Newark Bay Study Area

Crab and Clam Sampling and Analysis

Quality Assurance Project Plan

Baseline Human Health and
Ecological Risk Assessment

Tierra Solutions, Inc.

East Brunswick, New Jersey

October 2013

Revision 0

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Acronyms and Abbreviations

BERA	baseline ecological risk assessment
BHHERA	baseline human health and ecological risk assessment
BSAF	biota-sediment accumulation factor
CBR	critical body residue
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COPEC	constituent of potential ecological concern
CPG	Cooperating Parties Group
CSM	conceptual site model
DEAR	Phase I and Phase II Data Evaluation and Analysis Report (Tierra 2013b)
Deposition Report	Phase I and Phase II Sediment Deposition Report (Tierra 2011)
HHRA	human health risk assessment
LPRRP	Lower Passaic River Restoration Project
NBSA	Newark Bay Study Area
NOAA	National Oceanic and Atmospheric Administration
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzo-*p*-dioxin (dioxin)
PCDF	polychlorinated dibenzofuran (furan)
Problem Formulation	Newark Bay Study Area Problem Formulation (Tierra 2013a)
RI	Remedial Investigation
RI/FS	Remedial Investigation/Feasibility Study
SOP	standard operating procedure
SVOC	semivolatile organic compound
Tierra	Tierra Solutions, Inc.
TRV	toxicity reference value
USEPA	U.S. Environmental Protection Agency
VOC	volatile organic compound

1. Introduction

This document presents the Quality Assurance Project Plan (QAPP) for the proposed sampling and analysis of blue crab (*Callinectes sapidus*) and bivalve clam (e.g., *Mya arenaria, Macoma sp.*) tissue and co-located surface sediment in the Newark Bay Study Area (NBSA). This tissue and sediment collection event is anticipated to occur in fall 2013 and will commence sampling activities for the baseline human health and ecological risk assessment (BHHERA). The BHHERA is being conducted as part of Phase III of the Remedial Investigation for the NBSA. The BHHERA will address the goal to "*Determine the primary human and ecological receptors (endpoints) of PCDDs, PCDFs, PCBs, PAHs, pesticides, and metals contaminated sediments in the NBSA*" (U.S. Environmental Protection Agency [USEPA] 2004).

Agreements made during the June 28 to 29, 2011 BHHERA Workshop among USEPA, its Partner Agencies[1] and Tierra Solutions, Inc. (Tierra) resulted in the development of the Problem Formulation for the NBSA (Tierra 2013a). Following approval of the Problem Formulation, this QAPP is the first of several documents to be developed for the tissue and sediment sampling associated with the NBSA BHHERA. Specifically, tissue and sediment chemical data collected under this QAPP will support the following objectives of the BHHERA:

1. Evaluate whether exposure to site-related constituents of potential ecological concern (COPECs) in the NBSA poses unacceptable risks to benthic macroinvertebrates (represented by blue crab and bivalve clams).
2. Evaluate whether the consumption of shellfish from the NBSA poses unacceptable risks to human and ecological receptors.
3. Evaluate whether exposure to surface sediments in the Intertidal Areas of the NBSA poses unacceptable risks to human and ecological receptors.

The blue crab and bivalve clam tissue data and the co-located sediment data will be used in the baseline ecological risk assessment (BERA) as input parameters to a food web model, which will evaluate dietary exposure of COPECs to upper trophic level ecological receptors (i.e., birds and mammals). Surface sediment data will also be compared to appropriate toxicity benchmarks to qualitatively evaluate potential ecological risks to aquatic plants, fish, and reptiles in the NBSA. In addition, the clam tissue data and co-located sediment data will be used (to the extent practicable) to develop biota-sediment accumulation factors (BSAFs) for the NBSA.

The collection and analysis of blue crab and bivalve clam tissue will provide valuable data to evaluate potential human health risks from ingestion of these species. Sediment chemistry data will be used in the human health risk assessment (HHRA) to evaluate exposure to humans via multiple exposure pathways

[1] The Partner Agencies include the U.S. Army Corps of Engineers, New Jersey Department of Environmental Protection, New Jersey Department of Transportation, National Oceanic and Atmospheric Administration (NOAA), and U.S. Fish and Wildlife Service.

(e.g., direct contact, incidental ingestion). In addition, further tissue and sediment data are anticipated to be collected in the NBSA as part of the larger sampling program in 2014. These data will be combined with the data collected under this QAPP to assess risks to potential receptors in the NBSA.

2. Background Information

The NBSA is identified as Newark Bay and portions of the Hackensack River, Kill van Kull, and Arthur Kill. Pursuant to the Administrative Order on Consent under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA Index 02-2004-2010; USEPA 2004), a Remedial Investigation/Feasibility Study (RI/FS) is being conducted by Tierra, on behalf of Occidental Chemical Corporation (the successor to Diamond Shamrock Chemicals Company [formerly known as Diamond Alkali Company]), for the NBSA.

The NBSA is situated within one of the most urbanized and industrialized areas in the United States and is known to be contaminated with a number of chemicals including, but not limited to, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), pesticides, herbicides, volatile and semivolatile organic compounds (VOCs and SVOCs, respectively), polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans (PCDDs/PCDFs), and metals (NOAA 1995; USEPA 1998).

Due to the size and complexity of the NBSA, USEPA and Tierra agreed in 2005 that the RI would be implemented in multiple phases. Collectively, the Phase I and Phase II investigations gathered information on NBSA sediment, as described in the Phase I and Phase II Sediment Deposition Report, Revision 1 (Deposition Report; Tierra 2011) and the Draft Phase I and Phase II Data Evaluation and Analysis Report, Revision 1 (DEAR; Tierra 2013b). Other activities under the RI have included work related to the risk assessments (e.g., Draft Pathways Analysis Report [USEPA 2006] and Screening-Level Ecological Risk Assessment [USEPA 2008]). As part of the baseline risk assessment process, the Problem Formulation (Tierra 2013a) was developed to document the goals and focus of the baseline risk assessments, and define the questions to be addressed during these evaluations. Additional history and background information on the NBSA can be found in the aforementioned documents.

3. Data Use

The primary sample type that will be collected as part of this sampling event is tissue from blue crabs and bivalve clams, which will be collected from the Intertidal Areas of the NBSA. These tissue samples will be retained for chemical analyses. Surface sediment samples will also be collected from the same locations as the clam samples, from the top 6 inches (15 centimeters) of sediment. Surface sediment will be analyzed for the same chemical parameters that are evaluated in tissue, as well as herbicides and total extractable petroleum hydrocarbons (TEPH). In addition, sediment samples will also include analyses for wet chemistry (e.g., ammonia, sulfide), and physical parameters (e.g., grain size, percent moisture).

To evaluate bioaccumulation of COPECs in the NBSA, surface sediment data will be co-located and paired with clam samples to develop site-specific BSAFs for the NBSA.

3.1 Ecological Risk Assessment

The data described in this QAPP, in conjunction with data anticipated to be collected in 2014, will collectively support the BERA in evaluating the assessment endpoints for plant, benthic invertebrate, fish, bird, and mammal populations as presented in the Problem Formulation (Tierra 2013a) and summarized below:

Assessment Endpoint No. 1 - Survival and/or growth of aquatic plants and maintenance of plants as a food resource and habitat for fish and wildlife.

Sediment chemistry data collected as part of this sampling event will be used as a measurement endpoint to evaluate risks to aquatic plants by answering the following risk question: **Are COPEC concentrations in surface water and sediments from the NBSA greater than benchmarks for the survival and/or growth of aquatic plants?** To do this, measured chemical concentrations in sediment will be compared to toxicological benchmarks.

Assessment Endpoint No. 2 - Survival, growth, and/or reproduction of invertebrates.

Tissue chemistry data collected as part of this sampling event will be used as one measurement endpoint to evaluate risks to benthic invertebrates by answering the following risk question: **Are COPEC residues in invertebrate tissues from the NBSA greater than tissue benchmarks (e.g., critical body residues [CBRs]) for the survival, growth, and/or reproduction of invertebrates?** To do this, measured tissue chemical concentrations in blue crabs and clams will be compared to CBRs. As part of the assessment, whole body crab COPEC concentrations will be estimated by combining the results of the edible tissue and hepatopancreas tissue samples.

Sediment chemistry data collected as part of this sampling event will be used as one measurement endpoint to evaluate risks to benthic invertebrates by answering the following risk question: **Are COPEC concentrations in sediments from the biologically active zone greater than benchmarks**

for the survival, growth, and/or reproduction of invertebrates? To do this, measured chemical concentrations in sediment will be compared to toxicological benchmarks.

Assessment Endpoints No. 3 and 4 - Survival, growth, and/or reproduction of reptiles and fish, respectively.

Sediment chemistry data collected as part of this sampling event will be used as a measurement endpoint to evaluate risks to fish and reptiles by answering the following risk question: **Are COPEC concentrations in site sediment at levels that might adversely affect survival, growth, and/or reproduction of reptiles and fish?** To do this, measured chemical concentrations in sediment will be compared to toxicological benchmarks.

Assessment Endpoints No. 5 and 6 - Survival, growth, and/or reproduction of birds and mammals, respectively.

Tissue and sediment chemistry data collected as part of this sampling event will be used as input parameters for a food web model to estimate dietary intakes for select bird and mammal receptors by answering the following risk question: **Does the daily dose of COPECs received by birds and mammals from consumption of the tissues of prey species and from other media in the NBSA exceed the toxicity reference values (TRVs) for survival, growth, and/or reproduction of birds and mammals? If yes, what are the probabilities of effects of differing magnitude for survival, growth, and/or reproduction of birds and mammals?** To do this, measured chemical concentrations in sediment and blue crab and bivalve clam tissue will be incorporated into the food web model to estimate a daily dietary ingestion of chemicals. This value is then compared to the literature-based TRV to evaluate potential risks. If potential risks are evident (i.e., the resulting hazard quotient is greater than 1), a probabilistic food web model will be run to estimate the range of possible risks.

3.2 Human Health Risk Assessment

The tissue data collected during this sampling effort, combined with data anticipated to be collected in 2014, will be used to support the HHRA by estimating potential human exposures and assessing the potential impact of chemicals on human health via consumption of shellfish from the NBSA. Potential consumption scenarios are presented in the human health conceptual site model (CSM) included in the Problem Formulation (Tierra 2013a). Potential risks will be evaluated using hepatopancreas-only, edible muscle tissue only, and a combination of both (i.e., whole body). The HHRA will use these data as the basis for quantitatively evaluating the exposure of individuals under current and future scenarios for both cancer and non-cancer health effects following USEPA Superfund guidance, guidelines, and policies.

Sediment data collected during this sampling effort will also be used to support the HHRA by estimating potential human exposures and assessing the potential impact of chemicals on human health via dermal contact with, incidental ingestion of, and/or inhalation of VOCs from surface sediment, primarily in the

Intertidal Areas. Potential surface sediment exposure scenarios are presented in the human health CSM included in the Problem Formulation (Tierra 2013a).

4. Overview of Sampling Design and Locations

4.1 Biological Tissue

Blue crab and bivalve clam tissue samples will be collected from eight locations along the Intertidal Areas and surrounding shallow Subtidal Flats. The Intertidal Areas have sparse aquatic vegetation (e.g., *Spartina sp.*), and are expected to provide adequate habitat for bivalve clams and crustaceans. The existence and accessibility of the Intertidal Areas were confirmed during the Reconnaissance Survey in September 2013.

At each of the eight Intertidal Areas/shallow Subtidal Flats (Figure 1), one composite bivalve clam sample will be collected, for a total of eight clam samples. Clams will be collected by hand digging/shoveling or use of a clam rake. Clam samples will be shipped whole to the laboratory where the clams will be shucked, and soft tissue will be removed, processed and homogenized.

At each of the eight Intertidal Areas/shallow Subtidal Flats, one composite blue crab sample will also be collected. Crabs will be collected using baited crab traps. Bait will be typical blue crab bait purchased from a local bait shop. At least three crab traps will be deployed at each Intertidal Area; traps will be checked at least once every 12 hours. Retained crab samples will be shipped whole to the laboratory, where they will be processed and homogenized. Crab tissue samples will consist of hepatopancreas-only and edible muscle-only tissue. If sufficient hepatopancreas tissue is not available from each Intertidal Area, then hepatopancreas tissue from multiple Intertidal Areas within a zone (i.e., North, Central, South) will be composited to form a single sample. Samples would only be combined within a zone and will not consist of crabs from more than one zone.

4.2 Sediment

A single surface sediment sample (0 to 6 inches below sediment surface) will be collected from each Intertidal Area and surrounding shallow Subtidal Flats from the same general sampling locations where the clam samples were collected (total of eight sediment samples). Samples will be collected using techniques appropriate for water depth and substrate type (e.g., hand auger, ponar grab); the specific protocol and sampling equipment were evaluated in the field during the Reconnaissance Survey. The co-located sediment samples will be homogenized in the field, and submitted to a laboratory for chemical and physical analyses.

4.3 Sampling Locations

Sampling locations are located within each of eight Intertidal Areas and surrounding shallow Subtidal Flats. The Intertidal Areas have been identified and confirmed during the Reconnaissance Survey in September 2013.

4.4 Estimates of Sample Size

Samples sizes were determined by the number of Intertidal Areas in the NBSA. These areas were confirmed during the Reconnaissance Survey. Because this is an initial, preliminary sampling effort, additional data collected in 2014 will supplement data collected under this QAPP.

4.4.1 Biological Tissue Sample Composites

The target length of individual blue crabs for sampling is 4.5 inches (Tierra 2002, Windward 2009a). Based on this minimum size requirement and crab tissue regressions using blue crab data previously collected by Tierra (2002), each blue crab composite sample is anticipated to consist of approximately 25 blue crabs:

Muscle weight (g wet weight) = 1.36 x body length (mm) – 143.51

The target length, species, and number of bivalve clams per composite sample were identified following experimental sampling in the Intertidal Areas during the Reconnaissance Survey. While data from USEPA (2003) indicate a dominance of the bivalve community by softshell clams (*Mya arenaria*), additional bivalve species such as *Macoma balthica*, *Mulinia lateralis,* and/or *Mercenaria mercenaria* are also present, although in much smaller numbers. It is assumed that each composite sample will include at least 20 softshell clams, based on the following softshell clam length:weight regression from Beal (2002):

Wet tissue weight (g) = 0.4104 x body length (mm) + 0.5961

Composite samples of whole, intact blue crabs and bivalve clams will be submitted to the laboratory for homogenization and analysis. Composites will consist of crabs and clams that are of similar size for each composite (i.e., within 25 percent of the mean size for that composite). Clam samples will be composited by species. There will be a minimum of 5 organisms per composite sample; the maximum number of organisms per composite is variable. Crab samples will be composited by location with an equal number of male and female crabs when possible.

4.4.2 Sediment Sample Composites

Sediment will be collected from the surface (top 6 inches) at locations that are co-located with clam collection. Sediment will be homogenized and one composite sample per Intertidal Area will be submitted to the laboratory for analysis. Because clams will be collected from a general area within each of the eight Intertidal Areas, a sediment sample will be based on a composite of several individuals grabs from throughout the clam sampling area. An initial single sediment grab sample will be collected from the center of the clam sampling area for some target analytes (e.g., VOCs, acid volatile sulfides), as described in Standard Operating Procedure No. 9 (Appendix F). This sample will not be composited/homogenized with the rest of the collected sediment.

5. Analysis

The analyte list for the sediment and tissue collected under this sampling event is based on available data collected under the following programs:

- Newark Bay Phase I and II Sediment Investigations (Tierra 2011, 2013b)
- Lower Passaic River Restoration Project (LPRRP) RI/FS Cooperating Parties Group (CPG) Fish and Decapod Crustacean Tissue Collection (Windward 2009a)
- LPRRP RI/FS CPG Surface Sediment Sampling Program (Windward 2009b)

5.1 Biological Tissue

Exhibit 1 provides a summary of the targetchemical groups for tissue analysis under the CPG LPRRP, and identifies the targeted chemical groups that are proposed for crab and clam tissue analyses in the NBSA under this QAPP.

Exhibit 1. Chemical Groups for Tissue Analysis

Analyte Group	Lower Passaic River Restoration Project	Proposed for NBSA Phase III Investigation
Dioxins/Furans	X	X
Congener PCBs	X	X
Aroclor PCBs	X	X
Total Metals	X	X
Mercury	X	X
Methylmercury	X	X
SVOCs (including phthalates)	X	X
PAHs (including alkylated)	X	X
Lipids	X	X
Percent Moisture	X	X
Pesticides	X	X
Herbicides		
Butyltins	X	X

Note:
X = the analysis was conducted in previous tissue samples or is being proposed in future samples.

5.2 Sediment

Exhibit 2 provides a summary of the target chemical groups for sediment analysis under the CPG LPRRP and the Phase I and II of the Newark Bay Sediment Investigations, and identifies the targeted chemical groups that are proposed for sediment analyses in the NBSA under this QAPP.

Exhibit 2. Chemical Groups for Sediment Analysis

Analyte Group	Lower Passaic River Restoration Project	NBSA Phase I/II Sediment Investigation	Proposed for NBSA Phase III Investigation
Dioxins/Furans	X	X	X
Congener PCBs	X	X	X
Aroclor PCBs	X	X	X
Total Metals	X	X	X
Mercury	X	X	X
Methylmercury	X		X
Hexavalent Chromium	X	X	X
SVOCs (including phthalates)	X	X	X
PAHs (including alkylated)	X	X	X
VOCs	X	X	X
Percent Moisture	X	X	X
Pesticides	X	X	X
Herbicides	X	X	X
Butyltins	X	X	X
Total Petroleum Hydrocarbons	X	X	X
Total Organic Carbon	X	X	X
pH	X		X
Oxidation Reduction Potential			X
Total Sulfide	X		X
Total Phosphorus	X		X
Total Kjeldahl Nitrogen	X		X
AVS/SEM	X		X
Ammonia	X		X
Cyanide	X	X	X
Grain Size	X	X	X

Note:
X = the analysis was conducted in previous sediment samples or is being proposed in future samples.

6. Document Organization

This document was prepared using the Uniform Federal Policy for Quality Assurance Project Plans guidance (USEPA et al. 2005). Worksheet No. 2 identifies the location of each element of this QAPP. A brief summary of the information provided in this document is presented below.

Information about personnel and project organization related specifically to this QAPP, including personnel responsibilities, qualifications, and special training; as well as project organization, distribution, and communications pathways, are presented in Worksheet Nos. 3 through 8. A summary of the scoping sessions conducted for the development of this QAPP is presented in Worksheet No. 9.

The problem definition, project quality objectives, a summary of project tasks, and the project schedule and timeline for this QAPP are summarized in Worksheet Nos. 10, 11, 14, and 16, respectively. A summary of secondary data that may be used for the completion of this QAPP is provided in Worksheet No. 13. The field sampling design, rationale, and a list of proposed sampling locations are provided in Worksheet Nos. 17 and 18.

Information related to laboratory analyses, including performance criteria; reference limits and evaluations; analytical standard operating procedure (SOP) requirements; SOP references; instrument calibration, maintenance, testing, and inspection; QC samples; and analytical services are presented in Worksheet Nos. 12, 15, 19, 23, 24, 25, 28, and 30, respectively.

Field QC samples are summarized in Worksheet No. 20. Field sampling SOPs are presented in Appendices B through K of this document, and the location of each SOP is identified in Worksheet No. 21. Procedures for the calibration and maintenance of field equipment are presented in Worksheet No. 22. Field sample handling and custody procedures are provided in Worksheet Nos. 26 and 27, respectively.

A summary of the documents and records associated with this QAPP, from field sampling effort to the delivery of the data report, is presented in Worksheet No. 29. Internal and external assessments of the field activities, map preparation, laboratory analytical method compliance, data usability, and document review are described in Worksheet No. 31. Types of findings and corrective action responses are outlined in Worksheet No. 32. A summary of quality assurance (QA) management reports for this QAPP is provided in Worksheet No. 33. Verification of field sampling data, validation of laboratory analytical data, and an assessment of data usability are presented in Worksheet Nos. 34 through 37.

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QAPP Worksheets

QAPP Worksheet #1 (UFP-QAPP Manual Section 2.1): Title and Approval Page

Document Title: Crab and Clam Sampling and Analysis Quality Assurance Project Plan (Crab/Clam QAPP)

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QAPP Worksheet #2 (UFP-QAPP Manual Section 2.2.4): QAPP Identifying Information

Site Name/Project Name: Newark Bay Study Area/Crab and Clam Sampling and Analysis Quality Assurance Project Plan (Crab/Clam QAPP)

Site Location: Newark Bay, New Jersey
Site Number/Code: CERCLA Document No. 02-2004-2010
Operable Unit: 003
Contractor Name: ARCADIS
Contractor Number: Not Applicable (NA)
Contract Title: NA
Work Assignment Number: NA

1. Identify guidance used to prepare QAPP:

   ☐ USEPA, USDOD, and USDOE. 2005. Intergovernmental Data Quality Task Force. Uniform Federal Policy for Quality Assurance Project Plans. Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs. Part 1: UFP-QAPP Manual. USEPA 505-B-04-900A. Final Version 1. March.

2. Identify regulatory program: CERCLA.

3. Identify approval entity: USEPA Region 2

4. Indicate whether the QAPP is a generic or a project-specific QAPP. (circle one)

5. List dates of scoping sessions that were held:

   ☐ February 22, 2013
   ☐ July 22 and 29, 2013

6. List dates and titles of QAPP and Field Sampling Plan (FSP) documents written for previous site work, if applicable:

Tierra. 2005. Newark Bay Study Area Remedial Investigation Work Plan. Sediment Sampling and Source Identification Program, Newark Bay, New Jersey. Phase I. Revision 1. Volumes 1-3. September.
Tierra. 2007. Newark Bay Study Area Remedial Investigation Work Plan. Sediment Sampling and Source Identification Program, Newark Bay, New Jersey. Phase II. Revision 2, Amendment 1. November.
Tierra. 2011. Waste Characterization Quality Assurance Project Plan. Diamond Alkali Superfund Site, Operable Unit 1/CERCLA Non-Time-Critical Removal Action – Lower Passaic River Study Area and Newark Bay Study Area. Revision 1. December.
Tierra. 2012. Quality Assurance Project Plan for Newark Bay Study Area: Multi-beam and Single-beam Bathymetric Survey. Revision 1. December.
Tierra. 2013. Newark Bay Study Area. Reconnaissance Survey Work Plan. Baseline Human Health and Ecological Risk Assessment. September.

QAPP Worksheet #2 (UFP-QAPP Manual Section 2.2.4): QAPP Identifying Information (continued)

Additionally, the following QAPPs and documents were written for similar work on an adjacent site (Lower Passaic River) and were used to guide the scope of this QAPP:

Windward. 2009a. Lower Passaic River Restoration Project. Quality Assurance Project Plan. Fish and Decapod Crustacean Tissue Collection for Chemical Analysis and Fish Community Survey. Final. August 6.
Windward. 2009b. Lower Passaic River Restoration Project. Quality Assurance Project Plan. Surface Sediment Chemical Analyses and Benthic Invertebrate Toxicity and Bioaccumulation and Tissue Testing. Final. October 8.
Windward. 2011. 2009 Fish and Blue Crab Tissue Chemistry Data for the Lower Passaic River Study Area. Prepared for Cooperating Parties Group, Newark, NJ. September.
Tierra Solutions. 1999. Passaic River Study Area Ecological Sampling Plan. Quality Assurance Project Plan. Volume 2 of 6. Tierra Solutions, Inc. Newark, New Jersey.

7. List organizational partners (stakeholders) and connection with lead organization:

   This work will be performed under the requirements of the Administrative Order on Consent (CERCLA-02-2004-2010) (USEPA 2004) for the NBSA, with oversight conducted by USEPA and its partner agencies (NJDEP, NOAA, USFWS, USACE, NJDOT). Tierra (acting as Facility Coordinator), has engaged ARCADIS to prepare this document on its behalf.

8. List data users: see Worksheet #11.

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

QAPP Worksheet #2 (UFP-QAPP Manual Section 2.2.4): QAPP Identifying Information (continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information Relevant to Collection of Tissue and Sediment Data	Crosswalk to QAPP Worksheet No. or Related Documents
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	1
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	2
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	3 4
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	5 6 7 8
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps	9 9 10 Figure 1
2.6 Project Quality Objectives (PQOs) and Measurement Performance Criteria 2.6.1 Development of PQOs Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	11 12
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	13
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks -Reference Limits and Evaluation Table - Project Schedule/Timeline Table	14 15 16

QAPP Worksheet #2 (UFP-QAPP Manual Section 2.2.4): QAPP Identifying Information (continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information Relevant to Collection of Tissue and Sediment Data	Crosswalk to QAPP Worksheet No. or Related Documents
Measurement/Data Acquisition		
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	- Sampling Design and Rationale/Sample Location Map - Sample Locations and Methods/Standard Operating Procedure (SOP) Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control (QC) Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table	17 18 19 20 20 21 22
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	- Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	23 24 25
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample handling and Tracking System 3.3.3 Sample Custody	- Sample Collection Documentation Handling, Tracking, and Custody SOPs - Sample Container Identification - Sample Handling Flo Diagram - Example Chain-of-Custody Form and Seal	26 26 27 27
3.4 QC Samples 3.4.1 Sampling QC Samples 3.4.2 Analytical QC Samples	- QC Sample Tables - Screening/Confirmatory Analysis Decision Tree	28 28
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	- Project Documents and Records Table - Analytical Services Table	29 30

QAPP Worksheet #2 (UFP-QAPP Manual Section 2.2.4): QAPP Identifying Information (continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information Relevant to Collection of Tissue and Sediment Data	Crosswalk to QAPP Worksheet No. or Related Documents
Assessment/Oversight		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	- Assessment and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses	31 31 32
4.2 QA Management Reports	- QA Management Reports Table	33
4.3 Final Project Report		
Data Review		
5.1 Overview 5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment	34 35 36 37

QAPP Worksheet #3 (UFP-QAPP Manual Section 2.3.1): Distribution List

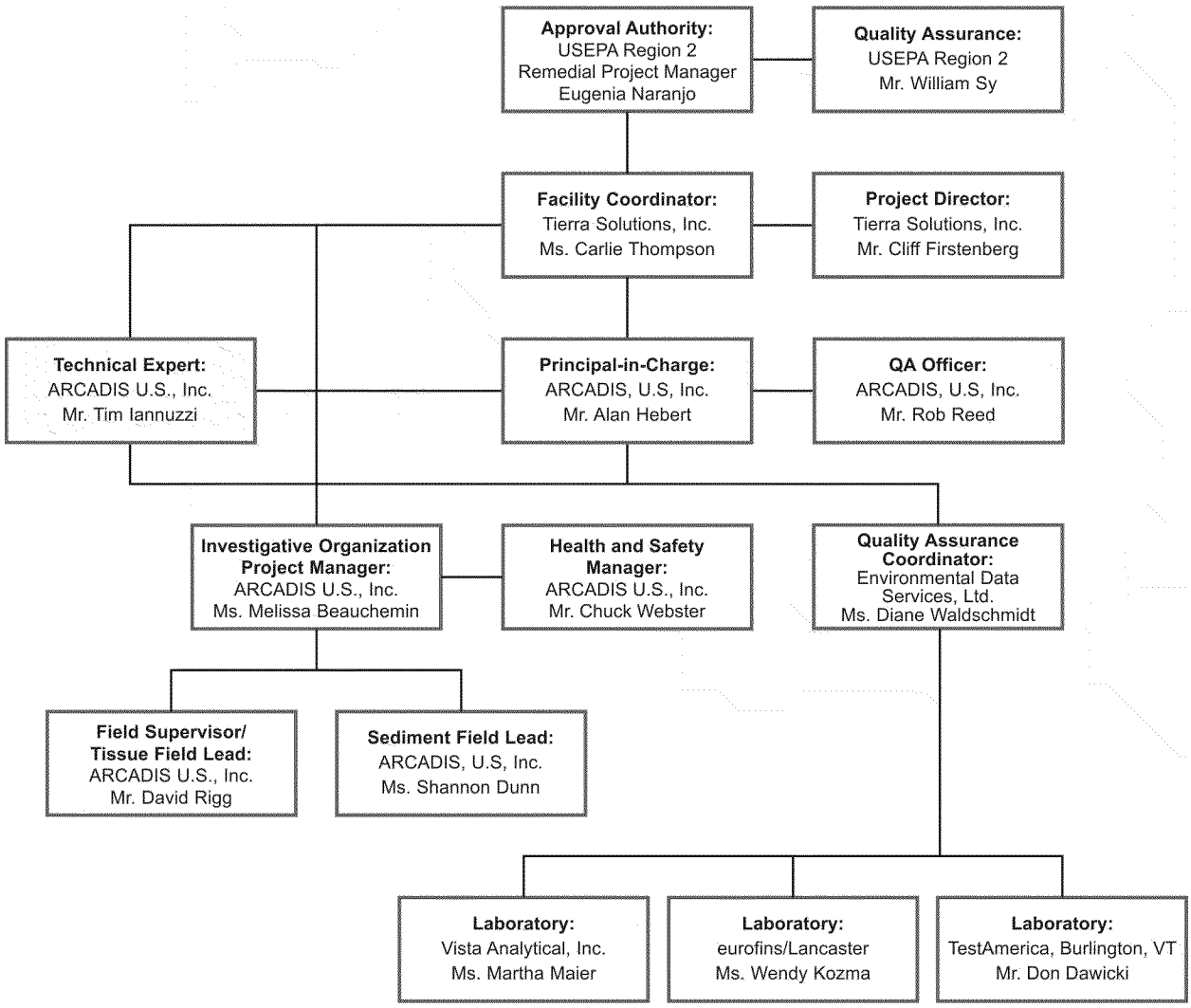
QAPP Recipients	Title	Organization	Telephone Number	Email Address
Eugenia Naranjo	Remedial Project Manager (RPM)	USEPA Region 2	212.637.3467	naranjo.eugenia@epamail.epa.gov
William Sy	USEPA Project QA Officer	USEPA Region 2	732.632.4766	sy.william@epa.gov
Marc Greenberg	Environmental Toxicologist	USEPA Headquarters	732.452.6413	greenberg.marc@epa.gov
Jay Nickerson	Site Remediation Manager	NJDEP	609.633.1448	jay.nickerson@dep.state.nj.us
Tim Kubiak	Assistant Supervisor of Environmental Contaminants	USFWS	609.646.9310, ext. 26	tim_kubiak@fws.gov
Bryce Wisemiller	Project Manager	USACE NY District	917.790.8307	bryce.w.wisemiller@usace.army.mil
Reyhan Mehran	Coastal Resource Coordinator	NOAA	212.637.3257	reyhan.mehran@noaa.gov
Carlie Thompson	Facility Coordinator	Tierra	732.246.5849	carlie.thompson@tierra-inc.com
Clifford Firstenberg	Project Director	Tierra	757.258.7720	clifford.firstenberg@tierra-inc.com
David Rigg	Field Supervisor	ARCADIS	518.250.7379	david.rigg@arcadis-us.com
Shannon Dunn	Sediment Field Lead	ARCADIS	206.726.4708	shannon.dunn@arcadis-us.com
Melissa Beauchemin	Project Manager	ARCADIS	978.322.4551	melissa.beauchemin@arcadis-us.com
Tim Iannuzzi	Technical Expert	ARCADIS	410.295.1205	tim.iannuzzi@arcadis-us.com
Alain Hebert	Principal-in-Charge	ARCADIS	609.366.9032	alain.hebert@arcadis-us.com
Chuck Webster	Health and Safety Manager	ARCADIS	315.671.9657	chuck.webster@arcadis-us.com
Rob Reed	Quality Assurance Officer	ARCADIS	315.671.9457	rob.reed@arcadis-us.com
Diane Waldschmidt	Quality Assurance Coordinator	Environmental Data Services, Ltd.	412.486.6989	dwaldschmidt@eds-us.net
Don Dawicki	Laboratory Project Manager	TestAmerica, Burlington, VT	802.923.1026	don.dawicki@testamericainc.com
Martha Maier	Laboratory Project Manager	Vista Analytical Laboratory	916.673.1520	mmaier@vista-analytical.com
Wendy Kozma	Laboratory Project Manager	eurofins/Lancaster Laboratories	717.656.2300 ext. 1522	wkozma@lancasterlabs.com

QAPP Worksheet #4 (UFP-QAPP Manual Section 2.3.2): Project Personnel Sign-Off Sheet

Project Personnel	Title	Organization	Telephone Number	Signature	Date QAPP Read

Note:
*Signature indicates that personnel have read the applicable QAPP sections and will perform the tasks as described.

QAPP Worksheet #5 (UFP-QAPP Manual Section 2.4.1): Project Organizational Chart



QAPP Worksheet #6 (UFP-QAPP Manual Section 2.4.2): Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (e.g., timing, pathways)
Field Sampling Communication	Field Supervisor	David Rigg	518.250.7379	Communicate daily, or as needed, with field personnel, Investigative Organization Project Manager, and QA Coordinator directly or via email or phone.
Communication with Investigative Organization Project Manager				
Communication with QA Coordinator				
Health and Safety Communication	Site Safety and Health Officer	David Rigg	518.250.7379	Communicate daily, or as needed, with field personnel directly, or via email or phone, on matters regarding health and safety.
Communication with Investigative Organization Project Manager	Facility Coordinator	Carlie Thompson	732.246.5849	Communicate daily, or as needed, with Investigative Organization Project Manager, and QA Coordinator directly or via email or phone.
Communication with Investigative Organization QA/QC Manager				
Communication with Facility Coordinator	Investigative Organization Project Manager	Melissa Beauchemin	978.322.4551	Communicate daily, or as needed, with Facility Coordinator via email or phone.
	QA Coordinator	Diane Waldschmidt	412.486.6989	
Communication with Analytical Laboratories	Investigative Organization Project Manager	Melissa Beauchemin	978.322.4551	Communicate, as needed, with Laboratory Project Managers regarding laboratory and analysis issues.
	QA Coordinator	Diane Waldschmidt	412.486.6989	
	Investigative Organization Biological Laboratory Coordinator	David Rigg	518.250.7379	

QAPP Worksheet #6 (UFP-QAPP Manual Section 2.4.2): Communication Pathways (continued)

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (e.g., timing, pathways)
Communication with USEPA	Facility Coordinator	Carlie Thompson	732.246.5849	Communicate with USEPA Project Manager, as needed, via email or phone.
Quality Status and Issues	QA Coordinator	Diane Waldschmidt	412.486.6989	Communicate with Facility Coordinator, as needed, via email or phone.
Sampling Vessel Operations	Field Supervisor	David Rigg	518.250.7379	Communicate with vessel captain regarding safety issues.

QAPP Worksheet #7 (UFP-QAPP Manual Section 2.4.3): Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications[a]
Carlie Thompson	Facility Coordinator	Tierra Solutions, Inc.	Program Manager	BS Chemical Engineering/9 years
Cliff Firstenberg	Project Director	Tierra Solutions, Inc.	Environmental Sciences Manager	MS Physical Oceanography, BA Earth and Planetary Sciences/30 years
Alain Hebert	Program Manager	ARCADIS U.S., Inc.	Principal-in-Charge	MS Environmental Engineering/20 years
Tim Iannuzzi	Technical Manager	ARCADIS U.S., Inc.	Senior Technical Expert	MS Biology, BS Biology/24 years
Melissa Beauchemin	Project/Task Manager	ARCADIS U.S., Inc.	Project/Task Manager	MS Environmental Science, BS Biology/11 years
David Rigg	Field Supervisor	ARCADIS U.S., Inc.	Senior Field Scientist / Technician Health and Safety Supervisor / Field Supervisor	MS Environmental Science, BS Environmental Science, AAS Environmental Ecological Toxicology/24 years
Shannon Dunn	Field Advisor (Sediment)	ARCADIS U.S., Inc.	Senior Field Advisor (Sediment)	MS Marine Environmental Science, BS Geology/14 years
Chuck Webster	Health and Safety Manager	ARCADIS U.S., Inc.	Health and Safety Advisor	MS Safety, BS Political Science/20 years
Dave Buys	Field Technician	ARCADIS U.S., Inc.	Field Scientist/Technician	MS Aquatic Ecology, BS Fisheries and Wildlife /16 years
Jason Vogel	Field Technician	ARCADIS U.S., Inc.	Field Scientist/Technician	BS Chemical Engineering, Environmental Engineering/15 years
Matt Frackelton	Field Technician	ARCADIS U.S., Inc.	Field Scientist/Technician	BA Anthropology, BS Political Science/9 years
Jessie Bluvias	Field Technician	ARCADIS U.S., Inc.	Field Scientist/Technician	MEM Coastal Environmental Management, BS Ecology and Natural Resources/3 years
Diane Waldschmidt	Director	Environmental Data Services, Ltd.	Quality Assurance Coordinator	BS Chemistry/22 years
Elise Francken	Senior Technical Specialist	Environmental Data Services, Ltd.	Quality Assurance Assistant	BS Chemistry/16 years
Don Dawicki	Customer Service/Project Manager	TestAmerica, Burlington, VT	Laboratory Project Manager	18 years
Martha Maier	Laboratory Manager	Vista Analytical, Inc.	Laboratory Project Manager	BS Chemistry/23 years
Wendy Kozma	Project Manager	eurofins/Lancaster Laboratories	Laboratory Project Manager	BS Environmental Science/21 years

Note:
[a] Resumes of all individuals are available upon request.

QAPP Worksheet #8 (UFP-QAPP Manual Section 2.4.4): Special Personnel Training Requirements Table

All field personnel assigned to the site must have successfully completed 40 hours of training for hazardous site work in accordance with Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120(e)(3) and be current with their 8-hour refresher training in accordance with OSHA 29 CFR 1910.120(e)(8). Documentation of OSHA training is required prior to personnel being permitted to work on site.

Personnel managing or supervising work on site will also have successfully completed 8 hours of Manager/Supervisor Training in accordance with OSHA 29 CFR 1910.120(e)(4).

Personnel assigned to the site must be enrolled in a medical surveillance program, which meets the requirements of OSHA 29 CFR 1910.120(f). Personnel must have successfully passed a periodic occupational physical and be medically cleared to work on a hazardous waste site and capable of wearing appropriate personal protective equipment. For employees potentially exposed more than 30 days per year, the frequency of periodic examinations will be annual. For employees potentially exposed less than 30 days per year, the frequency for periodic examinations will be once every 24 months.

It is the responsibility of the site worker's employer to provide their employees with the required training and medical monitoring prior to assigning them to work at the site. Each employer will be responsible for providing documentation of training and monitoring to the Field Supervisor prior to sending their employees to the site to work.

Additional health and safety information and records documentation information are detailed in the site-specific Health and Safety Plan.

QAPP Worksheet #9 (UFP-QAPP Manual Section 2.5.1): Project Scoping Session Participants Sheet, February 22, 2013

Project Name: Crab/Clam Sampling **Projected Date(s) of Sampling:** Fall 2013 **Project Managers:** Carlie Thompson (Tierra) /Melissa Beauchemin (ARCADIS)	**Site Name:** NBSA **Site Location:** Newark Bay, New Jersey
Date of Session: February 22, 2013 **Scoping Session Purpose:** To discuss measurement endpoints for the upcoming baseline ecological risk assessment of the NBSA	

Name	Affiliation	Phone #	Email Address	Project Role
Melissa Beauchemin	ARCADIS	978-322-4551	melissa.beauchemin@arcadis-us.com	Project Manager
Carlie Thompson	Tierra	732-246-5849	carlie.thompson@tierra-inc.com	Facility Coordinator
Cliff Firstenberg	Tierra	757-258-7720	clifford.firstenberg@tierra-inc.com	Project Director
Eugenia Naranjo	USEPA Reg 2	212-637-3467	naranjo.eugenia@epa.gov	Remedial Project Manager
Chuck Nace	USEPA Reg 2	212-637-4164	nace.charles@epa.gov	Ecological Risk Assessor
Marc Greenberg	USEPA Headquarters	732-452-6413	greenberg.marc@epa.gov	Ecological Risk Assessor
Norman Richardson	Battelle	781-869-1417	richardsonn@battelle.org	Contractor
Len Warner	Louis Berger Group	914-798-3721	lwarner@louisberger.com	Contractor
Tim Iannuzzi	ARCADIS	410-991-9754	tim.iannuzzi@arcadis-us.com	Technical Expert

Comments/Decisions:
- Refer to Meeting Minutes

QAPP Worksheet #9 (UFP-QAPP Manual Section 2.5.1): Project Scoping Session Participants Sheet, July 22, 2013

Project Name: Crab/Clam Sampling **Projected Date(s) of Sampling:** Fall 2013 **Project Managers:** Carlie Thompson (Tierra)/Melissa Beauchemin (ARCADIS)	**Site Name:** NBSA **Site Location:** Newark Bay, New Jersey
Date of Session: July 22, 2013 **Scoping Session Purpose:** Discuss sampling and analytical methods.	

Name	Affiliation	Phone #	Email Address	Project Role
Melissa Beauchemin	ARCADIS	978-322-4551	melissa.beauchemin@arcadis-us.com	Project Manager
David Rigg	ARCADIS	518-250-7379	david.rigg@arcadis-us.com	Field Supervisor
Tim Iannuzzi	ARCADIS	410-295-1205	tim.iannuzzi@arcadis-us.com	Technical Expert
Carlie Thompson	Tierra	732-246-5849	carlie.thompson@tierra-inc.com	Facility Coordinator
Cliff Firstenberg	Tierra	757-258-7720	clifford.firstenberg@tierra-inc.com	Project Director
Diane Waldschmidt	EDS	412-486-6989	dwaldschmidt@eds-us.net	QA Coordinator
Mark Harris	ToxStrategies	281-394-1567	mharris@toxstrategies.com	Human Health Risk Assessor

Comments/Decisions:

- Laboratories: decision to use Tierra laboratories (likely two or three laboratories).
- Analyte list: decision to keep in everything (include herbicides, Aroclors, and butyltins).
- VOCs do not accumulate in tissue, can eliminate from tissue. VOCs in sediment are limited (most screened out – see Problem Formulation Table 4-2) but will keep in for HHRA.
- Discuss process for co-located sediment/clam samples – composites based on areas where clams dug.
- Tissue processing to be done in laboratory with oversight by ARCADIS biologist.
- Discussed numbers of organisms to make composite to get enough tissue for all analyses (base on memorandum written for Passaic).

QAPP Worksheet #9 (UFP-QAPP Manual Section 2.5.1): Project Scoping Session Participants Sheet, July 29, 2013

Project Name: Crab/Clam Sampling **Projected Date(s) of Sampling:** Fall 2013 **Project Managers:** Carlie Thompson (Tierra) /Melissa Beauchemin (ARCADIS)	**Site Name:** NBSA **Site Location:** Newark Bay, New Jersey
Date of Session: July 29, 2013 **Scoping Session Purpose:** Discuss sampling and analytical methods	

Name	Affiliation	Phone #	Email Address	Project Role
Melissa Beauchemin	ARCADIS	978-322-4551	melissa.beauchemin@arcadis-us.com	Project Manager
Carlie Thompson	Tierra	732-246-5849	carlie.thompson@tierra-inc.com	Facility Coordinator
Cliff Firstenberg	Tierra	757-258-7720	clifford.firstenberg@tierra-inc.com	Project Director
Paul Bluestein	Tierra	732-246-3091	pjbluestein@tierra-inc.com	Alternate Facility Coordinator
Mark Harris	ToxStrategies	281-394-1567	mharris@toxstrategies.com	Human Health Risk Assessor
Diane Waldschmidt	EDS	412-486-6989	dwaldschmidt@eds-us.net	QA Coordinator
Elise Francken	EDS	412-486-6989	efrancken@eds-us.net	Data Validator/QA Assistant

Comments/Decisions:

- Estimated sample size for tissue for each analysis – total is approximately 200 grams.
- Need to prioritize analyte list (develop hierarchy).
  - Remove VOCs and TPH from tissue.
  - Remove herbicides from tissue (lack of methods).
  - Remove hexavalent chromium from tissue.
- The need for Aroclors was discussed. ARCADIS recommends keeping them in the analyte list for trends analysis, but as in the lower Passaic they will not be used in the risk assessment (PCB congener data are better for use in risk assessment). They were maintained in lower Passaic River; however, few data have been collected in NBSA for trend analysis.

QAPP Worksheet #10 (UFP-QAPP Manual Section 2.5.2): Problem Definition

The problem to be addressed by the project:

An understanding of the concentration of COPECs in benthic invertebrate tissues (specifically bivalve clams and blue crabs) and surficial sediments.

The environmental questions being asked:

1) Does exposure to site-related COPECs in the NBSA pose unacceptable risks to benthic macroinvertebrates (represented by blue crab and bivalve clams)?
2) Does the consumption of shellfish from the NBSA pose unacceptable risks to human and ecological wildlife receptors?
3) Does exposure to surface sediments in the intertidal areas of the NBSA pose unacceptable risks to human and ecological receptors?

Observations from any site reconnaissance reports:

No observations are available at this time, although a Reconnaissance Survey occurred in September, the results of which will be provided prior to the implementation of this QAPP.

A synopsis of secondary data or information from site reports:

The Intertidal Areas have been mapped and are depicted on Figure 1.

The classes of contaminants and the affected matrices:

There are several different classes of organic and inorganic contaminants in the NBSA, many of which may accumulate in crabs and clams. Crab (muscle-only and hepatopancreas-only samples) and clam tissue samples will be analyzed for the following analytes: polychlorinated biphenyl (PCB) congeners, PCB Aroclors, PCDDs/PCDFs, pesticides, polycyclic aromatic hydrocarbons (PAHs, including alkylated PAHs), semivolatile organic compounds (SVOCs, including phthalates), metals (including mercury, methylmercury, and butyltins), lipid content, and percent moisture. Worksheet No. 15 lists the specific analytes in each of these chemical classes that will be analyzed. Although volatile organic compounds (VOCs) and herbicides are identified as possible COPECs, VOCs were not identified as bioaccumulative chemicals by USEPA (2000a). Therefore, VOCs will not be analyzed in tissue samples. Herbicides are not included for analysis in tissue for the following reasons: 1) there are no published methods for herbicides in tissue, 2) herbicides are infrequently detected in recent studies, and 3) the likely levels of detection are below levels to be toxic to wildlife and the bioaccumulation potential is low.

Co-located surficial sediment samples will be analyzed for the same constituents as the tissue samples, as well as VOCs, herbicides, hexavalent chromium, total extractable petroleum hydrocarbons (TEPH), acid volatile sulfides/simultaneously extracted metals (AVS/SEM), cyanide, sulfide, Total Kjeldhal Nitrogen (TKN), total phosphorus, grain size, total organic carbon (TOC), percent moisture, pH, ammonia and oxidation reduction potential (ORP).

The analysis of PCB Aroclors is being conducted to provide data comparable to historical PCB Aroclor data for the purposes of trend analysis on the basis of PCB Aroclors. The PCB Aroclor data will not be used in either the HHRA or BERA due to the superior accuracy and precision of the PCB congener data. Total PCB concentrations will be calculated as the sum of PCB congeners and not the sum of PCB Aroclors.

QAPP Worksheet #10 (UFP-QAPP Manual Section 2.5.2): Problem Definition (continued)

The rationale for inclusion of chemical and non-chemical analyses:

Sediment and tissue COPEC concentrations will provide valuable information that will be used to perform the BERA and BHHRA for the NBSA.

Project decision conditions:

The conditions for project decisions (i.e., those decisions that may require communication between ARCADIS, Tierra, and USEPA during the field event) include the identification of species targeted for collection, the appropriate size of those species for collection, the prioritization of chemical analyses if insufficient tissue is collected, the prioritization for compositing (if necessary) in a given sampling area, and the need to relocate sampling locations.

The targeted sizes for each species collected will be based on relevancy of each species to the BERA and the BHHRA (presented in Worksheet No. 11). If there is a change in the target size of species, based on the Reconnaissance Survey or any initial field work, Tierra will be contacted immediately.

A pre-homogenization minimum tissue mass of 232 grams (g) and a post-homogenization mass of 207 g is needed, per sample, for analysis of all proposed chemical groups. A mass of 25 g was added to the sum of the minimum mass requirements for chemical analyses (207 g) to account for tissue lost during processing and homogenization, for a total pre-homogenization minimum mass of 232 g. The minimum mass requirements per analytical group are provided in the priority list below. Mass requirements have been optimized with each analytical laboratory, such that they are the lowest required to achieve the detection limits presented in Worksheet No. 15. The minimum mass does not include enough mass for re-extractions or matrix-specific quality control samples. If the pre-homogenization minimum tissue mass (232 g) for chemistry analysis cannot be obtained for a given species/location, the field effort will cease. If a post-homogenization minimum mass of 207 g is not obtained, the following priority list for the chemical analyses of tissue samples will be considered in conjunction with available sediment chemistry data collected:

1. PCDDs/PCDFs including percent lipids (50-g minimum mass)
2. PCB congeners (50-g minimum mass)
3. TAL metals including mercury and titanium (6-g minimum mass)
4. Methylmercury (1-g minimum mass)
5. Pesticides (15-g minimum mass)
6. Percent moisture (10-g minimum mass)
7. Semivolatile organics (15-g minimum mass)
8. Semivolatile organics SIM (15-g minimum mass)
9. Butyltins (30-g minimum mass)
10. PCB Aroclors (15-g minimum mass)

The targeted sample size for composite surface sediment samples is a total weight of 843 g for the following analytical requirements:

- PCDDs/PCDFs (30-g minimum mass)
- PCB congeners (30-g minimum mass)
- TAL metals and mercury and titanium (30-g minimum mass)
- Methylmercury (3-g minimum mass)
- Butyltins (30-g minimum mass)

QAPP Worksheet #10 (UFP-QAPP Manual Section 2.5.2): Problem Definition (continued)

- ☐ Chlorinated herbicides (50-g minimum mass)
- ☐ Semivolatile organics (30-g minimum mass)
- ☐ Semivolatile organics SIM (30-g minimum mass)
- ☐ PCB Aroclors (15-g minimum mass)
- ☐ Pesticides (15-g minimum mass)
- ☐ Volatile organics (15-g minimum mass)
- ☐ Cyanide (10-g minimum mass)
- ☐ TOC (10-g minimum mass)
- ☐ TEPH (30-g minimum mass)
- ☐ Hexavalent chromium (5-g minimum mass)
- ☐ pH (25-g minimum mass)
- ☐ Oxidation Reduction Potential (100-g minimum mass)
- ☐ Percent moisture (10-g minimum mass)
- ☐ Total sulfide (15-g minimum mass)
- ☐ Total phosphorus (20-g minimum mass)
- ☐ Total Kjedahl Nitrogen (20-g minimum mass)
- ☐ AVS/SEM (60-g minimum mass)
- ☐ Ammonia (10-g minimum mass)
- ☐ Grain size (250-g minimum mass)

QAPP Worksheet #11 (UFP-QAPP Manual Section 2.6.1): Project Quality Objectives/Systematic Planning Process Statements

Who will use the data?

The data collected under this QAPP will be used by Tierra and USEPA for CERCLA-related decisions, specifically for the BERA and the BHHRA, and by other interested parties (e.g., USACE, NJDEP, USFWS, NJDOT, NOAA) for other purposes, such as restoration planning.

What will the data be used for?

The data collected during this sampling effort will be used in risk-based decision-making for the RI/FS in the NBSA. Specifically, the data will be used to estimate potential human health and ecological risks to receptors that may be exposed to COPECs in the NBSA. The results of the baseline risk assessments will be used to inform remedial decision-making under CERCLA/National Contingency Plan and other appropriate regulations and future restoration planning.

BERA Assessment Endpoints:

Assessment Endpoint No. 1 – Survival and/or growth of aquatic plants and maintenance of plants as a food resource and habitat for fish and wildlife

Sediment chemistry data collected as part of this sampling event will be used as a measurement endpoint to evaluate risks to aquatic plants by answering the following risk question: **Are COPEC concentrations in sediments from the NBSA greater than benchmarks for the survival and/or growth of aquatic plants?** To do this, measured chemical concentrations in sediment will be compared to toxicological benchmarks.

Assessment Endpoint No. 2 – Survival, growth, and/or reproduction of invertebrates

Tissue chemistry data collected as part of this sampling event will be used as one measurement endpoint to evaluate risks to benthic invertebrates by answering the following risk question: **Are COPEC residues in invertebrate tissues from the NBSA greater than tissue benchmarks (e.g., critical body residues [CBRs]) for the survival, growth, and/or reproduction of invertebrates?** To do this, measured tissue chemical concentrations in blue crabs and clams will be compared to CBRs. As part of the assessment, whole body crab COPEC concentrations will be estimated by combining the results of the edible tissue and hepatopancreas tissue samples.

Sediment chemistry data collected as part of this sampling event will be used as one measurement endpoint to evaluate risks to benthic invertebrates by answering the following risk question: **Are COPEC concentrations in sediments from the BAZ greater than benchmarks for the survival, growth, and/or reproduction of invertebrates?** To do this, measured chemical concentrations in sediment will be compared to toxicological benchmarks.

Assessment Endpoints No. 3 and 4 – Survival, growth, and/or reproduction of fish and reptiles

Sediment chemistry data collected as part of this sampling event will be used as a measurement endpoint to evaluate risks to fish and reptiles by answering the following risk question: **Are COPEC concentrations in site sediment at levels that might adversely affect survival, growth, and/or reproduction of reptiles and fish?** To do this, measured chemical concentrations in sediment will be compared to toxicological benchmarks.

QAPP Worksheet #11 (UFP-QAPP Manual Section 2.6.1): Project Quality Objectives/Systematic Planning Process Statements (continued)

Assessment Endpoints No. 5 and 6 – Survival, growth, and/or reproduction of birds and mammals

Tissue and sediment chemistry data collected as part of this sampling event will be used as input parameters for a food web model to estimate dietary intakes for select bird and mammal receptors by answering the following risk question: **Does the daily dose of COPECs received by birds and mammals from consumption of the tissues of prey species and from other media in the NBSA exceed the toxicity reference values (TRVs) for survival, growth, and/or reproduction of birds and mammals? If yes, what are the probabilities of effects of differing magnitude for survival, growth, and/or reproduction of birds and mammals?** To do this, measured chemical concentrations in sediment and clam and crab tissue will be incorporated into a deterministic web model to estimate a daily dietary ingestion of chemicals. This value is then compared to the literature-based TRV to evaluate potential risks. If potential risks are evident (i.e., the resulting hazard quotient [HQ] is greater than 1), a probabilistic food web model will be run to estimate the range of possible risks.

HHRA Endpoints

The data collected during this sampling effort will also be used to support the BHHRA. In addition to the BERA risk questions outlined above, the BHHRA risk question relevant to this project is "What are the potential adverse effects of chemicals in the NBSA to human health under current and future exposure scenarios for both cancer and non-cancer health effects via shellfish consumption from the NBSA?"

For blue crabs, individual tissue type (muscle/hepatopancreas combined, hepatopancreas-only, and muscle-only) chemistry data will be collected. Muscle-only and hepatopancreas-only samples will be analyzed. The data will be combined to estimate a whole body concentration. The BHHRA will use data from combined blue crab muscle/hepatopancreas samples as the basis for quantitatively evaluating the RME of individuals under current and future exposure scenarios for both cancer and non-cancer health effects, following USEPA Superfund guidance, guidelines, and policies. Risks associated with the consumption of hepatopancreas-only and muscle-only tissue will be discussed qualitatively in the uncertainty section of the BHHRA.

What type of data are needed (matrix, target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)?

The number of clams and crabs in each composite sample will vary to meet the anticipated minimum sample mass requirements (232 g pre-homogenization, 207 g post-homogenization).

Sediment composite samples must meet the minimum sample size of 843 g total.

The targeted size range for blue crab is at least 4.5 inches (measured as the carapace width), based on the minimum legal catch size and depending on shed stage. The legal minimum is 3 inches for shedders, 3.5 inches for softshell, and 4.5 inches for hardshell (http://www.eregulations.com/newjersey/fishing/saltwater/marine-regulations-mollusks-crustaceans/). Crabs will be composited by intertidal location. However, in the event that sufficient hepatopancreas tissue cannot be collected to obtain the required tissue mass for analysis from one Intertidal Area, hepatopancreas tissue from more than one sampling location within a zone (i.e., North, Central, South) will be combined.

QAPP Worksheet #11 (UFP-QAPP Manual Section 2.6.1): Project Quality Objectives/Systematic Planning Process Statements (continued)

The targeted size of clams will be determined after the Reconnaissance Survey evaluates the dominant species and size of clams in the NBSA; however, the minimum size will be 1.5 inches per New Jersey regulation (http://www.eregulations.com/newjersey/fishing/saltwater/marine-regulations-mollusks-crustaceans/). Clams will be composited by species and location.

The sizes of all blue crabs and clams collected for each sample will be evaluated prior to compositing (if necessary), and individuals included in a given composite will be of similar size so that the smallest individual in a composite is no less than 75% of the length of the largest individual (USEPA 2000b). This target size requirement will be evaluated during the sampling event in conjunction with Tierra to determine if the range of individual sizes included in a composite needs to be increased or decreased to accommodate the level of effort of the sampling event. When possible, composite crabs will consist of approximately equal portions of each gender.

Crab and clam samples will be submitted on wet ice to the laboratory directly from the field. Upon receipt at the processing laboratory, clams will be frozen overnight and shucked the next day. Tissue from each composite sample will be homogenized. Composite crab samples will be processed by removing all soft (edible) tissue and hepatopancreas, which will be homogenized separately. Tissue homogenate will be shipped, as necessary, to subcontracted laboratories for analysis either on wet or dry ice.

The specific methodology and sampling equipment used for collecting sediment samples will be determined after experimental sampling during the Reconnaissance Survey. Sediment samples will be homogenized and composited in the field and shipped on wet ice to the laboratory directly from the field.

How "good" do the data need to be in order to support the environmental decision?

The data will be used to support decisions about the magnitude and spatial distribution of risks to human and ecological health. The data will be used to better define risk decisions for discrete endpoints. The data may also be used to support initial investigations of potential remedial options. Consequently, the data need to be collected with a design that specifically addresses the questions that are being posed (see above, "What will the data be used for?"). The conceptual framework of the design is more important than the actual number of samples collected because it is this framework that will be used to extrapolate from the data that are collected in order to draw conclusions about risks at the site.

It is inevitable that some tissue-sediment relationships will be highly variable, while others will not. However, if the data have been collected with a logical design to cover the range of variation in the controlling physical factors at the site, the interpretation of the results is more straightforward, even when the variance of relationships is high. Because a number of assumptions will be made when assessing risks, the data must be collected in a way that makes the evaluation of assumptions possible and allows assessments of the remaining uncertainty to be conducted in a way that enables decision makers to weigh the costs and benefits of proceeding with remedial action decisions.

How much data are needed (number of samples for each analytical group, matrix, and concentration)?

The overall number of crab and clam tissue samples (and co-located sediment samples) was identified based on the number of Intertidal Areas in the NBSA, recognizing that additional samples will be collected from other areas of the NBSA as part of subsequent sampling efforts in 2014.

The number of clams and crabs in each composite sample will vary to meet the anticipated minimum sample mass requirements (232 g pre-homogenization, 207 g post-homogenization) but is expected to be at least 25 blue crabs and 20 clams per sample.

QAPP Worksheet #11 (UFP-QAPP Manual Section 2.6.1): Project Quality Objectives/Systematic Planning Process Statements (continued)

Sediment composite samples must meet the minimum sample size of 843 g total.

The sizes of all blue crabs and clams collected for each sample will be evaluated prior to compositing (if necessary), and individuals included in a given composite will be of similar size so that the smallest individual in a composite is no less than 75% of the length of the largest individual (USEPA 2000b). This target size requirement will be evaluated during the sampling event in conjunction with Tierra to determine if the range of individual sizes included in a composite needs to be increased or decreased to accommodate the level of effort of the sampling event. When possible, composite crabs will consist of approximately equal portions of each gender.

The targeted size range for blue crab is at least 4.5 inches (measured as the carapace width), based on the minimum legal catch size and depending on shed stage. The legal minimum is 3 inches for shedders, 3.5 inches for softshell, and 4.5 inches for hardshell (http://www.eregulations.com/newjersey/fishing/saltwater/marine-regulations-mollusks-crustaceans/). Crabs will be composited by intertidal location. However, in the event that sufficient hepatopancreas tissue cannot be collected to obtain the required tissue mass for analysis from one Intertidal Area, hepatopancreas tissue from more than one sampling location within a zone (i.e., North, Central, South) will be combined.

The targeted size of clams will be determined after the dominant species and size of clams in the NBSA is evaluated during the Reconnaissance Survey; however, the minimum size will be 1.5 inches per New Jersey regulation (http://www.eregulations.com/newjersey/fishing/saltwater/marine-regulations-mollusks-crustaceans/). Clams will be composited by species and location.

Where, when, and how should the data be collected/generated?

The data will be collected from the Intertidal Areas of the NBSA. In late fall, as the weather and water temperatures cool, clams will burrow deep into the sediments, which may prevent them from being collected. In addition, as the weather and water temperatures cool, blue crabs seek deeper water, such as in the navigation channels or farther out into the New York/New Jersey Harbor. As such, these tissue data should be collected no later than mid-October in order to successfully obtain adequate tissue mass.

Samples will be collected by locating the Intertidal Area for sampling, through maps, and GPS coordinates (Figure 1). Upon reaching each area identified for sampling, locational information will be recorded on the field datasheet; a GPS will be used to record the location; general observations about the Intertidal Area will be documented, including presence/absence of vegetation, general substrate appearance, and texture; tidal stage; and time on station will be recorded. At least three baited crab traps will be deployed so that they are completely submerged underwater, equally spaced in the shallow Subtidal Flats surrounding the Intertidal Area. The time of release and GPS coordinates will be recorded.

Each Intertidal Area will be explored for the presence of clam holes. A clam rake and/or shovel will be used to locate buried clams. Holes will be dug until the required number of clams (at least 20) is collected. Clams will be identified, measured, and weighed. Once the minimum required weight (232 g) or number (e.g., 20) of a single species is met, clams will be composited by species and placed into appropriate-labeled sampling containers and stored on wet ice for shipment to the analytical laboratory. Clams not meeting the required length (1.5 inches) will be left at the site.

Following clam collection, a grab sample will be collected from the top 6 inches of sediment next to the first location of the retained clam. This sediment will be used for, but not be limited to, analyses of VOC, AVS/SEM, sulfide, and cyanide. Sediment will be placed in the appropriate sample containers with no headspace, preservative (as required), and sealed per the SOP. Additional sediment will be collected from the same area as clams were collected, homogenized, placed in the appropriate-labeled sample containers, and stored on wet ice for shipment to the analytical laboratory for the remaining chemical analyses. The specific methodology and sampling equipment used will be determined after experimental sampling during the Reconnaissance Survey.

QAPP Worksheet #11 (UFP-QAPP Manual Section 2.6.1): Project Quality Objectives/Systematic Planning Process Statements (continued)

Crab traps will be checked at least every 12 hours. Hard-shelled blue crabs meeting the required size limit (4.5 inches) will be inspected for gross anomalies. Whole, intact crabs retained for analysis will be measured, weighed, documented, euthanized, placed into the appropriate-labeled sampling container, and stored on wet ice for shipment to the analytical laboratory. At least 25 crabs per sample are required to obtain enough tissue mass for analysis.

Who will collect and generate the data?

As described in Worksheet No. 7, ARCADIS will provide the field sampling coordination and personnel required to conduct the tissue and sediment collection efforts. EDS will provide laboratory coordination and support.

How will the data be reported?

Daily activities will be communicated (e.g., telephone conversation, email) to Project Managers and the Facility Coordinator, as described in Worksheet No. 6.

An electronic database that includes the coordinates for the collection of each sediment and tissue sample will be provided. The database will include time of crab trap deployment and retrieval; time of collection; depth of clam collection or trap location; and species, length, and weight. Gender of all individual crabs collected for analysis will also be provided.

A data report(s) summarizing the sediment and tissue collection and analysis results will be provided 90 days after receipt of all Phase III validated chemical data. In addition, this report will include maps that present the sampling locations.

How will the data be archived?

Data records, forms, and notes will be scanned and stored electronically in a project file. Hard copies will be archived in Tierra's main office in East Brunswick, New Jersey. Similarly, the data reports will be issued and then archived electronically and as hard copies. The analytical results will also be provided, as electronic data deliverables (EDDs), to the project database. Multimedia electronic data deliverables (MEDDs) will be provided to USEPA Region 2 by EDS in their required format.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	PCDDs/PCDFs
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-1	Accuracy/Bias	All target compound concentrations must fall within range provided in Table 6 of L-1	Ongoing Precision and Recovery	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Accuracy/Bias	Per L-1	Initial Calibration	A
		Accuracy/Bias	Per L-1	Calibration Verification	A
		Accuracy/Bias	Per L-1	Labeled Compound Spike	A
		Accuracy/Bias	Per L-1	Labeled Internal Standards	A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (Continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	PCB Congeners
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-2	Accuracy/Bias	All target compound analytes contained in the OPR must have observed concentration values that fall within the acceptance ranges provided in Table 6 of L-2	Ongoing Precision and Recovery	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Accuracy/Bias	Per L-2	Initial Calibration	A
		Accuracy/Bias	Per L-2	Calibration Verification	A
		Accuracy/Bias	Per L-2	Labeled Compound Spike	A
		Accuracy/Bias	Per L-2	Labeled Internal Standards	A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	TAL Metals, Titanium
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-3, L-29	Accuracy/Bias	**Compound** All target analyes	**% Recovery** 80-120	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Instrument Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Mercury
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-4	Accuracy/Bias	% Recovery 80-120	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks and Instrument Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Methylmercury
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-5	Accuracy/Bias	% Recovery 67-133	Ongoing Precision and Recovery	A
		Accuracy/Bias	Per L-5	Initial Precision and Recovery	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks, Trip Blanks[d] and Instrument Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Accuracy/Bias	Per L-5	Initial Calibration	A
		Accuracy/Bias	Per L-5	Calibration Verification	A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Butyltins
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-6	Accuracy/Bias	**Surrogate Compounds** Tripentyltin	**% Recovery** 15-150	Surrogate Spikes	A
		Accuracy/Bias	**Compound** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 10-48 30-150 30-150 30-150	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Chlorinated Herbicides
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-7	Accuracy/Bias	**Surrogate Compounds** DCAA	**% Recovery** 50-120	Surrogate Spikes	A
		Accuracy/Bias	**Compound** All target analytes	**% Recovery** 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Semivolatile Organics
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-8	Accuracy/Bias	**Surrogate Compounds**	**% Recovery[d]**	Surrogate Spikes	A
			Phenol-d_5	10-110		
			2-Fluorobiphenyl	45-110		
			2-Fluorophenol	11-110		
			2,4,6-Tribromophenol	44-131		
			Nitrobenzene-d_5	35-110		
			p-Terphenyl-d_{14}	49-120		
		Accuracy/Bias	**Compound**	**% Recovery**	Laboratory Control Sample	A
			All target analytes	20-150		
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Semivolatile Organics (SIM)
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-9	Accuracy/Bias	**Surrogate Compounds**	**% Recovery**	Surrogate Spikes	A
			Acenaphthene-d_{10}	20-130		
			Chrysene-d_{12}	20-130		
			Naphthalene-d_8	20-130		
			Perylene-d_{12}	20-130		
			Phenanthrene -d_{12}	20-130		
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Aroclor PCBs
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-10	Accuracy/Bias	**Surrogate Compound** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-144 43-144	Surrogate Spikes	A
		Accuracy/Bias	**Compound** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Instrument Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Pesticides
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-11	Accuracy/Bias	**Surrogate Compound** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-155 43-135	Surrogate Spikes	A
		Accuracy/Bias	**Compound** All spiking compounds	**% Recovery** 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Instrument Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Volatile Organics
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-12	Accuracy/Bias	**Surrogate Compounds** 1,2-Dichloroethane-d_4 4-Bromofluorobenzene-d_4 Toluene-d_8 Dibromofluoromethane	**% Recovery** 59-150 63-143 61-145 65-150	Surrogate Spikes	A
		Accuracy/Bias	**Compound** All spiking compounds	**% Recovery** 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Trip Blanks[d]	A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Cyanide
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-13	Accuracy/Bias	% Recovery 75-125	Laboratory Control Sample	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks	A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	TOC
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-14	Accuracy/Bias	% Recovery 50-150	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks and Instrument Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	TEPH
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-16	Accuracy/Bias	**Surrogate Compounds** Chlorobenzene Ortho-terphenyl	**% Recovery** 60-120 60-120	Surrogate Spikes	A
		Accuracy/Bias	**Compound** #2 Diesel	**% Recovery** 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Hexavalent Chromium
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-17	Accuracy/Bias	% Recovery 75-125	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks, Instrument Blanks and Calibration Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	pH
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-19	Accuracy/Bias	pH acceptable range (6.82 to 6.92) when true value is 7.0	Laboratory Control Sample	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Total Phosphorus
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-23	Accuracy/Bias	% Recovery 91-114	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Total Kjeldhal Nitrogen
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-24	Accuracy/Bias	% Recovery 90-110	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A

See the last page of Worksheet #12-1 for a description of footnotes.

QAPP Worksheet #12-1 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Field/Rinse/Equipment Blank (continued)

Matrix	Field/Rinse/ Equipment Blank
Analytical Group[a]	Ammonia
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-3	L-26	Accuracy/Bias	% Recovery 90-110	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A

Notes:

[a] If information varies within an analytical group, separate by individual analyte.
[b] Reference number from Worksheet #21.
[c] Reference number from Worksheet #23.
[d] Trip blanks will be collected, travel with environmental samples, and be submitted for analysis in association with any samples collected for volatile organics or methylmercury analysis.

DCAA = 2,4-dichlorophenylacetic acid
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/dibenzofurans
PQL = project quantitation limit
QC = quality control
RPD = relative percent difference
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
TEPH = total extractable petroleum hydrocarbons
TOC = total organic carbon
USEPA = U.S. Environmental Protection Agency
% = percent

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment

Matrix	Sediment
Analytical Group[a]	PCDDs/PCDFs
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-1	Precision - Lab	**Compound** All target analytes	**RPD** <50	Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** All analytes	**% Recovery** 60-140	Matrix Spike	A
		Accuracy/Bias	All target compound concentrations must fall within the acceptance ranges provided in Table 6 of L-1		Ongoing Precision and Recovery	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Field Duplicate	S & A
		Accuracy/Bias	Per L-1		Initial Calibration	A
		Accuracy/Bias	Per L-1		Calibration Verification	A
		Accuracy/Bias	Per L-1		Labeled Compound Spike	A
		Accuracy/Bias	Per L-1		Labeled Internal Standards	A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	PCB Congeners
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-2	Precision - Lab	**Compound** 86 target analytes	**RPD** <50	Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** 86 target analytes	**% Recovery** 60-140	Matrix Spike	A
		Accuracy/Bias	All target compound concentrations must fall within the acceptance ranges provided in Table 6 of L-2		Ongoing Precision and Recovery	A
		Accuracy/Bias Contamination	No target compounds ≥ PQL		Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	PCB Congeners
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-2	Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A
		Accuracy/Bias	Per L-2	Initial Calibration	A
		Accuracy/Bias	Per L-2	Calibration Verification	A
		Accuracy/Bias	Per L-2	Labeled Compound Spike	A
		Accuracy/Bias	Per L-2	Labeled Internal Standards	A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	TAL Metals, Titanium
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-3, L-29	Accuracy/Bias	**Compound** All target analytes	**% Recovery** 75-125	Matrix Spike	A
		Accuracy/Bias	**Compound** All target analytes	**% Recovery** 80-120	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks, Field Blanks, Instrument Blanks, and Calibration Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision - Lab	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Mercury
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-4	Accuracy/Bias	% Recovery 80-120	Matrix Spike	A
		Accuracy/Bias	% Recovery 80-120	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks, Field Blanks, and Instrument Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by less than 2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Methylmercury
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-5	Precision - Lab	RPD <35%	Matrix Spike Duplicate	A
		Accuracy/Bias	**% Recovery** 65-135	Matrix Spike	A
		Accuracy/Bias	**% Recovery** 67-133	Ongoing Precision and Recovery	A
		Accuracy/Bias	Per L-5	Initial Precision and Recovery	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks, Field Blanks, Trip Blanks[d], and Instrument Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Methylmercury
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-5	Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A
		Accuracy/Bias	Per L-5	Labeled Compound Spike	A
		Accuracy/Bias	Per L-5	Initial Calibration	A
		Accuracy/Bias	Per L-5	Calibration Verification	A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Butyltins
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-6	Precision - Lab	RPD <30%		Matrix Spike Duplicate	A
		Accuracy/Bias	**Analyte** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 10-48 30-160 30-160 30-160	Matrix Spike	A
		Accuracy/Bias	**Analyte** Tripentyltin	**% Recovery** 30-120	Surrogate Spikes	A
		Accuracy/Bias	**Analyte** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 10-48 30-160 30-160 30-160	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Chlorinated Herbicides
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-7	Precision - Lab	**Compound** All target analytes	**RPD** <35	Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** All target analytes	**% Recovery** 60-140	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compounds** DCAA	**% Recovery** 50-120	Surrogate Spikes	A
		Accuracy/Bias	**Compound** All target analytes	**% Recovery** 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Semivolatile Organics
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-8	Precision - Lab	RPD <30%		Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** All target analytes	**% Recovery** 20-150	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compounds** Phenol-d_5 2-Fluorobiphenyl 2-Fluorophenol 2,4,6-Tribromphenol Nitrobenzene-d_5 p-Terphenyl-d_{14}	**% Recovery** 38-110 39-112 33-110 34-150 28-110 51-119	Surrogate Spikes	A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Semivolatile Organics
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-8	Accuracy/Bias	**Compound** All target analytes	**% Recovery** 20-150	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Semivolatile Organics (SIM)
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-9	Precision - Lab	**Compound** All target analytes	**RPD** <40	Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** All target analytes	**% Recovery** 60-140	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compounds** Acenaphthene-d_{10} Chrysene-d_{12} Naphthalene-d_8 Perylene-d_{12} Phenanthrene -d_{12}	**% Recovery** 20-130 20-130 20-130 20-130 20-130	Surrogate Spikes	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Field Duplicate	S & A

See the last page of Worksheet #12-2 for footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Aroclor PCBs
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-10	Precision - Lab	**Compound** Aroclor-1016 Aroclor-1260	**RPD** <15 <20	Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compound** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 30-150 30-150	Surrogate Spikes	A
		Accuracy/Bias	**Compound** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks, Field Blanks, and Instrument Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Pesticides
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-11	Precision - Lab	RPD <25%		Matrix Spike Duplicate	A
		Accuracy/Bias	**Compounds** All target compounds	**% Recovery** 50-150	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compounds** Tetrachloro-m-zylene Decachlorobiphenly	**% Recovery** 43-135 43-144	Surrogate Spikes	A
		Accuracy/Bias	**Compound** All target compounds	**% Recovery** 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Volatile Organics
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-12	Precision - Lab	RPD < 25%		Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** All target compounds	**% Recovery** 70-130	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compounds** 1,2-Dichloroethane-d_4 4-Bromofluorobenzene-d_4 Toluene-d_8 Dibromofluoromethane	**% Recovery** 70-141 65-131 72-123 71-141	Surrogate Spikes	A
		Accuracy/Bias	**Compound** All target compounds	**% Recovery** 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks, Field Blanks, and Trip Blanks[d]	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Cyanide
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-13	Accuracy/Bias	% Recovery 75-125	Matrix Spike	A
		Accuracy/Bias	% Recovery 75-125	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks, Field Blanks, and Instrument Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤20% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	TOC
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-15	Accuracy/Bias	**% Recovery** 70-130	Matrix Spike	A
		Accuracy/Bias	**% Recovery** 50-150	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks, Field Blanks, and Instrument Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤35%	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	TEPH
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-16	Precision - Lab	RPD <50%		Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** #2 Diesel	**% Recovery** 70-130	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compounds** Chlorobenzene Ortho-terphenyl	**% Recovery** 60-120 60-120	Surrogate Spikes	A
		Accuracy/Bias	**Compound** #2 Diesel	**% Recovery** 70-120	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by < 2X the PQL when detects are <5X PQL for both field duplicate samples		Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Hexavalent Chromium
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-17	Accuracy/Bias	% Recovery 75-125	Matrix Spike	A
		Accuracy/Bias	% Recovery 75-125	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks, Field Blanks, and Instrument Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤20% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	pH
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-18	Accuracy/Bias	pH acceptable range (6.82 to 6.92) when true value is 7.0	Laboratory Control Sample	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤25%	Laboratory Duplicate	A
		Precision - Overall	RPD ≤25%	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	% Moisture
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-20	Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤20%	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Total Sulfide
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-22	Precision - Lab	RPD <20%	Matrix Spike Duplicate	A
		Accuracy/Bias	**% Recovery** 75-125	Matrix Spike	A
		Accuracy/Bias	**% Recovery** 75-125	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤20% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Total Phosphorus
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-23	Precision - Lab	RPD <12%	Matrix Spike Duplicate	A
		Accuracy/Bias	**% Recovery** 75-125	Matrix Spike	A
		Accuracy/Bias	**% Recovery** 91-114	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤20% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Total Kjeldhal Nitrogen
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-24	Precision - Lab	RPD <20%	Matrix Spike Duplicate	A
		Accuracy/Bias	**% Recovery** 85-115	Matrix Spike	A
		Accuracy/Bias	**% Recovery** 90-110	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤20% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	AVS/SEM
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-25	Precision - Lab	RPD <20%	Matrix Spike Duplicate	A
		Accuracy/Bias	% Recovery 60-130	Matrix Spike	A
		Accuracy/Bias	% Recovery 60-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤20% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Ammonia
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-26	Precision - Lab	RPD <20%	Matrix Spike Duplicate	A
		Accuracy/Bias	**% Recovery** 75-125	Matrix Spike	A
		Accuracy/Bias	**% Recovery** 90-110	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks and Field Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤20% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Grain Size
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-27	Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤20% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A
		Precision - Overall	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Field Duplicate	S & A

See the last page of Worksheet #12-2 for a description of footnotes.

