	4°C
Herbicides, Chlorinated	EPA 8151A	14	50	G	4°C
PAHs	EPA 8310	14	20	G	4°C
SVOCs (BNAs)	EPA 8270C	14	20	G	4°C
Organotins	Krone et al	14	20	G	4°C
Volatile Organics	***Method***	***Holding Time (days)***	***Minimum Mass (g)***	***Container***	***Preservation***
Purgeable Halocarbons/Aromatics	EPA 8260B(8021B list)	14	10	G	4°C
VOCs / TPPH	EPA 8260B / LUFT GC/MS	14	10	G	4°C
VOCs / TPPH (Encore)	EPA 5035/8260B	48 hours	3/sample	Encore	4°C
VOCs / TPPH (TerraCore)	EPA 5035/8260B	14	3/sample	Pre-Weighed Vial	2x$NaHSO_4$/1xMeOH

Holding Times and Container-Type for Solid Samples					
LUFT	*Method*	*Holding Time (days)*	*Minimum Mass (g)*	*Container*	*Preservation*
TPH(g)/BTEX/MTBE	EPA 8015B GRO/8021B	14	5	G	4°C
TPH(g) (EnCore)	EPA 5035/8015B GRO	48 hours	2/sample	EnCore	4°C
TPH(g) (TerraCore)	EPA 5035/8015B GRO	14	3/sample	Pre-Weighed Vial	2x$NaHSO_4$/1xMeOH
TPH(d)	EPA 8015B DRO	14	10	G	4°C
TPH-CC	EPA 8015B(M)	14	10	G	4°C
TRPH	EPA 418.1(M)	28	5	G	4°C
STLC / TCLP / SPLP	*Method*	*Holding Time (days)*	*Minimum Mass (g)*	*Method Ext. After*	*After Ext. (days)*
Mercury	CCR T22.11.5.A-II / EPA 1311/1312	28	50 / 100	N/A	28
Metals	CCR T22.11.5.A-II / EPA 1311/1312	180	50 / 100	N/A	180
SVOCs	CCR T22.11.5.A-II / EPA 1311/1312	14	50 / 100	7	40
VOCs	CCR T22.11.5.A-II / EPA 1311/1312	14	50 / 25	N/A	7
TPH(g)	CCR T22.11.5.A-II / EPA 1311/1312	14	50 / 25	N/A	7
Holding Times and Container-Type for Air/Vapor Samples					
Analysis	*Method*	*Holding Time (days)*	*Minimum Volume (L)*	*Container*	*Preservation*
Fixed Gases	ASTM D1946	3 / 14	1 / 6	Tedlar Bag / Summa Can	Keep out of sunlight
Hydrocarbon Speciation	ASTM D2820	3 / 14	1 / 6	Tedlar Bag / Summa Can	Keep out of sunlight
Hydrogen Sulfide (H2S)	GC/FPD	24 hours	1	Tedlar Bag	Keep out of sunlight
Landfill Gases (NMOCs)	SCAQMD 25.1(M)	3 / 14	1 / 6	Tedlar Bag / Summa Can	Keep out of sunlight
TPH(g)	TO-3(M)	3 / 14	1 / 6	Tedlar Bag / Summa Can	Keep out of sunlight
VOCs	EPA TO-14A/TO-15	3 / 14	1 / 6	Tedlar Bag / Summa Can	Keep out of sunlight

Holding Time Container.xls Revision 02/21/2011

Appendix 11.3:

MISCELLANEOUS METHODS AND SUBCONTRACT INFORMATION

METALS		Title 22 or CAM Metals	Priority Pollutant Metals	TCLP or RCRA Metals	Gen Mineral Metals
Antimony	Sb	X	X		
Arsenic	As	X	X	X	
Barium	Ba	X		X	
Beryllium	Be	X	X		
Cadmium	Cd	X	X	X	
Calcium	Ca				X
Chromium	Cr	X	X	X	
Cobalt	Co	X			
Copper	Cu		X		
Iron	Fe				X
Lead	Pb	X	X	X	
Magnesium	Mg				X
Manganese	Mn				X
Mercury	Hg	X	X	X	
Molybdenum	Mo	X			
Nickel	Ni	X	X		
Potassium	K				X
Selenium	Se	X	X	X	
Silver	Ag	X	X	X	
Sodium	Na				X
Thallium	Tl	X	X		
Vanadium	V	X			
Zinc	Zn	X	X		

ICP SCAN METALS	
Ag	Silver
Al	Aluminum
As	Arsenic
B	Boron
Ba	Barium
Be	Beryllium
Bi	Bismuth
Ca	Calcium
Cd	Cadmium
Co	Cobalt
Cr	Chromium
Cu	Copper
Fe	Iron
Hg	Mercury
Li	Lithium
Mg	Magnesium
Mn	Manganese
Mo	Molybdenum
Na	Sodium
Ni	Nickel
P	Phosphorus, Total
Pb	Lead
S	Sulfur, Total
Sb	Antimony
Se	Selenium
Si	Silicon
Sn	Tin
Sr	Strontium
Ti	Titanium
Tl	Thallium
U	Uranium
Vn	Vanadium
Zn	Zinc

LUFT 5 METALS
Cadmium
Chromium
Lead
Nickel
Zinc

ANIONS	
Bromide	Br
Chloride	Cl
Fluoride	Fl
Nitrate	NO_3
Nitrite	NO_2
Ortho-Phosphate	o-PO_4
Sulfate	SO_4

OXYGENATES	
DIPE	Di-iso-Propyl Ether
ETOH	Ethanol
ETBE	Ethyl-t-Butyl Ether
MTBE	Methyl-t-Butyl Ether
TAME	Tert-Amyl-Methyl Ether
TBA	Tert-Butyl Alcohol

PRESERVATIVES
H_2SO_4: Sulfuric Acid
H_3PO_4: Phosphoric Acid
HCL: Hydrochloric Acid
HNO_3: Nitric Acid
$Na_2S_2O_3$: Sodium Thiosulfate
NaOH: Sodium Hydroxide
$ZnAc_2$: Zinc Acetate

SUBCONTRACT ANALYSES	
Asbestos	PLM
	TEM
Aquatic Toxicity/Bioassay	Acute
	Chronic
Dioxins/Furans	2,3,7,8-TCDD
	Dioxins and Furans
Geotechnical	Atterberg Limits
	Bromate
	Bulk Density
	FOC / TOC
	Grain Size
	Hydraulic Conductivity
	Particle Size
	Permeability
	Porosity
	Specific Gravity
Glycols	Ethylene Glycol
	Propylene Glycol
Helium	Helium (Air)
Microbiology	Total Coliform
	Fecal Coliform
	E. Coli
	Enterococcus
	Heterotrophic Plate Count (HPC)
	Gasoline-Degrading Bacteria
Pesticides/Herbicides	Carbamates/Urea Pesticides
	Triazines
Radiochemistry	Gross Alpha
	Gross Beta
	Radium
	Radon
TOX	Total Organic Halides (TOX)
VFA	Volatile Fatty Acids (AM23E)
VOCs	EPA 524.2
	Industrial Solvents

SHORTHOLD ANALYSES	
15 Minutes:	pH, TRC (Total Residual Chlorine), Diss. Sulfide
24-Hours:	ADA, Carbon Dioxide (CO2), Cr-6,
	Dissolved Metals (Lab to Filter), DOC,
	D.O. (Diss. Oxygen),
	Ferrous Iron (Fe+2), Formaldehyde,
	Hydrazine, Hydrogen Sulfide,
	Iodide, Odor, Redox Potential,
	Conductivity (120.1), Sulfite
36-Hours:	Bioassay (Aquatic Toxicity)
48-Hours:	BOD, Color, MBAS, Mercaptans, Nitrate,
	Nitrite, ortho-Phosphate,
	Settleable Solids, Thiosulfate
	Turbidity, EnCores (Soil)

Holding Time Container.xls Revision 01/06/2011

Appendix 11.4

SAMPLE ENTRY INTO LIMS PROCEDURE

1. LIMS LOG-IN

1.1. Open Log-in Program in Launcher.

1.1.1. Log in Initials.

1.1.2. Enter Password.

2. SAMPLE ENTRY

2.1. Select Sample Log-in Tab.

2.1.1. Click on "New Work Order".

2.1.2. Match work order that appeared with work order number on your (COC).

2.1.3. Adjust the numbers to reflect COC numbers if they don't match.

2.2. Click on "Client" button.

2.2.1. Scroll down to appropriate client.

2.2.2. Select client by clicking "okay".

2.3. When you choose the client, a client contact is defaulted into the appropriate spot.

2.3.1. Adjust the name if deemed necessary.

2.4. Enter Project I.D.

2.5. Enter P.O. # (If present on COC).

2.6. Enter Date and time received in appropriate spots.

2.7. Enter any special instructions if present.

2.8. Enter Quality Assurance Project Plan box (QAPP).

2.8.1. Refer to QAPP notebook for the numbers.

2.8.2. Note there are two slots for QAPP numbers. You must have at least 0000 entered if there are no applicable QAPP'S.

2.9. Regional Water Quality Control Board (RWQC B).

2.9.1. Check the box if you choose to indicate this on chemist sheets. This indicates detection limits set by the control board that chemists must use.

2.9.2. If RWQCB is selected then you must enter 0001 as QAPP number in either one of slots given.

2.10. Click on "Clear".

2.10.1. Computer will ask you if you want to clear the sample information screen.

2.10.2. Click on "yes" or "no" depending on what you need to do. "Yes" will clear all the fields so that you may enter new information.

2.11. Click on "Samples" tab.

2.11.1. Click on "New". (A new window will appear.)

2.11.1.1. In this window you will enter the sampling time and sampling date. (Note time is entered in military time.)

2.11.2. Enter the sample I.D. as it appears on the COC.

2.11.2.1. If you look in the field located directly located under the sample I.D. box, you will see the figures you entered for in the previous step for time and date sampled.

2.11.3. Go to Matrix box.

2.11.3.1. Click on the down arrow.

2.11.3.1.1. You will see the different types of matrices (Water, Soil/Solid, Air, Oil, Filter Paper, and Extract).

2.11.3.1.2. Once a matrix is chosen, you must enter the cursor into another field before the choice is accepted by LIMS. This is true for any data entered in any field.

2.12. Go to #Containers box.

2.12.1. Enter number of containers for that sample.

2.13. Adding / Deleting / Editing tests.

2.13.1. Adding Tests.

2.13.1.1. Click on "Add Test" button. (A new window will pop open.)

2.13.1.2. Scroll for the desired testcode. Note: You can change TAT on the bottom of the screen so that any test added would have that desired TAT.

2.13.1.3. Some tests require specific extraction codes.

2.13.1.3.1. The computer will automatically default to an extraction code.

2.13.1.3.2. If you need to change this you can when the extraction box opens after a testcode is selected.

2.13.1.3.3. For some tests, the extraction box doesn't open when the test is added. You can change the extraction for those by:

2.13.1.3.3.1. Clicking on the "Change XT" button. If extraction isn't normally done with a particular method the computer will prompt you. However, you can still change the extraction to the desired code.

2.13.1.4. Once you have the desired testcode, select it with the mouse and click the "Add test" button. Your test has been added.

2.13.2. Deleting Tests.

2.13.2.1. Choose the test to be deleted by clicking on the test.

2.13.2.1.1. Click "Delete 1 Test" if that is desired.

2.13.2.1.2. Click "Delete All Tests" if that is desired.

2.13.2.1.3. Computer will prompt you before the deletion is made. Make the decision when prompted.

2.13.3. Editing Tests.

2.13.3.1. To change any information entered, simply double click the field desired and if it is applicable the computer will allow this.

2.13.3.2. Change the information needed, but then remember to choose another field to make the change final.

2.13.3.3. If you forgot to change the TAT when you selected your analysis, you can still change them by doing the following:

2.13.3.3.1. Locate the test that needs to be changed.

2.13.3.3.2. Go to the TAT box.

2.13.3.3.3. Double click on the number and change it to the desired TAT. Note: Remember to change the field once the new data is entered and double check to make sure the change was accepted.

Appendix 11.5

COURIER PROCEDURE

1. SAMPLE PICK-UP / DELIVERY

1.1. Courier checks maintenance log of vehicle to be used and checks brakes, tires and scheduled maintenance.

1.2. Courier makes copy of the Courier Request Logbook for the schedule of pick-ups and deliveries as assigned by the dispatcher.

1.3. Courier loads up the vehicle with clean empty coolers with Temperature Blank, ice, and bubble bags for packaging the samples.

1.4. Courier checks and takes items for delivery to the client as requested in the Courier Request Log.

1.5. Courier informs dispatcher when a map is required and dispatcher will generate as needed.

1.6. If any time delays are encountered, the courier informs dispatcher of delay and they relay it to the Project Manager.

1.7. ►Courier then follows the ***standard*** sample receipt procedure as outlined in SOP-T100, ***Section 6***.

1.8. Upon arrival at pick-up location, courier will compare received samples versus COC and resolve discrepancies with client, if possible. If client is unavailable, proceed with pick-up as-is and inform sample control personnel upon arrival at laboratory.

1.9. Courier checks if containers are safely arranged inside the cooler otherwise, more packaging materials are added to avoid breakage.

1.10. Wet ice and Temperature Blank is added if necessary into the cooler.

1.11. When samples are not in cooler the samples are then placed in the cooler that the courier brought and documented on Sample Receipt Form how the sample was received.

1.12. The chain-of-custody is signed after following the procedure.

1.13. If the courier must make multiple stops, the samples are to be secured within the vehicle at each stop if the courier is to be away from the vehicle for any period of time. ("Away from the vehicle" is defined as out of eyesight. If the courier cannot fully view the vehicle at all times then they are deemed to be "away from the vehicle".)

1.13.1. This will involve locking the vehicle at each location and then verifying that the coolers and/or samples were not compromised while the courier was away from the vehicle. If there is evidence that the cooler and/or samples have been compromised (missing, damaged, etc.), the courier is to immediately contact the project manager at the laboratory who in turn will contact the client regarding the incident. Further instruction about returning the cooler/samples to the client, or heading in to the laboratory will be given after contact with the client.

1.13.2. If the coolers/samples are usually carried in the back of an open truck (not suggested), then the coolers must be put inside the cab of the vehicle and

then the vehicle locked if the courier is to be away from the vehicle for any period of time.

1.13.2.1. If coolers must be carried in the back of an open truck, the coolers must be secured by tying them down to the sides/bed of the truck.

1.14. The actual temperature of the Temperature Blank is taken upon arrival at the laboratory following the instructions included in this SOP.

1.15. All courier stops are recorded on the Courier Log.

2. COURIER TRAINING CHECKLIST

2.1. Courier has a driving record acceptable to CEL insurance carrier.

2.2. Courier has demonstrated thorough knowledge of proper and professional interaction with clients.

2.3. Courier has read and is knowledgeable of all SOPs applicable to Sample Control operations.

2.4. Courier clearly understands the sample receipt and log-in procedures including, but not limited to chain-of custody protocols.

2.5. Courier has been trained in sample and cooler handling procedures, including cushioning containers to prevent breakage and arresting coolers to prevent sudden shifting during transport.

2.6. Courier has demonstrated knowledge of proper safety procedures associated with driving and Sample Control operations involving hazardous sample handling.

Appendix 11.6

SUMMA CANISTER CHECK-IN PROCEDURE

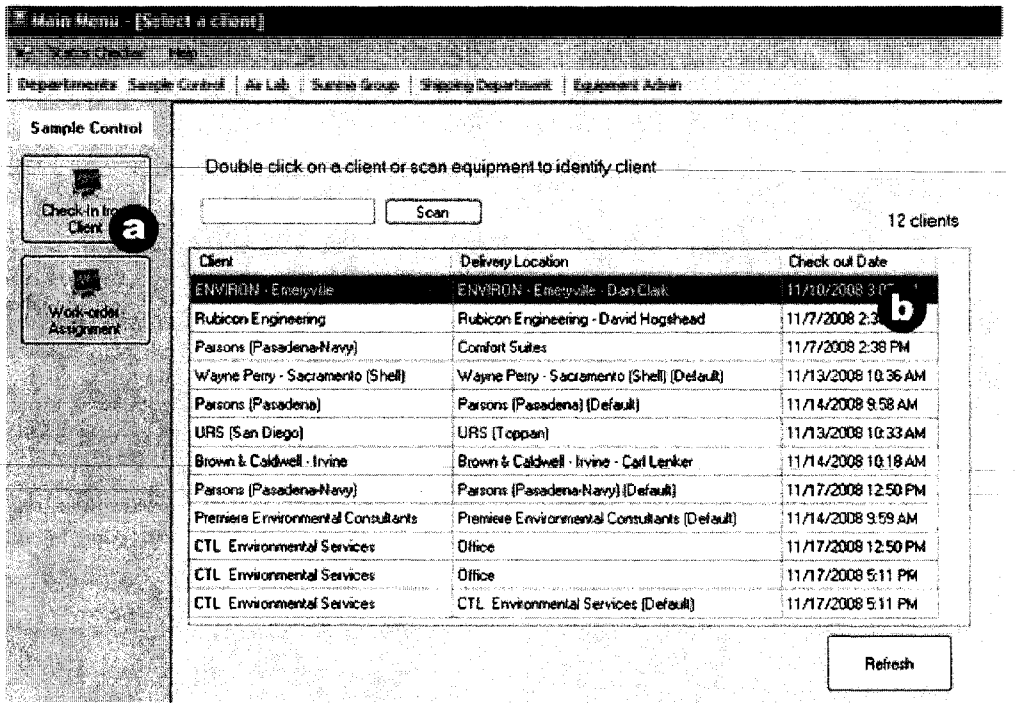
1. SUMMA CANISTER CHECK-IN

1.1. The Summa canister and flow controller equipment is managed using a barcode system. When Summas and/or flow controllers are returned to the laboratory by the client they need to be logged back into the equipment management system. To do this, follow these simple steps.

1.2. Open Launcher and click on the "New LIMS" button in the lower right hand corner of the home page. This will open another screen. On this screen there is a button labeled "Scanner". Click on this button and a screen called "Equipment Scanner" will appear. Click on "Sample Control" in the upper left hand portion of the screen and complete the following steps:

1.2.1. Click on "Check-In from Client" to load the form (see "a").

1.2.2. ►Double click on the client name or scan any equipment and let the system identify the client ***(see "b")***.

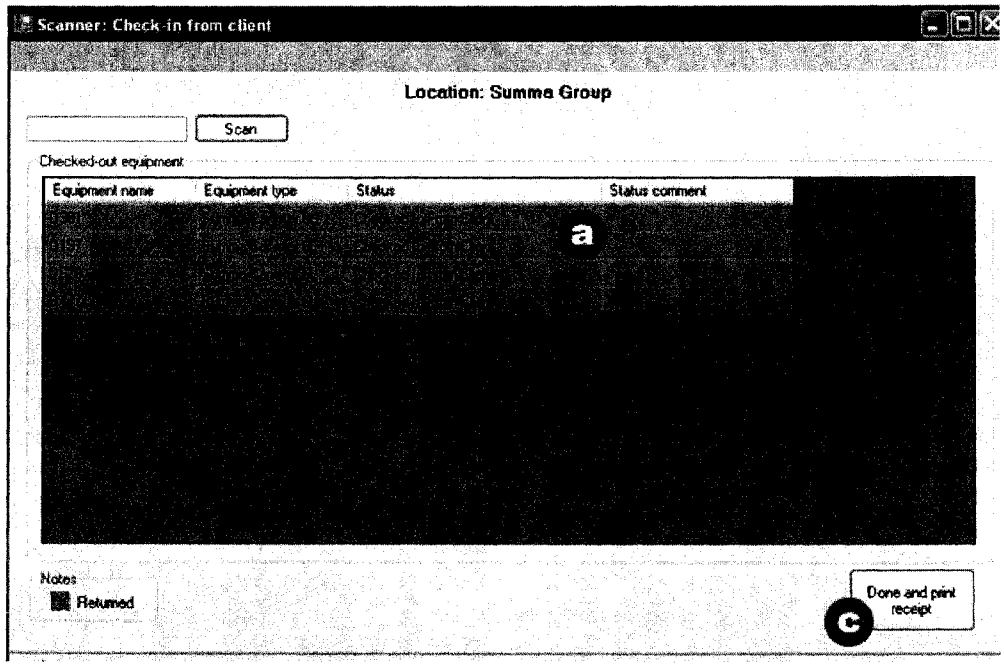


1.2.3. To scan equipment, use the barcode reader, and aim it at the barcode label applied to all Summa canisters and flow controllers.

1.2.3.1. The barcode appears as a small black and white icon on a thermal sticker. There is a letter and a 3 digit number also printed on the sticker.

1.2.4. Scan equipment to be checked-in. Returned equipment will be highlighted green.

1.2.5. ►After scanning all the equipment. Click on "Done and print receipt" to print a receipt ***(see "c")***.



1.2.6. If the client dropped off the Summa canisters/equipment, give them a copy of the return receipt.

1.2.7. A copy of the receipt is also given to the PM.

Appendix 11.7

NORTHERN CALIFORNIA REMOTE SAMPLE SHIPPING PROCEDURE

1. NORTHERN CALIFORNIA REMOTE SAMPLE SHIPPING PROCEDURE

1.1. This appendix is specific for shipping by Golden State Overnight (GSO) from their drop-off locations. General practices as described herein may be applicable to other shipping companies, but care must be taken to complete accompanying documentation as required by the particular vendor.

1.2. Prior to picking up any samples to be shipped, make sure to have the following in adequate supply for the anticipated number of samples and associated containers:

1.2.1. Coolers with ice and temperature blanks.

1.2.2. Large Ziploc bags.

1.2.3. Garbage bags, plastic.

1.2.4. Bubble bags / bubble wrap.

1.2.5. Strapping tape.

1.2.6. Shipping labels.

1.2.7. SDS (Saturday Deliver Service) stickers, if applicable.

2. SAMPLE RECEIPT

2.1. As with all sample pick-ups and receipt, the process begins with verification of the Chain of Custody (COC) and related sample identification.

2.1.1. All containers should be checked for cracks, breakage, and evidence of freezing, leakage or any other damage that could possibly compromise sample integrity.

2.1.2. Any identification of variance should be brought to the client's immediate attention and/or otherwise noted on the COC if a client representative is not present.

2.1.3. Upon completion of the verification, a copy of the original COC is left with the client or their representative, an additional copy is kept for record of shipment, and the original accompanies the samples to their destination. Refer to Appendix 11.4 for specific courier guidance.

3. PREPARING THE SHIPMENT

3.1. Upon receipt, samples should be placed in a shipping cooler with enough ice to insure that a temperature of ≤ 6.0°C is maintained during shipping.

3.1.1. Glass containers should be "bubble bagged" or "bubble wrapped" to protect from breakage.

3.1.2. Samples should be placed in Ziploc bags to prevent melting ice from infiltrating the sample.

3.1.3. Garbage bags may be used to contain the ice and water that accumulates from melting, but it is not necessary if the samples are securely Ziploc'd in individual or double bags.

3.2. Upon completion of packing the cooler(s), the original COC's should be signed as relinquished 'To GSO', sealed in a Ziploc bag, and taped to the underside of the lid to accompany the associated samples.

3.2.1. If multiple coolers are shipped, all COC's may be placed in one cooler as long as a record is made of how many coolers constitute the total shipment.

3.3. Once the COC's have been secured to the lid, close the cooler, and make sure that the lid is closed completely.

3.3.1. The lid should close without being 'forced'. Care should be taken to prevent containers from breaking from undue pressure caused by the weight of the ice and lid.

3.4. When the lid has been fully closed, use the strapping tape to seal the lid.

3.4.1. Make sure to use enough tape as the cooler and its contents will be handled as freight. Hence, it is important that the lid stays firmly in place for the duration of the trip.

4. ►LABELING

4.1. Fill out Sections 1, 2 (if necessary), 4, and 5 on a GSO shipping label for each cooler that will be shipped. ***See Appendix 11.8 for examples of the GSO shipping label.***

4.2. Enter the following information in Section 1:

4.2.1. Date: ***MM/DD/YY or MM-DD-YY.*** (This is the date that shipment will be delivered to GSO.)

4.2.2. Shipper's GSO Account No.: 9255. ***(This is the Calscience GSO account number. The account number is on pre-printed labels.)***

4.2.3. Company: CEL – Concord. ***(This is the Calscience Northern California Service Center.)***

4.2.4. Address: 5063 Commercial Circle.

4.2.5. Ste/Room: H.

4.2.6. City: Concord.

4.2.7. Zip Code: 94520-***8577***.

4.2.8. Sender's Name: ***(This is the name of the person who prepares the shipment.)***

4.2.9. Phone Number: 925-689-9022.

4.3. ***For coolers shipping to Calscience in Garden Grove***, if a pre-printed label is unavailable, enter the following information in Section 2:

4.3.1. Company: Calscience.

4.3.2. Name: Noel.

4.3.3. Phone Number: 714-895-549***4***.

4.3.4. Address: 7440 Lincoln Way.

4.3.5. City: Garden Grove.

4.3.6. Zip Code: 92841-***1427***.

4.4. For coolers shipping to a third party facility, enter the third party address and contact information in Section 2.

4.5. Check the box next to 'Package' in Section 4 and enter the estimated weight on the line. This field does not have to be accurate as GSO will weigh the cooler for billing purposes.

4.5.1. A large cooler containing water samples and ice typically weighs about 65 pounds. A large cooler containing soil samples and ice will be much heavier.

4.6. Indicate the type of delivery service requested in Section 5.

4.6.1. For shipments made from Monday through Thursday, check the 'Priority Overnight by 10:30 AM' box.

4.6.2. For shipments made on Fridays, must check the 'Saturday Delivery' box and affix an 'SDS' (Saturday Deliver Service) sticker to the top of each cooler.

4.7. Upon completion of all necessary information, remove the 'Shippers Copy' (the top copy) from the 3 ply label, and retain with the copies of COC for shipment tracking. Remove the label backing and firmly adhere the label to the top of the cooler.

5. DELIVERY

5.1. Coolers may be delivered to the following locations for direct shipment:

5.1.1. 896 National Drive, Sacramento, CA 95834-***1173 (see Appendix 11.9 for local area map)***.

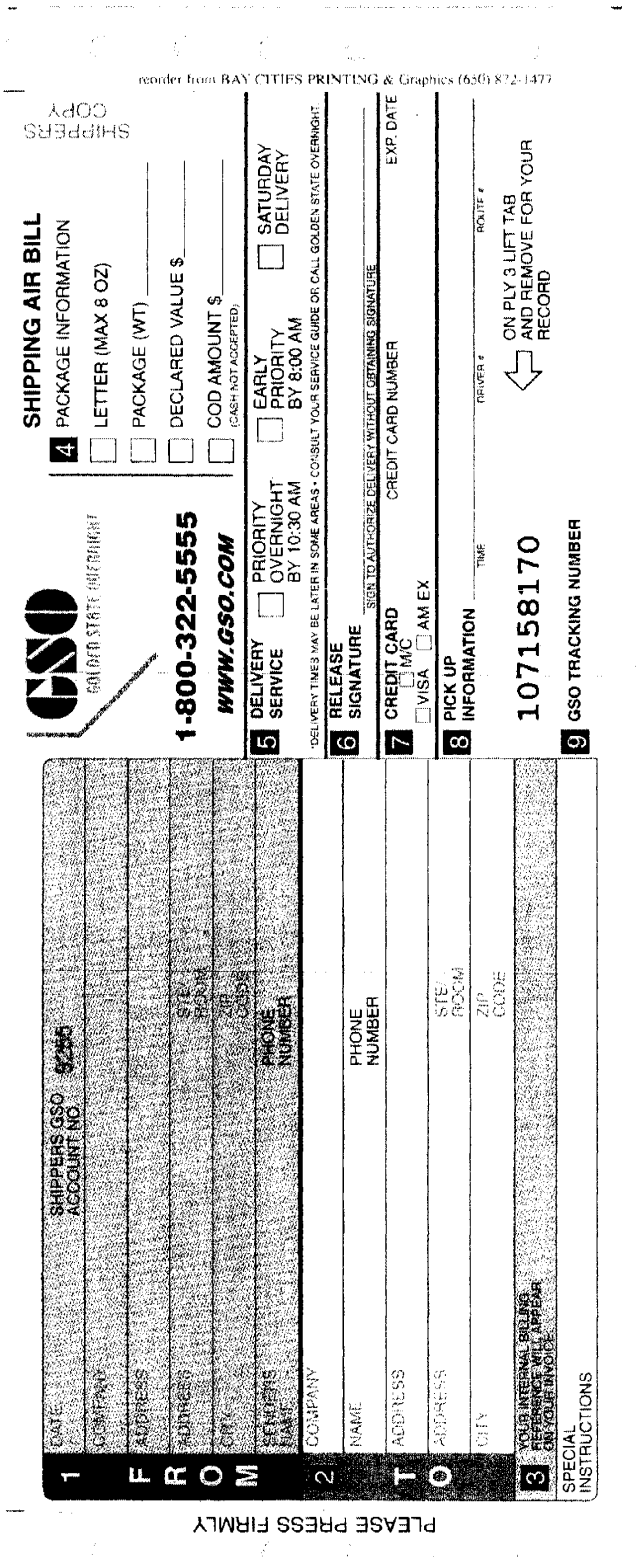
5.1.2. 4601 Malat Street, Oakland, CA 94601-***4903 (see Appendix 11.10 for local area map)***.

5.1.3. 2419 Zanker Road, San Jose, CA 95131-***1116 (see Appendix 11.11 for local area map)***.

5.2. These 3 facilities are regional distribution centers and will typically accept deliveries as late as 9:00 PM, but every effort should be made to arrive with sufficient time to process samples to be shipped overnight.

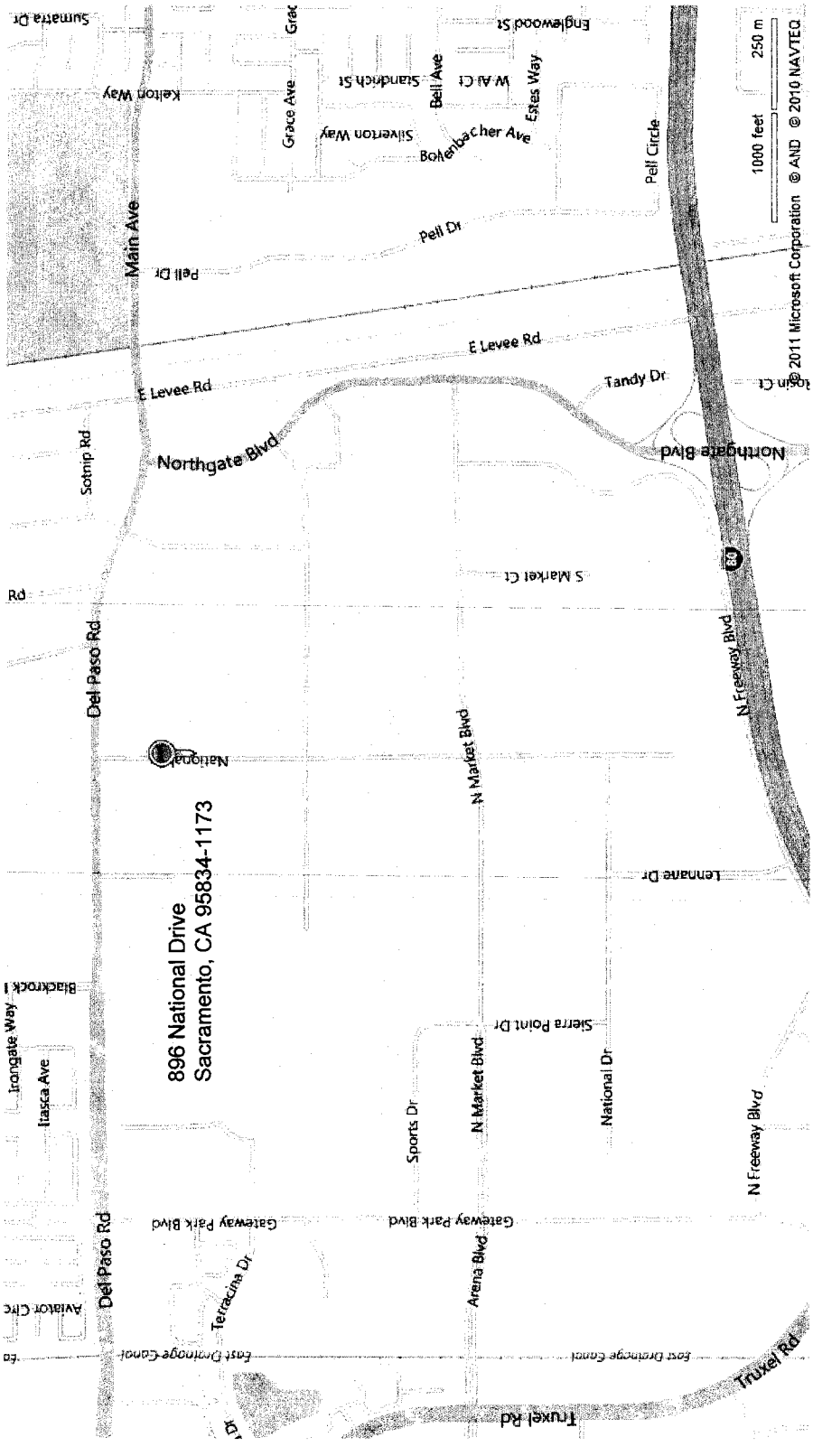
Appendix 11.8

EXAMPLE OF GSO SHIPPING LABEL


reorder from BAY CITIES PRINTING & Graphics (650) 872-1477
SHIPPERS COPY
GSO
GOLDEN STATE OVERNIGHT
SHIPPING AIR BILL
1-800-322-5555
WWW.GSO.COM
4 PACKAGE INFORMATION
LETTER (MAX 8 OZ)
PACKAGE (WT)
DECLARED VALUE $
COD AMOUNT $
(CASH NOT ACCEPTED)
5 DELIVERY SERVICE
PRIORITY OVERNIGHT BY 10:30 AM
EARLY PRIORITY BY 8:00 AM
SATURDAY DELIVERY
*DELIVERY TIMES MAY BE LATER IN SOME AREAS • CONSULT YOUR SERVICE GUIDE OR CALL GOLDEN STATE OVERNIGHT
6 RELEASE SIGNATURE
SIGN TO AUTHORIZE DELIVERY WITHOUT OBTAINING SIGNATURE
7 CREDIT CARD
M/C
VISA
AM EX
CREDIT CARD NUMBER
EXP. DATE
8 PICK UP INFORMATION
TIME
DRIVER #
ROUTE #
ON PLY 3 LIFT TAB AND REMOVE FOR YOUR RECORD
107158170
9 GSO TRACKING NUMBER
1 FROM
DATE
SHIPPERS GSO ACCOUNT NO.
COMPANY
ADDRESS
ADDRESS
STE. ROOM
CITY
ZIP CODE
SENDERS NAME
PHONE NUMBER
2 COMPANY
NAME
PHONE NUMBER
TO
ADDRESS
ADDRESS
STE. ROOM
CITY
ZIP CODE
3 YOUR INTERNAL BILLING REFERENCE WILL APPEAR ON YOUR INVOICE
SPECIAL INSTRUCTIONS
PLEASE PRESS FIRMLY

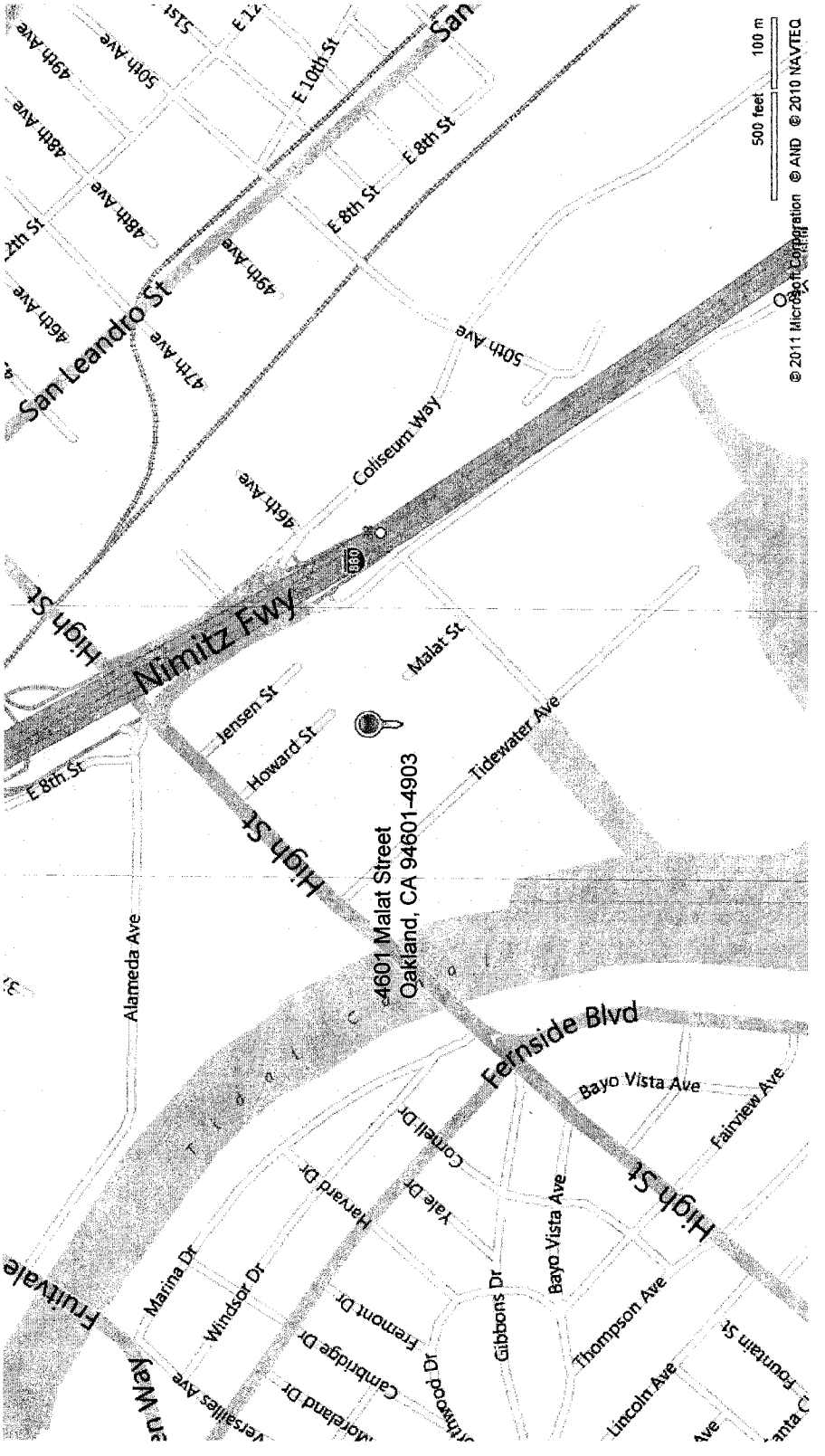
Appendix 11.9

GSO SACRAMENTO DISTRIBUTION CENTER LOCAL AREA MAP


896 National Drive
Sacramento, CA 95834-1173
Northgate Blvd
E Levee Rd
Pell Dr
Main Ave
Del Paso Rd
Truxel Rd
N Freeway Blvd
N Market Blvd
Gateway Park Blvd
Arena Blvd
Sports Dr
National Dr
Sierra Point Dr
Lennane Dr
S Market Ct
Tandy Dr
Pell Circle
Bohlenbacher Ave
Silverton Way
Grace Ave
Bell Ave
Estes Way
Sotnip Rd
Keiton Way
Sumatra Dr
Englewood St
Irongate Way
Itasca Ave
Blackrock
Aviator Cir
Terracina Dr
East Drainage Canal
© 2011 Microsoft Corporation © AND © 2010 NAVTEQ
1000 feet
250 m

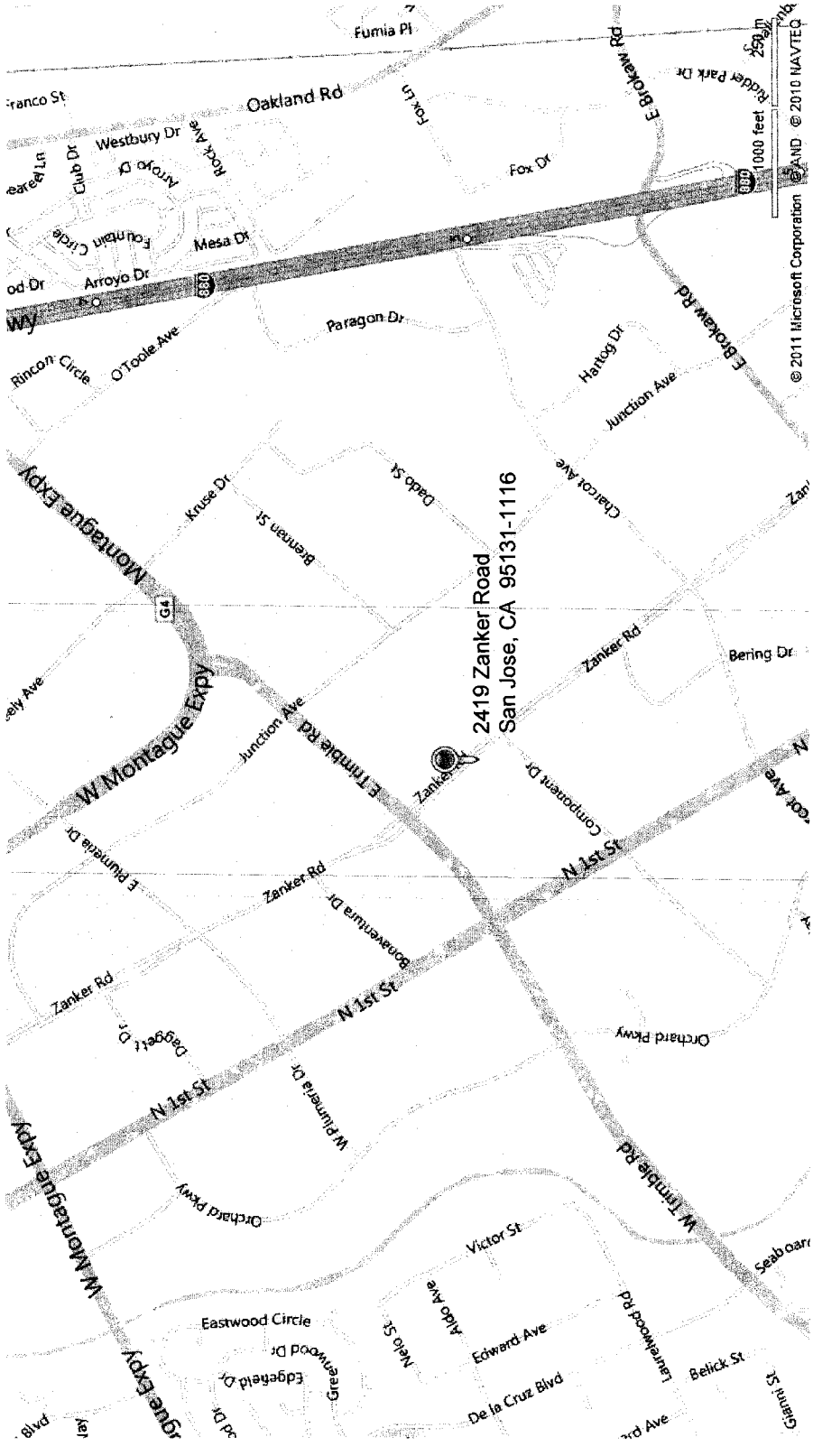
Appendix 11.10

GSO OAKLAND DISTRIBUTION CENTER LOCAL AREA MAP


4601 Malat Street
Oakland, CA 94601-4903
Nimitz Fwy
880
High St
Malat St
Jensen St
Howard St
Tidewater Ave
E 8th St
Alameda Ave
Fernside Blvd
Bayo Vista Ave
Fairview Ave
Cornell Dr
Yale Dr
Harvard Dr
Gibbons Dr
Thompson Ave
Lincoln Ave
Fremont Dr
Windsor Dr
Marina Dr
Fruitvale
Coliseum Way
46th Ave
47th Ave
48th Ave
49th Ave
50th Ave
San Leandro St
E 10th St
500 feet
100 m
© 2011 Microsoft Corporation © AND © 2010 NAVTEQ

Appendix 11.11

GSO SAN JOSE DISTRIBUTION CENTER LOCAL AREA MAP


2419 Zanker Road
San Jose, CA 95131-1116
Montague Expy
W Montague Expy
W Montague Expy
Oakland Rd
Fumia Pl
Fox Ln
Fox Dr
Westbury Dr
Club Dr
Arroyo Dr
Rock Ave
Mesa Dr
Fountain Circle
Paragon Dr
Rincon Circle
O'Toole Ave
Hartog Dr
Junction Ave
Kruse Dr
Brenan St
Dado St
Charcot Ave
Zanker Rd
Bering Dr
Junction Ave
E Trimble Rd
Zanker
Component Dr
N 1st St
E Plumeria Dr
Zanker Rd
Bonaventura Dr
Zanker Rd
Daggett Dr
Orchard Pkwy
W Plumeria Dr
W Trimble Rd
Victor St
Eastwood Circle
Edgefield Dr
Aldo Ave
Neto St
Edward Ave
De la Cruz Blvd
Laurelwood Rd
Belick St
E Brokaw Rd
Ruder Park Dr
1000 feet
250 m
© 2011 Microsoft Corporation
© 2010 NAVTEQ

Title : **EPA METHOD 3060A, ALKALINE DIGESTION FOR HEXAVALENT CHROMIUM**

Document No. : SOP-M224
Revision No. : 2.1
Supersedes : 2.0

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Revision 2.1 changes are noted in bold italicized typeface and preceded by a "►" marker.

APPROVED FOR RELEASE BY: _____ **MANAGEMENT** 8/18/9 **DATE**

_____ **QA DEPARTMENT** 08-18-09 **DATE**

ANNUAL SOP REVIEW

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

1. METHOD IDENTIFICATION

1.1. EPA Method 3060A, Alkaline Digestion for Hexavalent Chromium.

2. APPLICABLE MATRICES

2.1. This method is applicable to soils, sludges, sediments, and similar waste materials.

3. DETECTION LIMITS

3.1. Refer to the determinative method for detection limits.

4. SCOPE AND APPLICATION

4.1. EPA Method 3060A is an alkaline digestion procedure used to extract hexavalent chromium, Cr(VI).

4.2. Samples prepared by this method may be analyzed colorimetrically for Cr(VI) by Method 7196A (UV-VIS spectrophotometry) or Method 7199 (ion chromatography).

4.3. This method satisfies three criteria that must be met in order to quantify Cr(VI) in solids are:

4.3.1. The extracting solution must solubilize all forms of Cr(VI). The method's efficiency is evidenced through the recovery of the Cr(VI) spike.

4.3.2. The conditions of the extraction must not induce reduction of native Cr(VI) to Cr(III). Digestion in the alkaline environment minimizes the reduction of Cr(VI) to Cr(III).

4.3.3. The method must not cause oxidation of native Cr(III) contained in the sample to Cr(VI). The addition of Mg^{2+} in a phosphate buffer to the alkaline solution suppresses any oxidation, if oxidation is even observed.

5. METHOD SUMMARY

5.1. This method uses an alkaline digestion to solubilize both water-insoluble and water soluble Cr(VI) compounds in solid waste samples. The pH of the digestate must be carefully adjusted during the digestion procedure else re-digestion of the samples will be required.

5.2. A representative 2.5g (wet weight) sample is digested at 90–95°C in 0.28-M Na_2CO_3/0.5-M NaOH solution for 60 minutes to dissolve any Cr(VI) present and stabilize it against reduction to Cr(III).

6. DEFINITIONS

6.1. Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

6.2. Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

6.3. 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 NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

6.4. Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

6.5. Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

6.6. Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

6.7. Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

6.8. Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

6.9. Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.

6.10. Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

6.11. Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

6.12. Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

6.13. Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

6.14. Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

6.15. Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

6.16. Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

6.17. Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.

6.18. 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.

6.19. 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.

6.20. Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence.

6.21. Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

6.22. Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

6.23. Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

6.24. Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

7. INTERFERENCES

7.1. When analyzing a sample digest for Cr(VI), it may be appropriate to determine the reducing/oxidizing tendency of each sample matrix. This can be accomplished by characterization of each sample for additional analytical parameters such as pH, Fe(II), sulfide, Redox Potential, TOC, COD, and BOD. Analysis for these additional parameters establishes the tendency of CR(VI) to exist or not exist in the unspiked sample and assists in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria for total metals.

7.2. Certain substances, not typically found in the alkaline digests of soils, may interfere in the analytical methods for Cr(VI) following digestion if the concentrations of these interfering substances are high and the Cr(VI) is low. For the specific substances, please refer to Method 7196A or 7199, as appropriate.

7.3. For sample containing soluble Cr(III) concentrations greater than four times the laboratory Cr(VI) reporting limit, Cr(VI) results may be biased high due to method induced oxidation. The addition of Mg2+ in a phosphate buffer to the alkaline digestion solution has been shown to suppress this oxidation.

7.4. If the sample should go to dryness while being concentrated, low recoveries may result; therefore, the digestate must be discarded and the sample re-prepared.

8. ►SAFETY

8.1. ***Hexavalent chromium is a toxic form of the metal chromium. Chromium is a human poison by ingestion. Long term effects are localized itching, burning and dermatitis. Individuals with bronchial asthma can be affected. The metal is a severe irritant of the mucous membranes.***

8.2. Many metal salts are extremely toxic if inhaled or swallowed. ***Extreme care must be taken to ensure that samples and standards are handled properly and that all exhaust gases are properly vented.*** Wash hands thoroughly after handling.

8.3. All sample preparation activities should be performed in an operational fume hood appropriate for use with acids.

8.3.1. All operational fume hoods are to remain energized continuously in order to ***minimize acidic atmospheric or toxic gas buildup***.

8.4. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current

version of Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats are required to be worn in all designated laboratory areas. Protective gloves shall be worn when handling chemicals.

8.5. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

9. ►EQUIPMENT AND SUPPLIES

9.1. pH meter, ***capable of reading to 0.05 pH unit, equipped with ATC probe input and BNC electrode input connectors, Fisher Scientific*** accumet® Basic pH Meter, ***Fisher Scientific accumet® Basic AB 15 Bench-Top pH Meter***, or equivalent.

9.1.1. Electrode, ***0–14 pH range, glass pH membrane sensor, single porous ceramic junction, Ag/AgCl internal reference, rugged standard-size epoxy body, equipped with ATC probe, Fisher Scientific accumet® Liquid-Filled pH/ATC Epoxy Body Combination Electrode or equivalent***.

9.1.2. ***Electrode holder, adjustable height.***

9.2. ***Specimen containers, 4.5-oz (120-mL), with lids, high density polyethylene (HDPE) or polypropylene, disposable.***

9.3. Griffin beakers, 250-mL or other capacity, ***glass***.

9.4. Watch glasses, ribbed and non-ribbed, ***glass, appropriate diameter to cover beaker.***

9.5. Graduated cylinder, ***50-mL***, 100-mL, ***250-mL***, or other capacity, ***glass, Class A***.

9.6. ***Volumetric flasks, 100-mL and 1000-mL, glass, Class A.***

9.7. ***Pipetters, 10–100-µL, 100–1000-µL, 0.5–5.0-mL, and 1–10-mL, adjustable, with disposable tip.***

9.8. Hot plate, ***ceramic top, equipped with analog temperature and stirring control knobs***, capable of maintaining 90–95°C, ***Fisher Scientific Isotemp Basic Stirring Hotplate or equivalent***.

9.9. ***Stir bar, Teflon coated.***

9.10. Thermometer, ***equipped with holder and clamps***. Temperature range should include 90–95°C.

9.11. Drying oven, capable of maintaining 30°C.

9.12. Vacuum filtration apparatus:

9.12.1. ***Vacuum apparatus.***

9.12.2. ***Filter funnel, 300-mL capacity, polyphenylsulfone construction with magnetic seal, capable of supporting a 47-mm diameter filter, Pall Corporation 47-mm Magnetic Filter Funnel or equivalent.***

9.12.3. ***Filtration flask, 1000-mL, glass, with tubulation.***

9.12.4. ***Filter, 0.45-µm effective pore size, 47-mm diameter, hydrophilic polyvinylidene difluoride (PVDF) membrane, Millipore Durapore® Membrane Filter or equivalent.***

9.12.5. ***Filter, 0.7-µm or 1.0-µm effective pore size, 47-mm diameter, borosilicate glass microfiber, binder free, Whatman Grade GF/F Glass Microfiber Filter, Whatman Grade GF/B Glass Microfiber Filter, or equivalent.***

9.13. Balance, analytical, capable of weighing to the nearest ***0.1 mg***.

9.14. ***Balance, top loading, capable of weighing to the nearest 0.01 g.***

10. ►REAGENTS AND STANDARDS

10.1. Reagents

10.1.1. Reagent water, interferant free.

10.1.2. ***Sand, washed, sea or standard Ottawa.***

10.1.3. Sodium hydroxide, NaOH, ***white pellets and granules***, reagent grade or equivalent.

10.1.4. Sodium carbonate, Na_2CO_3, anhydrous, ***white granules***, reagent grade or equivalent.

10.1.5. Digestion solution.

10.1.5.1. Prepare the digestion solution by dissolving 20.00 ± 0.05 g of NaOH and 30.00 ± 0.05 g of anhydrous Na_2CO_3 in reagent water, and dilute to 1 L with additional reagent water.

10.1.5.2. ***Check the pH of the digestion solution prior to use.***

10.1.5.2.1. The pH must be ≥11.5. If the pH is < 11.5, discard the digestion solution and re-prepare.

10.1.5.3. The digestion solution must be prepared fresh monthly.

10.1.5.4. ***The digestion solution shall be stored in a plastic bottle at ambient temperature (20–25°C).***

10.1.6. Magnesium chloride, $MgCl_2$, hexahydrate, ***colorless crystals***, reagent grade or equivalent.

10.1.6.1. A mass of ***854*** mg of $MgCl_2 \cdot 6H_2O$ is approximately equivalent to 100 mg of Mg^{2+}.

10.1.7. ***Nitric acid, HNO_3, 68.0–70.0% (v/v) or 16-M, concentrated, clear colorless liquid, trace metals grade or equivalent.***

10.1.8. Nitric acid, HNO_3, 5.0-M.

10.1.8.1. ***Prepare the 5.0-M HNO_3 solution by slowly adding 62.5 mL of concentrated HNO_3 to 150 mL of reagent water and dilute to 200 mL with additional reagent water.***

10.1.8.1.1. Do not use the concentrated HNO_3 to prepare to prepare the 5.0-M HNO_3 solution if the liquid color has a yellow tinge. This is indicative of photoreduction of NO_3^- to NO_2^-, a reducing agent for Cr(VI).

10.1.8.2. The 5.0-M HNO_3 solution shall be stored ***in an amber glass bottle*** at ambient temperature (20–25°C).

10.1.9. ***Potassium phosphate, dibasic, K_2HPO_4, anhydrous, white crystals, certified, reagent grade or equivalent.***

10.1.10. ***Potassium phosphate, monobasic, KH_2PO_4, anhydrous, colorless to white crystals, certified, reagent grade or equivalent.***

10.1.11. Phosphate buffer, 0.5-M K_2HPO_4 / 0.5-M KH_2PO_4, pH = 7.

10.1.11.1. Prepare the phosphate buffer by dissolving 87.09 g of K_2HPO_4 and 68.04 g of KH_2PO_4 in 700 mL of reagent water, and dilute to 1 L with additional reagent water.

10.1.12. Potassium dichromate, $K_2Cr_2O_7$, ***orange-red crystals***, reagent grade or equivalent.

10.1.12.1. ***Dry $K_2Cr_2O_7$ at 105°C for 1 hour in an oven prior to stock standard preparation.***

10.1.12.2. ***The $K_2Cr_2O_7$ shall be stored in a plastic bottle at ambient temperature (20–25°C) under dry condition.***

10.1.13. Lead chromate, $PbCrO_4$, ***orange powder***, reagent grade or equivalent.

10.1.13.1. ***The $PbCrO_4$ shall be stored in a plastic bottle at ambient temperature (20–25°C) under dry condition.***

10.1.14. ***All reagents must be inspected and documented in the Chemicals and Supplies Verification Logbook prior to use.***

10.2. Standards

10.2.1. ***Manually-prepared stock standard solution, in a sealed amber glass bottle, containing 1000 mg/L of hexavalent chromium in reagent water (1.00-mL = 1.00-mg Cr^{6+}), is used to prepare the spike standard solution.***

10.2.1.1. Prepare the 1000-ppm Cr(VI) stock standard solution by dissolving 2.829 g of $K_2Cr_2O_7$ in reagent water and dilute to 1 L with additional reagent water.

10.2.1.2. ***The stock standard solution must be stored at ambient temperature (20–25°C) and replaced after six months or sooner.***

10.2.2. ***Spike Standard Solution***

10.2.2.1. Prepare the 100-ppm spike standard solution by diluting 10.0 mL of the 1000-ppm Cr(VI) stock standard to 100 mL with reagent water.

10.2.2.2. ***The spike standard solution must be prepared fresh daily.***

10.2.2.3. ***The spike standard solution is used to prepare quality control (QC) check samples such as soluble matrix spikes (MS/MSDs) and laboratory control samples (LCS/LCSDs).***

10.2.2.4. ***The hexavalent chromium for the spike standard solution must be of a source differing from that used for the initial calibration. If it is of the same source, then it must be of different lot.***

10.2.3. ***Spike Standard Solid***

10.2.3.1. ***Use $PbCrO_4$ as the spike standard solid.***

10.2.3.2. ***The spike standard solid is used to prepare quality control (QC) check samples such as insoluble matrix spikes (MS/MSDs).***

10.2.3.3. ***The hexavalent chromium for the spike standard solid must be of a source differing from that used for the initial calibration. If it is of the same source, then it must be of different lot.***

10.2.4. ***Refer to the specific SOPs of the determinative methods for additional standards.***

10.2.5. ***All stock standards must be inspected and documented in the Chemicals and Supplies Verification Logbook prior to use.***

11. ►SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

11.1. ***Solid samples should be collected in 2-oz or 4-oz pre-cleaned clear glass wide-mouth jars with Teflon-lined closures.***

11.1.1. Do not use stainless steel devices and containers to collect samples.

11.2. ***Samples should be maintained in a chill state (≤4°C) post sample collection until received at the laboratory. Samples should not be frozen (e.g., do not use dry ice as the refrigerant).***

11.3. ***Upon receipt, the samples are stored in a 0–6°C cooler.***

11.3.1. Solid samples must be digested within 30 days of sample collection.

11.3.2. All digested samples ***are then stored under dark and refrigerated (0–6°C) conditions and*** must be analyzed within a 7-day period post digestion.