QAPP Worksheet #12-2 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Sediment (continued)

Matrix	Sediment
Analytical Group[a]	Oxidation Reduction Potential
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-9	L-28	Accuracy/Bias	Acceptable range is the certified value ± 30mV	Laboratory Control Sample	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤25%	Laboratory Duplicate	A
		Precision - Overall	RPD ≤25%	Field Duplicate	S & A

Notes:

[a] If information varies within an analytical group, separate by individual analyte.
[b] Reference number from Worksheet #21.
[c] Reference number from Worksheet #23.
[d] Trip blanks will be collected, travel with environmental samples, and be submitted for analysis in association with any samples collected for volatile organics or methylmercury analysis.

AVS/SEM = acid volatile sulfide/simultaneously extracted metals
DCAA = 2,4-dichlorophenylacetic acid
mV = millivolts
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/dibenzofurans
PQL = project quantitation limit
QC = quality control
RPD = relative percent difference
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
TEPH = total extractable petroleum hydrocarbons
TOC = total organic carbon
USEPA = U.S. Environmental Protection Agency
% = percent

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue

Matrix	Tissue
Analytical Group[a]	PCDDs/PCDFs
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-1	Precision - Lab	**Compound** All target analytes	**RPD** <50	Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** All analytes	**% Recovery** 60-140	Matrix Spike	A
		Accuracy/Bias	All target compound concentrations must fall within the acceptance ranges provided in Table 6 of L-1		Ongoing Precision and Recovery	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks, Field Blanks, and Rinsate Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Accuracy/Bias	Per L-1		Initial Calibration	A
		Accuracy/Bias	Per L-1		Calibration Verification	A
		Accuracy/Bias	Per L-1		Labeled Compound Spike	A
		Accuracy/Bias	Per L-1		Labeled Internal Standards	A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	PCB Congeners
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-2	Precision - Lab	**Compound** 86 target analytes	**RPD** <50	Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** 86 target analytes	**% Recovery** 60-140	Matrix Spike	A
		Accuracy/Bias	All target compound concentrations must fall within the acceptance ranges provided in Table 6 of L-2		Ongoing Precision and Recovery	A
		Accuracy/Bias Contamination	No target compounds ≥ PQL		Method Blanks, Field Blanks, and Rinsate Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	PCB Congeners
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-2	Accuracy/Bias	Per L-2	Initial Calibration	A
		Accuracy/Bias	Per L-2	Calibration Verification	A
		Accuracy/Bias	Per L-2	Labeled Compound Spike	A
		Accuracy/Bias	Per L-2	Labeled Internal Standards	A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	TAL Metals, Titanium
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-3, L-29	Accuracy/Bias	**Compound** All target analytes	**% Recovery** 75-125	Matrix Spike	A
		Accuracy/Bias	**Compound** All target analytes	**% Recovery** 80-120	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks, Field Blanks, Rinsate Blanks, Instrument Blanks, and Calibration Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A
		Precision-Lab	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples		Laboratory Duplicate	A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	Mercury
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-4	Accuracy/Bias	**% Recovery** 80-120	Matrix Spike	A
		Accuracy/Bias	**% Recovery** 80-120	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks, Field Blanks, Rinsate Blanks, and Instrument Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	Methylmercury
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-5	Precision - Lab	RPD ≤35%	Matrix Spike Duplicate	A
		Accuracy/Bias	**% Recovery** 65-135	Matrix Spike	A
		Accuracy/Bias	**% Recovery** 67-133	Ongoing Precision and Recovery	A
		Accuracy/Bias	Per L-5	Initial Precision and Recovery	A
		Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks, Field Blanks, Rinsate Blanks, Trip Blanks[d] and Instrument Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Accuracy/Bias	Per L-5	Initial Calibration	A
		Accuracy/Bias	Per L-5	Calibration Verification	A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	Butyltins
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-6	Precision - Lab	RPD <30%		Matrix Spike Duplicate	A
		Accuracy/Bias	**Analyte** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 10-48 30-160 30-160 30-160	Matrix Spike	A
		Accuracy/Bias	**Analyte** Tripentyltin	**% Recovery** 30-120	Surrogate Spikes	A
		Accuracy/Bias	**Analyte** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 10-48 30-160 30-160 30-160	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks, Field Blanks, and Rinsate Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	Semivolatile Organics
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-8	Precision - Lab	RPD <30%		Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** All target compounds	**% Recovery** 20-150	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compounds** Phenol-d_5 2-Fluorobiphenyl 2-Fluorophenol 2,4,6-Tribromphenol Nitrobenzene-d_5 p-Terphenyl-d_{14}	**% Recovery** 38-110 39-112 33-110 34-150 28-110 51-119	Surrogate Spikes	A
		Accuracy/Bias	**Compound** All target compounds	**% Recovery** 20-150	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks, Field Blanks, and Rinsate Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	Semivolatile Organics (SIM)
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-9	Precision - Lab	**Compound** All target analytes	**RPD** <40	Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** All target analytes	**% Recovery** 60-140	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compounds** Acenaphthene-d_{10} Chrysene-d_{12} Naphthalene-d_8 Perylene-d_{12} Phenanthrene -d_{12}	**% Recovery** 20-130 20-130 20-130 20-130 20-130	Surrogate Spikes	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks, Field Blanks, and Rinsate Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	Aroclor PCBs
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-10	Precision - Lab	**Compound** Aroclor-1016 Aroclor-1260	**RPD** <15 <20	Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compound** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 30-150 30-150	Surrogate Spikes	A
		Accuracy/Bias	**Compound** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks, Field Blanks, and Instrument Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	Pesticides
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria		QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-11	Precision - Lab	RPD < 25%		Matrix Spike Duplicate	A
		Accuracy/Bias	**Compound** All spiking compounds	**% Recovery** 50-150	Matrix Spike	A
		Accuracy/Bias	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-135 43-144	Surrogate Spikes	A
		Accuracy/Bias	**Compound** All spiking compounds	**% Recovery** 70-130	Laboratory Control Sample	A
		Accuracy/Bias Contamination	No target compounds ≥PQL		Method Blanks, Field Blanks, and Rinsate Blanks	S & A
		Completeness	>90% sample collection, >90% laboratory analysis		Data Completeness Checks	S & A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	% Lipids
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-1	Accuracy/Bias Contamination	No target compounds ≥PQL	Method Blanks and Rinsate Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	Laboratory Duplicate	A

See the last page of Worksheet #12-3 for a description of footnotes.

QAPP Worksheet #12-3 (UFP-QAPP Manual Section 2.6): Measurement Performance Criteria – Tissue (continued)

Matrix	Tissue
Analytical Group[a]	% Moisture
Concentration Level	Low

Sampling Procedure[b]	Analytical Method/SOP[c]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
SOP-6, SOP-7, SOP-8	L-21	Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Checks	S & A
		Precision - Lab	RPD ≤20%	Laboratory Duplicate	A

Notes:

[a] If information varies within an analytical group, separate by individual analyte.
[b] Reference number from Worksheet #21.
[c] Reference number from Worksheet #23.
[d] Trip blanks will be collected, travel with environmental samples, and be submitted for analysis in association with any samples collected for volatile organics or methylmercury analysis.

PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/dibenzofurans
PQL = project quantitation limit
QC = quality control
RPD = relative percent difference
SIM = selective ion monitoring
TAL = Target Analyte List
SOP = Standard Operating Procedure
USEPA = U.S. Environmental Protection Agency
% = percent

QAPP Worksheet #13 (UFP-QAPP Manual Section 2.7): Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s)	Data Use	Limitations on Data Use
NBSA Sampling Performed by Others				
Tissue and Sediment Data	Contaminant Assessment and Reduction Program, available online at (http://www.carpweb.org/main.html)	Fish and blue crab tissue data and sediment data collected by NJDEP and NYSDEC between 2000 and 2004.	Tissue chemistry data were used to select the species appropriate for tissue-residue analysis.	Only two stations in Newark Bay; data are not recent (i.e., approximately 10 years old).
Tissue Data	NJDEP 2004 Routine Monitoring Program for Toxics in Fish: Year 2 – Estuarine and Marine Waters. NJDEP Crab Monitoring Program available online at (http://www.state.nj.us/dep/dsr/2004data.htm is the correct link).	NJDEP Crab Monitoring Program collected crab tissue.	All blue crab composite samples were analyzed for both muscle meat and hepatopancreas tissues. Each tissue (i.e., muscle, hepatopancreas) were analyzed for dioxins/furans, PCBs (congener and co-planar), organo-chlorinated pesticides, mercury, and lipids.	Only three stations in Newark Bay; data are not recent (i.e., approximately 10 years old).
Tissue and Sediment Data	Final Report. Sediment Quality of the NY/NJ Harbor System: a 5-year revisit (1993/4 – 1998) An Investigation under the Regional Environmental Monitoring and Assessment Program (REMAP) (USEPA 2003)	USEPA REMAP data includes benthic invertebrate numbers and species	Provides information on dominant clam/bivalve species in the NBSA. Sediment chemistry also collected.	Data are not recent (over 10 years old); benthic community assemblages and sediment chemistry may have changed since these data were collected.
Work Performed by Tierra on NBSA				
Sediment Data	Phase I and II Sediment Investigations	Tierra collected over 100 sediment cores at various intervals	Surficial sediment data (0 to 0.5 foot) screened to determine what chemicals to analyze for in surface sediment.	None.

QAPP Worksheet #14 (UFP-QAPP Manual Section 2.8.1): Summary of Project Tasks

Sampling Tasks:

Samples will be collected by locating the Intertidal Areas and associated shallow Subtidal Flats for sampling, through maps and GPS coordinates. Upon reaching each Intertidal Area for sampling, locational information will be recorded on the field datasheet; a GPS will be used to record the location; general observations about the Intertidal Area will be documented, including presence/absence of vegetation, general substrate appearance, and texture; tidal stage; and time on station will be recorded. At least three baited crab traps will be deployed so that they are completely submerged underwater, equally spaced around the Intertidal Area in the associated shallow Subtidal Flats. The time of release and GPS coordinates will be recorded.

The target length, species, and number of bivalve clams per composite sample will be identified following experimental sampling in the intertidal areas during the Reconnaissance Survey. While data indicate dominance of the bivalve community by *Mya arenaria*, additional bivalve species, such as *Macoma balthica*, *Mulinia lateralis,* and/or *Mercenaria mercenaria*, may also be prevalent. Each species varies in size; therefore, targeted species and sizes will be determined after the Reconnaissance Survey. It is assumed that each composite sample will include at least 20 clams of at least 1.5 inches (per NJDEP fishing regulations). The intertidal area will be explored for the presence of clam holes. A clam rake or shovel will be used to locate buried clams. Holes will be dug until the required number of clams is collected. Clams will be identified, measured, and weighed. Once the minimum required weight (232 g) of a single species is met, the clams will be placed into appropriate-labeled sampling containers and stored on wet ice for shipment to the analytical laboratory. Clams not meeting the required length (1.5 inches) will be left at the site.

Following clam collection, a grab sample will be collected from the top 6 inches of sediment next to the first location of the retained clam. This sediment will be used for, but not be limited to, analyses of VOC, AVS/SEM, sulfide, and cyanide. Sediment will be placed in the appropriate sample containers with no headspace, preservative (as required), and sealed per the SOP. Additional sediment will be scooped from the sediment dug out of each clam hole, homogenized, placed in the appropriate-labeled sample containers, and stored on wet ice for shipment to the analytical laboratory for the remaining chemical analyses. The specific methodology and sampling equipment used will be determined after the Reconnaissance Survey.

Crab traps will be checked at least every 12 hours. Hard-shelled blue crabs meeting the required size limit (4.5 inches, per NJDEP fishing regulations) will be inspected for gross anomalies. Whole, intact crabs retained for analysis will be measured, weighed, documented, placed into the appropriate-labeled sampling container, and stored on wet ice for shipment to the analytical laboratory. It is expected that at least 25 crabs will be required per sample to obtain enough tissue mass for analysis.

For a detailed description of tissue and sediment collection, refer to SOP Nos. 6, 7, and 9.

Analysis Tasks:

Following the tissue and sediment collection, samples will be shipped to the analytical laboratory for homogenization and analysis. The homogenate from the tissue samples will be shipped to subcontracted laboratories either on wet or dry ice. Tissue and sediment samples will be submitted for laboratory analysis of the chemicals listed in Worksheet No. 15.

Quality Control Tasks:

All field notes and forms completed during the field sampling task will be checked daily by the Field Supervisor. The Field Supervisor will also communicate daily with the Task QA/QC Manager to confirm objectives are being met.
As part of the QC process to assess the accuracy of species identification, specimens of each captured species will be collected and independently verified by a biologist who is not associated with the field task. Lengths and weights will be compiled in a table and reviewed as a QC step. Any lengths and weights that appear to be anomalous will be verified by a second team member by re-measuring. Sample identifications will be similarly verified.

QAPP Worksheet #14 (UFP-QAPP Manual Section 2.8.1): Summary of Project Tasks (continued)

Electronic sampling equipment (e.g., scale, GPS units) will be calibrated, maintained, tested, and inspected according to manufacturers' specifications, as necessary, to confirm they are functioning properly (refer to Worksheet No. 22).

Laboratory procedures will be carried out per the SOPs and analytical methodologies provided in the appendices of this document. In addition, field and laboratory quality control samples will be processed as indicated in Worksheets #28-1 and 28-2. Project quality objectives and measurement performance criteria will be monitored throughout project implementation and at the conclusion of field and analytical activities. Data produced will undergo data validation steps specified in Worksheets #34, 35 and 36. These data evaluation steps will be performed by the Quality Assurance Coordinator, an organization independent from those generating the data.

Secondary Data:

Other tissue and chemistry data that are summarized in Worksheet Nos. 10 and 13 will also be reviewed and potentially used to accomplish project objectives.

Data Management Tasks:

The data management task will include keeping accurate records of field activities and observations so that project team members using the data will have accurate and appropriate documentation. Field data will be stored in its native format and in the project sampling database. GPS data will also be downloaded and stored electronically in a project file. Laboratory analytical data will be loaded into the project sampling database, verified against the laboratory reports, merged with corresponding field data, and updated based on validation. Subsequently, the spatial data will be mapped for the data report.

Documentation and Records:

It is important that field activities be documented in an organized, chronologically accurate manner. All field activities will be recorded in a field notebook maintained by the Field Supervisor. The field notebook is intended to provide sufficient data and observations to enable participants to reconstruct events that occurred during the sampling period. Procedures for documentation are presented in SOP No. 10. All relevant forms and records are presented on Worksheet No. 29. In general, the following information must be recorded:

- ☐ The identities and affiliation of the personnel conducting field activities.
- ☐ Model numbers and serial numbers of instruments and/or equipment being used, will, to the extent available, be recorded in the field notebook.
- ☐ A description of the type of field work being conducted and the equipment used.
- ☐ The date and time the field activities were initiated and completed, with specific temporal information for each task (e.g., record the time activities commenced at each individual location, if applicable).
- ☐ The site where the field activities were conducted and also any locations within that site where work was performed (e.g., specific sampling sites, coordinates, depths).
- ☐ The general methodology used to conduct the activities.
- ☐ Communications with Project Managers and personnel regarding field activities.
- ☐ Field collected data (e.g., GPS measurements, catch totals).
- ☐ Daily health and safety briefings.
- ☐ Deviations from this Crab/Clam Sampling QAPP, SOP, or project HASP, reason for change, and any corrective actions taken. Corrective actions will be electronically documented on the Protocol Modification Form (Appendix A).
- ☐ Photos will be taken to document gross external abnormalities on captured crabs and clams. Photos will be taken of all species collected. When photos associated with sampling locations, field activities, or samples are taken, they will be documented in the field notebook, including the date, time, photographer, and brief description.

QAPP Worksheet #14 (UFP-QAPP Manual Section 2.8.1): Summary of Project Tasks (continued)

The Location Data Form and Specimen Data Form (Appendix A) will also be filled out electronically by field personnel to document sampling location information and gross external observations of collected organisms. A daily tally of all species that are caught will also be recorded in the Specimen Tally Form and Non-Target Species Tally Form (Appendix A). All tissue and sediment samples that will be analyzed will be recorded electronically in the Composite Sample Form (Appendix A) by field laboratory personnel or the project chemist.

A record of all personnel briefed on the HASP will be maintained by the Field Supervisor, Site Safety and Health Officer, or designee. The record will be archived at Tierra's office upon completion of the sampling efforts.

Laboratories will provide complete hard copy analytical data packages, as outlined in Worksheets #29-2 and #29-3. Specifications for electronic laboratory data deliverables are located in Worksheet #29-4.

Assessment/Audit Tasks:

Assessment/audit tasks will be conducted, as summarized below:

1. Laboratory Technical Systems Audit (one audit of each laboratory prior to use or again after period of inactivity associated with Tierra Solutions, Inc. exceeding 2 years).
2. Field Readiness Review. Refer to Worksheets #31 and 32.
3. Laboratory Readiness Review. Refer to Worksheets #31 and 32.
4. Data Validation.
5. Data Usability Assessment Report.

Data Review Tasks:

All field records will be reviewed by the Field Supervisor for completeness and accuracy and verified by the Task QA/QC Manager or a designee.

Contract laboratories will verify that all data are complete for samples received. All data package deliverable requirements will be met. Data will be validated by Environmental Data Services, Ltd. per the requirements specified in Worksheets #34, 35 and 36. A Data Validation Report will be produced for each sample delivery group and each analytical group.

Validated data and all related field logs/notes/records will be reviewed to assess total measurement error and determine overall usability of the data for project purposes. Data limitations will be determined and data will be compared to project quality objectives and required action limits. Corrective action will be initiated, as necessary. Final data will be placed in the database, with necessary qualifiers.

As part of data report preparation, chemical data will be reviewed to determine if differences related to species and/or location are evident. In addition, the data report will also undergo a senior and peer review process before the final draft is submitted to USEPA (see Worksheet Nos. 34 through 37 for relevant procedures).

A tissue chemistry data report will be prepared once all of the tissue chemistry results (including all Phase III data) have been validated. This data report will be provided to USEPA after 90 days of receipt of validated data and will include validation results.

A sediment chemistry data report will be prepared once all of the sediment chemistry results (including all Phase III data) have been validated. This data report will be provided to USEPA after 90 days of receipt of validated data and will include validation results.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water

Matrix: Field/Rinse/Equipment Blank
Analytical Group: PCDDs/PCDFs
Concentration Level: Low
Analytical Method/SOP Reference: L-1

Analyte	CAS Number	Study Action Level pg/L	Project Quantitation Limit pg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/L	Method QLs pg/L	MDLs pg/L	QLs pg/L
2,3,7,8-TCDD	1746-01-6	---	5.00	Not provided in method	10	0.474	5.00
1,2,3,7,8-PeCDD	40321-76-4	---	25.0	Not provided in method	50	1.69	25.0
1,2,3,6,7,8-HxCDD	57653-85-7	---	25.0	Not provided in method	50	2.75	25.0
1,2,3,4,7,8-HxCDD	39227-28-6	---	25.0	Not provided in method	50	2.19	25.0
1,2,3,7,8,9-HxCDD	19408-74-3	---	25.0	Not provided in method	50	1.39	25.0
1,2,3,4,6,7,8-HpCDD	35822-46-9	---	25.0	Not provided in method	50	3.06	25.0
OCDD	3268-87-9	---	50.0	Not provided in method	100	10.6	50.0
2,3,7,8-TCDF	51207-31-9	---	5.00	Not provided in method	10	0.651	5.00
1,2,3,7,8-PeCDF	57117-41-6	---	25.0	Not provided in method	50	1.92	25.0
2,3,4,7,8-PeCDF	57117-31-4	---	25.0	Not provided in method	50	2.40	25.0
1,2,3,6,7,8-HxCDF	57117-44-9	---	25.0	Not provided in method	50	2.94	25.0
1,2,3,7,8,9-HxCDF	72918-21-9	---	25.0	Not provided in method	50	1.22	25.0
1,2,3,4,7,8-HxCDF	70648-26-9	---	25.0	Not provided in method	50	1.79	25.0
2,3,4,6,7,8-HxCDF	60851-34-5	---	25.0	Not provided in method	50	0.934	25.0
1,2,3,4,6,7,8-HpCDF	67562-39-4	---	25.0	Not provided in method	50	1.39	25.0
1,2,3,4,7,8,9-HpCDF	55673-89-7	---	25.0	Not provided in method	50	2.73	25.0
OCDF	39001-02-0	---	50.0	Not provided in method	100	3.12	50.0

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/L	Project Quantitation Limit pg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/L	Method QLs pg/L	MDLs pg/L	QLs pg/L
PCB1	2051-60-7	---	5.0	82	200	3.72	5.0
PCB2	2051-61-8	---	5.0	4	10	4.05	5.0
PCB3	2051-62-9	---	5.0	88	200	3.69	5.0
PCB4/10	13029-08-8/33146-45-1	---	20	Not provided in method	Not provided in method	8.78	20
PCB6	25569-80-6	---	10.0	13	50	3.10	10.0
PCB5/8	16605-91-7/34883-43-7	---	20.0	Not provided in method	Not provided in method	11.0	20.0
PCB7/9	33284-50-3/34883-39-1	---	20.0	Not provided in method	Not provided in method	8.20	20.0
PCB11	2050-67-1	---	10.0	105	200	6.50	10.0
PCB12/13	2974-92-7/2974-90-5	---	20.0	28	100	12.1	20.0
PCB14	34883-41-5	---	20.0	31	100	5.17	20.0
PCB15	2050-68-2	---	10.0	183	500	5.71	10.0
PCB16/32	38444-78-9/38444-77-8	---	10.0	Not provided in method	Not provided in method	8.14	10.0
PCB17	37680-66-3	---	5.0	86	200	5.75	5.0
PCB18	37680-65-2	---	5.0	175	500	5.23	5.0
PCB19	38444-73-4	---	5.0	42	100	4.10	5.0
PCB20/21/33	38444-84-7/ 55702-46-0/38444-86-9	---	15.0	Not provided in method	Not provided in method	7.89	15.0
PCB22	38444-85-8	---	15.0	90	200	3.34	15.0
PCB23	55720-44-0	---	5.0	50	200	5.01	5.0
PCB24/27	55702-45-9/38444-76-7	---	10.0	Not provided in method	Not provided in method	9.54	10.0
PCB25	55712-37-3	---	5.0	55	200	3.89	5.0
PCB26	38444-81-4	---	5.0	83	200	3.05	5.0
PCB28	7012-37-5	---	5.0	192	500	2.59	5.0
PCB29	15862-07-4	---	5.0	83	200	4.11	5.0

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/L	Project Quantitation Limit pg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/L	Method QLs pg/L	MDLs pg/L	QLs pg/L
PCB30	35693-92-6	---	5.0	175	500	3.54	5.0
PCB31	16606-02-3	---	5.0	152	500	4.01	5.0
PCB34	37680-68-5	---	5.0	74	200	5.77	5.0
PCB35	37680-69-6	---	5.0	77	200	2.65	5.0
PCB36	38444-87-0	---	5.0	79	200	4.00	5.0
PCB37	38444-90-5	---	5.0	132	500	3.06	5.0
PCB38	53555-66-1	---	5.0	83	200	2.46	5.0
PCB39	38444-88-1	---	5.0	85	200	3.04	5.0
PCB40	38444-93-8	---	5.0	119	500	6.39	5.0
PCB41/64/71/ 72	52663-59-9/52663-58-8/ 41464-46-4/41464-42-0	---	20.0	Not provided in method	Not provided in method	21.0	20.0
PCB42/59	36559-22-5/74472-33-6	---	10.0	Not provided in method	Not provided in method	9.05	10.0
PCB43/49	70362-46-8/41464-40-8	---	10.0	Not provided in method	Not provided in method	8.31	10.0
PCB44	41464-39-5	---	5.0	195	500	5.21	5.0
PCB45	70362-45-7	---	5.0	51	200	5.07	5.0
PCB46	41464-47-5	---	5.0	101	200	5.26	5.0
PCB47	2437-79-8	---	5.0	195	500	6.18	5.0
PCB48/75	70362-47-9/32598-12-2	---	10.0	Not provided in method	Not provided in method	10.4	10.0
PCB50	62796-65-0	---	5.0	58	200	3.60	5.0
PCB51	68194-04-7	---	5.0	51	200	6.20	5.0
PCB52/69	35693-99-3/60233-24-1	---	10.0	Not provided in method	Not provided in method	8.05	10.0
PCB53	41464-41-9	---	5.0	58	200	4.89	5.0
PCB54	15968-05-5	---	5.0	118	500	3.54	5.0
PCB55	74338-24-2	---	5.0	120	500	3.24	5.0

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/L	Project Quantitation Limit pg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/L	Method QLs pg/L	MDLs pg/L	QLs pg/L
PCB56/60	41464-43-1/33025-41-1	---	10.0	Not provided in method	Not provided in method	7.59	10.0
PCB57	70424-67-8	---	5.0	125	500	5.38	5.0
PCB58	41464-49-7	---	5.0	127	500	3.90	5.0
PCB61/70	33284-53-6/32598-11-1	---	10.0	171	500	7.38	10.0
PCB62	54230-22-7	---	5.0	57	200	7.12	5.0
PCB63	74472-34-7	---	5.0	138	500	4.96	5.0
PCB65	33284-54-7	---	5.0	195	500	3.83	5.0
PCB67	73575-53-8	---	5.0	147	500	5.38	5.0
PCB68	73575-52-7	---	5.0	149	500	5.92	5.0
PCB73	74338-23-1	---	5.0	160	500	4.53	5.0
PCB74	32690-93-0	---	5.0	171	500	4.70	5.0
PCB76/66	70362-48-0/32598-10-0	---	10.0	Not provided in method	Not provided in method	9.14	10.0
PCB77	32598-13-3	---	5.0	169	500	3.76	5.0
PCB78	70362-49-1	---	5.0	171	500	6.73	5.0
PCB79	41464-48-6	---	5.0	173	500	5.24	5.0
PCB80	33284-52-5	---	5.0	175	500	3.49	5.0
PCB81	70362-50-4	---	5.0	177	500	5.49	5.0
PCB82	52663-62-4	---	5.0	133	500	2.75	5.0
PCB83	60145-20-2	---	5.0	217	500	5.29	5.0
PCB84/92	52663-60-2/52663-61-3	---	10.0	Not provided in method	Not provided in method	9.17	10.0
PCB85/116	65510-45-4/18259-05-7	---	10.0	104	200	10.4	10.0
PCB86	55312-69-1	---	5.0	149	500	5.08	5.0
PCB87/117/125	38380-02-8/ 68194-11-6/74472-39-2	---	15.0	Not provided in method	Not provided in method	13.7	15.0

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/L	Project Quantitation Limit pg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/L	Method QLs pg/L	MDLs pg/L	QLs pg/L
PCB88/91	55215-17-3/68194-05-8	---	10.0	118	500	12.9	10.0
PCB89	73575-57-2	---	5.0	195	500	6.32	5.0
PCB90/101	68194-07-0/37680-73-2	---	10.0	241	1000	11.7	10.0
PCB93	73575-56-1	---	5.0	221	500	9.86	5.0
PCB94	73575-55-0	---	5.0	121	500	3.81	5.0
PCB95/98/102	38379-99-6/ 60233-25-2/68194-06-9	---	15.0	221	500	14.7	15.0
PCB96	73575-54-9	---	5.0	210	500	6.69	5.0
PCB97	41464-51-1	---	5.0	149	500	4.48	5.0
PCB99	38380-01-7	---	5.0	217	500	6.25	5.0
PCB100	39485-83-1	---	5.0	221	500	5.72	5.0
PCB103	60145-21-3	---	5.0	225	500	5.66	5.0
PCB104	56558-16-8	---	5.0	228	500	4.08	5.0
PCB105	32598-14-4	---	5.0	109	200	3.57	5.0
PCB106/118	70424-69-0/31508-00-6	---	10.0	Not provided in method	Not provided in method	11.9	10.0
PCB107/109	70424-68-9/74472-35-8	---	10.0	Not provided in method	Not provided in method	9.64	10.0
PCB108/112	70362-41-3/74472-36-9	---	10.0	Not provided in method	Not provided in method	9.47	10.0
PCB110	38380-03-9	---	5.0	243	1000	8.81	5.0
PCB111/115	39635-32-0/74472-38-1	---	10.0	243	1000	7.05	10.0
PCB113	68194-10-5	---	5.0	241	1000	8.59	5.0
PCB114	74472-37-0	---	5.0	120	500	1.45	5.0
PCB119	56558-17-9	---	5.0	149	500	4.72	5.0
PCB120	68194-12-7	---	5.0	147	500	4.86	5.0
PCB121	56558-18-0	---	5.0	209	500	8.49	5.0

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/L	Project Quantitation Limit pg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/L	Method QLs pg/L	MDLs pg/L	QLs pg/L
PCB122	76842-07-4	---	5.0	117	500	3.23	5.0
PCB123	65510-44-3	---	5.0	150	500	3.88	5.0
PCB124	70424-70-3	---	5.0	200	1000	5.65	5.0
PCB126	57465-28-8	---	5.0	136	500	2.84	5.0
PCB127	39635-33-1	---	5.0	278	1000	2.51	5.0
PCB128/162	38380-07-3/39635-34-2	---	10.0	Not provided in method	Not provided in method	8.84	10.0
PCB129	55215-18-4	---	5.0	211	500	4.00	5.0
PCB130	52663-66-8	---	5.0	136	500	4.21	5.0
PCB131	61798-70-7	---	5.0	121	500	3.52	5.0
PCB132/161	38380-05-1/74472-43-8	---	10.0	Not provided in method	Not provided in method	8.00	10.0
PCB133/142	35694-04-3/41411-61-4	---	10.0	Not provided in method	Not provided in method	8.19	10.0
PCB134/143	52704-70-8/68194-15-0	---	10.0	134	500	9.00	10.0
PCB135	52744-13-5	---	5.0	112	500	6.51	5.0
PCB136	38411-22-2	---	5.0	91	200	5.37	5.0
PCB137	35694-06-5	---	5.0	300	1000	6.62	5.0
PCB138/163/164	35065-28-2/ 74472-44-9/74472-45-0	---	15.0	Not provided in method	Not provided in method	14.5	15.0
PCB139/149	56030-56-9/38380-04-0	---	10.0	Not provided in method	Not provided in method	13.7	10.0
PCB140	59291-64-4	---	5.0	196	500	5.29	5.0
PCB141	52712-04-6	---	5.0	93	200	3.72	5.0
PCB144	68194-14-9	---	5.0	167	500	7.35	5.0
PCB145	74472-40-5	---	5.0	317	1000	6.11	5.0
PCB146/165	51908-16-8/74472-46-1	---	10.0	Not provided in method	Not provided in method	7.47	10.0
PCB147	68194-13-8	---	5.0	179	500	9.07	5.0

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/L	Project Quantitation Limit pg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/L	Method QLs pg/L	MDLs pg/L	QLs pg/L
PCB148	74472-41-6	---	5.0	324	1000	7.02	5.0
PCB150	68194-08-1	---	5.0	328	1000	6.28	5.0
PCB151	52663-63-5	---	5.0	112	500	6.66	5.0
PCB152	68194-09-2	---	5.0	238	1000	6.92	5.0
PCB153	35065-27-1	---	5.0	130	500	5.59	5.0
PCB154	60145-22-4	---	5.0	112	500	6.85	5.0
PCB155	33979-03-2	---	5.0	339	1000	4.72	5.0
PCB156	38380-08-4	---	5.0	132	500	4.77	5.0
PCB157	69782-90-7	---	5.0	132	500	4.29	5.0
PCB158/160	74472-42-7/41411-62-5	---	10.0	Not provided in method	Not provided in method	9.25	10.0
PCB159	39635-35-3	---	5.0	348	1000	3.70	5.0
PCB166	41411-63-6	---	5.0	124	500	4.29	5.0
PCB167	52663-72-6	---	5.0	115	500	5.39	5.0
PCB168	59291-65-5	---	5.0	130	500	4.80	5.0
PCB169	32774-16-6	---	5.0	161	500	4.06	5.0
PCB170	35065-30-6	---	5.0	162	500	4.14	5.0
PCB171	52663-71-5	---	5.0	374	1000	2.56	5.0
PCB172	52663-74-8	---	5.0	377	1000	3.60	5.0
PCB173	68194-16-1	---	5.0	374	1000	4.64	5.0
PCB174	38411-25-5	---	5.0	186	500	4.74	5.0
PCB175	40186-70-7	---	5.0	383	1000	7.47	5.0
PCB176	52663-65-7	---	5.0	385	1000	5.20	5.0
PCB177	52663-70-4	---	5.0	141	500	3.25	5.0
PCB178	52663-67-9	---	5.0	221	500	4.14	5.0

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/L	Project Quantitation Limit pg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/L	Method QLs pg/L	MDLs pg/L	QLs pg/L
PCB179	52663-64-6	---	5.0	229	500	5.01	5.0
PCB180	35065-29-3	---	5.0	136	500	5.96	5.0
PCB181	74472-47-2	---	5.0	396	1000	3.36	5.0
PCB182/187	60145-23-5/52663-68-0	---	10.0	Not provided in method	Not provided in method	12.8	10.0
PCB183	52663-69-1	---	5.0	401	1000	6.46	5.0
PCB184	74472-48-3	---	5.0	403	1000	4.34	5.0
PCB185	52712-05-7	---	5.0	401	1000	3.20	5.0
PCB186	74472-49-4	---	5.0	407	1000	5.11	5.0
PCB188	74487-85-7	---	5.0	235	500	2.85	5.0
PCB189	39635-31-9	---	5.0	177	500	2.93	5.0
PCB190	41411-64-7	---	5.0	234	500	5.02	5.0
PCB191	74472-50-7	---	5.0	418	1000	5.01	5.0
PCB192	*74472-51-8*	---	5.0	420	1000	5.55	5.0
PCB193	69782-91-8	---	5.0	136	500	5.44	5.0
PCB194	35694-08-7	---	5.0	170	500	2.15	5.0
PCB195	52663-78-2	---	5.0	427	1000	4.41	5.0
PCB196/203	42740-50-1/52663-76-0	---	10.0	Not provided in method	Not provided in method	10.7	10.0
PCB197	33091-17-7	---	5.0	245	1000	6.88	5.0
PCB198	68194-17-2	---	5.0	203	500	6.36	5.0
PCB199	52663-75-9	---	5.0	203	500	6.52	5.0
PCB200	52663-73-7	---	5.0	245	1000	4.57	5.0
PCB201	40186-71-8	---	5.0	440	1000	4.25	5.0
PCB202	2136-99-4	---	5.0	442	1000	3.36	5.0
PCB204	74472-52-9	---	5.0	447	1000	5.41	5.0

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/L	Project Quantitation Limit pg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/L	Method QLs pg/L	MDLs pg/L	QLs pg/L
PCB205	74472-53-0	---	5.0	449	1000	3.66	5.0
PCB206	40186-72-9	---	5.0	451	1000	3.25	5.0
PCB207	52663-79-3	---	5.0	453	1000	3.35	5.0
PCB208	52663-77-1	---	5.0	455	1000	3.02	5.0
PCB209	2051-24-3	---	5.0	153	500	2.81	5.0

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: TAL Metals, Mercury, Methylmercury, Titanium
Concentration Level: Low
Analytical Method/SOP Reference: L-3, L-4, L-5, and L-29

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
Aluminum	7429-90-5	---	80	Not provided in method	Not provided in method	6	80
Antimony	7440-36-0	---	20	Not provided in method	Not provided in method	0.073	20
Arsenic	7440-38-2	---	2	Not provided in method	Not provided in method	0.092	2
Barium	7440-39-3	---	100	Not provided in method	Not provided in method	0.42	100
Beryllium	7440-41-7	---	2	Not provided in method	Not provided in method	0.092	2
Cadmium	7440-43-9	---	2	Not provided in method	Not provided in method	0.046	2
Calcium	7440-70-2	---	1000	Not provided in method	Not provided in method	18	1000
Chromium	7440-47-3	---	4	Not provided in method	Not provided in method	0.11	4
Cobalt	7440-48-4	---	10	Not provided in method	Not provided in method	0.049	10
Copper	7440-50-8	---	20	Not provided in method	Not provided in method	0.23	20
Iron	7439-89-6	---	200	Not provided in method	Not provided in method	8.3	200
Lead	7439-92-1	---	2	Not provided in method	Not provided in method	0.024	2
Magnesium	7439-95-4	---	1000	Not provided in method	Not provided in method	1.6	1000
Manganese	7439-96-5	---	4	Not provided in method	Not provided in method	1	4
Nickel	7440-02-0	---	20	Not provided in method	Not provided in method	0.63	20
Potassium	7440-09-7	---	1000	Not provided in method	Not provided in method	18	1000
Selenium	7782-49-2	---	2	Not provided in method	Not provided in method	0.32	2
Silver	7440-22-4	---	2	Not provided in method	Not provided in method	0.014	2
Sodium	7440-23-5	---	1000	Not provided in method	Not provided in method	25	1000
Titanium	7440-32-6	---	20	Not provided in method	Not provided in method	0.59	20
Thallium	7440-28-0	---	2	Not provided in method	Not provided in method	0.079	2
Vanadium	7440-62-2	---	4	Not provided in method	Not provided in method	0.3	4
Zinc	7440-66-6	---	20	Not provided in method	Not provided in method	0.5	20
Mercury	7439-97-6	---	0.2	Not provided in method	Not provided in method	0.05	0.2
Methylmercury	22967-92-6	---	0.05 ng/L	0.2 ng/L	0.06 ng/L	0.013 ng/L	0.05 ng/L

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Butyltins
Concentration Level: Low
Analytical Method/SOP Reference: L-6

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
Dibutyltin	14488-53-0	---	0.039	Not provided in method	0.039	0.0084	0.039
Monobuyltin	78763-54-9	---	0.5	Not provided in method	0.50	0.50	0.5
Tetrabutyltin	1461-25-2	---	0.05	Not provided in method	0.050	0.012	0.05
Tributyltin	36643-28-4	---	0.045	Not provided in method	0.045	0.012	0.045

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Chlorinated Herbicides
Concentration Level: Low
Analytical Method/SOP Reference: L-7

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
2,4-D	94-75-7	---	4	Not provided in method	2	0.45	4
2,4-DB	94-82-6	---	4	Not provided in method	2	0.46	4
2,4,5-TP(Silvex)	93-72-6	---	1	Not provided in method	1	0.11	1
2,4,5-T	93-76-5	---	1	Not provided in method	1	0.10	1

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Semivolatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-8

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
Benzaldehyde	100-52-7	---	25	Not provided in method	Not provided in method	0.78	25
Phenol	108-95-2	---	10	Not provided in method	Not provided in method	0.44	10
bis(2-Chloroethyl) ether	111-44-4	---	10	Not provided in method	Not provided in method	0.85	10
2-Chlorophenol	95-57-8	---	10	Not provided in method	Not provided in method	0.79	10
2-Methylphenol	95-48-7	---	10	Not provided in method	Not provided in method	0.51	10
2,2'-oxybis(1-Chloropropane)	108-60-1	---	10	Not provided in method	Not provided in method	0.85	10
Acetophenone	98-86-2	---	10	Not provided in method	Not provided in method	0.89	10
4-Methylphenol	106-44-5	---	10	Not provided in method	Not provided in method	1.2	10
N-Nitroso-di-n-propylamine	621-64-7	---	10	Not provided in method	Not provided in method	0.73	10
Hexachloroethane	67-72-1	---	10	Not provided in method	Not provided in method	0.78	10
Nitrobenzene	98-95-3	---	10	Not provided in method	Not provided in method	0.78	10
Isophorone	78-59-1	---	10	Not provided in method	Not provided in method	0.66	10
2-Nitrophenol	88-75-5	---	10	Not provided in method	Not provided in method	0.9	10
2,4-Dimethylphenol	105-67-9	---	10	Not provided in method	Not provided in method	0.75	10
bis(2-Chloroethoxy) methane	111-91-1	---	10	Not provided in method	Not provided in method	1.4	10
2,4-Dichlorophenol	120-83-2	---	10	Not provided in method	Not provided in method	0.93	10
4-Chloroaniline	106-47-8	---	10	Not provided in method	Not provided in method	0.5	10
Hexachlorobutadiene	87-68-3	---	10	Not provided in method	Not provided in method	0.78	10
Caprolactam	105-60-2	---	10	Not provided in method	Not provided in method	0.36	10
4-Chloro-3-methyphenol	59-50-7	---	10	Not provided in method	Not provided in method	1	10
Hexachlorocyclopentadiene	77-47-4	---	10	Not provided in method	Not provided in method	1.3	10
2,4,6-Trichlorophenol	88-06-2	---	10	Not provided in method	Not provided in method	0.64	10
2,4,5-Trichlorophenol	95-95-4	---	25	Not provided in method	Not provided in method	0.42	25
1,1'-Biphenyl	92-52-4	---	10	Not provided in method	Not provided in method	0.52	10

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Semivolatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-8

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
2-Chloronaphthalene	91-58-7	---	10	Not provided in method	Not provided in method	0.7	10
2-Nitroaniline	88-74-4	---	25	Not provided in method	Not provided in method	1.3	25
Dimethylphthalate	131-11-3	---	10	Not provided in method	Not provided in method	0.65	10
2,6-Dinitrotoluene	606-20-2	---	10	Not provided in method	Not provided in method	0.54	10
3-Nitroaniline	99-09-2	---	25	Not provided in method	Not provided in method	1.3	25
2,4-Dinitrophenol	51-28-5	---	25	Not provided in method	Not provided in method	1.6	25
4-Nitrophenol	100-02-7	---	25	Not provided in method	Not provided in method	1.1	25
Dibenzofuran	132-64-9	---	10	Not provided in method	Not provided in method	0.5	10
2,4-Dinitrotoluene	121-14-2	---	10	Not provided in method	Not provided in method	0.69	10
Diethylphthalate	84-66-2	---	10	Not provided in method	Not provided in method	0.73	10
4-Chlorophenyl-phenylether	7005-72-3	---	10	Not provided in method	Not provided in method	0.66	10
4-Nitroaniline	100-01-6	---	25	Not provided in method	Not provided in method	1.8	25
4,6-Dinitro-2-methylphenol	534-52-1	---	25	Not provided in method	Not provided in method	1.7	25
N-Nitrosodiphenylamine	86-30-6	---	12	Not provided in method	Not provided in method	0.63	12
1,2,4,5-Tetrachlorobenzene	95-94-3	---	10	Not provided in method	Not provided in method	0.67	10
4-Bromophenyl-phenylether	101-55-3	---	10	Not provided in method	Not provided in method	0.87	10
Atrazine	1912-24-9	---	10	Not provided in method	Not provided in method	0.74	10
Carbazole	86-74-8	---	10	Not provided in method	Not provided in method	0.69	10
Di-n-butylphthalate	84-74-2	---	10	Not provided in method	Not provided in method	0.68	10
Butylbenzylphthalate	85-68-7	---	10	Not provided in method	Not provided in method	1.3	10
3,3'-Dichlorobenzidene	91-94-1	---	10	Not provided in method	Not provided in method	1.2	10
bis-(2-Ethylhexyl)phthalate	117-81-7	---	10	Not provided in method	Not provided in method	1.1	10
Di-n-octylphthalate	117-84-0	---	10	Not provided in method	Not provided in method	0.99	10
2,3,4,6-Tetrachlorophenol	58-90-2	---	10	Not provided in method	Not provided in method	0.58	10

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Semivolatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-8

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
Azobenzene	103-33-3	---	10	Not provided in method	Not provided in method	0.54	10
Benzidine	92-87-5	---	25	Not provided in method	Not provided in method	6.8	25
Benzoic Acid	65-85-0	---	110	Not provided in method	Not provided in method	5.4	110
Pentachlorophenol	87-86-5	---	25	Not provided in method	Not provided in method	2.2	25
Pyridine	110-86-1	---	10	Not provided in method	Not provided in method	0.65	10

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Semivolatile Organic SIM
Concentration Level: Low
Analytical Method/SOP Reference: L-9

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
2-Methylnaphthalene	91-57-6	---	0.02	Not provided in method	Not provided in method	0.0012	0.02
Acenaphthylene	208-96-8	---	0.02	Not provided in method	Not provided in method	0.001	0.02
Acenaphthene	83-32-9	---	0.02	Not provided in method	Not provided in method	0.0011	0.02
Anthracene	120-12-7	---	0.02	Not provided in method	Not provided in method	0.0007	0.02
Benzo[b]fluoranthene	205-99-2	---	0.02	Not provided in method	Not provided in method	0.001	0.02
Benzo[k]fluoranthene	207-08-9	---	0.02	Not provided in method	Not provided in method	0.0011	0.02
Benzo[a]anthracene	56-55-3	---	0.02	Not provided in method	Not provided in method	0.0012	0.02
Benzo[a]pyrene	50-32-8	---	0.02	Not provided in method	Not provided in method	0.00051	0.02
Benzo{e}pyrene	192-97-2	---	0.02	Not provided in method	Not provided in method	0.0013	0.02
Indeno[1,2,3-c,d]-pyrene	193-39-5	---	0.02	Not provided in method	Not provided in method	0.0031	0.02
Dibenzo[a,h]anthracene	53-70-3	---	0.02	Not provided in method	Not provided in method	0.0038	0.02
Naphthalene	91-20-3	---	0.02	Not provided in method	Not provided in method	0.0015	0.02
C1-Naphthalenes	STL00916	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C2-Naphthalenes	STL00917	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C3-Naphthalenes	STL00918	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C4-Naphthalenes	STL00919	---	0.02	Not provided in method	Not provided in method	0.02	0.02
1-Methylnaphthalene	90-12-0	---	0.02	Not provided in method	Not provided in method	0.0015	0.02
Fluorene	86-73-7	---	0.02	Not provided in method	Not provided in method	0.0011	0.02
C1-Fluorenes	STL00913	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C2-Fluorenes	STL00914	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C3-Fluorenes	STL00915	---	0.02	Not provided in method	Not provided in method	0.02	0.02
Phenanthrene	85-01-8	---	0.02	Not provided in method	Not provided in method	0.00079	0.02
C1-Phenanthrenes/Anthracenes	STL00901	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C2-Phenanthrenes/Anthracenes	STL00902	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C3-Phenanthrenes/Anthracenes	STL00903	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C4-Phenanthrenes/Anthracenes	STL00904	---	0.02	Not provided in method	Not provided in method	0.02	0.02

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Semivolatile Organic SIM
Concentration Level: Low
Analytical Method/SOP Reference: L-9

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
Fluoranthene	206-44-0	---	0.02	Not provided in method	Not provided in method	0.00057	0.02
Pyrene	129-00-0	---	0.02	Not provided in method	Not provided in method	0.0012	0.02
C1-Fluoranthenes/pyrene	STL00912	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C2-Fluoranthenes/Pyrene	STL00968	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C3-Fluoranthenes/Pyrene	STL00969	---	0.02	Not provided in method	Not provided in method	0.02	0.02
Chrysene	218-01-9	---	0.02	Not provided in method	Not provided in method	0.00091	0.02
C1-Chrysenes	STL00905	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C2-Chrysenes	STL00906	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C3-Chrysenes	STL00907	---	0.02	Not provided in method	Not provided in method	0.02	0.02
C4-Chrysenes	STL00908	---	0.02	Not provided in method	Not provided in method	0.02	0.02
Perylene	198-55-0	---	0.02	Not provided in method	Not provided in method	0.00055	0.02
Benzo[g,h,i]perylene	191-24-2	---	0.02	Not provided in method	Not provided in method	0.002	0.02

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Aroclor PCBs
Concentration Level: Low
Analytical Method/SOP Reference: L-10

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
Aroclor-1016	12674-11-2	---	0.5	Not provided in method	1.0	0.031	0.5
Aroclor-1221	11104-28-2	---	0.5	Not provided in method	1.0	0.041	0.5
Aroclor-1232	11141-16-5	---	0.5	Not provided in method	1.0	0.065	0.5
Aroclor-1242	53469-21-9	---	0.5	Not provided in method	1.0	0.037	0.5
Aroclor-1248	12672-29-6	---	0.5	Not provided in method	1.0	0.034	0.5
Aroclor-1254	11097-69-1	---	0.5	Not provided in method	1.0	0.044	0.5
Aroclor-1260	11096-82-5	---	0.5	Not provided in method	1.0	0.03	0.5
Aroclor-1262	37324-23-5	---	0.5	Not provided in method	1.0	0.044	0.5
Aroclor-1268	11100-14-4	---	0.5	Not provided in method	1.0	0.02	0.5

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Pesticides
Concentration Level: Low
Analytical Method/SOP Reference: L-11

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
2,4'-DDD	53-19-0	---	0.016	Not provided in method	Not provided in method	0.00087	0.016
2,4'-DDE	3424-82-6	---	0.016	Not provided in method	Not provided in method	0.00095	0.016
2,4'-DDT	789-02-6	---	0.016	Not provided in method	Not provided in method	0.00084	0.016
4,4'-DDD	72-54-8	---	0.016	Not provided in method	Not provided in method	0.0011	0.016
4,4'-DDE	72-55-9	---	0.016	Not provided in method	Not provided in method	0.00077	0.016
4,4'-DDT	50-29-3	---	0.016	Not provided in method	Not provided in method	0.0043	0.016
Aldrin	309-00-2	---	0.016	Not provided in method	Not provided in method	0.00086	0.016
alpha-BHC	319-84-6	---	0.016	Not provided in method	Not provided in method	0.0013	0.016
alpha-Chlordane	5103-71-9	---	0.016	Not provided in method	Not provided in method	0.00096	0.016
beta-BHC	319-85-7	---	0.016	Not provided in method	Not provided in method	0.0069	0.016
delta-BHC	319-86-8	---	0.016	Not provided in method	Not provided in method	0.00093	0.016
Dieldrin	60-57-1	---	0.016	Not provided in method	Not provided in method	0.00074	0.016
Endosulfan I	959-98-8	---	0.016	Not provided in method	Not provided in method	0.00066	0.016
Endosulfan II	33213-65-9	---	0.016	Not provided in method	Not provided in method	0.0018	0.016
Endosulfan sulfate	1031-07-8	---	0.016	Not provided in method	Not provided in method	0.0011	0.016
Endrin	72-20-8	---	0.016	Not provided in method	Not provided in method	0.0043	0.016
Endrin aldehyde	7421-93-4	---	0.016	Not provided in method	Not provided in method	0.0017	0.016
Endrin ketone	53494-70-5	---	0.016	Not provided in method	Not provided in method	0.0053	0.016
gamma-BHC	58-89-9	---	0.016	Not provided in method	Not provided in method	0.00094	0.016

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Pesticides
Concentration Level: Low
Analytical Method/SOP Reference: L-11

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
gamma-Chlordane	5103-74-2	---	0.016	Not provided in method	Not provided in method	0.00076	0.016
Heptachlor	76-44-8	---	0.016	Not provided in method	Not provided in method	0.0011	0.016
Heptachlor epoxide	1024-57-3	---	0.016	Not provided in method	Not provided in method	0.00075	0.016
Hexachlorobenzene	118-74-1	---	0.016	Not provided in method	Not provided in method	0.00088	0.016
Methoxychlor	72-43-5	---	0.016	Not provided in method	Not provided in method	0.0011	0.016
Nonachlor(cis)	5103-73-1	---	0.016	Not provided in method	Not provided in method	0.00074	0.016
Nonachlor(trans)	39765-80-5	---	0.016	Not provided in method	Not provided in method	0.002	0.016
Oxychlordane	27304-13-8	---	0.016	Not provided in method	Not provided in method	0.0008	0.016
Mirex	2385-85-5	---	0.016	Not provided in method	Not provided in method	0.0061	0.016
Toxaphene	8001-35-2	---	0.2	Not provided in method	Not provided in method	0.38	0.2
Toxaphene Peak 1	STL00100	---	0.2	Not provided in method	Not provided in method	0.043	0.2
Toxaphene Peak 2	STL00109	---	0.2	Not provided in method	Not provided in method	0.043	0.2
Toxaphene Peak 3	STL00220	---	0.2	Not provided in method	Not provided in method	0.043	0.2
Toxaphene Peak 4	STL00083	---	0.2	Not provided in method	Not provided in method	0.043	0.2
Toxaphene Peak 5	STL00051	---	0.2	Not provided in method	Not provided in method	0.043	0.2

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Volatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-12

Analyte	CAS Number	Study Action Level µg/L	Project Quantitation Limit µg/L	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/L	Method QLs µg/L	MDLs µg/L	QLs µg/L
1,3-Dichlorobenzene	541-73-1	---	1	Not provided in method	5.0	0.18	1
1,4-Dichlorobenzene	106-46-7	---	1	Not provided in method	5.0	0.15	1
1,2-Dichlorobenzene	95-50-1	---	1	Not provided in method	5.0	0.15	1
1,2,4-Trichlorobenzene	120-82-1	---	1	Not provided in method	5.0	0.18	1

See the last page of Worksheet #15-1 for a description of footnotes.

QAPP Worksheet #15-1 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Water (continued)

Matrix: Field/Rinse/Equipment Blank
Analytical Group: Miscellaneous
Concentration Level: Low
Analytical Method/SOP Reference: L-14, L-16, L-17, L-23, L-24, L-26, L-13

Analyte	CAS Number	Study Action Level	Project Quantitation Limit	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs	Method QLs	MDLs	QLs
TOC	TOC	---	1.0 mg/L	Not provided in method	Not provided in method	0.24 mg/L	1.0 mg/L
TEPH	TEPH	---	0.30 mg/L	Not provided in method	Not provided in method	0.041 mg/L	0.30 mg/L
Hexavalent Chromium	18540-29-9	---	10 µg/L	Not provided in method	Not provided in method	5 µg/L	10 µg/L
Total Phosphorus	14365-44-2	---	0.1 mg/L	Not provided in method	Not provided in method	0.033 mgL	0.1 mg/L
Total Kjedahl Nitrogen	7727-37-9	---	5 mg/L	Not provided in method	Not provided in method	1.7 mgL	5 mg/L
Ammonia	766-41-7	---	1.0 mg/L	Not provided in method	Not provided in method	0.025 mg/L	0.2 mg/L
Cyanide	57-12-5	---	10 µg/L	Not provided in method	Not provided in method	1.6 µg/L	10 µg/L

Notes:

[a] Analytical MDLs and QLs are those documented in validated methods.
[b] Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

BHC = benzene hexachloride
CAS = Chemical Abstracts Service
HpCDD = heptachlorodibenzo-*p*-dioxin
HpCDF = heptachlorodibenzofuran
HxCDD = hexachlorodibenzo-*p*-dioxin
HxCDF = hexachlorodibenzofuran
MDL = method detection limit
mg/L = milligrams per liter
NA = not applicable
ng/L = nanogram per liter
OCDD = octachlorodibenzo-*p*-dioxin
OCDF = octachlorodibenzofuran
PCBs= polychlorinated biphenyls
PeCDD = pentachlorodibenzo-*p*-dioxin
PeCDF = pentachlorodibenzofuran
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
pg/L = picograms per liter
QL = quantitation limit
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
TCDD = tetrachlorodibenzo-*p*-dioxin
TCDF = tetrachlorodibenzofuran
TEPH = total extractable petroleum hydrocarbon
TOC = total organic carbon
µg/L = micrograms per liter

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment

Matrix: Sediment
Analytical Group: PCDDs/PCDFs
Concentration Level: Low
Analytical Method/SOP Reference: L-1

Analyte	CAS Number	Study Action Level ng/kg	Project Quantitation Limit ng/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs ng/kg	Method QLs ng/kg	MDLs ng/kg	QLs ng/kg
2,3,7,8-TCDD	1746-01-6	2.5e-4	0.5	Not provided in method	1	0.0510	0.5
1,2,3,7,8-PeCDD	40321-76-4	4.5e-4	2.5	Not provided in method	5	0.155	2.5
1,2,3,6,7,8-HxCDD	57653-85-7	4.5e-3	2.5	Not provided in method	5	0.269	2.5
1,2,3,4,7,8-HxCDD	39227-28-6	4.5e-3	2.5	Not provided in method	5	0.211	2.5
1,2,3,7,8,9-HxCDD	19408-74-3	4.5e-3	2.5	Not provided in method	5	0.215	2.5
1,2,3,4,6,7,8-HpCDD	35822-46-9	0.45	2.5	Not provided in method	5	0.284	2.5
OCDD	3268-87-9	15	5.0	Not provided in method	10	0.446	5.0
2,3,7,8-TCDF	51207-31-9	4.5e-3	0.5	Not provided in method	1	0.0830	0.5
1,2,3,7,8-PeCDF	57117-41-6	0.15	2.5	Not provided in method	5	0.203	2.5
2,3,4,7,8-PeCDF	57117-31-4	1.5e-3	2.5	Not provided in method	5	0.0905	2.5
1,2,3,6,7,8-HxCDF	57117-44-9	4.5e	2.5	Not provided in method	5	0.122	2.5
1,2,3,7,8,9-HxCDF	72918-21-9	4.5e	2.5	Not provided in method	5	0.139	2.5
1,2,3,4,7,8-HxCDF	70648-26-9	4.5e	2.5	Not provided in method	5	0.0983	2.5
2,3,4,6,7,8-HxCDF	60851-34-5	4.5e	2.5	Not provided in method	5	0.219	2.5
1,2,3,4,6,7,8-HpCDF	67562-39-4	0.45	2.5	Not provided in method	5	0.164	2.5
1,2,3,4,7,8,9-HpCDF	55673-89-7	0.45	2.5	Not provided in method	5	0.184	2.5
OCDF	39001-02-0	15	5.0	Not provided in method	10	0.240	5.0

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level ng/kg	Project Quantitation Limit ng/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs ng/kg	Method QLs ng/kg	MDLs ng/kg	QLs ng/kg
PCB1	2051-60-7	---	2.5	8	20	0.852	2.5
PCB2	2051-61-8	---	2.5	0.4	1	1.15	2.5
PCB3	2051-62-9	---	2.5	9	20	0.777	2.5
PCB4/10	13029-08-8/33146-45-1	---	10	Not provided in method	Not provided in method	1.75	10
PCB6	25569-80-6	---	5	1	5	1.17	5
PCB5/8	16605-91-7/34883-43-7	---	10	Not provided in method	Not provided in method	2.93	10
PCB7/9	33284-50-3/34883-39-1	---	10	2	5	1.76	10
PCB11	2050-67-1	---	5	10	20	1.32	5
PCB12/13	2974-92-7/2974-90-5	---	10	3	10	0.736	10
PCB14	34883-41-5	---	5	3	10	1.39	5
PCB15	2050-68-2	---	5	18	50	1.49	5
PCB16/32	38444-78-9/38444-77-8	---	2.5	Not provided in method	Not provided in method	1.75	2.5
PCB17	37680-66-3	---	5.0	9	20	0.768	5.0
PCB18	37680-65-2	---	5.0	17	50	0.749	5.0
PCB19	38444-73-4	---	5.0	4	10	1.07	5.0
PCB20/21/33	38444-84-7/ 55702-46-0/38444-86-9	---	7.5	Not provided in method	Not provided in method	4.94	7.5
PCB22	38444-85-8	---	2.5	9	20	1.98	2.5
PCB23	55720-44-0	---	2.5	5	20	1.21	2.5
PCB24/27	55702-45-9/38444-76-7	---	5.0	Not provided in method	Not provided in method	1.20	5.0
PCB25	55712-37-3	---	2.5	5	20	1.35	2.5
PCB26	38444-81-4	---	2.5	8	20	1.77	2.5
PCB28	7012-37-5	---	2.5	19	50	0.731	2.5
PCB29	15862-07-4	---	2.5	8	20	1.36	2.5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level ng/kg	Project Quantitation Limit ng/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs ng/kg	Method QLs ng/kg	MDLs ng/kg	QLs ng/kg
PCB30	35693-92-6	---	2.5	17	50	1.22	2.5
PCB31	16606-02-3	---	2.5	15	50	0.751	2.5
PCB34	37680-68-5	---	2.5	7	20	1.79	2.5
PCB35	37680-69-6	---	2.5	8	20	0.274	2.5
PCB36	38444-87-0	---	2.5	8	20	1.05	2.5
PCB37	38444-90-5	---	2.5	13	50	0.666	2.5
PCB38	53555-66-1	---	2.5	8	20	1.22	2.5
PCB39	38444-88-1	---	2.5	9	20	0.777	2.5
PCB40	38444-93-8	---	2.5	12	50	0.856	2.5
PCB41/64/71/ 72	52663-59-9/52663-58-8/ 41464-46-4/41464-42-0	---	10.0	Not provided in method	Not provided in method	2.49	10.0
PCB42/59	36559-22-5/74472-33-6	---	5.0	6	20	1.73	5.0
PCB43/49	70362-46-8/41464-40-8	---	5.0	Not provided in method	Not provided in method	1.41	5.0
PCB44	41464-39-5	---	2.5	19	50	1.27	2.5
PCB45	70362-45-7	---	2.5	5	20	1.15	2.5
PCB46	41464-47-5	---	2.5	10	20	0.577	2.5
PCB47	2437-79-8	---	2.5	19	50	2.16	2.5
PCB48/75	70362-47-9/32598-12-2	---	5.0	Not provided in method	Not provided in method	1.23	5.0
PCB50	62796-65-0	---	2.5	6	20	0.907	2.5
PCB51	68194-04-7	---	2.5	5	20	0.512	2.5
PCB52/69	35693-99-3/60233-24-1	---	5.0	Not provided in method	Not provided in method	1.58	5.0
PCB53	41464-41-9	---	2.5	6	20	1.35	2.5
PCB54	15968-05-5	---	2.5	12	50	0.538	2.5
PCB55	74338-24-2	---	2.5	12	50	0.411	2.5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level ng/kg	Project Quantitation Limit ng/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs ng/kg	Method QLs ng/kg	MDLs ng/kg	QLs ng/kg
PCB56/60	41464-43-1/33025-41-1	---	5.0	Not provided in method	Not provided in method	1.01	5.0
PCB57	70424-67-8	---	2.5	12	50	0.597	2.5
PCB58	41464-49-7	---	2.5	13	50	0.644	2.5
PCB61/70	33284-53-6/32598-11-1	---	5.0	17	50	1.56	5.0
PCB62	54230-22-7	---	2.5	6	20	0.583	2.5
PCB63	74472-34-7	---	2.5	14	50	0.640	2.5
PCB65	33284-54-7	---	2.5	19	50	0.666	2.5
PCB67	73575-53-8	---	2.5	15	50	0.653	2.5
PCB68	73575-52-7	---	2.5	15	50	0.867	2.5
PCB73	74338-23-1	---	2.5	16	50	1.04	2.5
PCB74	32690-93-0	---	2.5	17	50	0.640	2.5
PCB76/66	70362-48-0/32598-10-0	---	5.0	Not provided in method	Not provided in method	0.653	5.0
PCB77	32598-13-3	---	2.5	17	50	0.446	2.5
PCB78	70362-49-1	---	2.5	17	50	0.914	2.5
PCB79	41464-48-6	---	2.5	17	50	0.499	2.5
PCB80	33284-52-5	---	2.5	18	50	0.468	2.5
PCB81	70362-50-4	---	2.5	18	50	0.843	2.5
PCB82	52663-62-4	---	2.5	13	50	0.767	2.5
PCB83	60145-20-2	---	2.5	22	50	0.861	2.5
PCB84/92	52663-60-2/52663-61-3	---	5.0	12	50	1.38	5.0
PCB85/116	65510-45-4/18259-05-7	---	5.0	10	20	1.41	5.0
PCB86	55312-69-1	---	2.5	15	50	0.893	2.5
PCB87/117/125	38380-02-8/ 68194-11-6/74472-39-2	---	7.5	Not provided in method	Not provided in method	1.32	7.5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level ng/kg	Project Quantitation Limit ng/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs ng/kg	Method QLs ng/kg	MDLs ng/kg	QLs ng/kg
PCB88/91	55215-17-3/68194-05-8	---	5.0	12	50	1.78	5.0
PCB89	73575-57-2	---	2.5	19	50	0.855	2.5
PCB90/101	68194-07-0/37680-73-2	---	5.0	24	100	1.66	5.0
PCB93	73575-56-1	---	2.5	22	50	1.74	2.5
PCB94	73575-55-0	---	2.5	12	50	0.497	2.5
PCB95/98/102	38379-99-6/ 60233-25-2/68194-06-9	---	7.5	22	50	2.26	7.5
PCB96	73575-54-9	---	2.5	21	50	0.926	2.5
PCB97	41464-51-1	---	2.5	15	50	1.59	2.5
PCB99	38380-01-7	---	2.5	22	50	0.922	2.5
PCB100	39485-83-1	---	2.5	22	50	0.596	2.5
PCB103	60145-21-3	---	2.5	23	50	0.582	2.5
PCB104	56558-16-8	---	2.5	23	50	0.778	2.5
PCB105	32598-14-4	---	2.5	11	20	0.624	2.5
PCB106/118	70424-69-0/31508-00-6	---	5.0	Not provided in method	Not provided in method	1.22	5.0
PCB107/109	70424-68-9/74472-35-8	---	5.0	Not provided in method	Not provided in method	1.37	5.0
PCB108/112	70362-41-3/74472-36-9	---	5.0	Not provided in method	Not provided in method	0.896	5.0
PCB110	38380-03-9	---	2.5	24	100	0.332	2.5
PCB111/115	39635-32-0/74472-38-1	---	5.0	24	100	0.848	5.0
PCB113	68194-10-5	---	2.5	24	100	1.07	2.5
PCB114	74472-37-0	110	2.5	12	50	0.588	2.5
PCB119	56558-17-9	---	2.5	15	50	0.581	2.5
PCB120	68194-12-7	---	2.5	15	50	0.797	2.5
PCB121	56558-18-0	---	2.5	21	50	1.40	2.5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level ng/kg	Project Quantitation Limit ng/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs ng/kg	Method QLs ng/kg	MDLs ng/kg	QLs ng/kg
PCB122	76842-07-4	---	2.5	12	50	0.400	2.5
PCB123	65510-44-3	110	2.5	15	50	1.05	2.5
PCB124	70424-70-3	---	2.5	27	100	0.434	2.5
PCB126	57465-28-8	0.034	2.5	14	50	0.727	2.5
PCB127	39635-33-1	---	2.5	28	100	0.648	2.5
PCB128/162	38380-07-3/39635-34-2	---	5.0	Not provided in method	Not provided in method	0.352	5.0
PCB129	55215-18-4	---	2.5	21	50	0.401	2.5
PCB130	52663-66-8	---	2.5	14	50	1.18	2.5
PCB131	61798-70-7	---	2.5	12	50	0.490	2.5
PCB132/161	38380-05-1/74472-43-8	---	5.0	Not provided in method	Not provided in method	0.686	5.0
PCB133/142	35694-04-3/41411-61-4	---	5.0	Not provided in method	Not provided in method	0.968	5.0
PCB134/143	52704-70-8/68194-15-0	---	5.0	13	50	1.21	5.0
PCB135	52744-13-5	---	2.5	11	50	0.925	2.5
PCB136	38411-22-2	---	2.5	9	20	0.869	2.5
PCB137	35694-06-5	---	2.5	30	100	0.848	2.5
PCB138/163/164	35065-28-2/ 74472-44-9/74472-45-0	---	7.5	Not provided in method	Not provided in method	1.18	7.5
PCB139/149	56030-56-9/38380-04-0	---	5.0	Not provided in method	Not provided in method	1.91	5.0
PCB140	59291-64-4	---	2.5	20	50	1.65	2.5
PCB141	52712-04-6	---	2.5	9	20	0.675	2.5
PCB144	68194-14-9	---	2.5	17	50	1.53	2.5
PCB145	74472-40-5	---	2.5	32	100	0.429	2.5
PCB146/165	51908-16-8/74472-46-1	---	5.0	Not provided in method	Not provided in method	0.601	5.0
PCB147	68194-13-8	---	2.5	18	50	0.526	2.5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level ng/kg	Project Quantitation Limit ng/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs ng/kg	Method QLs ng/kg	MDLs ng/kg	QLs ng/kg
PCB148	74472-41-6	---	2.5	32	100	1.06	2.5
PCB150	68194-08-1	---	2.5	33	100	1.28	2.5
PCB151	52663-63-5	---	2.5	11	50	1.19	2.5
PCB152	68194-09-2	---	2.5	24	100	0.314	2.5
PCB153	35065-27-1	---	2.5	13	50	0.973	2.5
PCB154	60145-22-4	---	2.5	11	50	0.704	2.5
PCB155	33979-03-2	---	2.5	34	100	0.725	2.5
PCB156	38380-08-4	110	2.5	13	50	0.566	2.5
PCB157	69782-90-7	110	2.5	13	50	0.352	2.5
PCB158/160	74472-42-7/41411-62-5	---	5.0	Not provided in method	Not provided in method	0.590	5.0
PCB159	39635-35-3	---	2.5	35	100	0.790	2.5
PCB166	41411-63-6	---	2.5	12	50	0.434	2.5
PCB167	52663-72-6	---	2.5	11	50	0.652	2.5
PCB168	59291-65-5	---	2.5	13	50	0.734	2.5
PCB169	32774-16-6	0.11	2.5	16	50	0.808	2.5
PCB170	35065-30-6	---	2.5	16	50	0.734	2.5
PCB171	52663-71-5	---	2.5	37	100	1.05	2.5
PCB172	52663-74-8	---	2.5	38	100	0.701	2.5
PCB173	68194-16-1	---	2.5	37	100	1.06	2.5
PCB174	38411-25-5	---	2.5	19	50	0.425	2.5
PCB175	40186-70-7	---	2.5	38	100	0.678	2.5
PCB176	52663-65-7	---	2.5	39	100	1.34	2.5
PCB177	52663-70-4	---	2.5	14	50	0.825	2.5
PCB178	52663-67-9	---	2.5	22	50	0.638	2.5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level ng/kg	Project Quantitation Limit ng/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs ng/kg	Method QLs ng/kg	MDLs ng/kg	QLs ng/kg
PCB179	52663-64-6	---	2.5	23	50	0.990	2.5
PCB180	35065-29-3	---	2.5	14	50	0.710	2.5
PCB181	74472-47-2	---	2.5	40	100	0.255	2.5
PCB182/187	60145-23-5/52663-68-0	---	5.0	Not provided in method	Not provided in method	1.19	5.0
PCB183	52663-69-1	---	2.5	40	100	0.817	2.5
PCB184	74472-48-3	---	2.5	40	100	0.917	2.5
PCB185	52712-05-7	---	2.5	40	100	1.08	2.5
PCB186	74472-49-4	---	2.5	41	100	1.05	2.5
PCB188	74487-85-7	---	2.5	23	50	0.438	2.5
PCB189	39635-31-9	110	2.5	18	50	0.508	2.5
PCB190	41411-64-7	---	2.5	23	50	1.12	2.5
PCB191	74472-50-7	---	2.5	42	100	0.810	2.5
PCB192	74472-51-8	---	2.5	42	100	1.02	2.5
PCB193	69782-91-8	---	2.5	14	50	0.431	2.5
PCB194	35694-08-7	---	2.5	17	50	0.399	2.5
PCB195	52663-78-2	---	2.5	43	100	0.586	2.5
PCB196/203	42740-50-1/52663-76-0	---	5.0	Not provided in method	Not provided in method	1.77	5.0
PCB197	33091-17-7	---	2.5	25	100	0.324	2.5
PCB198	68194-17-2	---	2.5	20	50	1.18	2.5
PCB199	52663-75-9	---	2.5	20	50	0.626	2.5
PCB200	52663-73-7	---	2.5	25	100	1.19	2.5
PCB201	40186-71-8	---	2.5	44	100	2.28	2.5
PCB202	2136-99-4	---	2.5	44	100	0.541	2.5
PCB204	74472-52-9	---	2.5	45	100	0.871	2.5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level ng/kg	Project Quantitation Limit ng/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs ng/kg	Method QLs ng/kg	MDLs ng/kg	QLs ng/kg
PCB205	74472-53-0	---	2.5	45	100	0.392	2.5
PCB206	40186-72-9	---	2.5	45	100	0.540	2.5
PCB207	52663-79-3	---	2.5	45	100	0.561	2.5
PCB208	52663-77-1	---	2.5	46	100	0.432	2.5
PCB209	2051-24-3	---	2.5	15	50	0.640	2.5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: TAL Metals, Mercury, Methylmercury, Titanium
Concentration Level: Low
Analytical Method/SOP Reference: L-3, L-4, L-5, and L-29

Analyte	CAS Number	Study Action Level mg/kg	Project Quantitation Limit mg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs mg/kg	Method QLs mg/kg	MDLs mg/kg	QLs mg/kg
Aluminum	7429-90-5	7700	8	Not provided in method	Not provided in method	0.21	8
Antimony	7440-36-0	3.1	2	Not provided in method	Not provided in method	0.0082	2
Arsenic	7440-38-2	0.39	0.2	Not provided in method	Not provided in method	0.012	0.2
Barium	7440-39-3	48	10	Not provided in method	Not provided in method	0.023	10
Beryllium	7440-41-7	16	0.2	Not provided in method	Not provided in method	0.0049	0.2
Cadmium	7440-43-9	1.2	0.2	Not provided in method	Not provided in method	0.0075	0.2
Calcium	7440-70-2	---	100	Not provided in method	Not provided in method	1.2	100
Chromium	7440-47-3	81	0.4	Not provided in method	Not provided in method	0.014	0.4
Cobalt	7440-48-4	2.3	1	Not provided in method	Not provided in method	0.0017	1
Copper	7440-50-8	13.3	2	Not provided in method	Not provided in method	0.016	2
Iron	7439-89-6	5500	20	Not provided in method	Not provided in method	0.6	20
Lead	7439-92-1	10.6	0.2	Not provided in method	Not provided in method	0.0048	0.2
Magnesium	7439-95-4	---	100	Not provided in method	Not provided in method	0.34	100
Manganese	7439-96-5	180	0.4	Not provided in method	Not provided in method	0.019	0.4
Nickel	7440-02-0	21	2	Not provided in method	Not provided in method	0.005	2
Potassium	7440-09-7	---	100	Not provided in method	Not provided in method	1.5	100
Selenium	7782-49-2	1	0.2	Not provided in method	Not provided in method	0.035	0.2
Silver	7440-22-4	1	0.2	Not provided in method	Not provided in method	0.0016	0.2
Sodium	7440-23-5	---	100	Not provided in method	Not provided in method	0.63	100
Titanium	7440-32-6	0.078	2	Not provided in method	Not provided in method	0.076	2
Thallium	7440-28-0	14000	0.2	Not provided in method	Not provided in method	0.01	0.2
Vanadium	7440-62-2	39	0.4	Not provided in method	Not provided in method	0.023	0.4
Zinc	7440-66-6	6.62	2	Not provided in method	Not provided in method	0.11	2
Mercury	7439-97-6	0.037	0.033	Not provided in method	Not provided in method	0.0091	0.033
Methylmercury	22967-92-6	---	0.0001	Not provided in method	Not provided in method	0.000037	0.0001

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Butyltins
Concentration Level: Low
Analytical Method/SOP Reference: L-6

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
Dibutyltin	14488-53-0	1.8	1.3	Not provided in method	Not provided in method	0.40	1.3
Monobuyltin	78763-54-9	1.8	5	Not provided in method	Not provided in method	5	5
Tetrabutyltin	1461-25-2	1.8	1.7	Not provided in method	Not provided in method	0.44	1.7
Tributyltin	36643-28-4	1.8	1.5	Not provided in method	Not provided in method	0.67	1.5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Chlorinated Herbicides
Concentration Level: Low
Analytical Method/SOP Reference: L-7

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
2,4-D	94-75-7	1273	80	0.11	Not provided in method	5.5	80
2,4-DB	94-82-6	49000	80	Not provided in method	Not provided in method	6.1	80
2,4,5-TP (Silvex)	93-72-6	675	20	0.28	Not provided in method	2.1	20
2,4,5-T	93-76-5	12300	20	Not provided in method	Not provided in method	2.5	20

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Semivolatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-8

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
Benzaldehyde	100-52-7	---	330	Not provided in method	Not provided in method	16	330
Phenol	108-95-2	130	330	Not provided in method	Not provided in method	40	330
bis(2-Chloroethyl) ether	111-44-4	210	330	Not provided in method	Not provided in method	15	330
2-Chlorophenol	95-57-8	8	330	Not provided in method	Not provided in method	34	330
2-Methylphenol	95-48-7	55.4	330	Not provided in method	Not provided in method	40	330
2,2'-oxybis(1-Chloropropane)	108-60-1	---	330	Not provided in method	Not provided in method	16	330
Acetophenone	98-86-2	---	330	Not provided in method	Not provided in method	16	330
3 & 4-Methylphenol	106-44-5	670	670	Not provided in method	Not provided in method	74	670
N-Nitroso-di-n-propylamine	621-64-7	69	330	Not provided in method	Not provided in method	46	330
Hexachloroethane	67-72-1	73	330	Not provided in method	Not provided in method	34	330
Nitrobenzene	98-95-3	145	330	Not provided in method	Not provided in method	41	330
Isophorone	78-59-1	432	330	Not provided in method	Not provided in method	42	330
2-Nitrophenol	88-75-5	---	330	Not provided in method	Not provided in method	37	330
2,4-Dimethylphenol	105-67-9	304	330	Not provided in method	Not provided in method	52	330
bis(2-Chloroethoxy) methane	111-91-1	18000	330	Not provided in method	Not provided in method	15	330
2,4-Dichlorophenol	120-83-2	5	330	Not provided in method	Not provided in method	33	330
4-Chloroaniline	106-47-8	146	330	Not provided in method	Not provided in method	32	330
Hexachlorobutadiene	87-68-3	1.3	330	Not provided in method	Not provided in method	35	330
Caprolactam	105-60-2	---	330	Not provided in method	Not provided in method	35	330
4-Chloro-3-methyphenol	59-50-7	388	330	Not provided in method	Not provided in method	40	330
Hexachlorocyclopentadiene	77-47-4	901	330	Not provided in method	Not provided in method	65	330
2,4,6-Trichlorophenol	88-06-2	6	330	Not provided in method	Not provided in method	34	330
2,4,5-Trichlorophenol	95-95-4	3	830	Not provided in method	Not provided in method	34	830
1,1'-Biphenyl	92-52-4	1220	330	Not provided in method	Not provided in method	15	330

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Semivolatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-8

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
2-Chloronaphthalene	91-58-7	417	330	Not provided in method	Not provided in method	43	330
2-Nitroaniline	88-74-4	61000	830	Not provided in method	Not provided in method	37	830
Dimethylphthalate	131-11-3	4900000	330	Not provided in method	Not provided in method	15	330
2,6-Dinitrotoluene	606-20-2	39.8	330	Not provided in method	Not provided in method	30	330
3-Nitroaniline	99-09-2	24000	830	Not provided in method	Not provided in method	38	830
2,4-Dinitrophenol	51-28-5	6.21	830	Not provided in method	Not provided in method	140	830
4-Nitrophenol	100-02-7	13.3	830	Not provided in method	Not provided in method	84	830
Dibenzofuran	132-64-9	7300	330	Not provided in method	Not provided in method	16	330
2,4-Dinitrotoluene	121-14-2	14.4	330	Not provided in method	Not provided in method	24	330
Diethylphthalate	84-66-2	6	330	Not provided in method	Not provided in method	15	330
4-Chlorophenyl-phenylether	7005-72-3	---	330	Not provided in method	Not provided in method	15	330
4-Nitroaniline	100-01-6	24000	830	Not provided in method	Not provided in method	32	830
4,6-Dinitro-2-methylphenol	534-52-1	104	830	Not provided in method	Not provided in method	97	830
N-Nitrosodiphenylamine	86-30-6	99000	387	Not provided in method	Not provided in method	16	387
1,2,4,5-Tetrachlorobenzene	95-94-3	1252	330	Not provided in method	Not provided in method	40	330
4-Bromophenyl-phenylether	101-55-3	1230	330	Not provided in method	Not provided in method	18	330
Atrazine	1912-24-9	6.62	330	Not provided in method	Not provided in method	16	330
Carbazole	86-74-8	---	330	Not provided in method	Not provided in method	15	330
Di-n-butylphthalate	84-74-2	58	330	Not provided in method	Not provided in method	15	330
Butylbenzylphthalate	85-68-7	63	330	Not provided in method	Not provided in method	20	330
3,3'-Dichlorobenzidine	91-94-1	127	330	Not provided in method	Not provided in method	48	330
bis-(2-Ethylhexyl)phthalate	117-81-7	182.16	330	Not provided in method	Not provided in method	21	330
Di-n-octylphthalate	117-84-0	40600	330	Not provided in method	Not provided in method	23	330
2,3,4,6-Tetrachlorophenol	58-90-2	284	330	Notprovided in method	Not provided in method	37	330

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Semivolatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-8

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
Azobenzene	103-33-3	---	330	Not provided in method	Not provided in method	19	330
Benzidine	92-87-5	---	830	Not provided in method	Not provided in method	97	830
Benzoic Acid	65-85-0	---	830	Not provided in method	Not provided in method	56	830
Pentachlorophenol	87-86-5	---	830	Not provided in method	Not provided in method	68	830
Pyridine	110-86-1	106	330	Not provided in method	Not provided in method	36	330

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Semivolatile Organics SIM
Concentration Level: Low
Analytical Method/SOP Reference: L-9

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
2-Methylnaphthalene	91-57-6	70	0.67	Not provided in method	Not provided in method	0.055	0.67
Acenaphthylene	208-96-8	44	0.67	Not provided in method	Not provided in method	0.053	0.67
Acenaphthene	83-32-9	16	0.67	Not provided in method	Not provided in method	0.053	0.67
Anthracene	120-12-7	85	0.67	Not provided in method	Not provided in method	0.053	0.67
Benzo[b]fluoranthene	205-99-2	150	0.67	Not provided in method	Not provided in method	0.047	0.67
Benzo[k]fluoranthene	207-08-9	240	0.67	Not provided in method	Not provided in method	0.057	0.67
Benzo[a]anthracene	56-55-3	150	0.67	Not provided in method	Not provided in method	0.048	0.67
Benzo[a]pyrene	50-32-8	15	0.67	Not provided in method	Not provided in method	0.047	0.67
Benzo{e}pyrene	192-97-2	170000	0.67	Not provided in method	Not provided in method	0.067	0.67
Indeno[1,2,3-c,d]-pyrene	193-39-5	150	0.67	Not provided in method	Not provided in method	0.077	0.67
Dibenzo[a,h]anthracene	53-70-3	15	0.67	Not provided in method	Not provided in method	0.057	0.67
Naphthalene	91-20-3	160	0.67	Not provided in method	Not provided in method	0.057	0.67
C1-Naphthalenes	STL00916	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C2-Naphthalenes	STL00917	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C3-Naphthalenes	STL00918	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C4-Naphthalenes	STL00919	---	0.67	Not provided in method	Not provided in method	0.67	0.67
1-Methylnaphthalene	90-12-0	1600	0.67	Not provided in method	Not provided in method	0.055	0.67
Fluorene	86-73-7	19	0.67	Not provided in method	Not provided in method	0.055	0.67
C1-Fluorenes	STL00913	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C2-Fluorenes	STL00914	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C3-Fluorenes	STL00915	---	0.67	Not provided in method	Not provided in method	0.67	0.67
Phenanthrene	85-01-8	240	0.67	Not provided in method	Not provided in method	0.07	0.67
C1-Phenanthrenes/Anthracenes	STL00901	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C2-Phenanthrenes/Anthracenes	STL00902	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C3-Phenanthrenes/Anthracenes	STL00903	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C4-Phenanthrenes/Anthracenes	STL00904	---	0.67	Not provided in method	Not provided in method	0.67	0.67

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Semivolatile Organics SIM
Concentration Level: Low
Analytical Method/SOP Reference: L-9

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
Fluoranthene	206-44-0	600	0.67	Not provided in method	Not provided in method	0.061	0.67
Pyrene	129-00-0	665	0.67	Not provided in method	Not provided in method	0.037	0.67
C1-Fluoranthenes/pyrene	STL00912	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C2-Fluoranthenes/Pyrene	STL00968	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C3-Fluoranthenes/Pyrene	STL00969	---	0.67	Not provided in method	Not provided in method	0.67	0.67
Chrysene	218-01-9	384	0.67	Not provided in method	Not provided in method	0.057	0.67
C1-Chrysenes	STL00905	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C2-Chrysenes	STL00906	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C3-Chrysenes	STL00907	---	0.67	Not provided in method	Not provided in method	0.67	0.67
C4-Chrysenes	STL00908	---	0.67	Not provided in method	Not provided in method	0.67	0.67
Perylene	198-55-0	170000	0.67	Not provided in method	Not provided in method	0.046	0.67
Benzo[g,h,i]perylene	191-24-2	170	0.67	Not provided in method	Not provided in method	0.091	0.67

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Aroclor PCBs
Concentration Level: Low
Analytical Method/SOP Reference: L-10

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
Aroclor-1016	12674-11-2	7	17	Not provided in method	Not provided in method	5.6	17
Aroclor-1221	11104-28-2	140	17	Not provided in method	Not provided in method	4.3	17
Aroclor-1232	11141-16-5	140	17	Not provided in method	Not provided in method	3.3	17
Aroclor-1242	53469-21-9	220	17	Not provided in method	Not provided in method	6.7	17
Aroclor-1248	12672-29-6	30	17	Not provided in method	Not provided in method	2	17
Aroclor-1254	11097-69-1	60	17	Not provided in method	Not provided in method	2.8	17
Aroclor-1260	11096-82-5	5	17	Not provided in method	Not provided in method	2.4	17
Aroclor-1262	37324-23-5	---	17	Not provided in method	Not provided in method	1.5	17
Aroclor-1268	11100-14-4	---	17	Not provided in method	Not provided in method	1.4	17

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Pesticides
Concentration Level: Low
Analytical Method/SOP Reference: L-11

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
2,4'-DDD	53-19-0	2000	0.20	Not provided in method	Not provided in method	0.053	0.20
2,4'-DDE	3424-82-6	1400	0.20	Not provided in method	Not provided in method	0.057	0.20
2,4'-DDT	789-02-6	1700	0.20	Not provided in method	Not provided in method	0.067	0.20
4,4'-DDD	72-54-8	2	0.2	Not provided in method	Not provided in method	0.039	0.2
4,4'-DDE	72-55-9	2.2	0.2	Not provided in method	Not provided in method	0.041	0.2
4,4'-DDT	50-29-3	1	0.2	Not provided in method	Not provided in method	0.057	0.2
Aldrin	309-00-2	2	0.2	Not provided in method	Not provided in method	0.046	0.2
alpha-BHC	319-84-6	6	0.2	Not provided in method	Not provided in method	0.043	0.2
alpha-Chlordane	5103-71-9	3.24	0.2	Not provided in method	Not provided in method	0.053	0.2
beta-BHC	319-85-7	5	0.2	Not provided in method	Not provided in method	0.11	0.2
delta-BHC	319-86-8	3	0.2	Not provided in method	Not provided in method	0.043	0.2
Dieldrin	60-57-1	30	0.2	Not provided in method	Not provided in method	0.048	0.2
Endosulfan I	959-98-8	2.9	0.2	Not provided in method	Not provided in method	0.044	0.2
Endosulfan II	33213-65-9	14	0.2	Not provided in method	Not provided in method	0.046	0.2
Endosulfan sulfate	1031-07-8	34.6	0.2	Not provided in method	Not provided in method	0.069	0.2
Endrin	72-20-8	3	0.2	Not provided in method	Not provided in method	0.048	0.2
Endrin aldehyde	7421-93-4	480	0.2	Not provided in method	Not provided in method	0.046	0.2
Endrin ketone	53494-70-5	1800	0.2	Not provided in method	Not provided in method	0.15	0.2
gamma-BHC	58-89-9	3	0.2	Not provided in method	Not provided in method	0.041	0.2

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Pesticides
Concentration Level: Low
Analytical Method/SOP Reference: L-11

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
gamma-Chlordane	5103-74-2	3.24	0.2	Not provided in method	Not provided in method	0.05	0.2
Heptachlor	76-44-8	0.68	0.2	Not provided in method	Not provided in method	0.058	0.2
Heptachlor epoxide	1024-57-3	5	0.2	Not provided in method	Not provided in method	0.043	0.2
Hexachlorobenzene	118-74-1	---	0.20	Not provided in method	Not provided in method	0.048	0.20
Methoxychlor	72-43-5	13.6	0.2	Not provided in method	Not provided in method	0.084	0.2
Nonachlor(cis)	5103-73-1	---	0.20	Not provided in method	Not provided in method	0.055	0.20
Nonachlor(trans)	39765-80-5	---	0.20	Not provided in method	Not provided in method	0.046	0.20
Oxychlordane	27304-13-8	---	0.20	Not provided in method	Not provided in method	0.043	0.20
Mirex	2385-85-5	---	0.20	Not provided in method	Not provided in method	0.067	0.20
Toxaphene	8001-35-2	---	10	Not provided in method	Not provided in method	1.3	10
Toxaphene Peak 1	STL00100	---	10	Not provided in method	Not provided in method	1.3	10
Toxaphene Peak 2	STL00109	---	10	Not provided in method	Not provided in method	1.3	10
Toxaphene Peak 3	STL00220	---	10	Not provided in method	Not provided in method	1.3	10
Toxaphene Peak 4	STL00083	---	10	Not provided in method	Not provided in method	1.3	10
Toxaphene Peak 5	STL00051	---	10	Not provided in method	Not provided in method	1.3	10

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Volatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-12

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
1,3-Dichlorobenzene	541-73-1	1315	5	Not provided in method	Not provided in method	0.11	5
1,4-Dichlorobenzene	106-46-7	110	5	Not provided in method	Not provided in method	0.22	5
1,2-Dichlorobenzene	95-50-1	13	5	Not provided in method	Not provided in method	0.1	5
1,2,4-Trichlorobenzene	120-82-1	4.8	5	Not provided in method	Not provided in method	0.32	5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: AVS/SEM
Concentration Level: Low
Analytical Method/SOP Reference: L-25

Analyte	CAS Number	Study Action Level µmoles/g	Project Quantitation Limit µmoles/g	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µmoles/g	Method QLs µmoles/g	MDLs µmoles/g	QLs µmoles/g
Acid Volatile Sulfide	AVS	---	16	Not provided in method	Not provided in method	15	16
SEM - Cadmium	7440-43-9	---	0.12	Not provided in method	Not provided in method	0.019	0.12
SEM - Copper	7440-50-8	---	0.62	Not provided in method	Not provided in method	0.54	0.62
SEM - Lead	7439-52-1	---	0.25	Not provided in method	Not provided in method	0.11	0.25
SEM - Mercury	7439-97-6	---	0.0002	Not provided in method	Not provided in method	0.000066	0.0002
SEM - Nickel	7440-02-0	---	1	Not provided in method	Not provided in method	0.072	1
SEM - Zinc	7440-66-6	---	0.5	Not provided in method	Not provided in method	0.14	0.5

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Matrix: Sediment
Analytical Group: Miscellaneous
Concentration Level: Low
Analytical Method/SOP Reference: L-15, L-16, L-17, L-28, L-18, L-22, L-23, L-24, L-26, L-13, L-20, L-27

Analyte	CAS Number	Project Quantitation Limit mg/kg	Project Quantitation Limit mg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs mg/kg	Method QLs mg/kg	MDLs mg/kg	QLs mg/kg
TOC	TOC	---	1000	Not provided in method	Not provided in method	109	1000
TEPH	TEPH	---	20	10	Not provided in method	1.9	20
Hexavalent Chromium	18540-29-9	0.29	0.4	Not provided in method	Not provided in method	0.14	0.4
ORP	ORP	---	NA	Not provided in method	Not provided in method	NA	NA
pH	pH	---	NA	Not provided in method	Not provided in method	NA	NA
Total Sulfide	18496-25-8	---	30	Not provided in method	Not provided in method	6.0001	30
Total Phosphorus	14365-44-2	---	10	Not provided in method	Not provided in method	0.099	10
Total Kjedahl Nitrogen	7727-37-9	---	150	Not provided in method	Not provided in method	51	150
Ammonia	766-41-7	---	2	Not provided in method	Not provided in method	0.52	2
Cyanide	57-12-5	2.2	0.5	Not provided in method	0.5	0.029	0.5
% Moisture	Moist	---	0.5%	Not provided in method	Not provided in method	0,5%	0.5%
Grain Size	Grain	---	NA	Not provided in method	Not provided in method	NA	NA

See the last page of Worksheet #15-2 for a description of footnotes.

QAPP Worksheet #15-2 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Sediment (continued)

Notes:

[a] Analytical MDLs and QLs are those documented in validated methods.
[b] Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

AVS/SEM = acid volatile sulfide/simultaneously extracted metals
BHC = benzene hexachloride
CAS = Chemical Abstracts Service
DDD = dichlorodiphenyldichloroethane
DDE = dichlorodiphenyldichloroethylene
DDT = dichlorodiphenyltrichloroethane
HpCDD = heptachlorodibenzo-*p*-dioxin
HpCDF = heptachlorodibenzofuran
HxCDD = hexachlorodibenzo-*p*-dioxin
HxCDF = hexachlorodibenzofuran
MDL = method detection limit
NA = not applicable
ng/kg = nanograms per kilogram
OCDD = octachlorodibenzo-*p*-dioxin
OCDF = octachlorodibenzofuran
ORP = oxidation reduction potential
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
PeCDD = pentachlorodibenzo-*p*-dioxin
PeCDF = pentachlorodibenzofuran
QL = quantitation limit
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
TCDD = tetrachlorodibenzo-*p*-dioxin
TCDF = tetrachlorodibenzofuran
TEPH = total extractable petroleum hydrocarbon
TOC = total organic carbon
µg/kg = micrograms per kilogram
% = percent

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue

Matrix: Tissue
Analytical Group: PCDDs/PCDFs
Concentration Level: Low
Analytical Method/SOP Reference: L-1

Analyte	CAS Number	Study Action Level ng/kg	Project Quantitation Limit ng/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs ng/kg	Method QLs ng/kg	MDLs ng/kg	QLs ng/kg
2,3,7,8-TCDD	1746-01-6	0.024	0.20	Not provided in method	1	0.068	0.20
1,2,3,7,8-PeCDD	40321-76-4	0.024	1.00	Not provided in method	5	0.209	1.00
1,2,3,6,7,8-HxCDD	57653-85-7	0.24	1.00	Not provided in method	5	0.237	1.00
1,2,3,4,7,8-HxCDD	39227-28-6	0.24	1.00	Not provided in method	5	0.181	1.00
1,2,3,7,8,9-HxCDD	19408-74-3	0.24	1.00	Not provided in method	5	0.187	1.00
1,2,3,4,6,7,8-HpCDD	35822-46-9	2.4	1.00	Not provided in method	5	0.198	1.00
OCDD	3268-87-9	80	2.0	Not provided in method	10	0.321	2.0
2,3,7,8-TCDF	51207-31-9	0.24	0.20	Not provided in method	1	0.038	0.20
1,2,3,7,8-PeCDF	57117-41-6	0.8	1.00	Not provided in method	5	0.162	1.00
2,3,4,7,8-PeCDF	57117-31-4	0.08	1.00	Not provided in method	5	0.176	1.00
1,2,3,6,7,8-HxCDF	57117-44-9	0.24	1.00	Not provided in method	5	0.136	1.00
1,2,3,7,8,9-HxCDF	72918-21-9	0.24	1.00	Not provided in method	5	0.135	1.00
1,2,3,4,7,8-HxCDF	70648-26-9	0.24	1.00	Not provided in method	5	0.112	1.00
2,3,4,6,7,8-HxCDF	60851-34-5	0.24	1.00	Not provided in method	5	0.119	1.00
1,2,3,4,6,7,8-HpCDF	67562-39-4	2.4	1.00	Not provided in method	5	0.080	1.00
1,2,3,4,7,8,9-HpCDF	55673-89-7	2.4	1.00	Not provided in method	5	0.188	1.00
OCDF	39001-02-0	80	2.0	Not provided in method	10	0.255	2.0

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/g	Project Quantitation Limit pg/g	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/g	Method QLs pg/g	MDLs pg/g	QLs pg/g
PCB1	2051-60-7	1580	2.5	8	20	0.119	2.5
PCB2	2051-61-8	1580	2.5	0.4	1	0.160	2.5
PCB3	2051-62-9	1580	2.5	9	20	0.138	2.5
PCB4/10	13029-08-8/33146-45-1	1580	10	Not provided in method	Not provided in method	0.241	10
PCB6	25569-80-6	1580	5.0	1	5	0.092	5.0
PCB5/8	16605-91-7/34883-43-7	1580	10	Not provided in method	Not provided in method	0.134	10
PCB7/9	33284-50-3/34883-39-1	1580	10	2	5	0.168	10
PCB11	2050-67-1	1580	5.0	10	20	0.301	5.0
PCB12/13	2974-92-7/2974-90-5	1580	10	3	10	0.252	10
PCB14	34883-41-5	1580	5.0	3	10	0.095	5.0
PCB15	2050-68-2	1580	5.0	18	50	0.133	5.0
PCB16/32	38444-78-9/38444-77-8	1580	2.5	Not provided in method	Not provided in method	0.153	2.5
PCB17	37680-66-3	1580	5.0	9	20	0.111	5.0
PCB18	37680-65-2	1580	5.0	17	50	0.147	5.0
PCB19	38444-73-4	1580	5.0	4	10	0.080	5.0
PCB20/21/33	38444-84-7/ 55702-46-0/38444-86-9	1580	7.5	Not provided in method	Not provided in method	0.558	7.5
PCB22	38444-85-8	1580	2.5	9	20	0.204	2.5
PCB23	55720-44-0	1580	2.5	5	20	0.245	2.5
PCB24/27	55702-45-9/38444-76-7	1580	5.0	Not provided in method	Not provided in method	0.245	5.0
PCB25	55712-37-3	1580	2.5	5	20	0.294	2.5
PCB26	38444-81-4	1580	2.5	8	20	0.145	2.5
PCB28	7012-37-5	1580	2.5	19	50	0.161	2.5
PCB29	15862-07-4	1580	2.5	8	20	0.182	2.5

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/g	Project Quantitation Limit pg/g	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/g	Method QLs pg/g	MDLs pg/g	QLs pg/g
PCB30	35693-92-6	1580	2.5	17	50	0.123	2.5
PCB31	16606-02-3	1580	2.5	15	50	0.222	2.5
PCB34	37680-68-5	1580	2.5	7	20	0.214	2.5
PCB35	37680-69-6	1580	2.5	8	20	0.234	2.5
PCB36	38444-87-0	1580	2.5	8	20	0.246	2.5
PCB37	38444-90-5	1580	2.5	13	50	0.157	2.5
PCB38	53555-66-1	1580	2.5	8	20	0.197	2.5
PCB39	38444-88-1	1580	2.5	9	20	0.188	2.5
PCB40	38444-93-8	1580	2.5	12	50	0.119	2.5
PCB41/64/71/ 72	52663-59-9/52663-58-8/ 41464-46-4/41464-42-0	1580	10	Not provided in method	Not provided in method	0.555	10
PCB42/59	36559-22-5/74472-33-6	1580	5.0	6	20	0.235	5.0
PCB43/49	70362-46-8/41464-40-8	1580	5.0	Not provided in method	Not provided in method	0.229	5.0
PCB44	41464-39-5	1580	2.5	19	50	0.142	2.5
PCB45	70362-45-7	1580	2.5	5	20	0.137	2.5
PCB46	41464-47-5	1580	2.5	10	20	0.079	2.5
PCB47	2437-79-8	1580	2.5	19	50	0.142	2.5
PCB48/75	70362-47-9/32598-12-2	1580	5.0	Not provided in method	Not provided in method	0.197	5.0
PCB50	62796-65-0	1580	2.5	6	20	0.100	2.5
PCB51	68194-04-7	1580	2.5	5	20	0.082	2.5
PCB52/69	35693-99-3/60233-24-1	1580	5.0	Not provided in method	Not provided in method	0.197	5.0
PCB53	41464-41-9	1580	2.5	6	20	0.153	2.5
PCB54	15968-05-5	1580	2.5	12	50	0.085	2.5
PCB55	74338-24-2	1580	2.5	12	50	0.115	2.5

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/g	Project Quantitation Limit pg/g	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/g	Method QLs pg/g	MDLs pg/g	QLs pg/g
PCB56/60	41464-43-1/33025-41-1	1580	5.0	Not provided in method	Not provided in method	0.226	5.0
PCB57	70424-67-8	1580	2.5	12	50	0.057	2.5
PCB58	41464-49-7	1580	2.5	13	50	0.121	2.5
PCB61/70	33284-53-6/32598-11-1	1580	5.0	17	50	0.198	5.0
PCB62	54230-22-7	1580	2.5	6	20	0.122	2.5
PCB63	74472-34-7	1580	2.5	14	50	0.073	2.5
PCB65	33284-54-7	1580	2.5	19	50	0.154	2.5
PCB67	73575-53-8	1580	2.5	15	50	0.110	2.5
PCB68	73575-52-7	1580	2.5	15	50	0.134	2.5
PCB73	74338-23-1	1580	2.5	16	50	0.091	2.5
PCB74	32690-93-0	1580	2.5	17	50	0.087	2.5
PCB76/66	70362-48-0/32598-10-0	1580	5.0	Not provided in method	Not provided in method	0.205	5.0
PCB77	32598-13-3	240	2.5	17	50	0.071	2.5
PCB78	70362-49-1	1580	2.5	17	50	0.082	2.5
PCB79	41464-48-6	1580	2.5	17	50	0.062	2.5
PCB80	33284-52-5	1580	2.5	18	50	0.083	2.5
PCB81	70362-50-4	80	2.5	18	50	0.096	2.5
PCB82	52663-62-4	1580	2.5	13	50	0.078	2.5
PCB83	60145-20-2	1580	2.5	22	50	0.072	2.5
PCB84/92	52663-60-2/52663-61-3	1580	5.0	12	50	0.166	5.0
PCB85/116	65510-45-4/18259-05-7	1580	5.0	10	20	0.215	5.0
PCB86	55312-69-1	1580	2.5	15	50	0.106	2.5
PCB87/117/125	38380-02-8/ 68194-11-6/74472-39-2	1580	7.5	Not provided in method	Not provided in method	0.224	7.5

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/g	Project Quantitation Limit pg/g	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/g	Method QLs pg/g	MDLs pg/g	QLs pg/g
PCB88/91	55215-17-3/68194-05-8	1580	5.0	12	50	0.220	5.0
PCB89	73575-57-2	1580	2.5	19	50	0.114	2.5
PCB90/101	68194-07-0/37680-73-2	1580	5.0	24	100	0.141	5.0
PCB93	73575-56-1	1580	2.5	22	50	0.149	2.5
PCB94	73575-55-0	1580	2.5	12	50	0.109	2.5
PCB95/98/102	38379-99-6/ 60233-25-2/68194-06-9	1580	7.5	22	50	0.257	7.5
PCB96	73575-54-9	1580	2.5	21	50	0.127	2.5
PCB97	41464-51-1	1580	2.5	15	50	0.088	2.5
PCB99	38380-01-7	1580	2.5	22	50	0.141	2.5
PCB100	39485-83-1	1580	2.5	22	50	0.124	2.5
PCB103	60145-21-3	1580	2.5	23	50	0.093	2.5
PCB104	56558-16-8	1580	2.5	23	50	0.123	2.5
PCB105	32598-14-4	800	2.5	11	20	0.094	2.5
PCB106/118	70424-69-0/31508-00-6	1580	5.0	Not provided in method	Not provided in method	0.121	5.0
PCB107/109	70424-68-9/74472-35-8	1580	5.0	Not provided in method	Not provided in method	0.187	5.0
PCB108/112	70362-41-3/74472-36-9	1580	5.0	Not provided in method	Not provided in method	0.117	5.0
PCB110	38380-03-9	1580	2.5	24	100	0.089	2.5
PCB111/115	39635-32-0/74472-38-1	1580	5.0	24	100	0.130	5.0
PCB113	68194-10-5	1580	2.5	24	100	0.062	2.5
PCB114	74472-37-0	800	2.5	12	50	0.078	2.5
PCB119	56558-17-9	1580	2.5	15	50	0.069	2.5
PCB120	68194-12-7	1580	2.5	15	50	0.084	2.5
PCB121	56558-18-0	1580	2.5	21	50	0.119	2.5

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/g	Project Quantitation Limit pg/g	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/g	Method QLs pg/g	MDLs pg/g	QLs pg/g
PCB122	76842-07-4	1580	2.5	12	50	0.065	2.5
PCB123	65510-44-3	800	2.5	15	50	0.081	2.5
PCB124	70424-70-3	1580	2.5	27	100	0.079	2.5
PCB126	57465-28-8	0.24	2.5	14	50	0.112	2.5
PCB127	39635-33-1	1580	2.5	28	100	0.093	2.5
PCB128/162	38380-07-3/39635-34-2	1580	5.0	Not provided in method	Not provided in method	0.202	5.0
PCB129	55215-18-4	1580	2.5	21	50	0.094	2.5
PCB130	52663-66-8	1580	2.5	14	50	0.118	2.5
PCB131	61798-70-7	1580	2.5	12	50	0.090	2.5
PCB132/161	38380-05-1/74472-43-8	1580	5.0	Not provided in method	Not provided in method	0.178	5.0
PCB133/142	35694-04-3/41411-61-4	1580	5.0	Not provided in method	Not provided in method	0.112	5.0
PCB134/143	52704-70-8/68194-15-0	1580	5.0	13	50	0.161	5.0
PCB135	52744-13-5	1580	2.5	11	50	0.123	2.5
PCB136	38411-22-2	1580	2.5	9	20	0.135	2.5
PCB137	35694-06-5	1580	2.5	30	100	0.077	2.5
PCB138/163/164	35065-28-2/ 74472-44-9/74472-45-0	1580	7.5	Not provided in method	Not provided in method	0.242	7.5
PCB139/149	56030-56-9/38380-04-0	1580	5.0	Not provided in method	Not provided in method	0.209	5.0
PCB140	59291-64-4	1580	2.5	20	50	0.119	2.5
PCB141	52712-04-6	1580	2.5	9	20	0.090	2.5
PCB144	68194-14-9	1580	2.5	17	50	0.137	2.5
PCB145	74472-40-5	1580	2.5	32	100	0.130	2.5
PCB146/165	51908-16-8/74472-46-1	1580	5.0	Not provided in method	Not provided in method	0.163	5.0
PCB147	68194-13-8	1580	2.5	18	50	0.169	2.5

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/g	Project Quantitation Limit pg/g	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/g	Method QLs pg/g	MDLs pg/g	QLs pg/g
PCB148	74472-41-6	1580	2.5	32	100	0.105	2.5
PCB150	68194-08-1	1580	2.5	33	100	0.092	2.5
PCB151	52663-63-5	1580	2.5	11	50	0.115	2.5
PCB152	68194-09-2	1580	2.5	24	100	0.080	2.5
PCB153	35065-27-1	1580	2.5	13	50	0.107	2.5
PCB154	60145-22-4	1580	2.5	11	50	0.111	2.5
PCB155	33979-03-2	1580	2.5	34	100	0.052	2.5
PCB156	38380-08-4	800	2.5	13	50	0.074	2.5
PCB157	69782-90-7	800	2.5	13	50	0.086	2.5
PCB158/160	74472-42-7/41411-62-5	1580	5.0	Not provided in method	Not provided in method	0.198	5.0
PCB159	39635-35-3	1580	2.5	35	100	0.085	2.5
PCB166	41411-63-6	1580	2.5	12	50	0.067	2.5
PCB167	52663-72-6	800	2.5	11	50	0.089	2.5
PCB168	59291-65-5	1580	2.5	13	50	0.056	2.5
PCB169	32774-16-6	0.8	2.5	16	50	0.099	2.5
PCB170	35065-30-6	1580	2.5	16	50	0.094	2.5
PCB171	52663-71-5	1580	2.5	37	100	0.086	2.5
PCB172	52663-74-8	1580	2.5	38	100	0.076	2.5
PCB173	68194-16-1	1580	2.5	37	100	0.094	2.5
PCB174	38411-25-5	1580	2.5	19	50	0.091	2.5
PCB175	40186-70-7	1580	2.5	38	100	0.092	2.5
PCB176	52663-65-7	1580	2.5	39	100	0.116	2.5
PCB177	52663-70-4	1580	2.5	14	50	0.094	2.5
PCB178	52663-67-9	1580	2.5	22	50	0.085	2.5

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/g	Project Quantitation Limit pg/g	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/g	Method QLs pg/g	MDLs pg/g	QLs pg/g
PCB179	52663-64-6	1580	2.5	23	50	0.083	2.5
PCB180	35065-29-3	1580	2.5	Not provided in method	Not provided in method	0.072	2.5
PCB181	74472-47-2	1580	2.5	40	100	0.116	2.5
PCB182/187	60145-23-5/52663-68-0	1580	5.0	40	100	0.222	5.0
PCB183	52663-69-1	1580	2.5	40	100	0.119	2.5
PCB184	74472-48-3	1580	2.5	40	100	0.117	2.5
PCB185	52712-05-7	1580	2.5	40	100	0.111	2.5
PCB186	74472-49-4	1580	2.5	41	100	0.079	2.5
PCB188	74487-85-7	1580	2.5	23	50	0.067	2.5
PCB189	39635-31-9	1580	2.5	18	50	0.082	2.5
PCB190	41411-64-7	1580	2.5	23	50	0.076	2.5
PCB191	74472-50-7	1580	2.5	42	100	0.084	2.5
PCB192	74472-51-8	1580	2.5	42	100	0.073	2.5
PCB193	69782-91-8	1580	2.5	14	50	0.078	2.5
PCB194	35694-08-7	1580	2.5	17	50	0.084	2.5
PCB195	52663-78-2	1580	2.5	43	100	0.129	2.5
PCB196/203	42740-50-1/52663-76-0	1580	5.0	Not provided in method	Not provided in method	0.178	5.0
PCB197	33091-17-7	1580	2.5	25	100	0.108	2.5
PCB198	68194-17-2	1580	2.5	20	50	0.095	2.5
PCB199	52663-75-9	1580	2.5	20	50	0.166	2.5
PCB200	52663-73-7	1580	2.5	25	100	0.116	2.5
PCB201	40186-71-8	1580	2.5	44	100	0.130	2.5
PCB202	2136-99-4	1580	2.5	44	100	0.077	2.5
PCB204	74472-52-9	1580	2.5	45	100	0.107	2.5

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: PCB Congeners
Concentration Level: Low
Analytical Method/SOP Reference: L-2

Analyte	CAS Number	Study Action Level pg/g	Project Quantitation Limit pg/g	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs pg/g	Method QLs pg/g	MDLs pg/g	QLs pg/g
PCB205	74472-53-0	1580	2.5	45	100	0.070	2.5
PCB206	40186-72-9	1580	2.5	45	100	0.086	2.5
PCB207	52663-79-3	1580	2.5	45	100	0.129	2.5
PCB208	52663-77-1	1580	2.5	46	100	0.074	2.5
PCB209	2051-24-3	1580	2.5	15	50	0.075	2.5

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: TAL Metals, Mercury, Methylmercury, Titanium
Concentration Level: Low
Analytical Method/SOP Reference: L-3, L-4, L-5 and L-29

Analyte	CAS Number	Study Action Level mg/kg	Project Quantitation Limit mg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs mg/kg	Method QLs mg/kg	MDLs mg/kg	QLs mg/kg
Aluminum	7429-90-5	140	8	Not provided in method	Not provided in method	0.21	8
Antimony	7440-36-0	0.054	2	Not provided in method	Not provided in method	0.0082	2
Arsenic	7440-38-2	0.0021	0.2	Not provided in method	Not provided in method	0.012	0.2
Barium	7440-39-3	27	10	Not provided in method	Not provided in method	0.023	10
Beryllium	7440-41-7	0.27	0.2	Not provided in method	Not provided in method	0.0049	0.2
Cadmium	7440-43-9	0.14	0.2	Not provided in method	Not provided in method	0.0075	0.2
Calcium	7440-70-2	Essential Nutrient	100	Not provided in method	Not provided in method	1.2	100
Chromium	7440-47-3	200	0.4	Not provided in method	Not provided in method	0.014	0.4
Cobalt	7440-48-4	0.041	1	Not provided in method	Not provided in method	0.0017	1
Copper	7440-50-8	5.4	2	Not provided in method	Not provided in method	0.016	2
Iron	7439-89-6	95	20	Not provided in method	Not provided in method	0.6	20
Lead	7439-92-1	1.5	0.2	Not provided in method	Not provided in method	0.0048	0.2
Magnesium	7439-95-4	Essential Nutrient	100	Not provided in method	Not provided in method	0.34	100
Manganese	7439-96-5	19	0.4	Not provided in method	Not provided in method	0.019	0.4
Nickel	7440-02-0	2.7	2	Not provided in method	Not provided in method	0.005	2
Potassium	7440-09-7	Essential Nutrient	100	Not provided in method	Not provided in method	1.5	100
Selenium	7782-49-2	0.68	0.2	Not provided in method	Not provided in method	0.035	0.2
Silver	7440-22-4	0.68	0.2	Not provided in method	Not provided in method	0.0016	0.2
Sodium	7440-23-5	Essential Nutrient	100	Not provided in method	Not provided in method	0.63	100
Titanium	7440-32-6	---	2	Not provided in method	Not provided in method	0.076	2
Thallium	7440-28-0	0.0014	0.2	Not provided in method	Not provided in method	0.01	0.2
Vanadium	7440-62-2	0.68	0.4	Not provided in method	Not provided in method	0.023	0.4
Zinc	7440-66-6	41	2	Not provided in method	Not provided in method	0.11	2
Mercury	7439-97-6	0.0086	0.033	Not provided in method	Not provided in method	0.0091	0.033
Methylmercury	22967-92-6	0.0086	0.1 µg/kg	Not provided in method	Not provided in method	0.037 µg/kg	0.1 µg/kg

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: Butyltins
Concentration Level: Low
Analytical Method/SOP Reference: L-6

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
Dibutyltin	14488-53-0	41	1.3	Not provided in method	1.3	0.40	1.3
Monobuyltin	78763-54-9	41	5	Not provided in method	5.0	5	5
Tetrabutyltin	1461-25-2	41	1.7	Not provided in method	1.7	0.44	1.7
Tributyltin	36643-28-4	41	1.5	Not provided in method	1.5	0.67	1.5

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: Semivolatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-8

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
Benzaldehyde	100-52-7	1.35e4	660	Not provided in method	Not provided in method	16	660
Phenol	108-95-2	41	660	Not provided in method	Not provided in method	40	660
bis(2-Chloroethyl) ether	111-44-4	2.9	660	Not provided in method	Not provided in method	15	660
2-Chlorophenol	95-57-8	680	660	Not provided in method	Not provided in method	34	660
2-Methylphenol	95-48-7	6.76e3	660	Not provided in method	Not provided in method	40	660
2,2'-oxybis(1-Chloropropane)	108-60-1	---	660	Not provided in method	Not provided in method	16	660
Acetophenone	98-86-2	1.35e4	660	Not provided in method	Not provided in method	16	660
4-Methylphenol	106-44-5	1.40e4	1340	Not provided in method	Not provided in method	74	1340
N-Nitroso-di-n-propylamine	621-64-7	0.45	660	Not provided in method	Not provided in method	46	660
Hexachloroethane	67-72-1	79	660	Not provided in method	Not provided in method	34	660
Nitrobenzene	98-95-3	270	660	Not provided in method	Not provided in method	41	660
Isophorone	78-59-1	3.32e3	660	Not provided in method	Not provided in method	42	660
2-Nitrophenol	88-75-5	4.06e4	660	Not provided in method	Not provided in method	37	660
2,4-Dimethylphenol	105-67-9	2700	660	Not provided in method	Not provided in method	52	660
bis(2-Chloroethoxy) methane	111-91-1	410	660	Not provided in method	Not provided in method	15	660
2,4-Dichlorophenol	120-83-2	410	660	Not provided in method	Not provided in method	33	660
4-Chloroaniline	106-47-8	16	660	Not provided in method	Not provided in method	32	660
Hexachlorobutadiene	87-68-3	40	660	Not provided in method	Not provided in method	35	660
Caprolactam	105-60-2	6.76e4	660	Not provided in method	Not provided in method	35	660
4-Chloro-3-methyphenol	59-50-7	1.40e4	660	Not provided in method	Not provided in method	40	660
Hexachlorocyclopentadiene	77-47-4	810	660	Not provided in method	Not provided in method	65	660
2,4,6-Trichlorophenol	88-06-2	140	660	Not provided in method	Not provided in method	34	660
2,4,5-Trichlorophenol	95-95-4	1.40e4	1660	Not provided in method	Not provided in method	34	1660
1,1'-Biphenyl	92-52-4	390	660	Not provided in method	Not provided in method	15	660

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: Semivolatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-8

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
2-Chloronaphthalene	91-58-7	1.08e4	660	Not provided in method	Not provided in method	43	660
2-Nitroaniline	88-74-4	1400	1660	Not provided in method	Not provided in method	37	1660
Dimethylphthalate	131-11-3	1.10e5	660	Not provided in method	Not provided in method	15	660
2,6-Dinitrotoluene	606-20-2	140	660	Not provided in method	Not provided in method	30	660
3-Nitroaniline	99-09-2	160	1660	Not provided in method	Not provided in method	38	1660
2,4-Dinitrophenol	51-28-5	270	1660	Not provided in method	Not provided in method	140	1660
4-Nitrophenol	100-02-7	4.06e4	1660	Not provided in method	Not provided in method	84	1660
Dibenzofuran	132-64-9	140	660	Not provided in method	Not provided in method	16	660
2,4-Dinitrotoluene	121-14-2	10	660	Not provided in method	Not provided in method	24	660
Diethylphthalate	84-66-2	1.10e5	660	Not provided in method	Not provided in method	15	660
4-Chlorophenyl-phenylether	7005-72-3	---	660	Not provided in method	Not provided in method	15	660
4-Nitroaniline	100-01-6	160	1660	Not provided in method	Not provided in method	32	1660
4,6-Dinitro-2-methylphenol	534-52-1	11	1660	Not provided in method	Not provided in method	97	1660
N-Nitrosodiphenylamine	86-30-6	640	774	Not provided in method	Not provided in method	16	774
1,2,4,5-Tetrachlorobenzene	95-94-3	41	660	Not provided in method	Not provided in method	40	660
4-Bromophenyl-phenylether	101-55-3	---	660	Not provided in method	Not provided in method	18	660
Atrazine	1912-24-9	13.7	660	Not provided in method	Not provided in method	16	660
Carbazole	86-74-8	---	660	Not provided in method	Not provided in method	15	660
Di-n-butylphthalate	84-74-2	1.40e4	660	Not provided in method	Not provided in method	15	660
Butylbenzylphthalate	85-68-7	1.7	660	Not provided in method	Not provided in method	20	660
3,3'-Dichlorobenzidine	91-94-1	7	660	Not provided in method	Not provided in method	48	660
bis-(2-Ethylhexyl)phthalate	117-81-7	230	660	Not provided in method	Not provided in method	21	660
Di-n-octylphthalate	117-84-0	1600	660	Not provided in method	Not provided in method	23	660
2,3,4,6-Tetrachlorophenol	58-90-2	---	660	Not provided in method	Not provided in method	37	660

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: Semivolatile Organics
Concentration Level: Low
Analytical Method/SOP Reference: L-8

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
Azobenzene	103-33-3	29	660	Not provided in method	Not provided in method	19	660
Benzidine	92-87-5	0.014	1660	Not provided in method	Not provided in method	97	1660
Benzoic Acid	65-85-0	5.40e5	1660	Not provided in method	Not provided in method	56	1660
Pentachlorophenol	87-86-5	7.9	1660	Not provided in method	Not provided in method	68	1660
Pyridine	110-86-1	---	660	Not provided in method	Not provided in method	36	660

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: Semivolatile Organic SIM
Concentration Level: Low
Analytical Method/SOP Reference: L-9

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
2-Methylnaphthalene	91-57-6	540	1.34	Not provided in method	Not provided in method	0.055	1.34
Acenaphthylene	208-96-8	4.10e3	1.34	Not provided in method	Not provided in method	0.053	1.34
Acenaphthene	83-32-9	8.10e3	1.34	Not provided in method	Not provided in method	0.053	1.34
Anthracene	120-12-7	4.10e4	1.34	Not provided in method	Not provided in method	0.053	1.34
Benzo[a]anthracene	56-55-3	4.5	1.34	Not provided in method	Not provided in method	0.048	1.34
Benzo[b]fluoranthene	205-99-2	4.3	1.34	Not provided in method	Not provided in method	0.047	1.34
Benzo[k]fluoranthene	207-08-9	43	1.34	Not provided in method	Not provided in method	0.057	1.34
Benzo[a]pyrene	50-32-8	0.43	1.34	Not provided in method	Not provided in method	0.047	1.34
Benzo[e]pyrene	192-97-2	4.10e3	1.34	Not provided in method	Not provided in method	0.067	1.34
Indeno[1,2,3-c,d]-pyrene	193-39-5	4.3	1.34	Not provided in method	Not provided in method	0.077	1.34
Dibenzo[a,h]anthracene	53-70-3	0.43	1.34	Not provided in method	Not provided in method	0.057	1.34
Naphthalene	91-20-3	2.7e3	1.34	Not provided in method	Not provided in method	0.67	1.34
C1-Naphthalenes	STL00916	4.10e3	1.34	Not provided in method	Not provided in method	0.67	1.34
C2-Naphthalenes	STL00917	---	1.34	Not provided in method	Not provided in method	0.67	1.34
C3-Naphthalenes	STL00918	---	1.34	Not provided in method	Not provided in method	0.67	1.34
C4-Naphthalenes	STL00919	---	1.34	Not provided in method	Not provided in method	0.67	1.34
1-Methylnaphthalene	90-12-0	---	1.34	Not provided in method	Not provided in method	0.055	1.34
Fluorene	86-73-7	5.40e3	1.34	Not provided in method	Not provided in method	0.055	1.34
C1-Fluorenes	STL00913	---	1.34	Not provided in method	Not provided in method	0.67	1.34
C2-Fluorenes	STL00914	---	1.34	Not provided in method	Not provided in method	0.67	1.34
C3-Fluorenes	STL00915	---	1.34	Not provided in method	Not provided in method	0.67	1.34
Phenanthrene	85-01-8	4.10e3	1.34	Not provided in method	Not provided in method	0.07	1.34
C1-Phenanthrenes/Anthracenes	STL00901	4.10e3	1.34	Not provided in method	Not provided in method	0.67	1.34
C2-Phenanthrenes/Anthracenes	STL00902	4.10e3	1.34	Not provided in method	Not provided in method	0.67	1.34
C3-Phenanthrenes/Anthracenes	STL00903	---	1.34	Not provided in method	Not provided in method	0.67	1.34
C4-Phenanthrenes/Anthracenes	STL00904	---	1.34	Not provided in method	Not provided in method	0.67	1.34

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: Semivolatile Organics SIM
Concentration Level: Low
Analytical Method/SOP Reference: L-9

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
Fluoranthene	206-44-0	5.4e3	1.34	Not provided in method	Not provided in method	0.061	1.34
Pyrene	129-00-0	4.10e3	1.34	Not provided in method	Not provided in method	0.037	1.34
C1-Fluoranthenes/pyrene	STL00912	---	1.34	Not provided in method	Not provided in method	0.67	1.34
C2-Fluoranthenes/Pyrene	STL00968	---	1.34	Not provided in method	Not provided in method	0.67	1.34
C3-Fluoranthenes/Pyrene	STL00969	---	1.34	Not provided in method	Not provided in method	0.67	1.34
Chrysene	218-01-9	430	1.34	Not provided in method	Not provided in method	0.057	1.34
C1-Chrysenes	STL00905	---	1.34	Not provided in method	Not provided in method	0.67	1.34
C2-Chrysenes	STL00906	---	1.34	Not provided in method	Not provided in method	0.67	1.34
C3-Chrysenes	STL00907	---	1.34	Not provided in method	Not provided in method	0.67	1.34
C4-Chrysenes	STL00908	---	1.34	Not provided in method	Not provided in method	0.67	1.34
Perylene	198-55-0	4.10e3	1.34	Not provided in method	Not provided in method	0.046	1.34
Benzo[g,h,i]perylene	191-24-2	4.1e3	1.34	Not provided in method	Not provided in method	0.091	1.34

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: Aroclor PCBs
Concentration Level: Low
Analytical Method/SOP Reference: L-10

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
Aroclor-1016	12674-11-2	9.5	17	Not provided in method	Not provided in method	5.6	17
Aroclor-1221	11104-28-2	1.6	17	Not provided in method	Not provided in method	4.3	17
Aroclor-1232	11141-16-5	1.6	17	Not provided in method	Not provided in method	3.3	17
Aroclor-1242	53469-21-9	1.6	17	Not provided in method	Not provided in method	6.7	17
Aroclor-1248	12672-29-6	1.6	17	Not provided in method	Not provided in method	2	17
Aroclor-1254	11097-69-1	1.6	17	Not provided in method	Not provided in method	2.8	17
Aroclor-1260	11096-82-5	1.6	17	Not provided in method	Not provided in method	2.4	17
Aroclor-1262	37324-23-5	---	17	Not provided in method	Not provided in method	1.5	17
Aroclor-1268	11100-14-4	---	17	Not provided in method	Not provided in method	1.4	17

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: Pesticides
Concentration Level: Low
Analytical Method/SOP Reference: L-11

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
2,4'-DDD	53-19-0	13	0.2	Not provided in method	Not provided in method	0.053	0.2
2,4'-DDE	3424-82-6	9.3	0.2	Not provided in method	Not provided in method	0.057	0.2
2,4'-DDT	789-02-6	9.3	0.2	Not provided in method	Not provided in method	0.067	0.2
4,4'-DDD	72-54-8	13	0.2	Not provided in method	Not provided in method	0.039	0.2
4,4'-DDE	72-55-9	9.3	0.2	Not provided in method	Not provided in method	0.041	0.2
4,4'-DDT	50-29-3	9.3	0.2	Not provided in method	Not provided in method	0.057	0.2
Aldrin	309-00-2	0.19	0.2	Not provided in method	Not provided in method	0.046	0.2
alpha-BHC	319-84-6	0.50	0.2	Not provided in method	Not provided in method	0.043	0.2
alpha-Chlordane	5103-71-9	9.0	0.2	Not provided in method	Not provided in method	0.053	0.2
beta-BHC	319-85-7	1.8	0.2	Not provided in method	Not provided in method	0.11	0.2
delta-BHC	319-86-8	1.8	0.2	Not provided in method	Not provided in method	0.043	0.2
Dieldrin	60-57-1	0.20	0.2	Not provided in method	Not provided in method	0.048	0.2
Endosulfan I	959-98-8	810	0.2	Not provided in method	Not provided in method	0.044	0.2
Endosulfan II	33213-65-9	810	0.2	Not provided in method	Not provided in method	0.046	0.2
Endosulfan sulfate	1031-07-8	810	0.2	Not provided in method	Not provided in method	0.069	0.2
Endrin	72-20-8	41	0.2	Not provided in method	Not provided in method	0.048	0.2
Endrin aldehyde	7421-93-4	41	0.2	Not provided in method	Not provided in method	0.046	0.2
Endrin ketone	53494-70-5	41	0.2	Not provided in method	Not provided in method	0.15	0.2

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: Pesticides
Concentration Level: Low
Analytical Method/SOP Reference: L-11

Analyte	CAS Number	Study Action Level µg/kg	Project Quantitation Limit µg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs µg/kg	Method QLs µg/kg	MDLs µg/kg	QLs µg/kg
gamma-BHC	58-89-9	2.9	1.7	Not provided in method	Not provided in method	0.31	1.7
gamma-Chlordane	5103-74-2	9.0	1.7	Not provided in method	Not provided in method	0.28	1.7
Heptachlor	76-44-8	0.70	1.7	Not provided in method	Not provided in method	0.34	1.7
Heptachlor epoxide B (Cis)	1024-57-3	0.35	1.7	Not provided in method	Not provided in method	0.28	1.7
Hexachlorobenzene	118-74-1	2.0	NA	Not provided in method	Not provided in method	NA	NA
Methoxychlor	72-43-5	680	1.7	Not provided in method	Not provided in method	2.7	1.7
Nonachlor(cis)	5103-73-1	---	NA	Not provided in method	Not provided in method	NA	NA
Nonachlor(trans)	39765-80-5	---	NA	Not provided in method	Not provided in method	NA	NA
Oxychlordane	27304-13-8	9.0	NA	Not provided in method	Not provided in method	NA	NA
Mirex	2385-85-5	0.18	NA	Not provided in method	Not provided in method	NA	NA
Toxaphene	8001-35-2	2.9	170	Not provided in method	Not provided in method	28	170
Toxaphene Peak 1	STL00100	---	10	Not provided in method	Not provided in method	1.3	10
Toxaphene Peak 2	STL00109	---	10	Not provided in method	Not provided in method	1.3	10
Toxaphene Peak 3	STL00220	---	10	Not provided in method	Not provided in method	1.3	10
Toxaphene Peak 4	STL00083	---	10	Not provided in method	Not provided in method	1.3	10
Toxaphene Peak 5	STL00051	--	10	Not provided in method	Not provided in method	1.3	10

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Matrix: Tissue
Analytical Group: Miscellaneous
Concentration Level: Low
Analytical Method/SOP Reference: L-1, L-21

Analyte	CAS Number	Study Action Level mg/kg	Project Quantitation Limit mg/kg	Analytical Method[a]		Achievable Laboratory Limits[b]	
				MDLs mg/kg	Method QLs mg/kg	MDLs mg/kg	QLs mg/kg
% Lipids	% Lipids	---	NA	NA	NA	NA	NA
% Moisture	% Moist	---	NA	NA	NA	NA	NA

See the last page of Worksheet #15-3 for a description of footnotes.

QAPP Worksheet #15-3 (UFP-QAPP Manual Section 2.8.1): Reference Limits and Evaluation – Tissue (continued)

Notes:

[a] Analytical MDLs and QLs are those documented in validated methods.
[b] Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

BHC = benzene hexachloride
CAS = Chemical Abstracts Service
DDD = dichlorodiphenyldichloroethane
DDE = dichlorodiphenyldichloroethylene
DDT = Dichlorodiphenyltrichloroethane
HpCDD = heptachlorodibenzo-*p*-dioxin
HpCDF = heptachlorodibenzofuran
HxCDD = hexachlorodibenzo-*p*-dioxin
HxCDF = hexachlorodibenzofuran
MDL = method detection limit
mg/kg = milligrams per kilogram
NA = not applicable
ng/kg = nanograms per kilogram
OCDD = octachlorodibenzo-*p*-dioxin
OCDF = octachlorodibenzofuran
PCBs= polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
PeCDD = pentachlorodibenzo-*p*-dioxin
PeCDF = pentachlorodibenzofuran
QL = quantitation limit
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
TCDD = tetrachlorodibenzo-*p*-dioxin
TCDF = tetrachlorodibenzofuran
µg/kg = micrograms per kilogram
% = percent

QAPP Worksheet #16 (UFP-QAPP Manual Section 2.8.2): Project Schedule/Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Preparation of Reconnaissance Survey Work Plan	ARCADIS	June 20, 2013	July 2013	Reconnaissance Survey Work Plan	September 2013
Reconnaissance Survey	ARCADIS	September 11, 2013	September 15, 2013	Reconnaissance Survey Report	TBD
Preparation of Crab and Clam Sampling and Analysis QAPP	ARCADIS	July 15, 2013	October 2013	NBSA Crab/Clam QAPP	TBD
Field Readiness Review	TBD	2 weeks prior to field work	2 weeks prior to field work	Participant sign-off sheet	TBD
Laboratory Readiness Review	EDS	2 weeks prior to field work	2 weeks prior to field work	Participant sign-off sheet	TBD
Crab and Clam Sampling	TBD	October 2013	1 week after initiation	All samples submitted to laboratories	TBD
Laboratory Analysis	TestAmerica, Vista, eurofins/Lancaster Laboratories	During start of field work	4 weeks after completion of field work	Data Packages	TBD
Data Validation	EDS	Upon receipt of laboratory data	4 weeks after receipt of laboratory data	Data Verification/Validation Reports	TBD
Data Usability Assessment	EDS	Upon completion of data validation	4 weeks after completion of data validation	Data Quality and Usability Assessment Report	TBD
Preparation of Tissue and Sediment Data Reports	ARCADIS	Upon completion and receipt of all Phase III validated data	4 months after completion and receipt of validated data	Phase III Data Report(s)	TBD

QAPP Worksheet #17 (UFP-QAPP Manual Section 3.1.1): Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):

The sampling design is based on the number and accessibility of the Intertidal Areas and associated shallow Subtidal Flats, which will be determined during the Reconnaissance Survey. The Intertidal Areas have sparse aquatic vegetation (e.g., *Spartina* sp.) and may provide adequate habitat for shellfish and crustaceans. While eight Intertidal Areas have previously been identified in the NBSA, the feasibility of accessing and sampling these areas needs to be confirmed during the Reconnaissance Survey.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will and at what concentration levels, the sampling locations the number of samples to be collected, and the sampling frequency [Refer to Figure 1 and Worksheet No. 18 for details]:

Samples will be collected by locating the Intertidal Area for sampling, through maps, and GPS coordinates (Figure 1). Upon reaching each area identified for sampling, locational information will be recorded on the field datasheet; a GPS will be used to record the location; general observations about the Intertidal Area will be documented, including presence/absence of vegetation, general substrate appearance, and texture; tidal stage; and time on station will be recorded. At least three baited crab traps will be deployed so that they are completely submerged underwater, equally spaced in the shallow Subtidal Flats surrounding the Intertidal Area. The time of release and GPS coordinates will be recorded.

Each Intertidal Area will be explored for the presence of clam holes. A clam rake or shovel will be used to locate buried clams. Holes will be dug until the required number of clams is collected. Clams will be identified, measured, and weighed. Once the minimum required weight (232 g) of a single species is met (at least 20 clams), clams will be composited and placed into appropriate-labeled sampling containers and stored on wet ice for shipment to the analytical laboratory. Clams not meeting the required length (1.5 inches) will be left at the site.

Following clam collection, a grab sample will be collected from the top 6 inches of sediment next to the first location of the retained clam. This sediment will be used for, but not be limited to, analyses of VOC, AVS/SEM, sulfide, and cyanide. Sediment will be placed in the appropriate sample containers with no headspace, preservative (as required), and sealed per the SOP. Additional sediment will be collected from the same area as clams were collected, homogenized, placed in the appropriate-labeled sample containers, and stored on wet ice for shipment to the analytical laboratory for the remaining chemical analyses. The specific methodology and sampling equipment used will be determined after experimental sampling during the Reconnaissance Survey.

Crab traps will be checked at least every 12 hours. Hard-shelled blue crabs meeting the required size limit (4.5 inches) will be inspected for gross anomalies. Whole, intact crabs retained for analysis will be measured, weighed, documented, euthanized, placed into the appropriate-labeled sampling container, and stored on wet ice for shipment to the analytical laboratory. It is expected that at least 25 crabs will be required per sample to obtain enough tissue mass for analysis.

Details regarding the collection of tissue and sediment samples can be found in SOP Nos. 6, 7, and 9.

QAPP Worksheet #18-1 (UFP-QAPP Manual Section 3.1.1): Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ Identification Number	Matrix	Depth (units)	Method	Number of Samples	Sampling SOP Reference	Rationale for Sampling Location
Station 122	Tissue	NA	Clam fork/shovel Crab trap	1 clam whole-body 1 crab muscle 1 crab hepatopancreas	6, 7	Spatial coverage
Station 123	Tissue	NA	Clam fork/shovel Crab trap	1 clam whole-body 1 crab muscle 1 crab hepatopancreas	6, 7	Spatial coverage
Station 124	Tissue	NA	Clam fork/shovel Crab trap	1 clam whole-body 1 crab muscle 1 crab hepatopancreas	6, 7	Spatial coverage
Station 125	Tissue	NA	Clam fork/shovel Crab trap	1 clam whole-body 1 crab muscle 1 crab hepatopancreas	6, 7	Spatial coverage
Station 126	Tissue	NA	Clam fork/shovel Crab trap	1 clam whole-body 1 crab muscle 1 crab hepatopancreas	6, 7	Spatial coverage
Station 127	Tissue	NA	Clam fork/shovel Crab trap	1 clam whole-body 1 crab muscle 1 crab hepatopancreas	6, 7	Spatial coverage
Station 128	Tissue	NA	Clam fork/shovel Crab trap	1 clam whole-body 1 crab muscle 1 crab hepatopancreas	6, 7	Spatial coverage
Station 129	Tissue	NA	Clam fork/shovel Crab trap	1 clam whole-body 1 crab muscle 1 crab hepatopancreas	6, 7	Spatial coverage

Notes:
NA = not applicable

QAPP Worksheet #18-2 (UFP-QAPP Manual Section 3.1.1): Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ Identification Number	Matrix	Depth (units)	Method	Number of Samples	Sampling SOP Reference	Rationale for Sampling Location
Station 122	Sediment	0 to 0.5 (feet)	Hand core/auger	1 Surface Sediment Sample	9	Spatial coverage
Station 123	Sediment	0 to 0.5 (feet)	Hand core/auger	1 Surface Sediment Sample	9	Spatial coverage
Station 124	Sediment	0 to 0.5 (feet)	Hand core/auger	1 Surface Sediment Sample	9	Spatial coverage
Station 125	Sediment	0 to 0.5 (feet)	Hand core/auger	1 Surface Sediment Sample	9	Spatial coverage
Station 126	Sediment	0 to 0.5 (feet)	Hand core/auger	1 Surface Sediment Sample	9	Spatial coverage
Station 127	Sediment	0 to 0.5 (feet)	Hand core/auger	1 Surface Sediment Sample	9	Spatial coverage
Station 128	Sediment	0 to 0.5 (feet)	Hand core/auger	1 Surface Sediment Sample	9	Spatial coverage
Station 129	Sediment	0 to 0.5 (feet)	Hand core/auger	1 Surface Sediment Sample	9	Spatial coverage

QAPP Worksheet #19-1 (UFP-QAPP Manual Section 3.1.2): Analytical SOP Requirements – Water

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[a]	Sample Volume (mL)[b]	Containers (number and type)[c]	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)[d,e]
Water	PCDDs/PCDFs	Low	L-1	1 L	1 L, G	4°C	30 days to extraction, 45 days until analysis
Water	PCB Congeners	Low	L-2	1 L	1 L, G	4°C	30 days to extraction, 40 days until analysis
Water	TAL Metals, Titanium	Low	L-3, L-29	500	500 mL, P	4°C, HNO_3 to pH <2	6 months
Water	Mercury	Low	L-4	500	500 mL, P	4°C, HNO_3 to pH <2	28 days
Water	Methylmercury[f]	Low	L-5	250	250 mL, fluoropolymer	4°C, HCl to pH <2	48 hours to preservation, 28 days until analysis
Water	Chlorinated Herbicides	Low	L-7	1 L	1 L, G	4°C	7 days to extraction, 40 days until analysis
Water	Semivolatile Organics	Low	L-8	1 L	1 L, G	4°C	7 days to extraction, 40 days until analysis
Water	Semivolatile Organics SIM	Low	L-9	1 L	1 L, G	4°C	7 days to extraction, 40 days until analysis
Water	Aroclor PCBs	Low	L-10	1 L	1 L, G	4°C	7 days to extraction, 40 days until analysis
Water	Pesticides	Low	L-11	1 L	1 L, G	4°C	7 days to extraction, 40 days until analysis
Water	Volatile Organics	Low	L-12	3-40	40 mL vial, G no headspace	4°C, HCl to pH <2	14 days
Water	Cyanide	Low	L-13	250	500 mL, P	4°C, NaOH to pH >12	14 days
Water	TOC	Low	L-14	250	250 mL, P	4°C, H_2SO_4 to pH <2	14 days
Water	TEPH	Low	L-16	1 L	1 L, G	4°C, HCl to pH <2	14 days to extraction, 40 days until analysis
Water	Hexavalent Chromium	Low	L-17	70	250 mL, P	4°C	24 hours
Water	pH	Low	L-19	100	200 mL, P	4°C	24 hours
Water	Total Phosphorus	Low	L-23	500	500 mL, P	4°C, H_2SO_4 to pH <2	7 days
Water	Total Kjedahl Nitrogen	Low	L-24	500	500 mL, P	4°C, H_2SO_4 to pH <2	24 hours
Water	Ammonia	Low	L-26	200	500 mL, P	4°C, H_2SO_4 to pH <2	28 days

See the last page of Worksheet #19-1 for a description of footnotes.

QAPP Worksheet #19-1 (UFP-QAPP Manual Section 3.1.2): Analytical SOP Requirements – Water[a] (continued)

Notes:

[a] Analytical methods are as specified in Worksheet #23-1.
[b] These are minimum sample volume requirements for a single sample analysis. Extra volume will be needed in order to fulfill quality control sample requirements, such as matrix spike/matrix spike duplicate and/or to provide contingency volume for analysis or breakage.
[c] Samples for analyses having identical container and preservation requirements may be combined in the same container. Similarly, smaller- or larger-sized sample containers than those recommended here may be used as long as the quality, container material, and preservative specifications are met, and the containers or container used will hold sufficient mass/volume to meet the minimum requirements specified.
[d] Holding time is calculated from the date and time of sample collection, to the date and time of sample analysis (or extraction as noted).
[e] Maximum holding times listed are based upon those stipulated in corresponding data validation guidance located in Appendix C of this Crab and Clam Sampling and Analysis QAPP.
[f] Samples will be shipped at 4°C to the laboratory via overnight carrier on the same day they are collected. Preservation will occur at the laboratory within the specified holding time (immediately upon receipt) in order to minimize the sources of potential field contamination. Samples will be stored for a minimum of 48 hours at 4°C after preservation and prior to analysis to allow the acid to completely dissolve the metals adsorbed on the container walls.

°C = degrees Celsius
G = amber glass
H_2SO_4 = sulfuric acid
HCl = hydrochloric acid
HNO_3 = nitric acid
L = liter
mL = milliliters
NaOH = sodium hydroxide
P = plastic
PCB = polychlorinated biphenyl
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
SIM = selection ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
TEPH = total extractable petroleum hydrocarbons
TOC = total organic carbon

QAPP Worksheet #19-2 (UFP-QAPP Manual Section 3.1.2): Analytical SOP Requirements – Sediment

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[a]	Sample Mass (g)[b]	Containers (number and type)[c]	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)[d,e]
Sediment	PCDDs/PCDFs[f]	Low	L-1	30	8.8 oz, G	4°C	30 days to extraction, 40 days until analysis
Sediment	PCB Congeners[f]	Low	L-2	30	8.8 oz, G	4°C	30 days to extraction, 40 days until analysis
Sediment	TAL Metals, Titanium	Low	L-3, L-29				6 months
Sediment	Mercury	Low	L-4	30	8.8 oz, G	4°C	28 days
Sediment	Methylmercury	Low	L-5	3	8.8 oz, G	4°C	28 days
Sediment	Butyltins	Low	L-6	30	8.8 oz, G	4°C	7 days to extraction, 40 days to analysis
Sediment	Chlorinated Herbicides	Low	L-7	50			7 days to extraction, 40 days to analysis
Sediment	Semivolatile Organics	Low	L-8	30			7 days to extraction, 40 days to analysis
Sediment	Semivolatile Organics SIM	Low	L-9	30			7 days to extraction, 40 days to analysis
Sediment	Aroclor PCBs	Low	L-10	15			7 days to extraction, 40 days to analysis
Sediment	Pesticides	Low	L-11	15	2 (8.8) oz, G	4°C	7 days to extraction, 40 days to analysis
Sediment	Volatile Organics[g]	Low	L-12	15	3x (5 g) Encore™ Sampler, or equivalent, G	4°C	48 hours to extraction, 12 days until analysis
Sediment	Cyanide	Low	L-13	10	4 oz, G no headspace	4°C	14 days
Sediment	TOC	Low	L-15	10			14 days
Sediment	TEPH	Low	L-16	30	8.8 oz, G	4°C	14 days to extraction, 40 days to analysis
Sediment	Hexavalent Chromium	Low	L-17	5	8.8 oz, G	4°C	24 hours
Sediment	pH	Low	L-18	25	8.8 oz, G	4°C	24 hours
Sediment	% Moisture	Low	L-20	10	4 oz, G	4°C	28 days
Sediment	Total Sulfide	Low	L-22	15	4 oz, G no headspace	4°C	7 days
Sediment	Total Phosphorus	Low	L-23	20	8.8 oz, G	4°C	7 days
Sediment	Total Kjedahl Nitrogen	Low	L-24	20	4 oz, G	4°C	24 hours
Sediment	AVS/SEM	Low	L-25	60	2 oz, G no headspace	4°C	14 days
Sediment	Ammonia	Low	L-26	20	4 oz, G	4°C	28 days
Sediment	Grain Size	Low	L-27	250	16 oz, G	4°C	28 days
Sediment	ORP	Low	L-28	100	4 oz, G	4°C	24 hours

See the last page of Worksheet #19-2 for a description of footnotes.

QAPP Worksheet #19-2 (UFP-QAPP Manual Section 3.1.2): Analytical SOP Requirements – Sediment (continued)

Notes:

[a] Analytical methods are as specified in Worksheet #23.
[b] These are minimum sample mass requirements for a single sample analysis. Extra mass/volume will be needed in order to fulfill quality control sample requirements, such as matrix spike/matrix spike duplicate and/or to provide contingency mass/volume for re-analysis or breakage.
[c] Samples for analyses having identical container and preservation requirements may be combined in the same container. Similarly, smaller- or larger-sized sample containers than those recommended here may be used as long as the quality, container material, and preservative specifications are met, and the containers or container used will hold sufficient mass/volume to meet the minimum requirements specified.
[d] Holding time is calculated from the date and time of sample collection, to the date and time of sample analysis (or extraction as noted).
[e] Maximum holding times listed are based upon those stipulated in corresponding data validation guidance located in Appendix C of this Crab and Clam Sampling and Analysis QAPP.
[f] PCDDs/PCDFs and PCB congeners can both be combined in the same 8.8-oz container.
[g] A separate Percent Moisture sample must be collected with the VOCs.

°C = degrees Celsius
AVS/SEM = acid volatile sulfide/simultaneously extracted metals
G = amber glass
g = gram
ORP = oxidation reduction potential
oz = ounce
P = plastic
PCB = polychlorinated biphenyl
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
TEPH = total extractable petroleum hydrocarbons
TOC = total organic carbon
USEPA = U.S. Environmental Protection Agency
VOC = volatile organic compound
% = percent

QAPP Worksheet #19-3 (UFP-QAPP Manual Section 3.1.2): Analytical SOP Requirements – Clam & Crab Sample Collection

Crab Sample Separation	Matrix	Analytical Group	Clam & Crab Sample Separation SOP Reference[a]	Number of Shellfish[b]	Containers (number and type)[c]	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)[d]
TestAmerica Burlington, VT	Clam Sample	PCDDs/PCDFs, % Lipids, PCB Congeners, TAL Metal, Titanium, Mercury, Methylmercury, Butyltins, Semivolatile Organics, Semivolatile Organic SIMs, Aroclor PCBs, Pesticides, % Moisture	SOP-8	25	Ziploc®	shipped on wet ice frozen (-15□) upon receipt at laboratory	14 Days
	Crab Sample	PCDDs/PCDFs, % Lipids, PCB Congeners, TAL Metal, Titanium, Mercury, Methylmercury, Butyltins, Semivolatile Organics, Semivolatile Organic SIMs, Aroclor PCBs, Pesticides, % Moisture	SOP-8	20	Ziploc®	shipped on wet ice frozen (-15□) upon receipt at laboratory	14 Days

Notes:

[a] Referenced from the SOP references on Worksheet #21.
[b] Number of clams or crab bodies needed to achieve designated tissue weight for all tissue chemical analyisis. See Worksheet #10 outlining details for prioritization of chemical analysis if post homogenization weight isn't obtained. Extra clams or crab bodies will be needed in order to fulfill quality control sample requirements, such as matrix spike/matrix spike duplicate and/or to provide contingency mass/volume for re-analysis or breakage.
[c] Containers for clams or crab sample collection. Smaller or larger sized coolers may be used as long as the quality, container material will hold sufficient mass to meet the minium requirements specified. Containers for chemical analyses are in Worksheets #19-5 and 19-6.
[d] Holding time is calculated from the date and time of the crab sample collection, to the date and time of the tissue sample separation.

°C = degrees Celsius
g = gram
PCB = polychlorinated biphenyl
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/dibenzofurans
SIM = selective ion monitoring
SOP = standard operating procedure
TAL = Target Analyte List
% = percent

QAPP Worksheet #19-4 (UFP-QAPP Manual Section 3.1.2): Analytical SOP Requirements – Tissue – Clam

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[a]	Sample Mass (g)[b]	Containers (number and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)[c,d]
Tissue	PCDDs/PCDFs	Low	L-1	50	8.8 oz, G	-15°C	30 days to extraction, 40 days to analysis
Tissue	% Lipids	Low	L-1	NA	8.8 oz, G	-15°C	NA
Tissue	PCB Congeners	Low	L-2	50	8.8 oz, G	-15°C	30 days to extraction, 40 days to analysis
Tissue	TAL Metal, Titanium	Low	L-3, L-29	4		-15°C	6 months
Tissue	Mercury	Low	L-4	2	8.8 oz, G	-15°C	28 days
Tissue	Methylmercury	Low	L-5	1	8.8 oz, G	-15°C	28 days
Tissue	Butyltins	Low	L-6	30	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	Semivolatile Organics	Low	L-8	15	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	Semivolatile Organic SIM	Low	L-9	15	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	Aroclor PCBs	Low	L-10	15	8.8 oz, G	-15°C	14 days
Tissue	Pesticides	Low	L-11	15	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	% Moisture	Low	L-21	10	8.8 oz, G	-15°C	28 days

Notes:

[a] Analytical methods are as specified in Worksheet #23.
[b] These are minimum sample mass requirements for a single sample analysis post homogenization. Extra mass will be needed in order to fulfill quality control sample requirements, such as matrix spike/matrix spike duplicate and/or to provide contingency mass for re-analysis or breakage.
[c] Holding time is calculated from the date and time of samplehomogenization, to the date and time of sample analysis (or extraction as noted).
[d] Maximum holding times listed are based upon those stipulated in corresponding data validation guidance located in Appendix C of this Crab and Clam Sampling and Analysis QAPP.

°C = degrees Celsius
G = amber glass
g = gram
NA = not applicable
oz = ounce
PCB = polychlorinated biphenyl
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
% = percent

QAPP Worksheet #19-5 (UFP-QAPP Manual Section 3.1.2): Analytical SOP Requirements – Tissue – Crab–Heptapancreas

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[a]	Sample Mass (g)[b]	Containers (number and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)[c,d]
Tissue	PCDDs/PCDFs	Low	L-1	50	8.8 oz, G	-15°C	30 days to extraction, 40 days to analysis
Tissue	% Lipids	Low	L-1	NA	8.8 oz, G	-15°C	NA
Tissue	PCB Congeners	Low	L-2	50	8.8 oz, G	-15°C	30 days to extraction, 40 days to analysis
Tissue	TAL Metal, Titanium	Low	L-3, L-29	4		-15°C	6 months
Tissue	Mercury	Low	L-4	2	8.8 oz, G	-15°C	28 days
Tissue	Methylmercury	Low	L-5	1	8.8 oz, G	-15°C	28 days
Tissue	Butyltins	Low	L-6	30	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	Semivolatile Organics	Low	L-8	15	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	Semivolatile Organic SIM	Low	L-9	15	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	Aroclor PCBs	Low	L-10	15	8.8 oz, G	-15°C	14 days
Tissue	Pesticides	Low	L-11	15	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	% Moisture	Low	L-21	10	8.8 oz, G	-15°C	28 days

Notes:

[a] Analytical methods are as specified in Worksheet #23.
[b] These are minimum sample mass requirements for a single sample analysis post homogenization. Extra mass will be needed in order to fulfill quality control sample requirements, such as matrix spike/matrix spike duplicate and/or to provide contingency mass for re-analysis or breakage.
[c] Holding time is calculated from the date and time of sample homogenization, to the date and time of sample analysis (or extraction as noted).
[d] Maximum holding times listed are based upon those stipulated in corresponding data validation guidance located in Appendix C of this Crab and Clam Sampling and Analysis QAPP.

°C = degrees Celsius
G = amber glass
g = gram
NA = not applicable
oz = ounce
PCB = polychlorinated biphenyl
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
% = percent

QAPP Worksheet #19-6 (UFP-QAPP Manual Section 3.1.2): Analytical SOP Requirements – Tissue – Crab–Muscle

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[a]	Sample Mass (g)[b]	Containers (number and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)[c,d]
Tissue	PCDDs/PCDFs	Low	L-1	50	8.8 oz, G	-15°C	30 days to extraction, 40 days to analysis
Tissue	% Lipids	Low	L-1	NA	8.8 oz, G	-15°C	NA
Tissue	PCB Congeners	Low	L-2	50	8.8 oz, G	-15°C	30 days to extraction, 40 days to analysis
Tissue	TAL Metal, Titanium	Low	L-3, L-29	4		-15°C	6 months
Tissue	Mercury	Low	L-4	2	8.8 oz, G	-15°C	28 days
Tissue	Methylmercury	Low	L-5	1	8.8 oz, G	-15°C	28 days
Tissue	Butyltins	Low	L-6	30	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	Semivolatile Organics	Low	L-8	15	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	Semivolatile Organic SIM	Low	L-9	15	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	Aroclor PCBs	Low	L-10	15	8.8 oz, G	-15°C	14 days
Tissue	Pesticides	Low	L-11	15	8.8 oz, G	-15°C	7 days to extraction, 40 days to analysis
Tissue	% Moisture	Low	L-21	10	8.8 oz, G	-15°C	28 days

Notes:

[a] Analytical methods are as specified in Worksheet #23.
[b] These are minimum sample mass requirements for a single sample analysis post homogenization. Extra mass will be needed in order to fulfill quality control sample requirements, such as matrix spike/matrix spike duplicate and/or to provide contingency mass for re-analysis or breakage.
[c] Holding time is calculated from the date and time of sample homogenization, to the date and time of sample analysis (or extraction as noted).
[d] Maximum holding times listed are based upon those stipulated in corresponding data validation guidance located in Appendix C of this Crab and Clam Sampling and Analysis QAPP.

°C = degrees Celsius
G = amber glass
g = gram
NA = not applicable
oz = ounce
PCB = polychlorinated biphenyl
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
% = percent

QAPP Worksheet #20-1a (UFP-QAPP Manual Section 3.1.2): Sediment Field Quality Control Sample Summary

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[a]	Number of Samples Collected	Number of Field Duplicate Pairs	Number of MS[b]	Number of MSD or Duplicate	Number of Trip Blanks	Number of Rinse/ Field Blanks	Total Number of Samples to Lab[c]
Sediment	PCDDs/PCDFs	Low	L-1	8	1	1	1	NA	1	10
Sediment	PCB Congeners	Low	L-2	8	1	1	1	NA	1	10
Sediment	TAL Metals, Titanium	Low	L-3, L-29	8	1	1	1	NA	1	10
Sediment	Mercury	Low	L-4	8	1	1	1	NA	1	10
Sediment	Methylmercury	Low	L-5	8	1	1	1	5[d]	1	15
Sediment	Butyltins	Low	L-6	8	1	1	1	NA	1	10
Sediment	Chlorinated Herbicides	Low	L-7	8	1	1	1	NA	1	10
Sediment	Semivolatile Organics	Low	L-8	8	1	1	1	NA	1	10
Sediment	Semivolatile Organics SIM	Low	L-9	8	1	1	1	NA	1	10
Sediment	Aroclor PCBs	Low	L-10	8	1	1	1	NA	1	10
Sediment	Pesticides	Low	L-11	8	1	1	1	NA	1	10
Sediment	Volatile Organics	Low	L-12	8	1	1	1	5[d]	1	15
Sediment	Cyanide	Low	L-13	8	1	1	1	NA	1	10
Sediment	TOC	Low	L-15	8	1	1	1	NA	1	10
Sediment	TEPH	Low	L-16	8	1	1	1	NA	1	10
Sediment	Hexavalent Chromium	Low	L-17	8	1	1	1	NA	1	10
Sediment	pH	Low	L-18	8	1	NA	1	NA	0	9
Sediment	% Moisture	Low	L-20	8	1	NA	1	NA	0	9
Sediment	Total Sulfide	Low	L-22	8	1	1	1	NA	0	9
Sediment	Total Phosphorus	Low	L-23	8	1	1	1	NA	1	10
Sediment	Total Kjedahl Nitrogen	Low	L-24	8	1	1	1	NA	1	10
Sediment	AVS/SEM	Low	L-25	8	1	1	1	NA	0	9
Sediment	Ammonia	Low	L-26	8	1	1	1	NA	1	10
Sediment	Grain Size	Low	L-27	8	1	NA	1	NA	0	9
Sediment	ORP	Low	L-28	8	1	NA	2	NA	0	9

See the last page of Worksheet #20-1a for a description of footnotes.