11.4. ***Refer to the specific SOPs of the determinative methods for additional information on sample preservation and holding times.***

12. ►QUALITY CONTROL

12.1. ***Event Based Quality Control (LCS/LCSDs and MBs)***

12.1.1. ***Event based quality control consists of QC samples prepared and processed with each preparatory event. This consists of a laboratory control sample and laboratory control sample duplicate (LCS/LCSD) and a method blank (MB).***

12.1.2. ***One LCS consisting of the specified analytes spiked into washed sea sand is required every day digestions are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent. The LCSD is handled identically to the LCS.***

12.1.3. ***One MB consisting of washed sea sand is required every day digestions are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.***

12.1.4. ***The MB/LCS/LCSD is processed concurrently with the associated samples. In the processing of the MB/LCS/LCSD, reagents and procedures identical to those for actual samples are used.***

12.2. ***Matrix Based Quality Control (MS/MSDs)***

12.2.1. ***Matrix based quality control consists of QC samples prepared and processed using actual environmental samples. This consists of a matrix spike and matrix spike duplicate (MS/MSD).***

12.2.2. ***One soluble MS consisting of the actual sample matrix spiked with known concentrations of specific target analytes is required for every batch of 20 samples per matrix or portion thereof digested concurrently. The soluble MSD is handled identically to the soluble MS.***

12.2.3. ***One insoluble MS consisting of the actual sample matrix spiked with known concentrations of specific target analytes is required for every batch of 20 samples per matrix or portion thereof digested concurrently. The insoluble MSD is handled identically to the insoluble MS.***

12.2.4. ***The sample which is spiked for the MS/MSD is processed concurrently with the associated samples. In the processing of the***

MS/MSD, reagents and procedures identical to those for actual samples are used.

12.3. ***Refer to the specific SOPs of the determinative methods for additional information on quality control.***

13. ►CALIBRATION AND STANDARDIZATION

13.1. ***Analytical Balance***

13.1.1. ***Calibrate the analytical balance at 2 mg, 1 g, and 100 g using Class 2 weights.***

13.1.2. ***Calibration shall be within ± 10% at 2 mg (± 0.2 mg), or within ± 2% at 1 g (± 0.02 g) and at 100 g (± 2 g). If the values are not within these limits, recalibrate the balance.***

13.2. ***Top Loading Balance***

13.2.1. ***Calibrate the top loading balance at 1 g and 100 g using Class 2 weights.***

13.2.2. ***Calibration shall be within ± 2% at 1 g (± 0.02 g) and at 100 g (± 2 g). If the values are not within these limits, recalibrate the balance.***

13.3. ***Pipetter***

13.3.1. ***Calibrate the pipetter according to the procedure outlined in the Pipetter Calibration Check Logbook.***

13.4. ***Thermometer***

13.4.1. ***Calibrate the thermometer using an NIST certified thermometer. The calibration procedure shall adhere to the current revision of SOP-T042, "Thermometer Calibration.***

14. ►PROCEDURE

14.1. Adjust the temperature of each hot plate to 90−95°C. Use a calibrated thermometer and a temperature blank to verify the temperature.

14.1.1. A temperature blank consists of 50-mL of digestion solution.

14.2. ***Solid Sample Preparation***

14.2.1. Homogenize a solid sample ***as outlined in the current revision of SOP-M230.***

14.2.2. ***The amount of sample required for analytical determination is established by the determinative method or the project's Quality Assurance Project Plan (QAPP).***

14.2.2.1. ***The amount of sample generally required is 2.5 g nominal.***

14.3. ***Percent Dry Weight Determination***

14.3.1. ***When sample results are to be reported on a dry weight basis, a second portion of sample should be weighed at the same time as the portion used for analytical determination.***

14.3.2. ***Additional information regarding determination of solids content is provided in SOP-M700.***

14.4. ***pH and Oxidation Reduction Potential (ORP) Determination***

14.4.1. ***Measure the pH and ORP of a solid sample.***

14.4.1.1. ***Information regarding determination of pH in soils and waste samples is provided in SOP-M736.***

14.4.2. ***Adjust the ORP measurement based on reference electrode correction factor to yield Eh values.***

14.4.3. ***Plot the pH and Eh values to obtain the initial indication of the solid sample's reducing/oxidizing nature.***

14.4.3.1. ***An example of the Eh/pH phase diagram is shown in Appendix C.***

14.5. Measure 2.50 ± 0.10 g (wet weight) of the homogenized solid sample into a clean beaker. ***Record the mass of sample used to the nearest 0.01 g.***

14.5.1. ***For MB/LCS/LCSD, measure exactly 2.50 g of washed sea sand. Record the washed sea sand identification number.***

14.5.2. ***For soluble MS/MSD, measure exactly 2.50 g of solid sample in each analytical batch selected for spiking.***

14.5.3. ***For insoluble MS/MSD, measure exactly 2.50 g of solid sample in each analytical batch selected for spiking.***

14.6. ***Add the appropriate amount of the spike standard solution or solid to all matrix spikes and laboratory control samples.***

14.6.1. ***Add 1.0 mL of the 100-ppm spike standard solution to all soluble matrix spikes and laboratory control samples.***

14.6.2. ***Add 15 mg of the spike standard solid to all insoluble matrix spikes.***

14.7. Add 50 ± 1 mL of the digestion solution, ***854*** mg of $MgCl_2 \cdot 6H_2O$, and 0.5 mL of phosphate buffer to the sample.

14.7.1. For analytical techniques that can correct for oxidation/reduction of Cr, the addition of Mg^{2+} is optional.

14.8. ***Place one stir bar into the beaker and cover the beaker with a watch glass.***

14.9. Stir the sample continuously at ambient temperature for a minimum of 5 minutes.

14.10. Heat the sample to 90−95°C, and maintain the sample at 90−95°C for at least 60 minutes with continuous stirring.

14.11. Turn off the heat and cool the digestate to ambient temperature with continuous stirring.

14.12. ***Assemble a vacuum filtration apparatus with a clean 0.45-µm membrane filter.***

14.12.1. ***Place a graduated cylinder inside the filtration flask to collect filtrate.***

14.13. Transfer the contents in the beaker ***quantitatively to the filtration apparatus and apply vacuum to filter the digestate***.

14.14. ***Collect the filtered digestate in the graduated cylinder.***

14.14.1. ***Transfer the membrane filter and the remaining solids into a clean specimen container and label appropriately.***

14.14.2. Store the filtered solid ***at 0–6°C*** for possible use in assessing low Cr(VI) recoveries.

14.15. Rinse the beaker with 3 successive portions of reagent water ***and transfer the rinsates to the filtration apparatus. Collect the filtered rinsates in the same graduated cylinder***.

14.16. ***Transfer the contents in the graduated cylinder into a clean specimen container and labeled appropriately. Rinse the graduated cylinder and collect the rinsate in the same specimen container.***

14.17. ***Place one stir bar into the specimen container and stir the digestate continuously.***

14.18. Slowly add 5.0-M HNO_3 solution to the digestate dropwise with continuous stirring, and adjust to the desired pH range ***according to the determinative method***.

14.18.1. For EPA Method 7196A, the desired pH range is 7.5 ± 0.5.

14.18.2. For EPA Method 7199, the desired pH range is 9.0 ± 0.5.

14.19. ***Monitor the pH of the digestate using a pH meter.***

14.19.1. ***If the pH of the digestate is not within the desired pH range, discard the digestate and re-digest the solid sample.***

14.19.2. If overshooting the desired pH range occurs repeatedly, prepare a more diluted HNO_3 solution and repeat the digestion procedure.

14.20. If a flocculent precipitate forms in the digestate ***after adding HNO_3 solution, assemble a vacuum filtration apparatus with a clean 0.45-µm membrane filter and apply vacuum to*** filter the digestate.

14.20.1. If the 0.45-µm membrane filter becomes clogged, use a clean ***0.7-µm or 1.0-µm glass microfiber filter*** to prefilter the digestate.

14.21. Remove and rinse the stir bar. Collect the rinsate in the ***same specimen container***.

14.22. ***Quantitatively transfer the contents from the specimen container into a clean volumetric flask.***

14.23. Adjust the final volume of the digestate to 100 mL with reagent water ***and label appropriately***. The digestate may now be analyzed using the appropriate determinative method.

14.24. ***Thoroughly document all aspects of the digestion in a hexavalent chromium sample preparation logbook.***

15. ►CALCULATIONS

15.1. ***The dry weight (wet-based solids content) for a sample is calculated as follows:***

$$C_d = \frac{M_2 - M_0}{M_1 - M_0} \times 100$$

where: C_d = ***dry weight (wet-based solids content) in %.***
M_2 = ***mass of weighing dish and oven-dried sample in g.***
M_0 = ***mass of weighing dish in g.***
M_1 = ***mass of weighing dish and as-received sample in g.***

15.2. ***Refer to the determinative method for additional calculations.***

16. ►METHOD PERFORMANCE

16.1. ***A demonstration of analytical capability shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.***

16.2. ***Calibration protocols specified in Section 13., "Calibration and Standardization," shall be followed.***

16.3. ***Proficiency test sample results shall be used to evaluate the ability to produce accurate results.***

17. POLLUTION PREVENTION

17.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Calscience's Health, Safety, and Respiratory Protection Manual. In general, protective eyewear (e.g. safety glasses or goggles), and protective apparel (e.g. lab coats) and gloves are required to be worn when handling chemicals.

17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:

17.3.1. A NIOSH approved air purifying respirator with cartridges appropriate for the chemical handled.

17.3.2. Extended length protective gloves.

17.3.3. Face shield.

17.3.4. Full-length laboratory apron.

17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.

17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles. Alternatively, the Coordinator may utilize a self-contained breathing apparatus or other supplied air system if appropriately trained and approved by the Health and Safety Manager.

17.6. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

18. ►DATA ASSESSMENT AND ACCEPTANCE CRITERIA

18.1. The acceptance criteria for LCS/LCSD compounds ***are predetermined***. The lower and upper acceptance limits for %REC of each LCS/LCSD compound are ***80% and 120%, respectively. The RPD is ≤20%***. All LCS/LCSD compounds must be within acceptance limits.

18.1.1. If the LCS and/or LCSD %REC is outside of the acceptance limits high, the RPD is within acceptance limits, and all target analytes in the associated samples are not detected, the sample data can be reported without qualification.

18.1.2. The LCSD is only reported when the MS/MSD is unacceptable due to matrix interference effects, or when the LCS/LCSD is used in place of MS/MSD due to insufficient sample quantity.

18.2. Ideally, the concentration of target analyte in an MB should be less than the respective reporting limit (RL). If the concentration of target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs are as follows:

18.2.1. If the target analyte is found in the MB but not in the associated samples, report the sample and MB data without qualification.

18.2.2. If the target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination.

Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-digested and/or re-analyzed.

18.2.3. If ***hexavalent chromium*** is found in the MB, no positive sample results should be reported unless the concentration of the compound in the sample exceeds 10 times the MB level. Report the data with qualification indicating the analyte was present in the corresponding MB.

18.3. The acceptance criteria for MS/MSD compounds ***are predetermined. The lower and upper acceptance limits for %REC of each MS/MSD compound are 75% and 125%, respectively. The RPD is ≤25%.***

18.3.1. When the %REC and RPD of the MS/MSD compounds are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.

18.3.2. If the %REC and/or RPD of the MS/MSD compounds are not within the established acceptance limits, the analytical system performance shall be suspect.

18.4. Matrix effects or poor instrument performance/technique typically cause unacceptable %REC values. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.

18.5. ***Refer to the determinative method for additional data assessment and acceptance criteria.***

18.6. Additional information regarding internal quality control checks is provided in SOP-T020.

18.7. ***For EPA Method 7196A, all concentrations shall be reported in mg/kg (ppm) of Cr(VI) for soil and solid waste samples. For EPA Method 7199, all concentrations shall be reported in µg/kg (ppb) of Cr(VI) for soil and solid waste samples.***

18.8. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

19. CORRECTIVE ACTIONS

19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.

19.2. The Operations Manager, Project Manager, Quality Control Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If

previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.

19.3. Corrective actions are generally of two types, immediate and long-term actions.

19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.

19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented in the Corrective Action Record. Examples of this type of action include:

19.3.2.1. Remedial training of staff in technical skills, technique or implementation of operating procedures.

19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.

19.3.2.3. Revision of standard operating procedures.

19.3.2.4. Replacing personnel, as necessary.

19.4. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:

19.4.1. Define the problem.

19.4.2. Assign responsibility for investigating the problem.

19.4.3. Investigate and determine the cause of the problem.

19.4.4. Assign and accept responsibility for implementing the corrective action.

19.4.5. Determine effectiveness of the corrective action and implement correction.

19.4.6. Verify that the corrective action has eliminated the problem.

19.5. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

20. ►CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1. ***Refer to the determinative method for contingencies for out-of-control or unacceptable data.***

20.2. Out-of-control data are reviewed and verified by the technical director of the appropriate department. All samples associated with an unacceptable QC set are then subject to reanalysis, depending upon the QC type in question.

21. WASTE MANAGEMENT

21.1. The proper disposal of analytical samples and laboratory wastes is not only good laboratory practice, but also regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated, and either returned to the client (preferable) or placed into the proper laboratory waste stream.

21.2. Unused or remaining soil or liquid samples and all other solid or liquid wastes resulting from our laboratory operations are considered hazardous for disposal purposes.

21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal.

21.4. Each specific laboratory area shall maintain clearly labeled waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste.

21.5. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly.

21.6. In order to maintain accountability for all samples received by Calscience, when a sample is used in its entirety for analysis, the empty container(s) are returned to Sample Control for placement in analytical storage.

21.7. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Wastes."

22. ►REFERENCES

22.1. *Alkaline Digestion for Hexavalent Chromium*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1A, Method 3060A, USEPA, Revision 1, December 1996.

22.2. ***Quality Control, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter One, USEPA, Revision 1, July 1992.***

22.3. ***Choosing the Correct Procedure, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Two, USEPA, Revision 4, February 2007.***

22.4. ***Inorganic Analytes, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Three, USEPA, Revision 4, February 2007.***

23. ►TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

23.1. Appendix ***A***: Alkaline Digestion Flowchart.

23.2. Appendix ***B***: Quality Control Flowchart.

23.3. Appendix ***C***: Eh/pH Phase Diagram.

24. ►MODIFICATIONS

24.1. ***The following modifications from EPA Method 3060A are noted.***

24.1.1. ***Reagents and Standards***

24.1.1.1. ***Magnesium chloride hexahydrate is added to a solid sample to correct for oxidation/reduction of Cr in stead of anhydrous magnesium chloride.***

24.1.2. ***Quality Control***

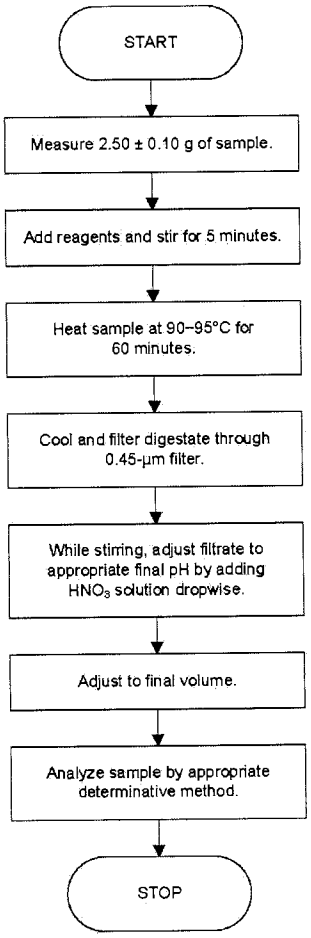
24.1.2.1. ***For LCS/LCSD, the spike standard solution is added to the matrix, not the digestion solution.***

Appendix A

ALKALINE DIGESTION FLOWCHART

Calscience Environmental Laboratories, Inc.

Alkaline Digestion Flowchart

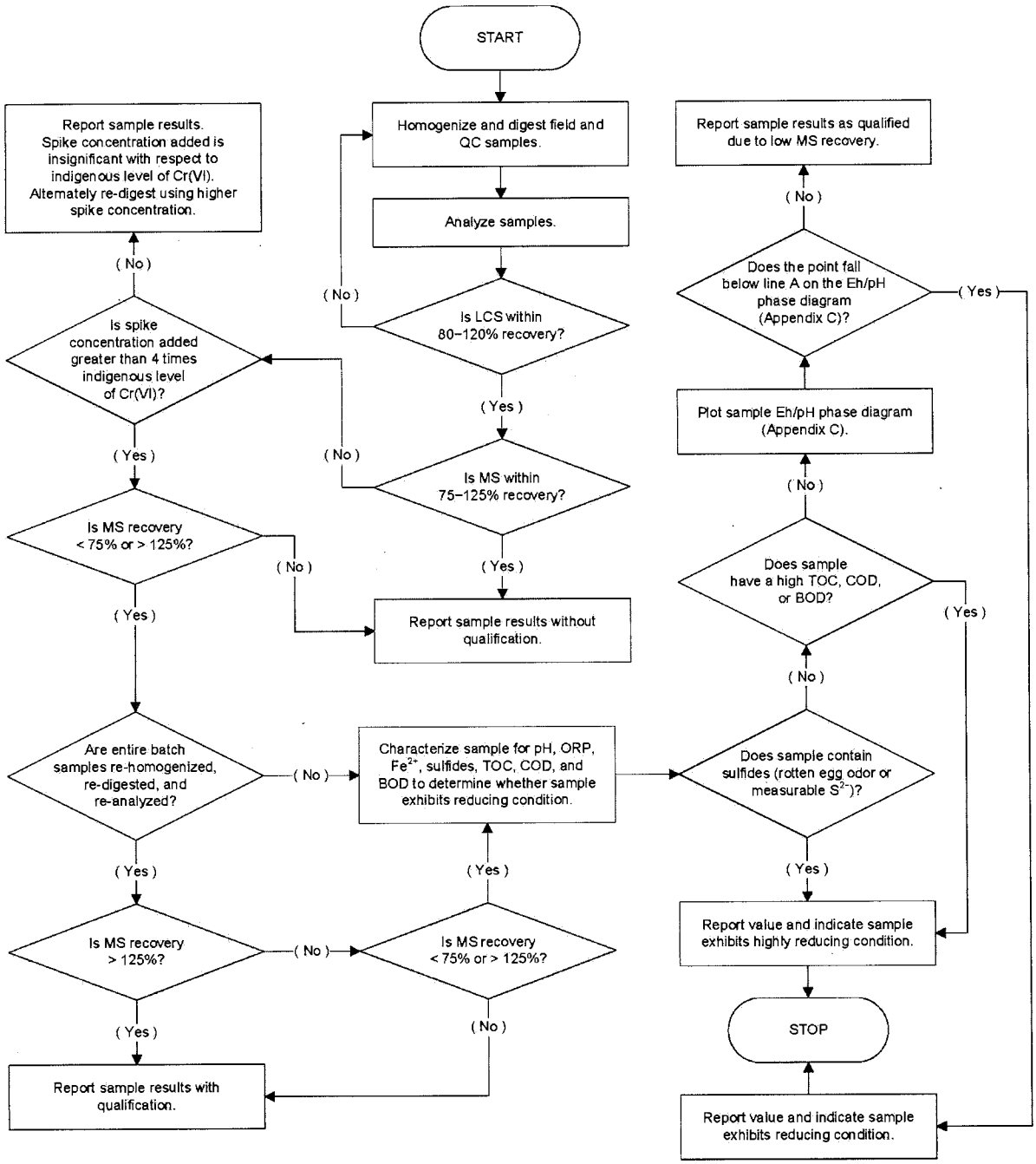


Appendix B

QUALITY CONTROL FLOWCHART

Calscience Environmental Laboratories, Inc.

Quality Control Flowchart



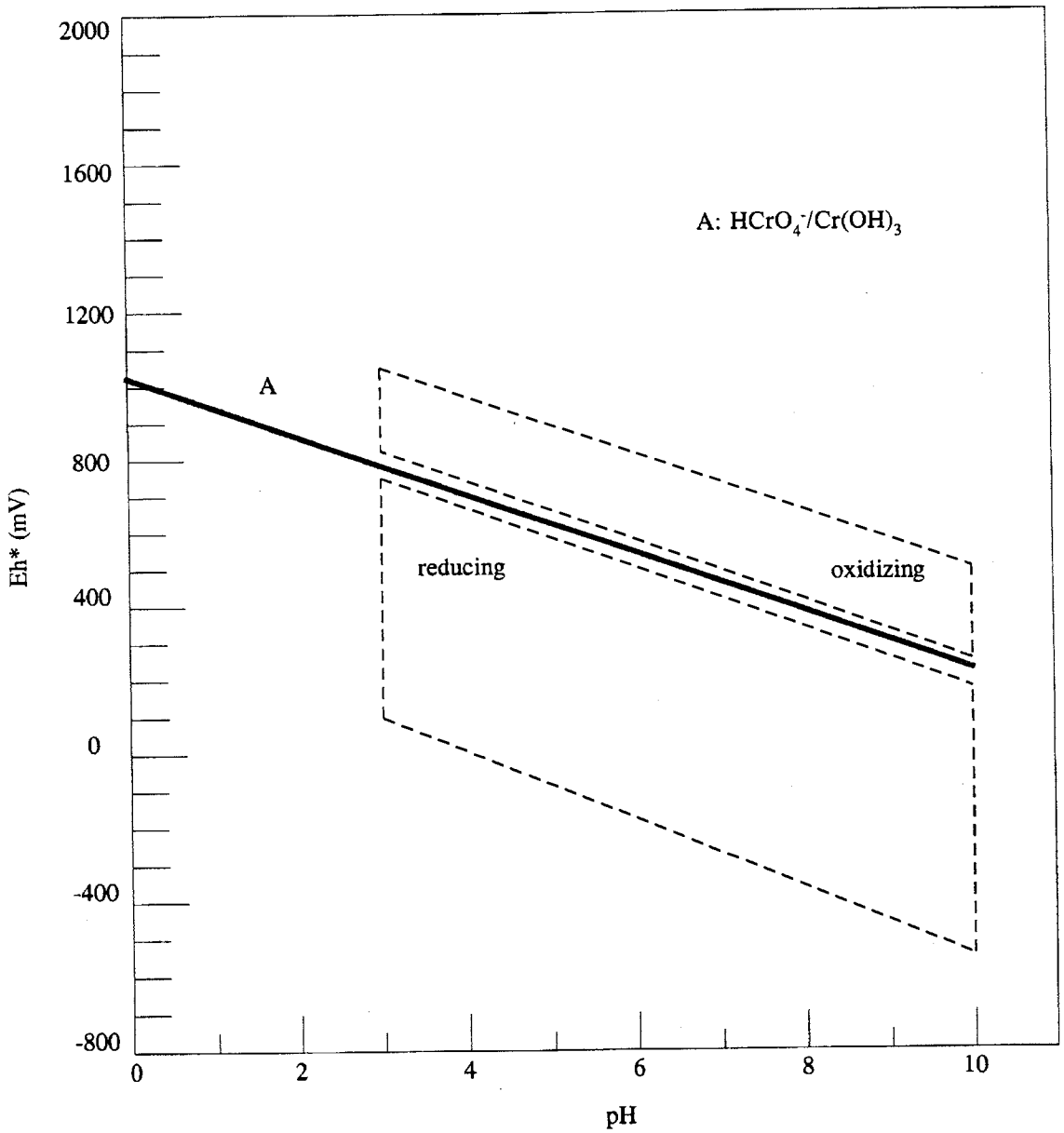
Appendix C

EH/PH PHASE DIAGRAM

Calscience Environmental Laboratories, Inc.

Eh/pH Phase Diagram

The dashed lines define Eh-pH boundaries commonly encountered in soils and sediments.



* Note the Eh values plotted on this diagram are corrected for the reference electrode voltage: 244 mV units must be added to the measured value when a separate calomel electrode is used, or 199 mV units must be added if a combination platinum electrode is used.

Title : **EPA METHOD 1311, TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)**

Document No. : SOP-M226
Revision No. : 2.4
Supersedes : 2.3

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Revision 2.4 changes are noted in bold italicized typeface and preceded by a "►" marker.

APPROVED FOR RELEASE BY: _____ 12/02/08
MANAGEMENT **DATE**

_____ 12/02/08
QA DEPARTMENT **DATE**

ANNUAL SOP REVIEW

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

1. METHOD IDENTIFICATION

1.1. EPA Method 1311, Toxicity Characteristic Leaching Procedure (TCLP).

2. APPLICABLE MATRICES

2.1. This method is applicable to liquid, solid and multiphasic wastes.

3. DETECTION LIMITS

3.1. ►Refer to the determinative ***(analytical)*** method for detection limit data.

4. SCOPE AND APPLICATION

4.1. EPA Method 1311 determines the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

4.1.1. The TCLP is designed to simulate the leaching a waste will undergo if disposed of in a sanitary landfill. The test is designed to simulate the leaching that takes place in a sanitary landfill only.

4.1.2. The approximate preparation time is 20 hours.

4.2. A subsample of a waste is extracted with the appropriate buffered acetic acid solution for 18 ± 2 hours. The TCLP extract (the extract obtained from the TCLP) is then analyzed to determine if any of the thresholds established for the 40 Toxicity Characteristic (TC) constituents (see Appendix A) have been exceeded or if the treatment standards established for the constituents listed in 40 CFR §268.40 have been met under the Land Disposal Restrictions (LDR) regulations.

4.2.1. If the TCLP extract contains any one of the TC constituents in an amount greater than or equal to the concentrations specified in 40 CFR §261.24, the waste possesses the characteristic of toxicity and is a hazardous waste.

4.2.2. If the TCLP extract contains constituents in an amount exceeding the concentrations specified in 40 CFR §268.40, the treatment standard for that waste has not been met, and further treatment is necessary prior to land disposal.

4.3. If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

4.4. If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

4.5. If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

4.6. ►This method is restricted to use by or under the supervision of analysts/***technicians*** experienced in the use of the instruments and apparatus required to execute the procedure.

5. METHOD SUMMARY

5.1. A preliminary evaluation is performed to determine if the waste is to be treated as a liquid or a solid.

5.1.1. For liquid wastes (i.e., wastes containing less than 0.5% dry solid material), the waste, after filtration through a 0.6−0.8-µm glass fiber filter, is defined as the TCLP extract.

5.1.2. For wastes containing greater than or equal to 0.5% solids, the liquid phase is separated from the solid phase by filtration and stored for later analysis. The solid phase is processed as follows:

5.1.2.1. The particle size of the solid phase is reduced, if necessary.

5.1.2.2. The solid phase is then extracted with an amount of TCLP extraction fluid equal to 20 times the mass of the solid phase for 18 ± 2 hours. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste.

5.1.2.2.1. A special extractor vessel is used for TCLP extraction when testing for volatile analytes.

5.1.2.3. Following extraction, the TCLP extract is separated from the solid material by filtration through a 0.6−0.8-µm glass fiber filter.

5.1.2.4. If compatible (i.e. multiple phases will not form on combination), the initial liquid phase of the waste is added to the TCLP extract, and these are analyzed together.

5.1.2.5. If incompatible, the initial liquid phase and the TCLP extract are analyzed separately, and the results are mathematically combined to yield a volume-weighted average concentration.

5.2. ►Refer to the appropriate determinative methods including EPA Methods 6010***C*** (metals), ***6020A (metals)***, 7196A/7199 (hexavalent chromium), 7470A/7471***B*** (mercury), 8081***B***, ***8021B***, 8151A, 8260B, and 8270***D*** for the analysis of the TCLP extract to identify and quantify the inorganic and organic analytes.

6. DEFINITIONS

6.1. Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

6.2. Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

6.3. 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 NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

6.4. Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

6.5. Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

6.6. Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

6.7. Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

6.8. Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

6.9. Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

6.10. Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.

6.11. Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

6.12. Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

6.13. Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

6.14. Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

6.15. Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

6.16. Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

6.17. Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

6.18. Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.

6.19. 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.

6.20. 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.

6.21. Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence.

6.22. Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated

observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

6.23. Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

6.24. Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

6.25. Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

6.26. Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

7. INTERFERENCES

7.1. Refer to the determinative method for potential interferences.

8. SAFETY

8.1. It is recommended that all filtrations be performed in a fume hood vented to the exterior of the laboratory.

8.2. For the safety of the analyst, cracked or broken glassware should be immediately discarded into a broken glassware receptacle. Broken glassware shall not be used in any step of the extraction.

8.3. ►To ensure the safety of the analyst during any possible emergency situation, ***it is recommended that*** chemists do not perform extractions alone. Another chemist shall be present during any extraction process.

8.4. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current version of Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats are required to be worn in all designated laboratory areas. Protective gloves shall be worn when handling chemicals.

8.5. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

9. EQUIPMENT AND SUPPLIES

9.1. Agitation apparatus:

9.1.1. Rotary agitator, capable of rotating extraction vessels in an end-over-end fashion at 30 ± 2 rpm, Associated Design and Manufacturing Company 3740-12-BRE Rotary Agitator or equivalent.

9.2. Zero-Headspace Extraction (ZHE) device:

9.2.1. ZHE vessel, 500−600-mL internal volume, type 316 stainless steel, consisting a 90−110-mm diameter glass fiber filter, a piston, Teflon or Viton O-rings, a top flange with a liquid inlet/outlet valve, and a bottom flange with a pressure relief valve and a pressure gauge, capable of withstanding 50 psi pressure requirement for phase separation, Associated Design and Manufacturing Company 3745-ZHE Zero Headspace Extractor or equivalent.

9.2.1.1. The ZHE device is used only when the waste is being tested for the mobility of volatile analytes.

9.2.1.2. The vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel.

9.2.1.3. The Viton O-rings, if any, in the device should be replaced frequently.

9.2.1.4. If it takes more pressure (> 15 psi) to move the piston, the Teflon or Viton O-rings should be replaced.

9.2.1.5. The device should be checked for leaks after every extraction by pressurizing the device to 50 psi, allowing it to stand unattended for 1 hour, and rechecking the pressure. If pressure is lost, check all fittings and replace the O-rings, if necessary. If leakage problems cannot be solved, replace the device.

9.2.2. Filter, 0.7-µm effective pore size, 90-mm diameter, borosilicate glass fiber without binder resin, Associated Design and Manufacturing Company SHWF-5044-0001 Glass Fiber Filter or equivalent.

9.2.3. Pressurizing gas, high purity nitrogen.

9.2.4. Syringe, 600-mL capacity, gas-tight, stainless steel, Associated Design and Manufacturing Company 3775-GTS Gas-Tight Syringe or equivalent.

9.2.4.1. The gas-tight syringe is used to transfer extraction fluid to and collect filtrate from the ZHE device.

9.3. Bottle extraction device:

9.3.1. Bottle extraction vessel, 2-L, borosilicate glass, polytetrafluoroethylene (Teflon), or high density polyethylene (HDPE).

9.3.1.1. Borosilicate glass and Teflon bottles may be used for evaluating the mobility of both organic and inorganic components.

9.3.1.2. HDPE bottles may be used only for evaluating the mobility of metals.

9.4. Filtration system:

9.4.1. Filter holder, 1.5-L minimum internal volume, type 316 stainless steel, capable of supporting a 142-mm diameter filter and withstanding 50+ psi pressure requirement for phase separation, Millipore Corporation YT30 142 HW Hazardous Waste Filtration System or equivalent.

9.4.1.1. Vacuum filtration can only be used for wastes with low solids content (< 10%), and for highly granular, liquid-containing wastes.

9.4.1.2. All other types of wastes should be filtered using positive pressure filtration.

9.4.2. Filter, 0.6−0.8-µm effective pore size, 142-mm diameter, borosilicate glass fiber without binder resin, Millipore Corporation AP40 142 50 Glass Fiber Filter or equivalent.

9.4.2.1. Do not use pre-filters.

9.4.2.2. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1:1 HNO_3 followed by three consecutive rinses with reagent water (a minimum of 1 L per rinse is recommended).

9.4.2.3. Glass fiber filters are fragile and should be handled with care.

9.4.3. Pressurizing gas, high purity nitrogen.

9.5. Vacuum filtration apparatus:

9.5.1. Vacuum apparatus.

9.5.2. Filter holder, ceramic, capable of supporting a 90-mm diameter filter.

9.5.3. Filtration flask, 500-mL, glass.

9.5.4. Filter, 0.6−0.8-µm effective pore size, 90-mm diameter, borosilicate glass fiber without binder resin, Whatman 1810-090 Acid Treated Low Metal TCLP Filter or equivalent.

9.5.4.1. Do not use pre-filters.

9.5.4.2. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1:1 HNO_3 followed by three consecutive rinses with reagent water.

9.5.4.3. Glass fiber filters are fragile and should be handled with care.

9.6. Syringe filtration apparatus:

9.6.1. ►Syringe, ***10-mL, polypropylene, eccentric tip***, disposable, ***BD Lab Syringe P/N 305462 or equivalent***.

9.6.2. Filter, 0.7-µm effective pore size, 30-mm diameter, borosilicate glass microfiber, polypropylene housing, disposable, National Scientific Company F2500-18 Target Syringe Filter or equivalent.

9.6.2.1. Do not use pre-filters.

9.6.2.2. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1:1 HNO_3 followed by three consecutive rinses with reagent water.

9.7. ►Filtrate containers:

9.7.1. ***Digestion tubes (vials), 3-oz (90-mL), 93-mm × 33-mm ID, graduated, snap closure, with hinge lids, polypropylene, disposable, Capitol Vial P/N 03EDM31/33 or equivalent.***

9.7.1.1. The 3-oz ***polypropylene*** vial is used to collect filtrate for metals analysis.

9.7.2. Bottles, 500-mL, amber glass, with Teflon-lined closures.

9.7.2.1. The 500-mL amber glass bottle is used to collect filtrate for semi-volatile solvent extraction.

9.7.3. Erlenmeyer flasks, 250-mL and 500-mL, glass.

9.7.3.1. The flask is used to collect filtrate for semi-volatile solvent extraction.

9.7.4. Vials, 4-mL, ***15-mm × 45-mm***, clear glass, ***with screw cap and*** Teflon-lined ***septa***, disposable.

9.7.4.1. The 4-mL clear glass vial is used to collect filtrate for volatile analysis.

9.7.5. Vials, 40-mL, ***28-mm × 95-mm***, amber glass, ***with screw cap and*** Teflon-lined ***septa***, disposable.

9.7.5.1. The 40-mL amber glass vial is used to collect filtrate for volatile analysis.

9.8. Grinding apparatus.

9.9. Sieve, ⅜-mesh (9.5-mm), stainless steel or Teflon.

9.10. Graduated cylinder, 100-mL, glass, Class A.

9.11. pH meter, accurate to ± 0.05 units at 25°C, equipped with ATC probe or equivalent, Fisher Scientific Accumet Basic AB15 pH Meter or equivalent.

9.12. pH indicator paper, narrow range. pH range should include the desired pH.

9.13. Beaker, 400-mL or 500-mL, glass.

9.14. Watch glass, appropriate diameter to cover beaker.

9.15. Stirring rod, glass.

9.16. Thermometer.

9.17. Hot plate, capable of maintaining 50°C.

9.18. Balance, top loading, capable of weighing to the nearest 0.01 g.

9.19. Pipetters, 10–100-µL, 100–1000-µL, 0.5–5.0-mL, and 1–10-mL, adjustable, with disposable tip.

9.20. ►***Dispensers, 1–10-mL, 2.5–25-mL, and 5–50-mL, adjustable.***

9.21. Solids content determination apparatus:

9.21.1. Watch glass, 90-mm diameter or larger.

9.21.2. Drying oven, capable of maintaining 104 ± 1°C.

9.21.3. Desiccator.

9.21.4. Forceps, blunt curved-tip, stainless steel.

10. ►REAGENTS AND STANDARDS

10.1. Reagents

10.1.1. Reagent water, interferant free, distilled or deionized.

10.1.2. Chips, Teflon.

10.1.3. Beads, glass.

10.1.4. Hydrochloric acid, HCl, ***36.5–38.0% (v/v)*** or 12-N, ***concentrated, colorless to pale yellow liquid***, trace metals grade or equivalent.

10.1.5. Hydrochloric acid, HCl, 1-N.

10.1.5.1. Prepare the 1-N HCl solution by diluting 8.3 mL of 12-N HCl to 100 mL with reagent water.

10.1.6. Nitric acid, HNO_3, ***68.0–70.0% (v/v)***, concentrated, ***clear to yellow liquid***, trace metals grade for equivalent.

10.1.7. Nitric acid, HNO_3, 1:1 ***or 1+1*** (v/v).

10.1.7.1. Prepare the 1:1 HNO_3 solution by ***slowly adding 1.25 L of concentrated HNO_3 to 1.25 L*** of reagent water.

10.1.8. Acetic acid, CH_3COOH, ***99.4% (v/v)***, glacial, ***clear, colorless liquid***, trace metals grade or equivalent.

10.1.9. Sodium hydroxide, NaOH, ***white pellets and granules***, reagent grade or equivalent.

10.1.10. Sodium hydroxide, NaOH, 1-N.

10.1.10.1. Prepare the 1-N NaOH solution by dissolving 160 g of NaOH in reagent water and dilute to 4 L with additional reagent water.

10.1.11. TCLP extraction fluid #1, pH at 4.93 ± 0.05.

10.1.11.1. Prepare the TCLP extraction fluid #1 by diluting ***120*** mL of glacial acetic acid and ***1380*** mL of 1-N NaOH solution to 20 L with reagent water.

10.1.11.2. Adjust the pH with additional glacial acetic acid or 1-N NaOH solution.

10.1.11.3. Record the volumes of the reagents to the nearest 1 mL in the preparation logbook.

10.1.12. TCLP extraction fluid #2, pH at 2.88 ± 0.05.

10.1.12.1. Prepare the TCLP extraction fluid #2 by diluting ***60*** mL of glacial acetic acid to ***10*** L with reagent water.

10.1.12.2. Adjust the pH with additional glacial acetic acid.

10.1.12.3. Record the volumes of the reagents to the nearest 1 mL in the preparation logbook.

10.1.13. All reagents must be inspected and documented ***in the Solvent/Standard Verification Logbook*** prior to use.

10.2. Standards

10.2.1. ***Refer to the specific SOPs of the determinative methods for standards.***

11. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

11.1. Do not add preservation chemicals to samples prior to TCLP extraction.

11.2. Refer to the specific SOPs of the determinative methods for additional information on sample collection, preservation, containers and holding times.

11.3. Refer to the specific SOPs of the determinative methods for extract preservation and holding times post TCLP extraction.

11.3.1. For hexavalent chromium, Cr(VI), determinations, no preservation chemicals are required. The TCLP extracts must be analyzed within 24 hours.

11.3.2. For other metallic analyte determinations, acidify TCLP extracts or portions of TCLP extracts with 1:1 HNO_3 to pH < 2.

11.3.2.1. If precipitation is observed upon addition of HNO_3 to a small aliquot of the TCLP extract, the remaining portion of the TCLP extract for metals analyses shall not be acidified, and the TCLP extract must be analyzed within 24 hours.

11.3.3. For organic analyte determinations, do not allow TCLP extracts or portions of TCLP extracts coming into contact with the atmosphere (i.e., no headspace) to prevent analyte losses.

11.3.4. TCLP extracts should be prepared within the appropriate period to allow for subsequent solvent extractions and determinative analyses (see Appendix B).

12. QUALITY CONTROL

12.1. ►Event Based Quality Control (***LCS/LCSDs and*** MBs)

12.1.1. Event based quality control consists of QC samples prepared and processed with each preparatory event. This consists of ***a laboratory control sample and laboratory control sample duplicate (LCS/LCSD) and*** a method blank (MB).

12.1.2. Aqueous Samples

12.1.2.1. ***One LCS consisting of the specified analytes spiked into the TCLP extract of clean reagent water is required every day for every batch of 20 samples per matrix or portion thereof, whichever is more frequent. The LCSD is handled identically to the LCS.***

12.1.2.2. One MB consisting of the TCLP extract of clean reagent water is required every day for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

12.1.3. Solid Samples

12.1.3.1. ***One LCS consisting of the specified analytes spiked into the TCLP extract of clean Teflon chips or glass beads is required every day for every batch of 20 samples per matrix or portion thereof, whichever is more frequent. The LCSD is handled identically to the LCS.***

12.1.3.2. One MB consisting of the TCLP extract of clean Teflon chips or glass beads is required every day for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

12.1.4. The MB/***LCS/LCSD*** is processed concurrently with the associated samples. In the processing of the MB/***LCS/LCSD***, reagents and procedures identical to those for actual samples are used.

12.2. Matrix Based Quality Control (MS/MSDs)

12.2.1. Matrix based quality control consists of QC samples prepared and processed using actual environmental samples. This consists of a matrix spike and matrix spike duplicate (MS/MSD).

12.2.2. One MS consisting of the TCLP extract of the actual sample matrix spiked with known concentrations of specific target analytes is required for every batch of 20 samples per matrix or portion thereof. The MSD is handled identically to the MS.

12.2.3. ►The sample which ***will be*** spiked for the MS/MSD is processed concurrently with the associated samples. In the processing of the MS/MSD, reagents and procedures identical to those for actual samples are used.

12.3. Refer to the specific SOPs of the determinative methods for additional information on quality control.

13. CALIBRATION AND STANDARDIZATION

13.1. Top Loading Balance

13.1.1. Calibrate the top loading balance at 1 g and 100 g using Class 2 weights.

13.1.2. Calibration shall be within ± 2% at 1 g (± 0.02 g) and at 100 g (± 2 g). If the values are not within these limits, recalibrate the balance.

13.2. Thermometer

13.2.1. Calibrate the thermometer using an NIST certified thermometer. The calibration procedure shall adhere to the current revision of SOP-T042, "Thermometer Calibration."

13.3. Pipetter

13.3.1. ***Calibrate the pipetter according to the procedure outlined in the Pipetter Calibration Check Logbook.***

13.4. ***Dispenser***

13.4.1. ***Calibrate the dispenser according to the procedure outlined in the Dispenser Calibration Check Logbook.***

13.5. pH Meter

13.5.1. Calibrate the pH meter daily prior to sample analysis at pH of 4.00, 7.00, and 10.00 using fresh buffer solutions and according to the instrument manufacturer's recommended procedures.

13.5.2. Verify the calibration with fresh second source buffer standard. The second source standard shall not differ from its expected value by more than 0.05 pH units. If this criterion is not met, recheck calibration or effect corrective action.

13.5.3. The theoretical slope shall be within 90−105% for analysis to proceed. If this criterion is not met, determine the cause of the problem, effect corrective action, and recalibrate, if necessary.

14. ►PROCEDURE

14.1. Solids Content Determination

14.1.1. Weigh a clean watch glass and a clean or acid-washed glass fiber filter. Record the mass to the nearest ***0.01*** g.

14.1.2. Weigh ***100–105*** g of sample. Record the mass to the nearest ***1*** g.

14.1.3. Assemble a vacuum filtration apparatus (Section ***9.5.***) with the pre-weighted filter and apply vacuum. Quantitatively transfer the sample to the filter holder. Spread the sample evenly over the surface of the filter. Continue to apply vacuum to filter the sample until all traces of water have been removed.

14.1.4. Remove the filter from the filter holder with forceps at the edge of the filter and carefully place on the pre-weighted watch glass without touching the solid residue or inverting the filter.

14.1.4.1. If sample result is to be reported on a dry-weight basis, dry the solid residue in 103–105°C oven for a minimum of one hour. Cool in the desiccator and weigh. Repeat the drying cycle until a constant mass is obtained or until the mass difference is within ± 1% from the previous weighing.

14.1.4.2. Oven-dried solid residue should not be used for subsequent TCLP extraction.

14.1.5. Weigh the sample including the filter and watch glass. Record the mass to the nearest ***0.01*** g. The formula for calculating the solids content is listed in Section 15.1.

14.1.6. Sample is deemed aqueous if the solids content is < 0.5%. Sample is deemed solid if the solids content is ≥0.5%.

14.2. TCLP Extraction for Metals or Semi-Volatile Analysis

14.2.1. TCLP Extraction Fluid Determination

14.2.1.1. Transfer 5.0 g of sample to a beaker.

14.2.1.2. Add 96.5 mL of reagent water and 3.5 mL of 1-N HCl to the beaker and stir briefly. Cover the beaker with a watch glass.

14.2.1.3. Place the beaker on a hot plate, heat to 50°C, and hold at 50°C for 10 minutes.

14.2.1.4. Cool the solution to room temperature and check the pH of sample with pH paper. If the pH paper shows a reading near 5, then the pH should be verified with a pH meter.

14.2.1.5. If the pH is < 5.0, use TCLP extraction fluid #1. If the pH is ≥ 5.0, use TCLP extraction fluid #2.

14.2.2. Measure the pH of ***the TCLP extraction fluid*** with a pH meter prior to use. Record the pH to the nearest 0.01 pH unit.

14.2.2.1. The pH of ***the TCLP extraction fluid*** is used to monitor ***for impurities and to verify integrity***.

14.2.3. Assemble a filtration system (Section 9.4.) with a clean or acid-washed 142-mm glass fiber filter according to the manufacturer's instructions to

filter sample. ***A vacuum filtration apparatus (Section 9.5.) may be utilized instead of a filtration system to filter sample.***

14.2.4. Measure sufficient amount of sample. Record the amount to the nearest ***1 mL or 1 g***.

14.2.4.1. For aqueous sample, ***measure exactly 100 mL*** of sample. ***Assume the density of aqueous sample is 1 g/mL.***

14.2.4.1.1. For MB/***LCS/LCSD, measure exactly 100 mL*** of clean reagent water.

14.2.4.1.2. For MS/MSD, ***measure exactly 100 mL*** of aqueous sample in each analytical batch selected for spiking.

14.2.4.2. For solid sample with 100% solids content, ***measure 100–105*** g of sample. For other solid and multiphasic sample, use the solids content (see Section 14.1.) to determine the amount of sample required. The formula for calculating the amount of sample is listed in Section 15.2.

14.2.4.2.1. For MB/***LCS/LCSD, measure 100–105 g*** of clean Teflon chips or glass beads.

14.2.4.2.2. For MS/MSD, ***measure 100–105 g (or the amount determined from calculation)*** of solid or multiphasic sample in each analytical batch selected for spiking.

14.2.4.3. The amount of sample required for analytical determination is established by the determinative method or the project's Quality Assurance Project Plan (QAPP).

14.2.5. Allow the solid phase of sample to settle. ***For solid sample with 100% solids content, proceed to Section 14.2.9.***

14.2.5.1. Centrifugation may be used as an aid to separate the liquid and solid phases.

14.2.5.2. If sample is centrifuged, decant and filter the liquid phase through the filtration system ***or vacuum filtration apparatus*** followed by filtration of the solid phase through the same filtration system ***or vacuum filtration apparatus***.

14.2.6. With the filter in place, quantitatively transfer both liquid and solid phases of sample to the filter holder. Spread the sample evenly over the surface of the filter.

14.2.7. Gradually apply pressure of 1–10 psi until pressurizing gas moves through the filter. ***If a vacuum filtration apparatus is used, gradually apply vacuum.***

14.2.7.1. If the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute

interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

14.2.7.2. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment.

14.2.7.3. If the pressurizing gas begins to move through the filter, or if liquid flow has ceased at 50 psi, stop the filtration.

14.2.8. Collect the filtrate in a clean filtrate container.

14.2.8.1. For aqueous sample, the filtrate is the TCLP extract.

14.2.8.1.1. Measure the pH of the filtrate with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4.

14.2.8.2. For solid and multiphasic sample, the filtrate is the TCLP extract of the liquid phase.

14.2.8.2.1. If compatible, combine the filtrates of both liquid and solid phases. Measure the pH of the combined filtrate with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4.

14.2.8.2.2. If not compatible, measure the pH of the filtrate of each phase with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4., analyze the filtrates separately, and combine the results mathematically.

14.2.9. Evaluate the particle size of the solid phase (i.e. the material in the filter holder).

14.2.9.1. If the solids are prepared for metals analyses, reduce the particle size of the solid by crushing, cutting, or grinding so that it can pass through a 9.5-mm sieve. The use of an appropriate graduated ruler is acceptable to determine the particle size.

14.2.9.2. If the solids are prepared for semi-volatile solvent extractions, refer to the specific SOPs of the extraction methods for additional information on sample homogenization and sieving.

14.2.10. Quantitatively transfer the solid phase including the filter into an appropriate bottle extraction vessel.

14.2.10.1. Use a 2-L borosilicate glass or Teflon bottle for evaluating mobility of both organic and inorganic analytes. Use a 2-L HDPE bottle only for evaluating the mobility of metals.

14.2.11. Refer to Section 14.2.1. for the type of the TCLP extraction fluid to be added to the bottle extraction vessel.

14.2.12. Determine the amount of the TCLP extraction fluid to be added to the bottle extraction vessel. The formula for calculating the amount of the TCLP extraction fluid is listed in Section 15.4.

14.2.12.1. For ***100*** g of sample with 100% solids content, the amount of the TCLP extraction fluid required is 2 L.

14.2.13. Slowly add the required amount of TCLP extraction fluid to the bottle.

14.2.14. Close the bottle tightly, and secure it in the rotary agitation apparatus.

14.2.15. Set the rotary agitation apparatus to rotate at 30 ± 2 rpm for 18 ± 2 hours. Maintain the room temperature at 23 ± 2°C during the extraction period.

14.2.15.1. Pressure may build up within the bottle extraction vessels during agitation for some types of wastes. To relieve excess pressure, the bottles may be periodically opened and vented into a fume hood.

14.2.16. Following the extraction, filter the extract as described in Sections 14.2.6. and 14.2.7. using the same filtration system ***or vacuum filtration apparatus*** with a new filter.

14.2.16.1. A syringe filtration apparatus ***(Section 9.6.)*** may be utilized instead of a filtration system ***or vacuum filtration apparatus*** to filter the extract.

14.2.16.1.1. ***Do not use a syringe filtration apparatus for extract designated for semi-volatile analysis.***

14.2.17. Collect the filtrate in a clean filtrate container.

14.2.17.1. For solid sample with 100% solids content, the filtrate is the TCLP extract.

14.2.17.1.1. Measure the pH of the filtrate with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4.

14.2.17.2. For other solid and multiphasic sample, the filtrate is the TCLP extract of the solid phase.

14.2.17.2.1. If compatible, combine the filtrates of both liquid and solid phases. Measure the pH of the combined filtrate with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4.

14.2.17.2.2. If not compatible, measure the pH of the filtrate of each phase with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4., analyze the filtrates separately, and combine the results mathematically.

14.3. TCLP Extraction for Volatile Analysis

14.3.1. TCLP Extraction Fluid Determination

14.3.1.1. Use only TCLP extraction fluid #1.

14.3.2. Measure the pH of ***the TCLP extraction fluid*** with a pH meter prior to use. Record the pH to the nearest 0.01 pH unit.

14.3.2.1. The pH of ***the TCLP extraction fluid*** is used to monitor ***for impurities and to verify integrity***.

14.3.3. Assemble the ZHE device according to the manufacturer's instructions, and secure a clean 90-mm glass fiber filter between the two support screens in the ZHE vessel.

14.3.4. Measure sufficient amount of sample. Record the amount to the nearest ***0.1 mL or*** 0.1 g.

14.3.4.1. For aqueous sample, measure exactly 25.0 ***mL*** of sample. ***Assume the density of aqueous sample is 1 g/mL.***

14.3.4.1.1. For MB/***LCS/LCSD, measure exactly 25.0 mL*** of clean reagent water.

14.3.4.1.2. For MS/MSD, ***measure exactly 25.0 mL*** of aqueous sample in each analytical batch selected for spiking.

14.3.4.2. For solid sample with 100% solids content, measure ***23.8–25.0*** g of sample. For other solid and multiphasic sample, use the solids content (see Section 14.1.) to determine the amount of sample required. The formula for calculating the amount of sample is listed in Section 15.3.

14.3.4.2.1. For MB/***LCS/LCSD, measure 23.8–25.0*** g of clean Teflon chips or glass beads.

14.3.4.2.2. For MS/MSD, ***measure 23.8–25.0 g (or the amount determined from calculation)*** of solid or multiphasic sample in each analytical batch selected for spiking.

14.3.4.3. The amount of sample required for analytical determination is established by the determinative method or the project's Quality Assurance Project Plan (QAPP).

14.3.5. For solid sample, reduce the particle size of the solid to < 9.5 mm by crushing, cutting, or grinding. Avoid the exposure of sample to the atmosphere to the extent possible.

14.3.5.1. Heat generated during particle size reduction may cause analyte losses. Hence, samples and appropriate reduction equipment should be refrigerated, if possible, to 4°C prior to particle size reduction.

14.3.5.2. Sieving of sample may cause analyte losses. The use of an appropriate graduated ruler is recommended to determine the particle size.

14.3.6. Quantitatively transfer both liquid and solid phases of sample quickly to the ZHE vessel. Do not centrifuge sample or allow the solid phase of sample to settle.

14.3.7. Place the ZHE device in the vertical position with the liquid inlet/outlet valve on top.

14.3.8. Apply gentle pressure (< 20 psi) and slowly open the liquid inlet/outlet valve (top flange) to force all headspace slowly out of the ZHE device.

14.3.8.1. For solid sample with 100% solids content, slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the ZHE device.

14.3.9. Quickly close the liquid inlet/outlet valve and discontinue the pressure at the first appearance of liquid from the liquid inlet/outlet valve.

14.3.9.1. If the filtration of a sample at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the ZHE device prior to filtration.

14.3.10. Attach an evacuated, clean filtrate collection syringe (Section 9.2.4.) to the liquid inlet/outlet valve, and open the valve.

14.3.11. Gradually apply pressure of 1−10 psi to force the liquid phase of sample into the filtrate collection syringe.

14.3.11.1. If no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

14.3.11.2. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment.

14.3.11.3. If liquid flow has ceased at 50 psi, and if no additional liquid has passed through the filter within a 2-minute period with continued pressure at 50 psi, stop the filtration.

14.3.12. Transfer the filtrate from the filtrate collection syringe to a clean filtrate vial without introducing any air bubbles within the vial while it is being filled.

14.3.12.1. If bubbling occurs during the transfer, the filtrate must be poured out and the vial refilled.

14.3.12.2. Completely fill and hermetically seal the filtrate vial such that when the vial is inverted, no headspace is visible.

14.3.12.3. For aqueous sample, the filtrate is the TCLP extract.

14.3.12.3.1. Measure the pH of the filtrate with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4.

14.3.12.4. For solid and multiphasic sample, the filtrate is the TCLP extract of the liquid phase.

14.3.12.4.1. If compatible, combine the filtrates of both liquid and solid phases. Measure the pH of the combined filtrate with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4.

14.3.12.4.2. If not compatible, measure the pH of the filtrate of each phase with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4., analyze the filtrates separately, and combine the results mathematically.

14.3.13. Determine the amount of the TCLP extraction fluid to be added to the ZHE vessel. The formula for calculating the amount of the TCLP extraction fluid is listed in Section 15.4.

14.3.13.1. For 25.0 g of sample with 100% solids content, the amount of the TCLP extraction fluid required is 500 mL.

14.3.14. Transfer the required amount of TCLP extraction fluid to the ZHE vessel using an evacuated, clean filtrate collection syringe.

14.3.15. After adding the TCLP extraction fluid, immediate close the liquid inlet/outlet value.

14.3.16. Manually rotate the ZHE device in an end-to-end fashion for 2−3 times. Place the ZHE device in the vertical position with the liquid inlet/outlet valve on top.

14.3.16.1. If necessary, apply gentle pressure (< 20 psi) and slowly open the liquid inlet/outlet valve (top flange) to force all headspace slowly out of the ZHE device.

14.3.16.2. Quickly close the liquid inlet/outlet valve and discontinue the pressure at the first appearance of liquid from the liquid inlet/outlet valve.

14.3.17. Secure the ZHE device in the rotary agitation apparatus.

14.3.18. Set the rotary agitation apparatus to rotate at 30 ± 2 rpm for 18 ± 2 hours. Maintain the room temperature at 23 ± 2°C during the extraction period.

14.3.19. Following the extraction, check the pressure behind the ZHE vessel piston by quickly opening and closing the pressure relief valve and noting the escape of gas.

14.3.19.1. If no gas release is observed, the device is leaking. Check the ZHE device for leaking as specified in Section 9.2.1.5., and perform the extraction again with a new aliquot of the sample.

14.3.20. Collect the filtrate using the same procedure as described in Sections 14.3.10. and 14.3.11.

14.3.21. Transfer the filtrate from the filtrate collection syringe to a clean filtrate vial without introducing any air bubbles within the vial while it is being filled.

14.3.21.1. If bubbling occurs during the transfer, the filtrate must be poured out and the vial refilled.

14.3.21.2. Completely fill and hermetically seal the filtrate vial such that when the vial is inverted, no headspace is visible.

14.3.21.3. For solid sample with 100% solids content, the filtrate is the TCLP extract.

14.3.21.3.1. Measure the pH of the filtrate with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4.

14.3.21.4. For other solid and multiphasic sample, the filtrate is the TCLP extract of the solid phase.

14.3.21.4.1. If compatible, combine the filtrates of both liquid and solid phases. Measure the pH of the combined filtrate with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4.

14.3.21.4.2. If not compatible, measure the pH of the filtrate of each phase with a pH meter. Record the pH to the nearest 0.01 pH unit. Proceed to Section 14.4., analyze the filtrates separately, and combine the results mathematically.

14.4. TCLP extract should be prepared for analysis and analyzed as soon as possible following TCLP extraction.

14.4.1. For hexavalent chromium, proceed to colorimetric or IC analysis. The TCLP extract must be analyzed within 24 hours.

14.4.2. For other metals, prepare the TCLP extract for acid digestion as follows:

14.4.2.1. Add the spike standard solution listed in the determinative method to all matrix spikes ***and laboratory control samples***.

14.4.2.2. Acidify the TCLP extract as described in Section 11.3.2., and proceed to acid digestion.

14.4.3. For semi-volatile organics, proceed to solvent extraction.

14.4.3.1. If solvent extraction is not to be performed within 24 hours, it is recommended to store the TCLP extract without headspace in a

pre-cleaned 500-mL amber glass bottle with Teflon-lined closure, and preserve the TCLP extract as an aqueous sample according to the specific SOP of the determinative method.

14.4.3.2. It is possible for the TCLP extract to generate some headspace in the form of micro bubbles during storage. The bubbles should not exceed ¼ in or 6 mm in diameter.

14.4.4. For volatile organics, proceed to purge-and-trap preparation and/or GC/MS analysis.

14.4.4.1. If purge-and-trap preparation and/or GC/MS analysis are not to be performed within 24 hours, it is recommended to store the TCLP extract without headspace in a pre-cleaned 40-mL amber glass vial with Teflon-lined closure, and preserve the TCLP extract as an aqueous sample according to the specific SOP of the determinative method.

14.4.4.2. It is possible for the TCLP extract to generate some headspace in the form of micro bubbles during storage. The bubbles should not exceed ¼ in or 6 mm in diameter.

14.5. Thoroughly document all aspects of the extraction in an extraction logbook. This logbook includes:

14.5.1. Extraction date, start time, and end time.

14.5.2. Sample initial mass.

14.5.3. Extraction fluid lot (or identification) number, type, and volume.

14.5.4. Pre-extraction pH and post-extraction pH.

14.5.5. ZHE leak check status.

14.5.6. Analyst comments which include encountered problems, pertinent observations, or conditions that could potentially impact data quality.

15. ►CALCULATIONS

15.1. The solids content for a sample is calculated as follows:

$$C_d = \frac{M_2 - M_1}{M_0} \times 100$$

where: C_d = solids content in %.
M_2 = mass of watch glass, filter, and solid residue (or oven-dried solid residue) of sample after filtration in g.
M_1 = mass of watch glass and filter in g.
M_0 = mass of sample prior to filtration in g.

15.2. The amount of sample required for TCLP extraction for metals or semi-volatile analysis is calculated as follows:

$$\frac{10000}{C_d} < M_x < \frac{10500}{C_d}$$

where: M_x = mass of sample prior to TCLP extraction in g.
C_d = solids content in %.

15.3. The amount of sample required for TCLP extraction for volatile analysis is calculated as follows:

$$\frac{2375}{C_d} < M_x < \frac{2500}{C_d}$$

where: M_x = mass of sample prior to TCLP extraction in g.
C_d = solids content in %.

15.4. The amount of TCLP extraction fluid is calculated as follows:

$$M_{EF} = \frac{20 \times C_d \times M_x}{100}$$

where: M_{EF} = amount of TCLP extraction fluid in g or mL.
Assume the density of TCLP extraction fluid is 1 g/mL.
C_d = solids content in %.
M_x = mass of sample prior to TCLP extraction in g.

15.5. The target analyte concentration for a sample based on simple volume-weighted average is calculated as follows:

$$C_x = \frac{(V_{LP} \times C_{LP}) + (V_{SP} \times C_{SP})}{V_{LP} + V_{SP}}$$

where: C_x = concentration of target analyte in sample in mg/L or µg/L.
V_{LP} = volume of sample liquid phase in L.
C_{LP} = concentration of target analyte in sample liquid phase in mg/L or µg/L.
V_{SP} = volume of sample solid phase in L.
C_{SP} = concentration of target analyte in sample solid phase in mg/L or µg/L.

Note: Concentrations and volumes must be in equivalent units.

15.6. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

16. METHOD PERFORMANCE

16.1. A demonstration of analytical capability shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

16.2. Calibration protocols specified in Section 13., "Calibration and Standardization," shall be followed.

16.3. Proficiency test sample results shall be used to evaluate the ability to produce accurate results.

17. POLLUTION PREVENTION

17.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Calscience's Health, Safety, and Respiratory Protection Manual. In general, protective eyewear (e.g. safety glasses or goggles), and protective apparel (e.g. lab coats) and gloves are required to be worn when handling chemicals.

17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:

17.3.1. A NIOSH approved air purifying respirator with cartridges appropriate for the chemical handled.

17.3.2. Extended length protective gloves.

17.3.3. Face shield.

17.3.4. Full-length laboratory apron.

17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.

17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles. Alternatively, the Coordinator may utilize a self-contained breathing apparatus or other supplied air system if appropriately trained and approved by the Health and Safety Manager.

17.6. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

18.1. Refer to the determinative method for data assessment and acceptance criteria.

18.2. Additional information regarding internal quality control checks is provided in SOP-T020.

18.3. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

19. CORRECTIVE ACTIONS

19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.

19.2. The Operations Manager, Project Manager, Quality Control Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.

19.3. Corrective actions are generally of two types, immediate and long-term actions.

19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.

19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented in the Corrective Action Record. Examples of this type of action include:

19.3.2.1. Remedial training of staff in technical skills, technique or implementation of operating procedures.

19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.

19.3.2.3. Revision of standard operating procedures.

19.3.2.4. Replacing personnel, as necessary.

19.4. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:

19.4.1. Define the problem.

19.4.2. Assign responsibility for investigating the problem.

19.4.3. Investigate and determine the cause of the problem.

19.4.4. Assign and accept responsibility for implementing the corrective action.

19.4.5. Determine effectiveness of the corrective action and implement correction.

19.4.6. Verify that the corrective action has eliminated the problem.

19.5. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

20. CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1. Refer to the determinative method for contingencies for out-of-control or unacceptable data.

20.2. Out-of-control data are reviewed and verified by the technical director of the appropriate department. All samples associated with an unacceptable QC set are then subject to reanalysis, depending upon the QC type in question.

21. WASTE MANAGEMENT

21.1. The proper disposal of analytical samples and laboratory wastes is not only good laboratory practice, but also regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated, and either returned to the client (preferable) or placed into the proper laboratory waste stream.

21.2. Unused or remaining soil or liquid samples and all other solid or liquid wastes resulting from our laboratory operations are considered hazardous for disposal purposes.

21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal.

21.4. Each specific laboratory area shall maintain clearly labeled waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste.

21.5. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly.

21.6. In order to maintain accountability for all samples received by Calscience, when a sample is used in its entirety for analysis, the empty container(s) are returned to Sample Control for placement in analytical storage.

21.7. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Wastes."

22. ►REFERENCES

22.1. *Toxicity Characteristic Leaching Procedure*, Test Methods for Evaluating Solid Waste (SW-846), Third edition, Volume 1C, Method 1311, USEPA, Revision 0, July 1992.

22.2. *Quality Control*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter One, USEPA, Revision 1, July 1992.

22.3. *Choosing the Correct Procedure*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Two, USEPA, Revision 4, ***February 2007***.

22.4. *Characteristics Introduction and Regulatory Definitions*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Seven, USEPA, Revision 4, November 2004.

22.5. *Toxicity Characteristic Leaching Procedure (TCLP) for VOCs, SVOCs, Chlorinated Pesticides and Herbicides, and Metals*, SW-846 Methods 1311, 8260B, 8270C, 8081, 8151A, 6010, and 7470/7471, Region 9 Quality Assurance Data Quality Indicator Tables, USEPA, January 2000.

22.6. *§261.24 Toxicity Characteristic*, Code of Federal Regulations, Title 40 Protection of Environment, Part 261 Identification and Listing of Hazardous Waste, Subpart C Characteristics of Hazardous Waste, 40 CFR, ***July 2008***.

22.7. *§268.40 Applicability of Treatment Standards*, Code of Federal Regulations, Title 40 Protection of Environment, Part 268 Land Disposal Restrictions, Subpart D Treatment Standards, 40 CFR, ***July 2008***.

23. TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

23.1. Appendix A: Maximum Concentration of Contaminants for Toxicity Characteristic.

23.2. ►Appendix B: Sample Maximum Holding Times.

23.3. Appendix C: Toxicity Characteristic Leaching Procedure Flowchart.

23.4. Appendix D: Rotary Agitator Diagram.

23.5. Appendix E: Zero-Headspace Vessel Diagram.

24. ►MODIFICATIONS

24.1. ***The following modifications from EPA Method 1311 are noted.***

24.1.1. ***Reagents and Standards***

24.1.1.1. ***The TCLP extraction fluid preparations are modified.***

24.1.2. ***Procedure***

24.1.2.1. ***The sequence of the pH check followed by the addition of 1-N HCl for TCLP extraction fluid determination is modified.***

Appendix A

MAXIMUM CONCENTRATION OF CONTAMINANTS FOR TOXICITY CHARACTERISTIC

Calscience Environmental Laboratories, Inc.

Maximum Concentration of Contaminants for Toxicity Characteristic

Contaminant	EPA Hazardous Waste (HW) Number	Chemical Abstracts Service (CAS) Number	Regulatory Level (mg/L)
Arsenic	D004	7440-38-2	5.0
Barium	D005	7440-39-3	100.0
Benzene	D018	71-43-2	0.5
Cadmium	D006	7440-43-9	1.0
Carbon tetrachloride	D019	56-23-5	0.5
Chlordane	D020	57-74-9	0.03
Chlorobenzene	D021	108-90-7	100.0
Chloroform	D022	67-66-3	6.0
Chromium	D007	7440-47-3	5.0
o-Cresol	D023	95-48-7	200.0 [1]
m-Cresol	D024	108-39-4	200.0 [1]
p-Cresol	D025	106-44-5	200.0 [1]
Cresol	D026		200.0 [1]
2,4-D	D016	94-75-7	10.0
1,4-Dichlorobenzene	D027	106-46-7	7.5
1,2-Dichloroethane	D028	107-06-2	0.5
1,1-Dichloroethylene	D029	75-35-4	0.7
2,4-Dinitrotoluene	D030	121-14-2	0.13 [2]
Endrin	D012	72-20-8	0.02
Heptachlor (and its hydroxide)	D031	76-44-8	0.008
Hexachlorobenzene	D032	118-74-1	0.13 [2]
Hexachloro-1,3-butadiene	D033	87-68-3	0.5
Hexachloroethane	D034	67-72-1	3.0
Lead	D008	7439-92-1	5.0
Lindane	D013	58-89-9	0.4
Mercury	D009	7439-97-6	0.2
Methoxychlor	D014	72-43-5	10.0
Methyl ethyl ketone	D035	78-93-3	200.0
Nitrobenzene	D036	98-95-3	2.0
Pentachlorophenol	D037	87-86-5	100.0
Pyridine	D038	110-86-1	5.0 [2]
Selenium	D010	7782-49-2	1.0
Silver	D011	7440-22-4	5.0
Tetrachloroethylene	D039	127-18-4	0.7
Toxaphene	D015	8001-35-2	0.5
Trichloroethylene	D040	79-01-6	0.5
2,4,5-Trichlorophenol	D041	95-95-4	400.0
2,4,6-Trichlorophenol	D042	88-06-2	2.0
2,4,5-TP (Silvex)	D017	93-72-1	1.0
Vinyl chloride	D043	75-01-4	0.2

[1] If o-, m-, and p-cresol concentrations cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level of total cresol is 200 mg/L.

[2] Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.

Appendix B

SAMPLE MAXIMUM HOLDING TIMES

Calscience Environmental Laboratories, Inc.

Sample Maximum Holding Time

Analytes	From Sample Collection To TCLP Extraction	From TCLP Extraction To Determinative Analysis
Hexavalent Chromium	24 hours	24 hours
Mercury	28 days	28 days
Other Metals	180 days	180 days
Volatiles in Water	14 days	14 days
Volatiles in Soil	14 days*	7 days

Analytes	From Sample Collection To TCLP Extraction	From TCLP Extraction To Solvent Extraction	From Solvent Extraction To Determinative Analysis
Semi-volatiles	14 days	7 days	40 days

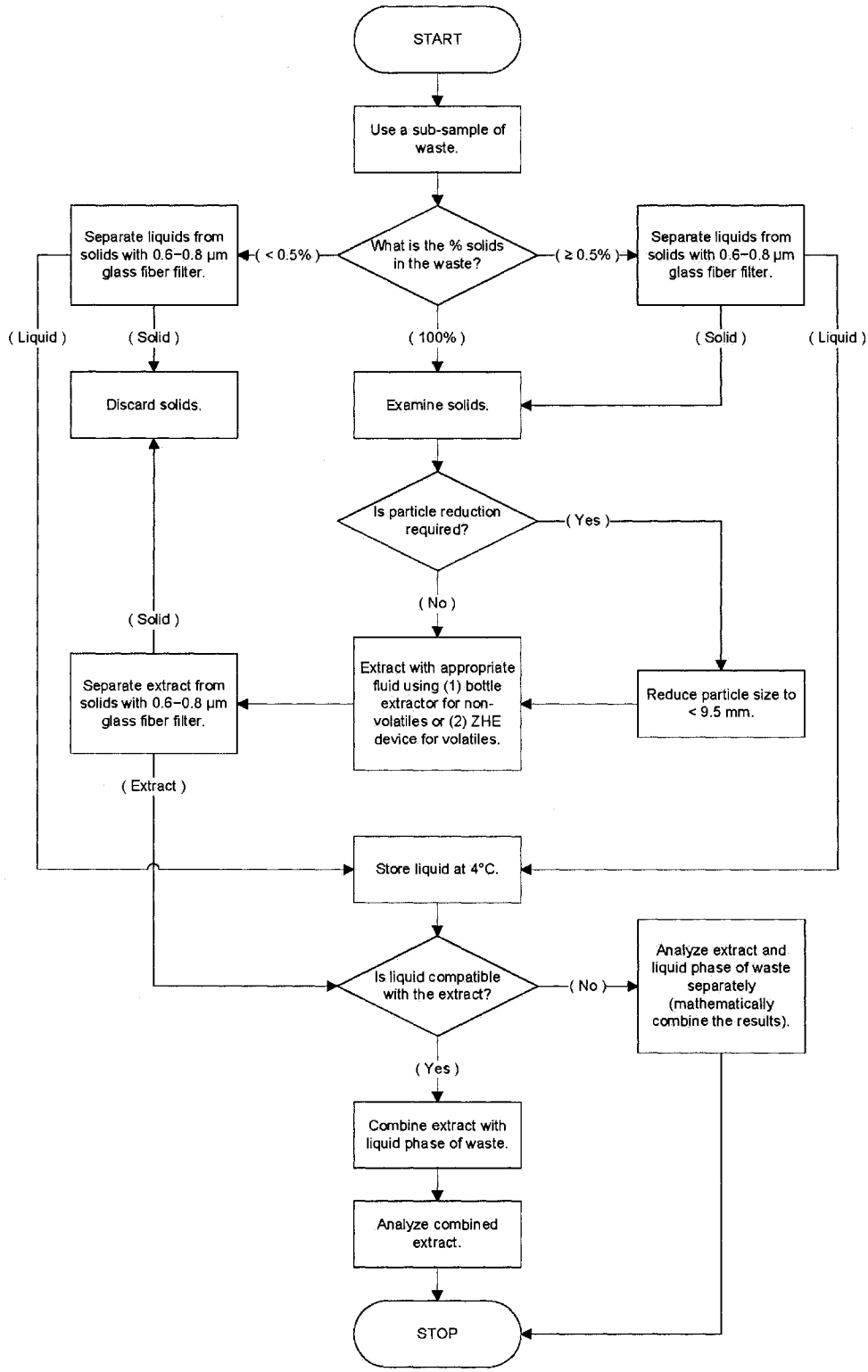
* For EPA Region 9 requirement, the holding time is 48 hours.

Appendix C

TOXICITY CHARACTERISTIC LEACHING PROCEDURE FLOWCHART

Calscience Environmental Laboratories, Inc.

Toxicity Characteristic Leaching Procedure Flowchart

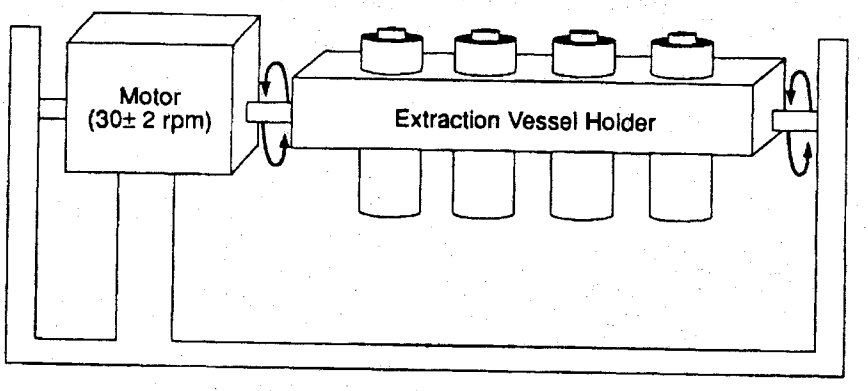


Appendix D

ROTATORY AGITATOR DIAGRAM

Calscience Environmental Laboratories, Inc.

Rotary Agitator Diagram

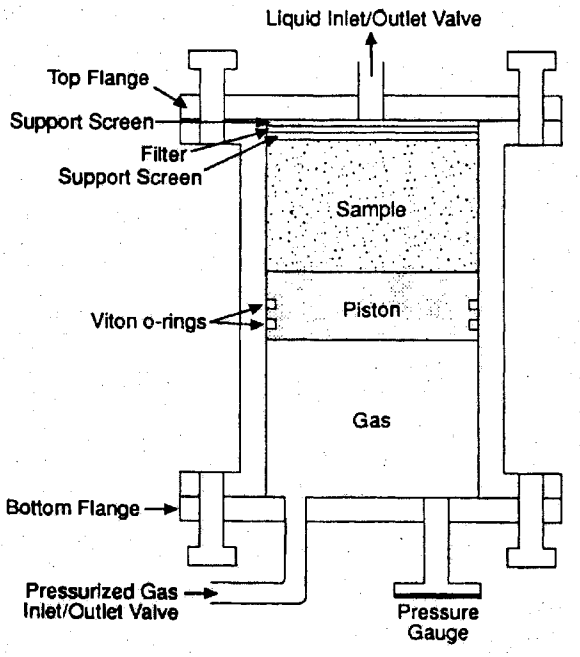


Appendix E

ZERO-HEADSPACE VESSEL DIAGRAM

Calscience Environmental Laboratories, Inc.

Zero-Headspace Vessel Diagram



Title : **CALIFORNIA CODE OF REGULATIONS, TITLE 22, DIVISION 4.5, CHAPTER 11, ARTICLE 5, §66261.126, APPENDIX II, WASTE EXTRACTION TEST (WET)**

Document No. : SOP-M228
Revision No. : 1.2
Supersedes : 1.1

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Revision 1.2 changes are noted in bold italicized typeface and preceded by a "►" marker.

APPROVED FOR RELEASE BY: _____ 12/02/08
MANAGEMENT **DATE**

_____ 12-02-08
QA DEPARTMENT **DATE**

ANNUAL SOP REVIEW

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

1. METHOD IDENTIFICATION

1.1. California Code of Regulations, Title 22, Division 4.5, Chapter 11, Article 5, §66261.126, Appendix II, Waste Extraction Test (WET).

2. APPLICABLE MATRICES

2.1. This method is applicable to liquid, solid and multiphasic wastes.

3. DETECTION LIMITS

3.1. ►Refer to the determinative ***(analytical)*** method for detection limit data.

4. SCOPE AND APPLICATION

4.1. CCR T22.11.5.A-II the Waste Extraction Test (WET) determines the amounts of inorganic and organic persistent and bioaccumulative toxic substances in liquid, solid, and multiphasic wastes.

4.1.1. The WET is designed to simulate the leaching a waste will undergo if disposed of in a sanitary landfill. This test is designed to simulate the leaching that takes place in a sanitary landfill only.

4.1.2. The approximate preparation time is 50 hours.

4.2. A subsample of a waste is extracted with the citric acid solution for 48 hours. The WET extract (the extract obtained from the WET) is then analyzed to determine if any of the soluble threshold limit concentrations (STLC) or the total threshold limit concentrations (TTLC) established for the inorganic and organic persistent and bioaccumulative toxic substances (see Appendix A) have been exceeded.

4.2.1. If a liquid waste contains any one of the inorganic and organic persistent and bioaccumulative toxic substances in an amount greater than or equal to the TTLC specified in CCR T22.11.3 §66261.24, the waste shall be classified as a hazardous waste.

4.2.2. If a liquid waste contains any one of the inorganic and organic persistent and bioaccumulative toxic substances in an amount less than the TTLC but greater than the STLC specified in CCR T22.11.3 §66261.24, the waste shall be filtered through a 0.45-µm membrane filter.

4.2.3. If the WET extract of a solid or multiphasic waste contains any one of the inorganic and organic persistent and bioaccumulative toxic substances in an amount greater than the STLC specified in CCR T22.11.3 §66261.24, the waste shall be classified as a hazardous waste.

4.3. ►This method is restricted to use by or under the supervision of analysts/***technicians*** experienced in the use of the instruments and apparatus required to execute the procedure.

5. METHOD SUMMARY

5.1. A preliminary evaluation is performed to determine if the waste is to be treated as a liquid or a solid.

5.1.1. For liquid wastes (i.e., wastes containing less than 0.5% dry solid material), the waste shall not be subject to the WET procedure.

5.1.1.1. The liquid waste shall be analyzed directly for the inorganic and organic persistent and bioaccumulative toxic substances (see Appendix A).

5.1.2. For wastes containing greater than or equal to 0.5% solids, the liquid phase is separated from the solid phase by filtration and stored for later analysis. The solid phase is processed as follows:

5.1.2.1. The particle size of the solid phase is reduced, if necessary.

5.1.2.2. The solid phase is then extracted with an amount of WET extraction fluid equal to 10 times the mass of the solid phase for 48 hours. The extraction fluid employed is a 0.2-M sodium citrate solution.

5.1.2.3. Following extraction, the WET extract is separated from the solid material by filtration through a 0.45-µm membrane filter.

5.1.2.4. If compatible (i.e. multiple phases will not form on combination), the initial liquid phase of the waste is added to the WET extract, and these are analyzed together.

5.1.2.5. If incompatible, the initial liquid phase and the WET extract are analyzed separately, and the results are mathematically combined to yield a volume-weighted average concentration.

5.2. ►Refer to the appropriate determinative methods including EPA Methods 6010***C*** (metals), ***6020A (metals)***, 7196A/7199 (hexavalent chromium), 7470A/7471***B*** (mercury), 8081***B***, ***8021B***, 8151A, 8260B, and 8270***D*** for the analysis of the SPLP extract to identify and quantify the inorganic and organic analytes.

6. DEFINITIONS

6.1. Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

6.2. Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

6.3. 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 NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the

start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

6.4. Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

6.5. Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

6.6. Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

6.7. Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

6.8. Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

6.9. Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

6.10. Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.

6.11. Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

6.12. Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

6.13. Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

6.14. Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

6.15. Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

6.16. Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

6.17. Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

6.18. Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.

6.19. 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.

6.20. 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.

6.21. Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence.

6.22. Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

6.23. Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

6.24. Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

6.25. Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

6.26. Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

7. INTERFERENCES

7.1. Refer to the determinative method for potential interferences.

8. SAFETY

8.1. It is recommended that all filtrations be performed in a fume hood vented to the exterior of the laboratory.

8.2. For the safety of the analyst, cracked or broken glassware should be immediately discarded into a broken glassware receptacle. Broken glassware shall not be used in any step of the extraction.

8.3. ►To ensure the safety of the analyst during any possible emergency situation, ***it is recommended that*** chemists do not perform extractions alone. Another chemist shall be present during any extraction process.

8.4. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current version of Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats are required to be worn in all designated laboratory areas. Protective gloves shall be worn when handling chemicals.

8.5. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

9. ►EQUIPMENT AND SUPPLIES

9.1. Agitation apparatus:

9.1.1. Rotary agitator, capable of rotating extraction vessels in an end-over-end fashion at 30 ± 2 rpm, Associated Design and Manufacturing Company 3740-12-BRE Rotary Agitator or equivalent.

9.2. Bottle extraction device:

9.2.1. Bottle extraction vessel, 500-mL and 1-L, borosilicate glass or high density polyethylene (HDPE).

9.2.1.1. Borosilicate glass bottles are used for evaluating the mobility of organic components.

9.2.1.2. HDPE bottles are used for evaluating the mobility of inorganic components.

9.3. Filtration system:

9.3.1. Filter holder, 1.5-L minimum internal volume, type 316 stainless steel, capable of supporting a 142-mm diameter filter and withstanding 50+ psi pressure requirement for phase separation, Millipore Corporation YT30 142 HW Hazardous Waste Filtration System or equivalent.

9.3.2. Filter, 0.45-µm effective pore size, 142-mm diameter, hydrophilic mixed cellulose esters membrane, Millipore Corporation HAWP 142 50 Membrane Filter or equivalent.

9.3.3. Prefilter, medium porosity, 0.8−8.0-µm effective pore size, 142-mm diameter, borosilicate glass fiber with binder resin, Millipore Corporation AP20 142 50 Glass Fiber Filter or equivalent.

9.3.3.1. Place the 142-mm glass fiber prefilter on top of the 142-mm membrane filter when assembling the filtration system.

9.3.4. Pressurizing gas, high purity nitrogen.

9.4. Vacuum filtration apparatus:

9.4.1. Vacuum apparatus.

9.4.2. Filter holder, ceramic, capable of supporting a 90-mm diameter filter.

9.4.3. Filtration flask, 500-mL, glass.

9.4.4. Filter, 0.45-µm effective pore size, 90-mm diameter, hydrophilic polyethersulfone membrane, Pall Corporation Supor-450 60206 Membrane Filter or equivalent.

9.4.5. Prefilter, 0.6−0.8-µm effective pore size, 90-mm diameter, borosilicate glass fiber without binder resin, Whatman 1810-090 Acid Treated Low Metal TCLP Filter or equivalent.

9.4.5.1. Place the 90-mm glass fiber prefilter on top of the 90-mm membrane filter when assembling the vacuum filtration apparatus.

9.5. Syringe filtration apparatus:

9.5.1. Syringe, ***10-mL, polypropylene, eccentric tip***, disposable, ***BD Lab Syringe P/N 305462 or equivalent***.

9.5.2. Filter, 0.45-µm effective pore size, 30-mm diameter, hydrophilic polyvinylidene difluoride (PVDF) membrane, polypropylene housing, disposable, National Scientific Company F2500-5 Target Syringe Filter or equivalent.

9.5.3. Prefilter, 1.0-µm effective pore size, 30-mm diameter, borosilicate glass microfiber, on top of 0.45-µm PVDF membrane filter.

9.6. Filtrate containers:

9.6.1. ***Digestion tubes (vials), 3-oz (90-mL), 93-mm × 33-mm ID, graduated, snap closure, with hinge lids, polypropylene, disposable, Capitol Vial P/N 03EDM31/33 or equivalent.***

9.6.1.1. The 3-oz plastic vial is used to collect filtrate for metals analysis.

9.6.2. Bottles, 500-mL, HDPE and amber glass, with Teflon-lined closures.

9.6.2.1. The 500-mL HDPE bottle is used to collect filtrate for hexavalent chromium or fluoride analysis.

9.6.2.2. The 500-mL amber glass bottle is used to collect filtrate for semi-volatile solvent extraction.

9.6.3. Erlenmeyer flasks, 250-mL and 500-mL, glass.

9.6.3.1. The flask is used to collect filtrate for semi-volatile solvent extraction.

9.6.4. Vials, 4-mL, ***15-mm × 45-mm***, clear glass, ***with screw cap and*** Teflon-lined ***septa***, disposable.

9.6.4.1. The 4-mL clear glass vial is used to collect filtrate for volatile analysis.

9.6.5. Vials, 40-mL, ***28-mm × 95-mm***, amber glass, ***with screw cap and*** Teflon-lined ***septa***, disposable.

9.6.5.1. The 40-mL amber glass vial is used to collect filtrate for volatile analysis.

9.7. Grinding apparatus.

9.8. Sieves, 10-mesh (2-mm) and 18-mesh (1-mm), ***stainless steel or Teflon***.

9.9. pH meter, accurate to ± 0.05 units at 25°C, equipped with ATC probe or equivalent, Fisher Scientific Accumet Basic AB15 pH Meter or equivalent.

9.10. pH indicator paper, narrow range. pH range should include the desired pH.

9.11. Balance, top loading, capable of weighing to the nearest 0.01 g.

9.12. ***Centrifuge, Fisher Scientific Centrific Benchtop Centrifuge Model 225 or equivalent.***

9.13. ***Centrifuge tubes, 50-mL, with lids, plastic, disposable.***

9.14. Pipetters, 10−100-µL, 100−1000-µL, 0.5−5.0-mL, and 1−10-mL, adjustable, with disposable tip.

9.15. ***Dispensers, 1−10-mL, 2.5−25-mL, and 5−50-mL, adjustable.***

9.16. ***Thermometer.***

9.17. Solids content determination apparatus:

9.17.1. Watch glass, 90-mm diameter or larger.

9.17.2. Drying oven, capable of maintaining 104 ± 1°C.

9.17.3. Desiccator.

9.17.4. Forceps, blunt curved-tip, stainless steel.

10. ►REAGENTS AND STANDARDS

10.1. Reagents

10.1.1. Reagent water, interferant free, distilled or deionized.

10.1.2. Chips, Teflon.

10.1.3. Beads, glass.

10.1.4. Nitric acid, HNO_3, ***68.0–70.0% (v/v)***, concentrated, ***clear to yellow liquid***, trace metals grade for equivalent.

10.1.5. Sodium hydroxide, NaOH, ***white pellets and granules***, reagent grade or equivalent.

10.1.6. Sodium hydroxide, NaOH, 4-N.

10.1.6.1. Prepare the 4-N NaOH solution by dissolving 640 g of NaOH in reagent water and dilute to 4 L with additional reagent water.

10.1.7. Citric acid, $C_6H_8O_7$, anhydrous, ***white crystalline powder, certified***, reagent grade or equivalent.

10.1.8. WET (STLC) extraction fluid #1, pH at 5.00 ± 0.10.

10.1.8.1. Prepare the WET (STLC) extraction fluid #1 by diluting 768.5 g of anhydrous citric acid and 2000 mL of 4-N NaOH solution to 20 L with reagent water.

10.1.8.2. Adjust the pH with additional anhydrous citric acid or 4-N NaOH solution.

10.1.8.3. Record the amounts of the reagents to the nearest 0.1 g or 1 mL in the preparation logbook.

10.1.8.4. The WET extraction fluid #1 is a 0.2-M sodium citrate solution used to determine the leachability of all inorganic and organic persistent and accumulative toxic substances <u>except</u> hexavalent chromium compounds.

10.1.9. WET (STLC) extraction fluid #2.

10.1.9.1. The WET extraction fluid #2 is reagent water used to determine the leachability of hexavalent chromium compounds.

10.1.10. All reagents must be inspected and documented ***in the Solvent/Standard Verification Logbook*** prior to use.

10.2. Standards

10.2.1. ***Refer to the specific SOPs of the determinative methods for standards.***

11. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

11.1. Do not add preservation chemicals to samples prior to WET extraction.

11.2. Refer to the specific SOPs of the determinative methods for additional information on sample collection, preservation, containers and holding times.

11.3. Refer to the specific SOPs of the determinative methods for extract preservation and holding times post WET extraction.

11.3.1. For hexavalent chromium, Cr(VI), determinations, no preservation chemicals are required. The WET extracts must be analyzed within 24 hours.

11.3.2. For other metallic analyte determinations, acidify WET extracts or portions of WET extracts with concentrated HNO_3 to 5% (v/v) of acid content.

11.3.3. For fluoride determinations, no preservation chemicals are required. The WET extracts must be analyzed within 24 hours.

11.3.4. For organic analyte determinations, do not allow WET extracts or portions of WET extracts coming into contact with the atmosphere (i.e., no headspace) to prevent analyte losses.

11.3.5. WET extracts should be prepared within the appropriate period to allow for subsequent solvent extractions and determinative analyses (see Appendix B).

12. QUALITY CONTROL

12.1. ►Event Based Quality Control (***LCS/LCSDs and*** MBs)

12.1.1. Event based quality control consists of QC samples prepared and processed with each preparatory event. This consists of ***a laboratory control sample and laboratory control sample duplicate (LCS/LCSD) and*** a method blank (MB).

12.1.2. Aqueous Samples

12.1.2.1. ***One LCS consisting of the specified analytes spiked into the WET extract of clean reagent water is required every day for every batch of 20 samples per matrix or portion thereof, whichever is more frequent. The LCSD is handled identically to the LCS.***

12.1.2.2. One MB consisting of WET extract of clean reagent water is required every day for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

12.1.3. Solid Samples

12.1.3.1. ***One LCS consisting of the specified analytes spiked into the WET extract of clean Teflon chips or glass beads is required every day for every batch of 20 samples per matrix or portion thereof, whichever is more frequent. The LCSD is handled identically to the LCS.***

12.1.3.2. One MB consisting of WET extract of clean Teflon chips or glass beads is required every day for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

12.1.4. The MB/***LCS/LCSD*** is processed concurrently with the associated samples. In the processing of the MB/***LCS/LCSD***, reagents and procedures identical to those for actual samples are used.

12.2. Matrix Based Quality Control (MS/MSDs)

12.2.1. Matrix based quality control consists of QC samples prepared and processed using actual environmental samples. This consists of a matrix spike and matrix spike duplicate (MS/MSD).

12.2.2. One MS consisting of WET extract of the actual sample matrix spiked with known concentrations of specific target analytes is required for every batch of 20 samples per matrix or portion thereof. The MSD is handled identically to the MS.

12.2.3. ►The sample which ***will be*** spiked for the MS/MSD is processed concurrently with the associated samples. In the processing of the MS/MSD, reagents and procedures identical to those for actual samples are used.

12.3. Refer to the specific SOPs of the determinative methods for additional information on quality control.

13. ►CALIBRATION AND STANDARDIZATION

13.1. Top Loading Balance

13.1.1. Calibrate the top loading balance at 1 g and 100 g using Class 2 weights.

13.1.2. Calibration shall be within ± 2% at 1 g (± 0.02 g) and at 100 g (± 2 g). If the values are not within these limits, recalibrate the balance.

13.2. ***Thermometer***

13.2.1. ***Calibrate the thermometer using an NIST certified thermometer. The calibration procedure shall adhere to the current revision of SOP-T042, "Thermometer Calibration."***

13.3. Pipetter

13.3.1. ***Calibrate the pipetter according to the procedure outlined in the Pipetter Calibration Check Logbook.***

13.4. ***Dispenser***

13.4.1. ***Calibrate the dispenser according to the procedure outlined in the Dispenser Calibration Check Logbook.***

13.5. pH Meter

13.5.1. Calibrate the pH meter daily prior to sample analysis at pH of 4.00, 7.00, and 10.00 using fresh buffer solutions and according to the instrument manufacturer's recommended procedures.

13.5.2. Verify the calibration with fresh second source buffer standard. The second source standard shall not differ from its expected value by more than 0.05 pH units. If this criterion is not met, recheck calibration or effect corrective action.

13.5.3. The theoretical slope shall be within 90–105% for analysis to proceed. If this criterion is not met, determine the cause of the problem, effect corrective action, and recalibrate, if necessary.

14. ►PROCEDURE

14.1. Solids Content Determination

14.1.1. Weigh a clean watch glass and a clean or acid-washed glass fiber filter. Record the mass to the nearest ***0.01*** g.

14.1.2. Weigh ***50.00 ± 2.50*** g of sample. Record the mass to the nearest ***0.01*** g.

14.1.3. Assemble a vacuum filtration apparatus (Section ***9.4.***) with the pre-weighted filter and apply vacuum. Quantitatively transfer the sample to the filter holder. Spread the sample evenly over the surface of the filter. Continue to apply vacuum to filter the sample until all traces of water have been removed.

14.1.4. Remove the filter from the filter holder with forceps at the edge of the filter and carefully place on the pre-weighted watch glass without touching the solid residue or inverting the filter.

14.1.4.1. If sample result is to be reported on a dry-weight basis, dry the solid residue in 103–105°C oven for a minimum of one hour. Cool in the desiccator and weigh. Repeat the drying cycle until a constant mass is obtained or until the mass difference is within ± 1% from the previous weighing.

14.1.4.2. Oven-dried solid residue should not be used for subsequent WET extraction.

14.1.5. Weigh the sample including the filter and watch glass. Record the mass to the nearest ***0.01*** g. The formula for calculating the solids content is listed in Section 15.1.

14.1.6. Sample is deemed aqueous if the solids content is < 0.5%. Sample is deemed solid if the solids content is ≥0.5%.

14.2. Analyze aqueous sample directly to determine whether the sample should be subject to filtration (see Section 4.2.2.).

14.2.1. If aqueous sample is subject to filtration, proceed to Section 14.4.

14.3. WET Extraction Fluid Determination

14.3.1. If Cr(VI) is not a target analyte, use WET extraction fluid #1.

14.3.2. If Cr(VI) is a target analyte, use WET extraction fluid #2 (reagent water).

14.4. Measure the pH of ***the WET extraction fluid*** with a pH meter prior to use. Record the pH to the nearest 0.01 pH unit.

14.4.1. The pH of ***the WET extraction fluid*** is used to monitor ***for impurities and to verify integrity***.

14.5. Assemble a filtration system (Section 9.3.) with a clean 142-mm membrane filter and a clean 142-mm glass fiber prefilter according to the manufacturer's instructions to filter sample. ***A vacuum filtration apparatus (Section 9.4.) may be utilized instead of a filtration system to filter sample.***

14.6. Measure sufficient amount of sample. Record the amount to the nearest ***0.1 mL or*** 0.1 g.

14.6.1. For aqueous sample, ***measure exactly*** 50.0 ***mL*** of sample. ***Assume the density of aqueous sample is 1 g/mL.***

14.6.1.1. For MB/***LCS/LCSD, measure exactly 50.0 mL*** of clean reagent water.

14.6.1.2. For MS/MSD, ***measure exactly 50.0 mL*** of aqueous sample in each analytical batch selected for spiking.

14.6.2. For solid and multiphasic sample, measure ***50.0 ± 2.5*** g of sample.

14.6.2.1. For MB/***LCS/LCSD, measure 50.0 ± 2.5 g*** of clean Teflon chips or glass beads.

14.6.2.2. For MS/MSD, ***measure 50.0 ± 2.5 g*** of solid or multiphasic sample in each analytical batch selected for spiking.

14.6.3. The amount of sample required for analytical determination is established by the determinative method or the project's Quality Assurance Project Plan (QAPP).

14.7. With the filter in place, quantitatively transfer both liquid and solid phases of sample to the filter holder. Spread the sample evenly over the surface of the filter.

14.8. Gradually apply pressure of 1−10 psi until pressurizing gas moves through the filter. ***If a vacuum filtration apparatus is used, gradually apply vacuum.***

14.8.1. If the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

14.8.2. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment.

14.8.3. If the pressurizing gas begins to move through the filter, or if liquid flow has ceased at 50 psi, stop the filtration.

14.9. Collect the filtrate in a clean filtrate container.

14.9.1. For aqueous sample, the filtrate is the WET extract. Proceed to Section 14.19.

14.9.2. For solid and multiphasic sample, the filtrate is the WET extract of the liquid phase.

14.9.2.1. If compatible, combine the filtrates of both liquid and solid phases. Proceed to Section 14.19.

14.9.2.2. If not compatible, proceed to Section 14.19., analyze the filtrates separately, and combine the results mathematically.

14.10. Evaluate the particle size of the solid phase (i.e. the material in the filter holder).

14.10.1. If the solids are prepared for inorganic analyses, reduce the particle size of the millable solid by crushing or grinding so that it can pass through a 2-mm sieve. Remove non-friable solid particles which are extraneous and irrelevant as hazardous constituents by mechanical means and discarded.

14.10.2. If the solids are prepared for organic analyses, reduce the particle size of the millable solid by crushing or grinding so that it can pass through a 1-mm sieve. Remove non-friable solid particles which are extraneous and irrelevant as hazardous constituents by mechanical means and discarded.

14.11. Quantitatively transfer the solid phase including the filter into an appropriate bottle extraction vessel.

14.11.1. Use a 500-mL or 1-L HDPE bottle for evaluating the mobility of inorganic analytes. Use a 500-mL or 1-L borosilicate glass bottle for evaluating mobility of organic analytes.

14.12. Refer to Section 14.3. for the type of the WET extraction fluid to be added to the bottle extraction vessel.

14.13. Determine the amount of the WET extraction fluid to be added to the bottle extraction vessel. The formula for calculating the amount of the WET extraction fluid is listed in Section 15.2.

14.13.1. For 50.0 g of sample with 100% solids content, the amount of the WET extraction fluid required is 500 mL.

14.14. Slowly add the required amount of WET extraction fluid to the bottle.

14.15. Close the bottle tightly, and secure it in the rotary agitation apparatus.

14.15.1. If volatile organics are to be analyzed, seal the bottle quickly after deaeration with nitrogen to prevent analyte losses.

14.16. Set the rotary agitation apparatus to rotate at 30 ± 2 rpm for 48 hours. Maintain the room temperature at 20–40°C during the extraction period.

14.17. Following the extraction, filter the extract as described in Sections 14.7. and 14.8. using the same filtration system ***or vacuum filtration apparatus*** with a new filter and a new prefilter.

14.17.1. A syringe filtration apparatus ***(Section 9.5.)*** may be utilized instead of a filtration system ***or vacuum filtration apparatus*** to filter the extract.

14.17.1.1. ***Do not use a syringe filtration apparatus for extract designated for organic analysis.***

14.17.2. Centrifugation may be used as an aid to separate the liquid and solid phases. If sample is centrifuged, decant and filter the liquid phase through the filtration system ***or vacuum filtration apparatus*** followed by filtration of the solid phase through the same filtration system ***or vacuum filtration apparatus***.

14.18. Collect the filtrate in a clean filtrate container.

14.18.1. For solid sample with 100% solids content, the filtrate is the WET extract. Proceed to Section 14.19.

14.18.2. For other solid and multiphasic sample, the filtrate is the WET extract of the solid phase.

14.18.2.1. If compatible, combine the filtrates of both liquid and solid phases. Proceed to Section 14.19.

14.18.2.2. If not compatible, proceed to Section 14.19., analyze the filtrates separately, and combine the results mathematically.

14.19. WET extract should be prepared for analysis and analyzed as soon as possible following WET extraction.

14.19.1. For hexavalent chromium, proceed to colorimetric or IC analysis. The WET extract must be analyzed within 24 hours.

14.19.2. For other metals, prepare the WET extract for acid digestion as follows:

14.19.2.1. Add the spike standard solution listed in the determinative method to all matrix spikes ***and laboratory control samples***.

14.19.2.2. Acidify the WET extract as described in Section 11.3.2., and proceed to ***metals analysis. Acid digestion prior to metals analysis is optional.***

14.19.3. For fluoride, proceed to distillation and/or ISE analysis. The WET extract must be analyzed within 24 hours.

14.19.4. For semi-volatile organics and organolead (organic lead compounds), proceed to solvent extraction.

14.19.4.1. If solvent extraction is not to be performed within 24 hours, it is recommended to store the WET extract without headspace in a

pre-cleaned 500-mL amber glass bottle with Teflon-lined closure, and preserve the WET extract as an aqueous sample according to the specific SOP of the determinative method.

14.19.4.2. It is possible for the WET extract to generate some headspace in the form of micro bubbles during storage. The bubbles should not exceed ¼ in or 6 mm in diameter.

14.19.5. For volatile organics, proceed to purge-and-trap preparation and/or GC/MS analysis.

14.19.5.1. If purge-and-trap preparation and/or GC/MS analysis are not to be performed within 24 hours, it is recommended to store the WET extract without headspace in a pre-cleaned 40-mL amber glass vial with Teflon-lined closure, and preserve the WET extract as an aqueous sample according to the specific SOP of the determinative method.

14.19.5.2. It is possible for the WET extract to generate some headspace in the form of micro bubbles during storage. The bubbles should not exceed ¼ in or 6 mm in diameter.

14.20. Thoroughly document all aspects of the extraction in an extraction logbook. This logbook includes:

14.20.1. Extraction date, start time, and end time.

14.20.2. Sample initial mass.

14.20.3. Extraction fluid lot (or identification) number, type, and volume.

14.20.4. Pre-extraction pH.

14.20.5. Analyst comments which include encountered problems, pertinent observations, or conditions that could potentially impact data quality.

15. ►CALCULATIONS

15.1. The solids content for a sample is calculated as follows:

$$C_d = \frac{M_2 - M_1}{M_0} \times 100$$

where: C_d = solids content in %.
M_2 = mass of watch glass, filter, and solid residue (or oven-dried solid residue) of sample after filtration in g.
M_1 = mass of watch glass and filter in g.
M_0 = mass of sample prior to filtration in g.

15.2. The amount of WET extraction fluid is calculated as follows:

$$M_{EF} = \frac{10 \times C_d \times M_x}{100}$$

where: M_{EF} = amount of WET extraction fluid in g or mL.
Assume the density of WET extraction fluid is 1 g/mL.
C_d = solids content in %.
M_x = mass of sample prior to WET extraction in g.

15.3. The target analyte concentration for a sample based on simple volume-weighted average is calculated as follows:

$$C_x = \frac{(V_{LP} \times C_{LP}) + (V_{SP} \times C_{SP})}{V_{LP} + V_{SP}}$$

where: C_x = concentration of target analyte in sample in mg/L.
V_{LP} = volume of sample liquid phase in L.
C_{LP} = concentration of target analyte in sample liquid phase in mg/L.
V_{SP} = volume of sample solid phase in L.
C_{SP} = concentration of target analyte in sample solid phase in mg/L.

Note: Concentrations and volumes must be in equivalent units.

15.4. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

16. METHOD PERFORMANCE

16.1. A demonstration of analytical capability shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

16.2. Calibration protocols specified in Section 13., "Calibration and Standardization," shall be followed.

16.3. Proficiency test sample results shall be used to evaluate the ability to produce accurate results.

17. POLLUTION PREVENTION

17.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Calscience's Health, Safety, and Respiratory Protection Manual. In general, protective eyewear (e.g. safety glasses or goggles), and protective apparel (e.g. lab coats) and gloves are required to be worn when handling chemicals.

17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:

17.3.1. A NIOSH approved air purifying respirator with cartridges appropriate for the chemical handled.

17.3.2. Extended length protective gloves.

17.3.3. Face shield.

17.3.4. Full-length laboratory apron.

17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.

17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles. Alternatively, the Coordinator may utilize a self-contained breathing apparatus or other supplied air system if appropriately trained and approved by the Health and Safety Manager.

17.6. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

18.1. Refer to the determinative method for data assessment and acceptance criteria.

18.2. Additional information regarding internal quality control checks is provided in SOP-T020.

18.3. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

19. CORRECTIVE ACTIONS

19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.

19.2. The Operations Manager, Project Manager, Quality Control Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.

19.3. Corrective actions are generally of two types, immediate and long-term actions.

19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.

19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented in the Corrective Action Record. Examples of this type of action include:

19.3.2.1. Remedial training of staff in technical skills, technique or implementation of operating procedures.

19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.

19.3.2.3. Revision of standard operating procedures.

19.3.2.4. Replacing personnel, as necessary.

19.4. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:

19.4.1. Define the problem.

19.4.2. Assign responsibility for investigating the problem.

19.4.3. Investigate and determine the cause of the problem.

19.4.4. Assign and accept responsibility for implementing the corrective action.

19.4.5. Determine effectiveness of the corrective action and implement correction.

19.4.6. Verify that the corrective action has eliminated the problem.

19.5. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

20. CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1. Refer to the determinative method for contingencies for out-of-control or unacceptable data.

20.2. Out-of-control data are reviewed and verified by the technical director of the appropriate department. All samples associated with an unacceptable QC set are then subject to reanalysis, depending upon the QC type in question.

21. WASTE MANAGEMENT

21.1. The proper disposal of analytical samples and laboratory wastes is not only good laboratory practice, but also regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated, and either returned to the client (preferable) or placed into the proper laboratory waste stream.

21.2. Unused or remaining soil or liquid samples and all other solid or liquid wastes resulting from our laboratory operations are considered hazardous for disposal purposes.

21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal.

21.4. Each specific laboratory area shall maintain clearly labeled waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste.

21.5. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly.

21.6. In order to maintain accountability for all samples received by Calscience, when a sample is used in its entirety for analysis, the empty container(s) are returned to Sample Control for placement in analytical storage.

21.7. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Wastes."

22. REFERENCES

22.1. *California Code of Regulations*, Title 22, Division 4.5, Chapter 11, Article 5, §66261.126, Appendix II, Waste Extraction Test (WET) Procedures.

22.2. *California Code of Regulations*, Title 22, Division 4.5, Chapter 11, Article 3, §66261.24, Characteristic of Toxicity.

22.3. *Toxicity Characteristic Leaching Procedure*, Test Methods for Evaluating Solid Waste (SW-846), Third edition, Volume 1C, Method 1311, USEPA, Revision 0, July 1992.

22.4. *Extraction Procedure (EP) Toxicity Test Method and Structural Integrity Test*, Test Methods for Evaluating Solid Waste (SW-846), Third edition, Volume 1C, Method 1310B, USEPA, Revision 2, November 2004.

23. TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

23.1. Appendix A: Persistent and Bioaccumulative Toxic Substances.

23.2. ►Appendix B: Sample Maximum Holding Times.

24. ►MODIFICATIONS

24.1. ***None.***

Appendix A

PERSISTENT AND BIOACCUMULATIVE TOXIC SUBSTANCES

Calscience Environmental Laboratories, Inc.

Persistent and Bioaccumulative Toxic Substances

Substances	STLC (mg/L)	TCLP (mg/kg, wet-weight)
Inorganic[a,b]		
Antimony and/or antimony compounds	15.	500.
Arsenic and/or arsenic compounds	5.0	500.
Barium and/or barium compounds (excluding barite)	100.	10000. [c]
Beryllium and/or beryllium compounds	0.75	75.
Cadmium and/or cadmium compounds	1.0	100.
Chromium (VI) compounds	5.	500.
Chromium and/or chromium (III) compounds	5. [d]	2500.
Cobalt and/or cobalt compounds	80.	8000.
Copper and/or copper compounds	25.	2500.
Fluoride salts	180.	18000.
Lead and/or lead compounds	5.0	1000.
Mercury and/or mercury compounds	0.2	20.
Molybdenum and/or molybdenum compounds	350.	3500. [e]
Nickel and/or nickel compounds	20.	2000.
Selenium and/or selenium compounds	1.0	100.
Silver and/or silver compounds	5.	500.
Thallium and/or thallium compounds	7.0	700.
Vanadium and/or vanadium compounds	24.	2400.
Zinc and/or zinc compounds	250.	5000.
Organic		
Aldrin	0.14	1.4
Chlordane	0.25	2.5
DDT, DDE, DDD	0.1	1.0
2,4-Dichlorophenoxyacetic acid	10.	100.
Dieldrin	0.8	8.0
Dioxin (2,3,7,8-TCDD)	0.001	0.01
Endrin	0.02	0.2
Heptachlor	0.47	4.7
Kepone	2.1	21.
Lead compounds (organic)		13.
Lindane	0.4	4.0
Methoxychlor	10.	100.
Mirex	2.1	21.
Pentachlorophenol	1.7	17.
Polychlorinated biphenyls (PCBs)	5.0	50.
Toxaphene	0.5	5.
Trichloroethylene	204.	2040.
2,4,5-Trichlorophenoxypropionic acid	1.0	10.

[a] STLC and TTLC values are calculated on the concentrations of the elements, not the compounds.

[b] In the case of elemental metals, the specified concentration limits apply only if the substances are in a friable, powdered or finely divided state.

[c] Excluding barium sulfate.

[d] If the soluble chromium is < 5 mg/L as determined by TCLP, but is ≥560 mg/L as determined by WET, and the waste is not otherwise identified as a RCRA hazardous waste pursuant to §66261.100, then the waste is a non-RCRA hazardous waste.

[e] Excluding molybdenum disulfide.

Appendix B

SAMPLE MAXIMUM HOLDING TIMES

Calscience Environmental Laboratories, Inc.

Sample Maximum Holding Time

Analytes	From Sample Collection To WET Extraction	From WET Extraction To Determinative Analysis
Hexavalent Chromium	24 hours	24 hours
Mercury	28 days	28 days
Other Metals	180 days	180 days
Fluoride	28 days	24 hours
Volatiles in Water	14 days	14 days
Volatiles in Soil	14 days*	7 days

Analytes	From Sample Collection To WET Extraction	From WET Extraction To Solvent Extraction	From Solvent Extraction To Determinative Analysis
Semi-volatiles	14 days	7 days	40 days

* For EPA Region 9 requirement, the holding time is 48 hours.

Title : **EPA METHOD 8081A, ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY**

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Revision 4.4 changes are noted in bold italicized typeface and preceded by a "►" marker. The method name change from EPA 8081B to 8081A is not so noted.

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ANNUAL SOP REVIEW

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE
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1. METHOD IDENTIFICATION

1.1. EPA Method 8081A, Organochlorine Pesticides by Gas Chromatography.

2. APPLICABLE MATRICES

2.1. This method is applicable to water, oil, soil, and solid wastes.

3. DETECTION LIMITS

3.1. The estimated quantitation limits (EQLs) for this method are as follows:

	Soil	Water	Oil	Wipe/Filter	Tissue
Pesticides	5 µg/kg (wet-weight)	0.1 µg/L	100 µg/kg	0.1 µg/sample	2 µg/kg
Chlordane	50 µg/kg (wet-weight)	1 µg/L	1000 µg/kg	1 µg/sample	
Toxaphene	100 µg/kg (wet-weight)	2 µg/L	2000 µg/kg	2 µg/sample	

3.2. The EQLs will be proportionally higher for sample extracts which require dilution or cleanup.

4. SCOPE AND APPLICATION

4.1. EPA Method 8081A is used to determine the concentrations of a number of common organochlorine pesticides in various matrices. The method is used to quantitate organochlorine pesticides without derivitization. The following compounds can be determined by this method:

aldrin	4,4'-DDD	endrin aldehyde
α-BHC	4,4'-DDE	endrin ketone
β-BHC	4,4'-DDT	heptachlor
γ-BHC (lindane)	dieldrin	heptachlor epoxide
δ-BHC	endosulfan I	methoxychlor
α-chlordane (cis-chlordane)	endosulfan II	toxaphene
γ-chlordane (trans-chlordane)	endosulfan sulfate	
chlordane	endrin	
2,4'-DDD (o,p'-DDD)	2,4'-DDT (o,p'-DDT)	cis-nonachlor
2,4'-DDE (o,p'-DDE)	hexachlorobenzene	trans-nonachlor

4.2. Upon client request, additional target analytes may be added to this analysis. However, it needs to be demonstrated that any added compounds lend themselves to EPA Method 8081A determination, either by regulatory reference or validation studies.

4.3. Extracts suitable for analysis by this method may also be analyzed for organophosphorus pesticides (EPA Method 8141B).

4.4. This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph (GC) and skilled in the interpretation of gas chromatograms.

5. METHOD SUMMARY

5.1. EPA Method 8081A describes chromatographic procedures that will allow for the separation of organochlorine pesticide compounds in the extract and their qualitative and quantitative analysis by gas chromatography. Detection is achieved using an electron capture detector (ECD).

5.2. Prior to performing this procedure, the appropriate sample preparation technique must be performed on each sample.

5.2.1. Aqueous samples are extracted via EPA Methods 3510C or 3520C at neutral pH using methylene chloride exchanged into hexane.

5.2.2. Solid samples are extracted via EPA Methods 3540C, 3545A, or 3550C using methylene chloride-acetone (1:1) exchanged into hexane, or via EPA Method 3545A using methylene chloride exchanged into hexane.

5.2.3. Solid samples for TCLP, SPLP, or STLC analysis are prepared using the appropriate mobility extraction method, and the resulting mobility-procedure extracts (leachates) are extracted via EPA Methods 3510C or 3520C at neutral pH using methylene chloride exchanged into hexane.

5.2.4. Oil samples are prepared in accordance with EPA Method 3580A using hexane as the diluent.

5.2.5. A variety of cleanup procedures may be applied to the extracts, depending on the nature of the target analytes and the matrix interferences.

5.3. Acceptable preparatory methods include, but are not limited to, the following:

Type of Sample Preparation	EPA Method No.	SOP No.
Separatory Funnel Liquid-Liquid Extraction	3510C	SOP-M200
Continuous Liquid-Liquid Extraction	3520C	SOP-M201
Soxhlet Extraction	3540C	SOP-M203
Pressurized Fluid Extraction	3545A	SOP-M204
Ultrasonic Extraction	3550**C**	SOP-M202
Waste Dilution	3580A	SOP-M205
Gel-Permeation Cleanup	3640A	SOP-M233
Cleanup	3600C(M)	SOP-M234
TCLP	1311	SOP-M226
SPLP	1312	SOP-M227
STLC (California Code of Regulations)	CCR T22.11.5.A-II	SOP-M228

6. DEFINITIONS

6.1. Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

6.2. Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

6.3. 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 NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

6.4. Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

6.5. Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

6.6. Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

6.7. Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

6.8. Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

6.9. Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.

6.10. Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

6.11. Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

6.12. Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

6.13. Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

6.14. Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

6.15. Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

6.16. Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

6.17. Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.

6.18. 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.

6.19. 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.

6.20. Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence.

6.21. Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

6.22. Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

6.23. Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

6.24. Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

6.25. Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

7. INTERFERENCES

7.1. Contamination by carryover can occur whenever high and low concentration level samples are analyzed sequentially. Suspected high level samples should be diluted and then analyzed at the end of the sequence to prevent carryover contamination. In addition, sample syringes should be thoroughly rinsed with solvent between sample injections.

7.2. Interference can also occur when "dirty" samples leave residue in the analytical column. To minimize this effect, guard columns should be used and cut frequently or replaced. Also, the analytical column can be "baked" after such samples. Other maintenance procedures include cleaning the inlet or replacing injection liner/seal.