QAPP Worksheet #20-1a (UFP-QAPP Manual Section 3.1.2): Sediment Field Quality Control Sample Summary (continued)

Notes:

[a] Referenced from the Analytical SOP References Table (Worksheet #23).
[b] Assumes MS/MSD will be collected at a rate of one (MS) and one (MSD) per up to 20 samples or per SDG (whichever is more frequent) for all constituents, except cyanide, which will have one MS and laboratory duplicate per up to 20 samples collected or per SDG, whichever is more frequent. Grain size, % moisture, pH, and % lipids will have laboratory duplicates, no MS/MSD, ORP will have two laboratory duplicates.
[c] This value excludes MS/MSD.
[d] The number of trip blanks may vary depending upon the duration of sampling. One trip blank is required to be transported with each shipment of samples when volatile organics or methylmercury will be analyzed.

AVS/SEM = acid volatile sulfide/simultaneously extracted metals
MS = matrix spike
MSD = matrix spike duplicate
NA = not applicable
ORP = oxidation reduction potential
PCB = polychlorinated biphenyl
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
SDG = sample delivery group
SOP = Standard Operating Procedure
TAL = Target Analyte List
TEPH = total extractable petroleum hydrocarbons
TOC = total organic carbon
% = percent

QAPP Worksheet #20-1b UFP-QAPP Manual Section 3.1.2): Tissue – Clam[a] Field Quality Control Sample Summary

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[b]	Number of Samples Collected	Number of Field Duplicate Pairs	Number of MS[c]	Number of MSD or Duplicates	Number of Trip Blanks	Number of Rinse/ Field Blanks	Total Number of Samples to Lab[d]
Tissue	PCDDs/PCDFs	Low	L-1	8	0	1	1	NA	2	10
Tissue	% Lipids	Low	L-1	NA[e]	NA	NA	NA	NA	0	0
Tissue	PCB Congeners	Low	L-2	8	0	1	1	NA	2	10
Tissue	TAL Metals, Titanium	Low	L-3, L-29	8	0	1	1	NA	2	10
Tissue	Mercury	Low	L-4	8	0	1	1	NA	2	10
Tissue	Methylmercury	Low	L-5	8	0	1	1	5[f]	2	15
Tissue	Butyltins	Low	L-6	8	0	1	1	NA	2	10
Tissue	Semivolatile Organics	Low	L-8	8	0	1	1	NA	2	10
Tissue	Semivolatile Organics SIM	Low	L-9	8	0	1	1	NA	2	10
Tissue	Aroclor PCBs	Low	L-10	8	0	1	1	NA	2	10
Tissue	Pesticides	Low	L-11	8	0	1	1	NA	2	10
Tissue	% Moisture	Low	L-21	8	0	NA	1	NA	0	9

Notes:

[a] Represents Clam tissue after shucking at TestAmerica, Burlington, VT. Homogenization will occur before aliquoting for each individual analytical group.
[b] Referenced from the Analytical SOP References Table (Worksheet #23).
[c] Assumes MS/MSD will be collected at a rate of one (MS) and one (MSD) per up to 20 samples or per SDG (whichever is more frequent) for all constituents, except for % moisture.
[d] This value excludes MS/MSD.
[e] The % lipids analysis is part of the analytical procedure for Method 1613B (dioxin analysis) and, therefore, requires no extra sample or quality control samples.
[f] The number of trip blanks may vary depending upon the duration of sampling. One trip blank is required to be transported with each shipment of methylmercury samples.
MS = matrix spike
MSD = matrix spike duplicate
NA = not applicable
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/dibenzofurans
SDG = sample delivery group
SOP = standard operating procedure
TEPH = total extractable petroleum hydrocarbons
% = percent

QAPP Worksheet #20-1c UFP-QAPP Manual Section 3.1.2): Tissue – Crab Heptapancreas[a] Field Quality Control Sample Summary

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[b]	Number of Samples Collected	Number of Field Duplicate Pairs	Number of MS[c]	Number of MSD or Duplicates	Number of Trip Blanks	Number of Rinse/ Field Blanks	Total Number of Samples to Lab[d]
Tissue	PCDDs/PCDFs	Low	L-1	8	0	1	1	NA	2	10
Tissue	% Lipids	Low	L-1	NA[e]	NA	NA	NA	NA	0	0
Tissue	PCB Congeners	Low	L-2	8	0	1	1	NA	2	10
Tissue	TAL Metals, Titanium	Low	L-3, L-29	8	0	1	1	NA	2	10
Tissue	Mercury	Low	L-4	8	0	1	1	NA	2	10
Tissue	Methylmercury	Low	L-5	8	0	1	1	5[f]	2	15
Tissue	Butyltins	Low	L-6	8	0	1	1	NA	2	10
Tissue	Semivolatile Organics	Low	L-8	8	0	1	1	NA	2	10
Tissue	Semivolatile Organics SIM	Low	L-9	8	0	1	1	NA	2	10
Tissue	Aroclor PCBs	Low	L-10	8	0	1	1	NA	2	10
Tissue	Pesticides	Low	L-11	8	0	1	1	NA	2	10
Tissue	% Moisture	Low	L-21	8	0	NA	1	NA	0	9

Notes:

[a] Represents Crab tissue after dissection and tissue organ separation at TestAmerica, Burlington, VT. Homogenization will occur before aliquoting for each individual analytical group.
[b] Referenced from the Analytical SOP References Table (Worksheet #23).
[c] Assumes MS/MSD will be collected at a rate of one (MS) and one (MSD) per up to 20 samples or per SDG (whichever is more frequent) for all constituents, except for % moisture.
[d] This value excludes MS/MSD.
[e] The % lipids analysis is part of the analytical procedure for Method 1613B (dioxin analysis) and, therefore, requires no extra sample or quality control samples.
[f] The number of trip blanks may vary depending upon the duration of sampling. One trip blank is required to be transported with each shipment of Methylmercury samples.
MS = matrix spike
MSD = matrix spike duplicate
NA = not applicable
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/dibenzofurans
SDG = sample delivery group
SOP = standard operating procedure
TEPH = total extractable petroleum hydrocarbons
% = percent

QAPP Worksheet #20-1d UFP-QAPP Manual Section 3.1.2): Tissue – Crab Muscle[a] Field Quality Control Sample Summary

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[b]	Number of Samples Collected	Number of Field Duplicate Pairs	Number of MS[c]	Number of MSD or Duplicates	Number of Trip Blanks	Number of Rinse/ Field Blanks	Total Number of Samples to Lab[d]
Tissue	PCDDs/PCDFs	Low	L-1	8	0	1	1	NA	2	10
Tissue	% Lipids	Low	L-1	NA[e]	NA	NA	NA	NA	0	0
Tissue	PCB Congeners	Low	L-2	8	0	1	1	NA	2	10
Tissue	TAL Metals, Titanium	Low	L-3, L-29	8	0	1	1	NA	2	10
Tissue	Mercury	Low	L-4	8	0	1	1	NA	2	10
Tissue	Methylmercury	Low	L-5	8	0	1	1	5[f]	2	15
Tissue	Butyltins	Low	L-6	8	0	1	1	NA	2	10
Tissue	Semivolatile Organics	Low	L-8	8	0	1	1	NA	2	10
Tissue	Semivolatile Organics SIM	Low	L-9	8	0	1	1	NA	2	10
Tissue	Aroclor PCBs	Low	L-10	8	0	1	1	NA	2	10
Tissue	Pesticides	Low	L-11	8	0	1	1	NA	2	10
Tissue	% Moisture	Low	L-21	8	0	NA	1	NA	0	9

Notes:

[a] Represents Crab tissue after dissection and tissue organ separation at TestAmerica, Burlington, VT. Homogenization will occur before aliquoting for each individual analytical group.
[b] Referenced from the Analytical SOP References Table (Worksheet #23).
[c] Assumes MS/MSD will be collected at a rate of one (MS) and one (MSD) per up to 20 samples or per SDG (whichever is more frequent) for all constituents, except for % moisture.
[d] This value excludes MS/MSD.
[e] The % lipids analysis is part of the analytical procedure for Method 1613B (dioxin analysis) and, therefore, requires no extra sample or quality control samples.
[f] The number of trip blanks may vary depending upon the duration of sampling. One trip blank is required to be transported with each shipment of methylmercury samples.
MS = matrix spike
MSD = matrix spike duplicate
NA = not applicable
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/dibenzofurans
SDG = sample delivery group
SOP = standard operating procedure
TEPH = total extractable petroleum hydrocarbons
% = percent

QAPP Worksheet #21 (UFP-QAPP Manual Section 3.1.2): Project Sampling SOP References Table

SOP Reference Number	Title, Revision Date, and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
1	Locating Sample Points Using a Hand- Held Global Positioning System, (October 2013), Revision 0	ARCADIS	Hand-held GPS unit	Y*	Appendix B
2	Positioning, (October 2013), Revision 0	ARCADIS	Trimble Geo XT (or similar unit) with related cable and power supply	Y*	Appendix C
3	Decontamination (October 2013), Revision 0	ARCADIS	Scoops, bowls, spoons, traps, scales, clam forks, and any equipment that comes into contact with any crabs, clams, or sediment	Y*	Appendix G
4	Management and Disposal of Residuals (October 2013), Revision 0	ARCADIS	Open-top drums, storage racks, and insulated coolers	Y*	Appendix H
5	Containers, Preservation, Handling, and Tracking of Samples for Analysis (October 2013), Revision 0	ARCADIS	COC forms, custody seals, sample containers, packaging supplies, and coolers	Y*	Appendix I
6	Crab Sampling (October 2013), Revision 0	ARCADIS	Sampling vessel, crab traps, and supplies	Y	Appendix D
7	Clam Sampling (October 2013), Revision 0	ARCADIS	Sampling vessel, clam forks, and supplies	Y	Appendix E
8	Crab and Clam Tissue Processing (October 2013), Revision 0	ARCADIS	Scalpel, tissue grinder, glove box	Y	Appendix O

QAPP Worksheet #21 (UFP-QAPP Manual Section 3.1.2): Project Sampling SOP References Table (continued)

SOP Reference Number	Title, Revision Date, and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
9	Sediment Sample Collection (October 2013), Revision 0	ARCADIS	Sediment grab samplers, hand auger, and/or corer and fathometer (or weighted demarcated line)	Y	Appendix F
10	Documenting Field Activities (October 2013), Revision 0	ARCADIS	Wireless recording device (e.g., laptop), bound waterproof logbooks, electronic field data forms, camera	Y*	Appendix J
11	Data Management (October 2013), Revision 0	ARCADIS		Y*	Appendix K
BR-EX-009	Homogenization of Biota and Tissue (August 2012), Revision 7	TestAmerica	Cutting board, meat grinder, glass jars, homogenizer, food processor, stainless steel knives	N	Appendix M (laboratory SOP)

Note: * indicates that these SOPs are based on previously approved SOPs; either from Newark Bay Study Area Phase I/II Sediment Investigations, or CPG's Lower Passaic River SOPs.

QAPP Worksheet #22 (UFP-QAPP Manual Section 3.1.2.4): Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment[a]	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GPS receiver	The GPS receiver is calibrated automatically using satellite signals each time it is powered on.	Keep one set of fresh batteries available at all times. Keep dirt and dust away from GPS receiver.	Vessel will be stationed at the check point to verify GPS position with known land-survey coordinates.	Confirm there are no cracks in the unit and that the antenna has not been damaged.	Each time unit is powered on.	GPS receiver is suitable for use if it is reporting coordinates, indicating it is receiving signals from three independent GPS satellites.	If unit will not obtain a coordinate lock, move to an unobstructed location. If no unobstructed location is available, consider recording position at nearby unobstructed location and measuring horizontal offset that can be used to correct the measured position later.	FS or designee	1
Sediment grab samplers	NA	Decontamination.	NA	Inspect for physical damage that may compromise effectiveness of sampler.	Daily, prior to use.	Sampler is undamaged.	Repair damage, if possible, or replace trap as necessary.	FS or designee	9
YSI 556 multi-probe meter	The YSI 556 meter is calibrated daily when in use.	Keep one set of fresh batteries available at all times. Protect probes from damage.	Calibrate against known standards.	Confirm that the o-ring is installed and in good condition.	Daily, prior to use.	Meter is undamaged and has no error messages.	Recalibrate, repair, and replace as necessary.	FS or designee	10
Crab traps/ clam rakes and forks	NA	Decontamination.	NA	Inspect for physical damage that may compromise effectiveness.	Daily, prior to use.	Equipment is undamaged.	Repair damage, if possible, or replace as necessary.	FS or designee	6,7

QAPP Worksheet #22 (UFP-QAPP Manual Section 3.1.2.4): Field Equipment Calibration, Maintenance, Testing, and Inspection Table (continued)

Field Equipment[a]	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Crab/clam measuring ruler	NA	Decontamination.	NA	Verify that measurement markings are not worn away and remain legible.	Daily, prior to use.	Measure to 1 mm accuracy.	Replace illegible rulers as necessary.	FS or designee	6,7
Electronic scale	Calibrate scale using calibration weights; calibration weights will bracket the expected crab or clam weights.	Decontamination; keep one set of fresh batteries available at all times.	NA	Inspect for physical damage that may compromise accuracy.	Daily, prior to use.	Measure to 1 g accuracy.	If scale cannot be calibrated, install new batteries and recalibrate. If scale can still not be calibrated, continue with planned activity and obtain a new scale at the earliest opportunity.	FS or designee	6,7

Notes:

[a] Refer to Project Sampling SOP References table (Worksheet #21).

FS = Field Supervisor
GPS = global positioning system
SOP = standard operating procedure

QAPP Worksheet #23-1 (UFP-QAPP Manual Section 3.2.1): Analytical Method/SOP References

Reference Number	Analytical Method/SOP	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
L-1	USEPA Method 1613B	Definitive	PCDDs/PCDFs	HRGC/HRMS	Vista Analytical	N
L-1	USEPA Method 1613B	Definitive	% Lipids (tissue)	Gravimetric	Vista Analytical	N
L-2	USEPA Method 1668A	Definitive	PCB Congeners	HRGC/HRMS	Vista Analytical	N
L-3	SW-846 Method 6020	Definitive	TAL Metals	ICP/MS	TestAmerica	N
L-4	SW-846 Method 7471B	Definitive	Mercury	CVAA	TestAmerica	N
L-5	USEPA Method 1630	Definitive	Methylmercury	CVAFS	TestAmerica, North Canton	N
L-6	SOP No. BR-GC-008, Rev.10 1/07/13	Definitive	Butyltins	GC/FPD	TestAmerica	N
L-7	SW-846 Method 8151A	Definitive	Chlorinated Herbicides	GC/ECD	TestAmerica, Pittsburgh	N
L-8	SW-846 Method 8270D	Definitive	Semivolatile Organics	GC/MS	TestAmerica	N
L-9	SW-846 Method 8270D	Definitive	Semivolatile Organics SIM	GC/MS SIM	TestAmerica	Y
L-10	SW-846 Method 8082	Definitive	Aroclor PCBs	GC/ECD	TestAmerica	N
L-11	SOP No. BR-PS-002, Rev.0 12/28/12	Definitive	Pesticides	GC/ECD	TestAmerica	N
L-12	SW-846 Method 8260B	Definitive	Volatile Organics	GC/MS	TestAmerica	N
L-13	Method 9010B, 9010C, 9012A, 9013	Definitive	Cyanide	Colorimetric	TestAmerica	N
L-14	SW-846 Method 9060A	Definitive	TOC (liquid)	Combustion or oxidation	TestAmerica	N
L-15	Lloyd Kahn USEPA Region 2	Definitive	TOC (solid)	Pyrolitic determination	TestAmerica	N
L-16	NJDEP OQA-QAM-025-10/91 Rev.6 4/24/06	Definitive	TEPH	GC/FID	TestAmerica	N
L-17	SOP Analysis #04725, 7825, 2432, Rev.15 8/12/10	Definitive	Hexavalent Chromium	Colorimetric	eurofins/Lancaster Laboratories	N
L-18	USEPA Method 9045D	Definitive	pH (solid)	pH meter	eurofins/Lancaster Laboratories	N
L-19	USEPA Method 9040C	Definitive	pH (liquid)	pH meter	TestAmerica	N
L-20	ASTM D2216	Definitive	% Moisture (solid)	Gravimetric	TestAmerica	N
L-21	SOP No. BR-WC-006 Rev.7 8/16/12	Definitive	% Moisture (tissue)	Gravimetric	TestAmerica	N
L-22	SW-846 Method 9030B/9034	Definitive	Total Sulfide	Iodometric	TestAmerica	N

See the last page of Worksheet #23-1 for a description of footnotes.

QAPP Worksheet #23-1 (UFP-QAPP Manual Section 3.2.1): Analytical Method/SOP References

Reference Number	Analytical Method/SOP	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
L-23	USEPA Method 365.3	Definitive	Total Phosphorus	Spectrophotometer	TestAmerica	N
L-24	USEPA Method 351.3	Definitive	Total Kjeldhal Nitrogen	Spectrophotometer	TestAmerica	N
L-25	USEPA Method-821-R-91-100	Definitive	AVS/SEM	Gravimetric Colorimetric/Electrode	TestAmerica	N
L-26	USEPA Method 350.2/350.3	Definitive	Ammonia	Membrane Electrode	TestAmerica	N
L-27	ASTM D422	Definitive	Grain Size (solid)	Mechanical hydrometer	TestAmerica	N
L-28	SOP Analysis #1821, Rev.10 8/12/10	Definitive	Oxidation Reduction Potential	Membrane Electrode	eurofins/Lancaster Laboratories	N
L-29	USEPA Method 6010C	Definitive	Titanium	ICP/AES	TestAmerica	N

Notes:

ASTM = American Society for Testing and Materials
AVS/SEM = acid volatile sulfide/simultaneously extracted metals
CLP = Contract Laboratory Program
CVAA = cold vapor atomic absorption
CVAFS = cold vapor atomic fluorescence spectrometry
GC = gas chromatography
GC/ECD = gas chromatography/electron capture detector
GC/FID = gas chromatography/flame ionization detector
GC/FPD = gas chromatography/flame photometric detector
GC/MS = gas chromatography/mass spectrometry
HRGC/HRMS = high resolution gas chromatography/high resolution mass spectrometry
ICP/AES = inductively coupled plasma/atomic emission spectrometry
ICP/MS = inductively coupled plasma/mass spectrometry
NJDEP = New Jersey Department of Environmental Protection
PCB = polychlorinated biphenyl
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
SIM = selective ion monitoring
SM = standard methods (for examination of water and wastewater)
SOP = standard operating procedures
TAL = Target Analyte List
TOC = total organic carbon
TEPH = total extractable petroleum hydrocarbons
USEPA = U.S. Environmental Protection Agency
% = percent

QAPP Worksheet #23-2 (UFP-QAPP Manual Section 3.2.1): Analytical Method/SOP References Additional Text to Supplement

Modifications to SW-846 8270 for Semivolatile Organics (Select Ion Monitoring [SIM] Analyses (L-9)

Method USEPA SW-846 8270 is modified to provide for operation of the gas chromatograph/mass spectrometer in selective ion monitoring (SIM) mode, thereby achieving lower levels of detection for specific semi-volatile compounds.

QAPP Worksheet #24 (UFP-QAPP Manual Section 3.2.2): Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[a]
GC/MS	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-8, L-9, L-12
ICP/MS	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-3
CVAFS	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-5
CVAA	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-4

See the last page of Worksheet #24 for a description of footnotes.

QAPP Worksheet #25 (UFP-QAPP Manual Section 3.2.3): Analytical Instrument and Equipment Maintenance, Testing, and Inspection

All analytical instrumentation/equipment must be inspected, maintained, and tested. Procedures for inspection, maintenance, and testing of laboratory instruments and equipment are provided in the laboratory's quality assurance (QA) manuals and associated laboratory Standard Operating Procedures (SOPs). Appropriate QA manuals and SOPs will be obtained and submitted to the U.S. Environmental Protection Agency for review prior to mobilization.

Documentation of inspections, maintenance, and testing of laboratory instruments and equipment will include details of observed problems, corrective measures, routine maintenance, and instrument repair, including information regarding the repair and the individual who performed the repair.

At a minimum, major instruments will be backed up by comparable (if not equivalent) instrument systems to avoid unscheduled downtime. An inventory of spare parts will also be available to minimize equipment/instrument downtime.

QAPP Worksheet #24 (UFP-QAPP Manual Section 3.2.2): Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[a]
HRGC/HRMS	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Martha Maier Vista Analytical	L-1, L-2
GC/ECD	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-7, L-10, L-11
GC/FPD	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-6
pH meter	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica Wendy Kozma eurofins/Lancaster Laboratories	L-18, L19

See the last page of Worksheet #24 for a description of footnotes.

QAPP Worksheet #24 (UFP-QAPP Manual Section 3.2.2): Analytical Instrument Calibration (continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[a]
Catalytic Combustion/Infrared Detector	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-14
GC/FID	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-16
Various Geotechnical	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-27
Analytical Balance	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica Wendy Kozma eurofins/Lancaster Laboratories	All

See the last page of Worksheet #24 for footnotes.

QAPP Worksheet #24 (UFP-QAPP Manual Section 3.2.2): Analytical Instrument Calibration (continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[a]
Spectrophometer	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica Wendy Kozma eurofins/Lancaster Laboratories	L-13, L-17, L-23, L-24, L-25, L-26
Pyrolitic Detection Instrumentation	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-15
Electrode	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Wency Kozma eurofins/Lancaster Laboratories	L-28
ICP/AES	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	All laboratory instruments and equipment must be calibrated according to the schedule and procedures specified in the analytical method. Analytical methods are provided in Appendix M.	Acceptance criteria for all laboratory instrument calibrations are provided in Appendix M.	Project samples may not be analyzed until all calibration acceptance criteria are met.	Don Dawicki TestAmerica	L-29

See the last page of Worksheet #24 for a description of footnotes.

QAPP Worksheet #24 (UFP-QAPP Manual Section 3.2.2): Analytical Instrument Calibration (continued)

Notes:

[a] From the Analytical SOP References Worksheet #23.

CVAA = cold vapor atomic absorption
CVAFS = cold vapor atomic fluorescence spectrometry
GC/ECD = gas chromatograph/electron capture detector
GC/FID = gas chromatograph/flame ionization detector
GC/FPD = gas chromatograph/flame photometric detector
GC/MS = gas chromatograph/mass spectrometer
HRGC/HRMS = high resolution gas chromatography/high resolution mass spectrometer
ICP/AES = inductively coupled plasma/atomic emissions spectrometry
ICP/MS = inductively coupled plasma/mass spectrometer
SOP = Standard Operating Procedure

QAPP Worksheet #26 (UFP-QAPP Manual Section 3.3): Sample Handling System

Sample Collection, Packaging, and Shipment	Sediment and Crab/Clam Tissue Samples
Sample collection (personnel/organization):	ARCADIS
Sample packaging (personnel/organization):	ARCADIS
Coordination of shipment (personnel/organization):	ARCADIS
Type of shipment/carrier:	Overnight carrier (FedEx, UPS, or equivalent) from field
Sample Receipt and Analysis	
Sample receipt (personnel/organization):	Contact at appropriate laboratory
Sample custody and storage (personnel/organization):	Contact at appropriate laboratory
Sample preparation (personnel/organization):	Contact at appropriate laboratory
Sample determinative analysis (personnel/organization):	Contact at appropriate laboratory
Sample Archiving	
Field sample storage (number of days from sample collection):	Contact at appropriate laboratory
Sample extract/digestate storage (number of days from extraction/digestion):	1 year until TBD authorizes disposal
Sample Disposal	
Personnel/organization:	TBD
Number of days from analysis:	1 year until TBD authorizes disposal

QAPP Worksheet #27 (UFP-QAPP Manual Section 3.3.3): Sample Custody Requirements

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

Surface sediment samples and crab/clamtissuesamplesfor chemistry analysis will be collected in the field and packaged for transport to the appropriate laboratories in coolers with ice. Refrigerated samples will be shipped on wet ice. Samples will be shipped via overnight delivery. The SOPs for collecting the sediment and tissue samples are discussed in further detail in Appendices D, E, and F. The samples will be shipped to the analytical laboratories for chemistry analysis. The original signed chain of custody (COC) forms will be placed in a sealable plastic bag, sealed, and taped to the inside lid of the cooler. Fiber tape will be wrapped completely around the cooler. On each side of the cooler, a "This Side Up" arrow label will be attached; a "Handle with Care" label will be attached to the top of the cooler, and the cooler will be sealed with a custody seal in two locations. An example COC form and custody seal are provided in Appendix I.

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

Each contracted laboratory will have a laboratory-specific SOP that details the procedures used to document sample receipt and custody within the laboratory. The following procedures must be addressed in the laboratory custody SOP:

- Each laboratory must have a designated sample custodian who accepts custody of the samples at the time of delivery to the laboratory and verifies that the information on the sample labels matches the information on the COC. The sample custodian must sign and date all appropriate receiving documents and note any discrepancies in sample documentation, as well as the condition of the samples at the time of receipt.
- Once the samples have been accepted by the laboratory, checked, and logged in, they must be maintained in accordance with laboratory custody and security requirements as outlined in the laboratory Quality Management Plan (QMP).
- To confirm traceability of samples during the analytical process, the laboratory will assign a sample identification (ID) number based on procedures outlined in the laboratory QMP or laboratory SOP.
- The following procedures, at a minimum, must be documented by the laboratory:
  - Sample extraction/preparation
  - Sample analysis
  - Data reduction
  - Data reporting

Laboratory personnel are responsible for sample custody until the samples are returned to the sample custodian.

When sample analysis and QC procedure are completed, any remaining sample must be stored in accordance with contractual terms. Data sheets, custody documents, and all other laboratory records must be retained in accordance with contractual agreements.

Final Evidence Files

Laboratory records, including all field- and laboratory-initiated COCs and other sample receiving records, sample preparation and analysis records, and the final data package, become part of the laboratory final evidence file and must be retained as required by the contractual agreement. An original copy of the data package and associated electronic deliverable must be provided to the investigative organization in accordance with the contractual agreement and will be retained by the investigative organization, including associated field records and other related correspondence.

QAPP Worksheet #27 (UFP-QAPP Manual Section 3.3.3): Sample Custody Requirements (continued)

Sample Identification Procedures:

The sediment samples and crab/clamtissuesampleswill be identified with the site name, time, date, sampling location, and field crew initials. Unique alphanumeric ID numbers will be assigned to each sample depending on the analysis. The sample identification schematic is as follows and as discussed in SOP 5 (Appendix I):

The first two characters will be “NB” to identify the project area (Newark Bay).

The second two characters will be “03” to identify that this sample was collected as part of Phase III.

The next characters will define the sample type (i.e., “SED” to identify that the sample collected is for sediment analysis). “CLM” and “CRB” identify it is a tissue sample (clam and crab, respectively).

The final three characters are the location ID number. As samples 1 through 121 have been collected as part of Phases I and II, locations collected as part of this sampling event will start at 122.

For example, a sediment sample collected at Intertidal Area 1 would be labeled 122: “NB03SED122.”

Chain of Custody Procedures:

COC procedures are documented in detail in Appendix I and summarized briefly below. Samples are considered to be in custody if they are: 1) in the custodian's possession or view, 2) in a secured place (under lock) with restricted access, or 3) in a container and secured with an official seal(s), such that the sample cannot be reached without breaking the seal(s). Custody procedures as defined in Appendix I will be used for all samples throughout the collection and transport process. Custody procedures will be initiated during sample collection. An electronic COC form will accompany samples to the analytical laboratory. Each person who has custody of the samples will sign the COC form and confirm that the samples are not left unattended unless properly secured.

The Field Supervisor (FS) will be responsible for all sample tracking and custody procedures for samples in the field. The FS will be responsible for final sample inventory and will maintain sample custody documentation. The FS will also complete COC forms prior to removing samples from the sampling area. At the end of each day, and prior to transfer, COC entries will be made for all samples. Information on the labels will be checked against sample log entries, and samples will be recounted. COC forms will accompany all samples. The COC forms will be signed at each point of transfer. Copies of all COC forms will be retained and included as appendices to QA/QC reports and data reports. Samples will be shipped in sealed coolers.

ARCADIS will confirm that COC forms are properly signed upon receipt of the samples and will note questions or observations concerning sample integrity on the COC forms. ARCADIS will contact the Facility Coordinator and Project QA/QC Manager immediately if discrepancies are discovered between the COC forms and the sample shipment upon receipt.

QAPP Worksheet #28-1a (UFP-QAPP Manual Section 3.4): Sediment Field Quality Control Samples

Matrix	Sediment
Analytical Group	PCDDs/PCDFs, PCB Congeners, TAL Metals, Mercury, Methylmercury, Butyltins, Chlorinated Herbicides, Semivolatile Organics, Semivolatile Organics (SIM), Aroclor PCBs, Pesticides, Volatile Organics, Cyanide, TOC, TEPH, Hexavalent Chromium, pH, % Moisture, Total Sulfide, Total Phosphorus, Total Kjeldhal Nitrogen, AVS/SEM, Ammonia, Grain Size, ORP, Titanium
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-1, L-2, L-3, L-4, L-5, L-6, L-7, L-8, L-9, L-10, L-11, L-12, L-13, L-15, L-16, L-17, L-18, L-20, L-22, L-23, L-24, L-25, L-26, L-27, L-28, L-29
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical, TestAmerica, eurofins/Lancaster Laboratories
Number of Samples Collected	See Worksheet #20

QC Sample	Frequency Number	Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Duplicates	1 field duplicate will be collected at a frequent of 1 per sample delivery group not to exceed 1 per 20 field samples per matrix and per method	Associated data will be critically assessed to determine the impact to data quality	Precision	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by less than 2X the PQL when detects are <5X PQL for both field duplicate samples
Trip Blank	1 trip blank will be included with each shipment of samples which contains volatile organics or methylmercury field samples	Associated data will be critically assessed to determine the impact to data quality	Accuracy/Bias Contamination	No target compounds ≥PQL
Field Blank[a]	1 per sampling event. Note field blanks will not be collected for pH, % moisture, total sulfide, AVS/SEM, grain size or ORP analytical groups.	Associated data will be critically assessed to determine the impact to data quality	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet 28-1b for description of footnotes.

QAPP Worksheet #28-1b (UFP-QAPP Manual Section 3.4): Tissue Field Quality Control Samples

Matrix	Tissue
Analytical Group	PCDDs/PCDFs, PCB Congeners, TAL Metals, Mercury, Methylmercury, Butyltins, Semivolatile Organics, Semivolatile Organics (SIM), Aroclor PCBs, Pesticides, % Lipids, % Moisture, Titanium
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-1, L-2, L-3, L-4, L-5, L-6, L-8, L-9, L-10, L-11, L-21, L-29
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical, TestAmerica
Number of Samples Collected	See Worksheet #20-1b

QC Sample	Frequency Number	Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Rinsate Blank	1 per sampling event. Note rinse blanks will not be collected for % lipids or % moisture analytical groups.	Associated data will be critically assessed to determine the impact to data quality	Accuracy/Bias Contamination	No target compounds ≥PQL
Trip Blank	1 trip blank will be included with each shipment of samples which contains methylmercury field samples. Note rinse blanks will not be collected for % lipids or % moisture analytical groups.	Associated data will be critically assessed to determine the impact to data quality	Accuracy/Bias Contamination	No target compounds ≥PQL
Field Blank[a]	1 per sampling event, Note field blanks will not be collected for % lipids or % moisture analytical groups.	Associated data will be critically assessed to determine the impact to data quality	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet 28-1b for description of footnotes.

QAPP Worksheet #28-1 (UFP-QAPP Manual Section 3.4): Field Quality Control Samples

Notes:

[a] Field blanks will be collected in association with sediment and tissue.

AVS/SEM = acid volatile sulfide/simultaneously extracted metals
ORP = oxidation reduction potential
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/dibenzofurans
PQL= project quantitation limit
QC = quality control
RPD = relative percent difference
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
TEPH = total extractable petroleum hydrocarbon
TOC = total organic carbon

Description of Terms:

Rinsate Blank

Rinsate blanks check for sample contamination caused by reuse of decontaminated homogenization equipment, as well as the sampling process and transportation.

Rinsate blanks are blanks collected by pouring de-ionized water or solvent, whichever is appropriate to the contaminants of interest, over the homogenization sampling equipment after it has been cleaned in the field. Rinsate blanks are submitted for testing each sampling event not to exceed more than one per day.

Field Duplicate

Field duplicates are co-located samples to be collected as separate, discrete samples, which are assigned separate and discrete sample numbers. Field duplicates are not to be homogenized together, as this would defeat the purpose of the field replicate.

Trip Blank

A trip blank is a sample matrix that is as free of volatile analytes as possible and is transported with the samples to the laboratory without being opened. This serves as a check on sample contamination during transport, shipping, and storage before analysis.

Field Blank

Field blanks are blanks collected by pouring de-ionized water or solvent through the sample collection equipment in the field. This serves as a check on contamination prior to sampling collection processing and transportation.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples

Matrix	Field/Rinse/Equipment Blank
Analytical Group	PCDDs/PCDFs
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-1
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Ongoing Precision and Recovery	1 per extraction batch[a]	All target compound concentrations must fall within range provided in Table 6 of L-1	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target compound concentrations must fall within range provided in Table 6 of L-1
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	PCDDs/PCDFs
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-1
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration	At initial setup or when corrective action is taken, which may change calibration per L-1	Per L-1	Investigate and recalibrate, if necessary, per L-1.	Accuracy/Bias	Per L-1
Calibration Verification	Prior to every 12-hour period, but following Column Performance Solution and at end of 12-hour period per L-1	Per L-1	Investigate and recalibrate, if necessary, per L-1.	Accuracy/Bias	Per L-1
Labeled Compound Spike	1 per sample	Per L-1	The laboratory should review their protocols and correct any that may have caused exceedance of QC acceptance limits refer to L-1.	Accuracy/Bias	Per L-1
Labeled Internal Standard	1 per sample	Per L-1	The laboratory should review their protocols and correct any that may have caused exceedance of QC acceptance limits refer to L-1.	Accuracy/Bias	Per L-1

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-2
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Ongoing Precision and Recovery	1 per extraction batch[a]	All target compound analytes contained in the OPR must have observed concentration values that fall within the acceptance ranges provided in Table 6 of L-2	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target compound analytes contained in the OPR must have observed concentration values that fall within the acceptance ranges provided in Table 6 of L-2
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-2
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration	At initial setup or when corrective action is taken, which may change calibration per L-2	Per L-2	The laboratory must follow all corrective actions required by L-2.	Accuracy/Bias	Per L-2
Calibration Verification	Prior to every 12-hour period, but following Column Performance Solution and at end of 12-hour period per L-2	Per L-2	The laboratory must follow all corrective actions required by L-2.	Accuracy/Bias	Per L-2
Labeled Compound Spike	1 per sample	Per L-2	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Refer to L-2.	Accuracy/Bias	Per L-2
Labeled Internal Standards	1 per sample	Per L-2	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Refer to L-2.	Accuracy/Bias	Per L-2

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	TAL Metals, Titanium
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-3, L-29
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
		Compounds	**% Recovery**			**Compounds**	**% Recovery**
Laboratory Control Sample	1 per extraction batch[a]	All target analytes	80-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target analytes	80-120
Method Blanks	1 per digestion/distillation batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-digest/re-distill and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	
Instrument Blanks	Per analytical method	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected sample digestates. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-4
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per extraction batch[a]	**% Recovery** 80-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-digest/re-distill and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.		**% Recovery** 80-120
Method Blanks	1 per digestion/distillation batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-digest/re-distill and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Instrument Blanks	Per analytical method	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected sample digestates. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Methylmercury
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-5
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica, North Canton
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Ongoing Precision and Recovery	1 per digestion/distillation batch[a]	**% Recovery** 67-133	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-digest/re-distill and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 67-133
Initial Precision and Recovery	At initial setup or when corrective action is taken, which may change calibration per L-5	Per L-5	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits.	Accuracy/Bias	Per L-5
Method Blanks	1 per digestion/distillation batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-digest/re-distill and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Methylmercury
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-5
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica, North Canton
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Instrument Blanks	Per analytical method	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected sample digestates. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Initial Calibration	At initial setup or when corrective action is taken, which may change calibration per L-5	Per L-5	The laboratory must follow all corrective actions required per L-5.	Accuracy/Bias	Per L-5
Calibration Verification	Per L-5	Per L-5	Investigate and recalibrate, if necessary, per L-5.	Accuracy/Bias	Per L-5

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Butyltins
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-6
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	1 per sample	**Surrogate Compound** Tripentyltin	**% Recovery** 15-150	The laboratories should evaluate potential corrective actions and implement, as appropriate. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compound** Tripentyltin	**% Recovery** 15-150
Laboratory Control Sample	1 per extraction batch[a]	**Compounds** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 40-48 30-150 30-150 30-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 40-48 30-150 30-150 30-150
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Chlorinated Herbicides
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-7
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica, Pittsburgh
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	1 per sample	**Surrogate Compound** DCAA	**% Recovery** 50-120	The laboratories should evaluate potential corrective actions and implement, as appropriate. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compound** DCAA	**% Recovery** 50-120
Laboratory Control Sample	1 per extraction batch[a]	**Compounds** All target analytes	**% Recovery** 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target analytes	**% Recovery** 70-130
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Semivolatile Organics
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-8
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	16 per sample	**Surrogate Compounds** Phenol-d_5 2-Fluorobiphenyl 2-Fluorophenol 2,4,6-Tribromophenol Nitrobenzene-d_5 p-Terphenyl-d_{14}	**% Recovery**[a] 10-110 45-110 11-110 44-131 35-110 49-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Phenol-d_5 2-Fluorobiphenyl 2-Fluorophenol 2,4,6-Tribromophenol Nitrobenzene-d_5 p-Terphenyl-d_{14}	**% Recovery** 10-110 45-110 11-110 44-131 35-110 49-120
Laboratory Control Sample	1 per extraction batch[b]	**Compounds** All target analytes	**% Recovery** 20-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	**Compounds** All target analytes	**% Recovery** 20-150

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Semivolatile Organics
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-8
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per extraction batch[b]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Semivolatile Organics (SIM)
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-9
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	5 per sample	**Surrogate Compounds**	**% Recovery**	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds**	**% Recovery**
		Naphthalenel-d_8	20-130			Naphthalenel-d_8	20-130
		Acenaphthene-d_{10}	20-130			Acenaphthene-d_{10}	20-130
		Phenanthrene-d_{10}	20-130			Phenanthrene-d_{10}	20-130
		Chrysene-d_{12}	20-130			Chrysene-d_{12}	20-130
		Perylene-d_{12}	20-130			Perylene-d_{12}	20-130
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Aroclor PCBs
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/ SOP Reference	L-10
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	2 per sample	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-144 43-144	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-144 43-144
Laboratory Control Sample	1 per extraction batch[a]	**Compounds** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Aroclor PCBs
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-10
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Instrument Blanks	1 per 12 hour sequence	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected sample extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Pesticides
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-11
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	2 per sample	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-135 43-135	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-135 43-135
Laboratory Control Sample	1 per extraction batch[a]	**Compounds** All target analytes	**% Recovery** 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target analytes	**% Recovery** 70-130

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Pesticides
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-11
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Instrument Blanks	1 per 12 hour sequence	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected sample extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Trip/Equipment Blank
Analytical Group	Volatile Organics
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/ SOP Reference	L-12
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	13 per sample	**Surrogate Compounds** 1,2-Dichloroethane-d_4 4-Bromofluorobenzene-d_4 Toluene-d_8 Dibromofluoromethane	**% Recovery** 59-150 63-143 61-145 65-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** 1,2-Dichloroethane-d_4 4-Bromofluorobenzene-d_4 Toluene-d_8 Dibromofluoromethane	**% Recovery** 59-150 63-143 61-145 65-150
Laboratory Control Sample	1 per extraction batch[a]	**Compounds** All target analytes	**% Recovery** 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target analytes	**% Recovery** 70-130
Method Blanks	1 per 12 hour sequence	No target compounds ≥ PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥ PQL	

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Cyanide
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-13
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 75-125
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	TOC
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-14
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	% Recovery 50-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	% Recovery 50-150
Method Blanks	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Instrument Blanks	1 per 10 determinations	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected field samples. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	TEPH
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-16
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	2 per sample	**Surrogate Compounds** Chlorobenzene Ortho-terphenyl	**% Recovery** 60-120 60-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Chlorobenzene Ortho-terphenyl	**% Recovery** 60-120 60-120
Laboratory Control Sample	1 per extraction batch[a]	**Compound** #2 Diesel	**% Recovery** 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compound** #2 Diesel	**% Recovery** 70-120
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Hexavalent Chromium
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-17
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	eurofins/Lancaster Laboratories
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 75-125
Instrument Blanks	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected field samples. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL

See the last page of Worksheet #28-2a for footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Total Phosphorus
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-23
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 91-114	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 91-114
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Total Kjeldhal Nitrogen
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-24
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 90-110	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 90-110
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Matrix	Field/Rinse/Equipment Blank
Analytical Group	Ammonia
Concentration Level	Low
Sampling SOP	SOP-3
Analytical Method/SOP Reference	L-26
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 90-110	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 90-110
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL

See the last page of Worksheet #28-2a for a description of footnotes.

QAPP Worksheet #28-2a (UFP-QAPP Manual Section 3.4): Field/Rinse/Equipment Blank Laboratory Quality Control Samples (continued)

Notes:

[a] A batch is defined as a group of up to 20 samples of the same matrix, prepared at the same time, using the same procedure.

DCAA = 2,4-dichlorophenylacetic acid
OPR = ongoing precision and recovery
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/dibenzofurans
PQL = project quantitation limit
QC = quality control
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
TEPH = total extractable petroleum hydrocarbons
TOC = total organic carbon
% = percent

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples

Matrix	Sediment
Analytical Group	PCDDs/PCDFs
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-1
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
		Compounds	**RPD**			**Compounds**	**RPD**
Matrix Spike Duplicate	1 per extraction batch[a]	All target analytes	<50	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	All target analytes	<50
		Compounds	**% Recovery**			**Compounds**	**% Recovery**
Matrix Spike	1 per extraction batch[a]	All target analytes	60-140	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target analytes	60-140

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	PCDDs/PCDFs
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-1
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Ongoing Precision and Recovery	1 per extraction batch[a]	All target compound concentrations must fall within the acceptance ranges provided in Table 6 of L-1	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target compound concentrations must fall within the acceptance ranges provided in Table 6 of L-1
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Initial Calibration	At initial setup or when corrective action is taken, which may change calibration per L-1	Per L-1	Investigate and recalibrate, if necessary, per L-1	Accuracy/Bias	Per L-1

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	PCDDs/PCDFs
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-1
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration Verification	Prior to every 12-hour period, but following Column Performance Solution and at end of 12-hour period per L-8	Per L-1	Investigate and recalibrate, if necessary, per L-1.	Accuracy/Bias	Per L-1
Labeled Compound Spike	1 per sample	Per L-1	The laboratory should review their protocols and correct any that may have caused exceedance of QC acceptance limits, refer to L-1.	Accuracy/Bias	Per L-1
Labeled Internal Standard	1 per sample	Per L-1	The laboratory should review their protocols and correct any that may have caused exceedance of QC acceptance limits, refer to L-1.	Accuracy/Bias	Per L-1

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-2
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compounds** 86 target analytes	**RPD** <50	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**Compounds** 86 target analytes	**RPD** <50
Matrix Spike	1 per extraction batch[a]	**Compounds** 86 target analytes	**% Recovery** 60-140	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** 86 target analytes	**% Recovery** 60-140

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-2
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Ongoing Precision and Recovery	1 per extraction batch[a]	All target compound concentrations must fall within the acceptance ranges provided in Table 6 per L-2	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target compound concentrations must fall within the acceptance ranges provided in Table 6 per L-2
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-2
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration	At initial setup or when corrective action is taken, which may change calibration per L-2	Per L-2	Investigate and recalibrate, if necessary, per L-2.	Accuracy/Bias	Per L-2
Calibration Verification	Prior to every 12-hour period, but following Column Performance Solution and at end of 12 hour period per L-2	Per L-2	Investigate and recalibrate, if necessary, per L-2.	Accuracy/Bias	Per L-2
Labeled Compound Spike	1 per sample	Per L-2	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Refer to protocol in L-2.	Accuracy/Bias	Per L-2
Labeled Internal Standards	1 per sample	Per L-2	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Refer to protocol in L-2.	Accuracy/Bias	Per L-2

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	TAL Metals, Titanium
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-3, L-29
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike	1 per extraction batch[a]	**Compounds** All target analytes	**% Recovery** 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target analytes	**% Recovery** 75-125
Laboratory Control Sample	1 per extraction batch[a]	**Compounds** All target analytes	**% Recovery** 80-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target analytes	**% Recovery** 80-120

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	TAL Metals, Titanium
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-3, L-29
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Instrument Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Laboratory Duplicate	1 per extraction batch[a]	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	Per L-5

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-4
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	1 per extraction batch[a]	**% Recovery** 80-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 80-120
Laboratory Control Sample	1 per extraction batch[a]	**% Recovery** 80-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 80-120

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-4
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Instrument Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Laboratory Duplicate	1 per extraction batch[a]	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	Per L-4

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Methylmercury
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-5
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica, North Canton
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	1 per extraction batch[a]	**RPD** ≤35%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤35%
Matrix Spike	1 per extraction batch[a]	**% Recovery** 65-135	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 65-135
Ongoing Precision and Recovery	1 per extraction batch[a]	**% Recovery** 67-133	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 67-133

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Methylmercury
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-5
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica, North Canton
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Precision and Recovery	At initial setup or when corrective action is taken, which may change calibration per L-5	Per L-5	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits.	Accuracy/Bias	Per L-5
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Methylmercury
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-5
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica, North Canton
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration	At initial setup or when corrective action is taken, which may change calibration per L-5	Per L-5	Investigate and recalibrate, if necessary, per L-5.	Accuracy/Bias	Per L-5
Calibration Verification	Prior to every 12-hour period, but following Column Performance Solution and at end of 12-hour period per L-5	Per L-5	Investigate and recalibrate, if necessary, per L-5.	Accuracy/Bias	Per L-5
Labeled Compound Spike	1 per sample	Per L-5	The laboratory should review their protocols and correct any that may have caused exceedance of QC acceptance limits, refer to L-5.	Accuracy/Bias	Per L-5

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Butyltins
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-6
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compounds** All target analytes	**RPD** <30	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision- Lab	**Compounds** All target analytes	**RPD** <30
Matrix Spike	1 per extraction batch[a]	**Compounds** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 10-48 30-160 30-160 30-160	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/ Bias	**Compounds** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 10-48 30-160 30-160 30-160
Surrogate Spikes	1 per sample	**Surrogate Compound** Tripentyltin	**% Recovery** 30-120	The laboratories should evaluate potential corrective actions and implement, as appropriate. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/ Bias	**Surrogate Compound** Tripentyltin	**% Recovery** 30-120

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Butyltins
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-6
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Laboratory Control Sample	1 per extraction batch[a]	**Compounds**	**% Recovery**	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds**	**% Recovery**
		Monobutyltin	10-48			Monobutyltin	10-48
		Dibutyltin	30-160			Dibutyltin	30-160
		Tributyltin	30-160			Tributyltin	30-160
		Tetrabutyltin	30-160			Tetrabutyltin	30-160
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Chlorinated Herbicides
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-7
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica, Pittsburgh
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compounds** All target analytes	**RPD** <35	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**Compounds** All target analytes	**RPD** <35
Matrix Spike	1 per extraction batch[a]	**Compounds** All target analytes	**% Recovery** 60-140	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target analytes	**% Recovery** 60-140
Surrogate Spikes	1 per sample	**Surrogate Compound** DCAA	**% Recovery** 50-120	The laboratories should evaluate potential corrective actions and implement, as appropriate. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compound** DCAA	**% Recovery** 50-120

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Chlorinated Herbicides
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-7
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica, Pittsburgh
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
		Compounds	**% Recovery**			**Compounds**	**% Recovery**
Laboratory Control Sample	1 per extraction batch[a]	All target analytes	70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target analytes	70-130
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Semivolatile Organics
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-8
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compounds** All target analytes	**RPD** <30	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**Compounds** All target analytes	**RPD** <30
Matrix Spike	1 per extraction batch[a]	**Compounds** All target analytes	**% Recovery** 20-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target analytes	**% Recovery** 20-150

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Semivolatile Organics
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-8
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	6 per sample	**Surrogate Compounds** Phenol-d_5 2-Fluorobiphenyl 2-Fluorophenol 2,4,6-Tribromphenol Nitrobenzene-d_5 p-Terphenyl-d_{14}	**% Recovery** 38-110 39-112 33-110 34-150 28-110 51-119	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Phenol-d_5 2-Fluorobiphenyl 2-Fluorophenol 2,4,6-Tribromphenol Nitrobenzene-d_5 p-Terphenyl-d_{14}	**% Recovery** 38-110 39-112 33-110 34-150 28-110 51-119
Laboratory Control Sample	1 per extraction batch[a]	**Compounds** All target analytes	**% Recovery** 20-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target analytes	**% Recovery** 20-150

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Semivolatile Organics
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-8
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Semivolatile Organics (SIM)
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-9
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compounds** All target analytes	**RPD** <40	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**Compounds** All target analytes	**RPD** <40
Matrix Spike	1 per extraction batch[a]	**Compounds** All target analytes	**% Recovery** 60-140	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/ Bias	**Compounds** All target analytes	**% Recovery** 60-140

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Semivolatile Organics (SIM)
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-9
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
		Surrogate Compounds	**% Recovery**			**Surrogate Compounds**	**% Recovery**
Surrogate Spikes	5 per sample	Acenaphthene-d_{10} Chrysene-d_{12} Naphthalene-d_8 Perylene-d_{12} Phenanthrene -d_{12}	20-130 20-130 20-130 20-130 20-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/ Bias	Acenaphthene-d_{10} Chrysene-d_{12} Naphthalene-d_8 Perylene-d_{12} Phenanthrene -d_{12}	20-130 20-130 20-130 20-130 20-130
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Aroclor PCBs
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-10
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compounds** Aroclor-1016 Aroclor-1260	**RPD** <15 <20	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision-Lab	**Compounds** Aroclor-1016 Aroclor-1260	**RPD** <15 <20
Matrix Spike	1 per extraction batch[a]	**Compounds** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130
Surrogate Spikes	2 per sample	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 30-150 30-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 30-150 30-150

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Aroclor PCBs
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-10
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
		Compound	**% Recovery**			**Compound**	**% Recovery**
Laboratory Control Sample	1 per extraction batch[a]	Aroclor-1016 Aroclor-1260	70-130 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	Aroclor-1016 Aroclor-1260	70-130 70-130
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	
Instrument Blanks	1 per 12 hour sequence	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected sample extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Pesticides
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-11
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compound** All target analytes	**RPD** <25	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**Compound** All target analytes	**RPD** <25
Matrix Spike	1 per extraction batch[a]	**Compound** All target analytes	**% Recovery** 50-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compound** All target analytes	**% Recovery** 50-150

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Pesticides
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-11
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	2 per sample	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-135 43-144	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-135 43-144
Laboratory Control Sample	1 per extraction batch[a]	**Compounds** All target compounds	**% Recovery** 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target compounds	**% Recovery** 70-130
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Volatile Organics
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-12
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compound** All target compounds	**RPD** <25	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, qualify data as needed.	Precision - Lab	**Compound** All target compounds	**RPD** <25
Matrix Spike	1 per extraction batch[a]	**Compound** All target compounds	**% Recovery** 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, qualify data as needed.	Accuracy/Bias	**Compound** All target compounds	**% Recovery** 70-130

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Volatile Organics
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-12
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	4 per sample	**Surrogate Compounds** 1,2-Dichloroethane-d_4 4-Bromofluorobenzene-d_4 Toluene-d_8 Dibromofluoromethane	**% Recovery** 70-141 65-131 72-123 71-141	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** 1,2-Dichloroethane-d_4 4-Bromofluorobenzene-d_4 Toluene-d_8 Dibromofluoromethane	**% Recovery** 70-141 65-131 72-123 71-141
Laboratory Control Sample	1 per extraction batch[a]	**Compounds** All target compounds	**% Recovery** 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target compounds	**% Recovery** 70-130

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Volatile Organics
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-12
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per 12 hour sequence	No target compounds ≥ PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥ PQL

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Cyanide
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-13
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	1 per digestion/distillation batch[a]	**% Recovery** 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-digest/re-distill and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 75-125
Laboratory Control Sample	1 per digestion/distillation batch[a]	**% Recovery** 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-digest/re-distill and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 75-125
Method Blanks	1 per digestion/distillation batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-digest/re-distill and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Cyanide
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-13
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Instrument Blanks	Per analytical method	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected sample digestates. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Laboratory Duplicate	1 per digestion/distillation batch[a]	RPD ≤20% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by less than 2X the PQL when detects are <5X PQL for both field duplicate samples.	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-digest/re-distill and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision-Lab	RPD ≤20% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by less than 2X the PQL when detects are <5X PQL for both field duplicate samples.

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	TOC
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-15
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	1 per analytical batch[a]	**% Recovery** 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 70-130
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 50-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 50-150

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	TOC
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-15
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Instrument Blanks	1 per 10 determinations	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected field samples. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤35%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤35%

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	TEPH
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-16
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**RPD** <50%		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision-Lab	**RPD** <50%	
Matrix Spike	1 per extraction batch[a]	**Compound** #2 Diesel	**% Recovery** 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compound** #2 Diesel	**% Recovery** 70-130
Surrogate Spikes	2 per sample	**Surrogate Compounds** Chlorobenzene Ortho-terphenyl	**% Recovery** 60-120 60-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Chlorobenzene Ortho-terphenyl	**% Recovery** 60-120 60-120

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	TEPH
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-16
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Laboratory Control Sample	1 per extraction batch[a]	**Compound** #2 Diesel	**% Recovery** 70-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compound** #2 Diesel	**% Recovery** 70-120
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Hexavalent Chromium
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-17
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	eurofins/Lancaster Laboratories
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	1 per analytical batch[a]	**% Recovery** 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 75-125
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 75-125

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Hexavalent Chromium
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-17
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	eurofins/Lancaster Laboratories
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL
Instrument Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤20%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤20%

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	pH
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-18
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	eurofins/Lancaster laboratories
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	pH acceptable range 6.82 to 6.93 when true value is 7.0	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	pH acceptable range 6.82 to 6.93 when true value is 7.0
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤25%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤25%

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	% Moisture
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-20
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤20%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤20%

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Total Sulfide
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-22
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	1 per extraction batch[a]	**RPD** <20%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** <20%
Matrix Spike	1 per analytical batch[a]	**% Recovery** 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 75-125

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Total Sulfide
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-22
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 75-125
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤20%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤20%

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Total Phosphorus
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-23
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	1 per analytical batch[a]	**RPD** ≤12%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤12%
Matrix Spike	1 per analytical batch[a]	**% Recovery** 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 75-125

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Total Phosphorus
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-23
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See of Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 91-114	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 91-114
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤12%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤12%

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Total Kjeldhal Nitrogen
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-24
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	1 per analytical batch[a]	**RPD** ≤20%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤20%
Matrix Spike	1 per analytical batch[a]	**% Recovery** 85-115	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 85-115

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Total Kjeldhal Nitrogen
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-24
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 90-110	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 90-110
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤20%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤20%

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	AVS/SEM
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-25
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per analytical batch[a]	**RPD** ≤20%		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤20%	
Matrix Spike	1 per analytical batch[a]	**Compounds** All target compounds	**% Recovery** 60-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compounds** All target compounds	**% Recovery** 60-130

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	AVS/SEM
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-25
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
		Compounds	**% Recovery**			**Compounds**	**% Recovery**
Laboratory Control Sample	1 per analytical batch[a]	All target compounds	60-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target compounds	60-130
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL	
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤20%		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤20%	

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Ammonia
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-26
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	1 per analytical batch[a]	**RPD** ≤20%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤20%
Matrix Spike	1 per analytical batch[a]	% Recovery 75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	% Recovery 75-125

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Ammonia
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-26
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	**% Recovery** 90-110	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 90-110
Method Blank	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	No target compounds ≥PQL
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤20%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤20%

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	Grain Size
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-27
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤20%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤20%

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Matrix	Sediment
Analytical Group	ORP
Concentration Level	Low
Sampling SOP	SOP-9
Analytical Method/SOP Reference	L-28
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	eurofins/Lancaster Laboratories
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per analytical batch[a]	Acceptance range is the certified value ± 30 mV	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	Acceptance range is the certified value ± 30 mV
Laboratory Duplicate	1 per analytical batch[a]	**RPD** ≤25%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**RPD** ≤25%

See the last page of Worksheet #28-2b for a description of footnotes.

QAPP Worksheet #28-2b (UFP-QAPP Manual Section 3.4): Sediment Laboratory Quality Control Samples (continued)

Notes:

[a] A batch is defined as a group of up to 20 samples of the same matrix, prepared at the same time, using the same procedure.

AVS/SEM = acid volatile sulfide/simultaneously extracted metals
DCAA = 2-4-Dichlorophenylacrtic acid
mV = millivolts
ORP = Oxidation Reduction Potential
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
PQL = project quantitation limit
QC = quality control
RPD = relative percent difference
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
TEPH = total extractable petroleum hydrocarbons
TOC = total organic carbon
% = percent

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples

Matrix	Tissue
Analytical Group	PCDDs/PCDFs
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-1
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compound** All target analytes	**RPD** <50	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision-Lab	**Compound** All target analytes	**RPD** <50
Matrix Spike	1 per extraction batch[a]	**Compound** All target analytes	**% Recovery** 60-140	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compound** All target analytes	**% Recovery** 60-140

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	PCDDs/PCDFs
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-1
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Ongoing Precision and Recovery	1 per extraction batch[a]	All target compound concentrations must fall within the acceptance ranges provided in Table 6 of L-1	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target compound concentrations must fall within the acceptance ranges provided in Table 6 of L-1
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Initial Calibration	At initial setup or when corrective action is taken, which may change calibration per L-1	Per L-1	Investigate and recalibrate, if necessary, per L-1	Accuracy/Bias	Per L-1

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	PCDDs/PCDFs
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-1
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See all of Worksheets #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration Verification	Prior to every 12-hour period, but following Column Performance Solution and at end of 12-hour period per L-1	Per L-1	Investigate and recalibrate, if necessary, per L-1.	Accuracy/Bias	Per L-1
Labeled Compound Spike	1 per sample	Per L-1	The laboratory should review their protocols and correct any that may have caused exceedance of QC acceptance limits, refer to L-1.	Accuracy/Bias	Per L-1
Labeled Internal Standard	1 per sample	Per L-1	The laboratory should review their protocols and correct any that may have caused exceedance of QC acceptance limits, refer to L-1.	Accuracy/Bias	Per L-1

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-2
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compound** 86 target analytes	**RPD** <50	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**Compound** 86 target analytes	**RPD** <50
Matrix Spike	1 per extraction batch[a]	**Compound** 86 target analytes	**% Recovery** 60-140	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compound** 86 target analytes	**% Recovery** 60-140

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-2
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Ongoing Precision and Recovery	1 per extraction batch[a]	All target compound concentrations must fall within the acceptance ranges provided in Table 6 per L-2	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target compound concentrations must fall within the acceptance ranges provided in Table 6 per L-2
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-2
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration	At initial setup or when corrective action is taken, which may change calibration per L-2	Per L-2	Investigate and recalibrate, if necessary, per L-2.	Accuracy/Bias	Per L-2
Calibration Verification	Prior to every 12-hour period, but following Column Performance Solution and at end of 12 hour period per L-2	Per L-2	Investigate and recalibrate, if necessary, per L-2.	Accuracy/Bias	Per L-2
Labeled Compound Spike	1 per sample	Per L-2	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Refer to protocol in L-2.	Accuracy/Bias	Per L-2
Labeled Internal Standards	1 per sample	Per L-2	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Refer to protocol in L-2.	Accuracy/Bias	Per L-2

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	TAL Metals, Titanium
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-3, L-29
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
		Compound	**% Recovery**			**Compound**	**% Recovery**
Matrix Spike	1 per extraction batch[a]	All target analytes	75-125	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target analytes	75-125
Laboratory Control Sample	1 per extraction batch[a]	All target analytes	80-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target analytes	80-120

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	TAL Metals, Titanium
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-3, L-29
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Instrument Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Laboratory Duplicate	1 per extraction batch[a]	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision-Lab	Per L-3

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-4
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	1 per extraction batch[a]	% Recovery 80-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	% Recovery 80-120
Laboratory Control Sample	1 per extraction batch[a]	% Recovery 80-120	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	% Recovery 80-120

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-4
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Instrument Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Laboratory Duplicate	1 per extraction batch[a]	RPD ≤50% when target is detected in both field duplicate samples at >5X PQL, or concentrations differ by <2X the PQL when detects are <5X PQL for both field duplicate samples	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	Per L-4

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Methylmercury
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-5
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica, North Canton
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	1 per extraction batch[a]	**RPD** ≤35%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision-Lab	**RPD** ≤35%
Matrix Spike	1 per extraction batch[a]	**% Recovery** 65-135	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**% Recovery** 65-135
Ongoing Precision and Recovery	1 per extraction batch[a]	All target compound concentrations must fall within the acceptance ranges provided in Table 6 per L-5	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target compound concentrations must fall within the acceptance ranges provided in Table 6 per L-5

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Methylmercury
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-5
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica, North Canton
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Instrument Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Methylmercury
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-5
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmercia, North Canton
No. of Samples Collected	See all of Worksheets #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration	At initial setup or when corrective action is taken, which may change calibration per L-5	Per L-5	Investigate and recalibrate, if necessary, per L-5.	Accuracy/Bias	Per L-5
Calibration Verification	Prior to every 12-hour period, but following Column Performance Solution and at end of 12-hour period per L-5	Per L-5	Investigate and recalibrate, if necessary, per L-5.	Accuracy/Bias	Per L-5

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Butyltins
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-6
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compound** All target analytes	**RPD** <30	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**Compound** All target analytes	**RPD** <30
Matrix Spike	1 per extraction batch[a]	**Compounds** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 10-48 30-160 30-160 30-160	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/ Bias	**Compounds** Monobutyltin Dibutyltin Tributyltin Tetrabutyltin	**% Recovery** 10-48 30-160 30-160 30-160
Surrogate Spikes	1 per sample	**Surrogate Compound** Tripentyltin	**% Recovery** 30-120	The laboratories should evaluate potential corrective actions and implement, as appropriate. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/ Bias	**Surrogate Compound** Tripentyltin	**% Recovery** 30-120

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Butyltins
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-6
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Laboratory Control Sample	1 per extraction batch[a]	**Compound**	**% Recovery**	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compound**	**% Recovery**
		Monobutyltin	10-48			Monobutyltin	10-48
		Dibutyltin	30-160			Dibutyltin	30-160
		Tributyltin	30-160			Tributyltin	30-160
		Tetrabutyltin	30-160			Tetrabutyltin	30-160
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Semivolatile Organics
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-8
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
		Compounds	**RPD**			**Compounds**	**RPD**
Matrix Spike Duplicate	1 per extraction batch[a]	All target analytes	<30	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	All target analytes	<30
		Compounds	**% Recovery**			**Compounds**	**% Recovery**
Matrix Spike	1 per extraction batch[a]	All target analytes	20-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target analytes	20-150

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Semivolatile Organics
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-8
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	6 per sample	**Surrogate Compounds** Phenol-d_5 2-Fluorobiphenyl 2-Fluorophenol 2,4,6-Tribromphenol Nitrobenzene-d_5 p-Terphenyl-d_{14}	**% Recovery** 38-110 39-112 33-110 34-150 28-110 51-119	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Phenol-d_5 2-Fluorobiphenyl 2-Fluorophenol 2,4,6-Tribromphenol Nitrobenzene-d_5 p-Terphenyl-d_{14}	**% Recovery** 38-110 39-112 33-110 34-150 28-110 51-119
Laboratory Control Sample	1 per extraction batch[a]	**Compounds** All target analytes	**% Recovery** 20-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision-Lab	**Compounds** All target analytes	**% Recovery** 20-150

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Semivolatile Organics
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-8
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-digest/re-distill and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Semivolatile Organics (SIM)
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-9
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
		Compounds	**RPD**			**Compounds**	**RPD**
Matrix Spike Duplicate	1 per extraction batch[a]	All target analytes	<40	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	All target analytes	<40
		Compounds	**% Recovery**			**Compounds**	**% Recovery**
Matrix Spike	1 per extraction batch[a]	All target analytes	60-140	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target analytes	60-140

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Semivolatile Organics (SIM)
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-9
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Surrogate Spikes	6 per sample	**Surrogate Compounds** Acenaphthene-d_{10} Chrysene-d_{12} Naphthalene-d_8 Perylene-d_{12} Phenanthrene -d_{12}	**% Recovery** 20-130 20-130 20-130 20-130 20-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Acenaphthene-d_{10} Chrysene-d_{12} Naphthalene-d_8 Perylene-d_{12} Phenanthrene -d_{12}	**% Recovery** 20-130 20-130 20-130 20-130 20-130
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Aroclor PCBs
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-9
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compound** Aroclor-1016 Aroclor-1260	**RPD** <15 <20	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision-Lab	**Compound** Aroclor-1016 Aroclor-1260	**RPD** <15 <20
Matrix Spike	1 per extraction batch[a]	**Compound** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compound** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130
Surrogate Spikes	2 per sample	**Surrogate Compound** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 30-150 30-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compound** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 30-150 30-150

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Aroclor PCBs
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-10
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Laboratory Control Sample	1 per extraction batch[a]	**Compound** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compound** Aroclor-1016 Aroclor-1260	**% Recovery** 70-130 70-130
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	
Instrument Blanks	1 per 12 hour sequence	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. Re-analyze affected sample extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Pesticides
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-11
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Matrix Spike Duplicate	1 per extraction batch[a]	**Compound** All target analytes	**RPD** <25	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision - Lab	**Compound** All target analytes	**RPD** <25
Matrix Spike	1 per extraction batch[a]	**Compound** All target analytes	**% Recovery** 50-150	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Compound** All target analytes	**% Recovery** 50-150
Surrogate Spikes	2 per sample	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-135 43-144	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	**Surrogate Compounds** Tetrachloro-m-xylene Decachlorobiphenyl	**% Recovery** 43-135 43-144

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	Pesticides
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-11
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits		Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
		Compound	**% Recovery**			**Compound**	**% Recovery**
Laboratory Control Samples	1 per extraction batch[a]	All target analytes	70-130	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias	All target analytes	70-130
Method Blanks	1 per extraction batch[a]	No target compounds ≥PQL		The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-extract and re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL	

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	% Lipids
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-1
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	Vista Analytical
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blanks	1 per analytical batch[a]	No target compounds ≥PQL	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Accuracy/Bias Contamination	No target compounds ≥PQL
Laboratory Duplicate	1 per analytical batch[a]	RPD ≤35%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision- Lab	RPD ≤35%

See the last page of Worksheet #28-2c for a description of footnotes.

QAPP Worksheet #28-2c (UFP-QAPP Manual Section 3.4): Tissue Laboratory Quality Control Samples (continued)

Matrix	Tissue
Analytical Group	% Moisture
Concentration Level	Low
Sampling SOP	SOP-6, SOP-7, SOP-8
Analytical Method/SOP Reference	L-21
Field Sampling Organization	ARCADIS U.S., Inc.
Analytical Organization	TestAmerica
No. of Samples Collected	See Worksheet #20

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Duplicate	1 per analytical batch[a]	RPD ≤20%	The laboratories should review their protocols and correct any that may have caused exceedance of the QC acceptance limits. If sufficient field sample is available, re-analyze affected samples. If insufficient sample is available, re-analyze extracts. Qualify data as needed.	Precision- Lab	RPD ≤20%

Notes:

[a] A batch is defined as a group of up to 20 samples of the same matrix, prepared at the same time, using the same procedure.

PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
PQL = project quantitation limit
QC = quality control
RPD = relative percent difference
SIM = selective ion monitoring
SOP = Standard Operating Procedure
TAL = Target Analyte List
% = percent

QAPP Worksheet #29-1 (UFP-QAPP Manual Section 3.5): Project Documents and Records

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records
Airbills	Water Quality Meter	Hard copy data package as specified in Worksheet #29-2.	Laboratory QA Plans
Chain of Custody Records	Calibration and Sampling Reading Documentation	Electronic data deliverable as specified in Worksheet #29-4.	Laboratory Certifications
Daily Log	Notebook		MDL Study Information
Employee and Visitor Log			Data Validation Reports
Equipment Calibration and Maintenance Log			Corrective Action Reports - Field and Laboratory
Photograph logs			Analytical Technical Systems Audit Reports
Refrigeration Unit Sign-in Sheet			Field Technical Systems Audit Reports
Sample Field Data Sheet			Telephone logs
Sample Processing Form			
Pre-Printed Sample Label			
Custody Seal			
SDG Tracking Log			
Notebook			
Crab/Clam Sampling QAPP			
Bills of Lading			

Notes:

Crab/Clam Sampling QAPP = Crab and Clam Sampling and Analysis Quality Assurance Project Plan
MDL = method detection limit
QA = quality assurance
SDG = sample delivery group

QAPP Worksheet #29-2 (UFP-QAPP Manual Section 3.5): Required Analytical Chemistry Data Deliverable Elements

Data Deliverable Elements	PCDDs/PCDFs	PCB Congeners	TAL Metals. Titanium	Mercury	Methylmercury	% Lipids	Butyltins	Chlorinated Herbicides	Semivolatile Organics	Semivolatile Organics (SIM)	Aroclor PCBs	Pesticides	Volatile Organics	Cyanide	TOC	TEPH	Hexavalent Chromium	pH	% Moisture	Total Sulfide	Total Phosphorus	Total Kjeldhal Nitrogen	AVS/SEM	Ammonia	Grain Size	ORP
INVENTORY SHEET (Org. and Inorg. DC-2 Form)	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
NARRATIVE (Org. Narrative, Inorg. Cover Page)	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
SHIPPING/RECEIVING DOCUMENTS AND INTERNAL LABORATORY CHAIN OF CUSTODY RECORDS:																										
Airbills	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Chain of Custody Records/Forms (Traffic Report)	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Miscellaneous Shipping/Receiving Records	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Internal Laboratory Sample Transfer Records and Tracking Sheets	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
SAMPLE DATA:																										
Tabulated Summary Form for Field Sample Results (Org. and Inorg. Form I)	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Reconstructed total ion chromatogram (RIC) for each sample									X	X			X													
Raw spectra of target compound and background-subtracted spectrum of target compound for each sample									X	X			X													
Chromatograms from both columns for each sample								X			X	X				X										
GC integration report or data system printouts and calibration plots for each sample								X			X	X				X										
PEST/PCB/HERB/TEPH Identification Tabulated Summary Form (Org. Form X)								X			X	X				X										
For pest/PCBs/HERB confirmed by GC/MS, copies of raw spectra and background-subtracted spectrum of target compounds								X			X	X														
Gel permeation chromatography sample chromatograms									X	X	X															
Sample preparation/extraction/digestion log (Inorg. Form XIII) and notebook pages	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Sample analysis run log (Inorg. Form XIV) and notebook pages	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
ICP/Mercury/Cyanide/TOC/Titanium raw data			X	X	X									X	X											

See the last page of Worksheet #29-2 for a description of footnotes.

QAPP Worksheet #29-2 (UFP-QAPP Manual Section 3.5): Required Analytical Chemistry Data Deliverable Elements (Continued)

Data Deliverable Elements	PCDDs/PCDFs	PCB Congeners	TAL Metals, Titanium	Mercury	Methylmercury	% Lipids	Butyltins	Chlorinated Herbicides	Semivolatile Organics	Semivolatile Organics (SIM)	Aroclor PCBs	Pesticides	Volatile Organics	Cyanide	TOC	TEPH	Hexavalent Chromium	pH	% Moisture	Total Sulfide	Total Phosphorus	Total Kjeldhal Nitrogen	AVS/SEM	Ammonia	Grain Size	ORP
STANDARDS DATA:																										
Method Detection Limit Study Tabulated Summary Form	X	X	X	X	X		X	X	X	X	X	X	X	X	X	X	X			X	X	X	X	X		
Initial Calibration Tabulated Summary Form (Org. Form VI, Inorg. Form IIA)			X	X	X		X	X	X	X	X	X	X	X		X	X			X	X	X	X	X		
Continuing Calibration Tabulated Summary Form (Org. Form VII, Inorg. Form IIA)			X	X	X		X	X	X	X	X	X	X	X		X	X			X	X	X	X	X		
RICs and quantitation reports for all GC/MS standards									X	X			X													
Pest/Herbicide/Aroclor/TEPH Calibration Verification Tabulated Summary Form (Org. Form VII, Pest-1 and Pest-2)								X			X	X				X										
Pest/Herbicide/Aroclor/TEPH Analytical Sequence Tabulated Summary From (Org. From VIII-Pest)								X			X	X				X										
GC chromatograms and data system printouts for all GC standards					X			X			X	X				X										
For Aroclors/Herbicide confirmed by GC/MS, copies of spectra for standards used								X			X															
GPC Calibration Tabulated Summary From (Org. Form IX, Pest-2)											X															
Florisil Cartridge Check Tabulated Summary Form (Org. From IX, Pes-1)											X															
ICP Interelement Correction Factors Summary Form (Inorg. Form XIA and XIB)			X	X	X																					
ICP Linear Ranges Tabulated Summary Form (Inorg. Form XII)			X	X	X																					
CRDL Standards for AA and ICP Tabulated Summary From (Inorg. Form IIB)			X	X	X																					
Standards preparation notebook pages	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X

See the last page of Worksheet #29-2 for a description of footnotes.

QAPP Worksheet #29-2 (UFP-QAPP Manual Section 3.5): Required Analytical Chemistry Data Deliverable Elements (Continued)

Data Deliverable Elements	PCDDs/PCDFs	PCB Congeners	TAL Metals, Titanium	Mercury	Methylmercury	% Lipids	Butyltins	Chlorinated Herbicides	Semivolatile Organics	Semivolatile Organics (SIM)	Aroclor PCBs	Pesticides	Volatile Organics	Cyanide	TOC	TEPH	Hexavalent Chromium	pH	% Moisture	Total Sulfide	Total Phosphorus	Total Kjeldhal Nitrogen	AVS/SEM	Ammonia	Grain Size	ORP
QC DATA:																										
Tuning and Mass Calibration Tabulated Summary Form (Org. Form V)									X	X			X													
Surrogate Percent Recovery Tabulated Summary Form (Org. From II)							X	X	X	X	X	X	X			X										
MS/MSD Recovery Tabulated Summary Form (Org. Form III and Inorganic Form V)							X	X	X	X	X	X	X			X				X	X	X	X	X		
Method Blank Tabulated Summary Form (Org. Form IV and Inorg. Form III)					X		X	X	X	X	X	X	X	X	X	X	X			X	X	X	X	X		
Internal Standard Area and RT Tabulated Summary Form (Org. Form VIII)					X				X	X			X													
QC Raw Data - RICs, chromatograms, quantitation reports, integration reports, mass spectra, etc.								X	X	X	X	X	X													
ICP Interference Check Sample Tabulated Summary Form (Inorg. Form IV)			X	X	X																					
Spike Sample Recovery Tabulated Summary Form (Inorg. Form VA)			X	X	X									X	X		X									
Post-Digest Spike Sample Recovery Tabulated Summary Form (Inorg. Form VB)			X	X	X									X			X									
Duplicates Tabulated Summary Form (Inorg. Form VI)			X	X	X									X	X		X									
Internal Laboratory Control Sample Tabulated Summary Form (Inorg. Form VII)			X	X	X									X	X		X									
Standard Addition Results Tabulated Summary Form (Inorg. Form VIII)			X	X	X															X	X	X	X	X		
ICP Serial Dilutions Tabulated Summary Form (Inorg. Form IX)			X	X	X																					
QC raw data	X	X	X	X	X		X	X	X	X	X	X	X	X	X	X	X	X		X	X	X	X	X	X	X
QC Sample preparation notebook pages	X	X	X	X	X		X	X	X	X	X	X	X	X	X	X	X	X		X	X	X	X	X	X	X
Data of Sample preparation and analysis	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Sample Holding Temperature Documentation	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Sample Preservation Verification Documents	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X

See the last page of Worksheet #29-2 for a description of footnotes.

QAPP Worksheet #29-2 (UFP-QAPP Manual Section 3.5): Required Analytical Chemistry Data Deliverable Elements (Continued)

Data Deliverable Elements	PCDDs/PCDFs	PCB Congeners	TAL Metals, Titanium	Mercury	Methylmercury	% Lipids	Butyltins	Chlorinated Herbicides	Semivolatile Organics	Semivolatile Organics (SIM)	Aroclor PCBs	Pesticides	Volatile Organics	Cyanide	TOC	TEPH	Hexavalent Chromium	pH	% Moisture	Total Sulfide	Total Phosphorus	Total Kjeldhal Nitrogen	AVS/SEM	Ammonia	Grain Size	ORP
MISCELLANEOUS DATA:																										
Airbills	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Chain of Custody Records/Forms (Traffic Report)	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Miscellaneous Shipping/Receiving Records	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Internal Laboratory Sample Transfer Records and Tracking Sheets	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
HIGH RESOLUTION MASS SPECTROMETRY:																										
Analysis Data Sheet	X	X																								
Confirmation analysis datasheet	X	X																								
Cleanup standard recoveries	X	X																								
Tabulated relative retention times for samples	X	X																								
Selected Ion Current Profiles (SICPs) for each sample	X	X																								
Method blank summarized results	X	X																								
SICPs for each method blank	X	X																								
Mass Spectrometer resolutions demonstration SICPs for each analysis shift	X	X																								
SICPs for 12-hour continuing calibration standard	X	X																								
GC resolution demonstration SICPs for each 12-hour sequence	X	X																								
Initial Calibration Relative Retention Times	X	X																								
Initial Calibration Response Factors	X	X																								
Initial Calibration Ion Abundance Ratios	X	X																								
SICPs for the Initial Calibration	X	X																								
Initial Precision and Recovery	X	X																								
Ongoing Precision and Recovery	X	X																								
MS/MSD Results and Spiked Level Summary	X	X																								

See the last page of Worksheet #29-2 for a description of footnotes.

QAPP Worksheet #29-2 (UFP-QAPP Manual Section 3.5): Required Analytical Chemistry Data Deliverable Elements (Continued)

Notes:

( ) = form number; refer to CLP SOW forms if CLP is used
AA = atomic absorption
AVS/SEM = acid volatile sulfide/simultaneously extracted metals
CLP = Contract Laboratory Program
CRDL = contract required detection limit
Herb = herbicides
GC = gas chromatography
GC/MS = gas chromatography/mass spectrometry
GPC = gel permeation chromatography
ICP = inductively coupled plasma
Inorg. = inorganic
MS/MSD = matrix spike/matrix spike duplicate
Org. = organic
ORP = oxidation reduction potential
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
Pest = pesticides
QC = quality control
RT = retention time
SIM = selective ion monitoring
SIPCs = selected ion current profiles
SOW = statement of work
TAL = Target Analyte List
TEPH = total petroleum hydrocarbons
TOC = total organic carbon
% = percent

QAPP Worksheet #29-3 (UFP-QAPP Manual Section 3.5): Standard Laboratory Data Qualifiers

The laboratory qualifiers and their stated definitions provided below will be used by the laboratories exclusively when reporting sample results for this Crab and Clam Sampling and Analysis Quality Assurance Project Plan[a].

Qualifier	Description
B	Inorganics – The reported value was obtained from an instrument reading that was less than the project quantitation limit (PQL). Organics – The associated analyte was also detected in the method blank.
D	The organic analyte was quantitated from a diluted analysis.
E	Inorganics – The reported value is estimated because of the presence of an interference. Organics – The associated compound concentration exceeded the calibration range of the instrument.
G	Organic data indicated the presence of a compound that meets the identification criteria; the result is below the PQL but above the method detection limit (MDL) or estimated detection limit (EDL), where appropriate.
N	The inorganic analysis is associated with a spike sample not within control limits.
P	The percent difference between the primary and confirmation column for pesticide/Aroclor analyses is greater than 25%.
U *	The analyte was analyzed for, but was not detected above the reported sample quantitation limit. The inorganic duplicate analysis was not within the established quality control limit.
S +	Inorganics - The reported value was determined by Method of Standard Additions (MSA). Inorganics - Correlation coefficient for MSA is less than 0.995.
W	Inorganics - The post-digestion spike for furnace atomic absorption analysis is out of control.
I	The laboratory indicated the presence of an interference during the sample analysis.

Other Data Reporting Requirements:

Only analytical data that are verified/validated (see Worksheets #34, #35, and #36) will be reported.

- Organic analytes detected below the PQL, but above the MDL/EDL, will be reported with a "G" flag, and organic analytes detected below the PQL and MDL/EDL as non-detects at the PQL.

- Inorganic analytes detected below the PQL, but above the MDL, will be reported with a "B" flag, and inorganic analytes detected below the PQL and MDL as non-detects at the MDL. All analytes measured in the air matrix will be reported as non-detects at the PQL. Results in the air matrix will not be reported down to the established MDL values.

Notes:

[a] A final deliverable will be provided with standard laboratory qualifiers consistent with U.S. Environmental Protection Agency Electronic Deliverable Region 2 MEDD Format Reference Manual (www.epa.gov/region02/superfund/medd.html).

QAPP Worksheet #29-4 (UFP-QAPP Manual Section 3.5): Example Format for Electronic Loading of Laboratory Files

A final deliverable will be provided with standard laboratory qualifiers consistent with U.S. Environmental Protection Agency Electronic Deliverable Region 2 MEDD Format Reference Manual (www.epa.gov/region02/superfund/medd.html).

QAPP Worksheet #30 (UFP-QAPP Manual Section 3.5): Analytical Services

Matrix: All
Concentration: Low

Analytical Group	Sample Locations/ID Numbers	Analytical SOP	Data Package[a] Turnaround Time	Laboratory/Organization
PCDDs/PCDFs	As specified in Worksheet #18	L-1	30 days	Martha Maier Vista Analytical 1104 Windfield Way El Dorado Hills, CA 95762 916-673-1520
% Lipids (tissue only)	As specified in Worksheet #18	L-1	30 days	Martha Maier Vista Analytical 1104 Windfield Way El Dorado Hills, CA 95762 916-673-1520
PCB Congeners	As specified in Worksheet #18	L-2	30 days	Martha Maier Vista Analytical 1104 Windfield Way El Dorado Hills, CA 95762 916-673-1520
TAL Metals	As specified in Worksheet #18	L-3	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
Mercury	As specified in Worksheet #18	L-4	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026

See the last page of Worksheet #30 for a description of footnotes.

QAPP Worksheet #30 (UFP-QAPP Manual Section 3.5): Analytical Services

Matrix: All
Concentration: Low

Analytical Group	Sample Locations/ID Numbers	Analytical SOP	Data Package[a] Turnaround Time	Laboratory/Organization
Methylmercury	As specified in Worksheet #18	L-5	30 days	Don Dawicki TestAmerica 4101 Shuffel Steet North West North Canton, OH 44720 802-923-1026
Butyltins	As specified in Worksheet #18	L-6	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
Chlorinated Herbicides	As specified in Worksheet #18	L-7	30 days	Don Dawicki TestAmerica 301 Alpha Drive RIDC Park Pittsburgh, PA 15238 802-923-1026
Semivolatile Organics	As specified in Worksheet #18	L-8	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
Semivolatile Organics SIM	As specified in Worksheet #18	L-9	30 days	Martha Maier Vista Analytical 1104 Windfield Way El Dorado Hills, CA 95762 916-673-1520

See the last page of Worksheet #30 for a description of footnotes.

QAPP Worksheet #30 (UFP-QAPP Manual Section 3.5): Analytical Services (continued)

Matrix: All
Concentration: Low

Analytical Group	Sample Locations/ID Numbers	Analytical SOP	Data Package[a] Turnaround Time	Laboratory/Organization
Aroclor PCBs	As specified in Worksheet #18	L-10	30 days	Don Dawicki TestAmercia 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
Pesticides	As specified in Worksheet #18	L-11	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
Volatile Organics	As specified in Worksheet #18	L-12	30days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
Cyanide	As specified in Worksheet #18	L-13	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
TOC	As specified in Worksheet #18	L-14, L-15	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026

See the last page of Worksheet #30 for a description of footnotes.

QAPP Worksheet #30 (UFP-QAPP Manual Section 3.5): Analytical Services (continued)

Matrix: All
Concentration: Low

Analytical Group	Sample Locations/ID Numbers	Analytical SOP	Data Package[a] Turnaround Time	Laboratory/Organization
TEPH	As specified in Worksheet #18	L-16	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
Hexavalent Chromium	As specified in Worksheet #18	L-17	30 days	Wendy Kozma eurofins/Lancaster Laboratories 2425 New Holland Pike Lancaster, PA 17605 717-656-2300 ext. 1522
pH	As specified in Worksheet #18	L-18, L-19	30 days	Wendy Kozma eurofins/Lancaster Laboratories 2425 New Holland Pike Lancaster, PA 17605 717-656-2300 ext. 1522
% Moisture	As specified in Worksheet #18	L-20, L-21	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
Total Sulfide	As specified in Worksheet #18	L-22	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026

See the last page of Worksheet #30 for a description of footnotes.

QAPP Worksheet #30 (UFP-QAPP Manual Section 3.5): Analytical Services (continued)

Matrix: All
Concentration: Low

Analytical Group	Sample Locations/ID Numbers	Analytical SOP	Data Package[a] Turnaround Time	Laboratory/Organization
Total Phosphorus	As specified in Worksheet #18	L-23	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
Total Kjeldhal Nitrogen	As specified in Worksheet #18	L-24	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
AVS/SEM	As specified in Worksheet #18	L-25	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026
Ammonia	As specified in Worksheet #18	L-26	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-929-1026
Grain Size	As specified in Worksheet #18	L-27	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026

See the last page of Worksheet #30 for a description of footnotes.

QAPP Worksheet #30 (UFP-QAPP Manual Section 3.5): Analytical Services (continued)

Matrix: All
Concentration: Low

Analytical Group	Sample Locations/ID Numbers	Analytical SOP	Data Package[a] Turnaround Time	Laboratory/Organization
ORP	As specified in Worksheet #18	L-28	30 days	Wendy Kozma eurofins/Lancaster Laboratories 2425 New Holland Pike Lancaster, PA 17605 717-656-2300 ext. 7522
Titanium	As specified in Worksheet #18	L-29	30 days	Don Dawicki TestAmerica 30 Community Drive, Suite 11 South Burlington, VT 05403 802-923-1026

Notes:

[a] Turnaround time based on day the last sample in the sample delivery group is received by laboratory.

AVS/SEM = acid volatile sulfide/simultaneously extracted metals
ORP = oxidation reduction potential
PCB = polychlorinated biphenyl
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
SOP = Standard Operating Procedure
TAL = Target Analyte List
TEPH = total extractable petroleum hydrocarbons
TOC = total organic carbon
% = percent

QAPP Worksheet #31 (UFP-QAPP Manual Section 4.1): Planned Project Assessments

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organization Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Field Sampling Technical Systems Audit	1 audit of each program task	External	TBD[a], Auditor	TBD[a], Auditor	David Rigg Field Supervisor ARCADIS U.S., Inc.	David Rigg Field Supervisor ARCADIS U.S., Inc.	Melissa Beauchemin Project Manager ARCADIS U.S., Inc.
Laboratory Technical Systems Audit	1 audit of each participating laboratory prior to use or again after a period of inactivity associated with Tierra exceeding 2 years	External	Environmental Data Services, Ltd.	Diane Waldschmidt Quality Assurance Coordinator Environmental Data Services, Ltd	Laboratory Project Manager	Laboratory Project Manager	Diane Waldschmidt Quality Assurance Coordinator Environmental Data Services, Ltd
Field Readiness Review	1 at sampling startup	Internal	ARCADIS U.S., Inc.	TBD[a] Task Manager ARCADIS U.S., Inc.	David Rigg Field Supervisor ARCADIS U.S., Inc.	David Rigg Field Supervisor ARCADIS U.S., Inc.	Melissa Beauchemin Project Manager ARCADIS U.S., Inc.
Laboratory Readiness Review	1 at sampling startup	Internal	TestAmerica Burlington, VT	Don DawickiLaboratory Project Manger TestAmerica Burlington, VT	Various Laboratory Personnel	Various Laboratory Personnel	Don DawickiLaboratory Project Manger TestAmerica Burlington, VT
Laboratory Readiness Review	1 at sampling startup	Internal	eurofins/Lancaster Laboratories Lancaster, PA	Wendy Kozma Laboratory Project Manager eurofins/Lancaster Laboratories Lancaster, PA	Various Laboratory Personnel	Various Laboratory Personnel	Wendy KozmaLaboratory Project Manager eurofins/Lancaster Laboratories Lancaster, PA
Laboratory Readiness Review	1 at sampling startup	Internal	Vista Analytical El Dorado Hills, CA	Martha Maier Laboratory Project Manager Vista Analytical El Dorado Hills, CA	Various Personnel Vista Analytical El Dorado Hills, CA	Various Personnel Vista Analytical El Dorado Hills, CA	Martha Maier Laboratory Project Manager Vista Analytical El Dorado Hills, CA

See the last page of Worksheet #31 for a description of footnotes.

QAPP Worksheet #31 (UFP-QAPP Manual Section 4.1): Planned Project Assessments

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organization Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Internal Field Audits	1 on first day during field operations	Internal	ARCADIS U.S., Inc.	TBD[a] ARCADIS U.S., Inc.	David Rigg Field Supervisor ARCADIS U.S., Inc.	David Rigg Field Supervisor ARCADIS U.S., Inc.	Melissa Beauchemin Project Manager ARCADIS U.S., Inc.
Data Validation	All analytical data collected is validated[b]	External	Environmental Data Services, Ltd.	Diane Waldschmidt Quality Assurance Coordinator Environmental Data Services, Ltd.	Field and Laboratory Contractors	Field and Laboratory Contractors	Diane Waldschmidt Quality Assurance Coordinator Environmental Data Services, Ltd.

Notes:

[a] All TBD information will be provided prior to the start of the project.
[b] All data collected will undergo full data validation (with the exception of grain size, % moisture, and % lipids) as outlined in Worksheet #36. Grain size, % moisture, and % lipids data collected will be verified per Standard Operating Procedure.

TBD = to be determined

QAPP Worksheet #32 (UFP-QAPP Manual Section 4.1.2): Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, and Organization)	Timeframe for Response
Field Readiness Review	Meeting and memo	David Rigg Field Supervisor ARCADIS U.S., Inc.	24 hours after review complete	Memo	Carlie Thompson Facility Coordinator Tierra Solutions, Inc. Eugenia Naranjo Remedial Project Manager USEPA Region 2	Immediate
Laboratory Readiness Review	Meeting and memo	Don Dawicki TestAmerica Laboratory Project Manger	24 hours after review complete	Memo	Carlie Thompson Facility Coordinator Tierra Solutions, Inc. Eugenia Naranjo Remedial Project Manager USEPA Region 2	Immediate
Laboratory Readiness Review	Meeting and memo	Martha Maier Laboratory Project Manager Vista Analytical	24 hours after review complete	Memo	Carlie Thompson Facility Coordinator Tierra Solutions, Inc. Eugenia Naranjo Remedial Project Manager USEPA Region 2	Immediate
Laboratory Readiness Review	Meeting and memo	Wendy Kozma eurofins/Lancaster Laboratories Laboratory Project Manager	24 hours after review complete	Memo	Carlie Thompson Facility Coordinator Tierra Solutions, Inc. Eugenia Naranjo Remedial Project Manager USEPA Region 2	Immediate
Internal Field Audit	Audit Report	TBD[a] ARCADIS U.S., Inc.	1 week after audit is complete	Memo	TBD[a] Project Manager ARCADIS U.S., Inc.	1 week

See the last page of Worksheet #32 for a description of footnotes.

QAPP Worksheet #32 (UFP-QAPP Manual Section 4.1.2): Assessment Findings and Corrective Action Responses (continued)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Time frame of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, and Organization)	Time frame for Response
Data Validation[b]	Validation Reports	All of those listed above as impacted	40 days after receipt of laboratory and field deliverables	Resubmission of missing or corrected documents	Carlie Thompson Facility Coordinator Tierra Solutions, Inc., Eugenia Naranjo Remedial Project Manager USEPA Region 2	1 week after request for completion/correction
Field Sampling Technical Systems Audit	Audit Report/ Corrective Action Form	TBD[a] ARCADIS U.S., Inc.,	1 week after audit is complete	Audit Report/ Corrective Action Form	Carlie Thompson Facility Coordinator Tierra Solutions, Inc., Eugenia Naranjo Remedial Project Manager USEPA Region 2	Immediate
Laboratory Technical Systems Audit	Audit Report/ Corrective Action Form	Impacted Laboratory Project Manager	1 week after audit is complete	Audit Report/ Corrective Action Form	Carlie Thompson Facility Coordinator Tierra Solutions, Inc., Eugenia Naranjo Remedial Project Manager USEPA Region 2	Immediate

Notes:

[a] All TBD information will be provided prior to the start of the project.
[b] All data collected will undergo full data validation (with the exception of grain size, % moisture, and % lipids) as outlined in Worksheet #36. Grain size, % moisture, and % lipids data collected will be verified per Standard Operating Procedure.

QMP = quality management plan
TBD = to be determined
USEPA = U.S. Environmental Protection Agency

QAPP Worksheet #33 (UFP-QAPP Manual Section 4.2): QA Management Reports

Type of Report	Frequency (e.g., daily, weekly, monthly, quarterly, annually)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Field Sampling Technical Systems Audit Reports	1 per audit conducted	Within 30 days of audit completion	TBD[a] ARCADIS U.S., Inc.	Carlie Thompson Facility Coordinator Tierra Solutions, Inc. Eugenia Naranjo Remedial Project Manager USEPA Region 2
Laboratory Technical Systems Audit	1 per audit conducted	Within 30 days of audit completion	Diane Waldschmidt Quality Assurance Coordinator Environmental Data Services, Ltd.	Carlie Thompson Facility Coordinator Tierra Solutions, Inc. Eugenia Naranjo Remedial Project Manager USEPA Region 2
Internal Field Audit Report	1 per audit conducted	Within 30 days of audit completion	TBD[a] ARCADIS U.S., Inc.	Carlie Thompson Facility Coordinator Tierra Solutions, Inc. Eugenia Naranjo Remedial Project Manager USEPA Region 2
Data Validation Reports	1 per sample delivery group, per analytical group	Within 40 days of receipt of final deliverables in the sample delivery group	Diane Waldschmidt Quality Assurance Coordinator Environmental Data Services, Ltd.	Carlie Thompson Facility Coordinator Tierra Solutions, Inc. Eugenia Naranjo Remedial Project Manager USEPA Region 2
Data Usability Assessment Report	1 after completion of validation task	Within 40 days of validation completion	Diane Waldschmidt Quality Assurance Coordinator Environmental Data Services, Ltd.	Carlie Thompson Facility Coordinator Tierra Solutions, Inc. Eugenia Naranjo Remedial Project Manager USEPA Region 2

Notes:

All data collected will undergo full data validation (with the exception of grain size, % moisture, and % lipids data) as outlined in Worksheet #36. Grain size, % moisture, and % lipids data collected will be verified per standard operating procedure.

[a] All TBD information will be provided prior to the start of the project.

TBD = to be determined
USEPA = U.S. Environmental Protection Agency

QAPP Worksheet #34 (UFP-QAPP Manual Section 5.2.1): Verification (Step I) Process

Verification Input	Action	Internal/ External	Organization Responsible for Verification
Chain of custody and shipping forms	Chain of custody/requests for analysis forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the chain of custody should be initialed by the reviewer, a copy of the chain of custody retained in the site file, and the original and remaining copies taped inside the cooler for shipment.	I/E	ARCADIS U.S., Inc. Environmental Data Services, Ltd.
Field notes	Field notes will be reviewed internally and placed in the project file.	I	ARCADIS U.S., Inc.
Corrective Action Reports	Corrective Action Reports (when necessary) will be verified for completeness and signed by the Project Coordinator and Quality Assurance Coordinator. Corrective actions must also be communicated to appropriate field staff, as well as project review personnel.	I	Tierra Solutions, Inc. Environmental Data Services, Ltd.
Documentation of deviation from sample collection methods	Verify completeness and accuracy prior to distribution and placement in the project file. Communicate deviations to pertinent field staff, as well as project review personnel.	I	ARCADIS U.S., Inc.
Field-generated electronic data deliverables[a]	Verify completeness and accuracy prior to distribution and placement in the project file.	I	ARCADIS U.S., Inc.
Identification of QC samples	Verify completeness and accuracy prior to distribution and placement in the project file.	I	ARCADIS U.S., Inc.
Observed climatological condition recordings	Verify completeness and accuracy prior to distribution and placement in the project file.	I	ARCADIS U.S., Inc.
Equipment decontamination records	Verify completeness and accuracy prior to distribution and placement in the project file.	I	ARCADIS U.S., Inc.
Sampling equipment calibration logs	Verify completeness and accuracy prior to distribution and placement in the project file.	I	ARCADIS U.S., Inc.
Field measurements documentation	Verify completeness and accuracy prior to distribution and placement in the project file.	I	ARCADIS U.S., Inc.

See the last page of Worksheet #34 for a description of footnotes.

QAPP Worksheet #34 (UFP-QAPP Manual Section 5.2.1): Verification (Step I) Process (continued)

Verification Input	Action	Internal/ External	Organization Responsible for Verification
Sample condition upon receipt and storage records	Chain of custody/requests for analysis forms and shipping documentation will be reviewed upon cooler receipt. The contents of each cooler will be checked against information provided on the chain of custody/request for analysis forms. Cooler temperatures and specified sample preservation will also be verified and recorded.	I/E	TBD Vista Analytical
Internal laboratory chain of custody records	A complete set of chain of custody records must be produced and submitted with the data package for each SDG.	I	TBD Vista Analytical
Case Narrative to include the following: Identification of QC samples Communication logs Corrective action reports Documentation of corrective action results Documentation of laboratory method deviations Signatures for laboratory sign-off	Verify completeness and accuracy prior to shipping the laboratory report.	I	TBD Vista Analytical
Laboratory data package deliverables as specified in Worksheet #29-2	All laboratory data packages will be verified by the laboratory performing the work for completeness and technical accuracy prior to shipping the laboratory report.	I	TBD Vista Analytical
Laboratory electronic data deliverables as specified in Worksheet #29-4	All laboratory electronic data deliverables will be verified by the laboratory performing the work for completeness and accuracy prior to shipping the laboratory report.	I	TBD Vista Analytical
Laboratory and field data packages and electronic data deliverables as specified in Worksheets #29-2, #29-3, and #29-4	All received data packages will be verified externally for completeness according to the data verification/validation procedures specified in Worksheet #36.	E	Environmental Data Services, Ltd.

Notes:

[a] Field-generated electronic data deliverables will be formatted and submitted in USEPA Region 2 MEDD format with the correct reference codes provided as per the USEPA Electronic Data Deliverable Valid Values Reference Manual (available at http://www.epa.gov/region02/superfund/medd.htm)

E = external in relation to data generator
I = internal in relation to data generator
QC = quality control
SDG = sample delivery group
TBD = to be determined
USEPA = U.S. Environmental Protection Agency

QAPP Worksheet #35 (UFP-QAPP Manual Section 5.2.2): Validation (Steps IIa and IIb) Process

Step IIa/IIb	Validation Input	Description of Validation Procedure	Organization Responsible for Validation
IIa	Data Deliverables and QAPP	Confirm that all required information on sampling and analysis from data verification was provided.	Environmental Data Services, Ltd.
IIa	Analytes	Confirm that required lists of analytes were reported as specified in this Crab/Clam QAPP.	Environmental Data Services, Ltd.
IIa	Chain of Custody	Examine the traceability of the data from time of sample collection until reporting of data. Examine chain of custody records against contract, method, or procedural requirements.	Environmental Data Services, Ltd.
IIa	Holding Times	Identify holding time criteria and either confirm that they were met or document deviations. Confirm that samples were analyzed within holding times specified in this Crab/Clam QAPP. If holding times were not met, confirm that deviations were documented, that appropriate notifications were made (consistent with procedural requirements), and that approval to proceed was received prior to analysis.	Environmental Data Services, Ltd.
IIa	Sample Handling	Confirm that required sample handling, receipt, and storage procedures were followed, and that any deviations were documented. Sample preservation and temperature will specifically be evaluated and documented.	Environmental Data Services, Ltd.
IIa	Sampling Methods and Procedures	Establish that required sampling methods were used and that any deviations were noted. Confirm that the sampling procedures and field measurements met performance criteria and that any deviations were documented.	Environmental Data Services, Ltd.
IIa	Analytical Methods and Procedures	Establish that required analytical methods were used and that any deviations were noted.	Environmental Data Services, Ltd.
IIa	Data Qualifiers	Determine that the laboratory data qualifiers were defined and applied as specified in this Crab/Clam QAPP.	Environmental Data Services, Ltd.
IIa	Laboratory Transcription	Authenticate accuracy of the transcription of analytical data (i.e., laboratory notebook to reporting form or instrument to laboratory information management system).	Environmental Data Services, Ltd.
IIa	Standards	Determine that standards are traceable and meet contract, method, or procedural requirements.	Environmental Data Services, Ltd.
IIa	Communication	Establish that required communication procedures were followed by field or laboratory personnel.	Environmental Data Services, Ltd.
IIb	Field Duplicates	Compare results of field duplicates with criteria established in this Crab/Clam QAPP.	Environmental Data Services, Ltd.
IIb	Project Quantitation Limits	Determine that quantitation limits were achieved, as outlined in this Crab/Clam QAPP.	Environmental Data Services, Ltd.

See the last page of Worksheet #35 for a description of footnotes.

QAPP Worksheet #35 (UFP-QAPP Manual Section 5.2.2): Validation (Steps IIa and IIb) Process (continued)

Step IIa/IIb	Validation Input	Description of Validation Procedure	Organization Responsible for Validation
IIb	Confirmatory Analyses	Evaluate agreement between results of confirmatory analyses.	Environmental Data Services, Ltd.
IIb	Performance Criteria	Evaluate QC data against project-specific performance criteria in this Crab/Clam QAPP (i.e., evaluate quality parameters beyond those outlined in the methods).	Environmental Data Services, Ltd.
IIb	Analytical Methods and Procedures	Evaluate results of required QC samples and compare with acceptance criteria established in this Crab/Clam QAPP.	Environmental Data Services, Ltd.
IIb	Data Validation or Verification Reports	Full data validation will be performed on all analytical parameters, with the exception of grain size. Summarize deviations from methods, procedures, or contracts. Summarize outcome of comparison of data to MPC in this Crab/Clam QAPP. Include qualified data and explanation of all data qualifiers.	Environmental Data Services, Ltd.

Notes:

Crab/Clam QAPP = Crab and Clam Sampling and Analysis Quality Assurance Project Plan
MPC = Measurement Performance Criteria
QC = quality control
QL = quantitation limit

QAPP Worksheet #36 (UFP-QAPP Manual Section 5.2.2.1): Validation Guidance Summary Table[a]

Concentration Level: Low

Analytical Group	Validation Criteria	Data Validator
PCDDs/PCDFs	USEPA Region 2 SOP HW-25, Revision 3	Environmental Data Services, Ltd.
PCB Congeners	EDS SOP: Congener PCB, Rev.3, 7/10	Environmental Data Services, Ltd.
TAL Metals	USEPA Region 2 SOP HW-2b, Rev.15, 12/12	Environmental Data Services, Ltd.
Mercury	USEPA Region 2 SOP HW-2b, Rev.15, 12/12	Environmental Data Services, Ltd.
Methylmercury	EDS SOP: Methyl Mercury by CVAFS, USEPA 1630, Rev.0, 7/10	Environmental Data Services, Ltd.
Butyltins	SOP Organotins Prep. 8/05	Environmental Data Services, Ltd.
Chlorinated Herbicides	USEPA Region 2 SOP HW-17, Revision 3	Environmental Data Services, Ltd.
Semivolatile Organic	USEPA Region 2 SOP HW-22 Rev.2, 6/01	Environmental Data Services, Ltd.
Semivolatile Organics (SIM)	USEPA Region 2 HW-35, Revision 2, 3/13	Environmental Data Services, Ltd.
Aroclor PCBs	USEPA Region 2 SOP HW-37, Revision 1	Environmental Data Services, Ltd.
Pesticides	USEPA Region 2 SOP HW-36 Rev.4, 5/13	Environmental Data Services, Ltd.
Volatile Organics	USEPA Region 2 SOP HW-24, Rev.1, 6/99	Environmental Data Services, Ltd.
Cyanide	USEPA Region 2 SOP HW-2, Revision 13	Environmental Data Services, Ltd.
TOC (solid/liquid)	EDS SOP: TOC-01 Rev. 2, 7/10	Environmental Data Services, Ltd.
TEPH	EDS SOP: TEPH-01, Rev. 3, 07/07	Environmental Data Services, Ltd.
Hexavalent Chromium	NJDEP SOP for Analytical Data Validation of Hexavalent Chromium, 5.A.10, Rev. 2, 8/05	Environmental Data Services, Ltd.
pH (solid/liquid)	USEPA Contract Laboratory Program National Functional guidelines for Inorganic Superfund Data Review, OSWER 9240.1-51 USEPA-540-R-10-011, 1/10	Environmental Data Services, Ltd.
% Moisture (solid/tissue)	EDS SOP V-14, Rev.2, 2/10 – Verification/Validation Geotechnical Data	Environmental Data Services, Ltd.
Total Sulfide	EDS SOP V-12 Rev.0, 1/09	Environmental Data Services, Ltd.
Total Phosphorus	USEPA Contract Laboratory Program National Functional guidelines for Inorganic Superfund Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 1/10	Environmental Data Services, Ltd.
Total Kjeidhal Nitrogen	USEPA Contract Laboratory Program National Functional guidelines for Inorganic Superfund Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 1/10	Environmental Data Services, Ltd.
AVS/SEM	USEPA Contract Laboratory Program National Functional guidelines for Inorganic Superfund Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 1/10	Environmental Data Services, Ltd.
Ammonia	USEPA Contract Laboratory Program National Functional guidelines for Inorganic Superfund Data Review, OSWER 9240.1-45 EPA-540-R-10-011, 1/10	Environmental Data Services, Ltd.
Grain Size (solids)	EDS SOP V-14, Rev.2, 2/10 – Verification/Validation Geotechnical Data	Environmental Data Services, Ltd.
% Lipids	Completeness Check	Environmental Data Services, Ltd.
ORP	Completeness Check	Environmental Data Services, Ltd.
Titanium	USEPA Region 2 SOP HW-2b, Rev.15, 12/12	Environmental Data Services, Ltd.

See the last page of Worksheet #36 for a description of footnotes.

QAPP Worksheet #36 (UFP-QAPP Manual Section 5.2.2.1): Validation Guidance Summary Table[a] (continued)

Notes:

[a] All validation and verification reference documents have been provided as appendices to this Crab and Clam Sampling and Analysis Quality Assurance Project Plan.

AVS/SEM = acid volatile sulfide/simultaneously extracted metals
EDS = Environmental Data Services, Ltd.
ORP = oxidation reduction potential
OSWER = Office of Solid Waste and Emergency Response
PCBs = polychlorinated biphenyls
PCDDs/PCDFs = polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
SOP = Standard Operating Procedure
SIM = selective ion monitoring
TAL = Target Analyte List
TEPH = total extractable petroleum hydrocarbons
TOC = total organic carbon
USEPA = U.S. Environmental Protection Agency
% = percent

QAPP Worksheet #37 (UFP-QAPP Manual Section 5.2.3): Usability Assessment

The Data Usability Assessment will be performed by Diane Waldschmidt, Director, Environmental Data Services, Ltd. Note that the Data Usability Assessment will be conducted on validated data. The results of the Data Usability Assessment will be presented in the final project report, which will be provided to U.S. Environmental Protection Agency for review and approval. The following items will be assessed and conclusions drawn based on their results.

Precision

Results of all laboratory and field duplicates will be evaluated based on the measurement performance criteria presented in Worksheets #12-1 through #12-3. A discussion will follow summarizing the results of the laboratory precision. Any conclusions about the precision of the analyses or sample collection techniques will be drawn and any limitations on the use of the data will be described.

Accuracy/Bias Contamination

Results for all laboratory method blanks and instrument blanks, as well as field rinsate equipment and trip blanks will be evaluated. The results for each analyte will be checked against the measurement performance criteria presented in Worksheets #12-1 through #12-3. A discussion will follow summarizing the results of the laboratory and field accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Overall Accuracy/Bias

The results for all matrix spike and surrogate standard spike analyses will be evaluated based on the requirements listed in Worksheets #12-1 through #12-3. A discussion will follow summarizing overall accuracy/bias. Any conclusions about the overall accuracy/bias of the analyses will be drawn and any limitations on the use of the data will be described.

Sensitivity

All analytical results reported will be evaluated to determine if adequate sensitivity was achieved. The results for each analyte will be cross-checked against the quantitation limits presented in Worksheets #15-1 through #15-3. Results for analytes that do not meet project quantitation limit criteria will be summarized. A discussion will follow detailing the results of the laboratory-sensitivity evaluation. Any conclusions about the sensitivity of the analyses will be drawn and any limitations on the use of the data will be described.

Representativeness

Representativeness is achieved through adherence to sampling and analytical procedures described in this Crab and Clam Sampling and Analysis Quality Assurance Project Plan and compliance with stipulated sample holding times. After evaluation of relative compliance with specified procedures and holding times, conclusions about data representativeness will be drawn and any limitations on the use of data will be described.

QAPP Worksheet #37 (UFP-QAPP Manual Section 5.2.3): Usability Assessment (continued)

Comparability

Data comparability will be assessed through evaluation of achieved sample-specific reporting limits, units of measure, and adherence to specified analytical methodologies and field/sample collection standard operating procedures specified in this Crab and Clam Sampling and Analysis Quality Assurance Project Plan. Evaluation of field procedures used will include assessment of the affects of any deviation from the established filed procedures to data usability. After the evaluations are completed, conclusions about data comparability will be drawn and any limitations on the use of data will be described.

Completeness

A completeness check will be done on all of the data generated. Field and analytical completeness criteria are presented in Worksheets #12-1 through #12-3. Field and analytical completeness will be calculated for each analytical group as follows:

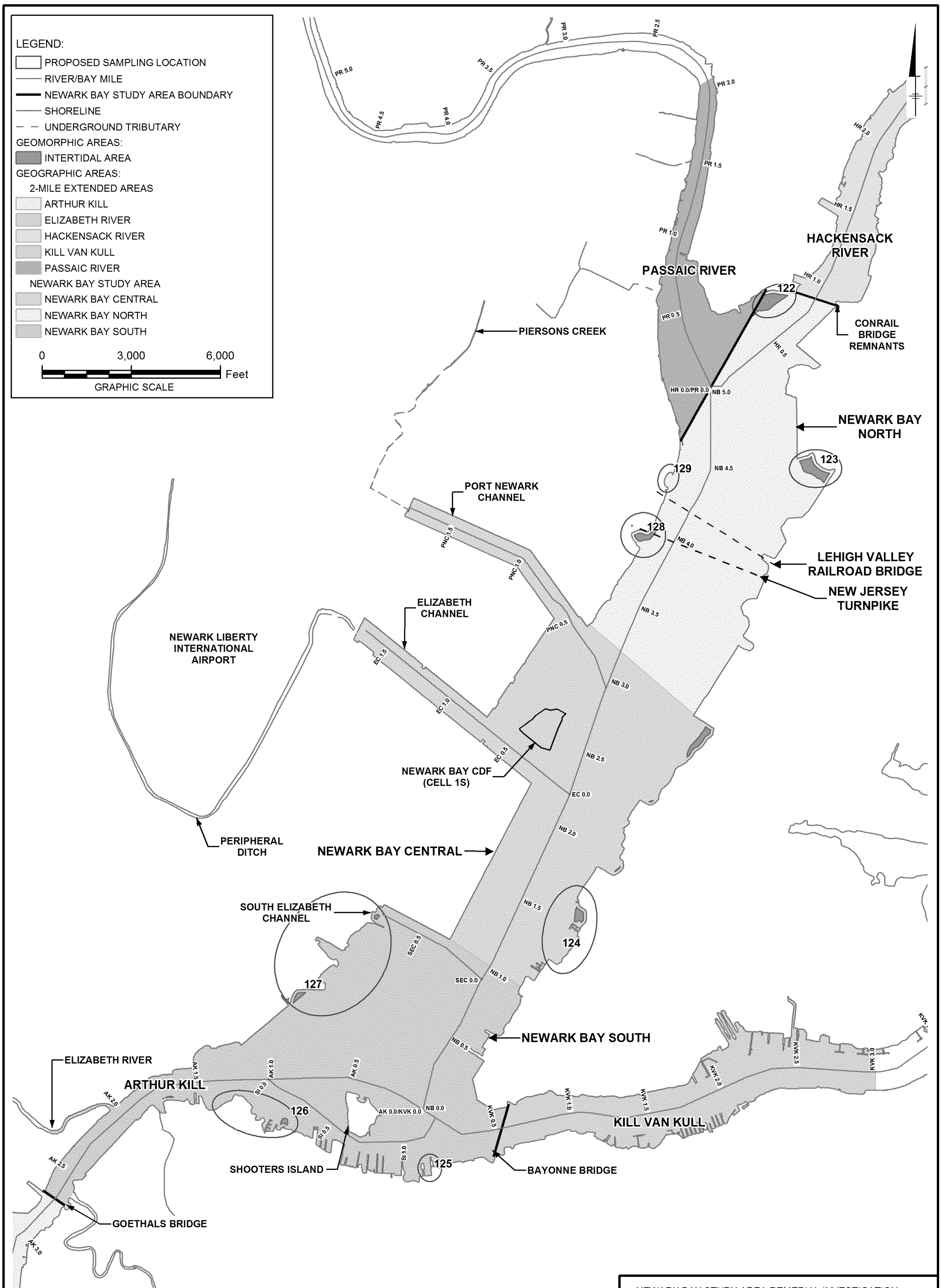
The field completeness will be calculated by the ratio of the number of samples received in acceptable condition by the laboratories to the number of samples planned to be collected as specified in this document. The equation for field completeness is:

$$\%\,Field\ Completeness = \frac{Number\ of\ Samples\ Received\ by\ Laboratories}{Total\ Number\ of\ Samples\ Planned\ to\ be\ Collected}\ x\ 100$$

The analytical completeness will be calculated by the ratio of total valid analytical data results (including estimated values) to the total number of analytical results requested on samples submitted for analysis. Valid analytical data results are defined as those that were not rejected during data validation, due to a significant quality assurance/quality control problem. The equation for analytical completeness is:

$$\%\ Analytical\ Completeness = \frac{Total\ Valid\ Analytical\ Data}{Analytical\ Data\ Obtained}\ x\ 100$$

Figures



NOTES:

1. HORIZONTAL DATUM: NEW JERSEY STATE PLANE COORDINATE SYSTEM, NAD83.
2. SHORELINE IS DIGITIZED FROM AERIAL PHOTO DATED JULY 2002 (INTRASEARCH, ENGLEWOOD, CO).
3. THE INTERTIDAL AND GEOGRAPHIC AREAS SHOWN ARE CONSISTENT WITH THOSE PRESENTED IN THE DEAR, REVISION 1 (TIERRA 2013b).
4. RIVER/BAY MILES CREATED BY DIVIDING RIVER CENTERLINE PROVIDED BY USEPA INTO 0.01-MILE SEGMENTS. HALF-MILE INCREMENTS ARE DISPLAYED ON THIS FIGURE.
5. ABBREVIATIONS ARE AS FOLLOWS:
   AK = ARTHUR KILL
   EC = ELIZABETH CHANNEL
   HR = HACKENSACK RIVER
   KVK = KILL VAN KULL
   NB = NEWARK BAY
   PNC = PORT NEWARK CHANNEL
   PR = PASSAIC RIVER
   SEC = SOUTH ELIZABETH CHANNEL
   SI = SHOOTERS ISLAND
6. THE NEWARK BAY STUDY AREA BOUNDARY PRESENTED WAS USEPA-APPROVED AND UTILIZED IN THE PHASE I AND II SEDIMENT INVESTIGATIONS, BUT MAY BE CHANGED BY USEPA FOR FUTURE INVESTIGATIONS UNDER THE RI/FS.

NEWARK BAY STUDY AREA REMEDIAL INVESTIGATION
CRAB AND CLAM SAMPLING AND ANALYSIS QAPP

PROPOSED CRAB AND CLAM SAMPLING LOCATIONS

OCTOBER 2013
REV 0

FIGURE
1

Appendix A

Data Forms

Composite Sample Form

Project Name: **Project No.:**

Date Composited: **Composited By:**

TISSUE TYPE	COMPOSITE ID	SPECIMEN ID	TISSUE SAMPLE ID	WEIGHT (g ww)

Comments:

Location Data Form

Project Name: **Project No.:**

Field Crew Initials:

Deployment Date & Time	Retrieval Date & Time	Location ID	Trap ID	Collection Method	Coordinates[a]		Depth (ft)
					Easting (X)[b]	Northing (Y)[b]	

[a] Field-observed coordinates.

[b] NAD83 New Jersey State Plane (ft).

Non-Target Species Tally Form

Project Name: **Project No.:**

Field Crew Initials:

Collection Date	Collection Time	Trap ID	Species	Length Min (mm)	Length Max (mm)	Total Weight (g ww)	Count	Comments

Protocol Modification Form

Project Name and Number: _____

Material to be Sampled: _____

Measurement Parameter: _____

_____

Standard Procedure for Field Collection & Laboratory Analysis (cite reference):

_____

_____

_____

_____

_____

Reason for Change in Field Procedure or Analysis Variation: _____

_____

_____

_____

_____

_____

Variation from Field or Analytical Procedure: _____

_____

_____

_____

_____

Special Equipment, Materials or Personnel Required: _____

_____

_____

_____

_____

_____

Initiator's Name: _____ Date: _____

Project Manager: _____ Date: _____

QA Manager: _____ Date: _____

USEPA Authority: _____ Date: _____

Specimen Tally Form

Project Name: **Project No.:**

Species Sampled: **Field Crew Initials:**

Collection Date	Collection Time	Trap ID	Specimen ID No.	Length (mm)	Weight (g)	Gender	Comments

SURFACE SEDIMENT COLLECTION FORM

Project Name: _____ **Project no.:** _____

Date: _____ **Weather:** _____

Sampling Method: _____ **Crew:** _____

GRAB DATA	**Location ID:**			
Latitude/Northing(Y):			**Longitude/Easting(X):**	
Grab time	**Bottom depth (m)**	**Penetration depth (cm)**	**Acceptable grab (Y/N)**	**Comments:**
SAMPLE DATA	**Sample ID:**			
Analyses needed before homogenization (circle): VOC sulfides AVS/SEM Other:				
Sediment type	**Sediment color**	**Sediment odor**		**Comments:** (i.e. redox potential discontinuity, organic matter, wood debris, shell fragments, sheen, fauna, field duplicate, rinsate blank, etc.)
cobble	brown surface	none	H_2S	
gravel	drab olive	slight	petroleum	
sand (F M C)	brown	moderate	other:	
silt	gray	strong		
clay	black			

GRAB DATA	**Location ID:**			
Latitude/Northing(Y):			**Longitude/Easting(X):**	
Grab time	**Bottom depth (m)**	**Penetration depth (cm)**	**Acceptable grab (Y/N)**	**Comments:**
SAMPLE DATA	**Sample ID:**			
Analyses needed before homogenization (circle): VOC sulfides AVS/SEM Other:				
Sediment type	**Sediment color**	**Sediment odor**		**Comments:** (i.e. redox potential discontinuity, organic matter, wood debris, shell fragments, sheen, fauna, field duplicate, rinsate blank, etc.)
cobble	brown surface	none	H_2S	
gravel	drab olive	slight	petroleum	
sand (F M C)	brown	moderate	other:	
silt	gray	strong		
clay	black			

Appendix B

SOP No. 1 – Locating Sample Points Using a Hand-Held Global Positioning System

Standard Operating Procedure No. 1

Locating Sample Points Using Hand-Held Global Positioning System

October 2013

Revision 0

1. Purpose and Scope **3**

2. Definition **3**

3. Equipment and Supplies **3**

4. Field Procedure **4**

5. Quality Control **5**

1. Purpose and Scope

The purpose of this procedure is to provide reference information regarding the collection and documentation of sample coordinates for the Newark Bay Study Area (NBSA) Remedial Investigation/Feasibility Study (RI/FS) using a global positioning system (GPS).

This standard operating procedure (SOP) may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by Tierra Solutions Inc.'s (Tierra's) Facility Coordinator and the United States Environmental Protection Agency Remedial Project Manager and On-Scene Coordinator. The ultimate procedure employed will be documented in the field notebook.

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Definition

GPS provides navigation and positioning information from a constellation of GPS satellites, operated by the United States Department of Defense. The system includes a control station and five monitoring locations that track each satellite. Information received by the monitoring stations is used to calculate satellite orbits and update the information sent to receivers. Satellite signals can be received by any GPS receiver on land or water or in the air. The system incorporates a minimum of 24 satellites, which are positioned around the world, such that six satellites are available at a given location, 24 hours a day. The NBSA RI/FS will use a handheld GPS unit to collect and record sampling location coordinates. The signals received by the hand-held GPS will produce locations with sub-meter accuracy.

3. Equipment and Supplies

The following equipment list contains materials that may be needed in carrying out the procedures contained in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- a hand-held differential global positioning system (DGPS) unit or equivalent model such as the Trimble® ProXH™, with sub-foot accuracy

- an additional DGPS unit with equivalent accuracy as the primary unit (described above) to be carried as a backup to the Trimble® unit in the case of malfunction or

loss (if necessary, the backup GPS unit will only be used temporarily until the primary unit can be replaced or repaired)

- ☐ AA or AAA batteries depending on the device
- ☐ USB port cable to download information

4. Field Procedure

Power on the GPS unit and wait several minutes for the GPS to locate the initial position via satellite. Confirm that the date and time are correct.

Locate the coordinate system information in the main menu and verify the following settings:

1. units = feet
2. coordinate system = New Jersey State Plane (easting and northing)
3. map datum = North American Datum of 1983 (NAD83)
4. north reference = magnetic north (Magnetic north will be used for navigational purposes; however, either magnetic or true north can be used to collect fixed coordinates. The north reference setting will be recorded in the field notebook.)

Confirm that the background map is set to North America. Record the date, time, and all relevant coordinate system information in the field notebook.

Once the unit has acquired the initial position and has indicated that it is ready, follow directions on the GPS to begin collecting sample coordinates.

At each sampling location, allow the GPS to receive satellite data for at least 1 minute before recording the sampling location. A minimum of three satellites is required for a three-dimensional reading, but four satellites are preferred. Save the location information at each sampling location. Record the date, time, and easting/northing (NAD83 New Jersey State Plane) in feet for each location in the field notebook. Readings will be stored in the GPS unit for easy downloading and also to reduce error.

The manufacturer's user's manual will be reviewed and referenced to address technical difficulties and/or malfunctions with the unit.

5. Quality Control

The GPS has quality control features within the system that maintain reliable readings. The GPS will indicate the number of satellites available, the strength of each satellite signal, and will not display coordinates for a given location if there is not a sufficient number of satellites available to take an accurate measurement. The GPS will also make sure that the satellite geometry is able to account for the three-dimensional position. The GPS averages data from satellites over time; therefore, waiting at least 1 minute before recording coordinates at each sampling location will provide a more accurate reading. To confirm the accuracy of the navigation system, a checkpoint will be located at a known point, such as a pier face, dock, piling, or similar structure that is accessible by the sampling vessel. At the beginning and end of each day, the vessel will be stationed at the check point, a GPS position reading will be taken, and the reading will be compared with the known land-survey coordinates. The two position readings should agree, within the limits of survey vessel operational mobility, to within 1 foot.

Appendix C

SOP No. 2 - Positioning

Standard Operating Procedure
No. 2

Positioning

October 2013

Revision 0

1. Purpose and Scope **3**

2. Procedures **3**

2.1 Equipment List 3

2.2 Positioning Vessel 4

2.2.1 Establishing Position at a Location 5

2.2.2 Adjusting Position 6

2.3 Calibration, Maintenance, and Use of Field Instruments 6

3. Quality Assurance **7**

4. Documentation **7**

5. References **7**

1. Purpose and Scope

The purpose of this document is to define the standard operating procedure (SOP) for positioning vessels for the Newark Bay Study Area Crab and Clam Sampling and Analysis Quality Assurance Project Plan (Crab/Clam QAPP). Positioning will be conducted to locate the vessel(s) and sample locations with sufficient accuracy and precision to meet project objectives during sampling activities.

This SOP describes the equipment, field procedures, materials, and documentation procedures necessary to position vessels and locate sampling locations. Specific information regarding proposed sampling locations is provided in the Crab/Clam QAPP.

This SOP may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by Tierra Solutions Inc.'s (Tierra's) Facility Coordinator and the United States Environmental Protection Agency Remedial Project Manager and On-Scene Coordinator. The ultimate procedure employed will be documented in the field notebook.

Other SOPs will be utilized with this procedure, including:

- SOP No. 10 – Documenting Field Activities.

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Procedures

Sampling activities will be conducted from a vessel or on land, depending on tidal stage and water depth. In accordance with the procedures outlined below, the vessels must be properly positioned and their position recorded before each activity can begin.

2.1 Equipment List

The following equipment list contains materials that may be needed in carrying out the procedures contained in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- personal protective equipment and other safety equipment, as required by the Health and Safety/Contingency Plan, Rev. 1 (Tierra 2007)

- ☐ vessel(s) adequate for Newark Bay conditions
- ☐ 25-watt marine VHF radio
- ☐ navigation charts and sample location figure
- ☐ differential global positioning system (DGPS) receivers (x2) with an accuracy of ±1 foot
- ☐ DGPS external antennas (x2)
- ☐ equipment user manuals
- ☐ table of target sampling location coordinates
- ☐ assorted nautical equipment (e.g., anchors, lines, personal flotation devices)
- ☐ field notebook
- ☐ Sample collection forms
- ☐ permanent marker or grease pencil.

2.2 Positioning Vessel

This section gives the step-by-step procedures for vessel positioning. Observations made during vessel positioning should be recorded in the logbook, as appropriate.

A DGPS will be used to establish locations during implementation of activities specified in the Crab/Clam QAPP. Two DGPS units will be required: one on board the vessel with a receiving antenna to be aligned with the deployment of the sampling apparatus, and the other at a known fixed location (monument or temporary benchmark) to provide corrections to the standard GPS signal.

While this SOP provides general guidance and procedural steps, personnel performing positioning activities also should follow the appropriate sections of the equipment user's manuals and have the manuals available for reference at all times.

The following procedures describe the steps to establish position at a location, as well as the steps to adjust the positioning for collection of additional samples.

2.2.1 Establishing Position at a Location

Preliminary Activities

1. Obtain the appropriate sample collection form(s). Complete the Daily Activity Log provided in SOP No. 10 – Documenting Field Activities.

2. Obtain the target sampling locations. For Phase III activities, these locations will have been selected prior to commencement of field activities, as described in the Crab/Clam QAPP. The location of each target sampling location will be established in the New Jersey State Plane Coordinate System with respect to the North American Datum of 1983.

3. Enter coordinates for the locations into the DGPS unit that will be on board the vessel as a waypoint.

Field Activities

1. Establish a DGPS base station over a shore-based marker prior to sampling operations. The operation and horizontal/vertical accuracy of the vessel-mounted DGPS will be verified at another shore-based marker by recording observed horizontal and vertical (XYZ) data and comparing these data to the published XYZ data for a given point. After initial DGPS system verification, a temporary benchmark may be established at a location convenient to the vessel to facilitate daily DGPS system performance verification. DGPS system performance verification will be conducted twice per day and documented in the field notebook and vessel data logger. The horizontal and vertical accuracy will be compared to shore-based markers to verify performance. Elevations will be recorded in North American Vertical Datum of 1988 with an accuracy of +/- 1 foot.

2. Verify receiving antenna is properly aligned with the sampling device.

3. Identify and approach actual sampling locations by using data from the DGPS unit in the navigation mode. The navigation mode provides information on heading, distance remaining, and time remaining. This information is based on the selected waypoint location and the present location of the vessel.

4. Anchor the vessel adjacent to the planned location, if desired.

5. Once the vessel is on location and secured, note the coordinates from the DGPS unit and check the coordinates to verify that the vessel is within the pre-determined range of the target location. For the initial attempt, confirm that the

coordinates are within 5 feet of the target. (For subsequent attempts, see Section 2.2.2 below.) If not acceptable, adjust the vessel's location and recheck the position. Repeat this process until the vessel's position is within acceptable range of the target. Record the final coordinates on the appropriate form.

6. Once the coordinates are acceptable, perform sampling activity at the location. Record final location coordinates on the appropriate form. Plot locations onto a master chart or use computer-based, real-time software to verify location.

7. At the end of the sampling day, check the data loaded onto the DGPS units to verify the existence of locations where data were collected.

2.2.2 Adjusting Position

The following steps will be used to adjust position for sampling activities:

1. Move vessel 10 feet from the initial sampling location and within a 50-foot radius of the target coordinates.

2. Check the coordinates to verify that the vessel is within 50 feet of the target coordinate and note coordinates on the Collection Form.

3. Once the coordinates are acceptable and recorded, collect samples. Record the final location.

4. Repeat Steps 1 through 3 until the appropriate number of samples is collected.

2.3 Calibration, Maintenance, and Use of Field Instruments

Prior to use, the DGPS units will be inspected in accordance with the Crab/Clam QAPP. DGPS units will be calibrated in accordance with the Crab/Clam QAPP, appropriate sections of the equipment user's manual, and as described in Section 2.2 of this SOP. Maintenance and use of DGPS units should follow the appropriate sections of the equipment user's manual. Field personnel will have the manual available for reference. Equipment inspection and maintenance will be recorded in the equipment maintenance log presented in Worksheet 22 of the Crab/Clam QAPP.

Despite virtually worldwide, 24-hour coverage, technical difficulties with GPS satellites can still occur. In the event of system-wide or other long-term problems with GPS (e.g., satellite failures), vessel positioning will be achieved using land-based methods. If a land-based method is selected, an SOP will be developed for its use.

3. Quality Assurance

For the Phase III Program, quality assurance (QA) activities for positioning procedures include verification of the sampling location by comparing the target coordinates specified in the Crab/Clam QAPP with coordinates entered into the DGPS, by plotting the coordinates on a master chart, and by performing the daily check with shore-based markers.

4. Documentation

Detailed positioning data will be recorded on the Sample Collection Forms provided in SOP No. 10 – Field Documentation. In addition, the following information will be recorded in a field notebook (at a minimum):

- notes on breaking position during sampling
- equipment calibration information
- summary of vessel activities.

5. References

Tierra. 2007. Newark Bay Study Area Remedial Investigation Work Plan [Rev. 1]. Volume 2 Health and Safety/Contingency Plan. September.

Appendix D

SOP No. 6 – Crab Collection

Standard Operating Procedure
No. 6

Crab Collection

October 2013

Revision 0

1. Purpose and Scope **3**

2. Procedures **3**

2.1 Equipment List 4

2.2 Collection Procedures 5

3. Quality Assurance/Quality Control **7**

4. Documentation **7**

5. References **8**

1. Purpose and Scope

The purpose of this document is to define the standard operating procedure (SOP) for collecting blue crab (*Callinectes sapidus*) in the Newark Bay Study Area (NBSA). .

This SOP provides descriptions of methodologies to be used for collection of blue crab for analysis. Blue crab samples will be analyzed for constituents as outlined in the Crab/Clam Sampling Quality Assurance Project Plan (Crab/Clam QAPP).

This SOP may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. The ultimate procedure employed will be documented in the field notebook.

Other SOPs will be utilized with this procedure, including:

- SOP No. 1 – Locating Sample Points Using Hand-Held Global Positioning System
- SOP No. 2 – Vessel Positioning
- SOP No. 3 – Decontamination
- SOP No. 5 – Containers, Preservation, Handling, and Tracking of Samples for Analysis
- SOP No. 10 – Documenting Field Activities.

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Procedures

Crab collection activities at the site will include the use of crab pots. The target species is blue crab (*Callinectes sapidus*), based on the survey information established during the September 2013 ARCADIS (2013) Reconnaissance Survey. All hard shell blue crabs kept for analysis will be at least the minimum legal catch size of 4.5 inches (measured as carapace width), per New Jersey Department of Environmental Protection (NJDEP) fishing regulations. To meet the target muscle tissue sample mass requirement of approximately 232 grams live weight, approximately 25 hard shell crabs will be targeted for collection at each location. The individual live muscle tissue weight is based on the estimates from studies conducted previously found in Tierra (2002) and Windward (2010). Using the minimum legal size of 4.5 inches for the carapace width and the tissue formula from Tierra (2002) and Windward (2010), an

estimate of approximately 11.5 grams per individual is calculated. At one location a quality assurance and quality control (QA/QC) laboratory sample (i.e., MS/MSD) will be collected. This QA/QC sample will require double the target tissue mass (approximately 464 grams) or approximately 50 crabs. Ideally, samples will have equal numbers of similar-sized male and female crabs. Estimates for hepatopancreas tissue are not well correlated to individual size. It is anticipated that hepatopancreas composites will be made up of multiple sampling locations within each of the two target zones (i.e., North and South) of the NBSA. Collection efforts will follow the general procedures described in this section.

2.1 Equipment List

The following list includes equipment and materials that may be needed to perform the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment not listed here may be required dependent on field conditions.

- ☐ personal protective equipment and other safety equipment, as required by the Health and Safety/ContingencyPlan, Rev. 1 (Tierra 2007)
- ☐ sampling vessel adequate for Newark Bay conditions
- ☐ station location map
- ☐ crab pot with dimensions meeting NJDEP fishing regulations[1]
- ☐ buoys (fluorescent and labeled with study information or Scientific Collector's Permit license information)
- ☐ weights for crab pots (5 to 10 pounds)
- ☐ bait (cut fish and/or chicken)

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[1] Crab pots will be a cube or rectangular shape no larger than 30 inches on a side with inward openings for the entrance of crabs. The material the pot is constructed of will have mesh not less than 1 inch across measured at its longest access. The openings into the interior of the pot will be oval and not larger than 7 inches wide and 4 inches high.

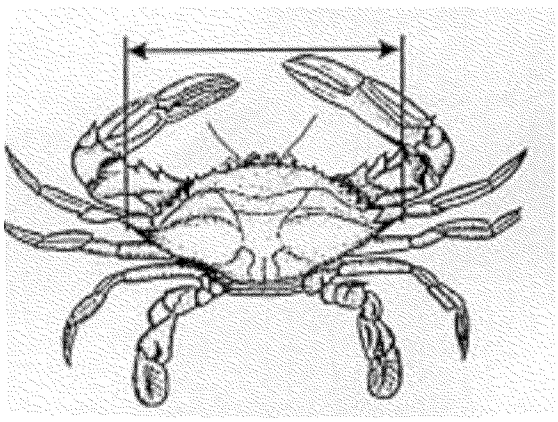
- 5 gallon bucket
- field data sheets
- fluorescent or reflective paint or tape
- shellfish license (or collection permit)
- field notebook
- crab gauge or ruler
- small hammer
- electronic scale
- large sealable Ziploc® bags
- laboratory-supplied sample coolers
- ice
- sample labels
- appropriate packaging materials and forms
- global positioning system (GPS).

2.2 Collection Procedures

Crabs will be collected as follows:

- Locate sample location according to SOPs No 1. – Locating Sample Points Using Hand-Held Global Positioning System and No. 2 – Vessel Positioning.
- Crab pots will be visibly marked with a buoy. The buoy will have a permanent identification tag bearing the license/collection permit number. The buoy will be marked with fluorescent/reflective paint or tape.
- A minimum of three crab pots will be placed at each intertidal station as displayed on the sampling station location figure in the Crab/Clam QAPP.

- Verify the crab pots have been decontaminated in accordance with SOP No. 3 – Decontamination, using the soap and water method.
- Upon arrival at target sampling locations, crab pots will be baited and checked to confirm they are working properly.
- Weights will be attached to crab pots prior to submersion to confirm that they rest on substrate and do not float.
- Crab pots will be checked at least once every 12 hours.
- Crab pots will be retrieved manually and placed on deck. Crabs will be removed from the pots and placed in a 5 gallon bucket.
- Use a crab gauge or ruler to measure the carapace length. Crabs that have any extensive damage to carapace (i.e., cracks or holes) will not be retained. Non-target species, as well as blue crabs not meeting NJDEP minimum size requirements, will be released. Minimum size requirements for blue crabs, as measured from point to point of the carapace, are as follows: peeler/shredder is 3 inches, soft shells are 3.5 inches, and hard shells are 4.5 inches.
- It is assumed that each crab sample will include at least 25 crabs.
- Crabs that are retained will be measured (carapace length, see inset below), weighed, sexed, and examined for any discoloration, abnormalities, or lesions. Photographs will be taken of any observed anomalies. All information will be recorded in the field notebook.



Blue crabs are measured by point to point of the carapace.

- Retained crabs will be quickly euthanized with a sharp blow to the area below the eyestalks on the underside of the shell using a clean instrument (e.g., a small hammer).

- Crabs will be placed in a labeled, sealable plastic bag. The bags will be kept on ice in a laboratory-supplied cooler.

- Crabs will be shipped overnight on ice in the coolers to the laboratory. Shipping and handling procedures are presented in SOP No. 5 – Containers, Preservation, Handling, and Tracking of Samples for Analysis.

3. Quality Assurance/Quality Control

The following quality assurance/quality control procedures will be performed during the collection events:

- An ecologist will make/confirm observations regarding habitat type, tide, water depth, and substrate type.

- Confirm all pertinent sample collection information is recorded in the field notebook and verify field sample identification label matches this information.

- Times of photographs and related observations will be noted in the field notebook.

Station locations in the NBSA will be marked both on maps in the field, as well as registered in a GPS unit.

4. Documentation

Documentation of crab collection activities will be conducted in accordance with SOP No. 10 – Documentation of Field Activities. Field personnel are responsible for documenting field activities related to the crab collection events. Observations and data will be recorded with ink in a field notebook with consecutively numbered pages. Information to be recorded in the field notebook will include, at a minimum:

- responsible person's name

- dates and times of activities

- identification and size and weight of each retained crab

- station locations

- information about each photograph (i.e., date, time, location).

5. References

Tierra Solutions. 2002. Passaic River Study Area fish community data. September 18, 2002. Tierra Solutions, Inc., Newark, NJ.

Tierra. 2007. Newark Bay Study Area Remedial Investigation Work Plan [Rev. 1]. Volume 2 Health and Safety/Contingency Plan. September.

Windward. 2010. Revised Sample Analysis Plan for Blue Crab Tissue for the Lower Passaic River Restoration Project Memorandum. February 8.

Appendix E

SOP No. 7 – Clam Collection

Standard Operating Procedure
No. 7

Clam Collection

October 2013

Revision 0

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1. Purpose and Scope

The purpose of this document is to define the standard operating procedure (SOP) for conducting bivalve clam collection in the Newark Bay Study Area (NBSA). This SOP gives descriptions of methodologies to be used for collection of clams for analysis.

This SOP may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Other SOPs will be utilized with this procedure, including:

- SOP No. 1 – Locating Sample Points Using Hand-Held Global Positioning System
- SOP No. 3 – Decontamination
- SOP No. 5 – Containers, Preservation, Handling, and Tracking of Samples for Analysis
- SOP No. 10 – Documentation of Field Activities.

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Procedures

Clam collection activities at the site will include the use of a basic clam shovel. Clams will be composited by species. The target species is softshell clams (*Mya arenaria*), based on the presence/dominance of bivalve clams established during the September 2013 Reconnaissance Survey. The target size range for clam samples is at least 1.5 inches, per New Jersey Department of Environmental Protection shell fishing regulations. To meet the target tissue sample mass requirement of approximately 232 grams live weight, approximately 20 softshell clams will be targeted for collection at each location. The individual live weight is based on the estimates from aquaculture studies for the softshell clam presented in Beal (2002). Using the minimum legal size of 1.5 inches for the soft shell clam and the Beal (2002) formula, an estimate of approximately 16 grams per individual is calculated. At one location a quality assurance and quality control (QAQC) laboratory sample (i.e., MS/MSD) will be collected. This QA/QC sample will require double the target tissue mass (approximately 464 grams) or approximately 40 softshell clams. Collection efforts will follow the general procedures described in this section.

2.1 Equipment List

The following list contains equipment and materials that may be needed to perform the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required depending on field conditions.

- ☐ personal protective equipment and other safety equipment, as required by the Health and Safety/ContingencyPlan, Rev. 1 (Tierra 2007)
- ☐ collection location map
- ☐ clam shovel
- ☐ bucket
- ☐ brush
- ☐ ruler or gauge
- ☐ electronic scale
- ☐ field data sheets
- ☐ fishing license (collection permit)
- ☐ field notebook
- ☐ field or shellfish guide
- ☐ large Ziploc® bags
- ☐ mesh bags
- ☐ laboratory-supplied cooler
- ☐ ice
- ☐ sample labels
- ☐ appropriate packaging materials and forms

- global positioning system (GPS)

2.2 Collection Procedures

Clam collection will be conducted as follows:

- Verify the clam shovel has been decontaminated in accordance with SOP No. 3 – Decontamination, using the soap and water method.

- Locate sample location according to SOPs No 1. – Locating Sample Points Using Hand-Held Global Positioning System.

- Start at the GPS coordinate and work outward to collect clams.

- Collect clams by use of a clam shovel. Look for holes and other signs of clams. When the presence of a clam is observed, place the shovel adjacent to the clam location, dig down and remove the clam.

- Verify the clam species using a field or shellfish guide. Use a ruler or clam gauge to measure the target species (softshell clam) from the anterior to posterior edge (longest axis) to see if the clam meets the minimum 1.5 inch legal size requirement. Non-target species, including clams not meeting minimum legal size requirements (1.5 inches), will be released.

- Clams that are retained (assumed to be at least 20 clams per sample) will be rinsed with ambient water and lightly scrubbed with a brush to remove sediment. Upon cleaning each individual, they will be placed in a labeled (sample location, collection time, number of individuals) mesh bag and placed on ice in a laboratory-supplied sample cooler.

- A photograph will be taken of each collection location, along with a photograph of the entire batch of collected clams. Information such as date, time, location (total spatial area for collection, measured in length and width of collection plot), GPS coordinates for edges of collection plot, collector, collection method, number of clams and species, and weather conditions will be recorded in the field notebook.

- At a secure location (e.g., staging area at the marina), each clam will be weighed and measured, and the information recorded in a field notebook.

- Clams will be placed in a labeled, sealable Ziploc® bag on ice in a laboratory-supplied cooler. All clams that will be composited together to form one sample will be placed together in the same Ziploc® bag. Clams will be composited by species.

- Clams will be shipped overnight to the analytical laboratory on wet ice in accordance with the shipping SOP No. 5 – Containers, Preservation, Handling, and Tracking of Samples for Analysis.

3. Quality Assurance/Quality Control

The following quality assurance/quality control procedures will be performed during the collection events:

- An ecologist will make/confirm observations regarding habitat types, tide, water depth, and substrate type.

- Confirm all pertinent sample collection information is recorded in the field notebook and verify field sample identification label matches this information. Times of photographs and related observations will also be noted in the field notebook.

- Collection locations in the NBSA will be marked both on maps in the field, as well as registered in a GPS unit.

4. Documentation

Documentation of clam collection activities will be conducted in accordance with SOP No. 10 – Documentation of Field Activities. Field personnel are responsible for documenting field activities related to the clam collection events. Observations and data will be recorded with ink in a field notebook with consecutively numbered pages. Information to be recorded in the notebook will include, at a minimum:

- responsible person's name

- dates and times of activities

- identification and size and weight of each clam

- collection locations

- information about each photograph (i.e., date, time, location).

5. References

Beal, B.F. 2002. Adding value to live, commercial size soft-shell clams (Mya arenaria L.) in Maine, USA: results from repeated, small-scale, field impoundment trials. Aquaculture 210: 119–135.

Tierra. 2007. Newark Bay Study Area Remedial Investigation Work Plan [Rev. 1]. Volume 2 Health and Safety/Contingency Plan. September.

Appendix F

SOP No. 9 – Sediment Sample Collection

Standard Operating Procedure
No. 9

Sediment Sample Collection

October 2013

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6. References **7**

1. Scope and Application

This standard operating procedure (SOP) describes the collection and field screening, logging, and subsequent sampling of sediment. The general procedures to be utilized in obtaining sediment samples are outlined below. This SOP should be followed for any collecting, processing, logging, and sampling of sediment.

This SOP may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by Tierra Solutions Inc.'s (Tierra's) Facility Coordinator and the United States Environmental Protection Agency Remedial Project Manager and On-Scene Coordinator. The ultimate procedure employed will be documented in field notebook.

Other SOPs will be utilized with this procedure, including:

- SOP No. 1 – Locating Sample Points Using Hand-Held Global Positioning System
- SOP No. 2 – Positioning
- SOP No. 3 – Decontamination
- SOP No. 4 – Management and Disposal of Residuals
- SOP No. 5 – Containers, Preservation, Handling, and Tracking of Samples for Analysis
- SOP No. 10 – Documenting Field Activities.

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Procedure

2.1. Equipment List

The following equipment will be needed during sediment sampling activities:

- personal protective equipment and other safety equipment, as required by the Health and Safety/ContingencyPlan, Rev. 1 (Tierra 2007)
- sampling vessel adequate for Newark Bay conditions

- ❑ aluminum foil pan or stainless steel bowl
- ❑ duct and packing tape
- ❑ Lexan® tubing with end caps
- ❑ 1-quart and 1-gallon freezer bags
- ❑ tape measure
- ❑ global positioning system (GPS)
- ❑ trowel
- ❑ hand auger
- ❑ garbage bags
- ❑ photoionization detector (PID)
- ❑ transport container with ice
- ❑ appropriate sample containers, labels, and forms
- ❑ field notebook
- ❑ erasable whiteboard
- ❑ digital camera.

2.2 Sediment Sampling

1. Don personal protective equipment (PPE), as required by the HASP.
2. Navigate to the sampling location in accordance with SOPs No 1. – Locating Sample Points Using Hand-Held Global Positioning System and No. 2 – Positioning.
3. Record sample location with the GPS.
4. Record the proposed sample location in the field notebook, including other appropriate information collected during sediment probing activities. Location,

date, time of collection, and description or surrounding area, as well as with a photograph will be recorded for each sample.

5. Based on the location conditions, determine which sampling method will be used. For areas saturated and/or submerged in water, a push core will be used. For areas that are dry, a hand auger and/or trowel will be used to collect the sample.

   a. Push Core

      i. Verify the sampler has been decontaminated in accordance with SOP No. 3 – Decontamination.

      ii. At each sample location, lower a section of Lexan® tube until it reaches the top of sediment. Measure the depth of water.

      iii. Push the Lexan® tube by hand 6 inches into the sediment or until refusal is encountered. Measure the depth of sediment.

      iv. Seal top of core tube to retain capillary pressure within barrel. Slowly pull the tube from the sediment. Before the bottom of the coring tube breaks the water surface, place a cap over the bottom to prevent the loss of material from the core tube. Place the cap on the core tube by reaching down into the water.

      v. Document appearance and recovery of the sample to confirm acceptability of the sample.

      vi. Pump the standing surface water from above the recovered sediment, being careful not to disturb the sediment-water interface.

      vii. Document the sampler penetration depth and recovery.

   b. Hand Auger/Trowel

      i. Verify the sampler has been decontaminated in accordance SOP No. 3 – Decontamination.

      ii. Use a trowel, spoon, or hand auger to collect a sample from the surface to 6 inches below the surface.

      iii. Use a tape measure to verify that the sample is collected to the correct depth and record the top and bottom depths from the ground surface.

iv. To prevent the hole from collapsing, it may be necessary to remove a wider section from the surface or use polyvinyl chloride (PVC) tubing or pipe to maintain the opening.

6. Place the sample in an aluminum foil pan or stainless steel bowl.

7. Immediately collect sediment for analysis of volatile compounds (e.g., volatile organic compounds [VOCs]). Collect these samples prior to screening or logging steps.

8. Collect a small aliquot (approximately 1 ounce [oz]) of sediment representative of the sediment sample and place into a zip-top bag. Perform PID headspace screening as follows:

   a. Allow sample to equilibrate to an ambient temperature for approximately 15 minutes.

   b. Insert 'sniffer' of the PID into the zip-top bag and read PID until value spikes and then stabilizes.

   c. Record the peak value displayed by the PID.

   d. The aliquot of sediment used for headspace testing will not be sent for laboratory analysis.

9. Photo-document the sample to provide reference for post-processing questions regarding descriptions of color/staining general texture, recovery, etc. Photos will include the sample identification, date, and time. The photo will also include a view of a tape measure for scale.

10. Describe sediment sample according to Unified Soil Classification System (USCS) and document any other observations, such as, but not limited to, color, type of organic materials present, odor[1], sheen, and staining.

11. Sediment samples will be homogenized in disposable aluminum foil pans or stainless steel bowls, prior to placement into proper laboratory jars.

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[1] Sediment should not be smelled intentionally, due to health and safety concerns. However, if an odor is observed during processing activities, it should be recorded.

12. Samples will be packaged and shipped overnight to the analytical laboratory on wet ice in accordance with the shipping SOP No. 5 – Containers, Preservation, Handling, and Tracking of Samples for Analysis.

3. Waste Management

Investigative-derived waste (IDW) generated during the sediment sampling activities and disposable equipment will be transported for off-site disposal in accordance with SOP No. 4 – Management and Disposal of Residuals.

4. Quality Assurance

Field documentation, data reporting requirements, sampling handling, custody requirements, packing, handling, and shipping requirements, including laboratory custody procedures, analytical methods, quality control requirements, and laboratory specific quality assurance/quality control requirements are stated in the Crab and Clam Sampling and Analysis Quality Assurance Project Plan (Crab/Clam QAPP).

5. Documentation

Documentation of sample handling activities will be conducted in accordance with SOP No. 10 – Documenting Field Activities. Upon collection of sediment samples, the sediment sample should be logged in the field notebook.

Digital photographs of typical sediment types observed at the site and any unusual features should be obtained whenever possible. All photographs should include a ruler or common object for scale. Photo location, depth, and orientation must be recorded in the daily log or field notebook, and a label showing this information in the photo is useful.

6. References

Tierra. 2007. Newark Bay Study Area Remedial Investigation Work Plan [Rev. 1]. Volume 2 Health and Safety/Contingency Plan. September.

Appendix G

SOP No. 3 – Decontamination

Standard Operating Procedure
No. 3

Decontamination

October 2013

Revision 0

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5. References **10**

1. Purpose and Scope

The purpose of this document is to define the standard operating procedure (SOP) for decontamination of equipment, instruments, and other materials used during implementation of the Newark Bay Study Area Crab/Clam Sampling Quality Assurance Project Plan (Crab/Clam QAPP). Decontamination is the process of neutralizing, washing, and rinsing exposed surfaces of equipment to minimize the potential for contaminant migration and/or cross-contamination. This procedure does not apply to personnel decontamination, which is described in the Health and Safety/Contingency Plan (HASCP), Rev. 1 (Tierra Solutions, Inc. [Tierra] 2007).

This SOP may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by Tierra's Facility Coordinator and the United States Environmental Protection Agency (USEPA) Remedial Project Manager and On-Scene Coordinator. The ultimate procedure employed will be documented in the field notebook.

Other SOPs will be utilized with this procedure, including:

- SOP No. 4– Management and Disposal of Residuals
- SOP No. 5 – Containers, Preservation, Handling, and Tracking of Samples for Analysis
- SOP No. 6 – Crab Sampling
- SOP No. 7 – Clam Sampling
- SOP No. 9 – Sediment Sample Collection
- SOP No. 10 – Documenting Field Activities.

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Procedures

2.1 Equipment List

The following equipment list contains materials that may be needed in carrying out the procedures contained in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- personal protective equipment (PPE) and other safety equipment, as required by the HASCP, Rev. 1 (Tierra 2007)
- bristle brushes
- wash/rinse tubs
- low phosphate detergent
- 10% nitric acid, ultrapure
- acetone, methanol, and hexane (pesticide grade or better in separate Teflon® bottles), as necessary
- deionized "analyte-free" water
- stainless steel bowls
- aluminum foil
- tap water (from any treated municipal water supply)
- high-pressure/steam cleaner
- sample container(s) for rinsate blank, if collected
- field notebook.

2.2 Sample Equipment and Decontamination

Sampling equipment (including newly purchased equipment) that comes into contact with the media to be sampled will be decontaminated prior to use in the field to eliminate or minimize cross-contamination. The frequency of decontamination is provided in SOP No. 6 – Crab Sampling, SOP No. 7 – Clam Sampling, and SOP No. 9

– Sediment Sample Collection. Sufficient decontaminated equipment must be available to be dedicated to the sampling locations planned for each day. Equipment will be decontaminated in the area designated for decontamination.

The decontamination procedure followed by USEPA Region 2 (USEPA 1989) – with the exception of the second tap water rinse being replaced with a deionized "analyte-free" water rinse – will be used prior to each sampling event for equipment that will come into contact with the media to be sampled. The USEPA Region 2 procedures are summarized below:

1. Wash and scrub with low phosphate detergent.

2. Rinse with tap water.

3. Rinse with 10% nitric acid (HNO_3), ultrapure.

4. Rinse with deionized "analyte-free" water.

5. Spray or rinse with acetone only or a methanol followed by hexane spray or rinse (solvents must be pesticide grade or better).

6. Rinse thoroughly with deionized ("analyte-free") water.

7. Air dry.

8. Wrap in aluminum foil, shiny side out, for temporary storage and transport.

Sampling equipment will be decontaminated as described in Section 2.2.1 below. Decontamination of the sampling equipment will be sufficient for collecting samples for the analysis of dioxin/furan congeners and homologues (PCDDs/PCDFs), including all other analytes, and will consist of a methanol rinse followed by a hexane rinse as described in Section 2.2.1.

Solvents used during decontamination activities will be collected and handled in accordance with residuals management procedures outlined in SOP No. 4 – Management and Disposal of Residuals.

Not all sampling equipment will require the full decontamination procedures listed in the USEPA Region 2 Comprehensive Environmental Response, Compensation, and Liability Act Quality Assurance Manual. Three levels of decontamination (i.e., solvent, soap and water, or ambient [Newark Bay] water decontamination) will be performed based on the usage of the sampling equipment as defined below.

2.2.1 Decontamination with Solvents

The following steps will be used to decontaminate small sampling equipment that will come into contact with sediment designated for chemical analysis (e.g., stainless steel trowels, spoons and bowls, Lexan® tube, and plastic caps for the core tubes):

- Personnel will dress in suitable PPE to reduce exposure to chemicals and contaminants Phase II RIWP HASCP Rev. 1 (Tierra, 2007).
- Residual sample media at the coring location (i.e., on the vessel) will be scraped off and the equipment rinsed with Newark Bay water.
- Residual sample media on equipment at the sample processing site will be scraped off and collected according to residuals management procedures outlined in SOP No. 4 – Management and Disposal of Residuals.
- Equipment will be placed in a wash tub or bucket containing Alconox (or other low-phosphate detergent) along with tap water, and scrubbed with a bristle brush or similar utensil. Equipment will be rinsed with tap water in a second wash tub or bucket, followed by a nitric acid rinse (for metals analyses), a deionized "analyte-free" water rinse, a methanol rinse followed by a hexane rinse (for organic analyses), and lastly with a deionized water rinse. Rinses shall utilize sufficient amounts of solvent/water to flush rather than just wet the surface. The volume of deionized water used during the rinse must be at least five times the volume of solvent used.
- Following decontamination, equipment will be placed in a clean area and allowed to air dry. Following air drying, the equipment will be wrapped in aluminum foil, shiny side out, until used for sample collection.
- Used decontamination water will be collected and handled in accordance with residuals management procedures outlined in SOP No. 7 – Management and Disposal of Residuals.