7.3. Solvents, reagents, glassware, and other sample processing equipment may yield discrete contaminants. This can lead to spurious peaks and/or an elevated baseline, resulting in possible misinterpretation of chromatograms.

7.4. Endrin and 4,4'-DDT are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with high boiling residue from sample injection or when the injector contains metal fittings.

7.4.1. Endrin and 4,4'-DDT breakdown to endrin aldehyde, endrin ketone, 4,4'-DDD, or 4,4'-DDE.

7.4.2. When such breakdown is observed, the corrective action may include, but is not limited to 1) cleaning and deactivating the injection port, 2) replacing the injection port liner, or 3) clipping the guard column.

7.5. Phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

7.5.1. Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

7.5.2. Exhaustive cleanup of solvents, reagents and glassware may be necessary to eliminate background phthalate ester contamination.

7.6. The presence of sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected

with sediment samples. EPA Method 3660B, Sulfur Cleanup, should be employed for sulfur removal prior to analysis.

7.6.1. The recovery of endrin aldehyde is drastically reduced when using the TBA procedure in EPA Method 3660B; hence, this compound must be determined prior to sulfur cleanup when it is an analyte of interest and the TBA procedure is to be used for cleanup.

7.6.2. The use of copper powder technique in EPA Method 3660B may adversely affect the recoveries of some organochlorine compounds and many organophosphorus compounds.

7.7. Waxes, lipids, and other high molecular weight materials can be removed by EPA Method 3640A, Gel-Permeation Cleanup.

7.8. Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides.

7.8.1. Certain co-eluting organophosphorus pesticides may be eliminated by the pesticide option of EPA Method 3640A.

7.8.2. Co-eluting chlorophenols may be eliminated by EPA Methods 3630C (Silica Gel Cleanup), 3620C (Florisil Cleanup), or 3610B (Alumina Cleanup).

7.9. Polychlorinated biphenyls (PCBs) may interfere with the analysis of the organochlorine pesticides. The problem may be most severe for the analysis of multiple-component analytes such as chlordane and toxaphene. If PCBs are known or expected to occur in samples, consult EPA Methods 3620C and 3630C to separate the pesticides from the PCBs.

8. SAFETY

8.1. Compounds covered by this method have been tentatively classified as known or suspected human carcinogens. Primary standards of these compounds and all other pesticides must be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when analysts handle high concentrations of these compounds.

8.2. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current version of Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats are required to be worn in all designated laboratory areas. Protective gloves shall be worn when handling chemicals.

8.3. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

9. EQUIPMENT AND SUPPLIES

9.1. Gas Chromatograph: Agilent 6890N Gas Chromatograph, Agilent 7890A Gas Chromatograph, or equivalent configured with the following components:

9.1.1. Splitless injection port.

9.1.2. Autoinjector, Agilent 7683 Series, Agilent 7683B Series, or equivalent.

9.1.3. PC based data system, Agilent GC ChemStation or equivalent.

9.2. Primary Detection Channel

9.2.1. Detector: Electron capture detector (ECD).

9.2.2. Analytical Column: 30-m × 0.32-mm ID, 0.50-µm film thickness, narrow-bore, capillary, silicone coated fused-silica, Restek Rtx®-CLPesticides or equivalent.

9.3. Confirmation Detection Channel

9.3.1. Detector: Electron capture detector (ECD).

9.3.2. Analytical Column: 30-m × 0.32-mm ID, 0.25-µm film thickness, narrow-bore, capillary, silicone coated fused-silica, Restek Rtx®-CLPesticides2 or equivalent.

9.4. Guard Column: 5-m × 0.32-mm ID, intermediate-polarity deactivated, uncoated fused-silica, Restek IP Deactivated Guard Column or equivalent.

9.5. Carrier Gas: Helium, He, 99.995%, compressed, Praxair 4.5 grade or equivalent.

9.6. Makeup Gas: Methane, CH_4, 5%, and argon, Ar, 95%, compressed, Praxair P-5 Mixture or equivalent.

9.7. Syringes, 10-µL, 25-µL, 50-µL, 100-µL, 250-µL, and 500-µL, gastight, Cemented Needle (N) termination, Hamilton 1700 Series or equivalent with NIST Traceable Certificate or equivalent documentation.

9.8. Storage vials, 15-mm × 45-mm (4-mL capacity), screw top, clear glass, with Teflon-lined screw caps and septa, disposable.

9.9. Autoinjector vials, 12-mm × 32-mm (2-mL capacity), crimp top, clear glass, with aluminum crimp caps and Teflon-lined septa, disposable.

10. REAGENTS AND STANDARDS

10.1. Reagents

10.1.1. Reagent water, interferant free.

10.1.2. Sand, washed, sea or standard Ottawa.

10.1.3. Sodium thiosulfate, $Na_2S_2O_3$, anhydrous, white solid, reagent grade or equivalent.

10.1.4. Sodium thiosulfate, $Na_2S_2O_3$, 10% (w/v).

10.1.4.1. Prepare the 10% $Na_2S_2O_3$ solution by dissolving 200 g of anhydrous $Na_2S_2O_3$ in reagent water and dilute to 2 L with additional reagent water.

10.1.5. Methylene chloride (or dichloromethane), CH_2Cl_2, clear colorless liquid, pesticide grade or equivalent.

10.1.6. Hexane, C_6H_{14}, clear colorless liquid, pesticide grade or equivalent.

10.1.7. Acetone, CH_3COCH_3, clear colorless liquid, pesticide grade or equivalent.

10.1.8. All reagents must be inspected and documented in the Chemicals and Supplies Verification Logbook prior to use.

10.2. Standards

10.2.1. Pre-certified stock standard solutions, each in sealed glass ampules, containing 2000/200/100 ppm of each single-component pesticide target analyte, 100/1000 ppm of chlordane, 100/1000 ppm of toxaphene, and 200 ppm of each surrogate are used to prepare calibration and check standards.

10.2.1.1. Prepare each working standard solution by diluting the appropriate volume of the stock standard to 4.0 mL with hexane.

10.2.1.2. The working standards are prepared as follows:

Analyte	Initial		Final	
	Conc. (ppm)	Volume (µL)	Conc. (ppm)	Volume (mL)
organochlorine pesticides	2000	8.0	4.0	4.0
hexachlorobenzene	100	160		
surrogates	200	160	8.0	
o,p'-DDD	100	80	2.0	4.0
o,p'-DDE	100	80		
o,p'-DDT	100	80		
cis-nonachlor	100	80		
trans-nonachlor	100	80		

Analyte	Initial		Final	
	Conc. (ppm)	Volume (µL)	Conc. (ppm)	Volume (mL)
organochlorine pesticides	200	80	4.0	4.0
hexachlorobenzene	100	160		
surrogates	200	160	8.0	
2,4'-DDD	100	80	2.0	4.0
2,4'-DDE	100	80		
2,4'-DDT	100	80		
cis-nonachlor	100	80		

Analyte	Initial		Final	
	Conc. (ppm)	Volume (µL)	Conc. (ppm)	Volume (mL)
trans-nonachlor	100	80	2.0	4.0
chlordane	1000	400	100	4.0
toxaphene	1000	400	100	4.0

10.2.1.3. The working standard solutions must be stored under dark and refrigerated conditions, and replaced after six months or sooner if comparison with check standards indicates a problem.

10.2.2. Degradation test stock standard solution containing 500 ppm each of 4,4'-DDT and endrin in methanol.

10.2.2.1. Prepare the 500-ppb degradation test working standard solution by diluting 5.0 µL of the 500-ppm degradation test stock standard to 5.0 mL with hexane.

10.2.2.2. Prepare the 50-ppb degradation test working standard solution by diluting 4.0 mL of the 500-ppb degradation test working standard to 40 mL with hexane.

10.2.2.3. Inject 2 µL of the 50-ppb degradation test working standard for degradation test.

10.2.3. Calibration standard solutions containing various concentrations of target analytes and surrogates in hexane.

10.2.3.1. Dilute the appropriate volumes of the working standards, the 100-ppm chlordane stock standard, and the 100-ppm toxaphene stock standard to 4.0 mL with hexane for initial calibration.

10.2.3.2. The calibration standards are prepared as follows:

Calibration Level (ppb)			Initial				Final	
			Concentration (ppm)		Volume (µL)		Volume (mL)	
A1	S	A2	A1 + S	A2	A1 + S	A2	A1 + S	A2
10	20	10	4.0 + 8.0	2.0	10	20	4.0	4.0
20	40	20	4.0 + 8.0	2.0	20	40	4.0	4.0
40	80	40	4.0 + 8.0	2.0	40	80	4.0	4.0
60	120	60	4.0 + 8.0	2.0	60	120	4.0	4.0
80	160	80	4.0 + 8.0	2.0	80	160	4.0	4.0

Note: A1 = Single-Component Analyte; S = Surrogate;
A2 = o,p'-(DDD, DDE, DDT) or Nonachlor

Calibration Level (ppb)		Initial				Final	
		Concentration (ppm)		Volume (µL)		Volume (mL)	
C	T	C	T	C	T	C	T
100	200	100	100	4.0	8.0	4.0	4.0
250	500	100	100	10	20	4.0	4.0
500	1000	100	100	20	40	4.0	4.0
750	1500	100	100	30	60	4.0	4.0
2000	4000	100	100	80	160	4.0	4.0

Note: C = Chlordane; T = Toxaphene

10.2.3.3. The mid-range standards are also used as the continuing calibration verification solutions.

10.2.4. Initial calibration verification (ICV) solutions containing 40 ppb of each single-component pesticide target analyte, 500 ppb of chlordane, 1000 ppb of toxaphene, and 80 ppb of each surrogate in hexane. The ICV solutions must be of a source differing from that used for the initial five-point calibration. If it is of the same source, then it must be of different lot.

10.2.4.1. Dilute the appropriate volumes of the second source working standards to 4.0 mL with hexane for initial calibration verification.

10.2.4.2. The ICV solution is prepared as follows:

Analyte	Calibration Level (ppb)	Initial		Final
		Conc. (ppm)	Volume (µL)	Volume (mL)
organochlorine pesticides	40	4.0	40	4.0
hexachlorobenzene				
surrogates	80	8.0		
2,4'-DDD	40	2.0	80	4.0
2,4'-DDE				
2,4'-DDT				
cis-nonachlor				
trans-nonachlor				
chlordane	500	100	20	4.0
toxaphene	1000	100	40	4.0

10.2.5. Continuing calibration verification (CCV) solutions containing mid-range concentrations of target analytes and surrogates in hexane. The CCV solutions are of a source same as that used for the initial five-point calibration.

10.2.5.1. Dilute the appropriate volumes of the working standards, the 100-ppm chlordane stock standard, and the 100-ppm toxaphene stock standard to 4.0 mL with hexane for continuing calibration verification.

10.2.5.2. The CCV solutions are prepared as follows:

Calibration Level (ppb)			Initial				Final	
			Concentration (ppm)		Volume (µL)		Volume (mL)	
A1	S	A2	A1 + S	A2	A1 + S	A2	A1 + S	A2
20	40	20	4.0 + 8.0	2.0	20	40	4.0	4.0
40	80	40	4.0 + 8.0	2.0	40	80	4.0	4.0
60	120	60	4.0 + 8.0	2.0	60	120	4.0	4.0

Note: A1 = Single-Component Analyte; S = Surrogate;
A2 = o,p'-(DDD, DDE, DDT) or Nonachlor

Calibration Level (ppb)		Initial				Final	
		Concentration (ppm)		Volume (µL)		Volume (mL)	
C	T	C	T	C	T	C	T
250	500	100	100	10	20	4.0	4.0
500	1000	100	100	20	40	4.0	4.0
750	1500	100	100	30	60	4.0	4.0

Note: C = Chlordane; T = Toxaphene

10.2.5.3. One CCV solution is used daily. The concentration of the CCV *solution must be varied at a minimum weekly.*

10.2.6. Surrogate stock standard solution containing 200 ppm each of decachloro-biphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TMX) in acetone.

10.2.6.1. Prepare the 2.0-ppm surrogate working standard solution by diluting 8.0 mL of the 200-ppm surrogate stock standard solution to 800 mL with acetone.

10.2.6.2. Add 500 µL of the 2.0-ppm surrogate working standard to each sample including the quality control (QC) check samples and method blanks prior to extraction.

10.2.7. Spike stock standard solutions containing 200/1000/100 ppm of each single-component pesticide target analyte, 1000 ppm of chlordane, and 1000 ppm of toxaphene in acetone or other acetone miscible solvent. The spike standard solution must be of a source differing from that used for the initial five-point calibration. If it is of the same source, then it must be of different lot.

10.2.7.1. Prepare the spike working standard solutions by diluting the appropriate volumes of the spike stock standard solutions with acetone.

10.2.7.2. The single-component pesticide spike working standard solutions are prepared as follows:

Analyte	Initial		Final	
	Conc. (ppm)	Volume (mL)	Conc. (ppm)	Volume (mL)
organochlorine pesticides	200	0.5	1.0	100
hexachlorobenzene	1000	0.1		
2,4'-DDD	100	0.1	1.0	10
2,4'-DDE	100	0.1		
2,4'-DDT	100	0.1		
cis-nonachlor	100	0.1		
trans-nonachlor	100	0.1		

10.2.7.3. The standard is used to prepare QC check samples such as matrix spikes (MS/MSDs) and laboratory control samples (LCS/LCSDs).

10.2.7.4. Add 500 µL of the single-component pesticide spike working standard to each MS/MSD and LCS/LCSD sample prior to extraction.

10.2.7.5. Add 5 µL of chlordane spike stock standard solution to each MS/MSD and LCS/LCSD sample prior to extraction.

10.2.7.6. Add 10 µL of toxaphene spike stock standard solution to each MS/MSD and LCS/LCSD sample prior to extraction.

10.2.7.7. Add 500 µL of the single-component pesticide spike working standard to each mobility-procedure extract designated as MS/MSD and LCS/LCSD prior to extraction.

10.2.8. All working standards must be replaced after six months (unless specified otherwise) or sooner if comparison with check standards indicates a problem.

10.2.9. All stock standards must be inspected and documented in the Chemicals and Supplies Verification Logbook prior to use.

11. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

11.1. Aqueous samples should be collected in 1-L pre-cleaned amber glass containers with Teflon-lined closures. Collect all samples in duplicate.

11.1.1. If the aqueous sample contains residual chlorine, add 4 mL of the 10% $Na_2S_2O_3$ solution per 1 L of sample. The 10% $Na_2S_2O_3$ solution may be added to the sample container prior to sample collection.

11.2. Solid samples should be collected in 4-oz or 8-oz pre-cleaned clear glass wide-mouth jars, or 6-in decontaminated stainless steel or brass sleeves with Teflon-lined closures.

11.3. Oil, wipe, and filter samples should be collected in 40-mL pre-cleaned clear or amber glass VOA vials with Teflon-lined closures.

11.4. Tissue samples should be collected in 4-oz (or other appropriate capacity) pre-cleaned clear glass or quartz wide-month jars with Teflon or aluminum foil-lined closures. Refer to SOP-M229 for additional information on sample collection, preservation, and containers.

11.5. Mobility-procedure extracts should be collected in 500-mL pre-cleaned amber glass containers with Teflon-lined closures.

11.5.1. If the mobility-procedure extract will not be solvent extracted within 24 hours, add 2 mL of the 10% $Na_2S_2O_3$ solution per 500 mL of sample.

11.5.2. Completely fill and hermetically seal the sample container with minimum headspace.

11.6. Aqueous, solid, oil, wipe, and filter samples shall be maintained in a chilled state (≤ 4°C) post sample collection until received at the laboratory. Aqueous, solid, oil, wipe, or filter samples should not be frozen (e.g., do not use dry ice as the refrigerant).

11.7. Tissue samples shall be maintained in a frozen state (≤ −20°C) post sample collection until received at the laboratory. Refer to SOP-M229 for additional information on sample collection and preservation.

11.8. Upon receipt, the aqueous, solid, oil, wipe, and filter samples are stored in a 0~6°C cooler, and the tissue samples are stored in a −10~−20°C freezer.

11.8.1. Aqueous samples must be solvent extracted within 7 days of sample collection.

11.8.2. Non-aqueous samples must be solvent extracted within 14 days of sample collection.

11.8.3. Mobility-procedure extracts must be solvent extracted within 7 days post mobility extraction.

11.8.3.1. Mobility-procedure extracts shall be stored in a 0~6°C cooler post mobility extraction if solvent extraction is not to be performed within 24 hours.

11.8.4. All solvent extracts are then stored under dark and refrigerated (0~6°C) conditions and must be analyzed within a 40-day period post extraction.

12. QUALITY CONTROL

12.1. Degradation Test

12.1.1. Prior to running the calibration standards, the degradation test standard solution must be analyzed and meet the following acceptance criteria:

12.1.1.1. The degradation (or percent breakdown) of 4,4'-DDT and endrin shall be ≤ 15% for each compound. The formula for calculating %B is listed in Section 15.14.

12.1.2. These criteria must be demonstrated every 12 hours.

12.1.3. If these criteria are not met, then the analytical system is deemed unacceptable for sample analysis to begin. Effect corrective action, rerun the degradation test, and recalibrate.

12.2. Initial Calibration (IC)

12.2.1. The initial five-point calibration must be established prior to the processing of sample extracts.

12.2.1.1. The calibration curve is established with five calibration standards.

12.2.2. The IC is deemed valid if the %RSD for each analyte is ≤ 20%.

12.2.3. If these criteria are not met, then the calibration is unacceptable for sample analysis to begin. Effect corrective action and recalibrate.

12.2.3.1. If the problem appears to be associated with a single calibration standard, then that one standard may be reanalyzed once within the same analytical shift prior to sample analysis.

12.3. Initial Calibration Verification (ICV)

12.3.1. The initial calibration is deemed valid if the %D for each analyte is ≤ 15%.

12.3.2. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to begin. An unacceptable ICV result indicates either a disagreement between like solutions from separate sources or a change in instrument conditions. Normally, this is caused when at least one of the solutions is no longer intact (representative of the stated concentration). Document the unacceptable result and reanalyze the ICV within 2 hours after the failed ICV. If the ICV criteria remain unacceptable, investigate, effect corrective action, which may include re-preparation of standard solutions or instrument maintenance, and recalibrate.

12.4. Continuing Calibration Verification (CCV)

12.4.1. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily prior to sample analysis, after every batch of 20 samples or portion thereof within a 12-hour shift, and at the end of sequence.

12.4.1.1. For EPA Region 9 requirement, a CCV standard must be analyzed daily prior to sample analysis, after every batch of 10 samples or portion thereof within a 12-hour shift, and at the end of sequence.

12.4.2. The initial calibration is deemed valid if the %D for each analyte is ≤ 15%.

12.4.3. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to resume. Document the unacceptable result and reanalyze the CCV within 2 hours after the failed CCV. If the CCV criteria remain unacceptable, effect corrective action and recalibrate.

12.5. Retention Time Window

12.5.1. Establishment of retention time window width is accomplished by making three injections of mid-range CCV standards throughout the course of a 72-hour period. Serial injections over a shorter period of time may result in narrow retention time window width that does not accurately account for variations over several days.

12.5.1.1. Retention time window width is ± 3S (where S is the standard deviation of the three retention times for that analyte/surrogate) or ± 0.030 minute, whichever is greater.

12.5.2. Establishment of retention time window position is accomplished by using the midpoint calibration standard once per initial calibration, and by using a mid-range CCV standard at the beginning of an analytical sequence.

12.5.2.1. When initial calibration is performed, daily retention time window for each analyte/surrogate is the retention time of the analyte/surrogate in the midpoint calibration standard ± 3S or ± 0.030 minute, whichever is greater.

12.5.2.2. When initial calibration is <u>not</u> performed, daily retention time window for each analyte/surrogate is the retention time of the analyte/surrogate in the mid-range CCV standard ± 3S or ± 0.030 minute, whichever is greater.

12.5.3. Retention time for each analyte/surrogate in the calibration verification standard is verified as follows:

12.5.3.1. When initial calibration is performed, the ICV standard and all CCV standards throughout the course of an analytical sequence within a 12-hour shift must fall within the daily retention time window established by the midpoint calibration standard.

12.5.3.2. When initial calibration is <u>not</u> performed, all succeeding CCV standards throughout the course of an analytical sequence within a 12-hour shift must fall within the daily retention time window established by the first CCV standard.

12.5.3.3. If these criteria are not met, determine the cause of the problem, effect corrective action, and re-establish the retention time window width and/or position, if necessary.

12.6. Event Based Quality Control (LCS/LCSDs and MBs)

12.6.1. Event based quality control consists of QC samples prepared and processed with each preparatory event. This consists of a laboratory control sample and laboratory control sample duplicate (LCS/LCSD) and a method blank (MB).

12.6.2. The acceptance criteria for LCS/LCSD compounds are as follows:

12.6.2.1. The lower and upper acceptance limits for %REC of each LCS/LCSD compound are 50% and 135%, respectively. The RPD is ≤ 25%.

12.6.2.2. All LCS/LCSD compounds must be within acceptance limits. However, if a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside of control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary. Lower and upper marginal exceedance (ME) limits can be established to determine when corrective action is necessary.

12.6.2.3. ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.

12.6.2.4. The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than 11 analytes.

12.6.2.5. The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number of Analytes Allowed in ME of the LCS Control Limit
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

12.6.2.6. Marginal exceedances must be random. If the same analyte exceeds the LCS control limit 2 out of 3 consecutive LCS, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.

12.6.3. Ideally, the concentrations of target analytes in an MB should be less than the respective reporting limits (RLs). If the concentration of any target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs are as follows:

12.6.3.1. If a target analyte is found in the MB, but not in the associated samples, report the sample and MB data without qualification.

12.6.3.2. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the

effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified, or rejected and the samples re-extracted and/or re-analyzed.

12.7. Matrix Based Quality Control (Surrogates and MS/MSDs)

12.7.1. Matrix based quality control consists of QC samples prepared and processed using actual environmental samples. This consists of a matrix spike and matrix spike duplicate (MS/MSD) and surrogates added to each sample.

12.7.2. The acceptance criteria for surrogate compounds are as follows:

12.7.2.1. The lower and upper acceptance limits for %REC of each surrogate compound in an aqueous sample are 50% and 135%, respectively. The lower and upper acceptance limits for %REC of each surrogate compound in a non-aqueous sample are 50% and 130%, respectively.

12.7.2.1.1. For EPA Region 9 requirement, the lower and upper acceptance limits for %REC of each surrogate compound are 60% and 150%, respectively.

12.7.2.2. If the surrogate compound recoveries are acceptable, report the surrogates and sample data without qualification.

12.7.2.3. If one or more surrogate recoveries are not acceptable, evaluation is not necessarily straightforward. The sample itself may produce effects due to factors such as interferences and high analyte concentration or a problem may have occurred during extraction or cleanup. The data alone cannot be used to evaluate the precision and accuracy of individual sample analysis. However, when exercising professional judgment, this data should be used in conjunction with other available QC information.

12.7.2.4. By itself, unacceptable surrogate recoveries do not invalidate sample data. The following must be accomplished if surrogate recoveries are not acceptable.

12.7.2.4.1. Check the surrogate standard solutions for degradation and contamination.

12.7.2.4.2. If the nonconformance is due to poor instrument performance or if the above actions fail to reveal the cause of the unacceptable surrogate(s) recovery, the same extract should be re-analyzed.

12.7.2.4.3. If incorrect procedures or degraded/contaminated standard solutions are determined to have not caused the unacceptable surrogate recoveries, the

affected sample(s) must be re-extracted and re-analyzed or, if insufficient sample remains, reference made to the associated MB surrogate recoveries and the sample data reported with qualification.

12.7.2.4.3.1. If, upon re-extraction and re-analysis, the surrogates remain unacceptable, matrix interference can be cited and reference made to the associated MB surrogate recoveries and the sample data reported with qualification.

12.7.2.4.3.2. If the MB surrogates are unacceptable, all associated sample data must be invalidated and all associated samples re-extracted and re-analyzed.

12.7.2.5. Where sample dilution is required, depending on the dilution factor, the surrogate recovery will be low or not detected. This is an expected occurrence and reference should be made to the MB surrogate recovery which must be reported to the client.

12.7.3. The acceptance criteria for MS/MSD compounds are as follows:

12.7.3.1. The lower and upper acceptance limits for %REC of each MS/MSD compound are 50% and 135%, respectively. The RPD is ≤ 25%.

12.7.3.2. When the %REC and RPD of the MS/MSD compounds are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.

12.7.3.3. If the %REC and/or RPD of the MS/MSD compounds are not within the established acceptance limits, the analytical system performance shall be suspect.

12.7.4. Unacceptable %REC values are typically caused by matrix effects or poor instrument performance/technique. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.

12.8. If the %REC or RPD of the MS/MSD and LCS/LCSD are unacceptable, all associated sample data must be invalidated and all associated samples re-extracted and re-analyzed.

12.9. Additional information regarding internal quality control checks is provided in SOP-T020.

13. CALIBRATION AND STANDARDIZATION

13.1. Prior to the analysis of field or QC sample extracts, the GC system must be shown to be resistant to the breakdown of 4,4'-DDT and endrin, and an acceptable five-point calibration established. The acceptance criteria for the parameters are listed in Sections 12.1. and 12.2.

13.2. Initial Calibration

13.2.1. Establish an acceptable five-point calibration curve. The acceptance criteria for the initial calibration are listed in Section 12.2.

13.2.1.1. Because of the sensitivity of the electron capture detector, always clean the injection port and column prior to performing the initial calibration.

13.2.1.2. Recalibration is required for the following maintenance procedures.

13.2.1.2.1. Change, replace, or reverse the analytical column.

13.2.2. After obtaining an acceptable five-point calibration curve and prior to processing field or QC sample extracts, an ICV standard must be analyzed to verify the initial calibration. The acceptance criteria for the ICV are listed in Section 12.3.

13.2.3. The initial five-point calibration and ICV shall include all anticipated target analytes for the duration of the use of the initial calibration.

13.3. Retention time window width for each analyte/surrogate is generated by running three mid-range CCV standards over a 72-hour period. Retention time window width determination shall be performed at method set-up, following column changes, after major instrument maintenance or when a significant retention time shift is suspected.

13.3.1. Document the serial number of the analytical column associated with the retention time window study.

13.3.2. Record the retention time in minutes for each analyte/surrogate to three decimal places.

14. PROCEDURE

14.1. Instrument Setup

14.1.1. Use the following GC operating conditions as guidance to establish the GC temperature program and flow rate necessary to separate the analytes of interest.

Description	GC Operating Condition
Front inlet mode	splitless
Carrier gas flow rate	1~3 mL/min at 8.73~10.15 psi
Makeup gas flow rate	30.0 mL/min
Detector temperature	300°C
Initial temperature	100°C
Temperature program	100°C to 200°C at 25.00°C/min 200°C to 225°C at 2.50°C/min 225°C to 300°C at 20.00°C/min
Final temperature	300°C, hold 6.25 min

14.1.2. Autoinjector is set to inject 2 µL of field or QC sample extract.

14.2. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily prior to sample analysis, after every batch of 20 samples or portion thereof within a 12-hour shift, and at the end of sequence. If the QC and retention time criteria are met, the initial calibration is assumed to be valid and sample analysis may resume. The acceptance criteria are listed in Section 12.4. and Section 12.5.3.

14.2.1. For EPA Region 9 requirement, refer to Section 12.4.1.1. for CCV frequency.

14.2.2. If a failed CCV is the first of the day, effect corrective action prior to analyzing any samples.

14.2.3. If a failed CCV is <u>not</u> the first of the day, effect corrective action and reanalyze all samples since the last acceptable CCV.

14.3. Following extraction by one of the methods specified in Section 5.2., the extracts for the QC and actual environmental samples are received in autoinjector vials. The autoinjector vials are then loaded onto the GC sample tray.

14.4. Standard and sample vials are loaded in the following or other logical order:

1) Degradation Test
2) Continuing Calibration Verification (CCV)
3) Laboratory Control Sample (LCS)
4) Laboratory Control Sample Duplicate (LCSD)
5) Method Blank (MB)
6) Samples (up to 20 per batch, including QC check samples and MBs)
7) Matrix Spike (MS)
8) Matrix Spike Duplicate (MSD)
9) Ending CCV

14.4.1. Item 1: An acceptable degradation test demonstrates that the chromatographic system is not causing breakdown of thermally labile compounds due to active sites (e.g., the injection port is contaminated or contains catalytic active sites). A degradation test meeting the acceptance criteria is required daily prior to sample analysis and every 12 hours thereafter during analysis.

14.4.2. Items 2 and 9: A CCV is used to verify the acceptance of the initial five-point calibration on a continuing basis. An acceptable CCV is required daily prior to sample analysis, after every batch of 20 samples or portion thereof within a 12-hour shift, and at the end of sequence.

14.4.2.1. For EPA Region 9 requirement, refer to Section 12.4.1.1. for CCV frequency.

14.4.2.2. More frequent (e.g., every 10 samples) calibration verification may be useful to minimize the number of sample extract re-analyses that would be required in the event of an unacceptable CCV.

14.4.3. Item 3: The LCS is a known matrix which has been spiked with known concentrations of specific target analytes. The purpose of the LCS is to demonstrate that the entire analytical process and systems are in control. The LCS is processed concurrently with the associated samples. In the processing of the LCS, reagents and procedures identical to those for actual samples are used.

14.4.3.1. For aqueous samples, the LCS consists of the specified compounds spiked into clean reagent water. For solid, oil, ***and tissue*** samples, the LCS consists of the specified compounds spiked into washed sea sand. For wipe samples, the LCS consists of the specified compounds spiked into unused gauze pad. For filter samples, the LCS consists of the specified compounds spiked into unused filter paper. ***For mobility-procedure extracts, the LCS consists of the specified compounds spiked into the mobility-procedure extract designated as LCS.***

14.4.3.2. One LCS is required every day ***preparatory methods (i.e., extractions, cleanups, etc.)*** are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

14.4.4. Item 4: The LCSD is handled identically to the LCS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the LCS in combination with the LCSD can be used to assess the precision of the analytical process. The measurement is expressed as relative percent difference (RPD). The formula for calculating RPD is listed in Section 15.6.

14.4.5. Item 5: The MB is a known matrix similar to the samples being analyzed which is processed concurrently with the associated samples. In the processing of the MB, reagents and procedures identical to those for actual samples are used (e.g., surrogates, etc.).

14.4.5.1. For aqueous samples, the MB consists of clean reagent water. For solid, oil, ***and tissue*** samples, the MB consists of washed sea sand. For wipe samples, the MB consists of unused gauze pad. For filter samples, the MB consists of unused filter paper.

For mobility-procedure extracts, the MB consists of the mobility-procedure extract designated as MB.

14.4.5.2. One MB is required every day preparatory methods (i.e., extractions, cleanups, etc.) are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

14.4.5.3. When samples that are processed together are analyzed on separate instruments or on separate analytical shifts, the MB associated with those samples must be analyzed on at least one of the instruments. A solvent blank consisting of hexane must be analyzed on all other instruments where the associated samples are analyzed to demonstrate that the instruments are not contributing contaminants to the samples.

14.4.6. Item 6: Up to 20 sample (including QC check sample and method blank) extracts per batch. Complex extracts should be sufficiently diluted or subjected to cleanup procedures to ensure that instrument is not contaminated. Dilution or cleanup of extracts will result in increased reporting limits.

14.4.6.1. All dilutions should keep the responses of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

14.4.7. Item 7: The MS is the actual sample matrix spiked with known concentrations of specific target analytes. The sample which is spiked for the MS is processed concurrently with the associated samples. In the processing of the MS, reagents and procedures identical to those for actual samples are used.

14.4.7.1. The purpose of the MS is to assess the effect of a sample matrix on the recovery of target analytes (i.e., assess the accuracy of the analytical measurements of the matrix). The measurement is expressed as percent recovery (%REC). The formula for calculating %REC is listed in Section 15.5.

14.4.7.2. One MS is required for every batch of 20 samples per matrix or portion thereof processed concurrently. This approach is considered "closed batch" as opposed to "open batch."

14.4.8. Item 8: The MSD is handled identically to the MS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the MS in combination with the MSD can be used to assess the precision of the analytical measurements. The measurement is expressed as relative percent difference (RPD). The formula for calculating RPD is listed in Section 15.6.

14.4.9. Solvent blanks may be added elsewhere in the sequence, as necessary (i.e., after suspected high concentration sample extracts), to check for potential carryover or cross-contamination.

14.5. Ensure that a sufficient amount of hexane is present in the autoinjector solvent rinse bottles and that a sufficient unused volume exists in the autoinjector waste bottles at the beginning of the sequence.

14.6. Edit the sequence in the data system. After all correct sample information is entered, save the sequence. After saving the sequence, record pertinent information in the instrument run logbook or on the sequence table printout.

14.7. Initiate the sequence.

14.8. Data Interpretation

14.8.1. Establish the daily retention time window for each analyte/surrogate (see Section 12.5.2.1. and Section 12.5.2.2.).

14.8.1.1. Tentative identification of an analyte/surrogate occurs when a peak from a sample extract falls within the daily retention time window.

14.8.1.1.1. For chlordane or toxaphene, choose a minimum of 5 characteristic peaks that are at least 25% of the height of the largest characteristic peak for the chlordane or toxaphene, and determine the retention time window of each characteristic peak.

14.8.1.2. Use the succeeding CCV standards analyzed throughout the course of an analytical sequence within a 12-hour shift to evaluate retention time stability (see Section 12.5.3.). If any analyte(s)/surrogate(s) in the CCV standard fall outside of their daily retention time window(s), determine the cause of the problem and effect appropriate corrective action.

14.8.1.2.1. If any analyte(s)/surrogate(s) in the single-component pesticide CCV standard fall outside of their daily retention time window(s), then all samples analyzed since the last acceptable CCV should be invalidated, corrective action effected, and the affected samples re-analyzed.

14.8.1.2.2. If any surrogate(s) in the chlordane or toxaphene CCV standard fall outside of their daily retention time window(s), then all samples analyzed since the last acceptable CCV should be invalidated, corrective action effected, and the affected samples re-analyzed.

14.8.2. Quantitation of a target analyte is based on a reproducible response of the detector within the calibration range and a direct proportionality of the magnitude of response between peaks in the sample extract and the calibration standards.

14.8.2.1. Proper quantitation requires the appropriate selection of a baseline from which the area of the characteristic peak(s) can be determined.

14.8.2.1.1. For toxaphene quantitation, a forced baseline or baseline-to-baseline integration across the entire target range is required to ensure the appropriate integration of toxaphene response.

14.8.2.2. Determine the concentration based on the initial calibration curve.

14.8.2.2.1. Calculate the concentration of each single-component pesticide target analyte in a sample extract using the average of the initial RFs and the area of the characteristic peak. The formula for calculating concentration is listed in Section 15.7.

14.8.2.2.2. Calculate the concentration of chlordane or toxaphene in a sample extract using the average of the initial RFs and the total area of the five predetermined peaks. The formula for calculating concentration is listed in Section 15.7.

14.8.2.2.3. Refer to Appendix A for examples of the chlordane and toxaphene predetermined peaks.

14.8.2.2.4. The data system is programmed to perform the calculation of concentration.

14.8.2.3. If the instrument response exceeds the calibration range, dilute the extract and reanalyze.

14.8.3. Tentative identification of a target analyte occurs when a peak from a sample extract falls within the analyte's retention time window. Confirmation is necessary when the composition of samples is not well characterized. Qualitative confirmation techniques are by second column with dissimilar stationary phase, GC/MS with Selected Ion Monitoring (SIM) or Full Scan mode, or GC data from two different detectors.

14.8.4. Second column confirmation is made on a "confirmation" channel configured with a column of dissimilar stationery phase and a second detector. The principle is that the retention time of the target analyte will differ between the primary and confirmation column and, unless the detected compound is the particular target analyte, it will not be observed within both retention time windows.

14.8.4.1. Report the higher result between the primary and confirmation column. The RPD between results must be ≤ 40%.

14.8.4.1.1. If one result is significantly higher (e.g., > 40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high

result. If no overlapping peaks are observed, examine the baseline parameters established by the instrument data system (or operator) during peak integration. A rising baseline may cause the mis-integration of the peak for the lower result.

14.8.4.1.2. If no anomalies are observed, review the chromatographic conditions. If there is no evidence of chromatographic problems, then it may be appropriate to report the lower result.

14.8.4.1.3. The data user must be advised of the disparity between the results on the two columns. Under some circumstances, including those involving in monitoring compliance with an action level or regulatory limit, further cleanup of the sample or additional analyses may be required when the two results in question span the action level or regulatory limit.

14.8.4.2. In cases where a peak is not observed in the confirmation column's retention time window, the analyte is reported as "ND."

14.8.4.3. A calibration curve and retention time window for each analyte/surrogate are also established and maintained for the confirmation channel. The calibration and quality control requirements for the confirmation channel are identical to those of the primary channel.

14.8.5. GC/MS confirmation is more reliable than second column confirmation. In this case, where confirmation is required by project requirements, the sample is re-analyzed on GC/MS. When GC/MS results indicate that a target analyte is not present, the GC result is reported as "ND."

14.8.6. Confirmation is required for all positive results unless the samples meet all of the following requirements:

14.8.6.1. All samples (liquid, solid, or oil) come from the same source (e.g., same monitoring well). However, samples of the same matrix from the same site but from differing sources (e.g., different monitoring wells) are not exempted.

14.8.6.2. All chemical parameters have been previously analyzed, identified, and confirmed by a second column with dissimilar stationary phase, GC/MS with Selected Ion Monitoring (SIM) or Full Scan mode, or GC data from two different detectors. Documentation of such must be maintained.

14.8.6.3. The resulting chromatograms are relatively simple and do not contain complex or overlapping peaks.

14.8.6.4. Chromatograms are largely unchanged from those for which confirmation was carried out.

14.8.7. Qualitative confirmation by a second GC column is not required for determining chlordane and toxaphene.

14.8.8. Manual integration of peaks shall adhere to the procedures and documentation policies outlined in the current revision of SOP-T023.

14.9. Recommended Instrument Maintenance

14.9.1. Perform the following tasks to remedy the column adsorption problem.

14.9.1.1. Inject an 800-ppb single-component pesticide standard solution to prime (or deactivate) the column.

14.9.1.2. Run one or more solvent blanks consisting of hexane until no carryover is observed prior to analyzing any standards or samples.

14.9.2. Perform the following tasks to eliminate the degradation problem.

14.9.2.1. For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter.

14.9.2.2. Break off the first few centimeters (up to 30 cm) of the injection port side of the column.

14.9.2.3. Check the injector temperature and lower it to 205°C, if necessary.

14.9.2.4. Remove the columns and solvent backflush according to the manufacturer's instructions.

14.9.2.5. If all else fail, it may be necessary to deactivate the metal injector body and/or replace the columns.

14.9.3. Perform the following tasks to rinse the analytical column.

14.9.3.1. Depending on the nature of the residues expected, the first rinse might be reagent water, followed by methanol and acetone, with methylene chloride as the final rinse. In some cases, methylene chloride may be the only solvent necessary.

14.9.3.2. After the final rinse, the analytical column should be filled with methylene chloride and remained flooded overnight to allow materials within the stationary phase to migrate into the solvent.

14.9.3.3. The analytical column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen passing through the column.

15. CALCULATIONS

15.1. The response factor is calculated as follows:

$$RF = \frac{A_x}{C_x}$$

where: RF = response factor for target analyte being measured.
A_x = area of the characteristic peak(s) for target analyte being measured.
C_x = concentration of target analyte being measured in µg/L.

15.2. The percent relative standard deviation is calculated as follows:

$$\%RSD = \frac{SD}{RF_{ave}} \times 100$$

where: %RSD = percent relative standard deviation.
SD = standard deviation of the RFs for the target analyte.
RF_{ave} = mean of the 5 initial RFs for the target analyte.

15.3. The percent difference of each analyte is calculated as follows:

$$\%D = \frac{|RF_{ave} - RF_{daily}|}{RF_{ave}} \times 100$$

where: %D = percent difference.
RF_{daily} = daily RF for the target analyte.
RF_{ave} = mean of the 5 initial RFs for the target analyte.

15.4. The recovery of each LCS compound is calculated as follows:

$$\%REC_{LCS} = \frac{C_{recovered}}{C_{added}} \times 100$$

where: $\%REC_{LCS}$ = percent recovery of target analyte in LCS (or LCSD).
$C_{recovered}$ = concentration of target analyte recovered.
C_{added} = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

15.5. The recovery of each MS compound is calculated as follows:

$$\%REC_{MS} = \frac{C_{recovered} - C_{sample}}{C_{added}} \times 100$$

where: $\%REC_{MS}$ = percent recovery of target analyte in MS (or MSD).
$C_{recovered}$ = concentration of target analyte recovered.
C_{sample} = concentration of target analyte in environmental sample used.

C_{added} = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

15.6. The relative percent difference is calculated as follows:

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where: RPD = relative percent difference between two measurements (C_1 and C_2).
C_1 = concentration of target analyte in measurement 1.
C_2 = concentration of target analyte in measurement 2.

Note: Concentrations must be in equivalent units.

15.7. The target analyte concentration for a sample extract is calculated as follows:

$$C_{ex} = \frac{A_x}{RF_{ave}}$$

where: C_{ex} = concentration of target analyte in extract in µg/L.
A_x = area of the characteristic peak(s) for target analyte.
RF_{ave} = mean of the 5 initial RFs for the target analyte.

15.8. The target analyte concentration for an aqueous sample is calculated as follows:

$$C_A = \frac{C_{ex} \times V_{ex} \times D}{V_A}$$

where: C_A = concentration of target analyte in aqueous sample in µg/L.
C_{ex} = concentration of target analyte in extract in µg/L.
V_{ex} = volume of extract in mL.
V_A = volume of aqueous sample ***solvent*** extracted in mL.
D = dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1.

15.9. The target analyte concentration for a solid (or oil) sample is calculated as follows:

$$C_S = \frac{C_{ex} \times V_{ex} \times D}{W_S}$$

where: C_S = concentration of target analyte in solid (or oil) sample in µg/kg.
C_{ex} = concentration of target analyte in extract in µg/L.
V_{ex} = volume of extract in mL.
W_S = mass of solid (or oil) sample ***solvent*** extracted in g.

D = dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1.

15.10. The target analyte concentration for a solid sample on a dry-weight basis is calculated as follows:

$$C_S = \frac{C_{ex} \times V_{ex} \times D}{W_S \times \left(\frac{C_{ss}}{100}\right)}$$

where:
C_S = concentration of target analyte in solid sample in µg/kg.
C_{ex} = concentration of target analyte in extract in µg/L.
V_{ex} = volume of extract in mL.
W_S = mass of solid sample solvent extracted in g.
C_{ss} = solids content in %.
D = dilution factor, if the extract was diluted prior to analysis. If no dilution was made, D = 1.

15.11. The target analyte concentration for a wipe (or filter) sample is calculated as follows:

$$C_W = C_{ex} \times V_{ex} \times D$$

where:
C_W = concentration of target analyte in wipe (or filter) sample in µg/sample.
C_{ex} = concentration of target analyte in extract in µg/L.
V_{ex} = volume of extract in L.
D = dilution factor, if the extract was diluted prior to analysis. If no dilution was made, D = 1.

15.12. The target analyte concentration for a tissue sample is calculated as follows:

$$C_T = \frac{C_{ex} \times V_{ex} \times D}{W_T}$$

where:
C_T = concentration of target analyte in tissue sample in µg/kg.
C_{ex} = concentration of target analyte in extract in µg/L.
V_{ex} = volume of extract in mL.
W_T = mass of tissue sample solvent extracted in g.
D = dilution factor, if the extract was diluted prior to analysis. If no dilution was made, D = 1.

15.13. The target analyte concentration for a mobility-procedure extract is calculated as follows:

$$C_{MP} = \frac{C_{ex} \times V_{ex} \times D}{V_{MP}}$$

where: C_{MP} = concentration of target analyte in mobility-procedure extract in µg/L.
C_{ex} = concentration of target analyte in extract in µg/L.
V_{ex} = volume of extract in mL.
V_{MP} = volume of mobility-procedure extract solvent extracted in mL. Unless specified otherwise, V_{MP} = 100.
D = dilution factor, if the extract was diluted prior to analysis. If no dilution was made, D = 1.

15.14. The percent breakdown is calculated as follows:

15.14.1. The percent breakdown of DDT is calculated as follows:

$$\%B_{DDT} = \frac{A_{DDD} + A_{DDE}}{A_{DDT} + A_{DDD} + A_{DDE}} \times 100$$

where: $\%B_{DDT}$ = percent breakdown of DDT.
A_{DDD} = degradation peak area of DDD.
A_{DDE} = degradation peak area of DDE.
A_{DDT} = peak area of DDT.

15.14.2. The percent breakdown of endrin is calculated as follows:

$$\%B_{endrin} = \frac{A_{aldehyde} + A_{ketone}}{A_{endrin} + A_{aldehyde} + A_{ketone}} \times 100$$

where: $\%B_{endrin}$ = percent breakdown of endrin.
$A_{aldehyde}$ = degradation peak area of aldehyde.
A_{ketone} = degradation peak area of ketone.
A_{endrin} = peak area of endrin.

15.15. Refer to the preparatory method(s) for additional calculations.

15.16. All concentrations shall be reported in µg/L (ppb) for aqueous samples, µg/kg (ppb) for oil, soil and solid waste samples, µg/sample for wipe and filter samples, and µg/kg (ppb) for tissue samples.

15.16.1. For EPA Region 9 requirement, report all concentrations in µg/L (ppb) for water samples, and µg/kg (ppb) on a dry-weight basis for soil samples.

15.17. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

16. METHOD PERFORMANCE

16.1. A demonstration of analytical capability shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

16.2. Calibration protocols specified in Section 13., "Calibration and Standardization," shall be followed.

16.3. Proficiency test sample results shall be used to evaluate the ability to produce accurate results.

17. POLLUTION PREVENTION

17.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Calscience's Health, Safety, and Respiratory Protection Manual. In general, protective eyewear (e.g. safety glasses or goggles), and protective apparel (e.g. lab coats) and gloves are required to be worn when handling chemicals.

17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:

17.3.1. A NIOSH approved air purifying respirator with cartridges appropriate for the chemical handled.

17.3.2. Extended length protective gloves.

17.3.3. Face shield.

17.3.4. Full-length laboratory apron.

17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.

17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles. Alternatively, the Coordinator may utilize a self-contained breathing apparatus or other supplied air system if appropriately trained and approved by the Health and Safety Manager.

17.6. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

18.1. The acceptance criteria for LCS/LCSD compounds are predetermined. The lower and upper acceptance limits for %REC of each LCS/LCSD compound are 50% and

135%, respectively. The RPD is ≤ 25%. All LCS/LCSD compounds must be within acceptance limits (see Section 12.6.2. for additional information).

18.1.1. If the LCS and/or LCSD %REC is outside of the acceptance limits high, the RPD is within acceptance limits, and all target analytes in the associated samples are not detected, the sample data can be reported without qualification.

18.1.2. The LCSD is only reported when the MS/MSD is unacceptable due to matrix interference effects, or when the LCS/LCSD is used in place of MS/MSD due to insufficient sample quantity.

18.2. Ideally, the concentrations of target analytes in an MB should be less than the respective reporting limits (RLs). If the concentration of any target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs are as follows:

18.2.1. If a target analyte is found in the MB but not in the associated samples, report the sample and MB data without qualification.

18.2.2. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-extracted and/or re-analyzed.

18.3. The acceptance criteria for surrogate compound recoveries are predetermined. The lower and upper acceptance limits for %REC of each surrogate compound in an aqueous sample are 50% and 135%, respectively. The lower and upper acceptance limits for %REC of each surrogate compound in a non-aqueous sample are 50% and 130%, respectively.

18.3.1. For EPA Region 9 requirement, refer to Section 12.7.2.1.1. for acceptance criteria.

18.3.2. If the surrogate compound recoveries are acceptable, report the surrogates and sample data without qualification.

18.3.3. If one or more surrogate recoveries are not acceptable, evaluation is not necessarily straightforward. The sample itself may produce effects due to factors such as interferences and high analyte concentration. This data alone cannot be used to evaluate the precision and accuracy of individual sample analysis. However, when exercising professional judgment, this data should be used in conjunction with other available QC information.

18.3.4. By itself, unacceptable surrogate recoveries do not invalidate sample data. The following must be accomplished if surrogate recoveries are not acceptable.

18.3.4.1. Check the surrogate standard solutions for degradation and contamination.

18.3.4.2. If the nonconformance is due to poor instrument performance or if the above actions fail to reveal the cause of the unacceptable surrogate(s) recovery, the same extract should be re-analyzed.

18.3.4.3. If incorrect procedures or degraded/contaminated standard solutions are determined to have not caused the unacceptable surrogate recoveries, the affected sample(s) must be re-extracted and re-analyzed or, if insufficient sample remains, reference made to the associated MB surrogate recoveries and the sample data reported with qualification.

18.3.4.3.1. If, upon re-extraction and re-analysis, the surrogates remain unacceptable, matrix interference can be cited and reference made to the associated MB surrogate recoveries and the sample data reported with qualification.

18.3.4.3.2. If the MB surrogates are unacceptable, all associated sample data must be invalidated and all associated samples re-extracted and re-analyzed.

18.3.5. Where sample dilution is required, depending on the dilution factor, the surrogate recovery will be low or not detected. This is an expected occurrence and reference should be made to the MB surrogate recovery which must be reported to the client.

18.4. The acceptance criteria for MS/MSD compounds are predetermined. The lower and upper acceptance limits for %REC of each MS/MSD compound are 50% and 135%, respectively. The RPD is ≤ 25%.

18.4.1. When the %REC and RPD of the MS/MSD compounds are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.

18.4.2. If the %REC and/or RPD of the MS/MSD compounds are not within the established acceptance limits, the analytical system performance shall be suspect.

18.5. Matrix effects or poor instrument performance/technique typically cause unacceptable %REC values. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.

18.6. Additional information regarding internal quality control checks is provided in SOP-T020.

18.7. All concentrations shall be reported in µg/L (ppb) for aqueous samples, µg/kg (ppb) for oil, soil and solid waste samples, µg/sample for wipe and filter samples, and µg/kg (ppb) for tissue samples.

18.7.1. For EPA Region 9 requirement, report all concentrations in µg/L (ppb) for water samples, and µg/kg (ppb) on a dry-weight basis for soil samples.

18.8. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

19. CORRECTIVE ACTIONS

19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.

19.2. The Operations Manager, Project Manager, Quality Control Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.

19.3. Corrective actions are generally of two types, immediate and long-term actions.

19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.

19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented in the Corrective Action Record. Examples of this type of action include:

19.3.2.1. Remedial training of staff in technical skills, technique or implementation of operating procedures.

19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.

19.3.2.3. Revision of standard operating procedures.

19.3.2.4. Replacing personnel, as necessary.

19.4. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:

19.4.1. Define the problem.

19.4.2. Assign responsibility for investigating the problem.

19.4.3. Investigate and determine the cause of the problem.

19.4.4. Assign and accept responsibility for implementing the corrective action.

19.4.5. Determine effectiveness of the corrective action and implement correction.

19.4.6. Verify that the corrective action has eliminated the problem.

19.5. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

20. CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1. Out-of-control data are reviewed and verified by the technical director of the appropriate department. All samples associated with an unacceptable QC set are then subject to reanalysis, depending upon the QC type in question.

20.1.1. MS/MSD: Acceptability of the MS/MSD recoveries is subject to the matrix and any anomalies associated with the subject batch. Failure of recoveries of an MS/MSD data set does not constitute an automatic reanalysis of the batch samples. Rather, it is acceptable to defer to the LCS/LCSD recoveries, to determine acceptance of the sample results.

20.1.2. LCS/LCSD: Because they denote whether the analytical system is operating within control, it is imperative that the LCS recoveries obtained are within acceptance criteria. If the recoveries fail for a given reported compound, the technical director confirms the unacceptable result.

20.1.2.1. If the LCS results are verified as acceptable, no corrective action is required.

20.1.2.2. If the LCS result is verified as out-of-control, and the subject compound is to be reported in samples within that analytical batch, the samples reported with that failed compound must be reanalyzed with a valid LCS recovery for the compound.

20.1.2.3. If the LCS result is verified as out-of-control, and the subject compound is NOT to be reported in the samples within that analytical batch, the samples are not subject to reanalysis. No corrective action is required for that batch.

21. WASTE MANAGEMENT

21.1. The proper disposal of analytical samples and laboratory wastes is not only good laboratory practice, but also regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated, and either returned to the client (preferable) or placed into the proper laboratory waste stream.

21.2. Unused or remaining soil or liquid samples and all other solid or liquid wastes resulting from our laboratory operations are considered hazardous for disposal purposes.

21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal.

21.4. Each specific laboratory area shall maintain clearly labeled waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste.

21.5. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly.

21.6. In order to maintain accountability for all samples received by Calscience, when a sample is used in its entirety for analysis, the empty container(s) are returned to Sample Control for placement in analytical storage.

21.7. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Wastes."

22. REFERENCES

22.1. ***►Organochlorine Pesticides by Gas Chromatography, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1B, Method 8081A, USEPA, Revision 1, December 1996.***

22.2. Determinative Chromatographic Separations, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1B, Method 8000C, USEPA, Revision 3, March 2003.

22.3. Quality Control, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter One, USEPA, Revision 1, July 1992.

22.4. Choosing the Correct Procedure, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Two, USEPA, Revision 4, February 2007.

22.5. Organic Analytes, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Four, USEPA, Revision 4, February 2007.

22.6. *Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs)*, SW-846 Method 8081 or 8080, Region 9 Quality Assurance Data Quality Indicator Tables, USEPA, December 1999.

23. TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

23.1. Appendix A: Quantitation Peaks for Single-Component and Multiple-Component Target Compounds.

23.2. Appendix B: Additional Quality Control Criteria for Department of Defense Project.

23.3. Appendix C: Control Limits for Department of Defense Project.

23.4. Appendix D: Modifications to Procedure for Low Level Reporting in Solid Matrices

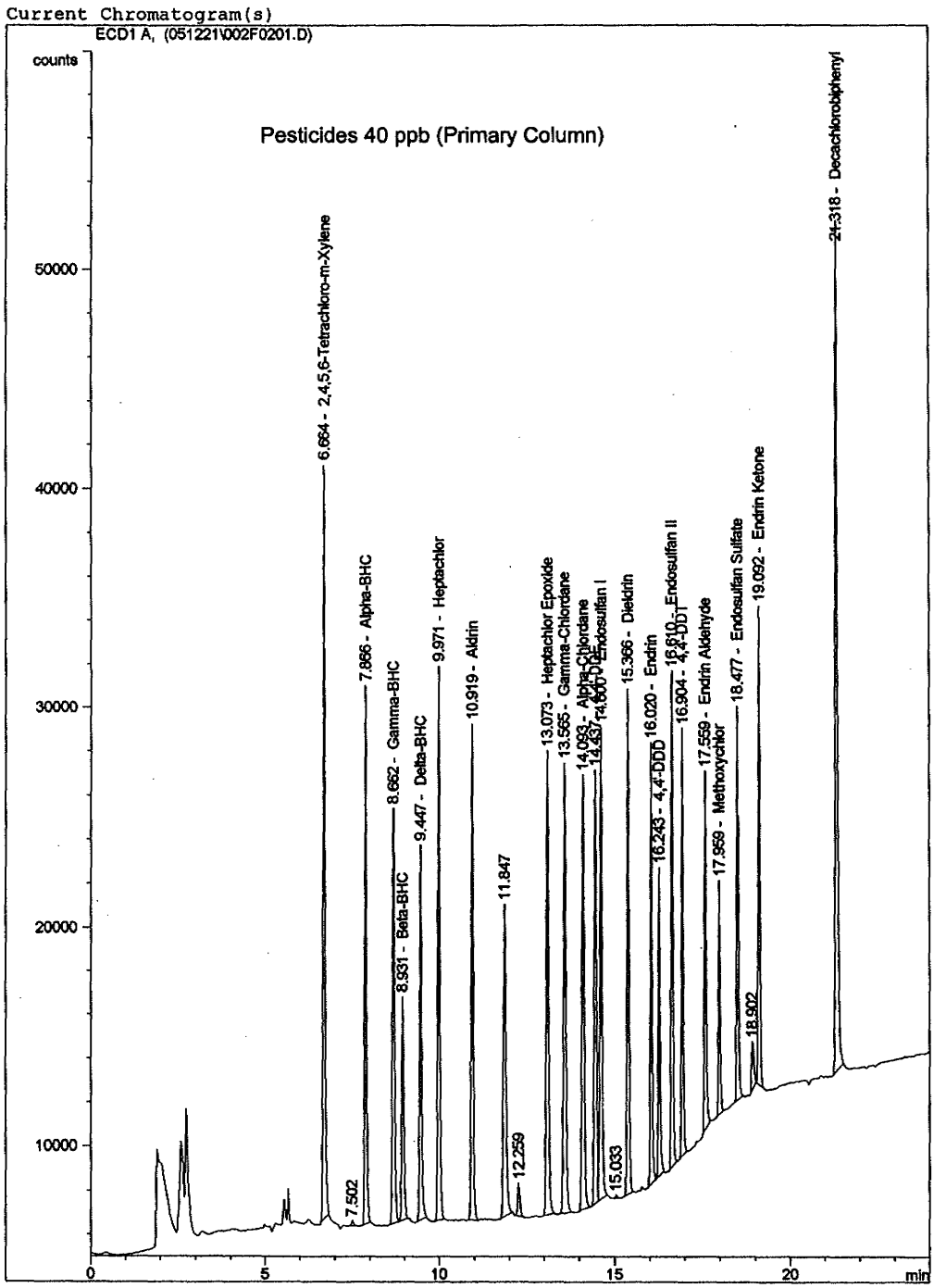
24. MODIFICATIONS

24.1. None.

Appendix A

QUANTITATION PEAKS FOR SINGLE-COMPONENT AND MULTIPLE-COMPONENT TARGET COMPOUNDS

Calscience Environmental Laboratories, Inc.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ECD1 A, (051221\002F0201.D)
counts
Pesticides 40 ppb (Primary Column)
6.664 - 2,4,5,6-Tetrachloro-m-Xylene
7.502
7.866 - Alpha-BHC
8.662 - Gamma-BHC
8.931 - Beta-BHC
9.447 - Delta-BHC
9.971 - Heptachlor
10.919 - Aldrin
11.847
12.259
13.073 - Heptachlor Epoxide
13.565 - Gamma-Chlordane
14.093 - Alpha-Chlordane
14.437 - Endosulfan I
14.600 - 4,4'-DDE
15.033
15.366 - Dieldrin
16.020 - Endrin
16.243 - 4,4'-DDD
16.610 - Endosulfan II
16.904 - 4,4'-DDT
17.559 - Endrin Aldehyde
17.959 - Methoxychlor
18.477 - Endosulfan Sulfate
18.902
19.092 - Endrin Ketone
21.318 - Decachlorobiphenyl
50000
40000
30000
20000
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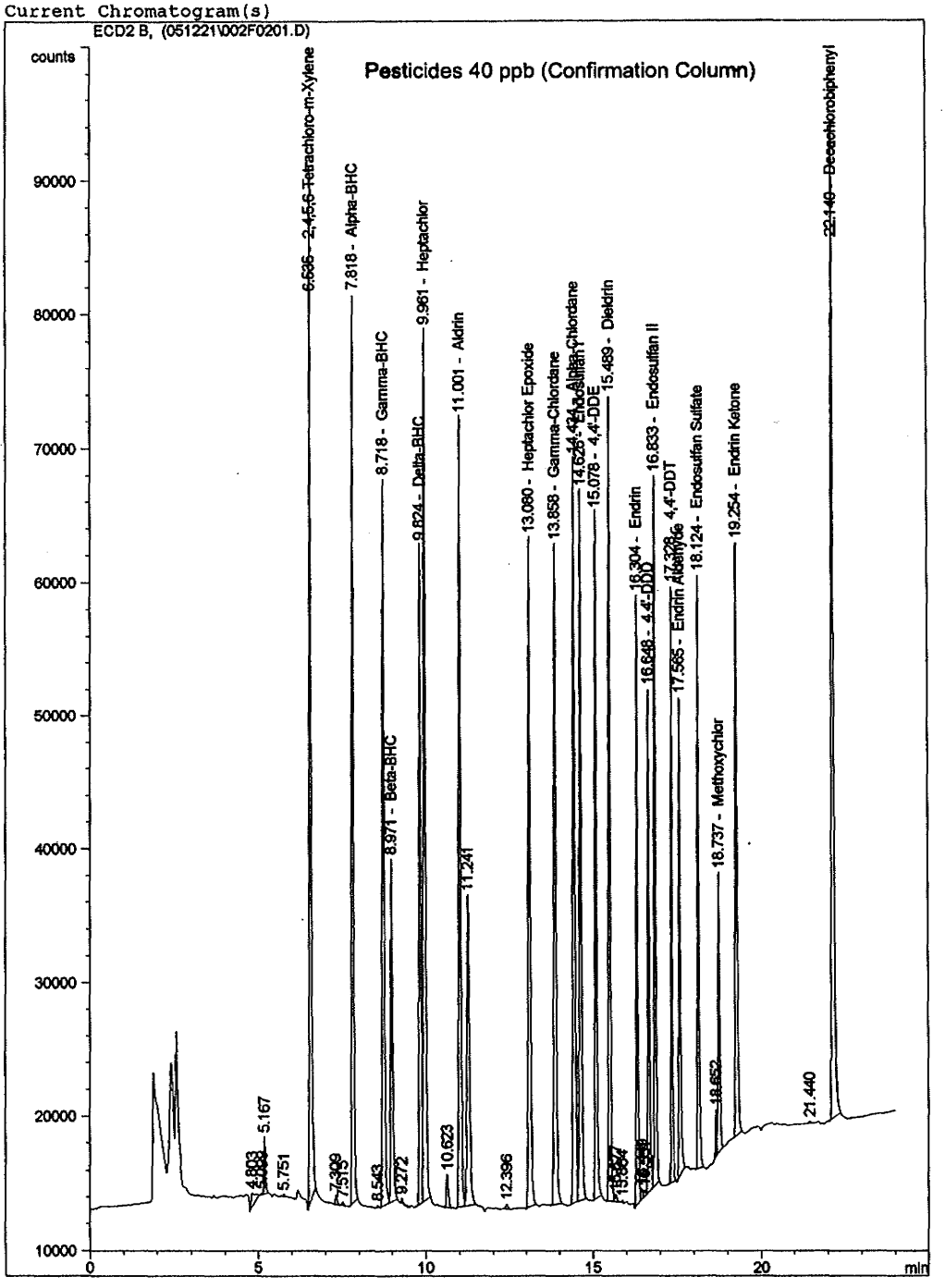
0
5
10
15
20
min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Pesticides 40 ppb (Confirmation Column)
counts
min
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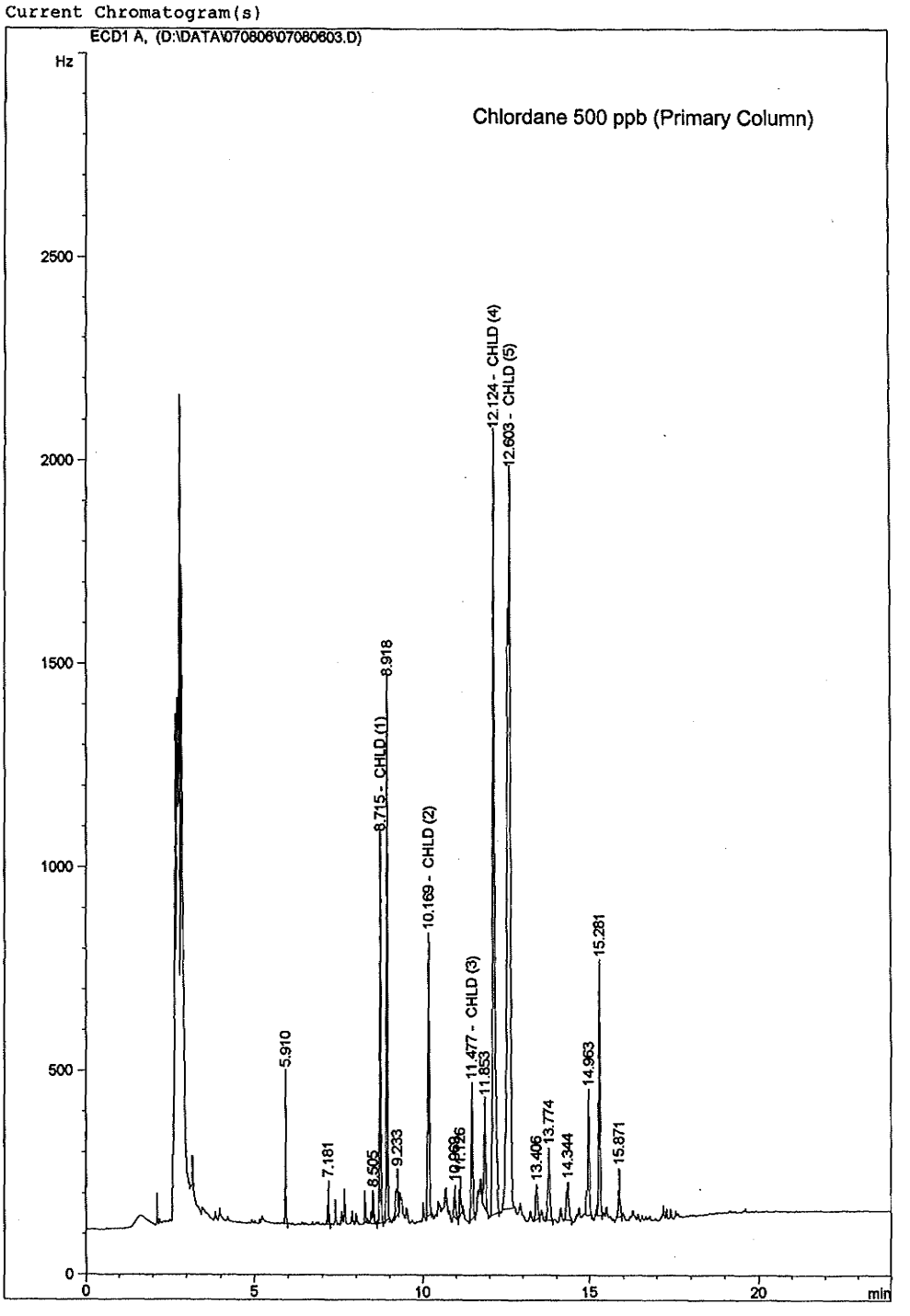
7.818 - Alpha-BHC
8.718 - Gamma-BHC
8.971 - Beta-BHC
9.961 - Heptachlor
9.624 - Delta-BHC
11.001 - Aldrin
11.241
13.080 - Heptachlor Epoxide
13.858 - Gamma-Chlordane
14.434 - Alpha-Chlordane
15.078 - 4,4'-DDE
15.489 - Dieldrin
16.304 - Endrin
16.648 - 4,4'-DDD
16.833 - Endosulfan II
17.565 - Endrin Aldehyde
17.728 - 4,4'-DDT
18.124 - Endosulfan Sulfate
18.737 - Methoxychlor
19.254 - Endrin Ketone
22.140 - Decachlorobiphenyl
21.440
5.167
5.751
12.396
10.623
8.543
8.272
7.515
18.652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2500
2000
1500
1000
500
0
5.910
7.181
8.505
8.715 - CHLD (1)
8.918
9.233
10.169 - CHLD (2)
11.477 - CHLD (3)
11.853
12.124 - CHLD (4)
12.603 - CHLD (5)
13.406
13.774
14.344
14.963
15.281
15.871
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5
10
15
20
min

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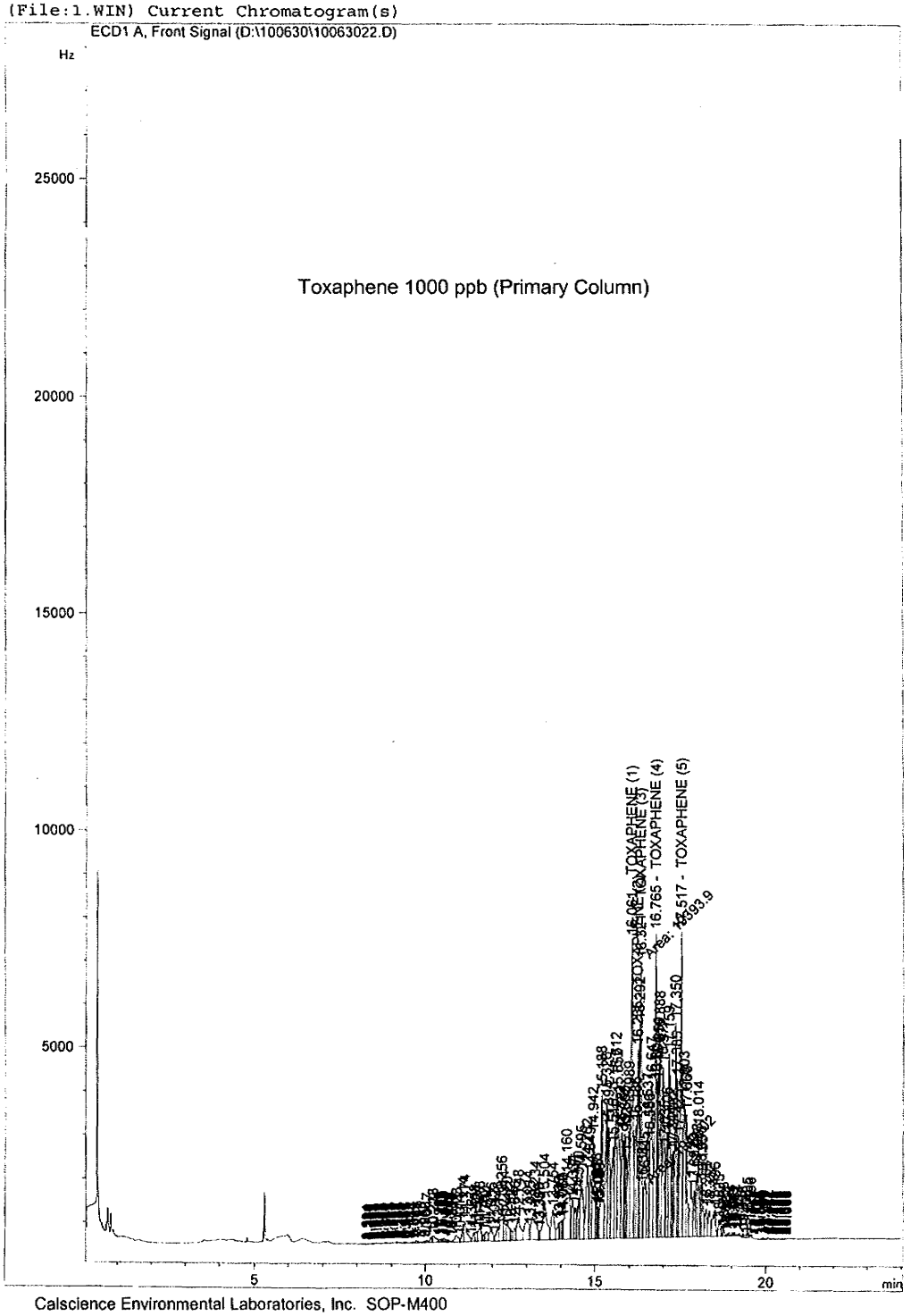

Appendix B

ADDITIONAL QUALITY CONTROL CRITERIA FOR DEPARTMENT OF DEFENSE PROJECT

Calscience Environmental Laboratories, Inc.

1. METHOD IDENTIFICATION

1.1. EPA Method 8081B, Organochlorine Pesticides by Gas Chromatography – Additional Quality Control Criteria for Department of Defense (DoD) Project.

2. DETECTION LIMITS

2.1. The quantitation limit must be set within the calibration range.

3. SCOPE AND APPLICATION

3.1. The quality control criteria and procedure described herein either supersede or are in addition to the standard quality control criteria and procedure.

4. STANDARDS

4.1. The spike standard solutions shall contain all anticipated target analytes.

4.2. The use of a standard from a second lot as the second source standard is acceptable when only one manufacturer of the calibration standard exists. "Manufacturer" refers to the producer of the standard, not the vendor.

5. QUALITY CONTROL

5.1. Method Detection Limit (MDL)

5.1.1. MDL study shall be performed at the initial test method setup, following a change in the test method that affects how the test is performed, or when a change in instrumentation that affects the sensitivity of the analysis.

5.1.2. MDL verification must be performed immediately following an MDL study and quarterly thereafter.

5.1.2.1. MDL verification sample shall be prepared by spiking an appropriate matrix at approximately 2–3 times the detection limit for single-analyte test, or 1–4 times the detection limit for multiple-analyte test.

5.1.2.2. MDL verification is deemed valid if the apparent signal to noise ratio of each analyte is at least 3 and the results must meet all method requirements for analyte identification (e.g., second column confirmation, pattern recognition, etc.).

5.1.2.3. If these criteria are not met, perform either one of the following tasks.

5.1.2.3.1. Repeat the MDL study and verification at a higher concentration. Set the MDL at the higher concentration.

5.1.2.3.2. Perform and pass 2 consecutive MDL verifications at a higher concentration. Set the MDL at the higher concentration.

5.1.3. No samples shall be analyzed without a valid MDL.

5.2. Continuing Calibration Verification (CCV)

5.2.1. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily prior to sample analysis, after every batch of 10 samples or portion thereof within a 12-hour shift, and at the end of sequence.

5.2.2. The concentration of the CCV standard shall be between the low point and the midpoint of the calibration range.

5.3. Retention Time Window

5.3.1. Establishment of retention time window position is accomplished by using the midpoint calibration standard once per initial calibration, and by using a low-to-midpoint CCV standard at the beginning of an analytical sequence.

5.3.1.1. When initial calibration is performed, daily retention time window for each analyte/surrogate is the retention time of the analyte/surrogate in the midpoint calibration standard ± 3S.

5.3.1.2. When initial calibration is <u>not</u> performed, daily retention time window for each analyte/surrogate is the retention time of the analyte/surrogate in the low-to-midpoint CCV standard ± 3S.

5.4. Event Based Quality Control (LCS/LCSDs and MBs)

5.4.1. Laboratory Control Samples (LCS/LCSDs)

5.4.1.1. The lower and upper acceptance limits for %REC of each LCS/LCSD compound in aqueous and solid matrices are listed in Appendix C.

5.4.1.2. Project-specific control limits shall be applied. If project-specific control limits are unavailable, DoD generated control limits shall be applied. If DoD generated control limits are unavailable, laboratory's in-house control limits shall be applied.

5.4.1.2.1. Laboratory's in-house control limits may not be greater than ± 3S of the average recovery.

5.4.1.3. All project-specific analytes of concern must be within control limits. No marginal exceedance is allowed for any project-specific analyte of concern. If a project-specific analyte of concern exceeds its control limit, determine the cause of the problem and effect corrective action.

5.4.2. Method Blanks (MBs)

5.4.2.1. The MB is considered to be contaminated if one of the following conditions is met.

5.4.2.1.1. The concentration of any target analyte in the MB exceeds 1/2 the RL, and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).

5.4.2.1.2. The concentration of any common laboratory contaminant in the MB exceeds RL, and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).

5.4.2.1.3. The MB result otherwise affects the sample results as per the test method requirements or the project specific data quality objectives (DQOs).

5.4.2.2. If the MB is contaminated, reprocess the samples associated with the failed MB in a subsequent preparation batch, except when the sample results are below the MDL.

5.4.2.2.1. If insufficient sample volume remains for reprocessing, the results shall be reported with the appropriate data qualifier (B-flag) for the specific analyte(s) in all samples associated with the failed MB.

5.5. Matrix Based Quality Control (Surrogates and MS/MSDs)

5.5.1. Surrogates

5.5.1.1. The lower and upper acceptance limits for %REC of each surrogate compound in aqueous and solid matrices are listed in Appendix C.

5.5.1.2. Project-specific control limits shall be applied. If project-specific control limits are unavailable, DoD generated control limits shall be applied. If DoD generated control limits are unavailable, laboratory's in-house control limits shall be applied.

5.5.2. Matrix Spikes (MS/MSDs)

5.5.2.1. The lower and upper acceptance limits for %REC of each MS/MSD compound in aqueous and solid matrices are listed in Appendix C. The RPD is ≤ 30%.

5.5.2.2. Project-specific control limits shall be applied. If project-specific control limits are unavailable, DoD generated control limits shall be applied. If DoD generated control limits are unavailable, laboratory's in-house control limits shall be applied.

5.5.2.2.1. Laboratory's in-house control limits may not be greater than ± 3S of the average recovery.

6. PROCEDURE

6.1. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily prior to sample analysis, after every batch of 10 samples or portion thereof within a 12-hour shift, and at the end of sequence.

6.2. Standard and sample vials are loaded in the following or other logical order:

1) Degradation Test
2) Continuing Calibration Verification (CCV)
3) Laboratory Control Sample (LCS)
4) Laboratory Control Sample Duplicate (LCSD)
5) Method Blank (MB)
6) Samples (up to 10 per batch, excluding QC check samples and MBs)
7) Matrix Spike (MS)
8) Matrix Spike Duplicate (MSD)
9) Ending CCV

6.2.1. Items 2 and 9: A CCV is used to verify the acceptance of the initial five-point calibration on a continuing basis. An acceptable CCV is required daily prior to sample analysis, after every batch of 10 samples or portion thereof within a 12-hour shift, and at the end of sequence.

6.2.2. Item 6: Up to 10 sample (excluding QC check sample and method blank) extracts per batch. Complex extracts should be sufficiently diluted or subjected to cleanup procedures to ensure that instrument is not contaminated. Dilution or cleanup of extracts will result in increased reporting limits.

6.2.3. Item 7: The MS is the actual sample matrix spiked with known concentrations of specific target analytes. The sample which is spiked for the MS is processed concurrently with the associated samples. In the processing of the MS, reagents and procedures identical to those for actual samples are used.

6.2.3.1. The sample selected for spiking must be one of the samples collected for the specific DoD project.

6.2.4. Item 8: The MSD is handled identically to the MS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the MS in combination with the MSD can be used to assess the precision of the analytical measurements. The measurement is expressed as relative percent difference (RPD).

6.3. Data Interpretation

6.3.1. The flagging criteria and data reporting procedure for second column confirmation are as follows:

6.3.1.1. If RPD is > 40%, apply the appropriate data qualifier (J-flag) and document in the case narrative.

6.3.1.2. Follow project-specific reporting requirements when reporting data.

6.3.1.2.1. If project-specific reporting requirements are unavailable, apply method-specific reporting requirements.

6.3.1.2.2. If method-specific reporting requirements are unavailable, report the results from the primary column or detector, unless there is a scientifically valid and documented reason for not doing so.

6.3.1.3. Identify unconfirmed results with the appropriate data qualifiers and document in the case narrative.

7. REFERENCES

7.1. Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.1, April 2009.

Appendix C

CONTROL LIMITS FOR DEPARTMENT OF DEFENSE PROJECT

Calscience Environmental Laboratories, Inc.

Appendix C

DoD Control Limits of LCS/LCSD/MS/MSD Compounds in Aqueous Matrix

Analyte	Control Limit		ME Limit	
	Lower	Upper	Lower	Upper
4,4'-DDD	25	150	10	170
4,4'-DDE	35	140	15	160
4,4'-DDT	45	140	30	155
Aldrin	25	140	10	155
α-BHC	60	130	50	140
α-Chlordane	65	125	55	135
β-BHC	65	125	55	135
δ-BHC	45	135	30	150
Dieldrin	60	130	50	140
Endosulfan I	50	110	40	120
Endosulfan II	30	130	10	150
Endosulfan sulfate	55	135	40	150
Endrin	55	135	45	145
Endrin aldehyde	55	135	40	150
Endrin ketone	75	125	70	135
γ-BHC	25	135	10	155
γ-Chlordane	60	125	50	135
Heptachlor	40	130	30	145
Heptachlor epoxide	60	130	50	140
Methoxychlor	55	150	40	165

Note: ME limits are applicable to LCS/LCSD compounds only.

Appendix C

DoD Control Limits of LCS/LCSD/MS/MSD Compounds in Solid Matrix

Analyte	Control Limit		ME Limit	
	Lower	Upper	Lower	Upper
4,4'-DDD	30	135	10	155
4,4'-DDE	70	125	60	135
4,4'-DDT	45	140	30	155
Aldrin	45	140	30	155
α-BHC	60	125	50	135
α-Chlordane	65	120	55	130
β-BHC	60	125	50	135
δ-BHC	55	130	45	145
Dieldrin	65	125	55	135
Endosulfan I	15	135	10	155
Endosulfan II	35	140	20	160
Endosulfan sulfate	60	135	50	145
Endrin	60	135	50	145
Endrin aldehyde	35	145	20	165
Endrin ketone	65	135	55	145
γ-BHC	60	125	50	135
γ-Chlordane	65	125	55	135
Heptachlor	50	140	35	155
Heptachlor epoxide	65	130	55	140
Methoxychlor	55	145	45	155

Note: ME limits are applicable to LCS/LCSD compounds only.

Appendix C

DoD Control Limits of Surrogate Compounds in Aqueous Matrix

Analyte	Control Limit	
	Lower	Upper
Decachlorobiphenyl	30	135
TMX	25	140

DoD Control Limits of Surrogate Compounds in Solid Matrix

Analyte	Control Limit	
	Lower	Upper
Decachlorobiphenyl	55	130
TMX	70	125

Appendix D

PROCEDURE MODIFICATIONS FOR LOW LEVEL REPORTING IN SOLID MATRICES

Calscience Environmental Laboratories, Inc.

1. ►PROCEDURE MODIFICATIONS FOR LOW LEVEL REPORTING IN SOLID MATRICES

1.1. *Sample Preparation*

1.1.1. ***Utilizing SOP M204 "EPA Method 3545, Pressurized Fluid Extraction (PFE)" the sample weight and final extract volume are modified as follows:***

1.1.1.1. ***Sample weight: Increased from 20 g to 50g***

1.1.1.2. ***Final extract volume: Decreased from 10 ml to 5 ml.***

1.2. *Calibration*

1.2.1. ***Calibration standard solutions containing various concentrations of target analytes and surrogates in hexane.***

1.2.1.1. ***Dilute the appropriate volumes of the working standards, the 100-ppm chlordane stock standard, and the 100-ppm toxaphene stock standard to 4.0 mL with hexane for initial calibration.***

1.2.1.2. ***The calibration standards are prepared as follows:***

Calibration Level (ppb)			Initial				Final	
			Concentration (ppm)		Volume (µL)		Volume (mL)	
A1	S	A2	A1 + S	A2	A1 + S	A2	A1 + S	A2
2	4	N/A	4.0 + 8.0	N/A	2	N/A	4.0	N/A
10	20	N/A	4.0 + 8.0	N/A	10	N/A	4.0	N/A
40	80	N/A	4.0 + 8.0	N/A	40	N/A	4.0	N/A
60	120	N/A	4.0 + 8.0	N/A	60	N/A	4.0	N/A
80	160	N/A	4.0 + 8.0	N/A	80	N/A	4.0	N/A

Note: A1 = Single-Component Analyte; S = Surrogate;
A2 = o,p'-(DDD, DDE, DDT) or Nonachlor

Calibration Level (ppb)		Initial				Final	
		Concentration (ppm)		Volume (µL)		Volume (mL)	
C	T	C	T	C	T	C	T
5	25	100	100	0.2	1.0	4.0	4.0
100	200	100	100	4.0	8.0	4.0	4.0
500	1000	100	100	20	40	4.0	4.0
750	1500	100	100	30	60	4.0	4.0
2000	4000	100	100	80	160	4.0	4.0

Note: C = Chlordane; T = Toxaphene

Title : **EPA METHOD 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION SPECTROMETRY (ICP-AES)**

Document No. : SOP-M601
Revision No. : 5.2
Supersedes : 5.1

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Revision 5.2 changes are noted in bold italicized typeface and preceded by a "►" marker.

APPROVED FOR RELEASE BY: _____ 05/12/11
MANAGEMENT **DATE**

_____ 05-12-11
QA DEPARTMENT **DATE**

ANNUAL SOP REVIEW

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

YEAR	GROUP LEADER	DATE	QA MANAGER	DATE

1. METHOD IDENTIFICATION

1.1. EPA Method 6010B, Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES).

2. APPLICABLE MATRICES

2.1. This method is applicable to groundwater samples, aqueous samples, TCLP/SPLP/WET extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes.

3. DETECTION LIMITS

3.1. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions.

3.2. The instrument detection limit data may be used to estimate instrument and method performance for other sample matrices.

4. SCOPE AND APPLICATION

4.1. EPA Method 6010B is used to determine trace elements, including metals, in solution. Except groundwater samples, all aqueous and solid matrices require acid digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion. Samples which are not digested must either use an internal standard or be matrix matched with the standards. If either option is used, instrument software should be programmed to correct for intensity differences of the internal standard between samples and standards.

4.2. The method is applicable to the elements listed in Appendix A. Appendix A also lists the recommended analytical wavelengths and estimated instrument detection limits for the elements in clean aqueous matrices with insignificant background interferences. Elements and matrices other than those listed in Appendix A may be analyzed by this method if performance at the concentrations of interest (see Section 12.) is demonstrated.

4.3. This method is restricted to use by or under the supervision of analysts experienced in the use of inductively coupled plasma emission spectrometer, skilled in the interpretation of atomic emission spectra, and knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

5. METHOD SUMMARY

5.1. EPA Method 6010B describes multielemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch.

Element-specific emission spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices.

5.2. Background correction is required for trace element determination. Background emission must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used should be as free as possible from spectral interference and should reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences identified in Section 7. should also be recognized and appropriate corrections made. Alternatively, multivariate calibration methods may be utilized. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

5.3. Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation methods. Acceptable preparatory methods include, but are not limited to, the following:

Sample Digestion/Preparation	Method No.	SOP No.
Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA/ICP	EPA 3005A	M220
Acid Digestion of Aqueous Samples/Extracts for Total Metals for Analysis by FLAA/ICP	EPA 3010A	M223
Acid Digestion of Sediments, Sludges, and Soils	EPA 3050B	M222
Toxicity Characteristic Leaching Procedure (TCLP)	EPA 1311	M226
Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312	M227
Waste Extraction Test Procedure (STLC/TTLC)	CCR T22.11.5.A-II	M228

5.4. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

6. DEFINITIONS

6.1. Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

6.2. Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

6.3. 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 NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the

start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

6.4. Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

6.5. Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

6.6. Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

6.7. Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

6.8. Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

6.9. Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

6.10. Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.

6.11. Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

6.12. Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

6.13. Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

6.14. Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

6.15. Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

6.16. Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

6.17. Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

6.18. Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.

6.19. 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.

6.20. 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.

6.21. Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence.

6.22. Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

6.23. Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

6.24. Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

6.25. Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

6.26. ►Terms Specific to ICP-AES Analysis

6.26.1. Linear Dynamic Range: The concentration range over which the analytical curve remains linear.

6.26.2. Method of Standard Addition (MSA): The standard-addition technique involves the use of the unknown and the unknown plus one or more known amounts of standard.

6.26.3. Optimum Concentration Range: A range, defined by limits expressed in concentration, below which scale expansion must be used and above which curve correction should be considered. This range will vary with the sensitivity of the instrument and the operating conditions employed.

6.26.4. Post Digestion (Matrix) Spike: A sample which has been extracted in the same manner as the other samples, but to which a known amount of target analytes has been added to the sample extractant. Post digestion spikes are used to evaluate the accuracy of the method without the losses incurred through the extraction process.

6.26.5. Sensitivity: The slope of the analytical curve, i.e., the functional relationship between emission intensity and concentration.

7. INTERFERENCES

7.1. Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

7.1.1. Compensation for background emission and stray light can usually be conducted by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be

included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

7.1.2. To determine the appropriate location for off-line background correction, the analyst must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100-mg/L single-element solutions are sufficient. However, for analytes such as iron that may be found in the sample at high concentration, a more appropriate test would be to use a concentration near the upper limit of the analytical range.

7.1.3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction **require** that the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive or positively biased determinations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Analysts may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off-line or on-line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Appendix B. For multivariate calibration methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

7.1.4. When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e., false positive analyte concentrations) arising from 100 mg/L of the interference element. For example, if As is to be determined at 193.696 nm in a sample containing approximately 10 mg/L of Al. According to Appendix B, 100 mg/L of Al will yield a false positive signal for an As level equivalent to approximately 0.01085 mg/L. Therefore, the presence of 10 mg/L of Al will result in a false positive signal for As equivalent to approximately 0.001085 mg/L. The analyst is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Appendix B. The

interference effects must be evaluated for each individual instrument, since the intensities will vary.

7.1.5. Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Analysts should continuously note that some samples may contain uncommon elements that could contribute spectral interferences.

7.1.6. The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences (see Appendix B) as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst shall utilize a computer routine for automatic correction on all analyses.

7.1.7. Analysts using sequential instruments must verify the absence of spectral interference by scanning over a range of 0.5 nm centered on the wavelength of interest for several samples. The range for lead, for example, would be from 220.6 to 220.1 nm. This procedure must be repeated whenever a new matrix is to be analyzed and when a new calibration curve using different instrumental conditions is to be prepared. Samples that show an elevated background emission across the range may be background corrected by applying a correction factor equal to the emission adjacent to the line or at two points on either side of the line and interpolating between them. An alternate wavelength that does not exhibit a background shift or spectral overlap may also be used.

7.1.8. If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and dividing by 10. If after the subtraction of the calibration blank, the apparent analyte concentration falls outside of this range, in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

7.1.9. When interelement corrections are applied, their accuracy should be verified daily, by analyzing spectral interference check solutions. The correction factors or multivariate correction matrices tested on a daily basis must be within the 20% criteria for 5 consecutive days. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change occurs, such as one in the torch, nebulizer, injector, or plasma conditions. Standard solutions should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

7.1.10. When interelement corrections are <u>not</u> used, verification of absence of interferences is required.

7.1.10.1. One method is to use a computer software routine for comparing the determinative data to established limits for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration (i.e., greater than the analyte instrument detection limit), or a false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

7.1.10.2. Another method is to analyze an interference check solution which contains similar concentrations of the major components of the samples (> 10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is ≥ 20% of the analyte concentration, the analyte must be determined (1) using analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

7.2. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using a peristaltic pump, by using an internal standard, or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization by using a tip washer, by using a high solids nebulizer, or by diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance. This may be accomplished with the use of mass flow controllers. The dilution test (see Section 12.12.) will help determine if a physical interference is present.

7.3. Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating

conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

7.3.1. The MSA should be used if an interference is suspected or a new matrix is encountered. When the MSA is used, standards are added at one or more levels to portions of a prepared sample. This technique compensates for enhancement or depression of an analyte signal by a matrix. It will not correct for additive interferences, such as contamination, interelement interferences, or baseline shifts. This technique is valid in the linear range when the interference effect is constant over the range, the added analyte responds the same as the endogenous analyte, and the signal is corrected for additive interferences.

7.3.2. An alternative to using the MSA is to use the internal standard technique. Add one or more elements that are both not found in the samples and verified to not cause an interelement spectral interference to the samples, standards, and blanks. Yttrium or scandium is often used. The concentration should be sufficient for optimum precision, but not so high as to alter the salt concentration of the matrix. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation. This technique is very useful in overcoming matrix interferences, especially in high solids matrices.

7.4. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to equal to or less than the method detection limit should be noted. Until the required rinse time is established, it is suggested that the rinse period be at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon the project specific data quality objectives (DQOs).

7.5. Analysts are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of

the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

7.6. The dashes in Appendix B indicate that no measurable interferences were observed even at higher interferant concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

7.7. The calibration blanks (Section 10.2.3.1.) may restrict the sensitivity of the detection limit or degrade the precision and accuracy of the analysis. Clean chemistry methods and procedures are necessary in reducing the magnitude and variability of the calibration blank.

8. SAFETY

8.1. All sample preparation activities should be performed in an operational fume hood appropriate for use with acids.

8.2. All operational fume hoods are to remain energized continuously in order to minimize acidic atmospheric buildup.

8.3. Many metal salts are extremely toxic if inhaled or swallowed. Extreme care must be taken to ensure that samples and standards are handled properly and that all exhaust gases are properly vented. Wash hands thoroughly after handling.

8.4. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current version of Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats are required to be worn in all designated laboratory areas. Protective gloves shall be worn when handling chemicals.

8.5. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

9. EQUIPMENT AND SUPPLIES

9.1. Inductively Coupled Argon Plasma Emission Spectrometer: Perkin-Elmer Optima 4300/5300 DV Optical Emission Spectrometer or equivalent configured with the following components:

9.1.1. Computer-controlled emission spectrometer with background correction.

9.1.2. Radio-frequency (RF) generator compliant with FCC regulations.

9.1.3. Mass-flow controller for argon nebulizer gas supply.

9.1.4. Peristaltic pump.

9.1.5. Autosampler, Perkin-Elmer AS 90 or equivalent.

9.1.6. Argon gas supply, high purity.

9.1.7. Nitrogen gas supply, high purity, for purge only.

9.1.8. PC based data system or equivalent.

9.2. Volumetric flasks, 100-mL, 500-mL, and 1000-mL, Class A.

9.3. Pipetters, 10–100-µL, 100–1000-µL, 0.5–5.0-mL, and 1–10-mL, adjustable, with disposable tip.

10. REAGENTS AND STANDARDS

10.1. Reagents

10.1.1. Hydrochloric acid, HCl, concentrated, trace metals grade for equivalent.

10.1.2. Hydrochloric acid, HCl, 1:1 (v/v).

10.1.2.1. Prepare the solution by slowly adding 500 mL of concentrated HCl to 400 mL of reagent water and diluting to 1 L with additional reagent water.

10.1.3. Nitric acid, HNO_3, concentrated, trace metals grade for equivalent.

10.1.4. Nitric acid, HNO_3, 1:1 (v/v).

10.1.4.1. Prepare the solution by slowly adding 500 mL of concentrated HNO_3 to 400 mL of reagent water and diluting to 1 L with additional reagent water.

10.1.5. Rinse blank, HCl-HNO_3-H_2O, 1:1:8 (v/v/v).

10.1.5.1. Prepare the rinse blank by slowly adding 1 part of concentrated HCl and 1 part of concentrated HNO_3 to 8 parts of reagent water.

10.1.5.2. The rinse blank consists of 10% (v/v) HCl and 10% (v/v) HNO_3 in reagent water.

10.1.5.3. The rinse blank is used to flush the system between standards and samples to minimize memory interferences (see Section 7.4.).

10.1.6. Reagent water, interferant free.

10.1.7. Chips, Teflon.

10.1.8. Beads, glass.

10.1.9. All reagents must be inspected and documented prior to use.

10.2. Standards

10.2.1. Stock Standard Solutions

10.2.1.1. Pre-certified stock standard solutions (ultra-high purity grade or equivalent), each in sealed polyethylene bottles, containing various concentrations of target analytes are used to prepare calibration and check standards.

10.2.1.2. Prior to preparing the calibration or check standards, analyze each stock standard solution separately to determine possible spectral interference or the presence of impurities.

10.2.2. Initial Calibration Standard Solutions

10.2.2.1. Prepare the initial calibration standard solutions by combining appropriate volumes of the stock standards in volumetric flasks (refer to Appendix C for preparation).

10.2.2.2. Add the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates.

10.2.2.2.1. If the addition of silver to the recommended acid combination initially results in a precipitate, then add 15 mL of reagent water and warm the flask until the solution clears. Cool and dilute to 100 mL with reagent water. For this acid combination, the silver concentration should be limited to 2 mg/L. Silver is stable under these conditions in a water matrix for 30 days, if protected from the light. Higher concentrations of silver require additional HCl.

10.2.2.3. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together.

10.2.2.4. Transfer the mixed standard solutions to FEP fluorocarbon or previously unused polyethylene or polypropylene bottles for storage.

10.2.3. Blanks

10.2.3.1. Calibration Blank (CB)

10.2.3.1.1. Prepare the CBs by acidifying reagent water to the same concentrations of the acids found in the standards and samples.

10.2.3.1.2. The CB consists of 5% (v/v) HCl and 6% (v/v) HNO_3 in reagent water.

10.2.3.1.3. The CB is used to establish the zero point of the calibration curve.

10.2.3.1.4. The CB is also used either as initial calibration blank (ICB) or as continuing calibration blank (CCB) to monitor contamination.

10.2.3.2. Method Blank (MB)

10.2.3.2.1. Prepare the MBs using the appropriate sample preparation methods (see Section 5.3.).

10.2.3.2.2. The MB is used to identify possible contamination resulting from either the acids or the equipment used during sample processing including filtration.

10.2.3.3. Both CB and MB are required for the analyses of samples prepared by any method other than EPA Method 3040A.

10.2.4. Initial Calibration Verification (ICV) Solutions

10.2.4.1. Prepare the ICV solutions in the same acid matrix by combining compatible elements from a source differing from that used for the initial one-point calibration, and at a concentration within the established linear dynamic range (refer to Appendix C for preparation and Section 12.3. for evaluation).

10.2.5. Continuing Calibration Verification (CCV) Solutions

10.2.5.1. Prepare the CCV solutions by diluting initial calibration standards with equal volume of reagent water (refer to Section 12.7. for evaluation).

10.2.5.2. The CCV solutions contain mid-point concentration of each target analyte.

10.2.6. Internal Standard Solution

10.2.6.1. Prepare the internal standard solution in the same acid matrix (refer to Appendix C for preparation).

10.2.6.2. The internal standard contains 5 ppm each of Ho, Tb, and Y. It is used to reduce or overcome interferences (see Section 7.2. and Section 7.3.).

10.2.7. Potential Interference Check Solution

10.2.7.1. Prepare the potential interference check solution in the same acid matrix.

10.2.7.2. The potential interference check solution contains 200 ppm each of Al, Ca, Cr, Cu, Fe, Mg, Mn, Tl, and V. It is used to establish the potential interference table (see Section 12.1.).

10.2.8. Daily Spectral Interference Check Solutions (ICS-AB and ICS-A)

10.2.8.1. Prepare the daily spectral interference check solutions in the same acid matrix (refer to Appendix C for preparation and Section 12.6. for evaluation).

10.2.8.2. The ICS-AB and ICS-A solutions contain known concentrations of interfering elements. They are used to verify the interelement correction factors.

10.2.9. Spike Standard Solutions

10.2.9.1. Prepare the spike standard solutions in the same acid matrix by combining compatible elements from a source differing from

that used for the initial one-point calibration, and at a concentration within the established linear dynamic range (refer to Appendix C for preparation).

10.2.9.2. The spike standard solutions are used to prepare QC check samples such as matrix spikes (MS/MSDs), post digestion spikes (PDSs), and laboratory control samples (LCS/LCSDs).

10.2.9.3. Add 250 µL of the spike standard to each 5-mL aliquot of MS/MSD leachate (TCLP/SPLP/WET extract) prior to dilution and acidification.

10.2.9.4. Add 250 µL of the spike standard to each 50-mL aliquot of aqueous MS/MSD and LCS/LCSD sample prior to digestion.

10.2.9.5. Add 500 µL of the spike standard to each 2-g aliquot of solid MS/MSD and LCS/LCSD sample prior to digestion.

10.2.9.6. Add 50 µL of the spike standard to each 10-mL aliquot of PDS sample after digestion.

10.2.10. Linear Dynamic Range Solutions

10.2.10.1. Prepare a minimum of three different concentrations of the linear dynamic range solutions in the same acid matrix by diluting the spike standard solutions or the stock standard solutions. The analyst determines the applicable concentrations.

10.2.10.2. The linear dynamic range solutions contain various concentrations of compatible elements. They are used to establish linear dynamic range (see Section 12.5.).

10.2.11. All working standards must be replaced after six months or sooner if comparison with check standards indicates a problem.

10.2.11.1. The stability of low level standards (i.e., < 1 ppm), must be demonstrated prior to use.

10.2.11.2. Prepare fresh standards as needed.

10.2.12. All stock standards must be inspected and documented prior to use.

11. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

11.1. Aqueous samples should be collected in 250-mL pre-cleaned high density polyethylene containers with Teflon-lined closures. Soil samples should be collected in 4-oz pre-cleaned clear glass wide-mouth jars with Teflon-lined closures.

11.1.1. Aqueous samples shall be preserved with 1:1 HNO_3 solution to pH < 2.

11.2. Samples should be maintained in a chilled state (≤ 4°C) post sample collection until received at the laboratory. Samples should not be frozen (e.g., do not use dry ice as the refrigerant).

11.3. Upon receipt, the samples are stored in a 4°C cooler.

11.4. Refer to Appendix D for additional information on sample holding time, digestion volumes and suggested collection volumes.

11.4.1. The sample amount required depends upon the sample preparation procedures (i.e. leaching and digestion) necessary for the analysis.

12. QUALITY CONTROL

12.1. Potential Interference Table

12.1.1. Following the initial instrument setup, the potential interference table (see Appendix B) must be established prior to initial calibration.

12.1.1.1. The potential interference table is established by analyzing the potential interference check solution (see Section 10.2.7.).

12.1.2. The potential interference table should be updated annually, when the daily spectral interference check is deemed unacceptable, or when an instrumentation change occurs.

12.2. Initial Calibration (IC)

12.2.1. The initial one-point calibration must be established daily prior to the processing of samples.

12.2.1.1. The calibration curve is established with one calibration blank and one high-level calibration standard.

12.2.2. ►The IC is deemed valid if the %RSD for each analyte is ≤ 5%.

12.2.3. If these criteria are not met, then the calibration is unacceptable for sample analysis to begin. Effect corrective action and recalibrate.

12.3. Initial Calibration Verification (ICV)

12.3.1. ►The initial calibration is deemed valid if the %RSD for each analyte is ≤ 5%, and the %D for each analyte is ≤ 10%.

12.3.2. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to begin. An unacceptable ICV result indicates either a disagreement between like solutions from separate sources or a change in instrument conditions. Normally, this is caused when at least one of the solutions is no longer intact (representative of the stated concentration). Investigate, effect corrective action, which may include re-preparation of standard solutions, and recalibration, if necessary.

12.4. Initial Calibration Blank (ICB)

12.4.1. The instrument operating condition is deemed satisfactory for sample analysis to begin if no analytes are detected at a concentration > RL (or the limit specified in the project specific DQO).

12.4.2. If these criteria are not met, no sample analysis shall begin. Determine the source of contamination. Reprepare and reanalyze the ICB.

12.5. Linear Dynamic Range

12.5.1. Following the initial instrument setup, the upper limit of the linear dynamic range for each analyte must be established for each wavelength utilized prior to initial calibration.

12.5.1.1. The upper range limit is established for each wavelength by determining the signal responses from a minimum of three, preferably five, different concentration standards across the range.

12.5.1.2. The ranges which may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made should be documented and kept on file.

12.5.2. Following the establishment of a valid initial calibration, the upper range limit must be checked every six months, and a new upper range limit should be determined whenever there is a significant change in instrument response.

12.5.2.1. The analyst should be aware that if an analyte that is present above its upper range limit is used to apply an interelement correction, the correction may not be valid and those analytes where the interelement correction has been applied may be inaccurately reported.

12.5.3. The upper range limit is deemed valid if the %D for each analyte in a high-level check standard analyzed and quantitated against the calibration curve is ≤ 10%.

12.5.4. Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self-absorption effects. Hence, non-linear second order curve may be used if the instrument allows it.

12.5.4.1. The effective range must be checked, and the correlation coefficient of the second order curve fit should be ≥ 0.995.

12.5.4.2. Non-linear response curves should be revalidated and recalculated every six months. These curves are much more sensitive to changes in operating conditions than the linear lines and should be checked whenever there have been moderate equipment changes.

12.6. Daily Spectral Interference Check (ICS-AB and ICS-A)

12.6.1. Following the establishment of a valid initial calibration, the daily spectral interference check solutions must be analyzed daily prior to sample analysis and at the end of sequence.

12.6.1.1. The daily spectral interference check solutions are utilized to verify either the accuracy of the interelement correction factors if interelement corrections are applied, or the absence of interferences if interelement corrections are not applied.

12.6.2. The daily spectral interference check is deemed acceptable if the %D for each analyte is ≤ 20%.

12.6.3. If these criteria are not met, no sample analysis shall begin. Determine the source of problem, effect corrective action, and reanalyze the ICS-AB and/or ICS-A.

12.6.4. All interelement spectral correction factors or multivariate correction matrices should be updated every six months, when the daily spectral interference check is deemed unacceptable, or when an instrumentation change occurs.

12.7. Continuing Calibration Verification (CCV)

12.7.1. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily prior to sample analysis, after every batch of 10 samples or portion thereof, and at the end of sequence.

12.7.2. The initial calibration is deemed valid if the %RSD for each analyte is ≤ 5%, and the %D for each analyte is ≤ 10%.

12.7.3. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to resume. Effect corrective action and reanalyze the CCV within 2 hours after the failed CCV. If the CCV criteria remain unacceptable, recalibrate.

12.8. Continuing Calibration Blank (CCB)

12.8.1. The instrument operating condition is deemed satisfactory for sample analysis to resume if no analytes are detected at a concentration > RL (or the limit specified in the project specific DQO).

12.8.2. If these criteria are not met, no sample analysis shall resume. Determine the source of contamination. Reprepare and reanalyze the CCB.

12.9. Event Based Quality Control (LCS/LCSDs and MBs)

12.9.1. Event based quality control consists of QC samples prepared and processed with each preparatory event. This consists of a laboratory control sample and laboratory control sample duplicate (LCS/LCSD) and a method blank (MB).

12.9.2. The acceptance criteria for LCS/LCSD elements are as follows:

12.9.2.1. The lower and upper acceptance limits for %REC of each LCS/LCSD element are 80% and 120%, respectively. The RPD is ≤ 20%.

12.9.2.2. All LCS/LCSD elements must be within acceptance limits. However, if a large number of analytes are in the LCS, it

becomes statistically likely that a few will be outside of control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary.

12.9.2.3. ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.

12.9.2.4. The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than 11 analytes.

12.9.2.5. The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number of Analytes Allowed in ME of the LCS Control Limit
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

12.9.2.6. Marginal exceedances must be random. If the same analyte exceeds the LCS control limit 2 out of 3 consecutive LCS, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.

12.9.2.7. If the problem was not related to the digestion process, then the LCS/LCSD and all associated sample digestates must be re-analyzed. If the failure was associated with the digestion process, then all associated samples must be re-processed and re-analyzed.

12.9.3. Ideally, the concentrations of target analytes in an MB should be less than the respective reporting limits (RLs). If the concentration of any target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs are as follows:

12.9.3.1. If a target analyte is found in the MB, but not in the associated samples, report the sample and MB data without qualification.

12.9.3.2. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the

effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified, or rejected and the samples re-processed and/or re-analyzed.

12.10. Matrix Based Quality Control (MS/MSDs and PDSs)

12.10.1. Matrix based quality control consists of QC samples prepared and processed using actual environmental samples. This consists of a matrix spike and matrix spike duplicate (MS/MSD) and a post digestion spike (PDS).

12.10.2. The acceptance criteria for MS/MSD elements are as follows:

12.10.2.1. The lower and upper acceptance limits for %REC of each MS/MSD element are 75% and 125%, respectively. The RPD is ≤ 20%.

12.10.2.1.1. If historical data is available, then the lower and upper acceptance limits for %REC and %RPD of each MS/MSD element are based upon the historical average recovery ± 3S that is updated at least annually.

12.10.2.2. When the %REC and RPD of the MS/MSD elements are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.

12.10.2.3. If the %REC and/or RPD of the MS/MSD elements are not within the established acceptance limits, the analytical system performance shall be suspect.

12.10.3. Unacceptable %REC values are typically caused by matrix effects or poor instrument performance/technique. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.

12.11. If the %REC or RPD of the MS/MSD and LCS/LCSD are unacceptable, all associated sample data must be invalidated and all associated samples re-processed and re-analyzed.

12.12. Dilution Test

12.12.1. If the analyte concentration is sufficiently high, an analysis of a 1:5 dilution should agree within ± 10% of the original determination.

12.12.2. If this criterion is not met, a physical or chemical interference effect shall be suspect. Perform recovery test.

12.13. Recovery Test (Post Digestion Spike Addition)

12.13.1. A PDS sample is prepared by adding the spike standard to a portion of a digested sample, or its dilution.

12.13.2. The acceptance criteria for PDS elements are as follows:

12.13.2.1. The lower and upper acceptance limits for %REC of each PDS element are 75% and 125%, respectively.

12.13.2.2. If the %REC of the PDS elements is not within the established acceptance limits, a matrix effect shall be suspect. Perform MSA on all samples within the batch.

12.14. Additional information regarding internal quality control checks is provided in SOP-T020.

13. CALIBRATION AND STANDARDIZATION

13.1. Initial Demonstration of Performance

13.1.1. Document the selection criteria for background correction points; analytical dynamic ranges, the applicable equations, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences.

13.1.2. Generate the data using the same instrument, operating conditions, and calibration routine to be used for sample analysis.

13.1.3. Keep the data on file and available for review.

13.2. Pipetter

13.2.1. Calibrate the pipetter according to the procedure outlined in the pipetter calibration logbook.

13.3. Spectrometer Initial Calibration

13.3.1. Establish an acceptable one-point calibration curve. The acceptance criteria for the initial calibration are listed in Section 12.2.

13.3.2. After obtaining an acceptable one-point calibration curve and prior to processing sample or QC digestates, an ICV standard and ICB must be analyzed to verify the initial calibration. The acceptance criteria for the ICV and ICB are listed in Section 12.3. and Section 12.4.

13.3.3. The initial one-point calibration and ICV should include all anticipated target analytes for the duration of the use of the initial calibration.

14. PROCEDURE

14.1. Instrument Setup

14.1.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable (usually requiring at least 30

minutes of operation) prior to calibration. Follow the instructions provided by the instrument manufacturer for operating conditions.

14.1.1.1. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality.

14.1.1.2. Deviations from instructions provided by the instrument manufacturer must be documented and approved by the Group Leader.

14.1.1.3. Use the following ICP-AES operating conditions as guidance.

Operating Condition	Aqueous Solution	Axial Plasma
Forward Power	1100–1450 watts	1100–1450 watts
Viewing Height	14–18 mm	
Argon Coolant Flow	15–19 L/min	15–19 L/min
Argon Nebulizer Flow	0.5–1.5 L/min	0.5–1.5 L/min
Sample Pumping Rate	1.0–3.0 mL/min	1.0–3.0 mL/min
Preflush Time	1 min	1 min
Measurement Time (Sequential Instrument)	~2 sec per wavelength peak	~2 sec per wavelength peak
Measurement Time (Simultaneous Instrument)	10 sec per sample	10 sec per sample

14.1.1.4. Repeatable interference correction factors can be achieved by adjusting the argon aerosol flow to reproduce the Cu/Mn intensity ratio at 324.754 nm and 257.610 nm respectively.

14.1.2. Refer to Appendix A for specific wavelengths. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference.

14.1.3. Optimize the plasma operating conditions prior to the use of the instrument. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure. This routine is not required on a daily basis, but only is required when first setting up a new instrument, or following a change in operating conditions. Apply the following procedure, or the instrument manufacturer's recommendations, to optimize the plasma operating conditions.

14.1.3.1. Ignite the radial plasma and select an appropriate incident RF power. Allow the instrument to become thermally stable (about 30 to 60 minutes of operation). While aspirating a 1000 µg/L solution of yttrium, follow the instrument manufacturer's instructions and adjust the aerosol carrier gas flow rate through the nebulizer so a definitive blue emission region of the plasma extends approximately from 5 to 20 mm above the top of the load coil. Record the nebulizer gas flow rate or pressure setting

for future reference. The yttrium solution can also be used for coarse optical alignment of the torch by observing the overlay of the blue light over the entrance slit to the optical system.

14.1.3.2. After establishing the nebulizer gas flow rate, determine the solution uptake rate of the nebulizer in mL/min by aspirating a known volume of a calibration blank for a period of at least three minutes. Divide the volume aspirated by the time in minutes and record the uptake rate. Set the peristaltic pump to deliver that rate in a steady even flow.

14.1.3.3. Profile the instrument to align it optically as it will be used during analysis. The following procedure can be used for both horizontal and vertical optimization in the radial mode, but is written for vertical.

14.1.3.3.1. Aspirate a solution containing 10 µg/L of several selected elements. As, Se, Tl, and Pb are the least sensitive of the elements and most in need of optimization. However, other elements may be used, based on the judgment of the analyst. V, Cr, Cu, Li and Mn also have been used with success.

14.1.3.3.2. Collect intensity data at the wavelength peak for each analyte at 1-mm intervals from 14 to 18 mm above the load coil. This region of the plasma is referred to as the analytical zone.

14.1.3.3.3. Repeat the process using the calibration blank. Determine the net signal to blank intensity ratio for each analyte for each viewing height setting. Choose the height for viewing the plasma that provides the best net intensity ratios for the elements analyzed or the highest intensity ratio for the least sensitive element.

14.1.3.3.4. Follow the instrument manufacturer's instructions for optimization in the axial mode.

14.1.4. The instrument operating condition finally selected as being optimum should provide the lowest reliable instrument detection limits (IDLs).

14.1.5. If either the instrument operating conditions (such as incident power or nebulizer gas flow rate) are changed, or a new torch injector tube with a different orifice internal diameter is installed, then the plasma and viewing height should be re-optimized.

14.1.6. After completing the initial optimization of operating conditions, and before analyzing samples, an interelement spectral interference correction routine to be used for sample analysis must be established and initially verified.

14.1.6.1. A general description of spectral interferences and the analytical requirements for background correction are discussed in Section 7.

14.1.6.2. The criterion for determining the presence of an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls beyond ± one reporting limit from zero. The upper control limit is the analyte instrument detection limit.

14.1.6.3. Once established, the entire routine must be verified every six months. Only a portion of the correction routine must be verified more frequently or on a daily basis. Initial and periodic verifications of the routine should be kept on file.

14.1.7. Before daily calibration, and after the instrument warm-up period, the nebulizer gas flow rate must be reset to the determined optimized flow. If a mass flow controller is being used, it should be set to the recorded optimized flow rate. In order to maintain valid spectral interelement correction routines, the nebulizer gas flow rate should be the same (< 2% change) from day to day.

14.1.8. For operation with organic solvents, the use of the auxiliary argon inlet is recommended, as is the use of solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power, to obtain stable operation and precise measurements.

14.1.9. Program the system to average at least duplicate readings on samples including the calibration standards, the calibration verification standards, the calibration blanks, the QC check samples and method blanks. Report the average.

14.1.9.1. If the %RSD for an analyte in a standard is > 5%, re-analyze the standard. If the %RSD criterion remains unacceptable, investigate, effect corrective action, which may include re-preparation of the standard solution, and recalibrate, if necessary.

14.1.9.2. If the %RSD for an analyte in a sample is > 20%, and the analyte concentration exceeds its RL, re-analyze the sample. If the %RSD criterion remains unacceptable, investigate and effect corrective action.

14.2. Establish sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

14.2.1. Establish method detection limits (MDLs) for all wavelengths utilized for each type of matrix analyzed and for each preparation method used and for each instrument. Additional information regarding determination of detection limits is provided in SOP-T006.

14.2.2. Establish the upper limit of the linear dynamic range for each wavelength utilized (see Section 12.5.).

14.2.3. Verify that the instrument configuration and operating conditions satisfy the analytical requirements, and maintain quality control data confirming instrument performance and analytical results.

14.3. Establish a calibration curve to cover the appropriate concentration range (see Section 13.3.).

14.4. Following the establishment of a valid initial calibration, an ICS-AB and ICS-A must be analyzed daily prior to sample analysis, and an ICS-AB must be analyzed at the end of sequence. The acceptance criteria are listed in Section 12.6.

14.4.1. If a failed ICS-AB/ICS-A is the first of the day, corrective action must be effected prior to analyzing any samples.