2.2.2 Decontamination with Soap and Water

The following steps will be used to decontaminate equipment that is not intended to collect samples for chemical analysis:

- Personnel will dress in suitable PPE to reduce exposure to contaminants as discussed in the HASCP, Rev. 1 (Tierra 2007).

- Residual sample media at the sampling location (i.e., on the vessel) will be scraped off and the equipment rinsed with Newark Bay water.

- Residual sample media on equipment at the sample processing site will be scraped off and collected according to residuals management procedures outlined in SOP No. 4 – Management and Disposal of Residuals.

- Equipment will be placed in a wash tub or bucket containing Alconox (or other low phosphate detergent) and tap water, and scrubbed with a bristle brush or similar utensil. Equipment will be rinsed with tap water in a second wash tub or bucket, and then rinsed again.

- Following decontamination, equipment will be placed in a dedicated clean area.

- Rinse water and detergent water will be replaced frequently. Used decontamination water will be collected and handled in accordance with residuals management procedures outlined in SOP No. 4 – Management and Disposal of Residuals.

2.2.3 Decontamination with Ambient (Newark Bay) Water

The following steps will be used to decontaminate sampling and support vessels, vessel anchors, lines, ropes, submersible pump and hose (not intended for sample collection), and buoy marker weights:

- Personnel will dress in suitable PPE to reduce exposure to contaminants (HASCP, Rev. 1 [Tierra 2007]).

- Equipment will be rinsed with Newark Bay water onboard the sampling vessel.

- Rinse water will not be contained.

Daily decontamination of the decks of the vessels will consist of a Newark Bay water washing as soon as possible after concluding work. Further wash-down with tap water at the marina is at the discretion of the boat's captain.

2.3 Field Instruments and Equipment

Instrumentation should be cleaned according to the manufacturer's instructions. Care will be taken to prevent damage to equipment. When possible, instruments that are difficult to decontaminate, such as cameras and data logging instruments, may be protectively wrapped to reduce or eliminate the need for decontamination.

2.4 Other Equipment Decontamination

Other sampling equipment that might be used that has had direct contact with sediments or wastes will be decontaminated at a designated area prior to leaving the site. The decontamination procedure will be as follows:

- Equipment will be wrapped or draped in plastic or placed in the plastic-lined cargo area of a truck for transport to the area designated for decontamination.
- Equipment will first be washed with a hot water, high-pressure spray or steam-cleaned.
- Equipment will then be rinsed, by hose or high pressure spray, with tap water.
- Wash and rinse water will be collected and handled in accordance with residuals management procedures outlined in SOP No. 4 – Management and Disposal of Residuals.

2.5 Equipment Leaving Newark Bay

Equipment leaving the site upon the completion of on-site investigation activities will be decontaminated according to Sections 2.2, 2.3, or 2.4 above.

2.6 Collection of Rinsate Blank

Rinsate blanks will be collected to assess the adequacy of equipment decontamination procedures. Rinsate blanks are submitted for testing each day a decontamination event is carried out (not to exceed one per day). Following sample collection and processing, all pieces of equipment (both used and unused) belonging to the equipment set will be decontaminated during the decontamination event. One set of equipment will be used throughout the sample collection and processing activities to confirm the rinsate blank corresponds to one set of samples (e.g., a given stainless steel bowl and spoon will be considered a set and will be used together throughout the sample processing activities).

The rinsate blank collection procedures for all analytes, except dioxin/furan analysis, are as follows:

- Pour analyte-free water over a representative set of sampling equipment (e.g., stainless steel bowl, stainless steel pan) after it has been decontaminated.

- Collect rinsate in a previously decontaminated stainless steel bowl and transfer rinsate to sample bottles for analysis.

- Preserve rinsate sample, as necessary, in accordance with SOP No. 5 – Containers, Preservatives, Handling, and Tracking of Samples for Analysis.

For the purposes of the Phase III Program, rinsate blank samples for dioxin/furan analysis will be collected using hexane, rather than spectrophotometric-grade trichloroethene (TCE) as specified by USEPA Region 2 (USEPA1989). As TCE is a potential target analyte for this RI Program, hexane was substituted to eliminate the potential for obtaining erroneous positive TCE results in collected field samples. Rinsate blank collection procedures for dioxin/furan analysis will be as follows:

- Following collection of the rinsate blank samples for all other analytes, pour hexane over a representative set of sampling equipment (e.g., stainless steel bowl, stainless steel pan) after it has been decontaminated.

- Collect rinsate in a previously decontaminated stainless steel bowl and transfer rinsate to sample bottles for analysis with other sediment samples.

- Preserve rinsate sample, as necessary, in accordance with SOP 5 – Containers, Preservatives, Handling, and Tracking of Samples for Analysis.

3. Quality Assurance

Decontamination quality assurance/quality control procedures described in Section 2.6 will be performed to assess the adequacy of equipment decontamination procedures. Rinsate blanks will be collected at the frequency specified in Worksheet No 28 of the Crab/Clam QAPP.

4. Documentation

Field personnel are responsible for documenting decontamination activities related to their on-site activities in accordance with SOP No. 10 – Documenting Field Activities. In addition to this, the following information should also be included in the field notebook (at a minimum):

- Information concerning items decontaminated and the procedure utilized.

- Information related to the collection of rinsate blank samples.

5. References

Tierra. 2007. Newark Bay Study Area Remedial Investigation Work Plan [Rev. 1]. Volume 2 Health and Safety/Contingency Plan. September.

USEPA. 1989. Comprehensive Environmental Response, Compensation, and Liability Act [CERCLA] Quality Assurance Manual, Revision 1. October.

Appendix H

SOP No. 4 – Management and Disposal of Residuals

Standard Operating Procedure No. 4

Management and Disposal of Residuals

October 2013

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1. Purpose and Scope

The purpose of this document is to define the standard operating procedure (SOP) for disposal of sediment, water, personal protective equipment (PPE), and other potentially contaminated materials generated during Newark Bay Study Area Crab and Clam Sampling and Analysis Quality Assurance Project Plan (Crab/Clam QAPP) operations.

This SOP provides procedures for handling potentially contaminated sediment, water, PPE, and other materials during coring and sampling activities through their ultimate disposal. Specific information regarding handling and disposal of residuals is provided in the Crab/Clam QAPP.

This SOP may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modifications to this SOP will be approved in advance by Tierra Solutions Inc.'s (Tierra's) Facility Coordinator and the United States Environmental Protection Agency (USEPA) Remedial Project Manager and On-Scene Coordinator. The ultimate procedure employed will be documented in the field notebook.

Other SOPs will be utilized in conjunction with this SOP, including:

- SOP No. 3 – Decontamination
- SOP No. 10 – Documenting Field Activities.

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Procedures

Potentially contaminated sediment, water, PPE, and other materials will be classified into three categories: 1) solid materials consisting of sediments, sediment samples returned from the laboratory, used core tubes, used PPE, and other materials used in the handling, processing, and storage of sediment (addressed in Section 2.2); 2) liquid wastes, such as wastewater, decontamination water, and aqueous samples returned from the laboratory (addressed in Section 2.3); and 3) spent and residual chemicals (liquids) from decontamination (addressed in Section 2.3). Sediment from samples that is not processed for chemical analysis may be either archived or disposed, and will be segregated and handled separately according to its classification. To the extent practical, liquids generated during sampling/decontamination operations should be

separated from the solid material. Each type of material should be handled in the manner described in this SOP.

2.1 Equipment List

The following equipment list contains materials that may be needed to carry out the procedures contained in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- ☐ PPE or other safety equipment, as required by the Health and Safety/Contingency Plan, Rev. 1 (Tierra 2007)
- ☐ 55-gallon open-top drums (Department of Transportation- [DOT-] approved) with lid
- ☐ 30-gallon (minimum) garbage bags
- ☐ permanent marking pens and/or paint pens
- ☐ labels
- ☐ duct tape
- ☐ storage racks
- ☐ small (cooler-size) storage containers
- ☐ walk-in cooler or refrigerated trailer
- ☐ chemical storage cabinet (meeting Occupational Safety and Health Administration and National Fire Protection Association Code 30 specifications/Factory Manual approved)
- ☐ field notebook
- ☐ indelible ink pens.

2.2 Solid Materials

2.2.1 Solid Residuals for Disposal

Solid residuals generated during field activities will be characterized for appropriate off-site disposal. Characterization procedures will be completed in accordance with the Waste Characterization QAPP (Tierra 2011). Solid residuals consist of two types of materials: non-sediment solid materials generated during the collection and processing of sediment, crab, and clam samples (e.g., used core tubes, aluminum foil, and PPE [e.g., gloves, Tyvek® suits, boot covers]) and sediment solid materials (i.e., left-over sediment not used for analyses including waste sediment and residual sediment). Non-sediment and sediment wastes will be segregated and temporarily stored in separate containers pending disposal. Loose sediment will be removed from non-sediment waste items prior to disposal and stored with sediment waste.

If recovered sediment is determined to be unusable, the sediment will be stored in an appropriate container for disposal as sediment waste. Sediment solid material will be placed in 55-gallon drums, labeled, and stored temporarily until disposal.

Non-sediment solid materials will be placed in 55-gallon drums, bulk bags, and/or a roll-off container and stored temporarily pending characterization and off-site disposal in accordance with the February 2006 IDW Management Plan (including any subsequent USEPA-approved modifications). All drums and bags containing solids residuals will be labeled and handled as described in Section 2.4 of this SOP.

2.3 Liquid Wastes

2.3.1 Waste Water

Waste water will be generated during sample collection and decontamination activities. Water mixed with detergent or chemicals will be treated and handled at the Lister Avenue groundwater treatment plant. Water from gross decontamination (e.g., to wash sediment from sampling equipment) will be allowed to flow overboard on the vessel or will be treated and handled at the Lister Avenue groundwater treatment plant. Sediment recovered during this process will be handled according to Section 2.2.1 of this SOP.

In the event that waste water is not treated through the Lister Avenue groundwater treatment plant, it will be stored at the Lister Avenue Site in appropriate containers pending characterization for off-site disposal. Modifications to the February 2006 IDW Management Plan will be submitted to the USEPA for review and approval for characterization and off-site disposal.

2.3.2 Chemical Liquid Wastes

Spent solvents, acids, and other residual chemicals generated during the decontamination process (SOP No. 3 – Decontamination) will be collected and treated through the Lister Avenue groundwater treatment plant in accordance with procedures outlined in the February 2006 IDW Management Plan.

In the event that these liquids are not treated through the Lister Avenue groundwater treatment plant, they will be stored at the Lister Avenue site in appropriate containers pending characterization for off-site disposal. Modifications to the February 2006 IDW Management Plan will be submitted to the USEPA for review and approval for characterization and off-site disposal.

2.4 Handling and Tracking of Solid Materials and Containers

As they are generated during field activities, waste sediment and other solid waste materials will be placed in DOT-approved 55-gallon drums or 30-gallon bags. Solid waste materials that are initially placed in bags may be bulked into 55-gallon drums for storage. The following procedures will be followed for storing sediment and other solid waste in these drums:

- A drum number will be assigned to each drum by the Field Supervisor or designee. The drum number will be clearly marked on multiple places on the drum.
- A log will be kept for each drum, listing the materials placed in that drum. All solid materials will be segregated based on the type of material (e.g., sediment, coring tubes, PPE, waste plastic, paper, foil) and, to the extent practicable, by where they were generated (e.g., location within Newark Bay).
- Drums will be closed or covered at the end of the day's work.
- Collection drums may be reused at the processing facility after emptying.
- Drums containing solid materials will be stored in a secured temporary facility until proper off-site disposal can be coordinated.

2.5 Handling and Tracking of Waste Water and Chemical Liquid Wastes and Containers

As they are generated during field activities, waste water and chemical liquid wastes will be treated directly through the on-site Lister Avenue groundwater treatment plant

and/or will be placed in separate DOT-approved 55-gallon drums. The following procedures will be followed for treating the liquids through the on-site groundwater treatment plant:

- A sump will be isolated in the processing area to collect and handle liquids.
- Liquids collected in the sump will be combined and treated with the liquids treated at the on-site groundwater treatment plant.
- Treated liquids will be monitored, characterized, and disposed in accordance with the requirements for the on-site groundwater treatment plant.

The following procedures will be followed for storing waste water and chemical liquid wastes in drums:

- A separate drum will be used for each non-commingled chemical. Another, separate drum will be used for chemicals and/or water that have been mixed.
- A drum number will be assigned to each drum by the Field Supervisor or designee. The drum number will be clearly marked on multiple places on the drum.
- A log will be kept for each drum, listing the materials placed in that drum.
- All drums will be closed or covered at the end of the day's work.
- Collection drums may be reused at the Sample Processing Area after emptying.
- Drums containing wastewater and chemical liquid wastes will be stored in a secured temporary facility until proper off-site disposal can be coordinated upon the completion of the sampling event.

2.6 Samples Returned from Off-Site Laboratories

Upon completion of the required chemical analyses, remaining sample material and sample containers from the laboratory will be returned to the Sample Processing Area. Returned sample material/containers will be transported under chain of custody procedures, and remain in custody until disposal. Upon receipt, the chain of custody form will be signed and the samples will be logged in by a project staff member. The approximate volume of sample material and the condition of the containers in which the samples are returned will be checked and recorded in a field notebook.

Samples will be separated into sediment and aqueous sample groups; empty sample containers will be grouped accordingly by sample matrix. Sediment samples will be placed in a DOT-approved 55-gallon drum and will be characterized and disposed off site in accordance with the procedures outlined in the February 2006 IDW Management Plan (including any subsequent USEPA-approved modifications).

Aqueous samples returned from the analytical laboratories will be treated through the Lister Avenue groundwater treatment plan in accordance with procedures outlined in the February 2006 IDW Management Plan (including any subsequent USEPA-approved modifications). In the event that these liquids are not treated through the Lister Avenue groundwater treatment plant, they will be stored at the Lister Avenue site in appropriate containers pending characterization for off-site disposal. Modifications to the February 2006 IDW Management Plan will be submitted to the USEPA for review and approval for characterization and off-site disposal.

3. Quality Assurance

Disposal procedures will be documented in a field notebook to confirm that disposal activities are conducted in accordance with the procedures outlined in the SOPs. Waste manifests will be obtained for solid and aqueous waste disposal to verify that proper transportation and disposal of these materials has occurred in accordance with the February 2006 IDW Management Plan (including any subsequent USEPA-approved modifications).

4. Documentation

Documentation of sample handling activities will be conducted in accordance with SOP No. 10 – Documenting Field Activities. In addition, the following information should be included in the field notebook (at a minimum):

- name of person performing residual management or disposal activities
- date and time of activity
- information coordinating container numbers for drums or bags containing solid materials with sample numbers, core boring numbers, or origin
- information coordinating origin of waste liquid (water or chemical[s]) with specific waste drum or tank

5. References

Tierra, 2006. Newark Bay Study Area Remedial Investigation Work Plan Sediment Investigation Program Investigation Derived Waste Management Plan [Rev. 0]. February.

Tierra. 2007. Newark Bay Study Area Remedial Investigation Work Plan [Rev. 1]. Volume 2 Health and Safety/Contingency Plan. September.

Tierra. 2011. Final Waste Characterization Quality Assurance Project Plan, Diamond Alkali Superfund Site, Operable Unit 1/CERCLA Non-Time-Critical Removal Action – Lower Passaic River Study Area and Newark Bay Study Area, Revision 1, December 2011.

Appendix I

SOP No. 5 – Containers, Preservation, Handling, and Tracking of Samples for Analysis

Standard Operating Procedure No. 5

Containers, Preservation, Handling, and Tracking of Samples for Analysis

October 2013

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Pre-Printed Sample Label

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1. Purpose and Scope

The purpose of this document is to define the standard operating procedure (SOP) for containerizing, preserving, handling, tracking, and shipping samples collected as part of the Newark Bay Study Area (NBSA) Crab/Clam Sampling Quality Assurance Project Plan (Crab/Clam QAPP). Samples may include sediment collected or generated for chemical analysis, radiochemical analysis, and associated quality assurance (QA) analysis. This SOP is intended to be complete enough so that: 1) the steps that could affect tracking, documentation, or integrity of samples are explained in sufficient detail and 2) different sampling personnel following these procedures will deliver samples to the laboratory that are equally reliable and consistent, and in compliance with regulatory agency requirements. Specific information regarding sample collection and analysis is found in the Crab/Clam QAPP.

This SOP may change depending on field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by Tierra Solutions, Inc. (Tierra) Facility Coordinator and the United States Environmental Protection Agency (USEPA) Remedial Project Manager and On-Scene Coordinator. The ultimate procedure employed will be documented in the field notebook.

Other SOPs will be utilized with this procedure, including:

- SOP No. 4 – Management and Disposal of Residuals
- SOP No. 10 – Documenting Field Activities.

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Procedures

2.1 Equipment List

The following equipment list contains materials that may be needed in carrying out the procedures contained in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, depending field conditions.

- personal protective equipment and other safety equipment, as required by the Health and Safety/ContingencyPlan, Rev. 1 (Tierra 2007)

- ☐ inert packing material (e.g., foam peanuts, vermiculite, cardboard)
- ☐ colorimetric pH test paper
- ☐ nitric acid (HNO_3) and pipette
- ☐ sample containers as specified in the Crab/Clam QAPP
- ☐ sample labels
- ☐ chain of custody
- ☐ Sample Delivery Group (SDG) Tracking Logs
- ☐ ice chest(s)
- ☐ custody seals
- ☐ indelible marking pens
- ☐ shipping tape
- ☐ sealable plastic bags
- ☐ temperature blanks (if not provided by the laboratory)
- ☐ field notebook
- ☐ ice or similar chilling source
- ☐ potassium iodide starch paper
- ☐ spatula
- ☐ sodium hydroxide (NaOH) and pipette
- ☐ plastic lining material
- ☐ clear tape.

2.2 Sample Identification and Labeling

2.2.1 Sample Identification Code

The standard sample identification number will consist of a unique 13-character string used to identify each sample collected and submitted to the laboratories for analysis, as follows:

Characters 1 and 2:	Two characters to describe the water body where the sample was collected (i.e., for Phase III this will be NB for Newark Bay).
Characters 3 and 4:	Two digits to describe the phase during which the sample was collected. For example, the Phase III SI Program will be described as “03.”
Characters 5, 6, and 7:	Three characters to describe the sample matrix. For example, this will be “SED” for sediment, “CRB” for crab, and “CLM” for clam. Duplicate samples will be identified in a logbook and transferred to the database.
Characters 8, 9, and 10:	A three-digit number to describe the sample collection location (sequentially numbered from 001 to 999). Note that core numbering for the Phase II Program ended at 121. For the Phase III Program, sediment sample locations will begin with 122.

Location Identifier

Location IDs are pre-assigned and are found in the Crab/Clam QAPP. The location ID and location information (coordinates) will be recorded (see SOP No. 10 – Documenting Field Activities).

Example of Sample Identification Code

Following are some examples of sediment and tissue identification numbers:

NB03SED122

Explanation:

Sample collected from Newark Bay, Phase III Program, sediment matrix, sample location 122

NB03CLM123

Explanation:

Sample collected from Newark Bay, Phase III Program, clam tissue, sample location 123

NB03CRB124

Explanation:

Sample collected from Newark Bay, Phase III Program, crab tissue, sample location 124

2.2.2 Sample Delivery Group Number Identification Code

The standard SDG number will consist of a unique six-character string used to identify each SDG submitted to the laboratories for analysis, as follows:

Characters 1 and 2:	Two characters to describe the Site. For the Phase III Program, this will be “NB” for Newark Bay.
Character 3:	One digit to describe the phase during which the sample was collected. For example, the Phase III Program will be described as “3.”
Characters 4 through 6:	A three-digit number to describe the SDG.

Following is an example of an SDG number:

NB3003

Explanation:

The samples in this SDG were collected from Newark Bay during the Phase III Program. The SDG was the third SDG submitted to the laboratory for analysis.

2.2.3 Quality Assurance Sample Identification Code

2.2.3.1 Field Duplicates

Field duplicates will be labeled by a unique 12-character string, as follows:

Characters 1 and 2:	Two characters to describe the site. For the Newark Bay Study Area Remedial Investigation sampling activities, "NB" will be used.
Characters 3 and 4:	Two digits to describe the phase during which the sample was collected. For example, the Phase III Program will be described as "03."
Characters 5, 6, and 7:	Three characters to describe the sample matrix. For example, this will be "SED" for sediment.
Characters 8, 9, and 10:	Three characters to signify a field duplicate. For the Phase III Program, this will be "DUP." Duplicate samples will be identified in a logbook and transferred to the database.
Characters 11 and 12:	A two- or three-digit number (proceeded by a hyphen) to describe the order in which field duplicates are collected within the project, sequentially numbered according to the following:

Following is an example of a field duplicate:

NB03SEDDUP-03

Explanation:

The sample was the third field duplicate collected for sediment during Phase III of the Newark Bay Study Area Remedial Investigation.

2.2.3.2 Rinsate Blanks

Rinsate blank samples will be labeled by a unique eight-character string. The first six characters will be the rinsate blank number (denoted sequentially from the beginning of the project). The last two characters will be "RB" for rinsate blank.

Following is an example of an SDG number:

NB3003RB

Explanation:

This rinsate blank was the third collected during the Phase III Program.

2.2.3.3 Trip Blanks

Trip blanks will be numbered by a unique 12-character string, as follows:

Characters 1 and 2:	Two characters to signify a trip blank. For the Phase III Program, these will be "TB."
Characters 3 through 10:	Eight characters to describe the date using a two-digit month, two-digit day, and four-digit year (e.g., MMDDYYYY).
Characters 11 and 12:	A two-digit number (preceded by a hyphen) to describe the order in which trip blanks were collected that day. Each cooler with sediment samples for volatile organic compound (VOC) analysis will contain a trip blank for VOC analyses.

The first trip blank will be "01" and will increase sequentially through the day.

Following in an example of a trip blank:

TB01012013-02

Explanation:

This sample is the second trip blank collected on January 1, 2013.

2.2.4 Sample Labeling

A label will be attached to each container used for sampling. An example of a preprinted sample label is attached to this SOP. When practical, the project number, sample matrix, laboratory designation, and sample identification code will be typed or printed onto the label before sampling. Once affixed to the sample container, the label will be protected from water and solvents with clear packing tape.

2.3 Sample Containers and Preservation

2.3.1 Sample Containers

To confirm that the appropriate sample quantities are collected in certified, pre-cleaned containers, sample containers for this project will be supplied from commercial suppliers or laboratories. Sample containers will be cleaned to the quality control standard defined in USEPA Office of Solid Waste and Emergency Response (OSWER) Directive #9240.0-05A.

Certification of sample container quality per the OSWER directive will be kept in the Newark Bay project file. The Crab/Clam QAPP summarizes container types that will be provided for samples collected.

Prior to use, the sample containers will be visually inspected for cracks, chips, or other damage. Damaged sample containers will not be used and will be disposed in the proper waste receptacle.

2.3.2 Sample Preservation

The contracted laboratory performing the analysis will provide certified, pre-cleaned containers for sediment and surface water containing a pre-determined amount of the required preservative(s) for rinsate blanks, as appropriate. In cases where field adjustment of pH is necessary, the procedures outlined below will be followed for the appropriate analysis.

When possible, the rinsate blank sample containers will be pre-preserved by the laboratory. The specific preservatives to be used for each chemical analysis are summarized in the Crab/Clam QAPP. Documentation of equipment and methods used in preservation and field-adjustment of pH will be maintained in a field notebook. The chemicals and amounts used will be recorded. If refrigeration is necessary, samples will be placed on ice after collection, and shipping containers will be packed with additional ice, if needed, prior to shipment via overnight carrier.

2.3.2.1 pH for Rinsate Blanks for Cyanide Analysis

Aqueous rinsate blank sample bottles for cyanide analysis will be pre-preserved with NaOH. Immediately following sample collection, the pH of the preserved sample will be determined and adjusted, if necessary, using the following procedure:

1. Close the bottle and gently invert it several times to mix the preservative with the sample.

2. Pour a small aliquot (a few drops) of the sample into a separate vial.

3. Test the aliquot in the vial with colorimetric pH paper appropriate to the pH being tested. If the pH of the sample is less than 12, increase the pH of the rinsate blank by adding 50% NaOH. Using a pipette, add 0.2 milliliters (mL) (four to five drops) of NaOH to the sample.

4. Close the bottle and gently invert it several times to mix the preservative with the sample.

5. Pour a small aliquot (a few drops) of the sample into a separate vial.

6. Repeat this process until the correct pH (greater than 12) is achieved. The aliquots used for testing the pH will be disposed in accordance with SOP No. 4 – Management and Disposal of Residuals. The amount, type, and procedures will be documented in the field notebook in accordance with SOP No. 10 – Documenting Field Activities.

2.3.2.2 pH for Rinsate Blanks for Metals Analysis

Aqueous rinsate blank sample bottles for metals analysis will be preserved with HNO_3. Immediately following sample collection, the pH of the preserved sample will be determined and adjusted, if necessary, using the following procedure:

1. Close the bottle and gently invert it several times to mix the preservative with the sample.

2. Pour a small aliquot (a few drops) of the sample into a separate vial.

3. Test the aliquot in the vial with colorimetric pH paper appropriate to the pH being tested. If the pH of the sample is greater than 2, lower the pH of the rinsate blank by adding HNO_3. Using a pipette, add 0.2 mL (four to five drops) of HNO_3 to the sample.

4. Close the bottle and gently invert it several times to mix the preservative with the sample.

5. Pour a small aliquot (a few drops) of the sample into a separate vial.

6. Repeat this process until the correct pH (less than 2) is achieved.

The separate aliquots used for testing the pH will be disposed in accordance with SOP No. 4 – Management and Disposal of Residuals. The amount, type, and procedures will be documented in the field notebook in accordance with SOP No. 10 – Documenting Field Activities.

2.3.2.3 pH for Rinsate Blanks for Total Organic Carbon Analysis

Aqueous rinsate blank sample bottles for total organic carbon analysis will be preserved with H_2SO_4. Immediately following sample collection, the pH of the preserved sample will be determined and adjusted, if necessary, using the following procedure:

1. Close the bottle and gently invert it several times to mix the preservative with the sample.

2. Pour a small aliquot (a few drops) of the sample into a separate vial.

3. Test the aliquot in the vial with colorimetric pH paper appropriate to the pH being tested. If the pH of the sample is greater than 2, lower the pH of the rinsate blank by adding H_2SO_4. Using a pipette, add 0.2 mL (four to five drops) of H_2SO_4 to the sample.

4. Close the bottle and gently invert it several times to mix the preservative with the sample.

5. Pour a small aliquot (a few drops) of the sample into a separate vial.

6. Repeat this process until the correct pH (less than 2) is achieved.

The separate aliquots used for testing the pH will be disposed in accordance with SOP No. 4 – Management and Disposal of Residuals. The amount, type, and procedures will be documented in the field notebook in accordance with SOP No. 10 – Documenting Field Activities.

2.3.2.4 pH for Rinsate Blanks for Total Extractable Petroleum Hydrocarbons Analysis

Aqueous rinsate blank sample bottles for total extractable petroleum hydrocarbons analysis will be preserved with HCl. Immediately following sample collection, the pH of the preserved sample will be determined and adjusted, if necessary, using the following procedure:

1. Close the bottle and gently invert it several times to mix the preservative with the sample.

2. Pour a small aliquot (a few drops) of the sample into a separate vial.

3. Test the aliquot in the vial with colorimetric pH paper appropriate to the pH being tested. If the pH of the sample is greater than 2, lower the pH of the rinsate blank by adding HCl. Using a pipette, add 0.2 mL (4 to 5 drops) of HCl to the sample.

4. Close the bottle and gently invert it several times to mix the preservative with the sample.

5. Pour a small aliquot (a few drops) of the sample into a separate vial.

6. Repeat this process until the correct pH (less than 2) is achieved.

The separate aliquots used for testing the pH will be disposed in accordance with SOP No. 4 – Management and Disposal of Residuals. The amount, type, and procedures will be documented in the field notebook in accordance with SOP No. 10 – Documenting Field Activities.

2.3.2.5 Preparation of pH for Rinsate Blanks for Volatile Organic Carbon Analysis

Aqueous rinsate blank sample bottles for VOC analysis will be pre-preserved with sufficient HCl to lower the rinsate blank below a pH of 2. Rinsate blanks for VOC analyses will be prepared using the following procedure:

1. Slowly pour rinsate blank into pre-prepared VOC vial until a meniscus is formed on the top of the vial.

2. Carefully cap the VOC vial and gently invert the vial to check for air bubbles.

3. Repeat process until no air bubbles are present. Dispose VOC samples with air bubbles in accordance with SOP No. 4 – Management and Disposal of Residuals.

2.4 Sample Handling and Shipping

Sample packaging and shipping will be done in accordance with applicable regulations, as described below.

1. After filling a sample container, affix cap and securely seal with clear tape (except for samples to be analyzed for VOCs) and complete the sample label. Apply the label to the sample container and cover with clear tape.

2. Clean the outside of each sample container by wiping it off with a clean paper towel. Verify that residual sediment has been removed from the outside of the container and from the area under and around the cap.

3. Seal each sample container inside a sealable plastic bag. Samples for VOC analysis will be packaged together in a sealed plastic bag.

4. Place samples on ice or similar chilling source immediately after collection.

5. Transfer the samples to a plastic-lined ice chest that will be used as a shipping container. Use inert packaging material (e.g., cardboard, vermiculite) to cushion the samples and minimize the potential for breakage. Seal the drains on the ice chest (if present) with shipping tape or plug the drains with silicone sealant or a similar inert substance.

6. Place a trip blank for VOCs analysis in each cooler containing sediment samples for VOCs analysis.

7. Ice chests will contain ice or similar chilling sources sufficient to maintain a temperature of 4° Celsius inside the cooler during transport. Use sufficient ice to accommodate reasonable delays in shipment. A temperature blank provided by the analytical laboratory with each cooler will be included in the shipment.

8. Complete sample tracking documentation as described in Section 2.5 of this SOP, and place the documents in a sealable plastic bag inside the ice chest, taped to the inside of the lid. Prior to sealing for shipment, check the list of samples against the container contents to verify the presence of each sample listed on the chain of custody.

9. Secure chest lid with shipping tape by covering the entire seal with tape. Complete information on the custody seal and affix the custody seal over the taped seal. An example of a custody seal is attached to this SOP.

10. For ice chests containing rinsate blanks for dioxin/furan analysis, affix flammable liquids labels to the outside of chest and carefully complete the dangerous goods declaration form, as required by the overnight carrier.

11. Transport the shipping container directly to the laboratory, the laboratory courier, or to the overnight carrier for overnight delivery. Samples will be shipped by close of the same day the sample was collected.

2.5 Sample Tracking

From the time of collection through transportation, the handling of samples will follow chain of custody procedures.

A sample is considered under the sampler's custody if one or more of the criteria are met:

- sample is in the sampler's possession
- sample is in the sampler's view after being in sampler's possession
- sample was in the sampler's possession and then locked up to prevent tampering
- sample is in a designated secure area.

Samples collected for analysis will be continuously tracked in the Sample Processing Area and while in transit to the laboratory by use of the following procedures below. The Sample Processing Area will be secured (locked) with limited access.

- Individual sample containers will be properly labeled and securely sealed before being placed in the container for shipment to the laboratory.
- Pertinent information will be entered on the chain of custody form in the field (see attached chain of custody form and form key). Assignment of the SDG number, matrix spike/matrix spike duplicate (MS/MSD) assignments, and the analyses requested for each sample will be made on both the SDG Tracking Log (see attached SDG Tracking Form) and the chain of custody form.
- The chain of custody form must include the following, as required by guidance in SW-846, Test Methods for Evaluating Solid Waste (USEPA 1993): 1) project name; 2) signatures of samplers; 3) sample number, date and time of collection, and grab or composite sample designation; 4) signatures of individuals involved in sample transfer; and 5) if applicable, the air bill or other shipping number.
- The completed chain of custody form will be signed, dated, enclosed in a sealable plastic bag with a copy of the SDG Tracking Log and placed in the container prior

to shipment. A copy of both documents will be retained by field personnel and stored in a dedicated binder. Additional copies will be distributed as follows:

- a copy will be faxed or emailed to the Facility Coordinator or the Facility Coordinator's designee
- a copy will be faxed or emailed to the data validator
- a copy will be faxed or emailed to the laboratory manager/client service representative at each laboratory being used.

- Samples will be considered in the sampler's custody while in his/her possession or within sight, or locked in a secure area prior to shipment. If the person packing the container and verifying the sample list is different than the sampler, both the sampler and the packer will sign the chain of custody form.

- Upon receipt at the laboratory, the designated laboratory sample custodian will sign the chain of custody form indicating receipt of the incoming field samples. The samples will be checked against the chain of custody form upon arrival at the laboratory. The receiving personnel will enter all arriving samples into a laboratory logbook. Any discrepancies between the samples and the chain of custody form(s), or any evidence of tampering with the shipping container or the custody seal will be immediately reported to the Facility Coordinator. The sample custodian will immediately check the temperature of the cooler upon arrival at the laboratory and record the measured temperature on the chain of custody form and in a laboratory logbook.

- A completed copy of the chain of custody form will be distributed to the following individuals on the day of sample receipt at the laboratory:

  - a copy will be faxed or emailed to the Facility Coordinator or the Facility Coordinator's designee
  - a copy will be faxed or emailed to the data validator
  - a copy will be faxed or emailed to the field office.

The original will be retained by the laboratory's sample custodian.

3. Documentation

3.1 Field Notes

Documentation of sample handling activities will be conducted in accordance with SOP No. 10 – Documenting Field Activities. The following information should also be included in the logbook (at a minimum):

- sample IDs collected on that day
- brief synopsis of types of equipment and methods used in collecting the samples
- details regarding the field adjustment of preservatives, if necessary.

3.2 Chain of Custody Documentation

Samples will be tracked through chain of custody documentation as described in Section 2.5 of this SOP.

4. References

Tierra. 2007. Newark Bay Study Area Remedial Investigation Work Plan [Rev. 1]. Volume 2 Health and Safety/Contingency Plan. September.

USEPA. 1993. SW-846, Test Methods for Evaluating Solid Waste, Third Ed., including Promulgated Update I, Chapter One.

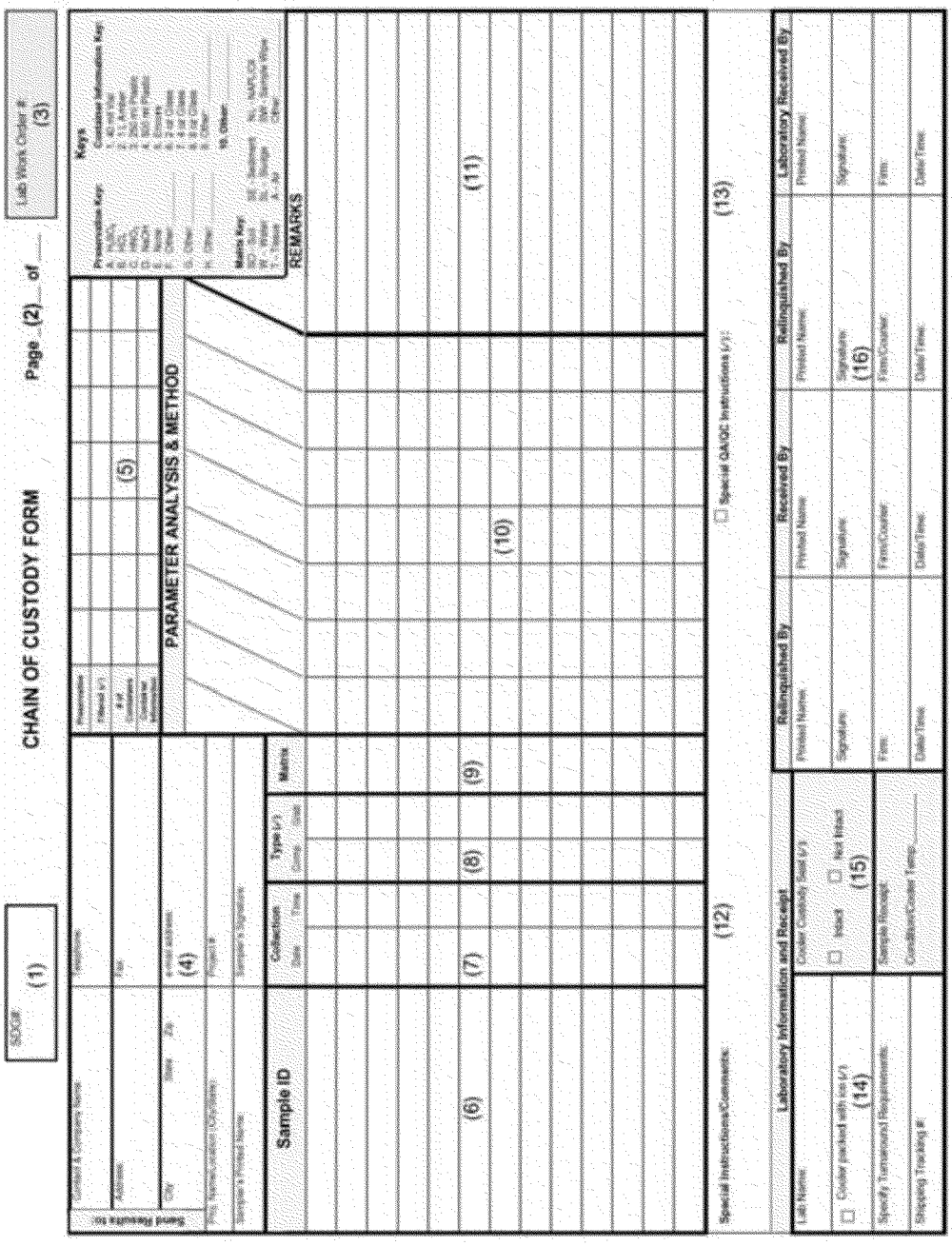
PRE-PRINTED SAMPLE LABEL

	PROJECT #: (1) PROJECT NAME: Newark Bay Phase III SI Program
SDG #: (2)	
SAMPLE #: (3)	GEOMORPHIC AREA: (4)
TIME SAMPLE COLLECTED: (5)	TIME SAMPLE COLLECTED: (6)
LABORATORY: (7)	
SAMPLE MATRIX: (8)	
ANALYSES REQUIRED: (9)	
PRESERVATIVE: (10)	
SAMPLER: (11)	
REMARKS: (12)	

Key:

(1) Company-specific project number, if appropriate
(2) SDG number
(3) Sample number (e.g., NB03SED122)
(4) Geomorphic area (e.g., Sub-tidal Flats)
(5) Date sample was collected from the bulk effluent (e.g., 1/1/2013)
(6) Time sample was collected from the bulk effluent (24-hour format)
(7) Laboratory used for analyses
(8) Sample matrix type (e.g., water)
(9) Analyses required for sample
(10) Preservative(s) used on sample (pre-preserved by the laboratory)
(11) Sampler name
(12) Remarks pertinent to proposed analyses

CHAIN OF CUSTODY FORM



CHAIN OF CUSTODY FORM KEY

(1) SDG number (as described in Section 2.2.2 of this SOP) (e.g., NB3003).

(2) Page number and total number of pages for the set of chain of custody form submitted with the samples for analysis.

(3) Analytical laboratory's internal work order number (to be completed by analytical laboratory).

(4) Address where the analytical results are to be sent, project identifiers (location and internal project numbers), and sampler's signature.

(5) Preservation methods and bottles.

(6) Sample ID (e.g., NB03SED122); refer to Section 2.2.1 of this SOP for sample and QA sample IDs.

(7) Date and time (24-hour format) of sample collection.

(8) Check if sample was a composite sample.

(9) Sample matrix (e.g., water).

(10) Provide analysis and method for which sample is being submitted. Check the appropriate box for which analyses the sample is being submitted.

(11) Provide any pertinent comments regarding the sample submitted for analyses (e.g., not enough sample volume for full analyses).

(12) Provide any special instructions to the analytical laboratory.

(13) Provide any special QA/QC instructions to the analytical laboratory.

(14) Provide details regarding the cooler shipment (analytical laboratory name, whether the cooler was packed with ice, turnaround requirements, and shipping tracking number).

(15) Provide details as to receipt of cooler (to be completed by analytical laboratory on receipt). Indicate if the chain of custody seal was intact and the cooler temperature upon receipt.

(16) Signatures for custody to be completed by sampler and analytical laboratory.

SDG TRACKING LOG

SDG Number _____(1)_____ **SDG Open Date** _____(3)_____

Sample Matrix _____(2)_____ **SDG Close Date** _____(4)_____

Sample #	Sample ID	MS/MSD	Comments
1	(5)	(6)	(7)
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
Rinsate Blank	(8)	N/A	(9)
Trip blank	(10)	N/A	(11)

Notes:
1. The SDG must not exceed 20 field samples. Rinsate blanks do not count towards the sample total. Check which of the 20 samples have been collected to include extra volume for MS/MSD and assign as such.
2. Three times the weights listed should be collected for laboratory QC (i.e., MS/MSD/internal laboratory duplicate).
3. Field duplicate is a separate sample, not to be confused with “internal lab duplicate.”

SDG TRACKING LOG KEY

(1) SDG number (as described in Section 2.2.2 of this SOP) (e.g., NB3003).

(2) Matrix of samples in this SDG (e.g., water).

(3) Date first sample in SDG is collected.

(4) Date last sample in SDG is collected.

(5) Sample ID (e.g., NB03SED122).

(6) Check if an MS or MSD analysis should be performed on this sample. If an MS or MSD is to be performed, note in the "Comments" column which analysis the MS/MSD should be performed for. If the sample is not to be analyzed for an MS/MSD, then leave blank.

(7) Provide any pertinent comments regarding the samples submitted for analyses (e.g., "MS for Herbicide").

(8) Rinsate blank ID as described in Section 2.2.3.2 of this SOP (e.g., NB3003RB).

(9) Provide any pertinent comments regarding the rinsate blank submitted for analyses.

(10) Trip blank ID as described in Section 2.2.3.3 of this SOP (e.g., TB01012013-02).

(11) Provide any pertinent comments regarding the trip blank submitted for analysis.

Custody Seal

SEALED BY

(1) _____

DATE (2) _____ **TIME** (3) _____

Key:

(1) Name of individual sealing ice chest.

(2) Date ice chest is sealed.

(3) Time ice chest is sealed (24-hour format).

Appendix J

SOP No. 10 – Documenting Field Activities

Standard Operating Procedure
No. 10

Documenting Field Activities

October 2013

Revision 0

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1. Purpose and Scope

The purpose of this document is to define the standard operating procedure (SOP) for the documentation of field activities associated with the Newark Bay Study Area (NBSA), including sample collection events, field measurements, and site visits. Appropriate documentation of field activities provides an accurate and comprehensive record of the work performed, sufficient for a technical peer to reconstruct the day's activities and determine that necessary requirements were met. Field records also provide evidence and support technical interpretations and judgments. The procedures and systems defined in this SOP help confirm that the records are identifiable (reference the project task/activity), retrievable, and protected from loss or damage.

NBSA field data will be recorded in field notebook entries, standardized forms, annotated maps, or photos. This SOP provides general guidance on field recordkeeping; additional details for specific procedures (e.g., chain of custody) are provided in the SOPs for the individual task.

This SOP may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by Tierra Solutions Inc.'s (Tierra's) Facility Coordinator and the United States Environmental Protection Agency Remedial Project Manager and On-Scene Coordinator. The ultimate procedure employed will be documented in the field notebook.

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Guidelines

The documentation of field activities at uncontrolled hazardous waste sites is governed by a variety of legal guidelines that must be understood prior to the commencement of field activities. It is imperative that the personnel who will be conducting the field activities understand how the overall constitutional, statutory, and evidentiary legal requirements apply to the site inspection documentation and to the rights of potentially responsible parties.

The description of and observations made during field activities often provide the basis for technical site evaluations and other related written reports. All records and notes generated in the field will be considered controlled evidentiary documents and may be subject to scrutiny in litigation. Consequently, it is essential that the Field Supervisor or designee pay attention to detail and document to the greatest extent practicable every aspect of the inspection.

Personnel designated as responsible for the documentation of field activities must be aware that all notes taken may provide the basis for the preparation of responses to legal interrogatories.

Field documentation must provide sufficient information and data to enable the reconstruction of field activities. A wireless field application using standardized electronic data forms may provide the basic means for documenting field activities.

Control and maintenance of wireless field applications used in the documentation of field activities is the responsibility of the Field Supervisor, and the transfer of responsibility (e.g., alternate Field Supervisor) must be documented.

3. Equipment and Materials

The following equipment list contains materials that may be needed in carrying out the procedures contained in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- ☐ standardized field data forms (electronic and printed copies)
- ☐ site maps (electronic and printed copies)
- ☐ clipboard
- ☐ three-ring binder or equivalent
- ☐ camera
- ☐ time piece
- ☐ hand-held electronic recording device (e.g., laptop)
- ☐ bound field notebook
- ☐ black, ballpoint pen or Sharpie® (or equivalent).

4. Procedures

4.1 General Requirements

The field records will contain sufficient detail so that the collection effort can be reconstructed without reliance on the collector's memory.

Pertinent field information will be recorded legibly in field notebook entries and/or in an appropriate standardized form (as described herein).

Field notebook entries will be signed and dated. No erasures or obliterations will be made. A single line (i.e., strikeout) will be drawn through incorrect entries and the corrected entry typed next to the original strikeout. Strikeouts are to be initialed and dated by the originator.

The field notebook will be a bound waterproof notebook with entries made in black ballpoint pen (or pencil, as necessary). All logbook entries will be electronically scanned at the end of each day or as frequently as possible and saved in the project files.

Entries will be factual and observational (i.e., no speculation or opinion), and will not contain any personal information or non-project-related entries. Abbreviations and acronyms will be defined.

Field information will be recorded without delay – information recorded significantly after the fact will be dated as such.

Field activities and other events pertinent to the field activities will be documented in chronological order. Times will be recorded using Eastern Standard Time (EST) or Eastern Daylight Savings Time (EDT) notation for each entry.

4.2 Field Notebook

The field notebook will be a bound waterproof notebook with entries made in black ballpoint pen (or pencil as necessary).

The title page of each logbook entry will contain the following:

- Tierra contact, Tierra office location, and phone number
- logbook entry number (corresponding to the number of days in the field event)
- project name and number
- start and end date and time of work covered by that field notebook entry.

A page header will appear on the first page of each field notebook entry (i.e., the beginning of notes for each day's events), and activities for each day will be recorded as a new logbook entry. The page header will include:

- name of author and other personnel on site (and affiliated organization if applicable)
- date
- time of arrival (military time)
- proposed activity (task)
- current weather and tidal conditions, and weather forecast for the day.

An abbreviated header, containing at least the date, will appear at the top of each additional page for the active date. Field forms require similar header information.

The field notebook will provide a chronology of events. At a minimum, documentation in a field notebook will include the following (unless documented on a standard form):

- names of visitor(s), including time of arrival and departure, the visitor's affiliation, and reason for visit
- summary of project-related communications, including names of people involved and time
- time daily work commences and ceases
- start and stop times of new tasks
- start and stop times of significant standby time (work interruptions)
- safety or other monitoring data, including units with each measurement
- deviations from approved scope of work, including the necessary approvals
- progress updates
- problems/delays encountered
- unusual events
- initials of author on every page

The field notebook will cross-reference the standardized field forms if necessary; however, whenever possible, details recorded on the standardized forms will not be replicated in the field notebook.

In the case of equipment malfunction or other unforeseen events, additional bound waterproof field notebooks will be carried by field personnel to serve as backup documentation methods. NBSA field notebooks will be dedicated to the project and will not be used for any other project or purpose. Separate and dedicated field notebooks will be kept for different operations running concurrently (e.g., sampling on board the vessel, processing at the field facility); individual tasks making up each operation will be maintained in the same field notebook, if possible. The cover and binding of each field notebook will be labeled to identify the operation and dates included with the notebook; each page in the field notebook will be consecutively numbered. Pages will not be removed or torn out of the notebook. If there are additional lines on the page at the end of the day's activities, a line will be drawn through the empty space, and initialed and dated, leaving no room for additional entries. Logbook entries will be electronically scanned at the end of each day, or as frequently as possible, and electronically saved as described in Section 4.6.

4.3 Standardized Forms

The information collected on any field forms will be collected and/or scanned and stored (if a printed form) electronically (described in Section 4.6).

The following rules apply to the standardized forms:

- Each form will be printed (if electronic), signed, and dated by the person completing the form and stored as described in Section 5.
- There will be no blank spaces on the form – unused spaces will have "not applicable" or "not available" explanations.
- Field forms require similar header information as field notebook entries (see Section B of this SOP).
- At the end of each day, or as frequently as possible, all forms completed will be saved as described in Section 4.6.

4.4 Maps and Drawings

Pre-existing maps and drawings that include notations made in the field (i.e., relocating of sample locations) will be referenced in the field notebook and, like all field records,

include the project/task name and number, site identification, and be signed or initialed and dated by the person who prepared them.

Maps and drawings will include compass orientation and scale. Sketches will include points of reference and distances to the reference points.

If notations are made on electronic map or drawing files, these will be referenced in the logbook as described above and initialed and dated by the person who prepared them. Notations made by hand on maps and drawings will be electronically scanned at the end of each day, or as frequently as possible, and electronically saved as described in Section 4.6.

4.5 Photographs and Other Photo Documentation

Photographs or videos may be taken by the field team to help document site conditions, sampling locations, or sample characteristics. Photographs and videos will be identified in the field notebook or on the electronic standard form by a unique numbering system. If photographs are collected using a digital camera, the file number, as well as the photograph number, will accompany the description of the photograph in the notebook. At a minimum, the date/time the photograph was taken, the general location, a brief description, and the photographer's name will be recorded. Additional information may include differential global positioning system coordinates, direction the photographer was facing, and/or weather conditions. If necessary, an object will be included to indicate the scale of the object in the photograph.

4.6 Electronic Files

Electronic recording devices may include data logging systems, personal digital assistants (PDAs), laptops, or tablet personal computers.

Sufficient backup systems will be in place to protect against electronic data loss. Information will be saved to a disk or backed up immediately upon completion. The backup disk or other media (CD, flash drive) will then be stored in a secure location separate from the laptop, tablet, or PDA.

Files will be uniquely identified and stored in the project files. File names should include the date, a description of the file contents or a unique title, and a version number (i.e., "YYYYMMDD_Name of documentV#). An unedited version of the file will be maintained, and all subsequent manipulations tracked.

5. Quality Assurance/Quality Control

Entries in the field forms will be double-checked by the samplers to verify that the information is correct.

Completed field forms will be reviewed periodically by the Facility Coordinator and/or Project Quality Assurance Manager or their designees to verify that the requirements are being met. At a minimum, this should occur at the end of each day. When the review is complete, the reviewer will append his/her initials and date to the pages reviewed for documentation purposes.

If information recorded in the field is transcribed to another format, the original record will be retained for comparison purposes.

6. Data and Records Management

Deviations to the procedures detailed in the SOP will be recorded in the field notebook.

Notebooks, field forms, chain of custody forms, and all other records associated with the activities described in this SOP will be ultimately maintained by the investigative organization.

Field notebook entries, field data forms, and chain of custody forms will be electronically stored once they have been completed and distributed (if necessary) at the end of each field day or as frequently as possible. Printed copies of these documents will be maintained in labeled three-ring binders or contained in some other organized manner that prevents loss in the field facility. Bound waterproof field notebooks will be electronically scanned and saved in project files at the end of each day, or as frequently as possible, to mitigate against the loss of historical entries should the notebook be lost in the field.

Distribution of daily forms will be performed according to the needs of the project team and at the direction of the Field Supervisor or designee.

The Field Supervisor is responsible for reviewing and approving the field records for accuracy, completeness, and conformance to the procedures in this SOP. The Field Supervisor is also responsible for confirming that the field records are distributed to the appropriate personnel during field activities, confirming that records are maintained properly on site, and for archiving the records upon completion of field activities.

Appendix K

SOP No. 11 – Data Management

Standard Operating Procedure
No. 11

Data Management

October 2013

Revision 0

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Attachment

Data Review Form

1. Purpose and Scope

The purpose of this document is to define the standard operating procedure (SOP) for data management. This SOP describes the procedures necessary to manage the field sampling and laboratory data. In addition to the sample collection efforts, all requirements set forth in this SOP are to apply to data collected as part of the sampling activities.

This SOP may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by Tierra Solutions Inc.'s (Tierra's) Facility Coordinator and the United States Environmental Protection Agency Remedial Project Manager and On-Scene Coordinator. The ultimate procedure employed will be documented in the field notebook.

Other SOPs will be utilized with this procedure, including:

- SOP No. 10 – Documenting Field Activities.

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Procedures

2.1 Data Handling and Management

The following sections trace the paths of data from generation to final use and storage, as well as the associated quality reviews for error detection that are performed to promote data integrity.

2.1.1 Field Data Management

2.1.1.1 Data Recording

Field data and information collected during sample collection may be recorded by hand using the associated forms or directly into an electronic field database (MS Access or equivalent), and will be recorded in accordance with requirements described in SOP No. 10 – Documenting Field Activities. Once sample collection data are transcribed into the electronic field database, the sample processing team may enter data into the associated forms by hand or within the electronic field database during sample processing activities. The electronic field database will be maintained for field parameter entries only (e.g., date, time, flow rates) and will not be joined with the

analytical database. In addition, a daily field notebook will be maintained per SOP No. 10 – Documenting Field Activities.

2.1.1.2 Data Review

Each day after the completion of field activities, an on-site review of the bulk sample collection data will be performed by a designated team member, who will be different from the originator of the field data. This review will include verifying:

- bulk samples were collected at pre-identified sampling locations following sampling procedures
- data and information, as well as associated forms, were completed in their entirety for each sample
- data and information entered into the database or forms are accurate and consistent with that recorded in the logbooks (i.e., sample ID, location, and time of collection).

Once these have been verified, the selected reviewer will sign and date hard copy forms and the field notebook. Hard copies from the electronic field database will be printed for placement in the project file. Additionally, electronic versions (in Portable Document Format [PDF] format, or equivalent) of all forms will be placed on a secure network drive accessible only to project personnel. The data will be sent to the ARCADIS office for review each day.

Upon receipt of daily documentation (including the electronic field database and scanned hard copy forms and notebook), the office data manager will perform a secondary review of the daily field data for consistency and accuracy.

Once the electronic field database has been reviewed and deemed accurate, a Daily Report will be generated by the office database team member and provided to the Facility Coordinator for review and comment. Upon approval by the Facility Coordinator, the Daily Report may be provided to USEPA.

If an electronic field database is maintained, the data sent from the site will be imported by the office data manager. If generated, the electronic field database will contain data collected during sample collection and sample processing activities for the entire project. The electronic field database will be placed on a secure network drive, accessible only to project personnel, for storage/filing purposes.

2.1.2 Analytical Data Management

2.1.2.1 Data Recording

The laboratory(ies) will prepare and retain full analytical and quality control (QC) documentation. Laboratory records (both handwritten and electronic) will be documented per the procedures contained in the laboratory-specific quality assurance (QA) manual. Once the hard copy data packages and Electronic Data Deliverables (EDDs) are ready, they will be verified by the laboratory for completeness and compliance prior to submittal to the Quality Assurance Coordinator (QAC, Environmental Data Services, Ltd. [EDS]).

2.1.2.2 Data Transformation and Data Reductions

Calculations involved in reducing raw data to reportable data are defined in the specified analytical methods. Automated data processing steps not provided in the procedures listed can be found in the laboratory-specific QA manuals. Verification of records and calculations generated by the laboratories will be performed as required tasks and documented by signature of reviewer/data validation contractor.

Significant digits for hard copy and electronic results will be reported as specified in the analytical method reference.

2.1.2.3 Data Transfer

Unverified/unvalidated data will be transferred electronically from the identified analytical laboratories to the QAC using the EDD format, specified in Worksheet No. 29 of the Crab and Clam Sampling and Analysis Quality Assurance Project Plan (Crab/Clam QAPP). The data will be transferred in Microsoft Excel spreadsheet format (or equivalent) via email or similar information transfer mechanism.

2.1.3 Quality Assurance Coordinator (EDS)

2.1.3.1 Data Recording

The QAC will document the data verification/validation tasks as described in Worksheet Nos. 34, 35, and 36 of the Crab/Clam QAPP.

2.1.3.2 Data Transfer

Once the analytical data have been reviewed and verified/validated, the Data Verification Reports will be generated. The QAC will transfer the EDDs containing

verified/validated sample results and completed Data Verification Reports to Tierra's Facility Coordinator. The EDD files will be transferred in Microsoft Excel (or equivalent) spreadsheets via email or similar information transfer mechanism, and Data Verification Reports will be transferred in hard copy or electronic PDF file format via FedEx or similar delivery service.

2.1.3.3 Review of Verified/Validated Laboratory Data and Agency Submittal

EDDs received by Tierra's Facility Coordinator will be saved to a secure network drive for storage/archive purposes. The EDD files will then be imported into a Microsoft Access (or equivalent) database, which will also be maintained on a secure network drive. Completed Data Verification Reports will be scanned or uploaded, as appropriate, and stored on a secure network drive as PDF files.

Importation of the verified/validated EDD files will be an automated process using macros and queries developed in an EQuIS database. Once EDDs have been entered into the analytical database, data output tables will be prepared so that the data can be checked for errors. This check will consist of a 10 percent comparison of the data output tables to marked-up Form 1s that have been initialed and dated by the reviewer upon completion of the data verification/validation task. This data review will be documented using the Data Review Form attached to this SOP.

Once EDDs have been entered into the analytical database and the data have been checked for errors, new EDDs will be created in the format required by USEPA Region 2. The EDDs will be created using various update and append queries in an EQuIS database. Once the EDDs have been reviewed for accuracy, they will be transmitted to USEPA via email or similar information transfer mechanism. Copies of the EDDs will be stored in the project file on compact disc and on the secure network drive in EQuIS format.

2.2 Data Tracking and Control

The following sections provide a discussion of the procedures for data tracking, storage, archiving, retrieval, and security for both hard copy and electronic data and information.

2.2.1 Data Storage, Archiving, and Retrieval

2.2.1.1 Hard Copy Files

Hard copies of project documentation and data will be placed in the project file, parts of which will exist in several locations, including:

- ARCADIS, Syracuse, New York and Cranbury, New Jersey
- Environmental Data System, Ltd., Pittsburgh, Pennsylvania
- Tierra Solutions, Inc., East Brunswick, New Jersey.

Such files will be maintained in secure locations within each facility. Hard copies of project documentation and data will be provided to USEPA upon request.

Duplicate copies of pertinent field-related correspondence/documentation will be maintained at the field office during field operations. Once such field operations have been completed, this documentation will be transferred to the project file.

Upon completion of the data verification/validation process, project documents and records will be stored in the project file. At such time that it is deemed appropriate to archive the project file, either in parts or in its entirety, files may be boxed and shipped off site to a secure document storage facility. The assigned barcode identifier for each box being archived will be logged into a tracking spreadsheet, as well as with a brief description of the contents of the box. Archived boxes will be retrieved from the document storage facility, if/when necessary, using the logged barcode identifier.

2.2.1.2 Electronic Files

Electronic data and information will be maintained and managed by the entities listed above using password-protected computers and on secure network drives with access limited to project personnel. Files will generally consist of the same components as the hard copy files.

Reports (including QA reports), analytical laboratory documentation, data verification reports, and field data will be in PDF (or equivalent). Additionally, electronic copies of EDDs containing verified/validated sample results will be filed in Microsoft Excel format (or equivalent), and daily and cumulative electronic field databases will be filed in Microsoft Access format (or equivalent).

2.2.2 Data Security

Hard copy information/data will be stored in secure areas within the three project file locations. Electronic data and information will be maintained and managed using password-protected computers and on secure network drives with access limited to project personnel.

2.3 Final Data Evaluation

Data evaluation activities may be performed by Tierra's Facility Coordinator. The computer programs that will be utilized during data evaluation include EQuIS, Microsoft Access, Microsoft Excel, MatLab, and ArcGIS or similar software (including ArcMap application [distributed by Environmental Systems Research Institute, Inc.]).

3. Quality Assurance

Appropriate QA/QC procedures will be followed during data management in accordance with SOP No. 10 – Documenting Field Activities and this SOP.

4. Documentation

Field and laboratory documentation will be maintained in accordance with SOP No.10 – Documenting Field Activities and this SOP.

5. References

USEPA. 1996. Method 1669, Sampling Ambient Water for Trace Metals at EPA Water Criterion Levels, U.S. Environmental Protection Agency, Office of Water Engineering and Analysis Division (4303), July 1996.

DATA REVIEW FORM

(Sheet 1 of 1)

Date: ___(1)____

SDG: ___(2)____

Analytical Group: _____(3)_____

Samples Reviewed: _____(4)_____

_____

Identified Errors:

_____(5)_____

_____

_____

_____

_____

_____

Corrective Actions Taken:

_____(6)_____

_____

_____

_____

_____

_____

Person Responsible: _____(7)_____

Attach original data table from analytical database, and final data table showing changes (if applicable) to this form for data tracking/filing purposes. Also, attach any communications (e.g., emails, conversation records) related to the data changes discussed above.

DATA REVIEW FORM KEY
(Sheet 1 of 1)

DESCRIPTION OF ITEMS:

(1) Date of data review (e.g., 1/1/2013).

(2) Sample delivery group number assigned to data package under review.

(3) Analytical group requested for analysis under review (e.g., chlorinated herbicides).

(4) Sample ID(s) of samples chosen for review.

(5) Errors identified during comparison of data table from database to initialed and dated Form 1s in Data Verification Reports.

(6) Actions taken to correct errors identified during comparison of data table from database to marked-up Form 1s in Data Verification Reports.

(7) Name of person entering information into this form.

Appendix L

Tissue and Sediment Thresholds Used to Establish Data Quality Levels

Screening Values for Tissue - Newark Bay

Chemical	CAS	Tissue USEPA RSL - Fish Ing (Risk of 10^{-6} or Hazard of 0.1) (mg/kg)	C/NC	Surrogate
Inorganics				
Aluminum	7429-90-5	140	nc	--
Antimony	7440-36-0	0.054	nc	--
Arsenic	7440-38-2	0.0021	ca	--
Asbestos	1332-21-4	TBD	--	--
Barium	7440-39-3	27	nc	--
Beryllium	7440-41-7	0.27	nc	--
Cadmium	7440-43-9	0.14	nc	--
Calcium	7440-70-2	Essential nutrient	--	--
Chromium [as Cr(III)]	7440-47-3	200	nc	--
Chromium (VI)	18540-29-9	0.0063	ca	--
Cobalt	7440-48-4	0.041	nc	--
Copper	7440-50-8	5.4	nc	--
Cyanide	57-12-5	0.081	nc	--
Erionite	66733-21-9	TBD	--	--
Iron	7439-89-6	95	nc	--
Lead	7439-92-1	TBD	--	--
Magnesium	7439-95-4	Essential nutrient	--	--
Manganese	7439-96-5	19	nc	--
Mercury	7439-97-6	TBD	--	--
Nickel	7440-02-0	2.7	nc	--
Potassium	2023695	Essential nutrient	--	--
Selenium	7782-49-2	0.68	nc	--
Silicon	7440-21-3	TBD	--	--
Silver	7440-22-4	0.68	nc	--
Sodium	7440-23-5	Essential nutrient	--	--
Thallium	7440-28-0	0.0014	nc	--
Tin	7440-31-5	81	nc	--
Titanium	7440-32-6	TBD	--	Titanium Tetrachloride
Vanadium	7440-62-2	0.68	nc	--
Zinc	7440-66-6	41	nc	--
Pesticides				
2,4-D	94-75-7	1.4	nc	--
2,4-DB	94-82-6	1.1	nc	--
2,4,5-T	93-76-5	1.4	nc	--
2,4,5-TP (Silvex)	93-72-1	1.1	nc	--
2,4'-DDD	53-19-0	0.013	ca	4,4'-DDD
2,4'-DDE	3424-82-6	0.0093	ca	4,4'-DDE
2,4'-DDT	784-02-0	0.0093	ca	4,4'-DDT
4-Nitrophenol	100-02-7	TBD	--	--
4,4'-DDD	72-54-8	0.013	ca	--
4,4'-DDE	72-55-9	0.0093	ca	--
4,4'-DDT	50-29-3	0.0093	ca	--
Aldrin	309-00-2	0.00019	ca	--
alpha-BHC	319-84-6	0.0005	ca	--
beta-BHC	319-85-7	0.0018	ca	--
Chlordane	12789-03-6	0.009	ca	--

Screening Values for Tissue - Newark Bay

Chemical	CAS	Tissue USEPA RSL - Fish Ing (Risk of 10^{-6} or Hazard of 0.1) (mg/kg)	C/NC	Surrogate
Chlordane,alpha (cis)	5103-71-9	0.009	ca	Chlordane
Chlordane,gamma (trans)	5103-74-2	0.009	ca	Chlordane
Chlorpyrifos	2921-88-2	0.14	nc	--
Dacthal	1861-32-1	1.4	nc	--
Dalapon	75-99-0	4.1	nc	--
Delta-BHC	319-86-8	0.0018	ca	Hexachlorocyclohexane, Technical
Dicamba	1918-00-9	4.1	nc	--
Dichloroprop	120-36-5	TBD	--	--
Dieldrin	60-57-1	0.0002	ca	--
Dinoseb	88-85-7	0.14	nc	--
Endosulfan I	959-98-8	0.81	nc	Endosulfan
Endosulfan II	33213-65-9	0.81	nc	Endosulfan
Endosulfan sulfate	1031-07-8	0.81	nc	Endosulfan
Endosulfan, alpha	95-99-98	0.81	nc	Endosulfan
Endrin	72-20-8	0.041	nc	--
Endrin aldehyde	7421-93-4	0.041	nc	Endrin
Endrin ketone	53494-70-5	0.041	nc	Endrin
Gamma-BHC (Lindane)	58-89-9	0.0029	ca	--
Heptachlor	76-44-8	0.0007	ca	--
Heptachlor epoxide	1024-57-3	0.00035	ca	--
Hexachlorobenzene	118-74-1	0.002	ca	--
MCPA	94-74-6	0.068	nc	--
MCPP	93-65-2	0.14	nc	--
Methoxychlor	72-43-5	0.68	nc	--
Mirex	2385-85-5	0.00018	ca	--
Mirex, photo-	39801-14-4	0.00018	ca	Mirex
Nonachlor, cis-	5103-73-1	TBD	--	--
Nonachlor, trans-	39765-80-5	TBD	--	--
Oxychlordane	27304-13-8	0.009	ca	Chlordane
Pentachlorophenol	87-86-5	0.0079	ca	--
Total Endosulfan	115-29-7	0.81	nc	--
Toxaphene	8001-35-2	0.0029	ca	--
PAHs				
1-Methylnaphthalene	90-12-0	0.11	ca	--
1-Methylphenanthrene	832-69-9	TBD	--	--
2-Methylnaphthalene	91-57-6	0.54	nc	--
2-Methylphenanthrene	2531-84-2	TBD	--	--
2,3,5-Trimethylnaphthalene	2245-38-7	TBD	--	--
2,6-Dimethylnaphthalene	581-42-0	TBD	--	--
3,6-Dimethylphenanthrene	1576-67-6	TBD	--	--
Acenaphthene	83-32-9	8.1	nc	--
Acenaphthylene	208-96-8	4.1	nc	--
Anthracene	120-12-7	41	nc	Pyrene
Benz(a)anthracene	56-55-3	0.0043	ca	--
Benzo(a)pyrene	50-32-8	0.00043	ca	--
Benzo(b)fluoranthene	205-99-2	0.0043	ca	--
Benzo(j)fluoranthene	205-82-3	0.0026	ca	--

Screening Values for Tissue - Newark Bay

Chemical	CAS	Tissue USEPA RSL - Fish Ing (Risk of 10^{-6} or Hazard of 0.1) (mg/kg)	C/NC	Surrogate
Benzo(e)pyrene	192-97-2	4.1	nc	Pyrene
Benzo(g,h,i)perylene	191-24-2	4.1	nc	Pyrene
Benzo(k)fluoranthene	207-08-9	0.043	ca	--
C1Naphthalenes	--	4.1	nc	Pyrene
C1Phenanthrenes	--	4.1	nc	Pyrene
C1Phenanthrenes/Anthracenes	--	4.1	nc	Pyrene
C2Phenanthrenes	--	4.1	nc	Pyrene
C2Phenanthrenes/Anthracenes	--	4.1	nc	Pyrene
Chrysene	218-01-9	0.43	ca	--
Dibenz(a,h)anthracene	53-70-3	0.00043	ca	--
Fluoranthene	206-44-0	5.4	nc	--
Fluorene	86-73-7	5.4	nc	--
High Molecular Weight PAHs	--	4.1	nc	Pyrene
Indeno(1,2,3-c,d)-pyrene	193-39-5	0.0043	ca	--
Low Molecular Weight PAHs	--	4.1	nc	Pyrene
Naphthalene	91-20-3	2.7	nc	--
Perylene	198-55-0	4.1	nc	Pyrene
Phenanthrene	85-01-8	4.1	nc	Pyrene
Pyrene	129-00-0	4.1	nc	--
Other Organics				
4-Chlorophenyl Phenyl Ether	7005-72-3	TBD	--	--
cis-1,4-Dichloro-2-butene	764-41-0	TBD	--	--
1,1-Dichloroethane	75-34-3	0.55	ca	--
1,1-Dichloroethene	75-35-4	6.8	nc	--
1,1-Dichloropropene	563-58-6	TBD	--	--
1,1,1-Trichloroethane	71-55-6	270	nc	--
1,1,1,2-Tetrachloroethane	630-20-6	0.12	ca	--
1,1,2-Trichloroethane	79-00-5	0.055	ca	--
1,1,2,2-Tetrachloroethane	79-34-5	0.016	ca	--
1,2-Dibromo-3-chloropropane	96-12-8	0.0039	ca	--
1,2-Dibromoethane	106-93-4	0.0016	ca	--
1,2-Dichlorobenzene	95-50-1	12	nc	--
1,2-Dichloroethane	107-06-2	0.035	ca	--
1,2-Dichloroethene (total)	540-59-0	1.2	nc	--
1,2-Dichloropropane	78-87-5	0.088	ca	--
1,2,3-Trichlorobenzene	87-61-6	0.11	nc	--
1,2,3-Trichloropropane	96-18-4	0.00011	ca	--
1,2,3,4-Tetrachlorobenzene	634-66-2	0.041	nc	1,2,4,5-Tetrachlorobenzene
1,2,3,5-Tetrachlorobenzene	634-90-2	0.041	nc	1,2,4,5-Tetrachlorobenzene
1,2,4-Trichlorobenzene	120-82-1	0.11	ca	--
1,2,4-Trimethylbenzene	95-63-6	TBD	--	--
1,2,4,5-Tetrachlorobenzene	95-94-3	0.041	nc	--
1,3-Dichlorobenzene	541-73-1	12	nc	1,2-Dichlorobenzene
1,3 Butadiene	106-99-0	0.00093	ca	--
1,3,5-Trichlorobenzene	108-70-3	0.11	ca	1,2,4-Trichlorobenzene
1,3,5-Trimethylbenzene	108-67-8	1.4	nc	--
1,4-Dichlorobenzene	106-46-7	0.58	ca	--

Screening Values for Tissue - Newark Bay

Chemical	CAS	Tissue USEPA RSL - Fish Ing (Risk of 10^{-6} or Hazard of 0.1) (mg/kg)	C/NC	Surrogate
1,4-Dioxane	123-91-1	0.032	ca	--
2-Chloroethylvinylether	110-75-8	TBD	--	--
2-Chlorophenol	95-57-8	0.68	nc	--
2-Chlorotoluene	95-49-8	2.7	nc	--
2-Hexanone	591-78-6	0.68	nc	--
2-Nitroaniline	88-74-4	1.4	nc	--
2-Nitrophenol	88-75-5	TBD	--	--
2-Naphthylamine	91-59-8	0.0018	ca	--
2,2-Dichloropropane	594-20-7	0.088	ca	1,2-Dichloropropane
2,4-Dichlorophenol	120-83-2	0.41	nc	--
2,4-Dimethylphenol	105-67-9	2.7	nc	--
2,4-Dinitrophenol	51-28-5	0.27	nc	--
2,4-Dinitrotoluene	121-14-2	0.01	ca	--
2,4,5-Trichlorophenol	95-95-4	14	nc	--
2,4,6-Trichlorophenol	88-06-2	0.14	nc	--
2,6-/2,7-Dimethylnaphthalene	--	TBD	--	--
2,6-Dinitrotoluene	606-20-2	0.14	nc	--
3-Nitroaniline	99-09-2	0.16	ca	4-Nitroaniline
3,3'-Dichlorobenzidine	91-94-1	0.007	ca	--
4-Bromophenyl Phenyl Ether	101-55-3	TBD	--	--
4-Chloro-3-Methylphenol	59-50-7	14	nc	--
4-Chloroaniline	106-47-8	0.016	ca	--
4-Chlorotoluene	106-43-4	2.7	nc	--
4-Methyl-2-Pentanone	108-10-1	11	nc	--
4-Methylphenol	106-44-5	14	nc	--
4-Nitroaniline	100-01-6	0.16	ca	--
4-Aminobiphenyl	92-67-1	0.00015	ca	--
4,6-Dinitro-2-methylphenol	534-52-1	0.011	nc	--
Acetone	67-64-1	120	nc	--
Acrolein	107-02-8	0.068	nc	--
Acrylonitrile	107-13-1	0.0058	ca	--
Allyl Chloride	107-05-1	0.15	ca	--
Aniline	62-53-3	0.55	ca	--
Azobenzene	103-33-3	0.029	ca	--
Benzal chloride	98-87-3	TBD	--	--
Benzene	71-43-2	0.06	ca	--
Benzidine	92-87-5	0.000014	ca	--
Benzo(b)thiophene	95-15-8	TBD	--	--
Benzoic Acid	65-85-0	540	nc	--
Benzotrichloride	98-07-7	0.00024	ca	--
Benzyl Alcohol	100-51-6	14	nc	--
Benzyl chloride	100-44-7	0.019	ca	--
Biphenyl	92-52-4	0.39	ca	--
Bis(2-chloro-1-methylethyl) ether	108-60-1	0.045	ca	--
Bis(2-Chloroethoxy)methane	111-91-1	0.41	nc	--
Bis(2-Chloroethyl)ether	111-44-4	0.0029	ca	--
Bis(2-Chloroisopropyl)ether	39638-32-9	TBD	--	--

Screening Values for Tissue - Newark Bay

Chemical	CAS	Tissue USEPA RSL - Fish Ing (Risk of 10^{-6} or Hazard of 0.1) (mg/kg)	C/NC	Surrogate
Bis(2-Ethylhexyl)phthalate	117-81-7	0.23	ca	--
Bis(chloromethyl) Ether	542-88-1	0.000014	ca	--
Bromobenzene	108-86-1	1.1	nc	--
Bromochloromethane	74-97-5	TBD	--	--
Bromoform	75-25-2	0.4	ca	--
Butyl Benzyl Phthalate	85-68-7	1.7	ca	--
Carbazole	86-74-8	TBD	--	--
Carbon Disulfide	75-15-0	14	nc	--
Carbon Tetrachloride	56-23-5	0.045	ca	--
Chlorobenzene	108-90-7	2.7	nc	--
Chlorodibromomethane	124-48-1	0.038	ca	--
Chloroethane	75-00-3	TBD	--	--
Chloroform	67-66-3	0.1	ca	--
Chloroprene	126-99-8	2.7	nc	--
cis-1,2-Dichloroethylene	156-59-2	0.27	nc	--
cis-1,3-Dichloropropene	10061-01-5	0.032	ca	Dichloropropene, 1,3-
Di-n-butyl Phthalate	84-74-2	14	nc	--
Di-n-Octyl Phthalate	117-84-0	1.6	nc	--
Dibenzofuran	132-64-9	0.14	nc	--
Dibenzothiophene	132-65-0	TBD	--	--
Dibromomethane	74-95-3	1.4	nc	--
Dibutyltin	1002-33-5	0.041	nc	--
Dichlorobromomethane	75-27-4	0.051	ca	--
Dichlorodifluoromethane	75-71-8	27	nc	--
Diethyl Phthalate	84-66-2	110	nc	--
Dimethyl phthalate	131-11-3	110	nc	Diethyl Phthalate
Ethyl Methacrylate	97-63-2	12	nc	--
Ethylbenzene	100-41-4	0.29	ca	--
Ethylene Oxide	75-21-8	0.01	ca	--
Formaldehyde	50-00-0	27	nc	--
Freon	76-13-1	4,100	nc	--
Hexachlorobutadiene	87-68-3	0.04	ca	--
Hexachlorocyclohexanes	608-73-1	0.0018	ca	--
Hexachlorocyclopentadiene	77-47-4	0.81	nc	--
Hexachloroethane	67-72-1	0.079	ca	--
Isobutyl Alcohol	78-83-1	41	nc	--
Isophorone	78-59-1	3.3	ca	--
Isopropylbenzene	98-82-8	14	nc	--
Methyl Bromide	74-83-9	0.19	nc	--
Methyl Chloride	74-87-3	TBD	--	--
Methyl Ethyl Ketone	78-93-3	81	nc	--
Methyl Iodide	74-88-4	TBD	--	--
Methyl Methacrylate	80-62-6	190	nc	--
Methyl-t-butyl Ether	1634-04-4	1.8	ca	--
Methylacrylonitrile	126-98-7	0.014	nc	--
Methylene Bromide	74-95-3	1.4	nc	--
Methylene Chloride	75-09-2	0.81	nc	--

Screening Values for Tissue - Newark Bay

Chemical	CAS	Tissue USEPA RSL - Fish Ing (Risk of 10^{-6} or Hazard of 0.1) (mg/kg)	C/NC	Surrogate
Methylmercury	22967-92-6	0.014	nc	--
Mineral Oils	8012-95-1	410	nc	--
Monobutyltin	78763-54-9	0.041	nc	Tri-n-butyltin
n-Butylbenzene	104-51-8	6.8	nc	--
N-nitroso-di-n-propylamine	621-64-7	0.00045	ca	--
N-nitrosodimethylamine	62-75-9	0.000062	ca	--
N-Nitrosodiphenylamine	86-30-6	0.64	ca	--
n-Propylbenzene	103-65-1	14	nc	--
Nitrobenzene	98-95-3	0.27	nc	--
O-Cresol	95-48-7	6.8	nc	--
p-Isopropyltoluene	99-87-6	14	nc	Isopropylbenzene
Pentachloroanisole	1825-21-4	TBD	--	--
Pentachlorobenzene	608-93-5	0.11	nc	--
Pentachlorophenol	87-86-5	0.0079	ca	--
Petroleum Hydrocarbons	--	TBD	--	--
Phenol	108-95-2	41	nc	--
Propionitrile	107-12-0	TBD	--	--
Pyridine	110-86-1	0.14	nc	--
sec-Butylbenzene	105-05-5	6.8	nc	N-Butylbenzene
Styrene	100-42-5	27	nc	--
tert-Butylbenzene	98-06-6	6.8	nc	N-Butylbenzene
Tetrabutyltin	1461-25-2	0.041	nc	Tri-n-butyltin
Tetrachloroethylene	127-18-4	0.81	nc	--
Tetrahydrofuran	109-99-9	120	nc	--
Toluene	108-88-3	11	nc	--
Total Xylenes	1330-20-7	27	nc	--
Trans-1,2,-Dichloroethene	156-60-5	2.7	nc	--
Trans-1,3-dichloropropene	542-75-6	0.032	ca	--
Trans-1,4-dichloro-2-butene	110-57-6	TBD	--	--
Tributyltin	56-35-9	0.041	nc	--
Trichloroethylene	79-01-6	0.068	nc	--
Trichlorofluoromethane	75-69-4	41	nc	--
Vinyl Acetate	108-05-4	140	nc	--
Vinyl Chloride	75-01-4	0.0044	ca	--
Non DL-PCBs				
Aroclor 1016	12674-11-2	0.0095	nc	--
Aroclor 1221	11104-28-2	0.0016	ca	--
Aroclor 1232	11141-16-5	0.0016	ca	--
Aroclor 1242	53469-21-9	0.0016	ca	--
Aroclor 1242/1016	53469-21-9 12674-11-2	0.0016	ca	Aroclor 1242
Aroclor 1248	12672-29-6	0.0016	ca	--
Aroclor 1254	11097-69-1	0.0016	ca	--
Aroclor 1254/1260	11097-69-1 11096-82-5	0.0016	ca	Aroclor 1242
Aroclor 1260	11096-82-5	0.0016	ca	--
PCB 1	2051-60-7	TBD	--	--
PCB 101/90	37680-73-2 68194-07-0	TBD	--	--
PCB 107	70424-68-9	TBD	--	--

Screening Values for Tissue - Newark Bay

Chemical	CAS	Tissue USEPA RSL - Fish Ing (Risk of 10^{-6} or Hazard of 0.1) (mg/kg)	C/NC	Surrogate
PCB 110/77	38380-03-9 32598-13-3	TBD	--	--
PCB 119	56558-17-9	TBD	--	--
PCB 128	38380-07-3	TBD	--	--
PCB 129	55215-18-4	TBD	--	--
PCB 130	52663-66-8	TBD	--	--
PCB 135	52744-13-5	TBD	--	--
PCB 136	38411-22-2	TBD	--	--
PCB 138/160	35065-28-2 33025-41-1	TBD	--	--
PCB 141/179	52712-04-6 52663-64-6	TBD	--	--
PCB 146	51908-16-8	TBD	--	--
PCB 149/123	38380-04-0 65510-44-3	TBD	--	--
PCB 15	2050-68-2	TBD	--	--
PCB 151	52663-63-5	TBD	--	--
PCB 153/132	35065-27-1 38380-05-1	TBD	--	--
PCB 158	74472-42-7	TBD	--	--
PCB 16/32	38444-78-9 38444-77-8	TBD	--	--
PCB 166	41411-63-6	TBD	--	--
PCB 170/190	35065-30-6 41411-64-7	TBD	--	--
PCB 171/202	52663-71-5 2136-99-4	TBD	--	--
PCB 172	52663-74-8	TBD	--	--
PCB 174	38411-25-5	TBD	--	--
PCB 175	40186-70-7	TBD	--	--
PCB 176/137	52663-65-7 35694-06-5	TBD	--	--
PCB 177	52663-70-4	TBD	--	--
PCB 178	52663-67-9	TBD	--	--
PCB 18/17	37680-65-2 37680-66-3	TBD	--	--
PCB 180	35065-29-3	TBD	--	--
PCB 183	52663-69-1	TBD	--	--
PCB 185	52712-05-7	TBD	--	--
PCB 187	52663-68-0	TBD	--	--
PCB 191	74472-50-7	TBD	--	--
PCB 193	69782-91-8	TBD	--	--
PCB 194	35694-08-7	TBD	--	--
PCB 195/208	52663-78-2 52663-77-1	TBD	--	--
PCB 197	33091-17-7	TBD	--	--
PCB 199	52663-75-9	TBD	--	--
PCB 200	52663-73-7	TBD	--	--
PCB 201/157/173	40186-71-8 69782-90-7 68194-16-1	TBD	--	--
PCB 203/196	52663-76-0 42740-50-1	TBD	--	--
PCB 205	74472-53-0	TBD	--	--
PCB 206	40186-72-9	TBD	--	--
PCB 207	52663-79-3	TBD	--	--
PCB 209	2051-24-3	TBD	--	--
PCB 22/51	38444-85-8 68194-04-7	TBD	--	--
PCB 24/27	55702-45-9 38444-76-7	TBD	--	--
PCB 25	55712-37-3	TBD	--	--
PCB 26	38444-81-4	TBD	--	--

Screening Values for Tissue - Newark Bay

Chemical	CAS	Tissue USEPA RSL - Fish Ing (Risk of 10^{-6} or Hazard of 0.1) (mg/kg)	C/NC	Surrogate
PCB 28	7012-37-5	TBD	--	--
PCB 29	15862-07-4	TBD	--	--
PCB 30	35693-92-6	TBD	--	--
PCB 31	16606-02-3	TBD	--	--
PCB 33/20	38444-86-9	TBD	--	--
PCB 39	38444-84-7	TBD	--	--
PCB 40	38444-93-8	TBD	--	--
PCB 41/64	52663-59-9 52663-58-8	TBD	--	--
PCB 42/59/37	36559-22-5 74472-33-6 38444-90-5	TBD	--	--
PCB 44	41464-39-5	TBD	--	--
PCB 45	70362-45-7	TBD	--	--
PCB 46	41464-47-5	TBD	--	--
PCB 47/75	2437-79-8 32598-12-2	TBD	--	--
PCB 48	70362-47-9	TBD	--	--
PCB 49	41464-40-8	TBD	--	--
PCB 52	35693-99-3	TBD	--	--
PCB 53	41464-41-9	TBD	--	--
PCB 55/91	74338-24-2 68194-05-8	TBD	--	--
PCB 56/60	74338-24-2 33025-41-1	TBD	--	--
PCB 63	74472-34-7	TBD	--	--
PCB 66	32598-10-0	TBD	--	--
PCB 67	73575-53-8	TBD	--	--
PCB 69	60233-24-1	TBD	--	--
PCB 7/9	33284-50-3 34883-39-1	TBD	--	--
PCB 70	32598-11-1	TBD	--	--
PCB 72	41464-42-0	TBD	--	--
PCB 74/61	32690-93-0 33284-53-6	TBD	--	--
PCB 8/5	34883-43-7 16605-91-7	TBD	--	--
PCB 82	52663-62-4	TBD	--	--
PCB 83	60145-20-2	TBD	--	--
PCB 84	52663-60-2	TBD	--	--
PCB 85	65510-45-4	TBD	--	--
PCB 87/115	38380-02-8 74472-38-1	TBD	--	--
PCB 92	52663-61-3	TBD	--	--
PCB 95/80	38379-99-6 33284-52-5	TBD	--	--
PCB 97	41464-51-1	TBD	--	--
PCB 99	38380-01-7	TBD	--	--
PCBs - Total Aroclors	1336-36-3	0.0016	ca	--
PCBs - Total Congeners	1336-36-3	0.0016	ca	--
Total PCBs	1336-36-3	0.0016	ca	--
DL-PCBs				
PCB 105	32598-14-4	0.0008	ca	--
PCB 114	74472-37-0	0.0008	ca	--
PCB 118	31508-00-6	0.0008	ca	--
PCB 123	65510-44-3	0.0008	ca	--
PCB 126	57465-28-8	0.00000024	ca	--
PCB 156	38380-08-4	0.0008	ca	--

Screening Values for Tissue - Newark Bay

Chemical	CAS	Tissue USEPA RSL - Fish Ing (Risk of 10^{-6} or Hazard of 0.1) (mg/kg)	C/NC	Surrogate
PCB 157	69782-90-7	0.0008	ca	--
PCB 167	52663-72-6	0.0008	ca	--
PCB 169	32774-16-6	0.0000008	ca	--
PCB 189	39635-31-9	0.0008	ca	--
PCB 77	32598-13-3	0.00024	ca	--
PCB 81	70362-50-4	0.00008	ca	--
Dioxins/Furans				
1,2,3,4,6,7,8-HpCDD	35822-46-9	0.0000024	ca	--
1,2,3,4,6,7,8-HpCDF	67562-39-4	0.0000024	ca	--
1,2,3,4,7,8-HxCDD	39227-28-6	0.00000024	ca	--
1,2,3,4,7,8-HxCDF	70648-26-9	0.00000024	ca	--
1,2,3,4,7,8,9-HpCDF	55673-89-7	0.0000024	ca	--
1,2,3,6,7,8-HxCDD	57653-85-7	0.00000024	ca	--
1,2,3,6,7,8-HxCDF	57117-44-9	0.00000024	ca	--
1,2,3,7,8-PeCDD	40321-76-4	0.000000024	ca	--
1,2,3,7,8-PeCDF	57117-41-6	0.0000008	ca	--
1,2,3,7,8,9-HxCDD	19408-74-3	0.00000024	ca	--
1,2,3,7,8,9-HxCDF	72918-21-9	0.00000024	ca	--
2,3,4,6,7,8-HxCDF	60851-34-5	0.00000024	ca	--
2,3,4,7,8-PeCDF	57117-31-4	0.00000008	ca	--
2,3,7,8-TCDD	1746-01-6	0.000000024	ca	--
2,3,7,8-TCDF	51207-31-9	0.00000024	ca	--
OCDD	3268-87-9	0.00008	ca	--
OCDF	39001-02-0	0.00008	ca	--

Abbreviations:

C - based on carcinogenic effects, CAS - Chemical Abstracts Service number, DL - dioxin-like, M - maximum contaminant level, mg/kg - milligram per kilogram, N - based on noncarcinogenic effects, NDL - non dioxin-like

PAH - polycyclic aromatic hydrocarbon, PBB - polybrominated biphenyl, PCB - polychlorinated biphenyl, PCN - polychlorinated naphthalene, RSL - regional screening level, TBD - to be determined

µg/L - microgram per liter, USEPA - U.S. Environmental Protection Agency

Notes:

The 4 essential nutrients lacking EPA RSLs will be screened from the HHRA.

Screening levels for chemicals lacking EPA RSLs will be determined at a later date (TBD).

RSLs reflect residential values from the November 2012 version of the RSLs.

Screening Values for Sediment

CAS Number	Analyte	Result Units	Screening Value	Basis
Dioxins/Furans (µg/kg)				
35822-46-9	1,2,3,4,6,7,8-HpCDD	µg/kg	0.45	Human Health (ca)
67562-39-4	1,2,3,4,6,7,8-HpCDF	µg/kg	0.45	Human Health (ca)
55673-89-7	1,2,3,4,7,8,9-HpCDF	µg/kg	0.45	Human Health (ca)
39227-28-6	1,2,3,4,7,8-HxCDD	µg/kg	0.045	Human Health (ca)
70648-26-9	1,2,3,4,7,8-HxCDF	µg/kg	0.045	Human Health (ca)
57653-85-7	1,2,3,6,7,8-HxCDD	µg/kg	0.045	Human Health (ca)
57117-44-9	1,2,3,6,7,8-HxCDF	µg/kg	0.045	Human Health (ca)
19408-74-3	1,2,3,7,8,9-HxCDD	µg/kg	0.045	Human Health (ca)
72918-21-9	1,2,3,7,8,9-HxCDF	µg/kg	0.045	Human Health (ca)
40321-76-4	1,2,3,7,8-PeCDD	µg/kg	0.0045	Human Health (ca)
57117-41-6	1,2,3,7,8-PeCDF	µg/kg	0.15	Human Health (ca)
60851-34-5	2,3,4,6,7,8-HxCDF	µg/kg	0.045	Human Health (ca)
57117-31-4	2,3,4,7,8-PeCDF	µg/kg	0.015	Human Health (ca)
1746-01-6	2,3,7,8-TCDD	µg/kg	0.0025	Ecological (Recommended by Partner Agencies)
51207-31-9	2,3,7,8-TCDF	µg/kg	0.045	Human Health (ca)
3268-87-9	OCDD	µg/kg	15	Human Health (ca)
39001-02-0	OCDF	µg/kg	15	Human Health (ca)
WHODIOXTEQ(B)	Total WHO Dioxin TEQ(Bird)	µg/kg	NA	NA
WHODIOXTEQ(F)	Total WHO Dioxin TEQ(Fish)	µg/kg	NA	NA
WHODIOXTEQ(H)	Total WHO Dioxin TEQ(Human/Mammal)	µg/kg	NA	NA
Inorganics (mg/kg)				
7429-90-5	Aluminum	mg/kg	7700	Human Health (nc)
7440-36-0	Antimony	mg/kg	3.1	Human Health (nc)
7440-38-2	Arsenic	mg/kg	0.39	Human Health (ca)
7440-39-3	Barium	mg/kg	48	Ecological (NJDEP ESC Table - Marine)
7440-41-7	Beryllium	mg/kg	16	Human Health (nc)
7440-43-9	Cadmium	mg/kg	1.2	Ecological (NJDEP ESC Table - Marine)
7440-70-2	Calcium	mg/kg	NA	NA
7440-47-3	Chromium	mg/kg	81	Ecological (Recommended by Partner Agencies)
7440-48-4	Cobalt	mg/kg	2.3	Human Health (nc)
7440-50-8	Copper	mg/kg	13.318	Ecological (Recommended by Partner Agencies)
7439-96-5-DIV	Divalent Manganese	mg/kg	NA	NA
18540-29-9	Hexavalent Chromium	mg/kg	0.29	Human Health (ca)
7439-89-6	Iron	mg/kg	5500	Human Health (nc)
7439-92-1	Lead	mg/kg	10.606	Ecological (Recommended by Partner Agencies)
7439-95-4	Magnesium	mg/kg	NA	NA
7439-96-5	Manganese	mg/kg	180	Human Health (nc)
7439-97-6	Mercury	mg/kg	0.037	Ecological (Recommended by Partner Agencies)
7440-02-0	Nickel	mg/kg	21	Ecological (NJDEP ESC Table - Marine)
7440-09-7	Potassium	mg/kg	NA	NA
7782-49-2	Selenium	mg/kg	1	Ecological (NJDEP ESC Table - Marine)
7440-22-4	Silver	mg/kg	1	Ecological (NJDEP ESC Table - Marine)
7440-23-5	Sodium	mg/kg	NA	NA
7440-28-0	Thallium	mg/kg	0.078	Human Health (nc)
7440-32-6	Titanium	mg/kg	14000	Human Health (nc)
7440-62-2	Vanadium	mg/kg	39	Human Health (nc)
7440-66-6	Zinc	mg/kg	6.62	Ecological (Recommended by Partner Agencies)
1191-48-6	Dibutyltin	µg/kg	1.8	Human Health (nc)
78763-54-9	Monobutyltin	µg/kg	1.8	Human Health (nc)
1461-25-2	Tetrabutyltin	µg/kg	1.8	Human Health (nc)
688-73-3	Tributyltin	µg/kg	1.8	Human Health (nc)
REAC-CN	Reactive Cyanide	µg/kg	NA	NA
57-12-5	Total Cyanide	µg/kg	2.2	Human Health (nc)
Pesticides/Herbicides (µg/kg)				
93-76-5	2,4,5-T	µg/kg	12300	Ecological (USEPA Reg 3 - Freshwater)
93-72-1	2,4,5-TP (Silvex)	µg/kg	675	Ecological (USEPA Reg 3 - Freshwater)
94-75-7	2,4-D	µg/kg	1273	Ecological (USEPA Reg 5)
94-82-6	2,4-DB	µg/kg	49000	Human Health (nc)
53-19-0	2,4'-DDD	µg/kg	2000	Human Health (ca)
3424-82-6	2,4'-DDE	µg/kg	1400	Human Health (ca)
789-02-6	2,4'-DDT	µg/kg	1700	Human Health (ca)
72-54-8	4,4'-DDD	µg/kg	2	Ecological (NJDEP ESC Table - Marine)
72-55-9	4,4'-DDE	µg/kg	2.2	Ecological (NJDEP ESC Table - Marine)
50-29-3	4,4'-DDT	µg/kg	1	Ecological (NJDEP ESC Table - Marine)

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Screening Values for Sediment

CAS Number	Analyte	Result Units	Screening Value	Basis
309-00-2	Aldrin	µg/kg	2	Ecological (Recommended by Partner Agencies)
319-84-6	Alpha-BHC	µg/kg	6	Ecological (NJDEP ESC Table - Freshwater)
5103-71-9	Alpha-Chlordane	µg/kg	3.24	Ecological (Recommended by Partner Agencies)
319-85-7	Beta-BHC	µg/kg	5	Ecological (NJDEP ESC Table - Freshwater)
5013-74-2	Beta-Chlordane	µg/kg	3.24	Ecological (Recommended by Partner Agencies)
57-74-9	Chlordane	µg/kg	3.24	Ecological (Recommended by Partner Agencies)
319-86-8	Delta-BHC	µg/kg	3	Ecological (NJDEP ESC Table - Freshwater)
60-57-1	Dieldrin	µg/kg	30	Human Health (ca)
959-98-8	Endosulfan I	µg/kg	2.9	Ecological (USEPA Reg 3 - Freshwater)
33213-65-9	Endosulfan II	µg/kg	14	Ecological (USEPA Reg 3 - Freshwater)
1031-07-8	Endosulfan sulfate	µg/kg	34.6	Ecological (NJDEP ESC Table - Freshwater)
72-20-8	Endrin	µg/kg	3	Ecological (Recommended by Partner Agencies)
7421-93-4	Endrin Aldehyde	µg/kg	480	Ecological (NJDEP ESC Table - Freshwater)
53494-70-5	Endrin Ketone	µg/kg	1800	Human Health (nc)
58-89-9	Gamma-BHC (Lindane)	µg/kg	3	Ecological (NJDEP ESC Table - Freshwater)
5566-34-7	Gamma-Chlordane	µg/kg	3.24	Ecological (Recommended by Partner Agencies)
76-44-8	Heptachlor	µg/kg	0.68	Ecological (Recommended by Partner Agencies)
1024-57-3	Heptachlor epoxide	µg/kg	5	Ecological (NJDEP ESC Table - Freshwater)
72-43-5	Methoxychlor	µg/kg	13.6	Ecological (Recommended by Partner Agencies)
TBHC	Total BHC	µg/kg	3	Ecological (Recommended by Partner Agencies)
TDDT-24-44	Total DDT (2,4 & 4,4)	µg/kg	1700	Human Health (ca)
TDDT-24	Total DDT (2,4)	µg/kg	1700	Human Health (ca)
TDDT-44	Total DDT (4,4)	µg/kg	1.58	Ecological (Recommended by Partner Agencies)
8001-35-2	Toxaphene	µg/kg	0.077	Ecological (NJDEP ESC Table - Freshwater)
TOT_AGChlor	Total Alpha + Gamma Chlordane	µg/kg	3.24	Ecological (Recommended by Partner Agencies)
PCBs (µg/kg)				
2051-60-7	PCB-1	µg/kg	NA	NA
39485-83-1	PCB-100	µg/kg	NA	NA
60145-21-3	PCB-103	µg/kg	NA	NA
56558-16-8	PCB-104	µg/kg	NA	NA
32598-14-4	PCB-105	µg/kg	110	Human Health (ca)
PCB-106/118	PCB-106/118	µg/kg	NA	NA
70424-68-9/7447	PCB-107/109	µg/kg	NA	NA
70362-41-3/7447	PCB-108/112	µg/kg	NA	NA
2050-67-1	PCB-11	µg/kg	NA	NA
38380-03-9	PCB-110	µg/kg	NA	NA
PCB-111/115	PCB-111/115	µg/kg	NA	NA
68194-10-5	PCB-113	µg/kg	NA	NA
74472-37-0	PCB-114	µg/kg	110	Human Health (ca)
56558-17-9	PCB-119	µg/kg	NA	NA
PCB-12/13	PCB-12/13	µg/kg	NA	NA
68194-12-7	PCB-120	µg/kg	NA	NA
56558-18-0	PCB-121	µg/kg	NA	NA
76842-07-4	PCB-122	µg/kg	NA	NA
65510-44-3	PCB-123	µg/kg	110	Human Health (ca)
70424-70-3	PCB-124	µg/kg	NA	NA
57465-28-8	PCB-126	µg/kg	0.034	Human Health (ca)
39635-33-1	PCB-127	µg/kg	NA	NA
PCB-128/162	PCB-128/162	µg/kg	NA	NA
55215-18-4	PCB-129	µg/kg	NA	NA
52663-66-8	PCB-130	µg/kg	NA	NA
61798-70-7	PCB-131	µg/kg	NA	NA
38380-05-1/7447	PCB-132/161	µg/kg	NA	NA
PCB-133/142	PCB-133/142	µg/kg	NA	NA
PCB-134/143	PCB-134/143	µg/kg	NA	NA
52744-13-5	PCB-135	µg/kg	NA	NA
38411-22-2	PCB-136	µg/kg	NA	NA
35694-06-5	PCB-137	µg/kg	NA	NA
35065-28-2/7447	PCB-138/163/164	µg/kg	NA	NA
PCB-139/149	PCB-139/149	µg/kg	NA	NA
34883-41-5	PCB-14	µg/kg	NA	NA
59291-64-4	PCB-140	µg/kg	NA	NA
52712-04-6	PCB-141	µg/kg	NA	NA
68194-14-9	PCB-144	µg/kg	NA	NA
74472-40-5	PCB-145	µg/kg	NA	NA

Screening Values for Sediment

CAS Number	Analyte	Result Units	Screening Value	Basis
51908-16-8/7447	PCB-146/165	µg/kg	NA	NA
68194-13-8	PCB-147	µg/kg	NA	NA
74472-41-6	PCB-148	µg/kg	NA	NA
2050-68-2	PCB-15	µg/kg	NA	NA
68194-08-1	PCB-150	µg/kg	NA	NA
52663-63-5	PCB-151	µg/kg	NA	NA
68194-09-2	PCB-152	µg/kg	NA	NA
35065-27-1	PCB-153	µg/kg	NA	NA
60145-22-4	PCB-154	µg/kg	NA	NA
33979-03-2	PCB-155	µg/kg	NA	NA
38380-08-4	PCB-156	µg/kg	110	Human Health (ca)
69782-90-7	PCB-157	µg/kg	110	Human Health (ca)
74472-42-7/4141	PCB-158/160	µg/kg	NA	NA
39635-35-3	PCB-159	µg/kg	NA	NA
PCB-16/32	PCB-16/32	µg/kg	NA	NA
41411-63-6	PCB-166	µg/kg	NA	NA
52663-72-6	PCB-167	µg/kg	110	Human Health (ca)
59291-65-5	PCB-168	µg/kg	NA	NA
32774-16-6	PCB-169	µg/kg	0.11	Human Health (ca)
37680-66-3	PCB-17	µg/kg	NA	NA
35065-30-6	PCB-170	µg/kg	NA	NA
52663-71-5	PCB-171	µg/kg	NA	NA
52663-74-8	PCB-172	µg/kg	NA	NA
68194-16-1	PCB-173	µg/kg	NA	NA
38411-25-5	PCB-174	µg/kg	NA	NA
40186-70-7	PCB-175	µg/kg	NA	NA
52663-65-7	PCB-176	µg/kg	NA	NA
52663-70-4	PCB-177	µg/kg	NA	NA
52663-67-9	PCB-178	µg/kg	NA	NA
52663-64-6	PCB-179	µg/kg	NA	NA
37680-65-2	PCB-18	µg/kg	NA	NA
35065-29-3	PCB-180	µg/kg	NA	NA
74472-47-2	PCB-181	µg/kg	NA	NA
PCB-182/187	PCB-182/187	µg/kg	NA	NA
52663-69-1	PCB-183	µg/kg	NA	NA
74472-48-3	PCB-184	µg/kg	NA	NA
52712-05-7	PCB-185	µg/kg	NA	NA
74472-49-4	PCB-186	µg/kg	NA	NA
74487-85-7	PCB-188	µg/kg	NA	NA
39635-31-9	PCB-189	µg/kg	110	Human Health (ca)
38444-73-4	PCB-19	µg/kg	NA	NA
41411-64-7	PCB-190	µg/kg	NA	NA
74472-50-7	PCB-191	µg/kg	NA	NA
74472-51-8	PCB-192	µg/kg	NA	NA
69782-91-8	PCB-193	µg/kg	NA	NA
35694-08-7	PCB-194	µg/kg	NA	NA
52663-78-2	PCB-195	µg/kg	NA	NA
PCB-196/203	PCB-196/203	µg/kg	NA	NA
33091-17-7	PCB-197	µg/kg	NA	NA
68194-17-2	PCB-198	µg/kg	NA	NA
52663-75-9	PCB-199	µg/kg	NA	NA
2051-61-8	PCB-2	µg/kg	NA	NA
38444-84-7/5570	PCB-20/21/33	µg/kg	NA	NA
52663-73-7	PCB-200	µg/kg	NA	NA
40186-71-8	PCB-201	µg/kg	NA	NA
2136-99-4	PCB-202	µg/kg	NA	NA
74472-52-9	PCB-204	µg/kg	NA	NA
74472-53-0	PCB-205	µg/kg	NA	NA
40186-72-9	PCB-206	µg/kg	NA	NA
52663-79-3	PCB-207	µg/kg	NA	NA
52663-77-1	PCB-208	µg/kg	NA	NA
2051-24-3	PCB-209	µg/kg	NA	NA
38444-85-8	PCB-22	µg/kg	NA	NA
55720-44-0	PCB-23	µg/kg	NA	NA
PCB-24/27	PCB-24/27	µg/kg	NA	NA

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Screening Values for Sediment

CAS Number	Analyte	Result Units	Screening Value	Basis
55712-37-3	PCB-25	µg/kg	NA	NA
38444-81-4	PCB-26	µg/kg	NA	NA
7012-37-5	PCB-28	µg/kg	NA	NA
15862-07-4	PCB-29	µg/kg	NA	NA
2051-62-9	PCB-3	µg/kg	NA	NA
35693-92-6	PCB-30	µg/kg	NA	NA
16606-02-3	PCB-31	µg/kg	NA	NA
37680-68-5	PCB-34	µg/kg	NA	NA
37680-69-6	PCB-35	µg/kg	NA	NA
38444-87-0	PCB-36	µg/kg	NA	NA
38444-90-5	PCB-37	µg/kg	NA	NA
53555-66-1	PCB-38	µg/kg	NA	NA
38444-88-1	PCB-39	µg/kg	NA	NA
PCB-4/10	PCB-4/10	µg/kg	NA	NA
38444-93-8	PCB-40	µg/kg	NA	NA
52663-59-9/5266	PCB-41/64/71/72	µg/kg	NA	NA
36559-22-5/7447	PCB-42/59	µg/kg	NA	NA
PCB-43/49	PCB-43/49	µg/kg	NA	NA
41464-39-5	PCB-44	µg/kg	NA	NA
70362-45-7	PCB-45	µg/kg	NA	NA
41464-47-5	PCB-46	µg/kg	NA	NA
2437-79-8	PCB-47	µg/kg	NA	NA
70362-47-9/3259	PCB-48/75	µg/kg	NA	NA
PCB-5/8	PCB-5/8	µg/kg	NA	NA
62796-65-0	PCB-50	µg/kg	NA	NA
68194-04-7	PCB-51	µg/kg	NA	NA
35693-99-3/6023	PCB-52/69	µg/kg	NA	NA
41464-41-9	PCB-53	µg/kg	NA	NA
15968-05-5	PCB-54	µg/kg	NA	NA
74338-24-2	PCB-55	µg/kg	NA	NA
PCB-56/60	PCB-56/60	µg/kg	NA	NA
70424-67-8	PCB-57	µg/kg	NA	NA
41464-49-7	PCB-58	µg/kg	NA	NA
25569-80-6	PCB-6	µg/kg	NA	NA
PCB-61/70	PCB-61/70	µg/kg	NA	NA
54230-22-7	PCB-62	µg/kg	NA	NA
74472-34-7	PCB-63	µg/kg	NA	NA
33284-54-7	PCB-65	µg/kg	NA	NA
73575-53-8	PCB-67	µg/kg	NA	NA
73575-52-7	PCB-68	µg/kg	NA	NA
PCB-7/9	PCB-7/9	µg/kg	NA	NA
74338-23-1	PCB-73	µg/kg	NA	NA
32690-93-0	PCB-74	µg/kg	NA	NA
PCB-76/66	PCB-76/66	µg/kg	NA	NA
32598-13-3	PCB-77	µg/kg	34	Human Health (ca)
70362-49-1	PCB-78	µg/kg	NA	NA
41464-48-6	PCB-79	µg/kg	NA	NA
33284-52-5	PCB-80	µg/kg	NA	NA
70362-50-4	PCB-81	µg/kg	11	Human Health (ca)
52663-62-4	PCB-82	µg/kg	NA	NA
60145-20-2	PCB-83	µg/kg	NA	NA
52663-60-2/5266	PCB-84/92	µg/kg	NA	NA
PCB-85/116	PCB-85/116	µg/kg	NA	NA
55312-69-1	PCB-86	µg/kg	NA	NA
38380-02-8/6819	PCB-87/117/125	µg/kg	NA	NA
PCB-88/91	PCB-88/91	µg/kg	NA	NA
73575-57-2	PCB-89	µg/kg	NA	NA
PCB-90/101	PCB-90/101	µg/kg	NA	NA
73575-56-1	PCB-93	µg/kg	NA	NA
73575-55-0	PCB-94	µg/kg	NA	NA
38379-99-6/6023	PCB-95/98/102	µg/kg	NA	NA
73575-54-9	PCB-96	µg/kg	NA	NA
41464-51-1	PCB-97	µg/kg	NA	NA
38380-01-7	PCB-99	µg/kg	NA	NA
12674-11-2	Aroclor-1016	µg/kg	7	Ecological (NJDEP ESC Table - Freshwater)

Screening Values for Sediment

CAS Number	Analyte	Result Units	Screening Value	Basis
11104-28-2	Aroclor-1221	µg/kg	140	Human Health (ca)
11141-16-5	Aroclor-1232	µg/kg	140	Human Health (ca)
53469-21-9	Aroclor-1242	µg/kg	220	Human Health (ca)
12672-29-6	Aroclor-1248	µg/kg	30	Ecological (NJDEP ESC Table - Freshwater)
11097-69-1	Aroclor-1254	µg/kg	60	Ecological (NJDEP ESC Table - Freshwater)
11096-82-5	Aroclor-1260	µg/kg	5	Ecological (NJDEP ESC Table - Freshwater)
37324-23-5	Aroclor-1262	µg/kg	NA	NA
11100-14-4	Aroclor-1268	µg/kg	NA	NA
TOT_PCB_ARO(7)	Total Aroclor PCBs (Sum of 7 Aroclors)	µg/kg	22.7	Ecological (Recommended by Partner Agencies)
TOT_PCB_ARO(9)	Total Aroclor PCBs (Sum of 9 Aroclors)	µg/kg	22.7	Ecological (Recommended by Partner Agencies)
TPCB Cong-209	Total PCB Congeners (209)	µg/kg	22.7	Ecological (Recommended by Partner Agencies)
TPCB CongNOAA89	Total PCB Congeners (NOAA 1989)	µg/kg	220	Human Health (ca)
WHOPCBTEQ(B)	Total PCB TEQ (Bird)	µg/kg	NA	NA
WHOPCBTEQ(F)	Total PCB TEQ (Fish)	µg/kg	NA	NA
WHOPCBTEQ(H)	Total PCB TEQ (Human/Mammal)	µg/kg	NA	NA
Semivolatile Organic Compounds (µg/kg)				
95-94-3	1,2,4,5-Tetrachlorobenzene	µg/kg	1252	Ecological (NJDEP ESC Table - Freshwater)
120-82-1	1,2,4-Trichlorobenzene	µg/kg	4.8	Ecological (NJDEP ESC Table - Marine)
95-50-1	1,2-Dichlorobenzene	µg/kg	13	Ecological (NJDEP ESC Table - Marine)
541-73-1	1,3-Dichlorobenzene	µg/kg	1315	Ecological (NJDEP ESC Table - Freshwater)
106-46-7	1,4-Dichlorobenzene	µg/kg	110	Ecological (NJDEP ESC Table - Marine)
90-12-0	1-Methylnaphthalene	µg/kg	16000	Human Health (ca)
540-54-5	2,2-oxybis(1-Chloropropane)	µg/kg	NA	NA
58-90-2	2,3,4,6-Tetrachlorophenol	µg/kg	284	Ecological (USEPA Reg 3 - Freshwater)
95-95-4	2,4,5-Trichlorophenol	µg/kg	3	Ecological (NJDEP ESC Table - Marine)
88-06-2	2,4,6-Trichlorophenol	µg/kg	6	Ecological (NJDEP ESC Table - Marine)
120-83-2	2,4-Dichlorophenol	µg/kg	5	Ecological (NJDEP ESC Table - Marine)
105-67-9	2,4-Dimethylphenol	µg/kg	304	Ecological (NJDEP ESC Table - Freshwater)
51-28-5	2,4-Dinitrophenol	µg/kg	6.21	Ecological (NJDEP ESC Table - Freshwater)
121-14-2	2,4-Dinitrotoluene	µg/kg	14.4	Ecological (NJDEP ESC Table - Freshwater)
606-20-2	2,6-Dinitrotoluene	µg/kg	39.8	Ecological (USEPA Reg 5)
91-58-7	2-Chloronaphthalene	µg/kg	417	Ecological (NJDEP ESC Table - Freshwater)
95-57-8	2-Chlorophenol	µg/kg	8	Ecological (NJDEP ESC Table - Marine)
91-57-6	2-Methylnaphthalene	µg/kg	70	Ecological (NJDEP ESC Table - Marine)
95-48-7	2-Methylphenol	µg/kg	55.4	Ecological (USEPA Reg 5)
88-74-4	2-Nitroaniline	µg/kg	61000	Human Health (nc)
88-75-5	2-Nitrophenol	µg/kg	NA	NA
108-39-4/106-44	3&4-Methylphenol	µg/kg	670	Ecological (USEPA Reg 3 - Freshwater)
91-94-1	3,3'-Dichlorobenzidine	µg/kg	127	Ecological (NJDEP ESC Table - Freshwater)
99-09-2	3-Nitroaniline	µg/kg	24000	Human Health (ca)
534-52-1	4,6-Dinitro-2-methylphenol	µg/kg	104	Ecological (USEPA Reg 5)
101-55-3	4-Bromophenyl phenyl ether	µg/kg	1230	Ecological (USEPA Reg 3 - Freshwater)
59-50-7	4-Chloro-3-Methylphenol	µg/kg	388	Ecological (USEPA Reg 5)
106-47-8	4-Chloroaniline	µg/kg	146	Ecological (USEPA Reg 5)
7005-72-3	4-Chlorophenyl phenyl ether	µg/kg	NA	NA
106-44-5	4-Methylphenol	µg/kg	670	Ecological (USEPA Reg 3 - Freshwater)
100-01-6	4-Nitroaniline	µg/kg	24000	Human Health (ca)
100-02-7	4-Nitrophenol	µg/kg	13.3	Ecological (NJDEP ESC Table - Freshwater)
83-32-9	Acenaphthene	µg/kg	16	Ecological (NJDEP ESC Table - Marine)
208-96-8	Acenaphthylene	µg/kg	44	Ecological (NJDEP ESC Table - Marine)
98-86-2	Acetophenone	µg/kg	NA	NA
120-12-7	Anthracene	µg/kg	85	Ecological (NJDEP ESC Table - Marine)
1912-24-9	Atrazine	µg/kg	6.62	Ecological (USEPA Reg 3 - Freshwater)
100-52-7	Benzaldehyde	µg/kg	NA	NA
56-55-3	Benzo(a)anthracene	µg/kg	150	Human Health (ca)
50-32-8	Benzo(a)pyrene	µg/kg	15	Human Health (ca)
205-99-2	Benzo(b)fluoranthene	µg/kg	150	Human Health (ca)
192-97-2	Benzo(e)pyrene	µg/kg	170000	Human Health (nc)
191-24-2	Benzo(g,h,i)perylene	µg/kg	170	Ecological (NJDEP ESC Table - Freshwater)
207-08-9	Benzo(k)fluoranthene	µg/kg	240	Ecological (NJDEP ESC Table - Freshwater)
92-52-4	Biphenyl	µg/kg	1220	Ecological (USEPA Reg 3 - Freshwater)
111-91-1	bis(2-Chloroethoxy)methane	µg/kg	18000	Human Health (nc)
111-44-4	bis(2-Chloroethyl)ether	µg/kg	210	Human Health (ca)
117-81-7	bis(2-ethylhexyl phthalate)	µg/kg	182.16	Ecological (Recommended by Partner Agencies)
85-68-7	Butyl benzyl phthalate	µg/kg	63	Ecological (NJDEP ESC Table - Marine)

Screening Values for Sediment

CAS Number	Analyte	Result Units	Screening Value	Basis
105-60-2	Caprolactam	µg/kg	NA	NA
86-74-8	Carbazole	µg/kg	NA	NA
218-01-9	Chrysene	µg/kg	384	Ecological (NJDEP ESC Table - Marine)
53-70-3	Dibenzo(a,h)anthracene	µg/kg	15	Human Health (ca)
132-64-9	Dibenzofuran	µg/kg	7300	Ecological (USEPA Reg 3 - Marine)
132-65-0	Dibenzothiophene	µg/kg	NA	NA
84-66-2	Diethyl phthalate	µg/kg	6	Ecological (NJDEP ESC Table - Marine)
131-11-3	Dimethylphthalate	µg/kg	4900000	Human Health (nc)
84-74-2	Di-n-Butylphthalate	µg/kg	58	Ecological (NJDEP ESC Table - Marine)
117-84-0	Di-n-Octylphthalate	µg/kg	40600	Ecological (USEPA Reg 5)
206-44-0	Fluoranthene	µg/kg	600	Ecological (NJDEP ESC Table - Marine)
86-73-7	Fluorene	µg/kg	19	Ecological (NJDEP ESC Table - Marine)
118-74-1	Hexachlorobenzene	µg/kg	20	Ecological (Recommended by Partner Agencies)
87-68-3	Hexachlorobutadiene	µg/kg	1.3	Ecological (NJDEP ESC Table - Marine)
77-47-4	Hexachlorocyclopentadiene	µg/kg	901	Ecological (NJDEP ESC Table - Freshwater)
67-72-1	Hexachloroethane	µg/kg	73	Ecological (NJDEP ESC Table - Marine)
193-39-5	Indeno(1,2,3-cd)pyrene	µg/kg	150	Human Health (ca)
78-59-1	Isophorone	µg/kg	432	Ecological (NJDEP ESC Table - Freshwater)
91-20-3	Naphthalene	µg/kg	160	Ecological (NJDEP ESC Table - Marine)
98-95-3	Nitrobenzene	µg/kg	145	Ecological (NJDEP ESC Table - Freshwater)
621-64-7	N-Nitroso-di-n-propylamine	µg/kg	69	Human Health (ca)
86-30-6	N-Nitrosodiphenylamine	µg/kg	99000	Human Health (ca)
87-86-5	Pentachlorophenol	µg/kg	17	Ecological (NJDEP ESC Table - Marine)
198-55-0	Perylene	µg/kg	170000	Human Health (nc)
85-01-8	Phenanthrene	µg/kg	240	Ecological (NJDEP ESC Table - Marine)
108-95-2	Phenol	µg/kg	130	Ecological (NJDEP ESC Table - Marine)
129-00-0	Pyrene	µg/kg	665	Ecological (NJDEP ESC Table - Marine)
110-86-1	Pyridine	µg/kg	106	Ecological (USEPA Reg 5)
THMW PAHs-SVOC	Total HMW PAHs-SVOC	µg/kg	170	Human Health (nc)
TLMW PAHs-SVOC	Total LMW PAHs-SVOC	µg/kg	170	Human Health (nc)
TOT_PAH_SVOC	TOTAL PAHs - SVOC	µg/kg	170	Human Health (nc)
TEPH	TEPH	µg/kg	NA	NA
TPH	Total Petroleum Hydrocarbons	µg/kg	NA	NA

Ecological screening values selected, based on the following hierarchy:

1. Recommended by Partner Agencies: Ecological sediment screening levels for saline waters —as recommended by the Partner Agencies for the lower Passaic River in email correspondence to Scott Kirchner of CDM dated May 26, 2011.
2. NJDEP ESC Table - Marine: NJDEP Ecological Screening Criteria (ESC) for Marine Sediment. March 2009.
3. NJDEP ESC Table - Freshwater: NJDEP Ecological Screening Criteria (ESC) for Freshwater Sediment. March 2009.
4. USEPA Reg 3 - Marine: USEPA Region 3 Marine Sediment Screening Benchmarks. July 2006.
5. USEPA Reg 3 - Freshwater: USEPA Region 3 Freshwater Sediment Screening Benchmarks. August 2006.
6. USEPA Reg 5: USEPA Region 5 Resource Conservation and Recovery Act (RCRA) Ecological Screening Levels. August 2003.

FOIA_07123_0003552_0474

Appendix M

Laboratory SOPs

Title: HOMOGENIZATION OF BIOTA & TISSUE

Approval Signatures:

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1.0 Scope and Application

This SOP describes the laboratory procedure for the whole body homogenization and extraction of biota and tissue samples by tissuemizer in preparation for analysis by a variety of chromatographic procedures.

NOTE: Pre-preparation of tissue such as dissection, sportsman filet, sample compositing, shucking, etc. may be performed by the laboratory per customer specification. These procedures are project specific and instructions are not included in this SOP.

2.0 Summary of Method

2.1 Homogenization

Tissue samples are homogenized using a titanium blade homogenizer. Biota samples are homogenized using stainless steel knives and/or a food processor. The homogenized sample(s) are transferred to labeled glass jars and stored in a freezer maintained at a temperature of -15°C (±5°C) in preparation for extraction.

The laboratory's standard procedure is to perform whole body homogenization of the tissue sample. Any customer specifications for dissection or homogenization of certain parts of the tissue sample, compositing multiple samples or other must be negotiated with the laboratory during project initiation and specific instructions for sample processing must be prepared and provided to the extraction laboratory by the laboratory PM. In the absence of instruction, the laboratory will homogenize the entire sample.

The homogenization procedure is a laboratory developed procedure based on the procedures described in the Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality.

2.2 Extraction

A portion of homogenized sample is mixed with anhydrous sodium sulfate then macerated for 3 minutes in an appropriate extraction solvent using the Tissumizer. The solvent layer decanted poured through sodium sulfate and collected in a collection vessel. The extraction is repeated two more times with fresh portions of extraction solvent. After extraction, the combined extracts are concentrated to an appropriate final volume using K-D Technique. Percent lipids are determined following procedures given in laboratory SOP BR-EX-016 Percent Lipid Determination and extract cleanup is performed when necessary.

The extraction procedure is a laboratory developed procedure based on the procedures described in the Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality.

3.0 Definitions

- Biota: flora and fauna. For this SOP, all reference to "biota" refers to plant material.

- Tissue: an aggregate of cells usually of a particular kind together with their intercellular substance that form one of the structural materials of a plant or animal. For this SOP, all reference to "tissue" refers to structural materials from an animal.

A list of general terms and definitions are provided in Appendix A.

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP BR-EX-017 Glassware Cleaning, and rinsed with acetone and methylene chloride prior to use.

5.0 Safety

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

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Homogenization Equipment

- Cutting Board- High density polyethylene 16X23"

- Homogenizer equipped with 55 mm Titanium Blade Omni International or equivalent.
- Food Processor Cuisinart or equivalent
- Stainless steel knives
- Glass Jars, wide mouth; 125 mL-1000 mL. ESS or equivalent.
- Meat Grinder

6.2 Extraction Equipment

- Tissuemizer equipped with a 20 mm x 195 Generator probe. Omni International PowerGen 700 or equivalent.
- Filter Funnels – 100 mm diameter for filtration/drying. Fisher Scientific or equivalent.
- No. 54 Filter paper. Whatman 18.5 cm, or equivalent.
- Beakers – 400 mL. Fisher Scientific or equivalent.

6.3 Extract Concentration (KD Apparatus)

- Concentrator Tube, 10 mL Graduated: ChemGlass Catalog # CG-1316-11 or equivalent
- Snyder Column: Three ball macro, AMK Catalog # SC2-01 or equivalent
- Snyder Column: Two ball micro, AMK Catalog # SC3-01 or equivalent
- Evaporation Flask: 500 mL attached to concentrator tube with clip, AMK Catalog Number KDF-500 or equivalent.
- Boiling Chips: Silicon carbide, approximately 10/40 mesh, solvent extracted in methylene chloride, Troemner Catalog # 133B or equivalent.
- Heating Mantle: Rheostat controlled for water bath capable of temperature control (±5°C). ChemGlass Catalog # PL3122 or equivalent.
- Water Bath, capable of temperature control to ±5°C. Barnstead Corporation Catalog # HM0500-HS1 or equivalent.
- Solvent Vapor Recovery System, Kontes K-54000-1006, K-547300-000, Ace Glass Catalog # 6614-30 or equivalent.

6.4 Miscellaneous

- Disposable Glass Pasteur Pipette and bulb: Fisher Scientific or equivalent.
- Top Loading balance: Capable of measuring to 0.01 gram accuracy, Mettler Model # PM4800 or equivalent.
- Vials and caps: 2, 4, 8, and 16 mL with Teflon lined septa and screw caps, Fisher Scientific or equivalent.
- Teflon and Stainless Steel Spatulas, Fisher Scientific or equivalent.
- Adjustable Pipette: Finnpipette or equivalent
- 0.5 mL – 2.0 mL Hamilton Gastight® syringes or equivalent.
- Paper towels

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate (Na_2SO_4), Granular Anhydrous: J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours.
- Methylene Chloride (CH_2C_{12}): Pesticide Quality, J.T Baker or equivalent.

- Hexane, (C_6H_{14}): Pesticide Quality, J.T. Baker or equivalent.
- Acetone, ($(CH_3)_2CO$): Pesticide Quality, J.T. Baker or equivalent.
- Reagent Water: RO water filtered through a Nanopure System.
- Alkaline Liquid Detergent: Contrex or equivalent.

Methylene Chloride/Acetone (1:1): In a 4 L amber glass bottle mix 2 L methylene chloride with 2 L acetone. Store the solution in a fume hood. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

Hexane /Acetone (1:1): In a 4 L amber glass bottle mix 2 L hexane with 2 L acetone. Store the solution in a fume hood. Assign an expiration date of 6 months from date of preparation unless the parent material expires earlier, in which case, use the earliest expiration date.

7.2 Standards

Purchase certified stock standards from commercial vendors and from these prepare surrogate and spiking solutions by diluting a known volume of the stock standard solutions in an appropriate solvent. Record the preparation of all standards in the LIMS module. The formulations for the preparation of surrogate and spiking standards are provided in analytical SOPs.

Unless otherwise specified in the analytical SOP store prepared in glass containers at 4°C or below and assign an expiration date of 6 months from the date of preparation unless the parent standards expire earlier in which case use the earliest expiration date.

Assay surrogate and spike solutions before each use to verify the made to concentration is within specifications. Maintain records of the assay per the procedure established for the work section.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP.

The laboratory recommends that tissue and biota samples be collected in glass jars or sealable plastic bags. Immediately following collection, biota samples should be iced to a temperature of 4°C (±2°C) and tissue samples should be frozen and maintained at a temperature of -15°C (±5°C) until the time of homogenization. After homogenization, all homogenized samples must be stored in a freezer maintained at a temperature of -15°C (±5°C).

Tissue and biota samples must be extracted within the holding time specified in the client's quality assurance plan. In the absence of client specifications, the following HT will be used:

SVOA GC/MS: 14 Days from Date of Collection
Pesticides: 14 Days from Date of Collection
PCB: 365 Days from Date of Collection
Mercury: 28 Days from Date of Collection
Metals: 180 Days from Date of Collection

After extraction any remaining sample should be returned immediately to the freezer or stored per the project specifications.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	AcceptanceCriteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Analytical SOP
Matrix Spike(s) MS/MSD	Client Request .	See Analytical SOP
Sample Duplicate (SD)	Client Request	See Analytical SOP

10.0 Procedure

10.1 Instrument Calibration

Check the calibration of the balance each day of use prior to use.

Check the calibration of the adjustable pipettes quarterly.

Perform periodic maintenance on the tissuemizer's generator probe as necessary. Maintenance may include but is not limited to the replacement of Teflon bearings and rotor shafts when a loud squealing noise is heard. Refer to the PowerGen 700 Homogenizer Instruction Manual for further guidance and for the manufacturer's recommended maintenance program.

10.2 Whole Body Homogenization of Tissue Samples

Remove the samples from storage and let the sample(s) thaw completely.

Wash all equipment with detergent and hot water, then rinse with reagent water and acetone prior to use and also after each sample.

Prepare a equipment blank to ensure the equipment is clean. To prepare the blank, transfer a representative amount of reagent water (similar to the size of the samples) to a clear glass jar. Let the water come into contact with all equipment that will be used to homogenize the samples. This process should be done randomly during homogenization to verify the cleaning process.

If possible obtain a glass jar large enough to accommodate the entire sample. Label the jar with the sample's lab ID and place the jar on the analytical balance. Tare the balance. Put on a pair of nitrile gloves and with your hands transfer the sample from the storage container to the labeled jar. Record the pre-homogenization weight of the sample in the worksheet.

Transfer the sample to a pre-cleaned cutting board. Cut the sample into 1-3" sections using a stainless steel knife then place the sections of samples in the labeled jar.

Insert the titanium blade into the jar and homogenize the sample at 2000-4000 RPM until the sample becomes slurry. Manual mixing with a stainless steel or Teflon spatula may be required

to insure complete homogenization. Remove the blade from the sample jar and scrape any remaining sample from the blade into the labeled jar. Place the jar on the top-loading balance and record the post homogenization weight in the worksheet.

NOTE: Samples greater than 12 inches or samples with weight measurements that exceed 1-2 pounds should be homogenized in a stainless steel meat grinder prior to use of the titanium blade. If the sample is extremely large homogenization with a knife may be necessary. If the homogenized tissue sample cannot fit into a single container, pass the sample through the meat grinder multiple times, homogenize the slurry and transfer to multiple containers.

10.3 Homogenization of Biota Samples

Wash all equipment with detergent and hot water, then rinse with reagent water and acetone prior to use and also after each sample.

Prepare a equipment blank to ensure the equipment is clean. To prepare the blank, transfer a representative amount of reagent water (similar to the size of the samples) to a clear glass jar. Let the water come into contact with all equipment that will be used to homogenize the samples. This process should be done randomly during homogenization to verify the cleaning process.

Remove the samples from storage and let warm to ambient temperature. Select a glass jar large enough to accommodate the entire sample. Label the jar with the sample's lab ID and place the jar on the top-loading balance. Tare the balance. Remove the biota sample from the storage container and place in the labeled jar. Record the pre-homogenization weight of the sample into the worksheet.

Remove the sample and place on a pre-cleaned cutting board. Slice the material into very fine sections using a stainless steel knife or a food processor. Return homogenized sample back into the jar and place on the top-loading balance. Record the post homogenization weight into the worksheet.

10.4 Extraction

Clean all glassware prior to use following the procedure given in laboratory SOP BR-EX-017. Label all glassware with field and QC samples ID numbers clearly and unambiguously during each step of the extraction procedure. Solvents will erase grease pens and "sharpie ink", so caution must be taken to ensure that the labels are not obliterated during the procedure.

Assemble a KD apparatus set-up and prepare a glass funnel for each sample to be extracted. Fold a 185 mm Whatman® 54 filter into quarters and place a filter in each funnel. Fill each funnel ~3/4 full with purified granular anhydrous sodium sulfate. Rinse the funnel with ~30 mL acetone and methylene chloride each and discard the solvent rinse. Place a prepared funnel onto each K-D setup.

Assemble the Tissuemizer by attaching the 20 mm x 195 mm-generator probe to the Tissumizer motor. Place the Tissuemizer in the fume hood and attach to the aluminum staging using clamps. Clean the Tissuemizer prior to use by running the generator probe for 10 seconds in a 400 mL beaker filled with ~ 200 mL of reagent water. Discard the reagent water and repeat with another aliquot of reagent water. Repeat the cleaning step two more times each with ~250 mL of acetone.

Mix the sample using a stainless steel or Teflon spatula. Place a labeled 400 mL beaker onto the top-loading balance and depress the "tare" button. Referring to the extraction condition spreadsheet, weigh out the appropriate amount of sample and record sample weight ± 1 gram into TALS. Repeat for all samples. Transfer two additional aliquots of the sample selected for the MS and MSD into labeled 400 mL beakers. Transfer the same weight of sodium sulfate each into labeled 400 mL beakers to serve as the method blank (MB) and laboratory control sample (LCS).

Add a sufficient volume of granular sodium sulfate to each sample and mix thoroughly with a stainless steel spatula until a free-flowing mixture is formed.

Add the appropriate volume of surrogate spike to each field sample and QC sample. Add the appropriate volume of spike solution to the laboratory control samples and the MS/MSD.

Add 100 mL of the appropriate extraction solvent to each beaker. Use 1:1 $MeCl_2$/Acetone for samples to be analyzed by GC/MS and 1:1 Hexane/Acetone for GC/ECD.

Refer to the Extractions Condition Worksheet to determine the type and amounts of solution added and the extraction solvent used for each test method.

Immerse the generator probe in the first sample beaker so that it is approximately ½" into the extraction solvent. Turn on the Tissuemizer. Adjust the speed on the motor until the solvent begins to vortex in the beaker, but does not splash out of the beaker. Extract the sample for 3 minutes. During extraction move the beaker in a circular motion to ensure that the entire sample is subject to extraction. Remove the beaker and decant the extraction solvent into the sample's corresponding funnel and K-D apparatus. Repeat the extraction 2 more times with ~100 mL of extraction solvent. After the 3rd extraction, pour the entire contents of the beaker into the funnel, rinse the beaker with more of the extraction solvent, and pour this into the funnel as well.

Rinse the funnel with ~30 mL of extraction solvent and allow the solvent to completely drain into the K-D apparatus. Remove the funnel from the K-D apparatus and discard the contents of the funnel. Clean the generator probe and repeat the extraction for each sample.

Concentrate the extracts following the procedure given in section 10.5 in preparation for percent lipids determination and extract cleanup. After concentration and prior to extract cleanup, set aside a 1 mL aliquot of the concentrated extract and determine the percent lipids following procedures given in laboratory SOP BR-EX-016 *Percent Lipids Determination.*

Perform extract cleanup as appropriate following procedures given in laboratory SOPs BR-EX-002 and BR-EX-011. Refer to extraction condition spreadsheet for details. After cleanup, concentrate the extracts following the procedure given in section 10.5.

Enter the extraction data into the TALS. Assemble any associated paperwork and submit the extracts to the supervisor for a final project check. After review is complete, relinquish the extracts to the appropriate analytical department and place in the refrigerated storage area.

Note: Immediately following concentration, all sample extracts must be stored in a refrigerator maintained at a temperature of 4ºC (±2°C) in order to maintain thermal preservation.

10.5 Extract Concentration (KD Apparatus)

10.5.1 Macro Concentration

Macro Snyder Column (K-D)

Add one or two clean boiling chips to the K-D evaporation flask and attach a three-ball Snyder column to the flask. Add ~1 mL of methylene chloride to the top of the column then place the K-D apparatus in a hot water bath (60-70°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in hot water vapor.

Attach the solvent vapor recovery glassware to the Snyder column. Adjust the vertical position of the apparatus and check the water bath temperature. The water bath temperature should be between 54.8 – 74.8°C when methylene chloride is the extraction solvent and 84-89°C when hexane is the extraction solvent. Higher water bath temperatures may be used so long as the recovery of target analytes is not impacted. The boiling point of each solvent is provided in the following table:

Solvent	Boiling Point	Water Bath Temperature
Hexane	69°C	84 – 89°C
Methylene Chloride	39.8°C	54.8 – 74.8°C

Monitor the concentration and do not let the extract evaporate to dryness. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with solvent.

When the apparent volume of the extract reaches desired amount remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

Micro Synder Column (K-D)

Add one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column to the tube. Place the concentrator tube into the water bath so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the concentrator tube and check the temperature of the water bath to ensure the proper temperature for the extract solvent.

Continuously monitor the distillation process to ensure sample extracts do not evaporate to dryness. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with solvent. Remove setup when desired sample volume is reached.

Nitrogen Blowdown

Nitrogen blow down may be used to concentrate extracts as needed.

Place the concentrator tube in a warm water bath maintained at a temperature of 35°C. Apply a steady stream of nitrogen until the desired final extract volume is achieved. Rinse the internal wall of the concentrator tube several times with the appropriate solvent during the evaporation and ensure the solvent level in the concentrator is positioned such to prevent water condensations. Monitor the concentration carefully and do not allow the extract to evaporate to dryness.

11.0 Calculations / Data Reduction

11.1 Data Review

11.1.1 Primary Review

Review the TALS worksheet for correctness and completeness. Record any problems encountered during the extraction process into TALS or complete a NCM, when necessary. Set aside the extracts and paperwork for secondary review.

11.1.2 Secondary Review

Review the TALS worksheet against the extraction conditions spreadsheet and/or project notes to ensure the extraction performed is consistent with project specifications. Authorize release of the extracts to the appropriate analytical department.

For additional guidance regarding the laboratory's protocol and required elements for data review refer to laboratory SOP BR-QA -019 *Data Review*.

12.0 Method Performance

12.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ)

Refer to the SOP for the test method for requirements for determination of LOD and LOQ. These procedures are not included in sample preparation SOPs.

12.2 Demonstration of Capabilities (DOC)

Each analyst must complete an Initial Demonstration of Capability prior to unsupervised performance of this method.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

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

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*. The following waste streams are produced when this method is carried out.

- Organic Solvents - Satellite container: 55 gallon covered and vented drum.
- Vials containing extracts - Satellite container: 5 gallon covered bucket in fume hood.

- Methylene Chloride-Waste-Satellite Container: 55 Gallon Waste Drum
- Sulfuric Acid Waste-Satellite Container: 2.5L Waste Bottle Labeled with appropriate acid type (sulfuric).
- Solid Waste-Satellite Container: Solid Waste 5 Gallon Plastic Bucket (inside fume hood)

15.0 References / Cross-References

- Comprehensive Descriptions of Trace Organic Analytical Methods given in the Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, National Status and Trends Program for Marine Environmental Quality.

- GERG Trace Organic Contaminant Analytical Techniques published in NOAA Technical Memorandum NOS Orca 71, Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Volume IV, Comprehensive Descriptions of Trace Organic Analytical Methods, July 1993.

- CW-E-M-001 *Corporate Environmental Health and Safety Manual*
- BR-EX-016 *Percent Lipid Determination*
- BR-EX-017 *Glassware Cleaning*
- BR-EX-002 *Extract Cleanup Procedure*
- BR-EX-011 *Gel-Permiation Cleanup*
- BR-QA -019 *Data Review*
- BR-QA-005 *Determination of LOD, LOQ, & RLs*
- BR-EH-001 *Hazardous Waste*

16.0 Method Modifications

Not applicable.

17.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions

18.0 Revision History

Revision 5, Effective Date 5/20/08:
- Title Page: Updated approval signatures.
- Section 6.0: Inserted vendor information
- Section 8.0: Inserted table
- Section 15.0: Added cross referenced methods with the SOP
- All Sections: Fixed typographical errors

Revision 6, Effective Date 05/20/10:
- Title Page: Updated approval signatures
- Section 6.1: Addition of Meat Grinder to equipment list
- Section 10.1: Changed pipette calibrations to be done quarterly
- All Sections: Fixed typographical errors
- All Sections: Changed benchsheets reference to extraction condition spreadsheet
- All Sections: Changed LIMS references to TALS

Revision 7, Effective Date 08/01/12

- Section 8.0: Updated Holding Time requirements
- Section 9.1: Changed frequency of MS/MSD to client request

Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

Appendix A: Terms and Definitions

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

Appendix N

Data Validations SOPs

VALIDATION SOP

PCDDs/PCDFs
USEPA Region 2 SOP HW-25
Rev. 3

SOP HW-25
Revision 3
September 2006

USEPA REGION II DATA VALIDATION SOP FOR EPA METHOD 1613, REVISION B
Tetra- through Octa-chlorinated Dioxins and Furans by Isotope Dilution (HRGC/HRMS)

PREPARED BY: George Karras DATE: 12/05/06
George Karras, Chemist HWSS

PEER REVIEWED BY: Russell Arnone DATE: 12/05/06
Russell Arnone, Chemist HWSS
Hazardous Waste Support Section

CONCURRED BY: Linda Mauel DATE: 12/5/06
Linda Mauel, Chief HWSS
Hazardous Waste Support Section

APPROVED BY: Robert Runyon DATE: 12/6/06
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ DATE: _____
Namer

Reviewed by: _____ DATE: _____
Name

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ATTACHMENT A
Data Assessment

1.0 Introduction

This method **was developed by the Engineering and Analysis division within the USEPA's Office of Science and Technology**. The method is used **for isomer specific determination** to detect the Tetra- through octa- chlorinated dibenzo-p-dioxins and dibenzofurans associated with the Clean Water Act (CWA, as amended 1987); the Resource Conservation and Recovery Act (RECRA, as amended 1986); **the Comprehensive Environmental Response,** the Compensation and Liability Act (as amended in 1986); **and the Safe Drinking Water Act** and other dioxin and furan compounds amenable to this method.

The dioxins and furans may be determined in water, soil, **sediment,** sludge, **tissue,** and other matrices using this method. The method is based on EPA, industry, and academic methods.

2.0 Applicability

The attached Standard Operating Procedure (SOP) is applicable to chlorinated dibenzodioxin and chlorinated dibenzofuran (CDD/CDF) data obtained using EPA Method **1613B**, Polychlorinated Dibenzodioxins (CDDs) and Polychlorinated Dibenzofurans (PCDFs) by Isotope Dilution using High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), **October 1994**. Its scope is to facilitate the data validation process of the data reported by the contracting laboratory and to ensure that the data is being reviewed in a unifor m manner. This SOP is based upon the quality control and quality assurance requirements specified in Method **1613B, October 1994.**

3.0 Responsibilities/Scope

3.1 The reviewer must be knowledgeable of the analytical method and its QC Criteria.

3.2 The reviewer must complete the following:

3.2.1 Data Assessment Checklist - The data reviewer must read each item carefully and must check yes if there is compliance, no if there is non compliance and N/A if the question is not applicable to the data.

3.2.2 Data Assessment Narrative - The data reviewer must present professional judgement and must express concerns and comments on the validity of the overall data package. The reviewer must explain the reasons for rejecting and/or qualifying the data. Example of Data Assessment format is provided in Attachment A.

3.2.3 **Communication** Record Log - All **communication must be in writing, and it** must be documen ted on the **Communication** Record Log Sheet. A photocopy of the **Communication** Record Log is attached to the Data Assessment package.

3.2.4 Paperwork - Upon completion of the review the following are to be maintained with the data package and returned to the authorized person :

a. completed data assessment checklist and narrative (original)
b. Two copies of the data assessment narrative
c. **Communication** record Log (original and copy)

3.3 Rejection of Data - All values determined to be unacceptable on the Dioxin/Furan Analysis Data Sheet (Form I) must be flagged with an "R". The qualifier R means that due to significant QA/QC problems the analysis is invalid and it provides no information as to whether the compound is present or not. Once the data are flagged with R any further review or consideration is unnecessary. The qualifier "J" is used to indicate that due to QA/QC problems the results are considered to be estimated. The qualifier "NJ" indicates that there is presumptive evidence for the presence of the compound at an estimated value.

The data reviewer must explain in the data assessment narrative why the data was qualified. He or she must also indicate all items of contract non-compliance. When 2,3,7,8- substituted TCDD, TCDF, PeCDD and PeCDF data are rejected (flagged "R") or qualified "J" the project officer must be notified promptly. If holding times have not been exceeded reanalysis of the affected samples may be requested. All qualifications and corrections on the Analysis Data Sheet must be made in red pencil.

4.0 Definitions

CALIBRATION SOLUTION: solutions containing known amounts of selected analytes, internal standards and recovery standards that are analyzed prior to sample analysis. The solutions are used to determine the ratio of the instrument response of the analytes to that of the appropriate internal standard and the internal standards to that of the recovery standards.

CALIBRATION VERIFICATION (VER): a mixture of known amounts of analytes that is analyzed every 12 hours to demonstrate continued acceptable GC/MS performance and establish the retention time window for each homologue.

CDD: Chlorinated Dibenzo-p-Dioxin. The isomers and congeners of tetra- through octa-chlorodibenzo-p-dioxin.

CDF: Chlorinated Dibenzofuran. The isomers and congeners of tetra- through octa-chlorodibenzofurans.

CLEAN-UP STANDARD: only one labeled analyte (2,3,7,8-TCDD) is added to all samples extracts prior to any Clean-up procedure. This standard is used to differentiate between losses of analytes or internal standards during extraction and losses that occur during the various Clean-up procedures.

CONGENER: elements of the same group in the periodic table.

DEFLECTIONS: bend or broadening of a peak

ESTIMATED DETECTION LIMIT (EDL): the concentration of a analyte required to produce a signal with peak height of at least 2.5 times the background signal level. The EDL is calculated for each 2,3,7,8 substituted isomer for which the response of the quantitation and confirmation ions is less than 2.5 times the background level.

ESTIMATED MAXIMUM POSSIBLE CONCENTRATION (EMPC): the concentration of a given analyte that would produce a signal with a given area peak. The EMPC is calculated for each 2,3,7,8 substituted isomer for which the response of the quantitation and/or confirmation ions has signal to noise in excess of 2.5 times the background level but does not meet identification criteria.

Field Blank: An aliquot of reagent water or other reference matrix that is placed in a sample container in the laboratoryor the field, and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blankis to determine if the field or sample transportingprocedures and environments have contaminated the sample.

FIELD CHAIN OF CUSTODY: see Traffic Report

GC: Gas chromatograph or gas chromatography.

GEL PERMEATION CHROMATOGRAPHY (GPC): removes many high molecular weight interferences that cause GC column performance to degrade. It may be used for all soil and sediment extracts and may be used for water extracts that are expected to contain high molecular weight organic compounds.

HOMOLOGUE: a member or members of a particular homologous series that has the same molecular weight but not necessarily the same structural arrangement. For example, the 28 pentachlorinated dibenzofurans are homologues.

HPLC: high performance liquid chromatography

HRGC/HRMS: high resolution gas chromatography/ high resolution mass spectrometry.

INITIAL CALIBRATION STANDARD SOLUTION (CS1-CS5): analysis of analytical standards for a series of different specified

concentrations. The initial calibration is used to define the linearity and dynamic range of the response of the mass spectrometer to the target compounds.

INITIAL PRECISION AND RECOVERY (IPR): must be performed by the laboratory to establish the ability to generate acceptable precision and accuracy **by analyzing four aliquots of the diluted PAR standard**. The standard deviation (s) of the concentration and the average concentration (x) for each unlabeled analyte must be within range established by the Method (Table **6**). **An IPR is performed prior to the first time this method is used and any time the method or instrumentation is modified.**

INTEGRATED ION CURRENT: electronic output to computer from instrument to provide a hard copy of area and height of a peak that may or may not be an analyte of interest.

INTERNAL STANDARDS (IS): labeled analytes are added to every sample and are present at the same concentration in every blank, quality control sample, and calibration solution. The IS are added to the sample before extraction and are used to measure the concentration of the analytes. In Method 1613 **B**, the ISs are $^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD.

ION ABUNDANCE RATIO: mathematical comparison of selected pair of ions stipulated by the method for each target analyte. The ratio between each pair of ions must fall within established limits. These ions are needed for the identification and quantitation of target analytes.

ISOMER: chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties. For example 1,2,3,4-TCDD and 2,3,7,8-TCDD are structural isomers.

LABELED ANALYTE (or analog): an analyte that has isotopically carbon added to its chemical structure. These compounds are used to established identification (retention time) and used for quantitation of unlabeled analytes.

MASS/CHARGE: usually expressed as m/z.

METHOD BLANK (MB): an analytical control consisting of all reagents, internal standards and surrogate standards that is carried through the entire analytical procedure. The MB is used to define the level of laboratory background contamination.

Minimum Level (ML): The level at which the entire analyticalsystem must give a recognizable signal and acceptable calibration point to the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and clean up procedures have been employed.

MAXIMUM CONCENTRATION LEVEL (MCL): Highest level of concentration for each analyte depending upon upper concentration of analyte. Usually used to determine upper level of the concentration range.

NON-CONGENER: elements not from the same group in the periodic table.

NON-2,3,7,8 SUBSTITUTED ANALYTES: analytes whose structure have positions other than 2,3,7,8.

ONGOING PRECISION AND RECOVERY (OPR): must be performed by the laboratory to establish the ability to maintain on a continuous basis, acceptable precision and accuracy. The standard deviation (s) of the concentration and the average concentration (x) for each unlabeled analyte must be within range established by the Method (Table **6**).

PAR: Precision and Recovery standard. Secondary standard that is diluted and spiked to form IPR and OPR. The standard deviation (s) of the concentration and the averageconcentration (x) for each unlabeled analyte must be withinrange established by the Method (Table 6).

PERCENT MOISTURE: an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at this degree including water. %M is determined from decanted samples and from samples that are not decanted.

PERCENT VALLEY: see Resolution

PERFLUOROKEROSENE (PFK): compound used to establish mass spectral instrument performance for dioxin/furan analysis.

PERFORMANCE EVALUATION MIXTURE (PEM): See Performance Evaluation (PE) Sample,

PERFORMANCE EVALUATION (PE) SAMPLE: a chemical waste, soil or water sample containing known amounts of unlabeled CDDs/PCDFs used for Quality Assurance programs. There are 3 types of PE's available. PEM Blank which consists of uncontaminated soil and used to monitor possible crossover contamination of samples in the field and laboratory. PEM Interference Fortified Blank which is a soil containing matrix interference and spiked by the laboratory with target compounds. A PEM sample(s) is a soil sample containing known amounts of unlabeled TCDD or a mixture of TCDD and other PCDD/PCDF isomers. These PEMs are used to monitor the laboratory's performance.

PCDPE: Polychlorinated Diphenylether: isomers having the same SICP and ion ratios identical to furan isomers and are monitored for interference in furan qualitative and quantitative analysis.

Quality Control Check Sample (QCS): A sample containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process.

RECOVERY: a determination of the accuracy of the analytical procedure made by comparing measured values from a fortified (spiked) sample against the known spiked values. Recovery is determined by the following equation:

$$\% \text{ Recovery} = \frac{\text{measured value}}{\text{known value}} \times 100\%$$

RELATIVE RETENTION TIME (RRT): ratio of the retention time of the analyte versus the retention time of the corresponding internal standard. RRT for each analyte must be within range established by the method.

RELATIVE RESPONSE (RR): the ratio of the area response of the mass spectrometer to a known amount of an analyte (unlabeled to labeled) versus a known concentration in standard solution, plotted using linear regression. The RR is used to determine instrument performance and is used in the quantitation calculations. RR are calculated using the following equation:

$$RR = \frac{(A_n^1 + A_n^2)\, C_l}{(A_l^1 + A_l^2)\, C_n}$$

$A_n^1 + A_n^2$ are the areas of the primary and secondary m/z's for the unlabeled compound.

$A_l^1 + A_l^2$ are the areas of the primary and secondary m/z's for the labeled compound.

C_l is the concentration of the labeled compound in the calibration standard.

C_n is the concentration of the unlabeled compound in the calibration standard.

Relative Standard Deviation (RSD): The standard deviation times 100 divided by the mean. Also termed "coefficient of variation".

RESPONSE FACTOR (RF): the ratio of the response of the mass spectrometer to a known amount of an analyte relative to that of a known amount of internal standard as measured in the initial and continuing calibrations. The RF is used to determine instrument

performance using correlation coefficient and is used in the quantitation calculations. RF are calculated using the following equation:

$$RF = \frac{(A_s^1 + A_s^2)\, C_{is}}{(A_{is}^1 + A_{is}^2)\, C_s}$$

$A_s^1 + A_s^2$ are the areas of the primary and secondary m/z's for the compound to be calibrated.

$A_{is}^1 + A_{is}^2$ are the areas of the primary and secondary m/z's for the internal standard.

C_s is the concentration of the compound in the calibration standard.

C_{is} is the concentration of the internal standard.

RESOLUTION: the separation between peaks on a chromatogram. Resolution is calculated by dividing the height of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RINSATE: a portion of the solvent that is used to rinse sampling equipment. The rinsate is later analyzed to demonstrate that samples were not contaminated during collection.

SAMPLE DELIVERY GROUP (SDG): a unit within a single case that is used to identify a group of samples for delivery. A SDG is a group of 20 or fewer samples within a case, received over a period of time up to 14 calendar days. Data from all samples in a SDG are due concurrently. A SDG is defined by one of the following, whichever occurs first:

- Case; or
- each 20 samples within a case; or
- each 14 day calendar period during which samples in a case are received, beginning with receipt of the first sample in the case or SDG.

SELECTED ION MONITORING (SIM): a mass spectrometric technique whereby ions with predetermined mass/charge ratios (m/z) are monitored, as opposed to scanning MS procedures in which all m/z's between two limits are monitored.

SICP: A plot of ion abundance versus time for each ion which provides the retention time, peak area and height. This information is used for identification and quantitation of target analyte.

SIGNAL TO NOISE (S/N) RATIO: the ratio of analyte signal to random background signal. To determine the ratio, display each characteristic ion using a window 100 scans wide, and draw a base line from the lowest point in the 100 scan window. The noise is defined as the height of the largest signal (excluding signal due to CDDs/PCDFs or other chemicals) within the 100 scan window. The signal is defined as the height of the PCDD/PCDF peak. If the data system determines the ratio, the Contractor shall demonstrate comparability between the above criteria and the automated S/N determination. Chemical noise is left to the judgement of the analyst.

2,3,7,8 SUBSTITUTED ANALYTES: analytes whose structure has other positions as well as the 2,3,7,8 positions.

TOXICITY EQUIVALENCY FACTOR (TEF): a method of converting concentrations of CDDs/PCDFs to an equivalent concentration of 2,3,7,8-TCDD to obtain an estimation of the toxicity of the entire sample. The concentrations can be found on Form I PCDD-2 in the DFLM01.1 Statement of Work for Dioxin Analysis.

TRAFFIC REPORT (TR): (may also be called Field Chain of Custody), a sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and documents sample condition and receipt by the laboratory.

TWELVE HOUR TIME PERIOD: the 12 hour time period begins with the injection of the CS3 solution on the DB-5 (or equivalent)

column or the injection of the column performance solution on the SP-2331 (or equivalent) column. The 12 hour period continues until 12:00 hours have elapsed according to the system clock. To be included in a given 12-hour time period, a sample or standard must be injected with 12:00 hours of the CS3 solution or the column performance solution.

UNLABEL ANALYTE: target compound that has not been isotopically altered.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR): the date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and sample traffic report.

WINDOW DEFINING MIXTURE (WDM): a mixture containing the first and last eluting isomer for each congener. The retention time for each first and last eluting isomer establishes the retention time window for each congener. All analytes in the standards (calibrations, internal standards, recovery standards, Clean-up standard) and identified analytes in samples must have a reported retention time within the established window. It is analyzed before any calibration standard, at the beginning of each 12 hour time period or when there is a shift greater than 10 seconds between retention time of recovery standards in standards or any analysis from retention time in recent calibration verification.

YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____
LAB:_____
SITE:_____

1.0 Data Completeness and Deliverables

1.1 Does the Traffic Report or Field Chain of Custody list all samples? [___] ___ ___

1.2 Is the Case Narrative present? [___] ___ ___

1.3 Are the Case Number and SDG numbers contained in the case narrative? [___] ___ ___

1.4 Do the Traffic Reports, Field Chain of Custody or Lab Case Narrative indicate problems with sample receipt, sample condition, analytical problems, or other comments affecting the quality of the data? ___ [___] ___

ACTION: Use professional judgement to evaluate the effect of the noted problems on the quality of the data.

ACTION: As per region II requirements, if any sample analyzed as a soil, contains 50% to 90% water, all data shall be flagged as estimated "J". If a soil sample Contains more than 90% water, then qualify positive hits "J", and non detects "UJ".

ACTION: If sample cooler temperature was greater than 10 C, then flag all positive hits "J" And non detects "UJ".

2.0 Reporting Requirements and Deliverables

2.1 All deliverables must be clearly labeled with the Case number and the associated sample/traffic number. Missing or illegible or incorrectly labeled items must be identified. The Project Officer must immediately be contacted and requested to ask laboratory to submit the missing or incorrect items.