14.4.2. If not, effect corrective action and reanalyze all samples since the last acceptable ICS-AB.

14.5. Following the establishment of a valid initial calibration, a CCV standard and CCB must be analyzed daily prior to sample analysis, after every batch of 10 samples or portion thereof, and at the end of sequence. If the QC criteria are met, the initial calibration is assumed to be valid and sample analysis may resume. The acceptance criteria are listed in Section 12.7. and Section 12.8.

14.5.1. If a failed CCV/CCB is the first of the day, corrective action must be effected prior to analyzing any samples.

14.5.2. If not, effect corrective action and reanalyze all samples since the last acceptable CCV/CCB.

14.6. Following digestion by one of the methods specified in Section 5.3., the digestates for the QC and actual environmental samples are received in autosampler vials. The autosampler vials are then loaded onto the ICP-AES sample tray.

14.6.1. Preliminary treatment of most matrices is necessary due to the complexity and variability of sample matrices.

14.6.2. All associated QC samples (MB/LCS/LCSD/MS/MSD) for the batch must undergo the same filtration and acidification procedures.

14.6.3. Groundwater samples which have been prefiltered and acidified will not need acid digestion.

14.6.4. Samples which are not digested must either use an internal standard or be matrix-matched with the standards.

14.7. Sample vials are loaded in the following or other logical order:

1) Calibration Blank (CB)
2) Initial Calibration Standard(s)
3) Initial Calibration Verification (ICV)
4) Initial Calibration Blank (ICB)
5) Interference Check Solution AB (ICS-AB)

6) Interference Check Solution A (ICS-A)
7) Continuing Calibration Verification (CCV)
8) Continuing Calibration Blank (CCB)
9) Method Blank (MB)
10) Laboratory Control Samples (LCS)
11) Laboratory Control Sample Duplicates (LCSD)
12) Samples (up to 10, including QC check samples and MBs)
13) Matrix Spike (MS)
14) Matrix Spike Duplicate (MSD)
15) Post Digestion Spike (PDS)
16) Ending ICS-AB
17) Ending CCV
18) Ending CCB

14.7.1. Item 1: The CB is a vial of acidified reagent water used to establish the zero point of the initial calibration curve.

14.7.1.1. Additional calibration blanks may also be added elsewhere in the sequence to rinse the analytical system.

14.7.1.2. The rinse time is set to one minute. Rinse time may be reduced through a suitable demonstration.

14.7.2. Item 2: The initial calibration standard(s) are high-level calibration standard(s) used to establish the initial calibration curve.

14.7.3. Item 3: The ICV is a second source standard used to verify the acceptance of the initial one-point calibration. An acceptable ICV is required daily after initial calibration.

14.7.4. Item 4: The ICB is a vial of acidified reagent water used to monitor contamination. An acceptable ICB is required daily after initial calibration verification.

14.7.5. Items 5, 6, and 16: The ICS-AB and ICS-A are used to verify the accuracy of the interelement correction factors. An acceptable ICS-AB is required daily prior to sample analysis and at the end of sequence. An acceptable ICS-A is required daily prior to sample analysis.

14.7.6. Items 7 and 17: A CCV is used to verify the acceptance of the initial one-point calibration on a continuing basis. An acceptable CCV is required daily prior to sample analysis, after every batch of 10 samples or portion thereof, and at the end of sequence.

14.7.7. Items 8 and 18: A CCB is a vial of acidified reagent water used to monitor contamination. An acceptable CCB is required daily prior to sample analysis, after every batch of 10 samples or portion thereof, and at the end of sequence.

14.7.8. Item 9: The MB is a known matrix similar to the samples being analyzed which is processed concurrently with the associated samples. In the processing of the MB, reagents and procedures identical to those for actual samples are used.

14.7.8.1. For aqueous samples, the MB consists of clean reagent water. For solid samples, the MB consists of clean Teflon chips (or glass beads).

14.7.8.2. One MB is required every day leachings/digestions are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

14.7.8.3. When samples that are leached/digested together are analyzed on separate instruments or on separate analytical shifts, the MB associated with those samples must be analyzed on at least one of the instruments. A solvent blank consisting of acidified reagent water must be analyzed on all other instruments where the associated samples are analyzed to demonstrate that the instruments are not contributing contaminants to the samples.

14.7.9. Item 10: The LCS is a known matrix which has been spiked with known concentrations of specific target analytes. The purpose of the LCS is to demonstrate that the entire analytical process and systems are in control. The LCS is processed concurrently with the associated samples. In the processing of the LCS, reagents and procedures identical to those for actual samples are used.

14.7.9.1. For aqueous samples, the LCS consists of the specified elements spiked into clean reagent water. For solid samples, the LCS consists of the specified elements spiked into clean Teflon chips (or glass beads).

14.7.9.2. One LCS is required every day leachings/digestions are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

14.7.10. Item 11: The LCSD is handled identically to the LCS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the LCS in combination with the LCSD can be used to assess the precision of the analytical process. The measurement is expressed as relative percent difference (RPD). The formula for calculating RPD is listed in Section 15.5.

14.7.11. Item 12: Up to 10 sample (including QC check sample and method blank) digestates per batch. Digestates should be sufficiently diluted if concentrations exceed the calibration range. Dilution of digestates will result in increased reporting limits.

14.7.12. Item 13: The MS is an actual sample matrix spiked with known concentrations of specific target analytes. The sample which is spiked for the MS is processed concurrently with the associated samples. In the processing of the MS, reagents and procedures identical to those for actual samples are used.

14.7.12.1. The purpose of the MS is to assess the effect of a sample matrix on the recovery of target analytes (i.e., assess the

accuracy of the analytical measurements of the matrix). The measurement is expressed as percent recovery (%REC). The formula for calculating %REC is listed in Section 15.4.

14.7.12.2. One MS is required for every batch of 20 samples per matrix or portion thereof digested/leached concurrently.

14.7.13. Item 14: The MSD is handled identically to the MS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the MS in combination with the MSD can be used to assess the precision of the analytical measurements. The measurement is expressed as relative percent difference (RPD). The formula for calculating RPD is listed in Section 15.5.

14.7.14. Item 15: The PDS is the same sample matrix from which the MS/MSD samples were prepared, and is spiked with known concentrations of specific target analytes post digestion. The sample which will be spiked for the PDS is processed concurrently with the associated samples. In the processing of the PDS, reagents and procedures identical to those for actual samples are used.

14.7.14.1. The purpose of the PDS is to access matrix effects. The measurement is expressed as percent recovery (%REC). The formula for calculating %REC is listed in Section 15.4.

14.7.14.2. One PDS is required for the occurrence of new or unusual matrix included within the batch, or for the failure of dilution test or matrix spike recovery.

14.7.15. Rinse blanks may be added elsewhere in the sequence to rinse the analytical system.

14.8. Ensure that a sufficient amount of rinse blank is present in the rinse blank bottle, and that a sufficient unused volume exists in the waste container at the beginning of the sequence.

14.9. Edit the sequence in the data system. After all correct sample information is entered, save the sequence. After saving the sequence, record pertinent information in the run logbook.

14.10. Initiate the sequence.

14.11. Dilution and recovery tests (see Section 12.12. and Section 12.13.) are recommended prior to reporting concentration data for the elements.

14.11.1. It is recommended that these tests be performed with each batch of samples prepared/analyzed to ensure that neither positive nor negative interferences are affecting the measurement of any element or distorting the accuracy of the reported values.

14.12. If spectral overlap is suspected, then the use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

14.13. Method of Standard Additions (MSA)

14.13.1. The standard addition technique involves adding known amounts of a standard solution to one or more aliquots of a digested sample. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. However, it will not correct for additive interferences which cause a baseline shift.

14.13.2. The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_s of a standard analyte solution of concentration C_s. To the second aliquot (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration C_x is calculated using the formula listed in Section 15.10. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

14.13.3. Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50% of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100% and 150% of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot is shown in Appendix E. A linear regression program may be used to obtain the intercept concentration.

14.13.4. For the results of the MSA technique to be valid, the following limitations must be taken into consideration:

14.13.4.1. The apparent concentrations from the calibration curve must be linear (correlation coefficient of 0.995 or greater) over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve.

14.13.4.2. The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

14.13.4.3. The determination must be free of spectral interference and corrected for nonspecific background interference.

14.14. Data Interpretation

14.14.1. Quantitation of a target analyte is based on a reproducible response of the spectrometer within the calibration range and a direct proportionality of the magnitude of response between intensities in the sample digestate and the calibration standard(s).

14.14.1.1. Proper quantitation requires the appropriate selection of a wavelength from which the intensity of an element can be determined.

14.14.1.2. Determine the concentration based on the initial calibration curve.

14.14.1.2.1. The data system is programmed to perform the calculation of concentration.

14.14.1.3. If the instrument response exceeds the calibration range, dilute the digestate and reanalyze.

15. CALCULATIONS

15.1. The percent relative standard deviation is calculated as follows:

$$\%RSD = \frac{SD}{I_{ave}} \times 100$$

where: %RSD = percent relative standard deviation.
SD = standard deviation of the intensity readings for the target analyte.
I_{ave} = mean of the intensity readings for the target analyte.

15.2. The percent difference of each analyte is calculated as follows:

$$\%D = \frac{|C_{expected} - C_{measured}|}{C_{expected}} \times 100$$

where: %D = percent difference.
$C_{expected}$ = concentration of target analyte expected.
$C_{measured}$ = concentration of target analyte measured.

Note: Concentrations must be in equivalent units.

15.3. The recovery of each LCS element is calculated as follows:

$$\%REC_{LCS} = \frac{C_{recovered}}{C_{added}} \times 100$$

where: $\%REC_{LCS}$ = percent recovery of target analyte in LCS (or LCSD).
$C_{recovered}$ = concentration of target analyte recovered.
C_{added} = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

15.4. The recovery of each MS element is calculated as follows:

$$\%REC_{MS} = \frac{C_{recovered} - C_{sample}}{C_{added}} \times 100$$

where: $\%REC_{MS}$ = percent recovery of target analyte in MS (or MSD/PDS).
$C_{recovered}$ = concentration of target analyte recovered.
C_{sample} = concentration of target analyte in environmental sample used.
C_{added} = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

15.5. The relative percent difference is calculated as follows:

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where: RPD = relative percent difference between two measurements (C_1 and C_2).
C_1 = concentration of target analyte in measurement 1.
C_2 = concentration of target analyte in measurement 2.

Note: Concentrations must be in equivalent units.

15.6. The slope and intercept of a linear calibration curve are calculated as follows:

$$M = \frac{I_s - I_b}{C_s - C_b} \qquad B = \frac{C_s I_b - C_b I_s}{C_s - C_b}$$

where: M = slope of the calibration curve.
B = intercept of the calibration curve.
I_s = intensity of calibration standard at a specific wavelength.
I_b = intensity of calibration blank at a specific wavelength.
C_s = concentration of calibration standard.
C_b = concentration of calibration blank.

Note: Concentrations must be in equivalent units.

15.7. The target analyte concentration for a sample digestate is calculated as follows:

$$C_x = \frac{I_x - B}{M}$$

where: C_x = concentration of target analyte in digestate in mg/L.
I_x = intensity of target analyte at a specific wavelength.
B = intercept of the calibration curve.
M = slope of the calibration curve.

15.8. The target analyte concentration for an aqueous sample is calculated as follows:

$$C_A = \frac{C_x \times V_x \times D}{V_A}$$

where: C_A = concentration of target analyte in aqueous sample in mg/L.
C_x = concentration of target analyte in digestate in mg/L.
V_x = volume of digestate in mL.
V_A = volume of aqueous sample digested in mL.
D = dilution factor, if the sample or digestate was diluted prior to analysis. If no dilution was made, D = 1.

15.9. The target analyte concentration for a solid sample is calculated as follows:

$$C_S = \frac{C_x \times V_x \times D}{W_S}$$

where: C_S = concentration of target analyte in solid sample in mg/kg.
C_x = concentration of target analyte in digestate in mg/L.
V_x = volume of digestate in mL.
W_S = mass of solid sample digested in g.
D = dilution factor, if the sample or digestate was diluted prior to analysis. If no dilution was made, D = 1.

15.10. The target analyte concentration from single-addition method is calculated as follows:

$$C_x = \frac{S_B \times V_s \times C_s}{(S_A - S_B) \times V_x}$$

where: C_x = concentration of target analyte in sample.
S_A = analytical signal (corrected for the blank) of solution A.
S_B = analytical signal (corrected for the blank) of solution B.
V_s = volume of target analyte in standard solution.
C_s = concentration of target analyte in standard solution.
V_x = volume of target analyte in sample.

Note: Concentrations and volumes must be in equivalent units.

15.11. All concentrations shall be reported in mg/L (ppm) for aqueous samples, and mg/kg (ppm) for soil and solid waste samples.

15.12. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

16. METHOD PERFORMANCE

16.1. A demonstration of analytical capability shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

16.2. Calibration protocols specified in Section 13., "Calibration and Standardization," shall be followed.

16.3. Proficiency test sample results shall be used to evaluate the ability to produce accurate results.

17. POLLUTION PREVENTION

17.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Calscience's Health, Safety, and Respiratory Protection Manual. In general, protective eyewear (e.g. safety glasses or goggles), and protective apparel (e.g. lab coats) and gloves are required to be worn when handling chemicals.

17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:

17.3.1. A NIOSH approved air purifying respirator with cartridges appropriate for the chemical handled.

17.3.2. Extended length protective gloves.

17.3.3. Face shield.

17.3.4. Full-length laboratory apron.

17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.

17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles. Alternatively, the Coordinator may utilize a self-contained breathing apparatus or

other supplied air system if appropriately trained and approved by the Health and Safety Manager.

17.6. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

18.1. The acceptance criteria for LCS/LCSD elements are predetermined. The lower and upper acceptance limits for %REC of each LCS/LCSD element are 80% and 120%, respectively. The RPD is ≤ 20%. All LCS/LCSD elements must be within acceptance limits (see Section 12.9.2. for additional information).

18.1.1. If the LCS and/or LCSD %REC is outside of the acceptance limits high, the RPD is within acceptance limits, and all target analytes in the associated samples are not detected, the sample data can be reported without qualification.

18.1.2. The LCSD is only reported when the MS/MSD is unacceptable due to matrix interference effects, or when the LCS/LCSD is used in place of MS/MSD due to insufficient sample quantity.

18.2. Ideally, the concentrations of target analytes in an MB should be less than the respective reporting limits (RLs). If the concentration of any target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs are as follows:

18.2.1. If a target analyte is found in the MB but not in the associated samples, report the sample and MB data without qualification.

18.2.2. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-processed and/or re-analyzed.

18.3. The acceptance criteria for MS/MSD elements are predetermined. The lower and upper acceptance limits for %REC of each MS/MSD element are 75% and 125%, respectively. The RPD is ≤ 20%.

18.3.1. Refer to Section 12.10.2.1.1. for acceptance criteria if historical data is available.

18.3.2. When the %REC and RPD of the MS/MSD elements are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.

18.3.3. If the %REC and/or RPD of the MS/MSD elements are not within the established acceptance limits, the analytical system performance shall be suspect.

18.4. The acceptance criteria for PDS elements are predetermined. The lower and upper acceptance limits for %REC of each PDS element are 75% and 125%, respectively.

18.4.1. When the %REC of the PDS elements are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The PDS data shall be reported with the corresponding sample data.

18.4.2. If the %REC of the PDS elements are not within the established acceptance limits, the MSA results shall be reported with the corresponding sample data.

18.5. Matrix effects or poor instrument performance/technique typically cause unacceptable %REC values. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.

18.6. Additional information regarding internal quality control checks is provided in SOP-T020.

18.7. All concentrations shall be reported in mg/L (ppm) for aqueous samples, and mg/kg (ppm) for soil and solid waste samples.

18.8. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

19. CORRECTIVE ACTIONS

19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.

19.2. The Operations Manager, Project Manager, Quality Control Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.

19.3. Corrective actions are generally of two types, immediate and long-term actions.

19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.

19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits.

The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented in the Corrective Action Record. Examples of this type of action include:

19.3.2.1. Remedial training of staff in technical skills, technique or implementation of operating procedures.

19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.

19.3.2.3. Revision of standard operating procedures.

19.3.2.4. Replacing personnel, as necessary.

19.4. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:

19.4.1. Define the problem.

19.4.2. Assign responsibility for investigating the problem.

19.4.3. Investigate and determine the cause of the problem.

19.4.4. Assign and accept responsibility for implementing the corrective action.

19.4.5. Determine effectiveness of the corrective action and implement correction.

19.4.6. Verify that the corrective action has eliminated the problem.

19.5. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

20. CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1. Out-of-control data are reviewed and verified by the technical director of the appropriate department. All samples associated with an unacceptable QC set are then subject to reanalysis, depending upon the QC type in question.

20.1.1. MS/MSD/PDS: Acceptability of the MS/MSD/PDS recoveries is subject to the matrix and any anomalies associated with the subject batch. Failure of recoveries of an MS/MSD/PDS data set does not constitute an automatic reanalysis of the batch samples. Rather, it is acceptable to defer to the LCS/LCSD recoveries, to determine acceptance of the sample results.

20.1.2. LCS/LCSD: Because they denote whether the analytical system is operating within control, it is imperative that the LCS recoveries obtained are within acceptability criteria. If the recoveries fail for a given reported element, the technical director confirms the unacceptable result.

20.1.2.1. If the LCS results are verified as acceptable, no corrective action is required.

20.1.2.2. If the LCS result is verified as out-of-control, and the subject element is to be reported in samples within that analytical batch, the samples reported with that failed element must be reanalyzed with a valid LCS recovery for the element.

20.1.2.3. If the LCS result is verified as out-of-control, and the subject element is NOT to be reported in the samples within that analytical batch, the samples are not subject to reanalysis. No corrective action is required for that batch.

21. WASTE MANAGEMENT

21.1. The proper disposal of analytical samples and laboratory wastes is not only good laboratory practice, but also regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated, and either returned to the client (preferable) or placed into the proper laboratory waste stream.

21.2. Unused or remaining soil or liquid samples and all other solid or liquid wastes resulting from our laboratory operations are considered hazardous for disposal purposes.

21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal.

21.4. Each specific laboratory area shall maintain clearly labeled waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste.

21.5. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly.

21.6. In order to maintain accountability for all samples received by Calscience, when a sample is used in its entirety for analysis, the empty container(s) are returned to Sample Control for placement in analytical storage.

21.7. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Wastes."

22. REFERENCES

22.1. *Inductively Coupled Plasma-Atomic Emission Spectrometry*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1A, Method 6010B, USEPA, Revision 2, December 1996.

22.2. *Inductively Coupled Plasma-Atomic Emission Spectrometry*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1A, Method 6010C, USEPA, Revision 3, November 2000.

22.3. *Quality Control*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter One, USEPA, Revision 1, July 1992.

22.4. *Choosing the Correct Procedure*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Two, USEPA, Revision 4, November 2000.

22.5. *Inorganic Analytes*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Three, USEPA, Revision 4, November 2000.

23. TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

23.1. Appendix A: Recommended Wavelengths and Estimated Instrumental Detection Limits.

23.2. Appendix B: Potential Interferences (Example), Analyte Concentration Equivalents Arising from Interference at the 100-mg/L Level.

23.3. Appendix C: Standard Solution Preparation.

23.4. Appendix D: Sample Holding Times, Required Digestion Volumes and Recommended Collection Volumes for Metal Determinations in Aqueous and Solid Samples.

23.5. Appendix E: Standard Addition Plot (Example).

23.6. Appendix F: Additional Quality Control Criteria for Navy Project.

Appendix A

RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Calscience Environmental Laboratories, Inc.

Appendix A

Recommended Wavelengths and Estimated Instrumental Detection Limits (IDLs)

Element	Detection Wavelength [a] (nm)	Estimated IDL [b] (µg/L)
Aluminum (Al)	308.215	30
Antimony (Sb)	217.582	21
Arsenic (As)	193.696	35
Barium (Ba)	233.527	0.87
Beryllium (Be)	313.042	0.18
Boron (B)	249.677 × 2	3.8
Cadmium (Cd)	226.502	2.3
Calcium (Ca)	317.933	6.7
Chromium (Cr)	267.716	4.7
Cobalt (Co)	228.616	4.7
Copper (Cu)	324.752	3.6
Iron (Fe)	273.955	4.1
Lead (Pb)	220.353	28
Lithium (Li)	610.362	2.8
Magnesium (Mg)	279.077	20
Manganese (Mn)	257.610	0.93
Molybdenum (Mo)	202.031	5.3
Nickel (Ni)	231.604 × 2	10
Phosphorus (P)	213.617	51
Potassium (K)	766.490	See note [c]
Selenium (Se)	196.026	50
Silica (SiO2)	251.611	17
Silver (Ag)	328.068	4.7
Sodium (Na)	589.592	19
Strontium (Sr)	407.771	0.28
Thallium (Tl)	190.801	27
Tin (Sn)	189.927	17
Titanium (Ti)	336.121	5.0
Vanadium (V)	292.402	5.0
Zinc (Zn)	213.857 × 2	1.2

[a] The wavelengths listed (where ×2 indicates second order) are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted (e.g., in the case of an interference) if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference (see Section 7.1.). In time, other elements may be added as more information becomes available and as required.

[b] The estimated instrumental detection limits shown are provided as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[c] Highly dependent on operating conditions and plasma position.

Appendix B

POTENTIAL INTERFERENCES (EXAMPLE)

Calscience Environmental Laboratories, Inc.

Appendix B

Potential Interferences (Example)

Analyte Concentration Equivalents Arising from Interference at the 100-mg/L Level [c]

Analyte		Wavelength (nm)	Interferant [ab] Al	Ca	Cr	Cu	Fe	Mg	Mn	Tl	V
Aluminum	Al	308.215	--	0.01926	0.01760	0.00290	--	0.00296	--	0.00470	0.63900
Antimony	Sb	206.836	--	--	1.50400	--	--	0.00116	--	--	--
Antimony	Sb	217.582	--	--	0.00910	--	--	0.00116	--	--	0.14900
Arsenic	As	188.979	--	--	--	0.00290	--	--	0.00440	0.00150	0.00040
Arsenic	As	193.696	--	0.00035	0.04780	--	--	--	--	--	--
Barium	Ba	233.527	0.00009	--	--	--	0.00403	0.00025	--	--	--
Beryllium	Be	313.042	--	--	0.00025	--	--	--	--	--	0.00650
Cadmium	Cd	226.502	--	--	--	--	0.00096	--	--	0.00005	0.00020
Calcium	Ca	317.933	0.00262	--	0.03450	0.00790	0.01350	0.02850	0.01330	0.00760	--
Chromium	Cr	267.716	0.00096	0.00009	--	0.02110	0.00186	0.00090	0.03080	0.00085	--
Cobalt	Co	228.616	0.00023	0.00001	--	0.00050	0.00166	0.00006	0.00045	0.00015	0.00050
Copper	Cu	324.752	0.00325	0.00229	--	--	--	0.00225	0.02680	0.04170	--
Iron	Fe	273.955	0.00617	--	0.01020	0.00660	--	0.00378	0.01140	0.00460	0.21900
Lead	Pb	220.353	--	--	--	0.00710	--	--	0.01530	0.00050	--
Magnesium	Mg	279.077	0.00066	0.00067	--	--	0.00049	--	--	0.00075	--
Manganese	Mn	257.610	--	0.00018	--	--	--	0.00041	--	0.00182	--
Molybdenum	Mo	202.031	0.00142	--	0.00625	0.00045	--	--	--	--	--
Nickel	Ni	231.604	0.00023	0.00002	0.00100	0.00010	0.00034	--	--	0.03940	0.00190
Phosphorus	P	213.617	--	--	--	0.87900	--	--	--	0.00430	0.01840
Potassium	K	766.490	--	--	--	--	--	0.00780	--	--	--
Selenium	Se	196.026	0.00667	--	--	0.00440	--	--	0.05840	--	0.00450
Silver	Ag	328.068	--	--	0.00040	0.00650	--	--	0.00045	--	--
Sodium	Na	589.592	0.09157	0.00801	0.10960	0.00580	--	0.13380	--	0.08005	--
Strontium	Sr	407.771	--	0.00258	0.00065	--	--	--	--	--	--
Thallium	Tl	190.801	0.00082	0.00378	0.04500	0.00445	0.00024	--	--	--	0.05500
Tin	Sn	189.927	0.00032	0.00301	--	0.00045	0.00059	--	0.00085	--	0.00165
Titanium	Ti	336.121	--	--	--	0.00015	0.00003	--	0.00010	0.00010	--
Vanadium	V	292.402	0.00011	--	--	--	--	0.00023	--	0.00025	--
Zinc	Zn	213.857	0.00041	0.00027	0.00285	0.05220	0.01660	0.00033	--	0.00100	--

[a] Dashes indicate that no interference was observed even when interferants were introduced at the following levels:

Al	- 200	mg/L	Mg	- 200	mg/L
Ca	- 200	mg/L	Mn	- 200	mg/L
Cr	- 1000	mg/L	Tl	- 1000	mg/L
Cu	- 1000	mg/L	V	- 1000	mg/L
Fe	- 200	mg/L			

[b] The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.

[c] Interferences will be affected by background choice and other interferences may be present.

Appendix C

STANDARD SOLUTION PREPARATION

Calscience Environmental Laboratories, Inc.

Appendix C

Standard Solution Preparation – Initial Calibration Standard

Initial Calibration Standard					
Element		Initial Conc. (ppm)	Initial Volume (mL)	Final Conc. (ppm)	Final Volume [a] (mL)
Aluminum	Al	200 + 2000	60 + 7.5	27	1000
Antimony	Sb	200 + 1000	15 + 6	9	1000
Arsenic	As	500	15	7.5	1000
Barium	Ba	2000	7.5	15	1000
Beryllium	Be	50 + 50	15 + 7.5	1.125	1000
Boron	B	100 + 1000	15 + 6	7.5	1000
Cadmium	Cd	100	15	1.5	1000
Calcium	Ca	1000	60	60	1000
Chromium	Cr	20	60	1.2	1000
Cobalt	Co	500	7.5	3.75	1000
Copper	Cu	250	7.5	1.875	1000
Iron	Fe	1000	7.5	7.5	1000
Lead	Pb	500	15	7.5	1000
Lithium	Li	100	60	6	1000
Magnesium	Mg	1000	15	15	1000
Manganese	Mn	100	15	1.5	1000
Molybdenum	Mo	200	6	1.2	1000
Nickel	Ni	20	60	1.2	1000
Phosphorus	P	10000	1.2	12	1000
Potassium	K	400 + 10000	60 + 3	54	1000
Selenium	Se	200	15	3	1000
Silicon	Si	2000	6	12	1000
Silver	Ag	50	15	0.75	1000
Sodium	Na	200 + 10000	60 + 6	72	1000
Strontium	Sr	10	60	0.6	1000
Thallium	Tl	200	15	3	1000
Tin	Sn	1000	6	6	1000
Titanium	Ti	200	6	1.2	1000
Vanadium	V	500	7.5	3.75	1000
Zinc	Zn	100 + 10000	15 + 0.35	5	1000
Bismuth [b]	Bi	1000	0.02	2	10
Sulfur [b]	S	1000	0.02	2	10

[a] Solvent for standard preparation is 5% (v/v) HCl + 6% (v/v) HNO_3. HCl and HNO_3 are concentrated trace metals grade acids.

[b] Bi and S standards are prepared separately.

Appendix C

Standard Solution Preparation – Initial Calibration Verification (ICV) Standard

Initial Calibration Verification (ICV) Standard					
Element		Initial Conc. (ppm)	Initial Volume (mL)	Final Conc. (ppm)	Final Volume [a] (mL)
Aluminum	Al	200	4	4	200
Antimony	Sb	200	2	2	200
Arsenic	As	500	2	5	200
Barium	Ba	100	2	1	200
Beryllium	Be	50	2	0.5	200
Boron	B	500	1	2.5	200
Cadmium	Cd	150	2	1.5	200
Calcium	Ca	1000	4	20	200
Chromium	Cr	20	4	0.4	200
Cobalt	Co	100	2	1	200
Copper	Cu	100	2	1	200
Iron	Fe	10000	2	100	200
Lead	Pb	500	2	5	200
Lithium	Li	100	4	2	200
Magnesium	Mg	1000	2	10	200
Manganese	Mn	100	2	1	200
Molybdenum	Mo	100 + 300	2 + 1	2.5	200
Nickel	Ni	20	4	0.4	200
Phosphorus	P	1000	1	5	200
Potassium	K	400	4	8	200
Selenium	Se	200	2	2	200
Silicon	Si	230	1	1.15	200
Silver	Ag	50	2	0.5	200
Sodium	Na	200 + 10000	4 + 1	54	200
Strontium	Sr	10	4	0.2	200
Thallium	Tl	200	2	2	200
Tin	Sn	10000	0.05	2.5	200
Titanium	Ti	1000	1	5	200
Vanadium	V	100	2	1	200
Zinc	Zn	150	2	1.5	200
Bismuth [b]	Bi	1000	0.01	1	10
Sulfur [b]	S	1000	0.01	1	10

[a] Solvent for standard preparation is 5% (v/v) HCl + 6% (v/v) HNO_3. HCl and HNO_3 are concentrated trace metals grade acids.

[b] Bi and S standards are prepared separately.

Appendix C

Standard Solution Preparation – Interference Check Standard AB (ICS-AB)

Interference Check Standard AB (ICS AB)					
Element		Initial Conc. (ppm)	Initial Volume (mL)	Final Conc. (ppm)	Final Volume [a] (mL)
Aluminum	Al	1200	10	24	500
Antimony	Sb	10000	0.05	1	500
Arsenic	As	1000	0.5	1	500
Barium	Ba	300	0.5	0.3	500
Beryllium	Be	100	0.5	0.1	500
Bismuth	Bi				
Boron	B	500	0.5	0.5	500
Cadmium	Cd	300	0.5	0.3	500
Calcium	Ca	6000	10	120	500
Chromium	Cr	300	0.5	0.3	500
Cobalt	Co	300	0.5	0.3	500
Copper	Cu	300	0.5	0.3	500
Iron	Fe	5000	10	100	500
Lead	Pb	1000	0.5	1	500
Lithium	Li				
Magnesium	Mg	3000	10	60	500
Manganese	Mn	200	0.5	0.2	500
Molybdenum	Mo	300	0.5	0.3	500
Nickel	Ni	300	0.5	0.3	500
Phosphorus	P				
Potassium	K	20000	0.5	20	500
Selenium	Se	500	0.5	0.5	500
Silicon	Si	230	0.5	0.23	500
Silver	Ag	300	0.5	0.3	500
Sodium	Na	1000	10	20	500
Strontium	Sr				
Thallium	Tl	1000	0.5	1	500
Tin	Sn				
Titanium	Ti	1000	0.5	1	500
Vanadium	V	300	0.5	0.3	500
Zinc	Zn	300	0.5	0.3	500

[a] Solvent for standard preparation is 5% (v/v) HCl + 6% (v/v) HNO_3. HCl and HNO_3 are concentrated trace metals grade acids.

Appendix C

Standard Solution Preparation – Interference Check Standard A (ICS-A)

Interference Check Standard A (ICS A)					
Element		Initial Conc. (ppm)	Initial Volume (mL)	Final Conc. (ppm)	Final Volume [a] (mL)
Aluminum	Al	1200	10	24	500
Calcium	Ca	6000	10	120	500
Iron	Fe	5000	10	100	500
Magnesium	Mg	3000	10	60	500
Sodium	Na	1000	10	20	500

[a] Solvent for standard preparation is 5% (v/v) HCl + 6% (v/v) HNO_3. HCl and HNO_3 are concentrated trace metals grade acids.

Standard Solution Preparation – Internal Standard

Internal Standard					
Element		Initial Conc. (ppm)	Initial Volume (mL)	Final Conc. (ppm)	Final Volume [a] (mL)
Holmium	Ho	1000	0.5	5	100
Terbium	Tb	1000	0.5	5	100
Yttrium	Y	1000	0.5	5	100

[a] Solvent for standard preparation is 5% (v/v) HCl + 6% (v/v) HNO_3. HCl and HNO_3 are concentrated trace metals grade acids.

Appendix C

Standard Solution Preparation – Spike Standards

Spike Standards 1 & 2					
Element		Initial Conc. (ppm)	Initial Volume (mL)	Final Conc. (ppm)	Final Volume [a] (mL)
Aluminum	Al	10000	10	100	1000
Antimony	Sb	10000	10	100	1000
Arsenic	As	10000	10	100	1000
Barium	Ba	10000	10	100	1000
Beryllium	Be	10000	10	100	1000
Boron	B	10000	10	100	1000
Cadmium	Cd	10000	10	100	1000
Calcium	Ca	10000	10	100	1000
Chromium	Cr	10000	10	100	1000
Cobalt	Co	10000	10	100	1000
Copper	Cu	10000	10	100	1000
Iron	Fe	10000	10	100	1000
Lead	Pb	5000 [b]	20	100	1000
Magnesium	Mg	10000	10	100	1000
Manganese	Mn	10000	10	100	1000
Molybdenum	Mo	10000	10	100	1000
Nickel	Ni	10000	10	100	1000
Phosphorus	P	10000	10	100	1000
Potassium	K	10000	100	1000	1000
Selenium	Se	10000	10	100	1000
Silicon	Si	10000	10	100	1000
Silver	Ag	10000	5	50	1000
Sodium	Na	10000	100	1000	1000
Strontium	Sr	10000	10	100	1000
Thallium	Tl	10000	10	100	1000
Tin	Sn	10000	10	100	1000
Titanium	Ti	10000	10	100	1000
Vanadium	V	10000	10	100	1000
Zinc	Zn	10000	10	100	1000
Bismuth [c]	Bi	1000	0.05	0.5	100
Sulfur [c]	S	1000	0.05	0.5	100
Lithium [c]	Li	10000	0.005	0.5	100

[a] Solvent for standard preparation is 5% (v/v) HCl + 6% (v/v) HNO_3. HCl and HNO_3 are concentrated trace metals grade acids.

[b] The 5000-ppm Pb spike solution is prepared from the 10000-ppm pre-certified stock standard solution.

[c] Bi ,S, and Li are spiked directly into 50-mL sample.

Appendix D

SAMPLE HOLDING TIMES, REQUIRED DIGESTION VOLUMES AND RECOMMENDED COLLECTION VOLUMES FOR METAL DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

Calscience Environmental Laboratories, Inc.

Appendix D

Sample Holding Times, Required Digestion Volumes and Recommended Collection Volumes for Metal Determinations in Aqueous and Solid Samples

Measurement		Digestion Volume (mL) [a, c]	Collection Volume (mL) [a, c]	Treatment/Preservative Holding Time [b]
Inorganic Analytes (except hexavalent chromium and mercury):				
Aqueous				
	Total	50	250	HNO_3 to pH < 2 6 months
	Dissolved	50	250	Filter on site HNO_3 to pH < 2 6 months
	Suspended	50	250	Filter on site 6 months
Solid				
	Total	2 g	4 oz	6 months
Hexavalent Chromium:				
Aqueous		50	250	24 hours Store at 4 ± 2°C until analyzed
Solid		2.5 g	4 oz	1 month to extraction 4 days after extraction Store at 4 ± 2°C until analyzed
Mercury:				
Aqueous				
	Total	50	250	HNO_3 to pH < 2 28 days
	Dissolved	50	250	Filter HNO_3 to pH < 2 28 days
Solid				
	Total	0.2 g	4 oz	28 days Store at 4 ± 2°C until analyzed

[a] Unless stated otherwise.

[b] Either glass or plastic containers may be used.

[c] Any sample volume reduction from the reference method's instructions must be made in the exact proportion as described in the method and representative sampling must be maintained.

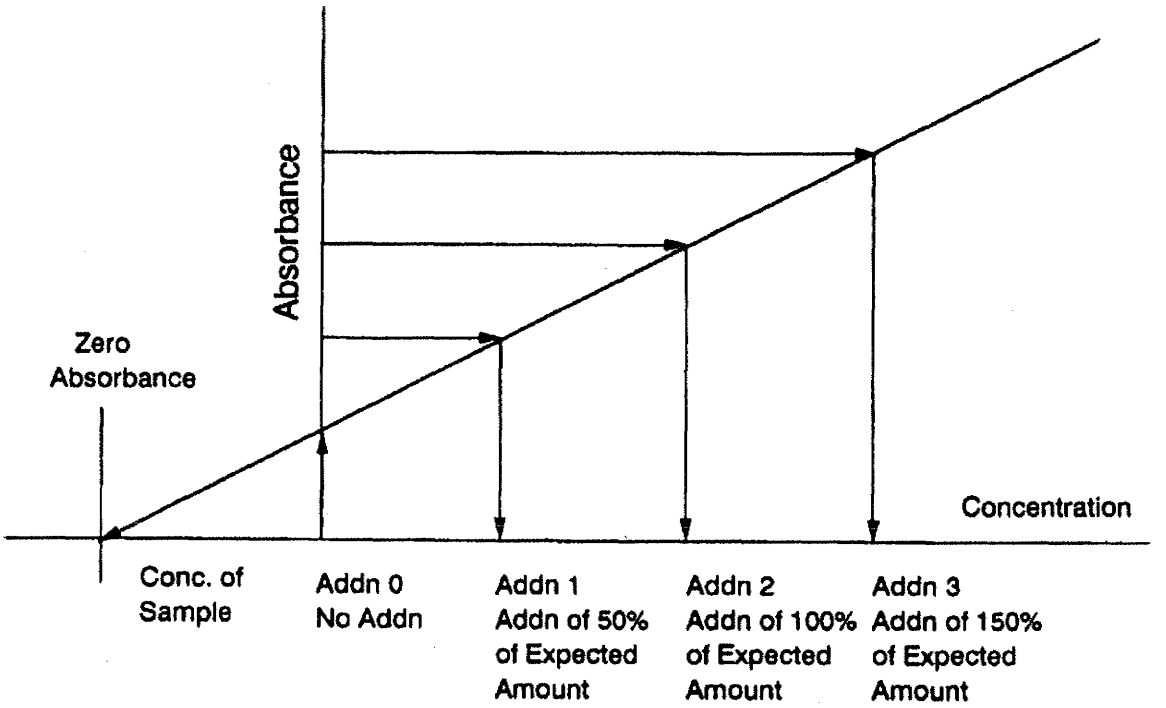
Appendix E

STANDARD ADDITION PLOT (EXAMPLE)

Calscience Environmental Laboratories, Inc.

Appendix E

Standard Addition Plot (Example)



Appendix F

ADDITIONAL QUALITY CONTROL CRITERIA FOR NAVY PROJECT

Calscience Environmental Laboratories, Inc.

1. METHOD IDENTIFICATION

1.1. EPA Method 6010B, Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES) – Additional Quality Control Criteria for Navy Project.

2. SCOPE AND APPLICATION

2.1. The quality control criteria and procedure described herein either supersede or are in addition to the standard quality control criteria and procedure.

3. STANDARDS

3.1. Stock Standard Solutions

3.1.1. Pre-certified stock standard solutions (ultra-high purity grade or equivalent), each in sealed polyethylene bottles, containing 500 ppb of each target analyte are used to prepare low-level calibration verification standards.

3.2. Low-Level Continuing Calibration Verification (LLCCV) Solutions

3.2.1. Prepare the LLCCV solutions in the same acid matrix by combining compatible elements.

3.2.2. The LLCCV solutions contain 5 ppb and 20 ppb of each target analyte.

3.2.2.1. The 20-ppb LLCCV solution is used to verify the RL of a target analyte with higher MDL.

3.3. The use of a standard from a second lot as the second source standard is acceptable when only one manufacturer of the calibration standard exists. "Manufacturer" refers to the producer of the standard, not the vendor.

4. QUALITY CONTROL

4.1. Method Detection Limit (MDL)

4.1.1. MDL study shall be performed annually, or verification checked quarterly for each analyte and matrix.

4.1.1.1. MDL verification must be performed immediately following an MDL study.

4.1.1.2. MDL verification is deemed valid if all analytes in the check sample are reliably detected and identified by method-specific criteria, or if the check sample produces a signal that is at least 3 times the instrument's noise level.

4.1.1.3. If these criteria are not met, run MDL verification check sample at higher level and set MDL higher or reconduct MDL study. No samples shall be analyzed without a valid MDL.

4.2. Instrument Detection Limit (IDL)

4.2.1. IDL study shall be performed at initial instrument setup and when there is significant change on instrument operating conditions.

4.2.2. The IDL for each analyte should be ≤ MDL.

4.2.3. No samples shall be analyzed without a valid IDL.

4.3. Initial Calibration Blank (ICB)

4.3.1. The instrument operating condition is deemed satisfactory for sample analysis to begin if no analytes are detected at a concentration > 2 × MDL.

4.3.2. If these criteria are not met, no sample analysis shall begin. Determine the source of contamination. Reprepare and reanalyze the ICB.

4.4. Daily Spectral Interference Check (ICS-AB and ICS-A)

4.4.1. Following the establishment of a valid initial calibration, the daily spectral interference check solutions must be analyzed daily prior to sample analysis.

4.4.2. The daily spectral interference check is deemed acceptable if the %D for each analyte in the ICS-AB standard is ≤ 20%, and the absolute value of the concentration for each non-spiked analyte in the ICS-A standard is < 2 × MDL (unless it is a verified trace impurity from one of the spiked analytes).

4.4.3. If these criteria are not met, no sample analysis shall begin. Determine the source of problem, effect corrective action, and reanalyze the ICS-AB and/or ICS-A.

4.5. Low-Level Continuing Calibration Verification (LLCCV)

4.5.1. Following the establishment of a valid initial calibration, a LLCCV standard must be analyzed daily prior to sample analysis.

4.5.2. The reporting limit (RL) is deemed valid if the %D for each analyte is ≤ 20%, and the analyte concentration is ≤ RL.

4.5.3. If these criteria are not met, the RL is deemed unacceptable for sample analysis to begin. Effect corrective action and reanalyze the LLCCV.

4.6. Continuing Calibration Blank (CCB)

4.6.1. The instrument operating condition is deemed satisfactory for sample analysis to resume if no analytes are detected at a concentration > 2 × MDL.

4.6.2. If these criteria are not met, no sample analysis shall resume. Determine the source of contamination. Reprepare and reanalyze the CCB. Reanalyze all samples since the last acceptable CCB.

4.6.2.1. The results shall be reported with the appropriate data qualifier (B-flag) for the specific analyte(s) in all samples associated with the failed CCB.

4.7. Event Based Quality Control (LCS/LCSDs and MBs)

4.7.1. Laboratory Control Samples (LCS/LCSDs)

4.7.1.1. The lower and upper acceptance limits for %REC of each LCS/LCSD element in aqueous matrix are as follows:

Analyte		Control Limit		ME Limit	
		Lower	Upper	Lower	Upper
Aluminum	Al	80	120	80	120
Antimony	Sn	80	120	80	120
Arsenic	As	80	120	80	120
Barium	Ba	80	120	80	120
Beryllium	Be	80	120	80	120
Cadmium	Cd	80	120	80	120
Calcium	Ca	80	120	80	120
Cobalt	Co	80	120	80	120
Copper	Cu	80	120	80	120
Iron	Fe	80	120	80	120
Lead	Pb	80	120	80	120
Magnesium	Mg	80	120	80	120
Manganese	Mn	80	120	80	120
Molybdenum	Mo	80	120	75	120
Nickel	Ni	80	120	80	120
Potassium	K	80	120	80	120
Selenium	Se	80	120	75	120
Silver	Ag	80	120	75	120
Sodium	Na	80	120	80	120
Thallium	Tl	80	120	80	120
Vanadium	V	80	120	80	120
Zinc	Zn	80	120	80	120

4.7.1.2. The lower and upper acceptance limits for %REC of each LCS/LCSD element in solid matrix are as follows:

Analyte		Control Limit		ME Limit	
		Lower	Upper	Lower	Upper
Aluminum	Al	80	120	75	120
Antimony	Sn	80	120	75	120
Arsenic	As	80	120	80	120
Barium	Ba	80	120	80	120
Beryllium	Be	80	120	80	120
Cadmium	Cd	80	120	80	120
Calcium	Ca	80	120	80	120
Cobalt	Co	80	120	80	120
Copper	Cu	80	120	80	120
Iron	Fe	80	120	80	120
Lead	Pb	80	120	80	120
Magnesium	Mg	80	120	80	120
Manganese	Mn	80	120	80	120
Molybdenum	Mo	80	120	75	120
Nickel	Ni	80	120	80	120

Analyte		Control Limit		ME Limit	
		Lower	Upper	Lower	Upper
Potassium	K	80	120	80	120
Selenium	Se	80	120	75	120
Silver	Ag	75	120	70	125
Sodium	Na	80	120	80	120
Thallium	Tl	80	120	80	120
Vanadium	V	80	120	80	120
Zinc	Zn	80	120	75	120

4.7.2. Method Blanks (MBs)

4.7.2.1. The concentration of a target analyte in an MB should be ≤ ½ RL. The concentrations of common laboratory contaminants should be < RL.

4.7.2.2. If these criteria are not met, investigate and eliminate the source of contamination.

4.7.2.3. Determine whether to reprocess the samples associated with the failed MB based on the following checks:

4.7.2.3.1. The concentration of a target analyte in the MB is ≥ RL as established by the test method or by regulation, and is > 1/10 of the amount measured in any sample.

4.7.2.3.2. The blank contamination affects the sample results as per the test method requirements or the individual project data quality objectives.

4.7.2.4. Any sample that meets either one or both of the checks described in Section 4.7.2.3. shall be reprocessed in a subsequent preparatory batch, except when the sample analysis resulted in a non-detect.

4.7.2.4.1. If no sample volume remains for reprocessing, the results shall be reported with the appropriate data qualifier (B-flag) for the specific analyte(s) in all samples associated with the failed MB.

4.8. Matrix Based Quality Control (MS/MSDs and PDSs)

4.8.1. Matrix Spikes (MS/MSDs)

4.8.1.1. The lower and upper acceptance limits for %REC of each MS/MSD element in aqueous matrix are as follows:

Analyte		Control Limit	
		Lower	Upper
Aluminum	Al	80	120
Antimony	Sn	80	120
Arsenic	As	80	120
Barium	Ba	80	120

Analyte		Control Limit	
		Lower	Upper
Beryllium	Be	80	120
Cadmium	Cd	80	120
Calcium	Ca	80	120
Cobalt	Co	80	120
Copper	Cu	80	120
Iron	Fe	80	120
Lead	Pb	80	120
Magnesium	Mg	80	120
Manganese	Mn	80	120
Molybdenum	Mo	80	120
Nickel	Ni	80	120
Potassium	K	80	120
Selenium	Se	80	120
Silver	Ag	80	120
Sodium	Na	80	120
Thallium	Tl	80	120
Vanadium	V	80	120
Zinc	Zn	80	120

4.8.1.2. The lower and upper acceptance limits for %REC of each MS/MSD element in solid matrix are as follows:

Analyte		Control Limit	
		Lower	Upper
Aluminum	Al	80	120
Antimony	Sn	80	120
Arsenic	As	80	120
Barium	Ba	80	120
Beryllium	Be	80	120
Cadmium	Cd	80	120
Calcium	Ca	80	120
Cobalt	Co	80	120
Copper	Cu	80	120
Iron	Fe	80	120
Lead	Pb	80	120
Magnesium	Mg	80	120
Manganese	Mn	80	120
Molybdenum	Mo	80	120
Nickel	Ni	80	120
Potassium	K	80	120
Selenium	Se	80	120
Silver	Ag	75	120
Sodium	Na	80	120
Thallium	Tl	80	120
Vanadium	V	80	120
Zinc	Zn	80	120

5. PROCEDURE

5.1. Following the establishment of a valid initial calibration, an LLCCV standard and CCB must be analyzed daily prior to sample analysis, and a CCV standard and CCB must be analyzed after every batch of 10 samples or portion thereof and at the end of sequence. If the QC criteria are met, the initial calibration is assumed to be valid and sample analysis may resume. Additional acceptance criteria are listed in Section 4.3., Section 4.5., and Section 4.6.

5.1.1. If LLCCV/CCB fails, corrective action must be effected prior to analyzing any samples.

5.1.2. If CCV/CCB fails, effect corrective action and reanalyze all samples since the last acceptable CCV/CCB.

5.2. Sample vials are loaded in the following or other logical order:

1) Calibration Blank (CB)
2) Initial Calibration Standard(s)
3) Initial Calibration Verification (ICV)
4) Initial Calibration Blank (ICB)
5) Interference Check Solution AB (ICS-AB)
6) Interference Check Solution A (ICS-A)
7) Low-Level Continuing Calibration Verification (LLCCV)
8) Continuing Calibration Blank (CCB)
9) Method Blank (MB)
10) Laboratory Control Samples (LCS)
11) Laboratory Control Sample Duplicates (LCSD)
12) Samples (up to 10, including QC check samples and MBs)
13) Matrix Spike (MS)
14) Matrix Spike Duplicate (MSD)
15) Post Digestion Spike (PDS)
16) ICS-AB
17) ICS-A
18) Continuing Calibration Verification (CCV)
19) Continuing Calibration Blank (CCB)

5.2.1. Items 5, 6, 16, and 17: The ICS-AB/ICS-A is used to verify the accuracy of the interelement correction factors. An acceptable ICS-AB/ICS-A is required daily prior to sample analysis and at the end of sequence.

5.2.2. Item 7: An LLCCV is used to verify the acceptance of the reporting limit on a continuing basis. An acceptable LLCCV is required daily prior to sample analysis.

5.2.3. Item 18: A CCV is used to verify the acceptance of the initial one-point calibration on a continuing basis. An acceptable CCV is required after every batch of 10 samples or portion thereof and at the end of sequence.

5.3. Dilution test is required daily for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

5.4. Recovery test (post digestion spike addition) is required when dilution test fails or when analyte concentration in all samples is < 50 × MDL.

6. REFERENCES

6.1. ►Department of Defense Quality Systems Manuals for Environmental Laboratories, ***Version 4.2, October 2010.***

Title : **EPA METHOD 7471A, MERCURY IN SOLID OR SEMISOLID WASTE (COLD-VAPOR TECHNIQUE)**

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Revision 3.3 changes are noted in bold italicized typeface and preceded by a "►" marker.

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ANNUAL SOP REVIEW

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1. METHOD IDENTIFICATION

1.1. EPA Method 7471A, Mercury in Solid or Semisolid Waste (Cold-Vapor Technique).

2. APPLICABLE MATRICES

2.1. ►This method is applicable to soils, sediments, bottom deposits, and sludge-type materials. ***It is also applicable to marine and freshwater tissues as a modified method.***

3. ►DETECTION LIMITS

3.1. ***The estimated quantitation limits (EQLs) for this method are as follows:***

	Solid	*Sediment/Tissue*
Mercury	*0.0835 mg/kg*	*0.0200 mg/kg*

3.2. ***The EQLs will be proportionally higher for samples which require reduced sample size.***

3.3. The instrument detection limit data may be used to estimate instrument and method performance for other sample matrices.

4. SCOPE AND APPLICATION

4.1. EPA Method 7471A is a cold-vapor atomic absorption procedure for measuring total (organic and inorganic) mercury. All samples must be subjected to an appropriate dissolution step prior to analysis.

4.1.1. If the dissolution procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix.

4.2. This method is restricted to use by or under the supervision of analysts experienced in the use of atomic absorption spectrometer, skilled in the interpretation of atomic absorption spectra, and knowledgeable in the correction of interferences described in this method.

5. METHOD SUMMARY

5.1. Cold-vapor atomic absorption (CVAA) technique is based on the absorption of radiation at the 253.7-nm wavelength by mercury vapor. The 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 atomic absorption spectrometer. Absorbance (peak height) is measured as a function of mercury concentration.

5.2. ►Prior to analysis, the appropriate sample preparation procedure ***(Appendix A)*** must be performed on each sample.

5.2.1. ***Solid, sediment, and tissue samples are digested with acids.***

6. DEFINITIONS

6.1. Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

6.2. Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

6.3. 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 NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

6.4. Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

6.5. Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

6.6. Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

6.7. Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

6.8. Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

6.9. Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.

6.10. Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

6.11. Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

6.12. Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

6.13. Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

6.14. Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

6.15. Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

6.16. Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

6.17. Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.

6.18. 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.

6.19. 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.

6.20. Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence.

6.21. Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data

and verified accurate by signature), the exact copy or exact transcript may be submitted.

6.22. Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

6.23. Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

6.24. Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

6.25. ►Terms Specific to Mercury Analysis

6.25.1. ***Contract Required Quantitation Limit (CRQL): Minimum level of quantitation acceptable under the contract Statement of Work (SOW).***

6.25.2. ***Lower Limit of Quantitation (LLOQ): The lowest point of quantitation, or in most cases, the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels based on the stated project requirements. Analysis of a standard prepared at the LLOQ concentration level or use of the LLOQ as the lowest point calibration standard provides confirmation of the established quantitation sensitivity of the method.***

6.25.3. Method of Standard Addition (MSA): ***An alternative calibration procedure employed when the signal response of the analyte of interest is different in a particular matrix than when it is in reagent water.*** The standard addition technique involves ***the addition of known amounts of the target analyte to each of a series of replicate sample aliquots. The final concentrations of the sample replicates should span the calibration range of the method. The analytical response versus the standard addition concentration for each of the replicates is plotted. After performing a linear regression, the curve is extrapolated to the x-axis. The analyte concentration in the original unspiked sample is equal to the inverse of the x-intercept.***

6.25.4. Post Digestion (Matrix) Spike: A sample which has been extracted in the same manner as the other samples, but to which a known amount of target analytes has been added to the sample extractant. Post digestion spikes are used to evaluate the accuracy of the method without the losses incurred through the extraction process.

6.25.5. Sensitivity: ***The ability of an analytical technique or instrument to discriminate between small differences in analyte concentration. For atomic absorption***, the concentration of metal, in mg/L, that produces ***a transmission*** of 1% is ***commonly employed to determine sensitivity***.

6.25.6. ***Total Mercury: The concentration of mercury determined in a sample following digestion with acids.***

7. ►INTERFERENCES

7.1. Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 ***mg/kg*** of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.

7.2. Copper may interfere; however, copper concentrations as high as 10 ***mg/kg*** has no effect on the recovery of mercury from spiked samples.

7.3. ***Samples*** high in chlorides require additional permanganate (as much as 25 mL) due to the fact that during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm.

7.3.1. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine ***hydrochloride*** reagent (25 mL).

7.3.2. ***Free chlorine may be removed by allowing a sample to stand for at least an hour under a hood.***

7.4. Certain volatile organic materials that absorb at the wavelength of 253.7 nm may also cause interference.

7.4.1. A preliminary run without reagents may be used to determine whether this type of interference is present.

8. ►SAFETY

8.1. ***Acids are corrosive. Many mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Extreme care must be exercised in the handling of acids and mercury standards.***

8.2. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current version of Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats are required to be worn in all designated laboratory areas. Protective gloves shall be worn when handling chemicals.

8.3. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

9. ►EQUIPMENT AND SUPPLIES

9.1. Atomic Absorption Spectrometer: PerkinElmer Flow Injection Mercury System (FIMS) 400 or equivalent configured with the following components:

9.1.1. ***Computer-controlled atomic absorption spectrometer, single-beam optical system, with 254-nm maximum sensitivity.***

9.1.2. ***Solar-blind detector.***

9.1.3. Mercury lamp, high intensity, ***low pressure***.

9.1.4. Absorption cell, ***240-mm × 7-mm OD × 4-mm ID***, quartz.

9.1.5. ***Two*** peristaltic pumps, ***20–120-rpm variable speed, computer controlled***.

9.1.6. ***Sample loop, 200-µL.***

9.1.7. Autosampler, PerkinElmer AS-90 Autosampler or equivalent.

9.1.8. Autosampler vessels, ***16***-mm ***OD*** (15-mL capacity), ***translucent*** polypropylene, ***disposable***.

9.1.9. ***Autosampler vessels, 30-mm OD (50-mL capacity), with screw caps, translucent polypropylene, disposable.***

9.1.10. PC based data system, ***PerkinElmer WinLab 32 for AA*** or equivalent, ***capable of automatic baseline offset correction***.

9.2. ***Carrier Gas: Argon, Ar, 99.998%, cryogenic liquid, Praxair Argon Cryogenic Liquid or equivalent.***

9.3. Graduated cylinders, ***100-mL or other capacity, glass***, Class A.

9.4. Volumetric flask, 100-mL ***or other capacity***, glass, Class A.

9.5. Pipetters, 100–1000-µL, 0.5–5.0-mL, ***and 1–10-mL, calibrated***, adjustable, with disposable tip.

9.6. ***Refer to Appendix A for additional equipment and supplies.***

10. ►REAGENTS AND STANDARDS

10.1. Reagents

10.1.1. Reagent water, interferant free, nano-pure.

10.1.2. Chips, Teflon.

10.1.3. Beads, glass.

10.1.4. Hydrochloric acid, HCl, ***32–35% or 34–37% (v/v)***, concentrated, ***clear colorless liquid, Fisher Scientific TraceMetal or Optima grade, EMD OmniTrace grade***, or equivalent.

10.1.5. Hydrochloric acid, HCl, 3% (v/v).

10.1.5.1. Prepare the 3% HCl solution by slowly adding ***60*** mL of concentrated HCl to 500 mL of reagent water and dilute to ***2*** L with additional reagent water.

10.1.5.2. The ***3% HCl*** solution is used as the carrier solution.

10.1.5.3. It is also used as a rinse blank to flush the system between standards and samples to minimize interferences.

10.1.6. Nitric acid, HNO_3, ***67–70% (v/v)***, concentrated, ***clear colorless to light yellow liquid, Fisher Scientific TraceMetal or Optima grade, EMD OmniTrace grade***, trace metals grade or equivalent.

10.1.7. ***Stannous chloride, $SnCl_2$, dihydrate, white crystalline powder, reagent grade or equivalent.***

10.1.8. Stannous chloride solution, ***$SnCl_2 \cdot 2H_2O/HCl$***.

10.1.8.1. Prepare the stannous chloride solution by adding 11 g of ***$SnCl_2 \cdot 2H_2O$*** to 1 L of 3% HCl solution.

10.1.8.2. The stannous chloride solution is used as the reducing agent.

10.1.9. ***Refer to Appendix A for additional reagents.***

10.1.10. All reagents must be inspected and documented ***in the Chemicals and Supplies Verification Logbook*** prior to use.

10.2. Standards

10.2.1. Stock Standard Solutions

10.2.1.1. Pre-certified stock standard solutions, ***99.990–99.999% source purity***, each in sealed polyethylene bottles, containing ***1000/100*** ppm of mercury are used to prepare calibration and check standards.

10.2.1.2. ***Prepare each mercury working standard solution by diluting the appropriate volume of the mercury stock standard and 5 mL of concentrated HNO_3 to 100 mL with reagent water.***

10.2.1.3. ***The working standards are prepared as follows:***

Analyte	Initial		Final	
	Conc (ppm)	Volume (mL)	Conc (ppm)	Volume (mL)
Hg	1000	0.2	2.0	100

Note: The working standard solution contains 5% (v/v) of HNO_3.

Analyte	Initial		Final	
	Conc (ppm)	Volume (mL)	Conc (ppm)	Volume (mL)
Hg	100	1.0	1.0	100

Note: The working standard solution contains 5% (v/v) of HNO_3.

10.2.1.4. ***The working standard solutions must be replaced after one month or sooner if comparison with check standards indicates a problem.***

10.2.2. Initial Calibration Standard Solutions

10.2.2.1. Measure 0.5 mL of the 2.0-ppm mercury working standard solution into a clean ***digestion tube***.

10.2.2.2. Add 10 mL of the ***diluted*** aqua regia solution to the ***digestion tube***.

10.2.2.3. Place the ***digestion tube*** in the ***pre-heated*** block digester, ***cover the digestion tube with a clean watch glass***, and heat for 2 minutes in the water bath maintained at 95°C.

10.2.2.4. ***Remove the digestion tube from the block digester and allow the digested standard solution to cool.***

10.2.2.5. Add 50 mL of reagent water and 15 mL of the 5% $KMnO_4$ solution to the ***digestion tube.***

10.2.2.6. ***Mix the contents of the digestion tube thoroughly.***

10.2.2.7. Place the ***digestion tube*** in the ***pre-heated*** block digester, ***cover the digestion tube with the same watch glass***, and continue to heat for 30 minutes in the water bath maintained at 95°C.

10.2.2.8. ***Remove the digestion tube from the block digester and allow the digested standard solution to cool.***

10.2.2.9. Add 6 mL of the sodium chloride-hydroxylamine hydrochloride solution to the ***digested standard solution*** to reduce excess permanganate.

10.2.2.10. Adjust the volume ***of the digested standard solution*** to 100 mL with calibration blank to obtain the 10.0-ppb initial calibration standard.

10.2.2.11. Dilute the appropriate volumes of the 10.0-ppb initial calibration standard to 20 mL with calibration blank to obtain other initial calibration standards.

10.2.2.12. ***Use the following calibration levels as guidance to prepare the initial calibration standards.***

Calibration Level (ppb)	Initial Conc (ppb)	Initial Volume (mL)	Final Volume (mL)
0.25	10.0	0.5	20.0
1.0	10.0	2.0	20.0
2.0	10.0	4.0	20.0
5.0	10.0	10.0	20.0
10.0	10.0	20.0	20.0

10.2.2.13. ***Use the following calibration levels as guidance to prepare the initial calibration standards for lower limit of quantitation.***

Calibration Level (ppb)	Initial Conc (ppb)	Initial Volume (mL)	Final Volume (mL)
0.025	10.0	0.05	20.0
0.25	10.0	0.5	20.0
1.0	10.0	2.0	20.0
2.0	10.0	4.0	20.0
5.0	10.0	10.0	20.0
10.0	10.0	20.0	20.0

10.2.2.14. The 2.0-ppb initial calibration standard is also used as the continuing calibration verification solution.

10.2.2.15. ***The initial calibration standard solutions must be prepared fresh daily.***

10.2.3. Calibration Blank (CB)

10.2.3.1. ***Designate a minimum of five clean digestion tubes for calibration blank preparation.***

10.2.3.2. Measure 0.5 mL of reagent water into each clean ***digestion tube***.

10.2.3.3. Add 10 mL of the ***diluted*** aqua regia solution to each ***digestion tube***.

10.2.3.4. Place each ***digestion tube*** in the ***pre-heated*** block digester, ***cover the digestion tube with a clean watch glass***, and heat for 2 minutes in the water bath maintained at 95°C.

10.2.3.5. ***Remove the digestion tubes from the block digester and allow the digested reagent water to cool.***

10.2.3.6. Add 50 mL of reagent water and 15 mL of the 5% $KMnO_4$ solution to each ***digestion tube.***

10.2.3.7. ***Mix the contents of each digestion tube thoroughly.***

10.2.3.8. Place each ***digestion tube*** in the ***pre-heated*** block digester, ***cover the digestion tube with the same watch glass***, and continue to heat for 30 minutes in the water bath maintained at 95°C.

10.2.3.9. ***Remove the digestion tube from the block digester and allow the digested reagent water to cool.***

10.2.3.10. Add 6 mL of the sodium chloride-hydroxylamine hydrochloride solution to the ***digested reagent water*** to reduce excess permanganate.

10.2.3.11. The CB is used to establish the zero point of the calibration curve or to dilute standards and samples.

10.2.3.12. The CB is also used either as initial calibration blank (ICB) or as continuing calibration blank (CCB) to monitor contamination.

10.2.4. Method Blank (MB)

10.2.4.1. Process the MBs ***using the appropriate sample preparation procedure***.

10.2.4.2. The MB is used to identify possible contamination resulting from either the reagents or the equipment used during sample processing.

10.2.5. Initial Calibration Verification (ICV) Solution

10.2.5.1. Measure 0.5 mL of the 1.0-ppm mercury working standard solution into a clean ***digestion tube***.

10.2.5.2. Add 10 mL of the ***diluted*** aqua regia solution to the ***digestion tube***.

10.2.5.3. Place the ***digestion tube*** in the ***pre-heated*** block digester, ***cover the digestion tube with a clean watch glass***, and heat for 2 minutes in the water bath maintained at 95°C.

10.2.5.4. ***Remove the digestion tube from the block digester and allow the digested standard solution to cool.***

10.2.5.5. Add 50 mL of reagent water and 15 mL of the 5% $KMnO_4$ solution to the ***digestion tube.***

10.2.5.6. ***Mix the contents of the digestion tube thoroughly.***

10.2.5.7. Place the ***digestion tube*** in the ***pre-heated*** block digester, ***cover the digestion tube with the same watch glass***, and continue to heat for 30 minutes in the water bath maintained at 95°C.

10.2.5.8. ***Remove the digestion tube from the block digester and allow the digested standard solution to cool.***

10.2.5.9. Add 6 mL of the sodium chloride-hydroxylamine hydrochloride solution to the ***digested standard solution*** to reduce excess permanganate.

10.2.5.10. Adjust the volume ***of the digested standard solution*** to 100 mL with calibration blank to obtain the 5.0-ppb ICV solution.

10.2.5.11. ***The ICV solution must be of a source differing from that used for the initial multi-point calibration. If it is of the same source, then it must be of different lot.***

10.2.5.12. ***The ICV solution must be prepared fresh daily.***

10.2.6. Continuing Calibration Verification (CCV) Solution

10.2.6.1. Dilute 4.0 mL of the 10.0-ppb initial calibration standard to 20 mL with calibration blank to obtain the 2.0-ppb CCV solution.

10.2.6.2. ***The CCV solution is of a source same as that used for the initial multi-point calibration.***

10.2.6.3. ***The CCV solution must be prepared fresh daily.***

10.2.7. ***Refer to Appendix A for additional standards.***

10.2.8. All stock standards must be inspected and documented ***in the Chemicals and Supplies Verification Logbook*** prior to use.

11. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

11.1. Solid samples should be collected in 4-oz ***or 8-oz*** pre-cleaned clear glass wide-mouth jars with Teflon-lined closures, ***or 6-in decontaminated brass or stainless steel sleeves with Teflon-lined closures***.

11.2. ***Tissue samples should be collected in 4-oz (or other appropriate capacity) pre-cleaned clear glass or quartz wide-month jars with Teflon or aluminum foil-lined closures. Refer to SOP-M229 for additional information on sample collection, preservation, and containers.***

11.3. Solid samples shall be maintained in a chilled state (≤ 4°C) post sample collection until received at the laboratory. Samples should not be frozen (e.g., do not use dry ice as the refrigerant).

11.4. ***Tissue samples shall be maintained in a frozen state (≤ −20°C) post sample collection until received at the laboratory. Refer to SOP-M229 for additional information on sample collection and preservation.***

11.5. Upon receipt, the solid samples are stored in a ***0~6***°C cooler, ***and the tissue samples are stored in a −10~−20°C freezer***.

11.5.1. ***Solid and tissue*** samples must be digested and analyzed within 28 days of sample collection.

12. ►QUALITY CONTROL

12.1. Initial Calibration (IC)

12.1.1. The initial ***multi-point*** calibration must be established daily prior to the processing of samples.

12.1.1.1. The calibration curve is established with one calibration blank and five ***or six*** calibration standards.

12.1.2. The IC is deemed valid if the correlation coefficient, r, for linear least squares regression of each analyte is ≥ 0.995.

12.1.3. If these criteria are not met, then the calibration is unacceptable for sample analysis to begin. Effect corrective action and recalibrate.

12.2. Initial Calibration Verification (ICV)

12.2.1. The initial calibration is deemed valid if the %D for each analyte is ≤ 10%.

12.2.2. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to begin. An unacceptable ICV result indicates either a

disagreement between like solutions from separate sources or a change in instrument conditions. Normally, this is caused when at least one of the solutions is no longer intact (representative of the stated concentration). ***Document the unacceptable result and reanalyze the ICV within 2 hours after the failed ICV. If the ICV criteria remain unacceptable***, investigate, effect corrective action, which may include re-preparation of standard solutions, and recalibrate.

12.3. Initial Calibration Blank (ICB)

12.3.1. The instrument operating condition is deemed satisfactory for sample analysis to begin if no analytes are detected at a concentration ≥ RL (or the limit specified in the project specific DQO).

12.3.2. If these criteria are not met, no sample analysis shall begin. Determine the source of contamination. Re-prepare and reanalyze the ICB.

12.4. Continuing Calibration Verification (CCV)

12.4.1. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily after every batch of 10 samples or portion thereof, and at the end of sequence.

12.4.2. The initial calibration is deemed valid if the %D for each analyte is ≤ 20%.

12.4.3. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to resume. ***Document the unacceptable result*** and reanalyze the CCV within 2 hours after the failed CCV. If the CCV criteria remain unacceptable, ***effect corrective action and*** recalibrate.

12.5. Continuing Calibration Blank (CCB)

12.5.1. The instrument operating condition is deemed satisfactory for sample analysis to resume if no analytes are detected at a concentration ≥ RL (or the limit specified in the project specific DQO).

12.5.2. If these criteria are not met, no sample analysis shall resume. Determine the source of contamination. Re-prepare and reanalyze the CCB.

12.6. ***Contract Required Quantitation Limit (CRQL) Check***

12.6.1. ***A CRQL check standard is analyzed immediately following the ICV/ICB analyses.***

12.6.1.1. ***The concentration of each analyte in the CRQL check solution shall be at the lowest calibration level.***

12.6.2. ***The linearity of the calibration curve is deemed verified if the recovery of each analyte is within 70–130%.***

12.6.3. ***CRQL check is performed per client request or project specific DQOs to verify the linearity of the calibration curve.***

12.7. ***Lower Limit of Quantitation (LLOQ) Check***

12.7.1. ***An LLOQ check sample is analyzed immediately following the ICV/ICB analyses.***

12.7.1.1. ***The concentration of each analyte in the LLOQ check sample is at the established laboratory reporting limit.***

12.7.1.2. ***The LLOQ check sample shall be carried through the entire preparation and analytical procedure.***

12.7.2. ***The lower limit of quantitation is deemed verified if each analyte is detected at within ± 30% of its expected value.***

12.7.3. ***LLOQ check is performed per client request or project specific DQOs to demonstrate the desired detection capability.***

12.8. Event Based Quality Control (LCS/LCSDs and MBs)

12.8.1. Event based quality control consists of QC samples prepared and processed with each preparatory event. This consists of a laboratory control sample and laboratory control sample duplicate (LCS/LCSD) and a method blank (MB).

12.8.2. The acceptance criteria for LCS/LCSD elements are as follows:

12.8.2.1. The lower and upper acceptance limits for %REC and RPD of each LCS/LCSD element are based upon the historical average recovery ± 3S that is updated at least annually.

12.8.2.1.1. ***If historical data is unavailable, the lower and upper acceptance limits for %REC of each LCS/LCSD element are 80% and 120%, respectively. The RPD is ≤ 20%.***

12.8.2.1.2. ***The acceptance limits derived from historical data should not be wider than ± 20% for accuracy and 20% for precision.***

12.8.2.2. All LCS/LCSD elements must be within acceptance limits. ***If the LCS/LCSD elements are not acceptable, determine the cause of the problem and effect corrective action.***

12.8.3. Ideally, the concentration of target analyte in an MB should be less than the respective ***limit specified in the project specific data quality objective (DQO). In the absence of project specific DQO, the concentration of target analyte in an MB should be less than or equal to one half of the respective RL. If regulatory limit is available, the concentration of target analyte in an MB should be less than 10% of the respective regulatory limit.*** If the concentration of target analyte exceeds its ***specified limit***, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs are as follows:

12.8.3.1. If the target analyte is found in the MB, but not in the associated samples, report the sample and MB data without qualification.

12.8.3.2. If the target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified, or rejected and the samples re-processed and/or re-analyzed.

12.9. Matrix Based Quality Control (MS/MSDs and PDSs)

12.9.1. Matrix based quality control consists of QC samples prepared and processed using actual environmental samples. This consists of a matrix spike and matrix spike duplicate (MS/MSD) and a post digestion spike (PDS).

12.9.2. The acceptance criteria for MS/MSD elements are as follows:

12.9.2.1. The lower and upper acceptance limits for %REC and RPD of each MS/MSD element are based upon the historical average recovery ± 3S that is updated at least annually.

12.9.2.1.1. ***If historical data is unavailable, the lower and upper acceptance limits for %REC of each MS/MSD element are 80% and 120%, respectively. The RPD is ≤ 20%.***

12.9.2.1.2. ***The acceptance limits derived from historical data should not be wider than ± 20% for accuracy and 20% for precision.***

12.9.2.2. When the %REC and RPD of the MS/MSD elements are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.

12.9.2.3. If the %REC and/or RPD of the MS/MSD elements are not within the established acceptance limits, the analytical system performance shall be suspect.

12.9.3. Unacceptable %REC values are typically caused by matrix effects or poor instrument performance/technique. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.

12.10. If the %REC or RPD of the MS/MSD and LCS/LCSD are unacceptable, all associated sample data must be invalidated and all associated samples re-processed and re-analyzed.

12.11. Dilution Test

12.11.1. If the analyte concentration is sufficiently high ***(minimally, a factor of 10 above the reporting limit after dilution)***, an analysis of a 1:5 dilution should agree within ± 10% of the original determination.

12.11.2. If this criterion is not met, a ***chemical or physical*** interference effect should be suspected. Perform ***post digestion spike addition***.

12.11.3. ***Dilution test is performed per client request or project specific DQOs.***

12.12. Post Digestion Spike Addition

12.12.1. A PDS sample is prepared by adding the spike standard to a portion of a digested sample, or its dilution. ***The spike addition should produce a concentration of 10–100 times the RL.***

12.12.2. The acceptance criteria for PDS elements are as follows:

12.12.2.1. The lower and upper acceptance limits for %REC of each PDS element are 85% and 115%, respectively.

12.12.2.2. If the %REC of a PDS element is not within the established acceptance limits, then matrix effects should be suspected. Perform MSA on all samples in the ***same preparation batch***.

12.12.3. ***Matrix effects are confirmed if the %REC values of both the MS/MSD and the PDS are unacceptable.***

12.12.4. ***Post digestion spike addition is performed per client request or project specific DQOs.***

12.13. Additional information regarding internal quality control checks is provided in SOP-T020.

13. ►CALIBRATION AND STANDARDIZATION

13.1. Pipetter

13.1.1. Calibrate the pipetter according to the procedure outlined in the ***current revision of SOP-T043, "Support Equipment – Calibration, Verification, Monitoring."***

13.2. Spectrometer Initial Calibration

13.2.1. Establish an acceptable ***multi***-point calibration curve. The acceptance criteria for the initial calibration are listed in Section 12.1.

13.2.2. After obtaining an acceptable ***multi***-point calibration curve and prior to processing ***field or*** QC sample digestates, an ICV standard and ICB must be analyzed to verify the initial calibration. The acceptance criteria for the ICV and ICB are listed in Section 12.2. and Section 12.3.

13.2.2.1. ***Per client request or project specific DQOs, a CRQL check standard must be analyzed immediately following the ICV/ICB analyses to verify the linearity of the calibration***

curve. The acceptance criteria for the CRQL are listed in Section 12.6.

13.2.2.2. ***Per client request or project specific DQOs, an LLOQ check sample must be analyzed immediately following the ICV/ICB analyses to verify the lower limit of quantitation. The acceptance criteria for the LLOQ are listed in Section 12.7.***

13.2.3. The initial ***multi***-point calibration and ICV shall include all anticipated target analytes for the duration of the use of the initial calibration.

14. ►PROCEDURE

14.1. Instrument Setup

14.1.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable (usually requiring at least 15 minutes of operation) prior to calibration. Follow the instructions provided by the instrument manufacturer for operating conditions.

14.1.1.1. ***Use the following CVAA operating conditions as guidance.***

Description	Operating Condition
Carrier gas flow	40~70 mL/min
Pump #1 speed	100 rpm
Pump #2 speed	120 rpm
Carrier solution / sample diluent (3.0% HCl)	9~11 mL/min
Reductant (1.1% $SnCl_2$ in 3.0% HCl)	5~7 mL/min
Reaction coil	110-mm × 1.0-mm ID
Wavelength	253.7 nm
Number of replicates	2

14.1.1.2. Autosampler is set to inject ***200*** µL of ***field*** or QC sample digestate.

14.1.2. Place the inlet of the blue/yellow carrier solution pump tube in the hydrochloric acid reservoir. Place the inlet of the red/red reductant pump tube in the stannous chloride reservoir. Position the sampling probe in the reagent water reservoir.

14.1.3. Check the carrier solution and reductant flow rates. The reductant flow rate should be approximately one half of the carrier solution flow rate.

14.1.3.1. Check the flow rate by placing the inlet of the tube in a graduated cylinder filled with reagent water, and then measure the volume decrease after one minute.

14.1.3.2. If the flow rate is not within the appropriate range, adjust the pump pressure for the tube until the flow rate is within range.

14.1.4. Program the system to average ***two integrations*** on each blank, standard, and sample. Report the average.

14.1.4.1. If the %RSD for an analyte in a standard is > 10%, ***document the unacceptable result*** and reanalyze the standard. If the %RSD criterion remains unacceptable, investigate, effect corrective action, which may include re-preparation of the standard solution, and recalibrate, if necessary.

14.1.4.2. If the %RSD for an analyte in a sample is > 20%, and the analyte concentration exceeds its RL, ***document the unacceptable result*** and reanalyze the sample. If the %RSD criterion remains unacceptable, investigate and effect corrective action.

14.2. Establish a calibration curve to cover the appropriate concentration range (see Section ***13.2.***).

14.3. Following the establishment of a valid initial calibration, a CCV standard and CCB must be analyzed daily after every batch of 10 samples or portion thereof, and at the end of sequence. If the QC criteria are met, the initial calibration is assumed to be valid and sample analysis may resume. The acceptance criteria are listed in Section 12.4. and Section 12.5.

14.3.1. ***If a failed CCV/CCB is the first of the day, effect corrective action and reanalyze all samples since the last acceptable ICV/ICB.***

14.3.2. If a failed CCV/CCB ***is <u>not</u> the first of the day***, effect corrective action and reanalyze all samples since the last acceptable CCV/CCB.

14.4. Following ***preparatory*** procedures specified in ***Appendix A***, the digestates for the QC and actual environmental samples are received in ***digestion tubes. After transferring aliquots of the digestates to autosampler vessels***, the autosampler vessels are then loaded onto the system sample tray.

14.5. ***Standard and*** sample vessels are loaded in the following or other logical order:

1) Calibration Blank (CB)
2) Initial Calibration Standards
3) Initial Calibration Verification (ICV)
4) Initial Calibration Blank (ICB)
5) ***CRQL or LLOQ Check (per client request or project specific DQOs)***
6) Method Blank (MB)
7) Laboratory Control Samples (LCS)
8) Laboratory Control Sample Duplicates (LCSD)
9) Samples (up to 10 per batch, including QC check samples and MBs)
10) Continuing Calibration Verification (CCV)
11) Continuing Calibration Blank (CCB)
12) Matrix Spike (MS)
13) Matrix Spike Duplicate (MSD)
14) ***Dilution Test Sample (per client request or project specific DQOs)***

15) Post Digestion Spike (PDS) ***(per client request or project specific DQOs)***
16) Samples (up to 10 per batch, including QC check samples and MBs)
17) Ending CCV
18) Ending CCB

14.5.1. Item 1: The CB is an aliquot of reagent water digestate used to establish the zero point of the initial calibration curve.

14.5.2. Item 2: The initial calibration standards are used to establish the initial calibration curve.

14.5.3. Item 3: The ICV is a second source standard used to verify the acceptance of the initial ***multi***-point calibration. An acceptable ICV is required daily after initial calibration.

14.5.4. Item 4: The ICB is an aliquot of reagent water digestate used to monitor contamination. An acceptable ICB is required ***immediately following ICV***.

14.5.5. ***Item 5: The CRQL check standard is used to verify the linearity of the calibration curve. The LLOQ check sample is used to verify the lower limit of quantitation. Per client request or project specific DQOs, an acceptable CRQL or LLOQ check is required immediately following ICV and ICB.***

14.5.6. Item ***6***: The MB is a known matrix similar to the samples being analyzed which is processed concurrently with the associated samples. In the processing of the MB, reagents and procedures identical to those for actual samples are used.

14.5.6.1. For solid ***and tissue*** samples, the MB consists of clean Teflon chips or glass beads.

14.5.6.2. One MB is required every day ***preparatory methods (i.e.,*** leachings, ***filtrations***, digestions, etc.) are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

14.5.6.3. When samples that are processed together are analyzed on separate instruments or on separate analytical shifts, the MB associated with those samples must be analyzed on at least one of the instruments. A solvent blank consisting of reagent water digestate must be analyzed on all other instruments where the associated samples are analyzed to demonstrate that the instruments are not contributing contaminants to the samples.

14.5.7. Item ***7***: The LCS is a known matrix which has been spiked with known concentration of specific target analyte. The purpose of the LCS is to demonstrate that the entire analytical process and systems are in control. The LCS is processed concurrently with the associated samples. In the processing of the LCS, reagents and procedures identical to those for actual samples are used.

14.5.7.1. For solid ***and tissue*** samples, the LCS consists of the specified element spiked into clean Teflon chips or glass beads.

14.5.7.2. One LCS is required every day ***preparatory methods (i.e.,*** leachings, ***filtrations***, digestions, etc.) are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

14.5.8. Item ***8***: The LCSD is handled identically to the LCS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the LCS in combination with the LCSD can be used to assess the precision of the analytical process. The measurement is expressed as relative percent difference (RPD). The formula for calculating RPD is listed in Section 15.5.

14.5.9. Items ***9*** and ***16***: Up to 10 sample (including QC check sample and method blank) digestates per batch. Digestates should be sufficiently diluted if concentrations exceed the calibration range. Dilution of digestates will result in increased reporting limits.

14.5.9.1. ***All dilutions should keep the responses of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.***

14.5.10. Items ***10*** and ***17***: A CCV is a standard used to verify the acceptance of the initial ***multi***-point calibration on a continuing basis. An acceptable CCV is required daily after every batch of 10 samples or portion thereof, and at the end of sequence.

14.5.11. Items ***11*** and ***18***: A CCB is an aliquot of reagent water digestate used to monitor contamination. An acceptable CCB is required ***immediately following CCV***.

14.5.12. Item ***12***: The MS is the actual sample matrix spiked with known concentration of specific target analyte. The sample which is spiked for the MS is processed concurrently with the associated samples. In the processing of the MS, reagents and procedures identical to those for actual samples are used.

14.5.12.1. The purpose of the MS is to assess the effect of a sample matrix on the recovery of target analyte (i.e., assess the accuracy of the analytical measurements of the matrix). The measurement is expressed as percent recovery (%REC). The formula for calculating %REC is listed in Section 15.4.

14.5.12.2. One MS is required for every batch of 20 samples per matrix or portion thereof ***processed*** concurrently.

14.5.13. Item ***13***: The MSD is handled identically to the MS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the MS in combination with the MSD can be used to assess the precision of the analytical measurements. The measurement is

expressed as relative percent difference (RPD). The formula for calculating RPD is listed in Section 15.5.

14.5.14. ***Item 14: The dilution test sample is prepared from the five-fold dilution of a high concentration sample post digestion. The high concentration sample is diluted to one-fifth of the original concentration post digestion to confirm that no interference is observed in the original sample.***

14.5.14.1. ***The purpose of the dilution test sample is to assess matrix effects.***

14.5.14.2. ***To comply with client request or project specific DQOs, one dilution test sample is required daily for every batch of 20 samples per matrix or portion thereof processed concurrently.***

14.5.15. Item ***15***: The PDS is the same sample matrix from which the MS/MSD samples were prepared, and is spiked with known concentration of specific target analyte post digestion. The sample which will be spiked for the PDS is processed concurrently with the associated samples. In the processing of the PDS, reagents and procedures identical to those for actual samples are used.

14.5.15.1. The purpose of the PDS is to confirm matrix effects. The measurement is expressed as percent recovery (%REC). The formula for calculating %REC is listed in Section 15.4.

14.5.15.2. ***The number of PDS required is based upon client request or project specific DQOs.***

14.5.16. Rinse blanks consisting of 3% HCl solution may be added elsewhere in the sequence to rinse the analytical system.

14.6. Ensure that sufficient amounts of 3% HCl solution and stannous chloride solution are present in the 3% HCl and stannous chloride reservoirs, respectively, and that a sufficient unused volume exists in the waste container at the beginning of the sequence.

14.7. Edit the sequence in the data system. After all correct sample information is entered, save the sequence. After saving the sequence, record pertinent information in the instrument run logbook ***or on the sequence table printout***.

14.7.1. ***Record the reagent and standard identification numbers on the sequence table printout.***

14.8. Initiate the sequence.

14.9. Data Interpretation

14.9.1. Quantitation of a target analyte is based on a reproducible response of the spectrometer within the calibration range and a direct proportionality of the magnitude of response between absorbances in the sample digestate and the calibration standards.

14.9.1.1. Proper quantitation requires the appropriate selection of a wavelength from which the absorbance of an element can be determined.

14.9.1.2. Determine the concentration based on the initial calibration curve.

14.9.1.2.1. The data system is programmed to perform the calculation of concentration ***via the Beer-Lambert Law***.

14.9.1.3. If the instrument response exceeds the calibration range, dilute the digestate and reanalyze.

14.10. Method of Standard Additions (MSA)

14.10.1. The standard addition technique involves adding known amounts of a standard solution to one or more aliquots of a ***processed*** sample. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. However, it will not correct for additive interferences which cause a baseline shift.

14.10.1.1. The MSA may be appropriate for analyses of ***digestates***, on analyses submitted as part of a delisting petition, whenever a new sample matrix is being analyzed, and on every batch that fails the ***post digestion spike addition***.

14.10.2. The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_s of a standard analyte solution of concentration C_s. To the second aliquot (labeled B) is added the same volume V_s of the ***digested reagent water***. The analytical signals of A and B, S_A ***and*** S_B, are measured and corrected for non-analyte signals. The unknown sample concentration C_x is calculated using the formula listed in Section ***15.9.*** V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

14.10.3. Improved results can be obtained by employing a series of standard additions. A series of standard solutions containing different known quantities of the analyte are added to equal volumes of the sample, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50% of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100% and 150% of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is

the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot is shown in Appendix ***B***. A linear regression program may be used to obtain the intercept concentration.

14.10.4. For the results of the MSA technique to be valid, the following limitations must be taken into consideration:

14.10.4.1. The apparent concentrations from the calibration curve must be linear (correlation coefficient of 0.995 or greater) over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve.

14.10.4.2. The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

14.10.4.3. The determination must be free of spectral interference and corrected for nonspecific background interference.

15. ►CALCULATIONS

15.1. The percent relative standard deviation is calculated as follows:

$$\%RSD = \frac{SD}{A_{ave}} \times 100$$

where: %RSD = percent relative standard deviation.
SD = standard deviation of the absorbances for the target analyte.
A_{ave} = mean of the absorbances for the target analyte.

15.2. The percent difference of each analyte is calculated as follows:

$$\%D = \frac{|C_{expected} - C_{measured}|}{C_{expected}} \times 100$$

where: %D = percent difference.
$C_{expected}$ = concentration of target analyte expected.
$C_{measured}$ = concentration of target analyte measured.

Note: Concentrations must be in equivalent units.

15.3. The recovery of each LCS element is calculated as follows:

$$\%REC_{LCS} = \frac{C_{recovered}}{C_{added}} \times 100$$

where: $\%REC_{LCS}$ = percent recovery of target analyte in LCS (or LCSD).
$C_{recovered}$ = concentration of target analyte recovered.
C_{added} = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

15.4. The recovery of each MS element is calculated as follows:

$$\%REC_{MS} = \frac{C_{recovered} - C_{sample}}{C_{added}} \times 100$$

where: $\%REC_{MS}$ = percent recovery of target analyte in MS (or MSD/PDS).
$C_{recovered}$ = concentration of target analyte recovered.
C_{sample} = concentration of target analyte in environmental sample used.
C_{added} = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

15.5. The relative percent difference is calculated as follows:

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where: RPD = relative percent difference between two measurements (C_1 and C_2).
C_1 = concentration of target analyte in measurement 1.
C_2 = concentration of target analyte in measurement 2.

Note: Concentrations must be in equivalent units.

15.6. The target analyte concentration for a solid sample is calculated as follows:

$$C_S = \frac{C_x \times V_x \times D}{W_S}$$

where: C_S = concentration of target analyte in solid sample in mg/kg.
C_x = concentration of target analyte in digestate in µg/L.
V_x = volume of digestate in mL.
W_S = mass of solid sample digested in mg.
D = dilution factor, if the digestate was diluted prior to analysis. If no dilution was made, D = 1.

15.7. ***The target analyte concentration for a solid sample on a dry-weight basis is calculated as follows:***

$$C_S = \frac{C_x \times V_x \times D}{W_S \times \left(\frac{C_{ss}}{100}\right)}$$

where: ***C_S = concentration of target analyte in solid sample in mg/kg.***
C_x = concentration of target analyte in digestate in µg/L.
V_x = volume of digestate in mL.
W_S = mass of solid sample digested in mg.
C_{ss} = solids content in %.
D = dilution factor, if the digestate was diluted prior to analysis. If no dilution was made, D = 1.

15.8. ***The target analyte concentration for a tissue sample is calculated as follows:***

$$C_T = \frac{C_x \times V_x \times D}{W_T}$$

where: ***C_T = concentration of target analyte in tissue sample in mg/kg.***
C_x = concentration of target analyte in digestate in µg/L.
V_x = volume of digestate in mL.
W_T = mass of tissue sample digested in mg.
D = dilution factor, if the digestate was diluted prior to analysis. If no dilution was made, D = 1.

15.9. The target analyte concentration from single-addition method is calculated as follows:

$$C_x = \frac{S_B \times V_s \times C_s}{(S_A - S_B) \times V_x}$$

where: C_x = concentration of target analyte in sample.
S_A = analytical signal (corrected for the blank) of ***sample aliquot*** A.
S_B = analytical signal (corrected for the blank) of ***sample aliquot*** B.
V_s = volume of target analyte in standard solution.
C_s = concentration of target analyte in standard solution.
V_x = volume of target analyte in sample.

Note: Concentrations and volumes must be in equivalent units.

15.10. All concentrations shall be reported in mg/kg (ppm) for soil and solid waste samples, ***and mg/kg (ppm) for tissue samples***.

15.10.1. ***To comply with client request or project specific DQOs, report all concentrations in mg/kg (ppm) on a dry-weight basis for soil and solid waste samples.***

15.11. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

16. METHOD PERFORMANCE

16.1. A demonstration of analytical capability shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

16.2. Calibration protocols specified in Section 13., "Calibration and Standardization," shall be followed.

16.3. Proficiency test sample results shall be used to evaluate the ability to produce accurate results.

17. POLLUTION PREVENTION

17.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Calscience's Health, Safety, and Respiratory Protection Manual. In general, protective eyewear (e.g. safety glasses or goggles), and protective apparel (e.g. lab coats) and gloves are required to be worn when handling chemicals.

17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:

17.3.1. A NIOSH approved air purifying respirator with cartridges appropriate for the chemical handled.

17.3.2. Extended length protective gloves.

17.3.3. Face shield.

17.3.4. Full-length laboratory apron.

17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.

17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles. Alternatively, the Coordinator may utilize a self-contained breathing apparatus or other supplied air system if appropriately trained and approved by the Health and Safety Manager.

17.6. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

18. ►DATA ASSESSMENT AND ACCEPTANCE CRITERIA

18.1. The acceptance criteria for LCS/LCSD elements vary depending upon historical data. The lower and upper acceptance limits for %REC and RPD of each LCS/LCSD element are based upon the historical average recovery ± 3S ***that is updated at least annually***. All LCS/LCSD elements must be within acceptance limits.

18.1.1. If the LCS and/or LCSD %REC is outside of the acceptance limits high, the RPD is within acceptance limits, and all target analytes in the associated samples are not detected, the sample data can be reported without qualification.

18.1.2. The LCSD is only reported when the MS/MSD is unacceptable due to matrix interference effects, or when the LCS/LCSD is used in place of MS/MSD due to insufficient sample quantity.

18.2. Ideally, the concentration of target analyte in an MB should be less than the respective ***limit specified in the project specific DQO. In the absence of project specific DQO, the concentration of target analyte in an MB should be less than or equal to one half of the respective RL. If regulatory limit is available, the concentration of target analyte in an MB should be less than 10% of the respective regulatory limit***. If the concentration of the target analyte exceeds its ***specified limit***, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs are as follows:

18.2.1. If the target analyte is found in the MB but not in the associated samples, report the sample and MB data without qualification.

18.2.2. If the target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-processed and/or re-analyzed.

18.3. The acceptance criteria for MS/MSD elements vary depending upon historical data. The lower and upper acceptance limits for %REC and RPD of each MS/MSD element are based upon the historical average recovery ± 3S ***that is updated at least annually***.

18.3.1. When the %REC and RPD of the MS/MSD elements are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.

18.3.2. If the %REC and/or RPD of the MS/MSD elements are not within the established acceptance limits, the analytical system performance shall be suspect.

18.4. The acceptance criteria for PDS elements are predetermined. The lower and upper acceptance limits for %REC of each PDS element are 85% and 115%, respectively.

18.4.1. If the %REC of the PDS element ***and the %REC of the MS/MSD elements*** are not within the established acceptance limits, ***matrix effects are confirmed. Perform MSA (see Section 14.10.) on all samples in the same preparation batch.***

18.5. Matrix effects or poor instrument performance/technique typically cause unacceptable %REC values. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.

18.6. Additional information regarding internal quality control checks is provided in SOP-T020.

18.7. All concentrations shall be reported in mg/kg (ppm) for soil and solid waste samples, ***and mg/kg (ppm) for tissue samples***.

18.7.1. ***To comply with client request or project specific DQOs, report all concentrations in mg/kg (ppm) on a dry-weight basis for soil and solid waste samples.***

18.8. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

19. CORRECTIVE ACTIONS

19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.

19.2. The Operations Manager, Project Manager, Quality Control Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.

19.3. Corrective actions are generally of two types, immediate and long-term actions.

19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.

19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented in the Corrective Action Record. Examples of this type of action include:

19.3.2.1. Remedial training of staff in technical skills, technique or implementation of operating procedures.

19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.

19.3.2.3. Revision of standard operating procedures.

19.3.2.4. Replacing personnel, as necessary.

19.4. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:

19.4.1. Define the problem.

19.4.2. Assign responsibility for investigating the problem.

19.4.3. Investigate and determine the cause of the problem.

19.4.4. Assign and accept responsibility for implementing the corrective action.

19.4.5. Determine effectiveness of the corrective action and implement correction.

19.4.6. Verify that the corrective action has eliminated the problem.

19.5. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

20. CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1. Out-of-control data are reviewed and verified by the technical director of the appropriate department. All samples associated with an unacceptable QC set are then subject to reanalysis, depending upon the QC type in question.

20.1.1. MS/MSD/PDS: Acceptability of the MS/MSD/PDS recoveries is subject to the matrix and any anomalies associated with the subject batch. Failure of recoveries of an MS/MSD/PDS data set does not constitute an automatic reanalysis of the batch samples. Rather, it is acceptable to defer to the LCS/LCSD recoveries, to determine acceptance of the sample results.

20.1.2. LCS/LCSD: Because they denote whether the analytical system is operating within control, it is imperative that the LCS recoveries obtained are within acceptability criteria. If the recoveries fail for a given reported element, the technical director confirms the unacceptable result.

20.1.2.1. If the LCS results are verified as acceptable, no corrective action is required.

20.1.2.2. If the LCS result is verified as out-of-control, and the subject element is to be reported in samples within that analytical batch, the samples reported with that failed element must be reanalyzed with a valid LCS recovery for the element.

20.1.2.3. If the LCS result is verified as out-of-control, and the subject element is NOT to be reported in the samples within that analytical batch, the samples are not subject to reanalysis. No corrective action is required for that batch.

21. WASTE MANAGEMENT

21.1. The proper disposal of analytical samples and laboratory wastes is not only good laboratory practice, but also regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated, and either returned to the client (preferable) or placed into the proper laboratory waste stream.

21.2. Unused or remaining soil or liquid samples and all other solid or liquid wastes resulting from our laboratory operations are considered hazardous for disposal purposes.

21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal.

21.4. Each specific laboratory area shall maintain clearly labeled waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste.

21.5. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly.

21.6. In order to maintain accountability for all samples received by Calscience, when a sample is used in its entirety for analysis, the empty container(s) are returned to Sample Control for placement in analytical storage.

21.7. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Wastes."

22. ►REFERENCES

22.1. *Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1A, Method 7471A, USEPA, Revision 1, September 1994.

22.2. *Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1A, Method 7471B, USEPA, Revision 2, ***February 2007***.

22.3. ***Flame Atomic Absorption Spectrophotometry, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1A, Method 7000B, USEPA, Revision 2, February 2007.***

22.4. *Quality Control*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter One, USEPA, Revision 1, July 1992.

22.5. *Choosing the Correct Procedure*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Two, USEPA, Revision 4, ***February 2007***.

22.6. *Inorganic Analytes*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Three, USEPA, Revision 4, ***February 2007***.

23. ►TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

23.1. ***Appendix A: Sample Preparation and Digestion Procedures.***

23.2. Appendix ***B***: Standard Addition Plot (Example).

23.3. Appendix ***C***: Additional Quality Control Criteria for ***Department of Defense*** Project.

23.4. ***Appendix D: Additional Quality Control Criteria for BP Project.***

24. ►MODIFICATIONS

24.1. ***The following modifications from EPA Method 7471A are noted.***

24.1.1. ***Reagents and Standards***

24.1.1.1. ***Standard preparations are modified.***

24.1.2. ***Quality Control***

24.1.2.1. ***The criteria of method blank are modified.***

24.2. ***The following modifications from EPA Method 7000B are noted.***

24.2.1. ***Quality Control***

24.2.1.1. ***The criteria of post digestion spike addition are modified.***

Appendix A

SAMPLE PREPARATION AND DIGESTION PROCEDURES

Calscience Environmental Laboratories, Inc.

1. METHOD IDENTIFICATION

1.1. EPA Method 7471A, Mercury in Solid or Semisolid Waste (Cold-Vapor Technique) – Sample Preparation and Digestion Procedures.

2. SCOPE AND APPLICATION

2.1. The procedure described herein is in addition to the standard procedure.

2.2. This method is restricted to use by or under the supervision of analysts/technicians experienced in the use of the equipments and apparatus required to execute the procedure.

3. METHOD SUMMARY

3.1. EPA Method 7471A provides digestion and cold-vapor atomic absorption conditions for the analysis of mercury in solid or semisolid waste.

4. INTERFERENCES

4.1. Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.

4.2. Samples high in chlorides require additional permanganate (as much as 25 mL) due to the fact that during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm.

5. SAFETY

5.1. Acids are corrosive. Many mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Extreme care must be exercised in the handling of acids and mercury standards.

5.2. All sample preparation activities must be performed in a fume hood vented to the exterior of the laboratory.

5.2.1. All operational fume hoods are to remain energized continuously in order to minimize acidic atmospheric or toxic gas buildup.

5.3. For the safety of the analyst, cracked or broken glassware should be immediately discarded into a broken glassware receptacle. Broken glassware shall not be used in any step of the digestion.

5.4. To ensure the safety of the analyst during any possible emergency situation, it is recommended that chemists do not perform digestions alone. Another chemist should be present during any digestion process.

5.5. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current

version of Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats are required to be worn in all designated laboratory areas. Protective gloves shall be worn when handling chemicals.

5.6. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

6. EQUIPMENT AND SUPPLIES

6.1. Digestion tubes (vials), 4-oz (120-mL), 90-mm × 43-mm ID, graduated, snap closure, with hinged lids, polypropylene, disposable, Capitol Vial P/N 04HPLS or equivalent.

6.2. Watch glass, ribbed or equivalent, glass, appropriate diameter to cover vial.

6.3. Dispensers, 1–10-mL, 2.5–25-mL, and 5–50-mL, calibrated, adjustable.

6.4. Thermometer, calibrated, capable of accurately measuring at 95°C.

6.5. Block digester, equipped with water bath, capable of maintaining 95°C.

6.6. Balance, top loading, calibrated, capable of weighing to the nearest 0.01 g.

6.7. Spatula, PTFE (preferred) or stainless-steel construction.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Reagent water, interferant free, nano-pure.

7.1.2. Chips, Teflon.

7.1.3. Beads, glass.

7.1.4. Diluted aqua regia solution, $HCl/HNO_3/H_2O$, 3:1:4 (v/v/v).

7.1.4.1. Prepare the diluted aqua regia solution by slowly adding 7.5 L of concentrated HCl and 2.5 L of concentrated HNO_3 to 8 L of reagent water and dilute to 20 L with additional reagent water.

7.1.4.2. The diluted aqua regia solution must be replaced after three months or sooner.

7.1.5. Sodium chloride, NaCl, fine white crystals, reagent grade or equivalent.

7.1.6. Hydroxylamine hydrochloride, $H_3NO{\cdot}HCl$, fine white crystalline powder, reagent grade or equivalent.

7.1.7. Sodium chloride-hydroxylamine hydrochloride solution, $NaCl\text{-}H_3NO{\cdot}HCl$.

7.1.7.1. Prepare the sodium chloride-hydroxylamine hydrochloride solution by dissolving 2400 g of NaCl and 2400 g of $H_3NO{\cdot}HCl$ in reagent water and dilute to 20 L with additional reagent water.

7.1.8. Potassium permanganate, $KMnO_4$, dark purple to green crystals, low mercury (≤ 0.05-ppm Hg), reagent grade or equivalent.

7.1.9. Potassium permanganate, $KMnO_4$, 5% (w/v).

7.1.9.1. Prepare the 5% $KMnO_4$ solution by dissolving 1000 g of $KMnO_4$ in 20 L of reagent water.

7.1.10. All reagents must be inspected and documented in the Chemicals and Supplies Verification Logbook prior to use.

7.2. Standards

7.2.1. Stock Standard Solution

7.2.1.1. Pre-certified stock standard solution, 99.990–99.999% source purity, in sealed polyethylene bottle, containing 1000 ppm of mercury is used to prepare check standards.

7.2.1.2. Prepare the 2.0-ppm mercury working standard solution by diluting 0.2 mL of the mercury stock standard and 5 mL of concentrated HNO_3 to 100 mL with reagent water.

7.2.2. Spike Standard Solution

7.2.2.1. Use the 2.0-ppm mercury working standard solution as the spike standard solution.

7.2.2.2. The spike standard is used to prepare QC check samples such as matrix spikes (MS/MSDs), post digestion spikes (PDSs), and laboratory control samples (LCS/LCSDs).

7.2.2.3. Add 250 µL of the spike standard to each 0.6-g aliquot of solid MS/MSD and LCS/LCSD sample prior to digestion.

7.2.2.4. Add 25 µL of the spike standard to each 10-mL aliquot of PDS sample after digestion.

7.2.3. Unless specified otherwise, all working standards must be replaced after one month or sooner if comparison with check standards indicates a problem.

7.2.4. All stock standards must be inspected and documented in the Chemicals and Supplies Verification Logbook prior to use.

8. CALIBRATION AND STANDARDIZATION

8.1. Top Loading Balance

8.1.1. Calibrate the top loading balance at 1 g and 100 g using Class 2 weights.

8.1.2. Calibration shall be within ± 2% or ± 0.02 g, whichever is greater. If the values are not within these limits, recalibrate the balance.

8.2. Thermometer

8.2.1. Calibrate the thermometer using an NIST certified thermometer. The calibration procedure shall adhere to the current revision of SOP-T043, "Support Equipment – Calibration, Verification, Monitoring."

8.3. Pipetter

8.3.1. Calibrate the pipetter according to the procedure outlined in the current revision of SOP-T043, "Support Equipment – Calibration, Verification, Monitoring."

8.4. Dispenser

8.4.1. Calibrate the dispenser according to the procedure outlined in the current revision of SOP-T043, "Support Equipment – Calibration, Verification, Monitoring."

9. PROCEDURE

9.1. Solid Sample Preparation

9.1.1. Homogenize a solid, soil, or sediment sample as outlined in the current revision of SOP-M230.

9.1.2. Measure triplicate 0.20-g (wet weight) aliquots (i.e., 0.60-g total) of the homogenized solid sample into a clean digestion tube. Record the mass of sample used to the nearest 0.01 g.

9.1.2.1. For MB/LCS/LCSD, measure 0.60 ± 0.03 g of clean Teflon chips or glass beads. Record the Teflon chip or glass bead identification number.

9.1.2.2. For MS/MSD, measure 0.60 ± 0.03 g of solid sample in each analytical batch selected for spiking.

9.1.3. Add 250 µL of the spike standard solution to all matrix spikes and laboratory control samples.

9.1.4. Proceed to Section 9.6. for digestion procedure.

9.2. Tissue Sample Preparation

9.2.1. Homogenize marine or freshwater tissue sample as outlined in the current revision of SOP-M229.

9.2.2. Measure 1.00 ± 0.05 g (wet weight) of the homogenized tissue sample into a clean digestion tube. Record the mass of sample used to the nearest 0.01 g.

9.2.2.1. For MB/LCS/LCSD, measure 1.00 ± 0.05 g of clean Teflon chips or glass beads. Record the Teflon chip or glass bead identification number.

9.2.2.2. For MS/MSD, measure 1.00 ± 0.05 g of tissue sample in each analytical batch selected for spiking.

9.2.3. Add 250 µL of the spike standard solution to all matrix spikes and laboratory control samples.

9.2.4. Proceed to Section 9.6. for digestion procedure.

9.3. CRQL Check Standard Preparation

9.3.1. Measure 12.5 µL of the 2.0-ppm mercury working standard solution into a clean digestion tube.

9.3.1.1. For lower limit of quantitation, measure 2.5 µL of the 1.0-ppm mercury working standard solution.

9.3.2. Proceed to Section 9.6. for digestion procedure.

9.4. Solid LLOQ Check Sample Preparation

9.4.1. Measure 12.5 µL of the 2.0-ppm mercury working standard solution and 0.60 ± 0.03 g of Teflon chips or glass beads into a clean digestion tube. Record the Teflon chip or glass bead identification number.

9.4.2. Proceed to Section 9.6. for digestion procedure.

9.5. Tissue LLOQ Check Sample Preparation

9.5.1. Measure 12.5 µL of the 2.0-ppm mercury working standard solution and 1.00 ± 0.05 g of Teflon chips or glass beads into a clean digestion tube. Record the Teflon chip or glass bead identification number.

9.5.2. Proceed to Section 9.6. for digestion procedure.

9.6. Digestion

9.6.1. Add 10 mL of the diluted aqua regia solution to the digestion tube.

9.6.2. Place the digestion tube in the pre-heated block digester, cover the digestion tube with a clean watch glass, and heat for 2 minutes in the water bath maintained at 95°C.

9.6.3. Remove the digestion tube from the block digester and allow the digestate to cool.

9.6.4. Add 50 mL of reagent water and 15 mL of the 5% $KMnO_4$ solution to the digestion tube.

9.6.5. Add additional portions of the 5% $KMnO_4$ solution to the digestion tube, if necessary, until the purple color persists for at least 15 minutes.

9.6.5.1. Ensure that equal amounts of permanganate are added to standards and blanks.

9.6.6. Mix the contents of the digestion tube thoroughly.

9.6.7. Place the digestion tube in the pre-heated block digester, cover the digestion tube with the same watch glass, and continue to heat for 30 minutes in the water bath maintained at 95°C.

9.6.8. Remove the digestion tube from the block digester and allow the digestate to cool.

9.6.9. Add 6 mL of the sodium chloride-hydroxylamine hydrochloride solution to the digestate to reduce excess permanganate.

9.6.10. Adjust the final volume of the digestate to 100 mL with calibration blank.

9.6.11. Transfer a sufficient volume of the digestate to an autosampler vessel and label appropriately. The digestate may now be analyzed.

9.6.11.1. For post digestion spike addition, add 25 µL of the spike standard to 10.0 mL of digestate designated as PDS.

9.7. Thoroughly document all aspects of the digestion in the Mercury Sample Preparation Logbook. This logbook includes, but is not limited to:

9.7.1. Digestion date, start time, and finish time.

9.7.1.1. The start and stop time of each sample digestion must be accurately recorded in the logbook. If all samples were started and finished at the same time, then an 'arrow down' approach may be used in the logbook. If a batch is open, and a sample is added on after the digestion process of the other batch samples has begun, then the actual start and stop time for each additional sample must be recorded in the logbook.

9.7.1.2. All samples must undergo the entire digestion process regardless when the digestion process was started.

9.7.2. Sample matrix, initial volume, and final volume.

9.7.3. Digestion temperature.

9.7.4. Reagent and supply lot (or identification) numbers.

9.7.5. Standard lot (or identification) number, concentration, and volume added.

9.7.6. Analyst comments which include encountered problems, pertinent observations, or conditions that could potentially impact data quality.

10. MODIFICATIONS

10.1. The following modifications from EPA Method 7471A are noted.

10.1.1. Reagents and Standards

10.1.1.1. Reagents and standard preparations are modified.

10.1.1.2. Aqua regia preparation and holding time are modified.

10.1.2. Procedure

10.1.2.1. The final volume of the digestate is modified.

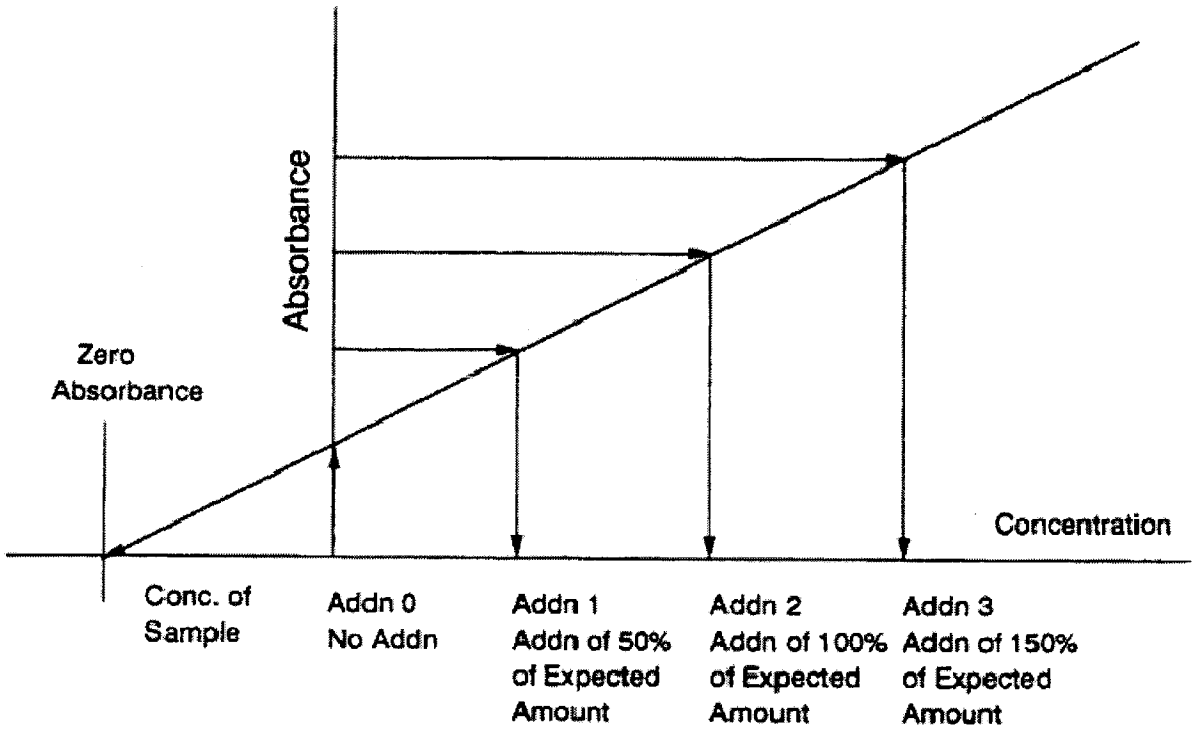
Appendix B

STANDARD ADDITION PLOT (EXAMPLE)

Calscience Environmental Laboratories, Inc.

Appendix B

Standard Addition Plot (Example)



Appendix C

ADDITIONAL QUALITY CONTROL CRITERIA FOR DEPARTMENT OF DEFENSE PROJECT

Calscience Environmental Laboratories, Inc.

1. METHOD IDENTIFICATION

1.1. ►EPA Method 7471A, Mercury in Solid or Semisolid Waste (Cold-Vapor Technique) – Additional Quality Control Criteria for ***Department of Defense (DoD)*** Project.

2. ►DETECTION LIMITS

2.1. ***The quantitation limit must be set within the calibration range.***

3. SCOPE AND APPLICATION

3.1. The quality control criteria and procedure described herein either supersede or are in addition to the standard quality control criteria and procedure.

4. STANDARDS

4.1. ►***The spike standard solutions shall contain all anticipated target analytes.***

4.2. The use of a standard from a second lot as the second source standard is acceptable when only one manufacturer of the calibration standard exists. "Manufacturer" refers to the producer of the standard, not the vendor.

5. ►QUALITY CONTROL

5.1. Method Detection Limit (MDL)

5.1.1. MDL study shall be performed ***at the initial test method setup, following a change in the test method that affects how the test is performed, or when a change in instrumentation that affects the sensitivity of the analysis***.

5.1.2. MDL verification must be performed immediately following an MDL study ***and quarterly thereafter***.

5.1.2.1. ***MDL verification sample shall be prepared by spiking an appropriate matrix at approximately 2–3 times the detection limit for single-analyte test, or 1–4 times the detection limit for multiple-analyte test.***

5.1.2.2. ***MDL verification is deemed valid if the apparent signal to noise ratio of each analyte is at least 3 and the results must meet all method requirements for analyte identification.***

5.1.2.3. If these criteria are not met, ***perform either one of the following tasks***.

5.1.2.3.1. ***Repeat the MDL study and verification at a higher concentration. Set the MDL at the higher concentration.***

5.1.2.3.2. ***Perform and pass 2 consecutive MDL verifications at a higher concentration. Set the MDL at the higher concentration.***

5.1.3. No samples shall be analyzed without a valid MDL.

5.2. Initial Calibration Blank (ICB)

5.2.1. The instrument operating condition is deemed satisfactory for sample analysis to begin if no analytes are detected at a concentration > ***MDL***.

5.2.2. If these criteria are not met, no sample analysis shall begin. Determine the source of contamination. Re-prepare and reanalyze the ICB.

5.3. ***Continuing Calibration Verification (CCV)***

5.3.1. ***The concentration of the CCV standard shall be between the low point and the midpoint of the calibration range.***

5.4. Continuing Calibration Blank (CCB)

5.4.1. The instrument operating condition is deemed satisfactory for sample analysis to resume if no analytes are detected at a concentration > ***MDL***.

5.4.2. If these criteria are not met, no sample analysis shall resume. Determine the source of contamination. Re-prepare and reanalyze the CCB. Reanalyze all samples since the last acceptable calibration blank.

5.4.2.1. The results shall be reported with the appropriate data qualifier (B-flag) for the specific analyte(s) in all samples associated with the CCB.

5.5. Event Based Quality Control (LCS/LCSDs and MBs)

5.5.1. Laboratory Control Samples (LCS/LCSDs)

5.5.1.1. The lower and upper acceptance limits for %REC of each LCS/LCSD element in solid matrix are 80% and 120%, respectively.

5.5.1.2. ***Project-specific control limits shall be applied. If project-specific control limits are unavailable, DoD generated control limits shall be applied. If DoD generated control limits are unavailable, laboratory's in-house control limits shall be applied.***

5.5.1.2.1. ***Laboratory's in-house control limits may not be greater than ± 3S of the average recovery.***

5.5.1.3. ***All project-specific analytes of concern must be within control limits. If a project-specific analyte of concern exceeds its control limit, determine the cause of the problem and effect corrective action.***

5.5.2. Method Blanks (MBs)

5.5.2.1. ***The MB is considered to be contaminated if one of the following conditions is met.***

5.5.2.1.1. The concentration of any target analyte in the MB ***exceeds 1/2 the RL***, and is greater than 1/10 the amount measured in any sample ***or 1/10 the regulatory limit (whichever is greater)***.

5.5.2.1.2. ***The concentration of any common laboratory contaminant in the MB exceeds RL, and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).***

5.5.2.1.3. The MB result otherwise affects the sample results as per the test method requirements or the project specific data quality objectives (DQOs).

5.5.2.2. ***If the MB is contaminated, reprocess the samples associated with the failed MB in a subsequent preparation batch, except when the sample results are below the MDL.***

5.5.2.2.1. If ***insufficient*** sample volume remains for reprocessing, the results shall be reported with the appropriate data qualifier (B-flag) for the specific analyte(s) in all samples associated with the failed MB.

5.6. Matrix Based Quality Control (MS/MSDs)

5.6.1. Matrix Spikes (MS/MSDs)

5.6.1.1. The lower and upper acceptance limits for %REC of each MS/MSD element in solid matrix are 80% and 120%, respectively. ***The RPD is ≤ 20%.***

5.6.1.2. ***Project-specific control limits shall be applied. If project-specific control limits are unavailable, DoD generated control limits shall be applied. If DoD generated control limits are unavailable, laboratory's in-house control limits shall be applied.***

5.6.1.2.1. ***Laboratory's in-house control limits may not be greater than ± 3S of the average recovery.***

6. ►PROCEDURE

6.1. ***Standard and sample vessels are loaded in the following or other logical order:***

1) ***Calibration Blank (CB)***
2) ***Initial Calibration Standards***

3) ***Initial Calibration Verification (ICV)***
4) ***Initial Calibration Blank (ICB)***
5) ***Method Blank (MB)***
6) ***Laboratory Control Samples (LCS)***
7) ***Laboratory Control Sample Duplicates (LCSD)***
8) ***Samples (up to 10 per batch, including QC check samples and MBs)***
9) ***Continuing Calibration Verification (CCV)***
10) ***Continuing Calibration Blank (CCB)***
11) ***Matrix Spike (MS)***
12) ***Matrix Spike Duplicate (MSD)***
13) ***Samples (up to 10 per batch, including QC check samples and MBs)***
14) ***Ending CCV***
15) ***Ending CCB***

6.1.1. ***Item 11: The MS is the actual sample matrix spiked with known concentration of specific target analyte. The sample which is spiked for the MS is processed concurrently with the associated samples. In the processing of the MS, reagents and procedures identical to those for actual samples are used.***

6.1.1.1. ***The sample selected for spiking must be one of the samples collected for the specific DoD project.***

6.1.2. ***Item 12: The MSD is handled identically to the MS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the MS in combination with the MSD can be used to assess the precision of the analytical measurements. The measurement is expressed as relative percent difference (RPD).***

7. REFERENCES

7.1. ►Department of Defense Quality Systems Manuals for Environmental Laboratories, Version ***4.2, October 25, 2010***.

Appendix D

ADDITIONAL QUALITY CONTROL CRITERIA FOR BP PROJECT

Calscience Environmental Laboratories, Inc.

1. METHOD IDENTIFICATION

1.1. EPA Method 7471A, Mercury in Solid or Semisolid Waste (Cold-Vapor Technique) – Additional Quality Control Criteria for BP Project.

2. SCOPE AND APPLICATION

2.1. The quality control criteria and procedure described herein either supersede or are in addition to the standard quality control criteria and procedure.

3. STANDARDS

3.1. The spike standard solutions shall contain all anticipated target analytes.

3.2. The use of a standard from a second lot as the second source standard is acceptable if written warranties demonstrating that the standards from two different lots are not prepared from the same reference materials are obtained from the manufacturer. "Manufacturer" refers to the producer of the standard, not the vendor.

4. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

4.1. Holding time begins at the date and time the sample is collected as documented on the chain of custody record.

5. QUALITY CONTROL

5.1. Method Detection Limit (MDL)

5.1.1. MDL study should be performed annually for each analyte and matrix.

5.1.2. MDL verification must be performed immediately following an MDL study.

5.1.2.1. MDL verification sample must be spiked at the calculated MDL value.

5.1.2.2. MDL verification is deemed valid if all analytes in the verification sample are recovered.

5.1.3. At a minimum, the MDL study shall be performed on a single instrument, and the calculated MDL value must be confirmed by analyzing an MDL verification sample on all instruments used to analyze BP samples.

5.2. Initial Calibration Blank (ICB)

5.2.1. The absolute value of the concentration of a target analyte in an ICB shall be $\leq 2 \times$ MDL.

5.3. Continuing Calibration Verification (CCV)

5.3.1. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily after every 10 analyses or portion thereof, and at the end of sequence.

5.3.2. Documentation for multiple (more than one) injection trials of CCV standards must include the reason for the multiple trials, the corrective action and which trial was used and why.

5.4. Continuing Calibration Blank (CCB)

5.4.1. The absolute value of the concentration of a target analyte in a CCB shall be ≤ 2 × MDL.

5.5. Low-Level Reporting Limit (LLRL) Check

5.5.1. An LLRL check standard must be analyzed daily prior to sample analysis, every 8 hours thereafter during analysis, and at the end of sequence.

5.5.1.1. The concentration of each analyte in the LLRL check solution shall be within 2 × RL.

5.5.2. The low-level reporting limit is validated if the recovery of each analyte is within 50−150%.

5.6. Event Based Quality Control (LCS/LCSDs and MBs)

5.6.1. Method Blanks (MBs)

5.6.1.1. Contamination observed in the MB at a concentration greater than 1/2 of the RL is acceptable if either one of the following conditions is met.

5.6.1.1.1. No contaminant is detected in the associated samples.

5.6.1.1.2. The concentration of the contaminant in the associated samples is greater than 10 times the concentration in the MB.

5.7. Multiple Integrations

5.7.1. Evaluation of %RSD between multiple integrations (replicates) for each sample analysis is required.

5.7.2. Sample with analyte concentration greater than the RL must be reanalyzed if the %RSD is > 20%.

6. PROCEDURE

6.1. All solid samples must undergo a documented thorough sample homogenization (i.e. formal cone and quartering) using inert utensils/mixing platforms that will not interfere with the target analytes.

6.2. Following the establishment of a valid initial calibration, a CCV standard and CCB must be analyzed daily after every 10 analyses or portion thereof, and at the end of

sequence. If the QC criteria are met, the initial calibration is assumed to be valid and sample analysis may resume.

6.2.1. If a failed CCB is the first of the day, and the concentration of a target analyte in a sample bracketed by the unacceptable ICB or CCB is less than or equal to 10 times the absolute value of the concentration of the target analyte in the ICB or CCB, reanalyze the sample.

6.2.2. If a failed CCB is not the first of the day, and the concentration of a target analyte in a sample bracketed by the unacceptable CCB is less than or equal to 10 times the absolute value of the concentration of the target analyte in the CCB, reanalyze the sample.

6.3. Standard and sample vessels are loaded in the following or other logical order:

1) Calibration Blank (CB)
2) Initial Calibration Standards
3) Initial Calibration Verification (ICV)
4) Initial Calibration Blank (ICB)
5) LLRL Check
6) Method Blank (MB)
7) Laboratory Control Samples (LCS)
8) Laboratory Control Sample Duplicates (LCSD)
9) Samples (up to 10 analyses, excluding ICV or CCV analyses)
10) Continuing Calibration Verification (CCV)
11) Continuing Calibration Blank (CCB)
12) Matrix Spike (MS)
13) Matrix Spike Duplicate (MSD)
14) Samples (up to 10 analyses, excluding CCV analyses)
15) Ending CCV
16) Ending CCB

6.3.1. Item 5: The LLRL check standard is used to validate the low-level reporting limit. An acceptable LLRL check is required daily prior to sample analysis, every 8 hours thereafter during analysis, and at the end of sequence.

6.3.2. Item 6: The MB is a known matrix similar to the samples being analyzed which is processed concurrently with the associated samples. In the processing of the MB, reagents and procedures identical to those for actual samples are used.

6.3.2.1. One MB is required every day preparatory methods (i.e., leachings, filtrations, digestions, etc.) are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent, within an 8-hour preparation event.

6.3.3. Item 7: The LCS is a known matrix which has been spiked with known concentration of specific target analyte. The purpose of the LCS is to demonstrate that the entire analytical process and systems are in control. The LCS is processed concurrently with the associated samples. In the

processing of the LCS, reagents and procedures identical to those for actual samples are used.

6.3.3.1. One LCS is required every day preparatory methods (i.e., leachings, filtrations, digestions, etc.) are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent, within an 8-hour preparation event.

6.3.4. Item 8: The LCSD is handled identically to the LCS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the LCS in combination with the LCSD can be used to assess the precision of the analytical process. The measurement is expressed as relative percent difference (RPD).

6.3.5. Item 12: The MS is the actual sample matrix spiked with known concentration of specific target analyte. The sample which is spiked for the MS is processed concurrently with the associated samples. In the processing of the MS, reagents and procedures identical to those for actual samples are used.

6.3.5.1. One MS is required for every batch of 20 samples per matrix or portion thereof processed concurrently within an 8-hour preparation event.

6.3.5.2. The sample selected for spiking must be one of the samples collected for the specific BP project. If 10 or fewer BP samples are received, and no sample is designated for MS/MSD analyses, then a non-BP sample may be used.

6.3.5.3. Samples identified as trip or field blanks cannot be used for MS/MSD.

6.3.6. Item 13: The MSD is handled identically to the MS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the MS in combination with the MSD can be used to assess the precision of the analytical measurements. The measurement is expressed as relative percent difference (RPD).

7. REFERENCES

7.1. Technical Requirements for Environmental Laboratory Analytical Services, BP Laboratory Management Program (LaMP), Revision 09, July 30, 2007.

Title: **EPA METHOD 7196A, Hexavalent Chromium (Colorimetric)**

Document No. : SOP-M716
Revision No. : 1.0
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APPROVED FOR RELEASE BY: _____ 05/21/03
MANAGEMENT **DATE**

_____ 05/21/03
QA DEPARTMENT **DATE**

1. METHOD IDENTIFICATION

1.1. Method 7196A, Hexavalent Chromium (Colorimetric)

2. APPLICABLE MATRICES

2.1. This method may be applied to drinking water, ground water, domestic and industrial wastes, digestates, or extracts.

3. DETECTION LIMITS

3.1. The estimated quantitation limits EQLs for this method are approximately 0.2 mg/kg (wet weight) for soil or solid waste and 0.005 mg/L for aqueous samples. The EQLs will be proportionally higher for samples that require dilution.

4. SCOPE AND APPLICATION

4.1. This method includes the measurement of dissolved hexavalent chromium in drinking water, ground water, domestic and industrial wastes, digestates, or extracts.

4.2. The method is applicable to measurement of dissolved hexavalent chromium above 0.02mg/l.

5. METHOD SUMMARY

5.1. Dissolved hexavalent chromium is determined colorimetrically by its reaction with 1,5-diphenylcarbazide in an acid solution, which produces a red-violet color. The reaction is very sensitive, the absorbancy index per gram atom of chromium being approximately 40,000 @ 540nm. Addition of excess 1,5-diphenylcarbazide yields the red-violet color and its absorbance is measured @ 540nm.

5.2. For soil samples, prior to performing this procedure, the appropriate sample preparation technique must be performed on each sample. Acceptable preparatory methods include the following:

Type of Sample Preparation	EPA Method No.	SOP No.
Alkaline Digestion	3060A	SOP-M224
TCLP	1311	SOP-M226
SPLP	1312	
WET	CAC, T22, §66261.24, App.2	

6. DEFINITIONS

6.1. Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

6.2. Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

6.3. 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 NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

6.4. Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

6.5. Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

6.6. Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

6.7. Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

6.8. Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

6.9. Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.

6.10. Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

6.11. Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

6.12. Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

6.13. Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

6.14. Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

6.15. Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

6.16. Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

6.17. Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.

6.18. 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.

6.19. 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.

6.20. Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence.

6.21. Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

6.22. Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

6.23. Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

7. INTERFERENCES

7.1. The hexavalent chromium reaction with 1,5-diphenylcarbazide is usually free from interferences. However, certain substances may produce significant interference if the hexavalent chromium concentration is low. Hexavalent molybdenum and mercury salts also react to form color with 1,5-diphenylcarbazide. However, the red-violet intensities produced are much lower than those for hexavalent chromium at pH=2. Concentrations of up to 200 mg/l of molybdenum and mercury can be tolerated. Vanadium interferes strongly with concentrations exceeding 10 times the hexavalent chromium concentration.

7.2. Iron in concentrations greater than 1 mg/L may produce a yellow color, but the color is not intense and does not pose difficulty @ 540nm.

8. SAFETY

8.1. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current version of Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats are required to be worn in all designated laboratory areas. Protective gloves shall be worn when handling chemicals.

8.2. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

8.3. Hexavalent chromium has been tentatively classified as known or suspected human carcinogen.

9. EQUIPMENT AND SUPPLIES

9.1. HP Model 8453 Diode Array Spectrophotometer or equivalent

9.2. Analytical balance, capable of weighing to the nearest 0.0001g

9.3. Drying oven

9.4. Volumetric flasks, 100ml and 1000ml

9.5. Volumetric pipettes, 10ml

9.6. Graduated cylinder, 50ml

10. REAGENTS AND STANDARDS

10.1. REAGENTS

10.1.1. Reagent Water: Distilled or deionized water

10.1.2. Sulfuric Acid, 10%(v/v), reagent or spectro grade: Dilute 10ml of H_2SO_4 to 100ml with reagent water

10.1.3. 1,5-Diphenylcarbazide Solution: Dissolve 250mg of 1,5-diphenylcarbazide into 50ml of acetone. Store in an amber container. Discard upon any sign of discoloration.

10.2. STANDARDS

10.2.1. Potassium Dichromate Stock Solution: Dissolve 141.1mg of dried reagent grade $K_2Cr_2O_7$ in reagent water and dilute to 1 liter (1mL = 50µg Cr).

10.2.2. Potassium Dichromate Standard Solution: Dilute 0.5ml, 1.0ml, and 2.0ml of the stock solution (Section 10.2.1 above) to 100ml to prepare 0.25 mg/l (ppm), 0.50ppm, and 1.00ppm standards.

10.2.3. Second Source (ICV/LCS/LCSD) Standards: A midrange standard (0.50ppm) prepared from a source (manufacturer) which differs from that of the stock standard solution.

11. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

11.1. For most accurate analyses, sampling containers should be free of metallic contaminants. Samples can be collect-ed in plastic or glass containers.

11.2. Samples should be kept cool at >0 °C to ≤6°C without preservative.

11.3. For water samples or extracts, a holding time of 24 hours applies. For extracts, the holding time commences at the completion of the extraction process. For digestates, the holding time is seven (7) days and commences at the completion of the digestion process.

12. QUALITY CONTROL

12.1. Matrix-Based Quality Control (MS/MSDs)

12.1.1. Matrix-based quality control consists of QC samples prepared and processed using actual environmental samples. This consists of a matrix spike and matrix spike duplicate (MS/MSD).

12.1.2. The acceptance criteria for MS/MSDs are as follows:

12.1.2.1. When the %REC and RPD of the MS/MSD are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method

for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.

12.1.2.2. If the %REC and/or RPD of the MS/MSD compound is not within the established acceptance limits, the analytical system performance shall be suspect.

12.1.2.2.1. Unacceptable %REC values are typically caused by matrix effects or poor instrument performance/technique. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.

12.1.2.2.2. If the %REC or RPD of the MS/MSD and LCS/LCSD are unacceptable, all associated sample data must be invalidated and all associated samples re-analyzed.

12.2. Event-Based Quality Control (LCS/LCSDs, CCBs, and MBs)

12.2.1. Event-based quality control consists of QC samples prepared and processed with each preparatory event. This consists of a laboratory control sample and laboratory control sample duplicate (LCS/LCSD) and a method blank (MB).

12.2.2. The acceptance criteria for the LCS/LCSD vary depending upon historical data. The upper and lower acceptance limits for %REC and RPD of the LCS/LCSD are the historical average recovery ±3S. The LCS/LCSD must be within acceptance limits. If the LCS/LCSD is not acceptable, the problem must be identified and corrected.

12.2.2.1. If the LCS and/or LCSD %REC is outside of the acceptance limits high, the RPD is within acceptance limits, and the target analyte in the associated samples is not detected, the sample data can be reported without qualification.

12.2.2.2. The LCSD is only reported when the MS/MSD is unacceptable due to matrix interference effects, or when the LCS/LCSD is used in place of MS/MSD due to insufficient sample quantity.

12.2.3. Ideally, the concentration of the target analyte in a method blank (MB) should be less than its reporting limit (RL). If the concentration of the target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs is as follows:

12.2.3.1. If the target analyte is found in the MB, but not in the associated samples, report the sample and MB without qualification.

12.2.3.2. If the target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data

should be qualified, or rejected and the samples re-extracted and re-analyzed.

12.3. Verification

12.3.1. For every sample matrix analyzed, verification is required to ensure that neither a reducing condition nor chemical interference is affecting color development. This must be accomplished by analyzing a second 25-ml aliquot of the pH-adjusted filtrate that has been spiked with Cr(VI). The amount of spike added should double the concentration found in the original aliquot. Under no circumstances should the increase be less than 30 ug Cr(VI) per liter. To verify the absence of interferences, the spike recovery must be between 85% and 115%.

12.3.2. If addition of the spike extends the concentration beyond the calibration curve, the analysis (verification) solution should be diluted with blank solution and the calculated results adjusted accordingly.

12.3.3. If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed.

12.3.4. If the interference persists after sample dilution, report LCS/LCSD.

12.4. Analyze all extracts and all samples that suffer from matrix interferences by the method of standard additions (section 14.12 of this SOP).

12.5. Additional information regarding internal quality control checks is provided in SOP-T020.

13. CALIBRATION AND STANDARDIZATION

13.1. Initial Calibration

13.1.1. An acceptable initial calibration must be established prior to the processing of samples.

13.1.2. The initial three-point calibration curve is assumed to be valid if the RSD ≤20%. If this criterion is not met, then the calibration is unacceptable for sample analysis to begin. Effect corrective action and re-calibrate.

13.1.3. The initial calibration verification (ICV) is deemed acceptable if the %D ≤15%. If this criterion is not met, the initial calibration is deemed unacceptable for sample analysis to begin. An unacceptable ICV result indicates a disagreement between like solutions from separate sources. This is usually caused when at least one of the solutions is no longer intact (representative of the stated concentration). Investigate and effect corrective action. Recalibrate, if necessary.

13.2. Continuing Calibration Blank

13.2.1. Following establishment of a valid initial calibration, a continuing calibration blank (CCB) must be analyzed prior to every batch of samples or portion thereof and at the end of every sequence. The acceptance criteria for CCBs are ≤MDL where MDL represents the current MDL value for the target analyte. It the CCB fails, it should be re-analyzed. If it fails again, the analytical system is considered out of

control and the cause must be determined and corrected prior to analysis of samples.

13.3. Continuing Calibration Verification

13.3.1. Following establishment of a valid initial calibration, a calibration verification (CV) standard must be analyzed prior to every batch of 20 samples or portion thereof and at the end of every sequence. If the %D ≤15% for the analyte in the CV, then the calibration is assumed to be valid and sample analysis may continue. If this criterion is not met, the CV is deemed unacceptable. Reanalyze the CV. If the %D remains unacceptable, effect corrective action and reanalyze the CV and/or recalibrate.

14. PROCEDURE

14.1. Prior to the analysis of samples, a valid initial three-point calibration curve must be established. The concentrations of the standards are 0.1, 0.50, and 1.00ppm Cr(VI). The acceptance criteria are listed below in Section 12, Quality Control.

14.2. To verify the calibration, after obtaining an acceptable three-point calibration curve and prior to processing samples, an initial calibration verification (ICV) must be analyzed to verify the initial calibration standards. The ICV shall be at mid-level concentration and shall be of a source other than that of the initial calibration. The acceptance criteria for the ICV are listed below in Section 12, Quality Control.

14.3. Satisfactory instrument baseline is continually assured through the analysis of continuing calibration blanks (CCBs). CCBs are reagent water analyzed directly without any preparatory steps. The required frequency of CCBs is at the beginning of every sequence, every 20 samples or portion thereof, and at the end of every sequence. The acceptance criteria for CCBs are listed below in Section 12, Quality Control.

14.4. Daily calibration is established by the analysis of a calibration verification (CV) standard. The CV standard is the midpoint concentration of the same standard used for initial calibration of the target analyte. If the CV is acceptable, then the calibration is assumed to be valid and sample analysis may continue. The required frequency of CVs is at the beginning of every sequence, every 20 samples or portion thereof, and at the end of every sequence. The acceptance criteria are listed in Section 12, Quality Control.

14.5. Transfer 95ml of the sample into a 100ml volumetric flask.

14.6. Add 2ml of 1,5-diphenylcarbazide solution and mix by inversion.

14.7. Add a few drops of 10% H_2SO_4, dilute to volume with reagent water, mix by inversion, and allow 5 – 10 minutes for full color development.

Note: If the sample is turbid, measure sample concentration without adding 1,5-diphenylcarbazide and subtract the concentration reading from the sample concentration measured previously.

14.8. The blanks, standards, and samples are analyzed using the UV/VIS spectrophotometer by transferring solution into a cell and measuring the absorbance at 540nm.

14.9. The samples are analyzed in the following order.

1. Method Blank (MB)
2. CCB
3. CV
4. Laboratory Control Sample (LCS)
5. Laboratory Control Sample Duplicate (LCSD)
6. Samples (up to 20 per batch)
7. Matrix Spike (MS)
8. Matrix Spike Duplicate (MSD)
9. Ending CCB
10. Ending CCV

14.9.1. Item 1: The MB is a known matrix similar to the samples being analyzed which is processed concurrently with the associated samples. In the processing of the MB, reagents and procedures identical to those for actual samples are used. The MB consists of reagent water.

14.9.1.1. One MB is required for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.

14.9.2. Item 2: A CCB is used to verify the acceptability of instrument baseline and to isolate sources of contamination in the MB that may occur. An acceptable CCB is required prior to sample analysis, every 20 samples thereafter, and at the end of every sequence.

14.9.3. Item 3: A CV is used to verify the acceptance of the initial three point calibration on a continuing basis. An acceptable CV is required prior to sample analysis, every 20 samples thereafter, and at the end of every sequence.

14.9.3.1. More frequent (e.g., every 10 samples) calibration verification may be useful to minimize the number of sample extract re-analyses that would be required in the event of an unacceptable CV.

14.9.4. Item 4: A LCS is a known matrix that has been spiked with a known concentration of the target analyte. The purpose of the LCS is to demonstrate that the entire analytical process and systems are in control. The LCS is processed concurrently with the associated samples. In the processing of the LCS, reagents and procedures identical to those for actual samples are used. The LCS consists of the target analyte spiked into reagent water.

14.9.4.1. A LCS is required for every batch of 20 samples per matrix or portion thereof whichever is more frequent.

14.9.5. Item 5: The LCSD is handled identically to the LCS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the LCS in combination with the LCSD can be used to assess the precision of the analytical process expressed as relative percent difference (RPD).

14.9.5.1. One LCSD is required for every batch of 20 samples per matrix or portion thereof.

14.9.6. Item 6: Up to 20 samples per batch. Please note that the samples should be sufficiently diluted such that the absorbance is within that of the high and low standard. In diluting or performing cleanups, it should be understood that increased reporting limits may result.

14.9.7. Item 7: The MS is the actual matrix spiked with a known concentration of the target analyte. The sample that is spiked for the MS is processed concurrently with the associated samples. In the processing of the MS, reagents and procedures identical to those for actual samples are used.

14.9.7.1. The purpose of a MS is to assess the effect of a sample matrix on the recovery of the target analyte (i.e., assess the accuracy of the analytical measurements of the matrix). The measurement is expressed as percent recovery (%REC) of the spiked compound.

14.9.7.2. One MS is required for every batch of 20 samples per matrix or portion thereof analyzed concurrently. This approach is considered "closed batch" as opposed to "open batch".

14.9.8. Item 8: The MSD is handled identically to the MS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the MS in combination with the MSD can be used to assess the precision of the analytical measurements. The measurement is expressed as relative percent difference (RPD).

14.9.9. Item 9: See Section14.9.2. An acceptable CCB is required at the completion of every analytical sequence.

14.9.10. Item 10: See Section 14.9.3. An acceptable CV is required at the completion of every analytical sequence.

14.10. The instrument data system displays the concentration readings. Print the data and record the pertinent information in the run logbook.

14.11. Data Interpretation

14.11.1. Instrument detector response is in absorbance converted to concentration. If the concentration of any sample exceeds that associated with the highest standard, then appropriate dilution and re-analysis of the sample must be performed.

14.11.2. Quantitation of the target analyte is based on a reproducible response of the detector within the calibration range and a direct proportionality of the absorbance readings in the sample and the calibration standards.

14.11.2.1. Proper quantitation requires the appropriate selection of a wavelength from which the absorbance of the target analyte expressed as concentration can be determined.

14.11.2.2. Determine the concentration based on the initial calibration curve.

14.11.2.3. If the instrument response exceeds the calibration range, dilute the sample and reanalyze or a smaller sample aliquot should be used prepare the sample for Cr(VI) determination.

14.12. Method of Standard Additions (MSA)

14.12.1. The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

14.12.1.1. The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_s of a standard analyte solution of concentration C_s. To the second aliquot (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated as: $C_x = S_B V_x C_s/(S_A-S_B)V_X$ where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and C_S should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

14.12.1.2. Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of the interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 1 (copied from the EPA method referenced in section 22 of this SOP). A linear regression program may be used to obtain the intercept concentration.

14.12.1.2.1. For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

14.12.1.2.1.1. The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best

results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

14.12.1.2.1.2. The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard additions should respond in a similar manner as the analyte.

14.12.1.2.1.3. The determination must be free of spectral interference and corrected for nonspecific background interference.

15. CALCULATIONS

15.1. Response factors are calculated as follows:

$$RF = \frac{C_x}{A_x}$$

where: RF = response factor for target analyte being measured.
A_x = absorbance of the target analyte being measured.
C_x = concentration of the target analyte being measured in mg/l.

15.2. The percent relative standard deviation is calculated as follows:

$$\%RSD = \frac{SD}{RF_{ave}} \times 100$$

where: %RSD = percent relative standard deviation.
SD = standard deviation of the three initial RFs for the target analyte.
RF_{ave} = mean of the three initial RFs for the target analyte.

15.3. The percent difference of each analyte is calculated as follows:

$$\%D = \frac{|RF_{ave} - RF|}{RF_{ave}} \times 100$$

where: %D = percent difference (or percent drift) of each target analyte.
RF = target analyte's daily RF.
RF_{ave} = mean of the 3 initial RFs for the target analyte.

15.4. The recovery of LCS compounds is calculated as follows:

$$\%REC_{LCS} = \left(\frac{C_{recovered}}{C_{added}}\right) \times 100$$

where: $\%REC_{LCS}$ = percent recovery of target analyte in LCS (or LCSD).
$C_{recovered}$ = concentration of target analyte recovered.
C_{added} = concentration of target analyte added.

15.5. The recovery of the MS compounds is calculated as follows:

$$\%REC_{MS} = \left(\frac{C_{recovered} - C_{sample}}{C_{added}}\right) \times 100$$

where: $\%REC_{MS}$ = percent recovery of target analyte in MS (or MSD).
$C_{recovered}$ = concentration of target analyte recovered.
C_{sample} = concentration of target analyte in the sample used.
C_{added} = concentration of target analyte added.

15.6. The relative percent difference is calculated as follows:

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where: RPD = relative percent difference between C_1 and C_2.
C_1 = concentration of target analyte recovered in measurement 1.
C_2 = concentration of target analyte recovered in measurement 2.

15.7. Compound concentration in the sample extract is calculated as follows:

$$C_x = \frac{C_e \times V \times D}{W}$$

where: C_x = concentration of the sample in mgCr(VI)/kg.
C_e = concentration of Cr(VI) in the digestate or extract in ppm.
V = final digestate or extract volume in mls.
D = dilution factor.
W = sample weight in g.

15.8. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

16. METHOD PERFORMANCE

16.1. The data shown in Table 1 (from the published method referenced in this SOP) were obtained from records of state and contractor laboratories. The data are intended to show the precision of the combined sample preparation and analysis method.

17. POLLUTION PREVENTION

17.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Calscience's Health, Safety, and Respiratory Protection Manual. In general, protective eyewear (e.g. safety glasses or goggles), and protective apparel (e.g. lab coats) and gloves are required to be worn when handling chemicals.

17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:

17.3.1. A NIOSH approved air-purifying respirator with cartridges appropriate for the chemical handled.

17.3.2. Extended length protective gloves.

17.3.3. Face shield.

17.3.4. Full-length laboratory apron.

17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.

17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles. Alternatively, the Coordinator may utilize a self-contained breathing apparatus or other supplied air system if appropriately trained and approved by the Health and Safety Manager.

17.6. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

18.1. The acceptance criteria for LCS/LCSD compounds vary depending upon historical data. The upper and lower acceptance limits for %REC and RPD of each LCS/LCSD compound are based upon the historical average recovery ±3S. All LCS/LCSD compounds must be within acceptance limits. If one or more LCS/LCSD compounds are not acceptable, the problem must be identified and corrected.

18.1.1. If the LCS and/or LCSD %REC is outside of the acceptance limits high, the RPD is within acceptance limits, and all target analytes in the associated

samples are not detected, the sample data can be reported without qualification.

18.1.2. The LCSD is only reported when the MS/MSD is unacceptable due to matrix interference effects, or when the LCS/LCSD is used in place of MS/MSD due to insufficient sample quantity.

18.2. Ideally, the concentration of target analytes in a MB should be less than the respective reporting limits (RLs). If the concentration of any target analyte exceeds its RL, the source of contamination must be investigated and, if possible, eliminated. The acceptance criteria for MBs is as follows:

18.2.1. If a target analyte is found in the MB but not in the associated samples, report the sample and MB data without qualification.

18.2.2. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-extracted and/or re-analyzed.

18.3. The acceptance criteria for MS/MSDs are as follows:

18.3.1. When the %REC and RPD of the MS/MSD compounds are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.

18.3.2. If the %REC and/or RPD of the MS/MSD compounds are not within the established acceptance limits, the analytical system performance shall be suspect.

18.4. Matrix effects or poor instrument performance/technique typically causes unacceptable % REC values. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.

18.5. Additional information regarding internal quality control checks is provided in SOP-T020.

18.6. All concentrations shall be reported in mg/L (ppm) for water samples and mg/kg (ppm) for oil, soil and solid waste samples.

18.7. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

19. CORRECTIVE ACTIONS

19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results,

analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.

19.1. The Operations Manager, Project Manager, Quality Control Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.

19.1. Corrective actions are generally of two types, immediate and long-term actions.

19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.

19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented in the Corrective Action Record. Examples of this type of action include:

19.3.2.1. Remedial training of staff in technical skills, technique or implementation of operating procedures.

19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.

19.3.2.3. Revision of standard operating procedures.

19.3.2.4. Replacing personnel, as necessary.

19.1. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:

19.4.1. Define the problem.

19.4.2. Assign responsibility for investigating the problem.

19.4.3. Investigate and determine the cause of the problem.

19.4.4. Assign and accept responsibility for implementing the corrective action.

19.4.5. Determine effectiveness of the corrective action and implement correction.

19.4.6. Verify that the corrective action has eliminated the problem.

19.1. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

20. CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

20.1. Out-of-control data are reviewed and verified by the technical director of the appropriate department. All samples associated with an unacceptable QC set is then subject to reanalysis, depending upon the QC type in question.

20.2.1. MS/MSD: Acceptability of the MS/MSD recoveries are subject to the matrix and any anomalies associated with the subject batch. Failure of recoveries an MS/MSD data set does not constitute an automatic reanalysis of the batch samples. Rather, it is acceptable to defer to the LCS/LCSD recoveries, to determine acceptance of the sample results.

20.2.2. LCS/LCSD: Because they denote whether the analytical system is operating within control, it is imperative that the LCS recoveries obtained are within acceptability criteria. If the recoveries fail for a given reported compound, the technical director confirms the unacceptable result.

20.2.2.1. If the LCS results are verified as acceptable, no corrective action is required.

20.2.2.2. If the LCS result is verified as out-of-control, and the subject compound is to be reported in samples within that analytical batch, the samples reported with that failed compound must be reanalyzed with a valid LCS recovery for the compound.

20.2.2.3. If the LCS result is verified as out-of-control, and the subject compound is NOT to be reported in the samples within that analytical batch, the samples are not subject to reanalysis. No corrective action is required for that batch.

21. WASTE MANAGEMENT

21.1. The proper disposal of analytical samples and laboratory wastes is not only good laboratory practice, but also regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated, and either returned to the client (preferable) or placed into the proper laboratory waste stream.

21.2. Unused or remaining soil or liquid samples and all other solid or liquid wastes resulting from our laboratory operations are considered hazardous for disposal purposes.

21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal.

21.4. Each specific laboratory area shall maintain clearly labeled waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste.

21.5. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly.

21.6. In order to maintain accountability for all samples received by Calscience, when a sample is used in its entirety for analysis, the empty container(s) are returned to Sample Control for placement in analytical storage.

21.7. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Waste."

22. REFERENCES

22.1. US EPA, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Volume 1A, Method 7196A, Hexavalent Chromium (Colorimetric), Revision 1, July 1992.

22.2. US EPA, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Volume IA, Method 7000A, Atomic Absorption Methods, Revision 1, July 1992.

23. TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

23.1. Flowchart of Method Steps: US EPA, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Volume 1A, Method 7196A, Hexavalent Chromium (Colorimetric), Revision 1, July 1992.

23.2. Table 1. Method Performance Data, US EPA, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Volume 1A, Method 7196A, Hexavalent Chromium (Colorimetric), Revision 1, July 1992.

23.3. Figure 1, Standard Addition Plot, US EPA, Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Volume IA, Method 7000A, Atomic Absorption Methods, Revision 1, July 1992.

Flowchart of Method Steps
US EPA, Test Methods for Evaluating Solid Waste
SW-846, Third Edition, Volume 1A,
Method 7196A, Hexavalent Chromium (Colorimetric), Revision 1, July 1992.

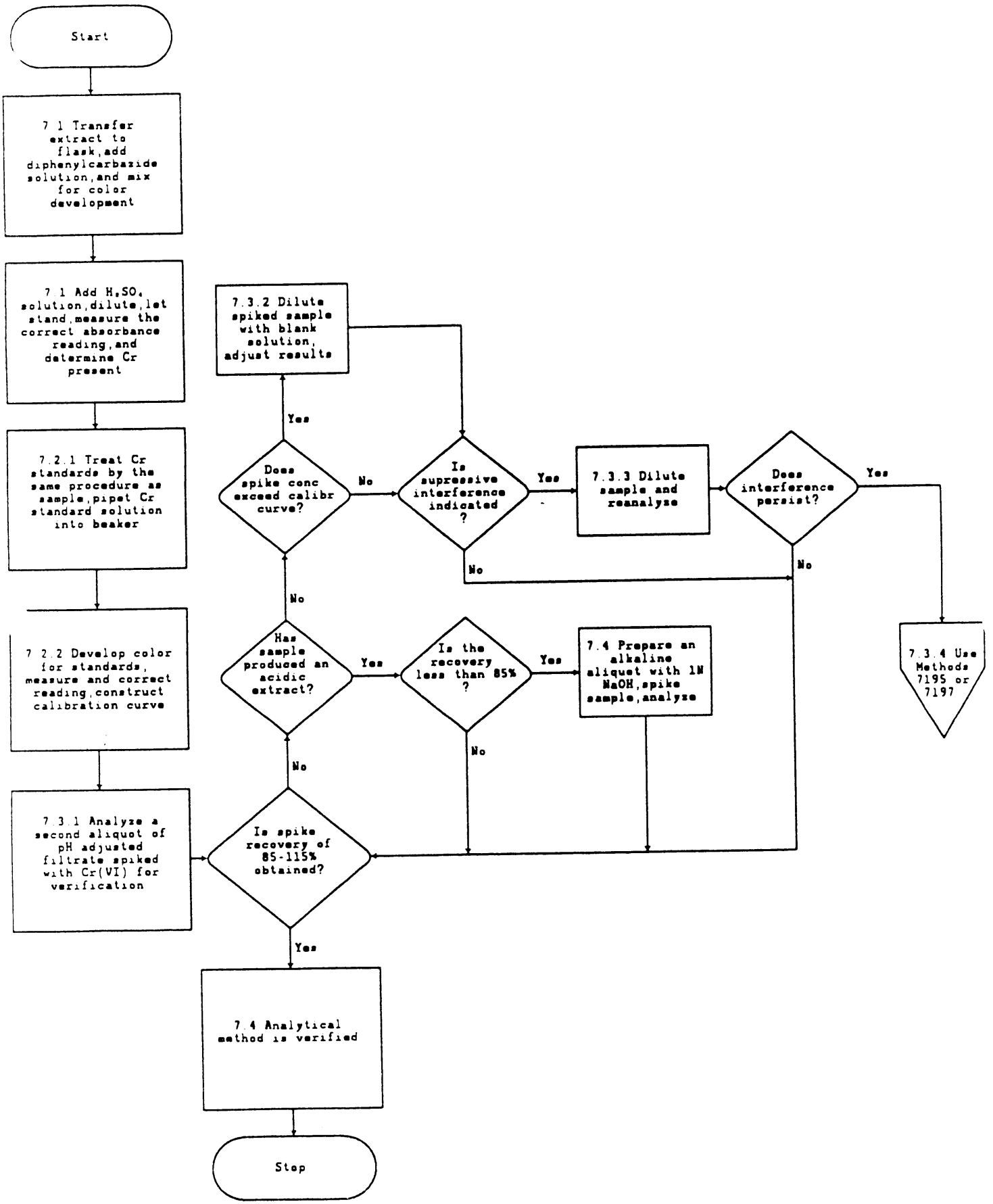

Start
7.1 Transfer extract to flask, add diphenylcarbazide solution, and mix for color development
7.1 Add H_2SO_4 solution, dilute, let stand, measure the correct absorbance reading, and determine Cr present
7.2.1 Treat Cr standards by the same procedure as sample, pipet Cr standard solution into beaker
7.2.2 Develop color for standards, measure and correct reading, construct calibration curve
7.3.1 Analyze a second aliquot of pH adjusted filtrate spiked with Cr(VI) for verification
Is spike recovery of 85-115% obtained?
Yes
7.4 Analytical method is verified
Stop
No
Has sample produced an acidic extract?
Yes
Is the recovery less than 85% ?
Yes
7.4 Prepare an alkaline aliquot with 1N NaOH, spike sample, analyze
No
No
Does spike conc exceed calibr curve?
Yes
7.3.2 Dilute spiked sample with blank solution, adjust results
No
Is supressive interference indicated ?
Yes
7.3.3 Dilute sample and reanalyze
Does interference persist?
Yes
7.3.4 Use Methods 7195 or 7197
No
No

Table 1. Method Performance Data

US EPA, Test Methods for Evaluating Solid Waste,SW-846, Third Edition, Volume 1A

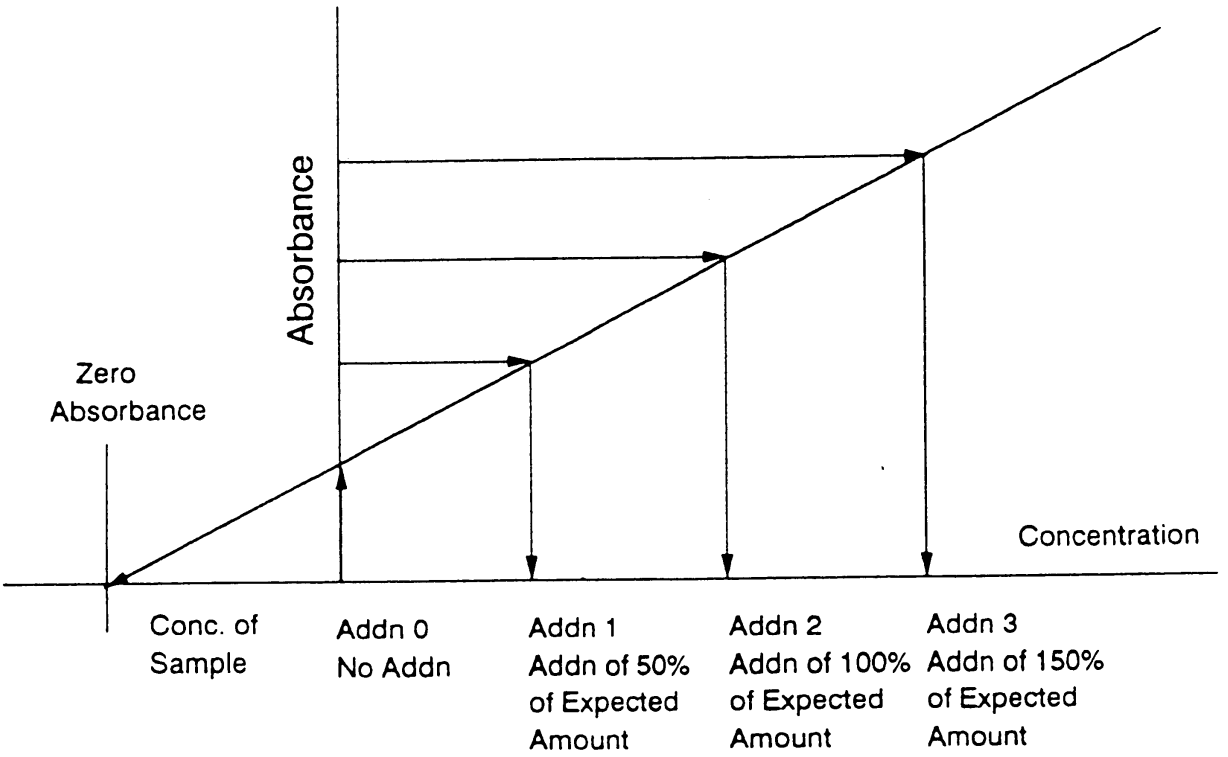
Method 7196A, Hexavalent Chromium (Colorimetric), Revision 1, July 1992.

TABLE 1. METHOD PERFORMANCE DATA

Sample Matrix	Preparation Method	Laboratory Replicates
Wastewater treatment sludge	Not known	0.096, 0.107 ug/g
Sediment from chemical storage area	3060	115, 117 ug/g

Figure 1, Standard Addition Plot
US EPA Test Methods for Evaluating Solid Waste
SW-846, Third Edition, Volume IA
Method 7000A, Atomic Absorption Methods
Revision 1, July 1992.

FIGURE 1.
STANDARD ADDITION PLOT



COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

6010B S TTLC (1474)

METHOD: EPA 6010B

MATRIX: Solid

COMPOUND NAME	MDL	RL	UNITS
Antimony	0.3061180	0.75000	mg/kg
Arsenic	0.3979205	0.75000	mg/kg
Barium	0.2361168	0.50000	mg/kg
Beryllium	0.0894367	0.25000	mg/kg
Cadmium	0.0883012	0.50000	mg/kg
Chromium	0.0878085	0.25000	mg/kg
Cobalt	0.0872733	0.25000	mg/kg
Copper	0.2022842	0.50000	mg/kg
Lead	0.1810761	0.50000	mg/kg
Molybdenum	0.0777007	0.25000	mg/kg
Nickel	0.1150345	0.25000	mg/kg
Phosphorus	0.4587909	5.00000	mg/kg
Selenium	0.3509483	0.75000	mg/kg
Silver	0.1174230	0.25000	mg/kg
Thallium	0.2319970	0.75000	mg/kg
Vanadium	0.0973926	0.25000	mg/kg
Zinc	0.2422880	1.00000	mg/kg
Aluminum	0.5756699	2.50000	mg/kg
Lithium	0.2127227	2.50000	mg/kg
Bismuth	0.2364717	5.00000	mg/kg
Calcium	0.7917464	5.00000	mg/kg
Iron	0.3956605	5.00000	mg/kg
Magnesium	0.4298655	5.00000	mg/kg
Manganese	0.2193937	0.25000	mg/kg
Potassium	6.6022051	25.00000	mg/kg
Sodium	5.5899844	25.00000	mg/kg
Strontium	0.0952685	1.50000	mg/kg
Tin	0.3715347	2.50000	mg/kg
Titanium	0.3051934	1.50000	mg/kg
Boron	0.6257641	1.00000	mg/kg
Silicon	0.4140232	5.00000	mg/kg
Sulfur, Total	0.7975848	5.00000	mg/kg

QC RECOVERY CONTROL LIMITS

METHOD: EPA 6010B

TESTCODE: 6010B_S_TTLC

(1474)

MS , LCS COMPOUNDS

COMPOUND NAME	MS/MSD REC CONTROL LIMITS	MS/MSD RPD CONTROL LIMITS	LCS/LCSD REC CONTROL LIMITS	LCS/LCSD RPD CONTROL LIMITS
Antimony	50-115	0-20	80-120	0-20
Arsenic	75-125	0-20	80-120	0-20
Barium	75-125	0-20	80-120	0-20
Beryllium	75-125	0-20	80-120	0-20
Cadmium	75-125	0-20	80-120	0-20
Chromium	75-125	0-20	80-120	0-20
Cobalt	75-125	0-20	80-120	0-20
Copper	75-125	0-20	80-120	0-20
Lead	75-125	0-20	80-120	0-20
Mercury	76-136	0-16	82-124	0-16
Molybdenum	75-125	0-20	80-120	0-20
Nickel	75-125	0-20	80-120	0-20
Phosphorus, Total	75-125	0-20	80-120	0-20
Selenium	75-125	0-20	80-120	0-20
Silver	75-125	0-20	80-120	0-20
Thallium	75-125	0-20	80-120	0-20
Vanadium	75-125	0-20	80-120	0-20
Zinc	75-125	0-20	80-120	0-20
Aluminum	75-125	0-20	80-120	0-20
Bismuth	75-125	0-20	80-120	0-20
Calcium	75-125	0-20	80-120	0-20
Iron	75-125	0-20	80-120	0-20
Magnesium	75-125	0-20	80-120	0-20
Manganese	75-125	0-20	80-120	0-20
Potassium	75-125	0-20	80-120	0-20
Silicon	75-125	0-20	80-120	0-20
Sodium	75-125	0-20	80-120	0-20
Strontium	75-125	0-20	80-120	0-20
Tin	75-125	0-20	80-120	0-20
Tungsten	75-125	0-20	80-120	0-20
Gold	75-125	0-20	80-120	0-20
Titanium	75-125	0-20	80-120	0-20
Boron	75-125	0-20	80-120	0-20
Sulfur, Total	75-125	0-20	80-120	0-20
Phosphorus	75-125	0-20	80-120	0-20
Lithium	75-125	0-20	80-120	0-20

SURROGATES

COMPOUND NAME	REC CONTROL LIMITS

COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

HG TTLC (2323)

METHOD: EPA 7471A

MATRIX: Solid

COMPOUND NAME	MDL	RL	UNITS
Mercury	0.0038984	0.08350	mg/kg

QC RECOVERY CONTROL LIMITS

METHOD: EPA 7471A

TESTCODE: HG_TTLC

(2323)

MS , LCS COMPOUNDS

COMPOUND NAME	MS/MSD REC CONTROL LIMITS	MS/MSD RPD CONTROL LIMITS	LCS/LCSD REC CONTROL LIMITS	LCS/LCSD RPD CONTROL LIMITS
Mercury	71-137	0-14	85-121	0-10

SURROGATES

COMPOUND NAME	REC CONTROL LIMITS

COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

6010B STLC (4512)

METHOD: EPA 6010B

MATRIX: Aqueous

COMPOUND NAME	MDL	RL	UNITS
Antimony	0.0787440	0.15000	mg/L
Arsenic	0.0438341	0.15000	mg/L
Barium	0.0296291	0.10000	mg/L
Beryllium	0.0262172	0.10000	mg/L
Cadmium	0.0268885	0.10000	mg/L
Chromium	0.0270571	0.10000	mg/L
Cobalt	0.0294518	0.10000	mg/L
Copper	0.0266534	0.10000	mg/L
Lead	0.0405958	0.10000	mg/L
Molybdenum	0.0277965	0.10000	mg/L
Nickel	0.0298379	0.10000	mg/L
Selenium	0.0699064	0.15000	mg/L
Silver	0.0139201	0.05000	mg/L
Thallium	0.0290618	0.15000	mg/L
Vanadium	0.0244008	0.10000	mg/L
Zinc	0.0352122	0.10000	mg/L
Aluminum	0.1235668	0.50000	mg/L
Bismuth	XXXXX	0.00000	mg/L
Calcium	0.1177116	1.00000	mg/L
Iron	0.1013501	1.00000	mg/L
Magnesium	0.0336474	1.00000	mg/L
Manganese	0.0270313	0.05000	mg/L
Potassium	1.0306524	5.00000	mg/L
Sodium	1.0309070	5.00000	mg/L
Strontium	0.0277385	0.30000	mg/L
Tin	0.0193041	0.50000	mg/L
Tungsten	XXXXX	0.00000	mg/L
Gold	XXXXX	0.00000	mg/L
Titanium	0.0267072	0.30000	mg/L
Boron	0.0475515	0.20000	mg/L

COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

6010B W T-S (5592)

METHOD: EPA 6010B

MATRIX: Aqueous

COMPOUND NAME	MDL	RL	UNITS
Lithium	0.0401534	0.50000	mg/L
Sulfur, Total	0.0927546	0.50000	mg/L
Antimony	0.0787440	0.15000	mg/L
Arsenic	0.0438341	0.15000	mg/L
Barium	0.0296291	0.10000	mg/L
Beryllium	0.0262172	0.10000	mg/L
Cadmium	0.0268885	0.10000	mg/L
Chromium	0.0270571	0.10000	mg/L
Cobalt	0.0294518	0.10000	mg/L
Copper	0.0266534	0.10000	mg/L
Lead	0.0405958	0.10000	mg/L
Molybdenum	0.0277965	0.10000	mg/L
Nickel	0.0298379	0.10000	mg/L
Selenium	0.0699064	0.15000	mg/L
Silver	0.0139201	0.05000	mg/L
Thallium	0.0290618	0.15000	mg/L
Vanadium	0.0244008	0.10000	mg/L
Aluminum	0.1235668	0.50000	mg/L
Calcium	0.1177116	1.00000	mg/L
Iron	0.1013501	1.00000	mg/L
Magnesium	0.0336474	1.00000	mg/L
Manganese	0.0270313	0.05000	mg/L
Potassium	1.0306524	5.00000	mg/L
Sodium	1.0309070	5.00000	mg/L
Strontium	0.0277385	0.30000	mg/L
Tin	0.0193041	0.50000	mg/L
Titanium	0.0267072	0.30000	mg/L
Boron	0.0475515	0.20000	mg/L
Silicon	0.2788283	0.50000	mg/L
Zinc	0.0352122	0.10000	mg/L

COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

HG STLC (4358)

METHOD: EPA 7470A

MATRIX: Aqueous

COMPOUND NAME	MDL	RL	UNITS
Mercury	0.0002265	0.00500	mg/L

COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

HG T-S (4359)

METHOD: EPA 7470A

MATRIX: Aqueous

COMPOUND NAME	MDL	RL	UNITS
Mercury	0.0002265	0.00500	mg/L

COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

Cr6 S (4346)

METHOD: **EPA 7196A** **MATRIX: Solid**

COMPOUND NAME	MDL	RL	UNITS
Chromium, Hexavalent	0.2154	0.8000	mg/kg

COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

7196A S STLC (4840)

METHOD: EPA 7196A

MATRIX: Solid

COMPOUND NAME	MDL	RL	UNITS
Chromium, Hexavalent	0.0114	0.0200	mg/L

COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

PEST S (5157)

METHOD: EPA 8081A

MATRIX: Solid

COMPOUND NAME	MDL	RL	UNITS
Aldrin	1.5455	5.0000	ug/kg
Alpha-BHC	1.4710	5.0000	ug/kg
Beta-BHC	1.2748	5.0000	ug/kg
Chlordane	20.0313	50.0000	ug/kg
4,4'-DDD	1.2899	5.0000	ug/kg
4,4'-DDE	1.5023	5.0000	ug/kg
4,4'-DDT	1.6400	5.0000	ug/kg
Delta-BHC	1.5902	5.0000	ug/kg
Dieldrin	1.1340	5.0000	ug/kg
Endosulfan I	1.7807	5.0000	ug/kg
Endosulfan II	0.8766	5.0000	ug/kg
Endosulfan Sulfate	1.3149	5.0000	ug/kg
Endrin	1.0084	5.0000	ug/kg
Endrin Aldehyde	0.9768	5.0000	ug/kg
Endrin Ketone	1.5022	5.0000	ug/kg
Gamma-BHC	1.1426	5.0000	ug/kg
Heptachlor	1.1149	5.0000	ug/kg
Heptachlor Epoxide	0.9187	5.0000	ug/kg
Methoxychlor	0.8367	5.0000	ug/kg
Total Pesticides	0.0000	5.0000	ug/kg
Toxaphene	42.3570	100.0000	ug/kg

QC RECOVERY CONTROL LIMITS

METHOD: EPA 8081A

TESTCODE: PEST_S

(5157)

MS , LCS COMPOUNDS

COMPOUND NAME	MS/MSD REC CONTROL LIMITS	MS/MSD RPD CONTROL LIMITS	LCS/LCSD REC CONTROL LIMITS	LCS/LCSD RPD CONTROL LIMITS
4,4'-DDD	50-135	0-25	50-135	0-25
4,4'-DDE	50-135	0-25	50-135	0-25
4,4'-DDT	50-135	0-25	50-135	0-25
Aldrin	50-135	0-25	50-135	0-25
Alpha-BHC	50-135	0-25	50-135	0-25
Beta-BHC	50-135	0-25	50-135	0-25
Delta-BHC	50-135	0-25	50-135	0-25
Dieldrin	50-135	0-25	50-135	0-25
Endosulfan I	50-135	0-25	50-135	0-25
Endosulfan II	50-135	0-25	50-135	0-25
Endosulfan Sulfate	50-135	0-25	50-135	0-25
Endrin	50-135	0-25	50-135	0-25
Endrin Aldehyde	50-135	0-25	50-135	0-25
Gamma-BHC	50-135	0-25	50-135	0-25
Heptachlor	50-135	0-25	50-135	0-25
Heptachlor Epoxide	50-135	0-25	50-135	0-25
Methoxychlor	50-135	0-25	50-135	0-25

SURROGATES

COMPOUND NAME	REC CONTROL LIMITS
Decachlorobiphenyl	50-135
2,4,5,6-Tetrachloro-m-Xylene	50-135

COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

STLC PEST (4632)

METHOD: EPA 8081A

MATRIX: Solid

COMPOUND NAME	MDL	RL	UNITS
Alpha-BHC	0.2796	1.0000	ug/L
Gamma-BHC	0.2994	1.0000	ug/L
Beta-BHC	0.2998	1.0000	ug/L
Heptachlor	0.2633	1.0000	ug/L
Delta-BHC	0.2859	1.0000	ug/L
Aldrin	0.2667	1.0000	ug/L
Heptachlor Epoxide	0.2504	1.0000	ug/L
Endosulfan I	0.2782	1.0000	ug/L
Dieldrin	0.2852	1.0000	ug/L
4,4'-DDE	0.2656	1.0000	ug/L
Endrin	0.3067	1.0000	ug/L
Endrin Aldehyde	0.2647	1.0000	ug/L
4,4'-DDD	0.2708	1.0000	ug/L
Endosulfan II	0.2715	1.0000	ug/L
4,4'-DDT	0.2674	1.0000	ug/L
Endosulfan Sulfate	0.2916	1.0000	ug/L
Methoxychlor	0.2510	1.0000	ug/L
Chlordane	3.3050	10.0000	ug/L
Toxaphene	5.9203	10.0000	ug/L
Endrin Ketone	0.2443	1.0000	ug/L

QC RECOVERY CONTROL LIMITS

METHOD: EPA 8081A

TESTCODE: STLC_PEST

(4632)

MS , LCS COMPOUNDS

COMPOUND NAME	MS/MSD REC CONTROL LIMITS	MS/MSD RPD CONTROL LIMITS	LCS/LCSD REC CONTROL LIMITS	LCS/LCSD RPD CONTROL LIMITS
4,4'-DDT	50-135	0-25	50-135	0-25
Dieldrin	50-135	0-25	50-135	0-25
Endosulfan I	50-135	0-25	50-135	0-25
Endrin	50-135	0-25	50-135	0-25
Gamma-BHC	50-135	0-25	50-135	0-25
Heptachlor	50-135	0-25	50-135	0-25

SURROGATES

COMPOUND NAME	REC CONTROL LIMITS
Decachlorobiphenyl	50-130
2,4,5,6-Tetrachloro-m-Xylene	50-130

COMPOUND LISTING / MDL / CALC REPORTING LIMIT FOR TESTCODE

TCLP PEST S (564)

METHOD: EPA 8081A

MATRIX: Solid

COMPOUND NAME	MDL	RL	UNITS
Dieldrin	0.2852	1.0000	ug/L
Gamma-BHC	0.2994	1.0000	ug/L
Heptachlor	0.2633	1.0000	ug/L
Heptachlor Epoxide	0.2504	1.0000	ug/L
Endrin	0.3067	1.0000	ug/L
Methoxychlor	0.2510	1.0000	ug/L
Chlordane	3.3050	10.0000	ug/L
Toxaphene	5.9203	10.0000	ug/L
Endrin Ketone	0.2443	1.0000	ug/L

QC RECOVERY CONTROL LIMITS

METHOD: EPA 8081A

TESTCODE: TCLP_PEST_S

(564)

MS , LCS COMPOUNDS

COMPOUND NAME	MS/MSD REC CONTROL LIMITS	MS/MSD RPD CONTROL LIMITS	LCS/LCSD REC CONTROL LIMITS	LCS/LCSD RPD CONTROL LIMITS
Endrin	50-135	0-25	50-135	0-25
Gamma-BHC	50-135	0-25	50-135	0-25
Heptachlor	50-135	0-25	50-135	0-25

SURROGATES

COMPOUND NAME	REC CONTROL LIMITS
Decachlorobiphenyl	50-135
2,4,5,6-Tetrachloro-m-Xylene	50-135