2.2 The following forms were taken from the CLP SOW, DFLM01.1 and should be specified in the Project Plan. Laboratories will not always use the exact CLP format for the forms. A comparison of CLP forms must be made against the Laboratory's version. Some information may not be found on the exact form as the CLP version but may be located on another form. As long as the information is present and accessible, it is not a problem. Are these forms (CLP or lab's version) present?

a. Sample Data Summary (Form I CDD-1) [___] ___ ___

b. CDD/CDF Toxicity Equivalency Factor (Form I, CDD-2) [___] ___ ___

c. Second Column Confirmation Summary (Form I, CDD-3) [___] ___ ___

d. Total Homologue Concentration Summary (Form II CDD) [___] ___ ___

e. CDD/CDF Spiked Sample Summary (Form III CDD-1) [___] ___ ___

f. CDD/CDF Duplicate Sample Summary (Form III CDD-2) [___] ___ ___

	YES	NO	N/A
g. CDD/CDF Method Blank Summary (Form IV-CDD)	[___]	___	___
h. CDD/CDF Window Defining Mix Summary (Form V-CDD-1)	[___]	___	___
I. Chromatographic Resolution Summary (Form V CDD-2)	[___]	___	___
j. CDD/CDF Analytical Sequence Summary (Form V CDD-3)	[___]	___	___
k. Initial Calibration (Form VI, CDD-1, CDD-2)	[___]	___	___
l. Continuing Calibration (Form VII, CDD-1, Form VII, CDD-2)	[___]	___	___

ACTION: If forms are missing, contact the Project Officer to confirm which forms if any were specified in the Project Plan. If the forms are required, inform the Project Officer or obtain written permission to contact the lab for explanation/resubmittal. If the lab cannot provide missing deliverables, assess the effect on the validity of the data. Note in the Data Assessment.

2.3 GC/MS Displays
Are the following GC/MS displays present?

	YES	NO	N/A
a. Standard and sample SIM chromatograms. SIM and TIC chromatograms must list date and time of analysis; the file name; sample number; and instrument I.D. number	[___]	___	___
b. Percent peak resolution valley	[___]	___	___
c. Window Defining Mixture raw data	[___]	___	___
d. SIM mass chromatograms must display quantitation ion, confirmation ion, and polychlorinated diphenylether ion, where applicable.	[___]	___	___
e. Integrated area and peak height must be listed for all peaks 2.5 times above background	[___]	___	___

ACTION: If deliverables are missing, contact the Project Officer to request explanation/resubmittals or obtain written permission to contact the lab for explanation/resubmittal. If the lab cannot provide missing deliverables, assess the effect on the validity of the data. Note in the Data Assessment.

2.4 Are the following Chain of Custody Records and in-house Laboratory Control Documents present?

	YES	NO	N/A
a. Chain of Custody Records	[___]	___	___
b. Sample Shipment Records	[___]	___	___
c. Sample log-in sheets	[___]	___	___
d. GC/MS Standard and Sample Run Log in chronological order	[___]	___	___
e. Sample Extraction Log	[___]	___	___

ACTION: If deliverables are missing, contact the Project Officer to request explanation/resubmittals

YES NO N/A

or obtain written permission to contact the lab for explanation/resubmittal. If the lab cannot provide missing deliverables, assess the effect on the validity of the data. Note in the Data Assessment.

2.5 Was the sample data package paginated and one sided? [___] ___ ___

ACTION: If no, document difficulties of reviewing data caused by lack of pagination in Data Assessment.

3.0 Holding Times

3.1 Have **samples been analyzed within proper holding times**?

a. For aqueous samples, 30 days from VTSR to extraction? [___] ___ ___

b. For soil/sediment samples, 30 days from VTSR to extraction? [___] ___ ___

c. For **fish and tissue samples, one (1) year** from VTSR to extraction? [___] ___ ___

d. For all samples 45 days from time of extraction to time of analysis? [___] ___ ___

ACTION: If holding times are exceeded, flag all **positive hits** as estimated ("J"), **and non-detects as estimated "UJ"**. Holding time criteria do not apply to PE samples.
If holding times are grossly exceeded (e.g. by greater than two times the specified Technical holding times), either on the first analysis or upon reanalysis, flag positive hits as estimated "J", and flag non-detects as unusable "R".

Note: **The data reviewer must note whether or not technical and contractual holding times were met.**

4.0 Instrument Performance

4.1 Mass Calibration - Mass calibration of the MS must be performed prior to analyzing calibration solutions, blanks, samples, and QC samples. A static resolving power of at least 10,000 (10% valley definition) must be demonstrated at appropriate masses before any analysis is performed. Static resolving power checks must be performed at the beginning and at the end of each 12 hour period of operation. Include in the narrative, minimum required resolving power of 10000 was obtained for perfluorokerosene (PFK) ion 380.9760. This is done by first measuring peak width at 5% of the maximum. This should not exceed 100 ppm, i.e., it should not exceed 0.038, for ion 380.9760. Resolving power, then is calculated using the formula,

Resolving Power = m/□m□= 380.9760/0.038 = 10025.

NOTE: The mass calibration is generally not reported. Improper mass calibration may be detected by examining ion abundance ratios for initial and continuing calibration standards. If the mass calibration is not properly performed, the standards will not have ion abundance ratios within criteria.

4.2 Window Defining **Solution**/ Isomer Specificity Test Standards

The Window Defining **Solution** must contain the first and the last isomers of each homologue CDD/CDF, (the labeled and internal standards are optional). The solution also should contain a series of other TCDD analytes for the purpose of documenting the chromatographic resolution.

4.2.1 For analyses on a DB-5 (or equivalent) GC column, the chromatographic resolution is evaluated by the analysis of Isomer Specificity Test Standards at the beginning

	YES	NO	N/A
of every 12 hour period. Was this performed accordingly?	[___]	___	___

ACTION: If the Isomer Specificity Test Standards was not analyzed at the required frequency, use professional judgement to determine the effect on the quality of the data. Document in Data Assessment under contract non-compliance.

4.2.2	Were all peaks labeled and identified on the Selected Ion Current Profiles (SICPs)?	[___]	___	___
4.2.3	Did the absolute retention time of the internal standards $^{13}C_{12}$-1,2,3,4-TCDD exceed 25.0 minutes on the DB-5 column and 15.0 minutes on the DB-225 column? (Method 1613**B**, Section **10**.2.4)	[___]	___	___
4.2.4	Are the relative retention times of native and labeled CDD's and CDF's within the limits given in Table 2 of the method. (Method 1613 **B**, Section **15**.4.1.2)	[___]	___	___

ACTION: If no for sections 4.2.2, 4.2.3 and 4.2.4, assess the effect on the validity of the data. Note in the Data Assessment.

4.2.5 For DB-5 or equivalent, (Method 1613 **B**, Section **15**.4.2.2) the peak separation between the unlabeled 2,3,7,8-TCDD and the peaks representing any other TCDD analyte shall be resolved with a valley of ☐≥25 percent.
Was this criteria met? [___] ___ ___

% Valley = (x/y) x (100)

Y = The peak height of 2,3,7,8-TCDD analyte

X = The distance from the baseline to the bottom of the valley between the adjacent peaks.

ACTION: If the percent valley criteria are not met, qualify all positive data "J". Do not qualify non-detects.

4.2.6 Is the last eluting tetra chlorinated congener (1,2,8,9-TCDD) and the first eluting penta chlorinated congener (1,3,4,6,8-PeCDF) separated properly, since they elute within 15 seconds of each other? [___] ___ ___

ACTION: If one of the congener is missing, report that in the Data Assessment.

5.0 Initial 5-Point Calibration

The initial calibration standard solutions (CS1-CS5) must be analyzed prior to any sample analysis. However, initial calibration should be analyzed when the CS3 Calibration Verification (VER) or Isomer Specificity Test Standard do not meet performance criteria. The initial calibration standards must be analyzed on the same instrument using the same GC/MS conditions that were used to analyze the Window Defining **Solution** and the Isomer Specificity Test Standards.

Was the initial calibration performed at the frequency specified above? [___] ___ ___

5.1 The method allows the Laboratory to perform quantitative analysis by isotope dilution and internal standard, or to combine calibration solutions.

1. Isotope Dilution: performed for the fifteen 2,3,7,8-substituted CDDs and CDFs unlabeled analytes with labeled analytes added to the samples prior to extraction and for 1,2,3,7,8,9- HXCDD and OCDF (see sections 5.2.8 and 5.2.9). The relative response (RR) is calculated and the percent

YES NO N/A

coefficient of variation must be □20% over the 5 point range **(1613B sec. 10.5.4)** to use the average **relative response** for quantitation, otherwise a calibration curve must be used..

2. Calibration by Internal Standard: performed for non-2,3,7,8 substituted compounds having no labeled analytes in this method and for measurement of labeled compounds for intra laboratory statistics.. The response factor (RF) is calculated and the percent coefficient of variation must be □35% over the 5 point range **(1613B sec. 10.6.3)** to use the average response factor for quantitation, otherwise a calibration curve must be used.

3. Combined Calibration: performed by using solutions containing unlabeled, labeled compounds and internal standards. The requirements of each of the above methods are used. This method allows the laboratory to produce a single set of curves for isotope dilution and internal standard method.

5.1.1 The following MS/DS conditions must be used:

5.1.1.1 Mass calibration as per Section 4.1? [___] ___ ___

5.1.1.2 Were SIM data acquired for each of the ions listed in Table **8**, including interfering ions? (see analytical method) [___] ___ ___

5.2 Were the following GC criteria met?

5.2.1 The chromatographic resolution between the 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of □≤25 percent on the primary analysis (DB-5) column (**1613B** sec. **15.4.2.2**). [___] ___ ___

5.2.2 The chromatographic resolution between the 2,3,7,8-TCDF and the peaks representing any other unlabeled TCDF isomers must be resolved with a valley of □≤25 percent on the confirmation (DB-225 or SP2330) analysis column. [___] ___ ___

5.2.3 For all calibration solutions, the relative retention time of peaks representing an unlabeled 2,3,7,8- substituted CDD or CDF must be within the limits given in table 2 of the Method. The retention times of the peaks representing non-2,3,7,8- substituted CDD or CDF's must fall within the retention time windows established by the Window Defining **Solution**. In addition, the absolute retention times of internal standards, $^{13}C_{12}$1,2,3,4-TCDD and $^{13}C_{12}$1,2,3,7,8,9-HxCDD shall not change by more than 15 seconds between the CS3 analysis and the analysis of any other standard. [___] ___ ___

5.2.4 **Are** the two SIM ions for each homolog must maximize simultaneously and within 2 seconds of the corresponding labeled analyte ions? **(1613B sec. 16.1)** [___] ___ ___

5.2.5 The relative ion abundance criteria for CDDs/CDFs listed in **Table 9** (see analytical method) must be met. [___] ___ ___

5.2.6 For all calibration solutions the signal to noise ratio (S/N) for the GC signal present in every SICP, including the ones for the labeled standards must be □≥10. [___] ___ ___

5.2.7 The percent relative standard deviations (% RSD) for the mean response factors (RRF) from the 17 unlabeled standards must be □≤20%, and those for the 15 labeled reference compounds must be □≤35%. [___] ___ ___

YES NO N/A

5.2.8 Labeled analyte 1,2,3,7,8,9-HxCDD is used as an internal standard in this method, and can not be used to quantitate corresponding unlabeled analyte. The unlabeled 1,2,3,7,8,9-HxCDD must be quantitated using the average of the responses of the labeled analytes of 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD. The concentration of the unlabeled 1,2,3,7,8,9-HxCDD is corrected for the average recovery of the other HxCDD's. Was the unlabeled 1,2,3,7,8,9-HxCDD quantitated correctly? [___] ___ ___

5.2.9 The labeled analog of OCDF is not added to the sample because of a potential interference. Unlabeled OCDF is quantitated against the labeled OCDD. The concentration of the unlabeled OCDF is corrected for the recovery of the labeled OCDD. Was the unlabeled OCDF correctly quantitated against the labeled OCDD. [___] ___ ___

ACTION:

1. If mass calibration criteria as specified in Section 4.1 was not met, note in Data Assessment.

2. If the selected monitoring ions specified in **Table 8** were not used for data acquisition, the lab must be contacted by the Project Officer for an explanation. If an incorrect ion was used, reject "R" all the associated data.

3. If the 25% percent valley for TCDD requirement was not met, quality positive data "J". Do not qualify non-detects. The tetra and penta (dioxins and furans) are affected. Heptas, Hexas and Octas are not affected.

4. If the ion abundance ratio for an analyte is outside the limits, flag the results for that analyte "R" (reject).

5. If the ion abundance ratio for an internal or labeled standard falls outside the QC limits flag the associated positive hits with "J". No effect on the non-detects.

6. If the signal to noise ratio (S/N) is below control limits, use professional judgement to determine quality of the data.

7. If the %RSD for each unlabeled analyte exceeds 20%, or the %RSD for each labeled analyte exceeds 35%, flag the associated sample positive results for that specific analyte as estimated ("J"). No effect on the non-detect data.

8. If 1,2,3,7,8,9-HxCDD was not calculated using the correct HxCDD response (average) factor, either manually recalculate the values for all standards and samples or contact Project Officer to request resubmittals from the laboratory.

9. If OCDF was not calculated using the correct response factor (OCDD), either manually recalculate the values for all standards and data or contact Project Officer to request resubmittals from the laboratory.

10. Non compliance of any other criteria specified above should be evaluated using professional judgement.

5.2.10 Spot check response factor calculations and ion ratios. Ensure that the correct quantitation ions for the unlabeled CDDs/CDFs and labeled standards were used. In addition, verify that the appropriate labeled standard was used for each analyte.

To recalculate the response factor, use the equation:

YES NO N/A

For target compounds (unlabeled analytes with corresponding labeled analytes):

$$RR = \frac{(A_{n1} + A_{n2}) \times Q_l}{(A_{l1} + A_{l2}) \times Q_n}$$

For labeled analytes, Internal standards and cleanup standard listed in Table 6 of method 1613:

$$RF = \frac{(A_{l1} + A_{l2}) \times Q_{is}}{(A_{is1} + A_{is2}) \times Q_l}$$

Note: There is only one m/z for $^{37}Cl_4$2,3,7,8-TCDD.

$A_{n1} + A_{n2}$ = integrated areas of the two quantitation ions of analytes of interest. (Target analyte, unlabeled compounds)

$A_{l1} + A_{l2}$ = integrated areas of the two quantitation ions of the appropriate labeled analytes compound.

$A_{is1} + A_{is2}$= integrated areas of the two quantitation ions of the appropriate internal standard.

Q_n = quantity of the unlabeled PCDD/PCDF analyte injected [pg]

Q_l = quantity of the appropriate labeled analytes compound [pg]

Q_{is} = quantity of the appropriate internal standard injected [pg]

ACTION: If calculations were not performed correctly, notify the Project Officer to initiate resubmittals from the laboratory.

6.0 System and Laboratory Performance (Calibration Verification and Isomer Specificity Test Standard)

At the beginning of a 12 hour shift during which analyses are performed, GC/MS system performance and calibration are verified for all unlabeled and labeled compounds. For these tests the calibration verification (VER) standard and the isomer specificity test standards shall be used to verify all performance criteria.

Only if the laboratory meets all performance criteria may samples, blanks, and precision and recovery standards be analyzed.

6.1 Calibration Verification

6.1.1 Was the relative ion abundance for CDDs/CDFs listed in **Table 9** of the analytical method met? (Method 1613**B**, Section **15**.3.2) [___] ___ ___

6.1.2 Were the peaks representing each unlabeled and labeled compound in the verification standard present with signal to noise ratio (S/N) of $\geq$ 10? **(Method 1613B, Section 15.3.3)** [___] ___ ___

6.1.3 For each compound, was the concentration within the limit in Table **6** of the method? (Method 1613**B**, Section **15**.3.5) [___] ___ ___

6.1.4 Were the absolute retention time of the internal standards $^{13}C_{12}$-1,2,3,4- TCDD and $^{13}C_{12}$1,2,3,7,8,9- HxCDD within $\pm$ 15 seconds of the retention times obtained during calibration? (Method 1613**B**, Section **15**.4.1.1) [___] ___ ___

	YES	NO	N/A
6.1.5 Were the relative retention times of the unlabeled and labeled CDDs and CDFs within the limits given by Table 2 of the method? (Method 1613 **B**, Section **15**.4.2.2)	[___]	___	___
6.2 Isomer Specificity Test Standard			
6.2.1 Was the chromatographic resolution between 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers resolved with a valley of $\leq$ 25 percent on the primary analysis (DB-5) column? (Method 1613 **B**, Section **15**.4.2.2)	[___]	___	___
6.2 Was the chromatographic resolution between 2,3,7,8- TCDF and the peaks representing any other unlabeled TCDF isomers resolved with a valley of $\leq$ 25 percent on the confirmation (DB-225 or SP2330) analysis.	[___]	___	___

ACTION:

1. If the ion abundance ratio for an analyte is outside the limits, flag the results for that analyte "R" (reject).

2. If the signal noise ratio (S/N) is below control limits, use professional judgement to determine the quality of the data.

3. If an analyte concentration fell outside the acceptance criteria listed in **Table 6** of the method.

   A. If the acceptance criteria for each unlabeled analyte and/or for each labeled analyte exceeds the range, flag the associated sample positive results for that specific analyte as estimated ("J"). No effect on the non-detect data.

   B. If the acceptance criteria for each unlabeled analyte and/or for each labeled analyte is below the range, flag the associated sample positive results as well as non-detects for that specific analyte as estimated ("J").

   C. If the acceptance criteria for each unlabeled analyte and/or for each labeled analyte is excessively below, $\leq$ 10% of the range, at the minimum, flag the associated sample positive results as well as non-detects for that specific analyte as estimated ("J"). However the validator may use professional judgement to accept or reject positive data and non-detects.

4. If the 25 percent valley for TCDD and TCDF requirement was not met, qualify positive data "J". Do not qualify non-detects. The tetras and pentas (dioxin and furans) are affected. Heptas, Hexas and Octas are not affected.

5. Non compliance of any other criteria specified above, in the method should be evaluated using professional judgement.

6.3 Spot check response factor calculations and ion ratios. Ensure that the correct quantitation ions for the unlabeled CDDs/CDFs and labeled standards were used. In addition, verify the appropriate labeled standard was used for each analyte.

7.0 Sample Data

NOTE: Any qualifications such as "J" applied to target compounds should be also applied to their associated total congeners concentration column.

7.1 Were the following MS/DS conditions used?

		YES	NO	N/A
7.1.1	SIM data were acquired for each of the ions listed in **Table 8** (see analytical method) including diphenylether interfering ions.	[___]	___	___
7.2	Were the following identification criteria met?			
7.2.1	For the 2,3,7,8 substituted analytes found present and the corresponding labeled compound or internal standard in the sample extract, must show relative retention times at the peak height within the limits given in Table 2. (Method 1613 **B**, Section **16**.4)	[___]	___	___
7.2.2	For non-2,3,7,8 substituted compounds (tetra through octa) found present, the retention time must be within the window established by the Window Defining **Solution**, for the corresponding homologue (Method 1613**B**, Section **16**.4)	[___]	___	___
7.2.3	All specified ions listed in **Table 8** for each isomer found present and the associated labeled compounds must be present in the SICP. The two SIM ions for the analyte, the labeled compound, and the internal standard must maximize simultaneously.(± 2 sec.) (Method 1613**B**, Section **16**.1)	[___]	___	___
7.2.4	The integrated ion current for each characteristic ion of the analyte identified as positive, must be at least 2.5 times background noise and must not have saturated the detector. (Method 1613**B**, Section **16**.2)	[___]	___	___
7.2.5	The integrated ion current for the labeled compounds, internal standards, and cleanup standard characteristic ions must be at least 10 times background noise. (Method 1613 **B**, Section **16**.2)	[___]	___	___
7.2.6	The relative ion abundance criteria for all CDDs/CDFs found present must be **within the limits of Table 9, or ☐10% of the ratio in the midpoint CS3 calibration or calibration verification (VER) whichever is most recent.**	[___]	___	___
7.2.7	The relative retention time of the unlabeled 2,3,7,8-substituted PCDD or PCDF must be within the limits given in Table 2 (Method 1613**B, Section 16.4**).	[___]	___	___
7.2.8	The relative ion abundance criteria for the labeled compounds, cleanup, and internal standard must be met (Table **9** - Method 1613**B**).	[___]	___	___
7.2.9	The analyte concentration must be within the calibration range. If not, dilution should have been made to bring the concentration within the calibration range. Was this criterion met?	[___]	___	___

NOTE: The analytical method clearly states that samples containing analytes having concentrations higher than 10 times the upper MCLs should be analyzed using a less sensitive, high resolution GC/low resolution MS method.

7.2.10	The identification of a GC peak as a PCDF can only be made if no signal having a S/N ☐$.5 is detected at the same time in the corresponding polychlorinated diphenylether (PCDPE) channel. Was the above condition met?	[___]	___	___

ACTION: 1. If the selected monitoring ions specified in Table 3 were not used for data acquisition, the lab must be contacted by the Project Officer for an explanation. If an incorrect ion was used, reject "R" all the associated data.

YES NO N/A

2. If the retention time of an analyte falls outside the retention time windows established by the associated Window Defining Mixture take the following action:

   A. If the analyte has a corresponding labeled analyte and is within 2 seconds of the labeled analyte, no action taken on positive data or non-detects.

   B. If the analyte has a corresponding labeled analyte and is outside 2 seconds of the labeled analyte, use professional judgement to determine qualifications for positive data or non-detects. At a minimum, "J" or "JN" positive data.

   C. If the analyte does not have a corresponding labeled analyte and is outside 2 seconds of the matching unlabeled analyte from the associated calibration, use professional judgement to determine qualifications for positive data or non-detects. At a minimum, "J" or "JN" positive data.

   D. If analyte meets identification criteria (7.2.2, 7.2.4, 7.2.5, 7.2.7) but does not meet ion abundance ratio criteria (7.2.8) and is not a labeled analog, the sample must be reanalyzed on a confirmation column. If confirmation analysis was not perform, reject **"R"** the failing analyte.

3. If the criteria listed in section 7.2.4 and 7.2.5 are not met but all other criteria are met, qualify all positive data of the specific analyte with "J".

4. If the analytes reported positive do not meet criteria for section 7.2.6, reject **"R"** all positive data for these analytes. Change the positive values to EMPC (Estimated Maximum Possible Concentration). Flag "J"

5. If the labeled compounds, internal standards and cleanup standards do not meet ion abundance criteria section 7.2.6. and 7.2.7. (Table **8** - analytical method) but they meet all other criteria, flag all corresponding data with "J".

6. If the lab reported values exceeding the calibration range flag those values with "J".

7. If peak deflections >50% are visible qualify particular compound with "J".

8. If PCDF was detected but an interfering PCDPE was also detected (see Section 7.2.9) and concentration not corrected for the interference, cross out the PCDF data. The reported value of PCDF is changed to EMPC.

9. If the lab did not monitor for PCDPEs, qualify all positive furan data "JN".

7.2.10 Spot check calculations for positive data and verify that the same labeled compounds used to calculate RFs were used to calculate concentration and EMPC. Ensure that the proper CDDs/CDFs and labeled compounds were used.

To recalculate the concentration of individual CDD/CDF analytes in the sample use the following equation:

All Matrices other than water

$$Cn\ (pg/g) = \frac{(A_{n1} + A_{n2}) \times Q_l}{W \times (A_{l1} + A_{l2}) \times RR}$$

Water

$$Cn\ (pg/L) = \frac{(A_{n1} + A_{n2}) \times Q_l}{}$$

YES NO N/A

$V \times (A_{l1} + A_{l2}) \times RR$

Where:

$A_{n1} + A_{n2}$ = integrated areas of the two quantitation ions of analyte of interest. (Target analyte)

$A_{l1} + A_{l2}$ = integrated areas of the two quantitation ions of the appropriate labeled analyte compound.

W = Weight (g) of sample extracted

V = Volume (L) of sample extracted

Q_l = Quantity (pg) of the appropriate labeled compound added to the sample prior to extraction.

RR = Calculated relative response from initial calibration. (see section 5.2.10)

ACTION: If the spot check calculations yielded positive hit concentrations with $\leq$ 15% Difference from those reported in Form I, correct manually. If the difference between the validator's value and the form 1's values are > 15% contact the Project Officer to request from the laboratory for an explanation and a copy of the laboratory's calculations.

7.3 Clean-up procedures

Clean-up may not be necessary for relatively clean samples (drinking waters, ground waters etc). If the matrix required clean-up, the laboratory has 4 different procedures to choose from. Before using any clean-up procedure, the laboratory must demonstrate that the Initial Precision and Recovery requirements of the method can be met using the clean-up procedure.

A labeled clean-up standard $^{37}Cl_4$2,3,7,8-TCDD is added to the sample just before the back extraction with base and acid procedure. This occurs before any recommended clean-up procedures are initiated.

7.3.1 Was the percent recovery of the clean-up standard within the recommended range **listed on Table 6 of the Analytical method?** [___] ___ ___

ACTION: If no, and the recovery is less than 25%, qualify all data as estimated "J". If recovery is 0 %, qualify all positive data as estimated "J" and reject "R" all non-detects for that sample.

7.3.2 Check the chromatograms that clean-up procedure was needed for each sample. Were any clean-up procedures needed for either water or soil samples? ___ [___] ___

ACTION: If yes, check extraction log to verify which clean-up procedures if any were performed. The laboratory is not limited to only one procedure.

1. If no clean-up was performed and the chromatograms indicated that some should have been performed. Use professional judgement to assess the effect on the interference on the validity of the data. Document lack of required clean-up for complex samples in Data Assessment.

2. If one type of clean-up was performed, but the chromatograms indicate that additional clean-up should have been utilized. Use professional judgement to assess the effect on the interference on the validity of the data. Document lack of additional clean-up for complex samples in Data Assessment.

7.3.3 If clean-up procedures were used, did the Laboratory perform clean-up procedures on the

	YES	NO	N/A
Initial Precision and Recovery samples as required by the method?	[___]	___	____

ACTION: If no, Use professional judgement to assess the effect of the interference on the validity of the data. Document lack of IPR documentation for clean-up procedures in Data Assessment.

8.0 Estimated Detection Limits (EDL) ***If required for the project***

8.1 Was an EDL calculated for each 2,3,7,8-substituted analyte that was not identified regardless of whether other non-2,3,7,8 substituted analytes were present? [___] ___ ___

ACTION: 1. If EDL or EMPC of an analyte which was not reported as a positive hit is missing, correct manually or contact the Project Officer to request from the laboratory corrections.

8.2 Use the equation below to check EDL calculations:

ALL MATRICES OTHER THAN WATER

$$EDL\ (pg/g) = \frac{2.5 \times Qis \times (Hx^1 + Hx^2) \times D}{W \times (His^1 + His^2) \times RR}$$

WATER

$$EDL\ (pg/L) = \frac{2.5 \times Qis \times (Hx^1 + Hx^2) \times D}{V \times (His^1 + His^2) \times RR}$$

Where:

Hx^1 and Hx^2 = peak heights of the noise for both quantitation ions of the 2,3,7,8-substituted isomer of interest.

His^1 and His^2 = peak heights of both the quantitation ions of the appropriate internal standards.

D = dilution factor

Qis, RR, W and V are previously defined.

NOTE: The validator should check the EDL data to verify that peak heights and not areas were used for this calculation. If the area algorithm was used, the validator should contact the Project Officer to request recalculations from the laboratory.

ACTION: If the spot check calculations yielded EDLs or EMPCs with $\leq$ 15% Difference from those reported in Form I, correct manually. If the difference between the validator's value and the Form I's values are > 15% contact the Project Officer to request from the laboratory for an explanation and a copy of the laboratory's calculations.

9.0 Estimated Maximum Possible Concentration (EMPC) ***If required for the project***

9.1 Was an EMPC calculated for 2,3,7,8-substituted analytes that had S/N ratio for the quantitation and confirmation ions greater than 2.5, but did not meet all the identification criteria? [___] ___ ___

9.2 Use the equation below to check EMPC calculations:

YES NO N/A

All Matrices other than water

$$\text{EMPC (pg/g)} = \frac{(A_{n1} + A_{n2}) \times Q_I \times D}{W \times (A_{I1} + A_{I2}) \times RR}$$

Water:

$$\text{EMPC (pg/L)} = \frac{(A_{n1} + A_{n2}) \times Q_I \times D}{V \times (A_{I1} + A_{I2}) \times RR}$$

Action: 1. If EDL or EMPC of an analyte which was not reported as a positive hit is missing, correct manually or contact the Project Officer to request from the laboratory corrections.

2. If the spot check calculations yielded EDLs or EMPCs with $\leq$ 15% Difference from those reported in Form I, correct manually. If the difference between the validator's value and the Form I's values are > 15% contact the Project Officer to request from the laboratory for an explanation and a copy of the laboratory's calculations.

3. If EDLs or EMPCs for the most toxic analytes (TEF $\geq$ 0.05) are above reporting limits, contact the project office to recommend sample reanalysis.

10.0 Method Blanks

10.1 Has a method blank per matrix been extracted and analyzed with each batch of 20 samples? [___] ___ ___

10.2 If samples of some matrix were analyzed in different events (i.e. different shifts or days) has one blank for each matrix been extracted and analyzed for each event? [___] ___ ___

10.3 Acceptable method blanks must not contain any signal of 2,3,7,8-TCDD, or 2,3,7,8-TCDF, equivalent to a minimum levels listed in Table 2 **or above one third the regulatory compliance level.** Was this criteria met? (Method 1613**B**, Section **9**.5.2) [___] ___ ___

10.4 For other 2,3,7,8- substituted CDD/CDF isomers of each homologue, the allowable concentration in the method blank is less than minimum level listed in Table 2 (< 5 ng/Kg for soils and 50 pg/L for waters). Was this criteria met? [___] ___ ___

ACTION: 1. If the proper number of method blanks were not analyzed, document in Data Assessment. If the validator feels that the validity of the data is seriously compromised and validation of data without the method blanks would be flawed then notify the Project Officer. If decision is made to proceed with the validation process, consider the following actions: no action taken on non-detected analytes. If an analyte has a reported concentration that is > 5 times the EDL, qualify "J" and all concentrations $\leq$ 5 times the EDL are qualified "R" due to possibility of contamination.

2. If the method blank is contaminated with 2,3,7,8-TCDD, 2,3,7,8-TCDF, 1,2,3,7,8-PeCDD, 1,2,3,7,8-PeCDF or 2,3,4,7,8-PeCDF at a concentration higher than the minimum levels in Table 2, reject all contaminant compound positive data for the associated samples "R" and notify the Project Officer to initiate reanalysis.

3. A. If the method blank is contaminated with any of the analytes mentioned in Action # 2 at a concentration of less than the minimum levels in Table 2 specified in the method or of any other 2,3,7,8-substituted analytes at any concentration and the concentration in the sample is less than five times the concentration in the blank, transfer the sample results to the EMPC/EDL column and cross-out the value in

YES NO N/A

the concentration column in order to present the data as a non-detect.

B. If the concentration in the sample is higher than five times the contamination concentration in the blank, no action is needed.

11.0 Labeled Compound Recoveries

11.1 Were the samples spiked with all the labeled compounds as specified in the method? [___] ___ ___

11.2 Have labeled compounds' recoveries been within the required limits? [___] ___ ___

11.3 If not, were samples reanalyzed? [___] ___ ___

ACTION: 1. If the labeled compound recovery was below 25 percent, reject "R" all associated non-detect data (EMPC/EDL) and flag with "J" the positive data for the associated compound.

2. If the labeled compound recovery is above the upper limit (150 percent) flag associated positive data with "J". No effect on non-detects.

3. If the labeled compound recovery is less than 10%, qualify positive hits and non-detects associated with the failed labeled compound "R" (Reject). When highly toxic analytes (TEF $\geq$ 0.05) are affected, notify Project Officer to initiate reanalysis.

Recalculate the percent recovery for each labeled standard in the sample extract, Rec_l, using the formula:

$$\% \, Rec_l = \frac{(A_{l1} + A_{l2}) \times Q_{is} \times 100}{(A_{is1} + A_{is2}) \times RF \times Q_l}$$

$A_{l1} + A_{l2}$ = integrated areas of the two quantitation ions of the appropriate labeled compound.

$A_{is1} + A_{is2}$= integrated areas of the two quantitation ions of the appropriate internal standard.

Q_l = quantity of the appropriate labeled compound

Q_{is} = quantity of the appropriate internal standard injected

RF was defined, previously.

12.0 Internal Standard Area Response

There is no method criterion for the Internal Standard area response. However, because it is very critical in determining instrument sensitivity, the Internal Standard area response should be checked for every sample. The two standards $^{13}C_{12}$1,2,3,4-TCDD and $^{13}C_{12}$1,2,3,7,8,9-HxCDD are referred to as Internal Standards in this method. In other Dioxin methods, the two standards are called Recovery Standards.

12.1 Are the internal standard areas for every sample and blank within the upper and lower limits of each associated initial calibration CS3?

Area upper limit= +100% of internal standard area.
Area lower limit= -50% of internal standard area. [___] ___ ___

12.2 Is the retention time of each internal standard within 15 seconds of the associated initial calibration CS3 standard? [___] ___ ___

YES NO N/A

ACTION: 1. If the internal standard area is outside the upper or lower limits, flag all related positive and non-detect data (EMPC/EDL) with "J" regardless whether the lab's labeled compound recoveries met specifications or not.

2. If extremely low area counts (<25%) are reported, flag all associated non-detect data as unusable "R" and the positive data "J".

3. If the retention time of the internal standards differs by more than 15 seconds from the initial calibration CS3, use professional judgement to determine the effect on the results. A time shift of more than 15 seconds may cause certain analytes to elute outside the retention time window established by the GC window defining/column performance check solution. A constant shift could be also the result of a leak.

NOTE: Action 1 and 2 are recommendations only since this criterion is not a method requirement. These guidelines are based on other methods, previously validated data packages and Region II recommendations. If method blanks have low area responses as well as the samples, the validator should seriously consider qualifying the data for this criterion. Action 3 is a method requirement.

13.0 Second Column Confirmation

13.1 Any sample in which 2,3,7,8-TCDF is identified on a DB-5 column, must have a confirmation analysis (Method 1613**B**, section **16.5**). Was a second column confirmation performed? [___] ___ ___

13.2 Was the sample extract reanalyzed on a 30 m DB-225, fused silica capillary column, for 2,3,7,8-TCDF using the GC/MS conditions given in Section **10.1.1** of the analytical method? [___] ___ ___

NOTE: The concentration of 2,3,7,8-TCDF obtained from the primary column (DB-5) should only be used for qualification, due to better QC data associated with the primary column. Also note that the confirmation and quantitation of 2,3,7,8-TCDF may be accomplished on a SP-2330 GC column.

ACTION: If confirmation is missing, use professional judgement, or contact the Project Officer for assistance.

13.3 Did the second column meet the calibration and linearity specification in Sections 5.0 and 6.0 above? [___] ___ ___

ACTION: If no, refer to section 5.0 and 6.0 for appropriate action.

13.4 Was the % D of the quantitation results of the two columns less than 50? [___] ___ ___

ACTION: Note in data assessment the differences, use professional judgement to decide which column data to report for TCDF. No other action is needed since this is not a method requirement but a technical recommendation.

14.0 Sample Reanalysis

14.1 The Project Officer will evaluate the need for reanalyzing the samples with qualified data based on site-specific Data Quality Objectives.

14.2 Due to a variety of situations (see below) that may occur during sample analysis, the laboratory is required to reanalyze or re-extract and reanalyze certain samples. If a reanalysis was required but was not performed, contact the Project Officer to initiate reanalysis. List in data assessment all re-extractions and reanalyses

YES NO N/A

and identify the CDD/CDF sample data summaries which must be used by the data user (when more than one analysis is submitted for a sample).

Lab must re-extract and/or re-analyzed samples when the following criteria are not met:

1. Contaminated method blank at concentrations above the minimum levels (Table 2)

2. Labeled compound recoveries outside acceptable ranges **listed on Table 6 of Analytical method.**

3. Exceedance of calibration range by an analyte (dilution or re-extract using a smaller aliquot).

4. Recovery of labeled compounds outside acceptable limits **listed on Table 6 of the Analytical method** in a diluted sample (re-extracted using a smaller aliquot).

ACTION: For criteria 1, 2, or 3, notify the Project Officer to discuss possible re-analysis of sample by the laboratory.

For criteria 4, If the calibration was verified and the re-extracted sample still does not meet labeled recovery requirements, then the method does not apply to the sample. The results are not reportable for regulatory purposes (Method 1613**B**, section **18.4.4**). Notify the Project Officer of problem to initiate re-analysis of sample using a different method. Document in Data Assessment.

15.0 Precision and Recovery (PAR)

The laboratory is required to show initial demonstration of capability, to evaluate and document data quality. Laboratory performance is compared to established performance criteria to determine if results of analyses meet the performance characteristics of the method.

The laboratory must perform and submit data to establish the ability to generate acceptable precision and accuracy.

15.1 Did the laboratory analyzed an Initial Precision and Recovery (IPR) standard as outlined in section **9**.2 required by the analytical method? [___] ___ ___

ACTION: If no, contact the Project Officer to request resubmittals from the laboratory.

If data is not available, discuss with the Project Officer the feasibility of continuing with validation. If a decision is made to proceed with validation, use professional judgement. All data at a minimum should be qualified as estimated "J". Technically according to the method, data and system performance is unacceptable for all compounds. Analyses should not have continued as per the method. Document under contract non-compliance in Data Assessment.

15.2 Did the IPR standard deviation (s) and average concentration (x) passed criteria as outlined in Table **6** of the method? [___] ___ ___

ACTION: If no, refer to action from section 15.1.

The laboratory must analyzed an Ongoing Precision and Recovery standard (OPR) periodically, at the beginning of 12 hour shift after the analysis of the CS3 calibration verification (VER), and before the analysis of any sample in each set .

15.3 Was the Ongoing Precision and Recovery (OPR) standard analyzed at the required frequency? [___] ___ ___

15.4 Did the OPR standard passed the concentration criteria limits in Table **6** of the method? [___] ___ ___

YES NO N/A

ACTION: If no, refer to action from section 15.1. All samples that do not have a passing OPR standard are potentially affected for that analyte.

The following sections may be incorporated in the validation process on a case by case basis depending upon the requirements of the Project Plan. Sometimes a laboratory will provide data for some of the following sections on a routine basis. If not a requirement of the Project Plan, then professional judgement is needed to qualify data based on additional information.

16.0 Isomer Specificity and Toxicity Equivalency Factor (TEF)

NOTE: The TEF value concentrations can be found in the DFLM01.1 Statement of Work for Dioxin Analysis Form I PCDD-2.

When calculating the 2,3,7,8-TCDD Toxicity Equivalency of a sample only those 2,3,7,8 substituted isomers that were positively identified in the sample must be included in the calculations. The sum of the TEF adjusted concentration is used to determine when a second column confirmation is required to achieve analyte specificity.

16.1 Did the lab include EMPC or EDL values in the toxicity equivalency calculations? [___] ___ ___

16.2 Were all samples, whose toxicity equivalency exceeded the required values were reanalyzed on a confirmation column to establish analyte specificity? [___] ___ ___

ACTION: 1. If yes, the toxicity equivalency calculations were not calculated properly, notify the Project Officer to arrange for laboratory resubmittals.

2. If the toxicity equivalency exceeded the required limits (0.7 µg/Kg for soil/ sediment, 7 ng/L for aqueous and 7 µg/Kg for chemical waste samples), and the lab failed to reanalyze the samples on a specific secondary column, notify Project Officer. Reanalysis may be initiated.

NOTE: Any qualifications such as "J" applied to target compounds should be also applied to their associated total congeners concentration.

17.0 Rinsate Blank *(Region 2 QA guidelines recommend rinse blanks for all projects)*

17.1 One rinsate blank should be collected for each batch of 20 soil samples or one per day whichever is more frequent. Were rinsate blanks collected at the above frequency? [___] ___ ___

17.2 Do any rinsate blanks show the presence of 2,3,7,8-TCDD, 2,3,7,8-TCDF, and 1,2,3,7,8-PeCDD at amounts > .5 µg/L or any other analyte at levels > 1 µg/L? [___] ___ ___

ACTION: If any rinsate blank was found to be contaminated with any of the CDDs/CDFs notify the Project Officer to discuss what proper action must be taken.

If any qualification is needed due to rinsate blank contamination, follow the guidelines outlined under Method Blanks, section 10, Actions 2 and 3.

18.0 Field Blanks

18.1 The field blanks are PEM samples (blind blanks) supplied to Laboratory at the frequency of one field blank per 20 samples or one per samples collected over a period of one week, which ever comes first. A typical "field blank" will consist of uncontaminated soil. The field blanks are used to monitor possible cross contamination of samples in the field and in the laboratory.

Were the following conditions met?

		YES	NO	N/A
18.2	Acceptable field blanks must not contain any signal of 2,3,7,8-TCDD, 2,3,7,8-TCDF, 1,2,3,7,8-PeCDD and 1,2,3,7,8-PeCDF equivalent to a concentration of > 20 ng/Kg.	[___]	___	___
18.3	For other 2,3,7,8 substituted CDD/CDF analytes of each homologue the allowable concentration in the field blank is less than the upper MCLs listed in the method.	[___]	___	___

ACTION: When the field blank is found to be contaminated with target compounds, apply the same action as described for the Method Blank, section 10, Actions 2 and 3.

NOTE: Ask Project Officer to verify that the PEM blank (field blank) did not contain any CDD/CDF analytes and ask their assistance in the evaluation of the PEM field blank.

19.0 PEM Interference Fortified Blanks

NOTE: This type of blank may not be available at this time. In many cases, laboratories will substitute matrix spike/matrix spike duplicate (MS/MSD). If a PEM Interference Fortified blank(s) were not analyzed but MS/MSD data were submitted, skip this section and go onto to section 21.

19.1	One known blank usually an interference fortified soil/sediment sample is supplied to the Laboratory. The frequency of this QC sample is one per group of 20 environmental samples or one per samples collected over one week period, whichever occurs first. The sample is spiked by the laboratory with the appropriate volume of the matrix spiking solution and then extracted and analyzed with other samples.			
19.2	Was a fortified PEM blank analyzed at the frequency described above?	[___]	___	___
19.3	Was the percent recovery of 2,3,7,8-TCDD and other 2,3,7,8-substituted compounds within the 50 to 150 percent control limits?	[___]	___	___

ACTION:
1. If the recovery of a 2,3,7,8-substituted analytes falls outside the 50-150 percent control limit, flag all positive and non-detect data of the same and related analytes in the same homolog series with "J". However, if the recovery is below 20%, qualify all associated non-detects "R" and positive hits as "J". Notify the Project Officer. Reanalysis may be initiated.
2. If no fortified PEM blank was analyzed, use professional judgement to assess data validity.

20.0 Matrix Spike (MS) Field Sample

Note: Matrix spike is not required by this method although Labs may routinely perform this analysis as part of internal QA/QC and submit this data as part of the package. Verify requirements with Project Officer.

20.1	Was a matrix spike analyzed at the frequency of one per SDG samples per matrix?	[___]	___	___
20.2	Was the percent recovery of 2,3,7,8-TCDD and other 2,3,7,8-substituted CDDs/CDFs within 60 to 140 percent?	[___]	___	___

ACTION: If problems such as interferences are observed, use professional judgement to assess the quality of the data. The 60-140% limits of the matrix spike data may be used to flag data of the spiked sample only. The matrix spike data of the PE blank sample are more important and must be used primarily in data validation.

20.3	Was a matrix spike duplicate analyzed as per section 11.1 and 11.2?	[___]	___	___

YES NO N/A

ACTION: No action required. A matrix spike duplicate is not required. Use professional judgement if there is a large difference in concentrations reported between MS and MSD. Qualifications if any, can only be performed on the sample that was used for this criteria.

21.0 Environmental Duplicate Samples *(recommended in Region 2 for all Projects)*

NOTE: Do not confuse an environmental duplicate with a matrix spike duplicate. An environmental duplicate is a sample that has been divided into 2 parts (extracted and analyzed as two different samples) or as 2 separate samples from the same location sent by the sampling crew. This sample is not spike with any additional compounds other than those compounds required by the method for analysis of all routine samples.

21.1 For every batch of 20 samples or samples collected over a period of one week, whichever is less, there must be a sample designated as duplicate. Were duplicate samples collected at the above frequency? [___] ___ ___

21.2 Did results of the duplicate samples agree within 25% relative difference for 2,3,7,8-substituted analytes and 50% for the rest of the analytes? [___] ___ ___

ACTION: The duplicate results can be used in conjunction of other QC data. Use professional judgement.

22.0 REFERENCES

The following references are cited in Method 1613. They are important references for technical information and are submitted here as part of this method's documentation.

1. "Analytical Procedures and Quality Assurance Plan for the Determination of PCDD/PCDF in Fish", USEPA Environmental Research Laboratory, 6201 Congdon Boulevard, Duluth, NH 55804, April 1988.

2. Barnes, Donald G., Kutz, Frederick W., and Bottimore, David P., "Update of Toxicity Equivalency Factors (TEFs) for Estimating Risks Associated with Exposure to Mixtures of Chlorinated Dibenzo-p-dioxins and dibenzofurans (CDDs/CDFs)", Risk Assessment Forum, USEPA, Washington, DC 20460, February 1989.

3. Lamparski, L.L., and Nestrick, T.J., "Determination of Tetra-, Hexa-, Hepta-, and Octachlorodibenzo-p-dioxin Isomers in Particulate Samples at Parts per Trillion Levels", Analytical Chemistry, 52: 2045-2054, 1980.

4. "Measurment of 2,3,7,8-Tetrachlorinated Dibenzo-p-dioxin (TCDD) and 2,3,7,8-Tetrachlorinated Dibenzofurans (TCDF) in Pulp, Sludges, Process Samples and Waste-waters from Pulp and Paper Mills", Wright State University, Dayton, OH 45435, June 1988.

5. Method 1613-Revision **B-** Tetra through Octa- chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, USEPA, Washington, DC, 20460, October **1994**

6. "Method 613--2,3,7,8-tetrachlorodibenzo-p-dioxin", 40 CFR 136 (49 FR 43234), October 26, 1984, Section 4.1.

7. "NCASI Procedures for the Preparation and Isomer Specific Analysis of Pulp and Paper Industry Samples for 2,3,7,8 TCDD and 2,3,7,8 TCDF", National Council of the Paper Industry for Air and Stream Improvement, 260 Madison Avenue, New York, NY 10016, Technical Bulletin No.551, Pre-release Copy, July 1988.

8. Provost, L.P., and Elder, R.S., "Interpretation of Percent Recovery Data", American Laboratory, 15: 56-83, 1983

YES NO N/A

9. Stanley, John S., and Sack, Thomas M., "Protocol for the Analysis of 2,3,7,8-Tetrachlorodibenzo-p-dioxin by High Resolution Gas Chromatography/High Resolution Mass Spectrometry", USEPA EMSL, Las Vegas, Nevada 89114, EPA 600/4-86-004, January 1986.

10. Tondeur, Yves, "Method 8290: Analytical Procedures and Quality Assurance for Multimedia Analysis of Polychlorinated Dibenzo-p-dioixin and Dibenzofurans hy High Resolution Gas Chromatography/High Resolution Mass Spectrometry", USEPA ENSL, Las Vegas, Nevada, June 1987.

11. Tondeur, Yves, "Proposed GC/MS Methodology for the Analysis of CDDs and CDFs in Special Analytical Services Samples", Triangle Laboratories, Inc., 801-10 Capitola Dr., Research Triangle Park, NC 27713, January 1988; updated by personal communication September 1988.

ATTACHMENT A

CDFs/CDD DATA ASSESSMENT

SDG No.
LABORATORY:
SITE:

DATA ASSESSMENT

The current Functional Guidelines for evaluating dioxin/furans organic data have been applied.

All data are valid and acceptable except those analytes which have been qualified with a "J" (estimated), "N" (presumptive evidence for the presence of the material), "U"(non-detects), "R" (unusable), or "JN"(presumptive evidence for the presence of the material at an estimated value). All action is detailed on the attached sheets.

Two facts should be noted by all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems, the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they can not be relied upon, even as a last resort. The second fact to keep in mind is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.

Reviewer's
Signature:_____ Date:__/__/200_

Verified By:_____ Date:___/___/200_

GENERAL COMMENTS:

HOLDING TIME:

BLANK CONTAMINATION:

WINDOW DEFINING MIXTURE:

ION ABUNDANCE:

CALIBRATIONS:

RESOLUTION:

LABELED STANDARDS PERFORMANCE:

INTERNAL STANDARDS:

PEAK IDENTIFICATION:

MATRIX SPIKE/ ENVIRONMENTAL DUPLICATE:

CONFIRMATIONS:

OTHER QC OUT OF SPECIFICATION:

SYSTEM PERFORMANCE AND OVERALL ASSESSMENT:

CONTRACT PROBLEMS NON-COMPLIANCE:

RE-EXTRACTION, REANALYSIS OR DILUTIONS:

DO NOT USE **USE**

FIELD DOCUMENTS:

VALIDATION SOP

PCB Congener

EDS SOP: Congener PCB, Rev.3, 7/10

Environmental Data Services
Congener PCBs (USEPA METHOD 1668A)
Data Validation Checklist

SITE:

DATE:

SDG:

	Y	N	N/A
Data Completeness and Deliverables			
Are the Field Chain of Custody Forms present for all samples?	_____	_____	_____
Are the Narrative, extraction logs, % solid worksheet, analysis logs and Cover Letter present?	_____	_____	_____
Do the Field Chain of Custody Reports or Lab Case Narrative indicate problems with sample receipt, sample condition, analytical procedures, or other comments regarding the quality of the data?	_____	_____	_____

ACTION: Use professional judgment to evaluate the effect of the noted problems on the quality of the data.

ACTION: If any solid sample analyzed contains 50% to 90% water, all data shall be flagged as estimated "J". If a solid sample contains more than 90% water, then qualify positive hits "J" and non-detects "R".

Preservation Requirements			
Is the cooler temperature ≤ 10°C for aqueous and soil samples from the time of collection until receipt at the laboratory?	_____	_____	_____

ACTION: If cooler temperature >10°C, flag non-detects as "UJ" and detects as "J".

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	Y	N	N/A
Reporting Requirements and Deliverables			
Are the following forms present?			
Toxic CB Congener Sample Data Summary (Form I CB-1)?	_____	_____	_____
Toxic CB Congener Toxicity Equivalency (Form I CB-2)?	_____	_____	_____
CB Congener Sample Data Summary (Form I CB-3)?	_____	_____	_____
NOTE: Form I is used for tabulating and reporting sample analyses, including dilutions, reanalyses, blanks, LCS/Ongoing Precision and Recovery (OPR) and requested matrix spike and matrix spike duplicates for target compounds.			
CB Congener Total Homologue Concentration Summary (Form II CB)?	_____	_____	_____
NOTE: Form II is used to report the concentration of the mono- through nano-chloro biphenyl homologue for each sample.			
CB Congener Method Blank Summary (Form IV CB-1)?	_____	_____	_____
NOTE: Form IV summarizes the sample associated with each method blank analysis.			
CB Congener Descriptor Switching Resolution Summary (Form V CB-1)?	_____	_____	_____
NOTE: Form V CB-1 is used to report the descriptor switching windows for each level of chlorination for each 12-hour time period and to summarize the date and time of analysis, including dilutions, reanalysis, standards, blanks, and requested MS/MSD associated with each analysis of the instrument performance check solution.			
CB Congener Ion Abundance Ratio Summary (Form V CB-2)?	_____	_____	_____
CB Congener (Labeled) Ion Abundance Ion Ratio Summary (Form V CB-3)?	_____	_____	_____
NOTE: Form V CB-2 & CB-3 are used to report the ion abundance ratios and signal-to-noise (S/N) ratios for the congeners contained in the LOC/WDM for each 12-hour time period.			
Toxic CB Congener Initial Calibration Response Factor Summary (Form VI CB-1 & CB-2)?	_____	_____	_____
Individual Congener Initial Calibration Response Factor Summary (Form VI CB-3)?	_____	_____	_____

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	Y	N	N/A
NOTE: Form VI is used to report the relative response factors (RRF), average RRF, % RSD and RRT for the five or six-point initial calibration at the specific concentration levels.			
Toxic CB Congener Continuing Calibration Summary (Form VII CB-1)?	_____	_____	_____
Toxic CB Congener (Labeled) Continuing Calibration Summary (Form VII CB-2)?	_____	_____	_____
Individual Congener Continuing Calibration Summary (Form VII CB-3)?	_____	_____	_____
Toxic CB Congener Continuing Calibration Time Summary (Form VII CB-4)?	_____	_____	_____
Toxic CB Congener (Labeled) Continuing Calibration Summary (Form VII CB-5)?	_____	_____	_____
Individual Congener Continuing Calibration Summary (Form VII CB-6)?	_____	_____	_____
NOTE: Form VII is used to report the calibration verification of the HRGC/HRMS system by the analysis of specific calibration verification standard(s). This form is required for each continuing calibration (one every 12 hours of sample analysis).			
CB Congener Analytical Sequence (Form VIII CB)?	_____	_____	_____
NOTE: Form VIII is used to report the analytical sequence for CB congener analyses.			
ACTION: If deliverables are missing, contact the laboratory to request explanation/resubmittal. Note in the Data Assessment.			

Holding Times

Were the following holding times met:

Aqueous samples were extracted within 30 days of sample collection	_____	_____	_____
Solid samples were extracted within 30 days of sample collection	_____	_____	_____
All samples were analyzed within 40 days of extraction	_____	_____	_____

ACTION: If holding times are exceeded, flag all data as estimated "J". Holding time criteria do not apply to PE samples.

If holding times from collection to extraction, or from extraction to analysis have been grossly exceeded, use professional judgment to determine whether non-detects shall be rejected.

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Column Performance	**Y**	**N**	**N/A**
Column performance is assessed based on the evaluation of data obtained from the daily (12-hr. shift) diluted combined 209 congener solution.			
The retention time for decachlorobiphenyl (PCB 209) must be greater than 55 minutes.	_____	_____	_____
The column, SPB-Octyl, must uniquely resolve congeners 34 from 23 and 187 from 182, and congeners 156 and 157 must co-elute within 2 seconds at peak maximum.	_____	_____	_____

An alternative Column Performance criteria when using DB-1 is as follows:

a. The retention time for decachlorobiphenyl must be greater than 55 minutes
b. The column must resolve the following congerers:
   i. 28,31 with a valley height less than 50 percent
   ii. 66,70,74,80 with a valley height less than 40 percent
   iii. 123,118 with a valley height less than 40 percent
   iv. 156,157 with a valley height less than 40 percent

NOTE: Unique resolution means a valley height of less than 40% of the shorter of the two peaks.

ACTION: If column performance criteria were not met, a detailed discussion of the problems observed and potential effects on analytical data obtained should be included in the data assessment narrative.

Initial 5-Point Calibration

The initial calibration standard solutions (CS1-CS5) must be analyzed prior to any sample analysis. These calibration standards should include all CBs listed at the concentrations specified in Table 5 of Method 1668A. They do not have to be analyzed daily provided the continuing calibration standard met all criteria. The calibration standards must be analyzed on the same instrument using the same GC/MS conditions that were used to analyze samples.	_____	_____	_____
SIM data were acquired for each of the ions listed in Table 7, Method 1668A	_____	_____	_____

The following criteria must be met:

For all target analyte PCBs with associated labeled analogs, the two ions for each CB must maximize simultaneously and within 3 seconds of the corresponding labeled isomer ions.	_____	_____	_____

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	Y	N	N/A
For the other CBs, the 2 ions for each of the CBs being quantified must maximize simultaneously.	_____	_____	_____
The relative ion abundance criteria listed in Table 8, Method 1668A must be met.	_____	_____	_____
The relative ion abundance criteria for the labeled internal and recovery standards listed in Table 8, Method 1668A must be met.	_____	_____	_____
For all calibration solutions, the signal to noise ration (S/N) for all ions of the native compounds must be greater than 10.	_____	_____	_____
For the internal and recovery standards, the signal to noise ratio for all ions must be greater than 10.	_____	_____	_____
The percent relative standard deviation (%RSD) of the five RRFs (CS1-CS5) for the CBs must not be greater than 20 percent.	_____	_____	_____

Spot- check response factor calculations and ion ratios. Ensure that the correct quantitation ions for the unlabeled CBs and internal standards were used. In addition verify that the appropriate internal standard was used for each isomer.

> ACTION: If required information was not supplied, notify the lab. If unavailable, reject all associated sample data. If any calibration curve standards fail to meet any acceptance criteria, check for re-calibration and re-analysis.

Calibration by Isotopic Dilution Procedure
(Native CBs included in the Toxics/LOC CBs list)

To recalculate the response factor for targets quantitated based on isotopic dilution, use the equation:

$$RRFn = \frac{(An^1 + An^2) \times Cis}{(Ais^1 + Ais^2) \times Cn}$$

Where:

A_n^1 and A_n^2	=	*integrated areas of the two quantitation ions of isomer of interest*
A_{is}^1 and A_{is}^2	=	*integrated areas of the two quantitation ions of the appropriate labeled compound.*
C_{is}	=	*The concentration of the labeled compound in the calibration standard.*
Cn	=	*The concentration of the native compound in the calibration standard.*

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Calibration by Internal Standard **Y** **N** **N/A**
(Native CBs not included in the Toxics/LOC CBs list)

Internal standard calibration is performed at a single point using the diluted combined 209-congener solution (Table 3, Method 1668A).

To calculate the response factor for targets based on the internal standard method, use the equation:

$$RF = \frac{(A1_S + A2_s)\, C_i s}{(A1is + A2is)\, C_s}$$

Where:

$A1_s$ and $A2_s$	=	*The areas of the primary and secondary m/z's for the PCB.*
$A1_{is}$ and $A2_{is}$	=	*The areas of the primary and secondary m/z's for the internal standard.*
C_{is}	=	*The concentration of the internal standard.*
C_s	=	*The concentration of the compound in the calibration standard.*

Continuing Calibration

At the beginning of each12-hour shift during which analyses are performed, GC/MS system performance and calibration are verified for all native CBs and labeled compounds. For these tests, analysis of the Cs-3 calibration verification (VER) standard (Section 7.10.1 and Table 5, Method 1668A) and the diluted compound 209-congener solution (Section 7.10.2.2 and Table 5, Method 1668A) must be used to verify all performance criteria. Adjustment and/or recalibration (Section 10, Method1668A) must be performed until all performance criteria are met. Only after all performance criteria are met, may samples, blanks, IPRs and OPRs be analyzed.

Was the continuing calibration run at the required frequency? _____ _____ _____

ACTION: If the continuing calibration standard was not analyzed at the required frequency, reject all the data.

For native Toxics and LOC CBs, the two SIM ions for each PCB must maximize simultaneously and within 3 seconds of the corresponding ions of the labeled isomers. _____ _____ _____

The two ions for each of the CBs being quantified must maximize simultaneously _____ _____ _____

	Y	N	N/A
For the continuing calibration standard solution, the signal to noise ratio (S/N) for the CB ions shall be greater than 10.	_____	_____	_____
The absolute retention times of the labeled Toxics/LOC/window defining standard congeners in the verification test must be within ± 15 seconds of the respective retention times in the calibration. Has this criteria been met?	_____	_____	_____
The relative retention times of native CBs and labeled compounds in the verification test must be within their respective RRT limits in Table 2, Method 1668A. Has the criteria been met?	_____	_____	_____
For the internal standards and the recovery standards, the signal to noise ratio (S/N) shall be greater than 10.	_____	_____	_____
The relative ion abundance criteria, Table 8, Method 1668A for all PCBs native shall be met.	_____	_____	_____
Evaluate the concentrations of the Toxics/LOC CBs in the CS-3 VER standard.			
For each compound, compare the concentration with the calibration verification limit in Table 6, Method 1668A. If all compounds meet the acceptance criteria, calibration has been verified and analysis of standards and sample extracts may proceed. Was this criteria met?	_____	_____	_____

Spot-check continuing calibration concentrations and ion ratios. Verify that the appropriate quantitation ions were used for the unlabeled PCBs and internal standards.

> ACTION: If the required continuing calibration fail to meet any of the acceptance criteria, check for recalibration and reanalysis. If not performed, reject ("R" flag) all data associated with the analyte out of specification in the continuing calibration.

Sample Data

SIM data were acquired for each of the ions listed in Table 7, Method1668A	_____	_____	_____

Identification Criteria

A. For native Toxics and LOC CBs, the two ions for each of the PCB's being quantified must maximize simultaneously and within 3 seconds of the corresponding labeled standard.

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	Y	N	N/A
For the remaining CBs, the two ions for each of the CBs being quantified must maximize simultaneously.	_____	_____	_____
B. The integrated ion current for each characteristic ion of the analyte identified as positive must be at least 2.5 times background noise and must have not saturated the detector.	_____	_____	_____
C. The integrated ion current for the internal and recovery standard characteristic ions must be at least 10 times background noise.	_____	_____	_____
D. The relative ion abundance criteria (Table 8, Method 1668A) for all PCB's found present must be met or ion ratios must be within ± 15% of the most recent CS-3 determination.	_____	_____	_____
E. The relative retention time of the peak for a positively identified CB must be within the RRT QC limits specified in Table 2, Method 1668A, or, if an alternate column or column system is employed, within its respective RRT QC limits for the alternate column or column system.	_____	_____	_____

ACTION: Reject ("R" flag) all positive data for the analytes which do not meet criteria listed in section A.

If the criteria listed in section B are not met but all other criteria are met, qualify all positive data of the specific analyte with "J".

If the requirements listed in section C are not met but all other requirements are met qualify the positive data of the corresponding analytes with "J".

If the analytes reported positive do not meet ion abundance criteria, section D, or do not meet RRT criteria, Section E, reject "R" all positive data for these analytes. Change the positive values to EMPC (estimated maximum possible concentration).

	Y	N	N/A
Spot check calculations for positive data and verify that the same internal standards used to calculate RRFs were used to calculate concentration.	_____	_____	_____

Compute the concentrations in the extract of the Native Toxics/LOC CBs, using the RRs from the calibration data and following equation:

$$C_{ex}\ (ng/mL) = \frac{(A1_n + A2_n)\ C_I}{(A1_I + A2_I)\ RR}$$

Where:

C_{ex} = *The concentration of the PCB in the extract, and the other terms as defined in Section 10.5.1, Method 1668A.*

Internal Standard Quantitation and Labeled Compound Recovery

Compute the concentrations in the extract of the native compounds other than those in the Native Toxics/LOC standard, in the Labeled cleanup standard, and in the Labeled injection internal standard (except for Labeled CB178), using the response factors determined from the calibration data and the following equation:

$$C_{ex}\ (ng/mL) = \frac{(A1_s + A2_s)\ C_{is}}{(A1_{is} + A2_{is})\ RF}$$

Where:

C_{ex} = *The concentration of the labeled compound in the extract. The other terms are defined on page 7 under calibration by Internal Standard.*

Using the concentration in the extract determined above, compute the percent recovery of the Labeled Toxics/LOC/window-defining CBs and the Labeled cleanup standard CBs using the following equation:

$$Recovery\ (\%) = \frac{Concentration\ found\ (ug/mL)\ x\ 100}{Concentration\ spiked\ (ug/mL)}$$

The concentration of a native CB in the solid phase of the sample is computed using the concentration of the compound in the extract and the weight of the solids as follows:

$$Concentration\ in\ solid\ (ng/kg) = \frac{(C_{ex}\ x\ V_{ex})}{W_s}$$

Where:

C_{ex} = *The concentration of the compound in the extract.*

V_{ex} = *The extract volume in mL.*

W_s = *The sample weight (dry weight) in kg.*

The concentration of a native CB in the solid phase of the sample is computed using the concentration of the compound in the extract and the weight of the solids as follows:

$$Concentration\ in\ solid\ (ng/kg) = \frac{(C_{ex}\ x\ V_{ex})}{W_s}$$

Where:

C_{ex} = *The concentration of the compound in the extract.*

V_{ex} = *The extract volume in mL.*

W_s = *The sample weight (dry weight) in kg.*

	Y	N	N/A

The concentration of a native CB in the aqueous phase of the sample is computed using the concentration of the compound in the extract and the volume of water extracted as follows:

$$\text{Concentration in aqueous phase (pg/L)} = \frac{(C_{ex} \times V_{ex})}{V_s}$$

Where:

C_{ex} = *The concentration of the compound in the extract.*

V_{ex} = *The extract volume in mL.*

V_s = *The sample volume in liters.*

Method Blanks

Has a method blank been extracted and analyzed with each batch of 20 samples or less, per matrix? _____ _____ _____

Was the method blank analyzed immediately after the OPR? _____ _____ _____

ACTION: If the proper number of method blanks were not analyzed, notify the contractor. If the reviewer is unavailable, reject all positive sample data. However, the reviewer may also use professional judgment to accept or reject positive sample data if no blank was run.

Were any PCBs found at a concentration greater than the PQL in the associated method blank? _____ _____ _____

ACTION: If an analyte has a reported concentration that is > 5 times the PQL, qualify "J" and all concentrations ≤ 5 times the PQL are qualified "U" due to possibility of contamination.

If the congener concentration in the sample is less than 5x the concentration in the blank, flag the congener reported value with a “U” qualifier.

If the concentration of the congener in the sample is greater than 5x the concentration in the blank, no action is needed.

Rinsate Blank

Was a rinsate blank submitted for analysis for each analytical batch not to exceed 20 samples? _____ _____ _____

Do any rinsate blanks contain concentrations of PCBs above the PQL? _____ _____ _____

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	Y	N	N/A
ACTION: If an analyte has a reported concentration that is > 5x the PQL, qualify "J" and all concentrations ≤ 5x the PQL are qualified "U" due to possibility of contamination.			
If the congener concentration in the sample is less than 5x the concentration in the blank, flag the congener reported value with a "U" qualifier.			
If the concentration of the congener in the sample is greater than 5x the concentration in the blank, no action is needed.			

Labeled Compound Recoveries

Were the samples spiked with all the internal standards as specified in the method?	_____	_____	_____
Were internal standard recoveries within the method required limits (Table 6, Method1688A)?	_____	_____	_____
If not, were samples reanalyzed?	_____	_____	_____

ACTION: If the internal standard recovery was outside the acceptance limits (Table 6, Method 1668A) "J" flag all associated positive results.

Labeled Injection Internal Standards

Internal standard areas for every sample and blank within the upper and lower limits of each associated continuing calibration?	_____	_____	_____

Area upper limit = +100% of internal standard area.
Area lower limit = -50% of internal standard area.

Is the retention time of each internal standard within 10 seconds of the associated daily calibration standard?	_____	_____	_____

ACTION: If the internal standard area is outside the upper or lower limits flag all related positive and non-detect data with "J" regardless of whether or not the labeled standard recoveries met specifications.

If extremely low area counts (<25%) are reported, qualify all associated non-detect data as unusable "R" and the positive data "J."

If the retention time of the internal standard differs by more than 10

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	Y	N	N/A
seconds from the daily calibration, use professional judgment to determine the effect on the results. A time shift of more than 10 seconds may cause certain analytes to elute outside the retention time window established by the window defining mix.			

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Sample ID of the sample chosen for MS/MSD analysis: _____

For every sample delivery group of 20 or fewer environmental samples was one MS/MSD pair analyzed?	_____	_____	_____

NOTE: In order for the MS/MSD to be appropriate for use in qualifying or rejecting associated sample data, the spike level must be within the range of 25% to 400% of the reported sample concentration for either the aqueous or soil sample..

Is the MS recovery within the 60 to 140% acceptance range for either an aqueous or solid sample?	_____	_____	_____

ACTION: qualify the value for the analyte in the sample used for the MS/MSD as estimated "J". Use professional judgment in association with signal to noise ratios and internal standard recoveries for the associated sample data to determine the effect on the quality of the associated sample data.

Does the precision of the MS/MSD analyses meet the $\leq$ 50% RPD criteria?	_____	_____	_____

ACTION: Qualify the value for that analyte in the sample used for the MS/MSD as estimated "J"; use professional judgment in association with signal to noise ratios and internal standard recoveries for the associated sample data to determine the effect on the quality of the associated sample data.

Field Duplicate Samples

Sample IDs of the field duplicate pair: _____

For every batch of 20 samples or less collected there must be a sample designated as duplicate.	_____	_____	_____

NOTE: For Aqueous: RPD $\leq$ 20% when target is detected in both field duplicate samples at $\geq$ 5XPQL or concentrations differ by less than 2X the PQL when detects are < 5X PQL for both field duplicate samples.

For Soil: RPD ≤ 50% when target is detected in both field duplicate samples at ≥ 5x PQL, or concentration differs by less than 2x the PQL when detects are < 5x PQL for both field duplicate samples.

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	Y	N	N/A
ACTION: The duplicate results must be used in conjunction with other QC data. If no hits are reported, precision may be assessed from the internal standard recoveries.			

Ongoing Precision and Recovery (OPR)

For every batch of 20 samples or less processed by the laboratory, was an OPR processed?	_____	_____	_____
Do the calculated percent recoveries for all CBs in the associated OPR meet the acceptance limits provided in Table 6, Method 1668A?	_____	_____	_____

ACTION: If the laboratory failed to process an OPR sample with the associated field samples, qualify all target analyte results as estimated “J”.

ACTION: If any CB OPR percent recoveries failed to meet the acceptance criteria listed in Table 6, Method 1668A, qualify detects sample results as estimated "J".

Initial Precision and Recovery (IPR)

Was an initial precision and accuracy demonstration performed for the appropriate matrix as per section 9.2 of Method 1668B?	_____	_____	_____
Were the results of the IPR evaluation acceptance when compared to the acceptance limits for each analyte listed in Table 6, Method166A?	_____	_____	_____

ACTION: If IPR date was not provided by the laboratory, contact the lab to obtain the results of the IPR study.

ACTION: If the results of the laboratories IPR study do not meet the acceptance criteria for performance tests listed in Table 6, Method 1668A, contact the lab to initiate remediation of technical difficulties.

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Data Validation Qualifiers

Qualifier	Description
J	Estimated value (bias undetermined) – The analyte was positively identified; but the associated numerical value is the approximate concentration of the analyte in the sample.
JH	Estimated value (potential high bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential high bias, of the analyte in the sample.
JL	Estimated value (potential low bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential low bias, of the analyte in the sample.
UJ	Estimated non-detect - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
UJL	Estimated non-detect (potential low bias) – The analyte was not detected and the report sample quantitation limit is biased low.
UJH	Estimated non-detect (potential high bias) – The analyte was not detected and the reported sample quantitation limit is biased high.
M	The analytical result reported was obtained from a sediment sample found to contain between 50 and 90 percent moisture and had no other data qualifiers added during the data validating process.
NJ	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
NJH	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration with a potential high bias, of the analyte concentration.
EMPC	Estimated Maximum Possible Concentration (EMPC).
R	The sample results are rejected. Due to a significant QA/QC problem, the analysis is invalid and provides no information as to whether the analyte is present or not.

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VALIDATION SOP

TAL Metals, Mercury and Titanium

USEPA Region 2 SOP HW-2b, Rev.15, 12/12

Hazardous Waste Support Section
SOP NO. HW-2b Revision 15
ICP-MS Data Validation

Approvals:

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Approving Official Annual Review — Date

NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (hereafter referred to as USEPA) and other governmental employees. They do not constitute rule making by USEPA, and may not be relied upon to create a substantive or procedural right enforceable by any other person. The Government may take action that is at variance with the policies and procedures in this manual.

This document can be obtained from the USEPA's Region 2 Quality Assurance website at:

http://www.epa.gov/region2/qa/documents.htm

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ACRONYMS

ASB	Analytical Services Branch
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
CO	Contracting Officer
CRQL	Contract Required Quantitation Limit
DF	Dilution Factor
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EDM	EXES Data Manager
ESAT	Environmental Services Assistance Team
EXES	Electronic Data eXchange and Evaluation System
HWSS	Hazardous Waste Support Section
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICS	Interference Check Sample
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
MDL	Method Detection Limit
NIST	National Institute of Standards and Technology
OSRTI	Office of Superfund Remediation and Technology Innovation
OSWER	Office of Solid Waste and Emergency Response
PE	Performance Evaluation
%D	Percent Difference
%R	Percent Recovery
%S	Percent Solids
PO	Project Officer
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center Coordinator
SDG	Sample Delivery Group
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TR/COC	Traffic Report/Chain of Custody Documentation
USEPA	United States Environmental Protection Agency

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TARGET ANALYTE LIST

Al Aluminum
Sb Antimony
As Arsenic
Ba Barium
Be Beryllium
Cd Cadmium
Ca Calcium
Cr Chromium
Co Cobalt
Cu Copper
Fe Iron
Pb Lead
Mg Magnesium
Mn Manganese
Ni Nickel
K Potassium
Se Selenium
Ag Silver
Na Sodium
Tl Thallium
V Vanadium
Zn Zinc

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INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the validity of analytical data generated through the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) ISM01.X Inorganic Superfund Methods (Multi-Media, Multi-Concentration), hereinafter referred to as the ISM01.2 SOW, and any future editorial revisions of ISM01.2. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process.

The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and QC criteria established in the SOW, and (b) the validity and extent of bias of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to site-specific technical requirements, the need to facilitate the progress of specific projects, and availability for re-sampling.

The reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as professional judgment, should also be used to determine the ultimate validity of data, especially in those cases where all data does not meet specific technical criteria.

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DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

DATA PACKAGE INSPECTION

For data obtained through the Contract Laboratory Program (CLP), the EXES Data Manager (EDM) is a useful tool in the data review process. For more information about EDM, please refer to the following Sample Management Office (SMO) website:

https://epasmoweb.fedcsc.com/help/guides/Submit%20and%20Inspect%20Data%20Quick%20Guide%20%28EXES%29.pdf

EDM will identify any missing and/or incorrect information in the data package. The CLP laboratory may submit a reconciliation package for any missing items or to correct data.
If there are any concerns regarding the data package, contact the CLP Project Officer (CLP PO) from the Region where the samples were taken. For personnel contact information, please refer to the following CLP website:

http://www.epa.gov/superfund/programs/clp/contacts.htm

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HWSS DATA VALIDATION PROCESS

After downloading the data package from EDM, the data validator will use the recommendations in this SOP as well as their own professional judgment to validate the data.

The data will be saved in the following location, under the appropriate case number folder:

G:\DESADIV\HWSS\DATA VALIDATION

The file naming conventions will consist of

A. case number i.e., 12345
B. SDG name i.e., MBXY12
C. level of validation performed i.e., S2BVE

Examples: **12345_MBXY12_S2BVE.xls**

12345_MBXY12_S2BVEM.xls

When data validation is completed, the data package is uploaded for the client to download from the HWSS data delivery website:

https://epaqpx.rtp.epa.gov/hwssclpdeliverables

The completed data package includes the Executive Narrative (see Appendix B for template), the Sample Summary Report (see Appendix C for example), and the Electronic Data Deliverable (EDD) (see Appendix D for a list of the column headers included in this document).

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PRELIMINARY REVIEW

This document is for the review of analytical data generated through the ISM01.2 SOW and any future editorial revisions of ISM01.2. To use this document effectively, the reviewer should have an understanding of the analytical method and a general overview of the Sample Delivery Group (SDG) or sample Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the sample data package [e.g., Modified Analysis requests, Traffic Report/Chain of Custody (TR/COC) documentation, SDG Narratives, etc.].

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The reviewer should contact the appropriate Regional Contract Laboratory Program Project Officer (CLP PO) to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

The SDGs or Cases routinely have unique samples that require special attention from the reviewer. These include field blanks and trip blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The sampling records (e.g., TR/COC records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken, and
2. The complete list of samples with information on:
    a. Sample matrix;
    b. Field blanks*;
    c. Field duplicates*;
    d. Field spikes*;
    e. PE samples*;
    f. Shipping dates;
    g. Preservatives;
    h. Types of analysis; and
    i. Laboratories involved.

    * If applicable.

The TR/COC documentation includes sample descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

The laboratory's SDG Narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, preservation, and unusual events should be documented in the SDG Narrative. The reviewer should also inspect any email or telephone/communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office (SMO), and the USEPA Region.

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An Example Analytical Sequence for ICP-MS

Tune
S0
S
S
S
S
S
ICV
ICB
LCS
ICSA
ICSAB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

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Preservation and Holding Times

Action:

NOTE: Apply the action to each sample for which the preservation or holding time criteria was not met.

1. If the pH of aqueous/water metal samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH of the sample to ≤ 2 at the time of sample receipt. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of the metal(s) of interest. Qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-), and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data, based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-), and qualify non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment samples, it is left to the discretion of the data reviewer whether to apply 180 day holding time criteria to soil/sediment samples. If they are applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.
5. When holding times are grossly exceeded, note it for Contract Laboratory Program Project Officer (CLP PO) action.

Table 1. Technical Holding Time Actions for ICP-MS Analysis

Preservation & Holding Time Results	Action for Samples
Aqueous/water metals samples received with pH > 2 and pH not adjusted	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Aqueous/water Metals > 180 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Soil/sediment Metals > 180 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

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ICP-MS Tune Analysis

Action:

NOTES: For ICP-MS tunes that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

1. If the ICP-MS instrument was not tuned prior to calibration, the sample data should be qualified as unusable (R).
2. If the tuning solution was not analyzed or scanned at least 5x consecutively or the tuning solution does not contain the required analytes spanning the analytical range, the reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be recorded in the Data Review Narrative and noted for Contract Laboratory Program Project Officer (CLP PO) action.
3. If the resolution of the mass calibration is not within 0.1 u for any isotope in the tuning solution, qualify all analyte results that are ≥ Method Detection Limit (MDL) associated with that isotope as estimated (J), and all non-detects associated with that isotope as estimated (UJ). The situation should be recorded in the Data Review Narrative and noted for CLP PO action.
4. If the %RSD exceeds 5% for any isotope in the tuning solution, qualify all sample results that are ≥ MDL associated with that tune as estimated (J), and all non-detects associated with that tune as estimated (UJ). The situation should be recorded in the Data Review Narrative and noted for CLP PO action.

Table 2. ICP-MS Tune Actions for ICP-MS Analysis

ICP-MS Tune Results	Action for Samples
Tune not performed	Qualify all results as unusable (R)
Tune not performed properly	Use professional judgment
Resolution of mass calibration not within 0.1u	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)
% RSD > 5%	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)

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Calibration

Table 3. Acceptance Criteria for ICV and CCV Standards

Analytical Method	Inorganic Analytes	ICV/CCV Low Limit (% of True Value)	ICV/CCV High Limit (% of True Value)
ICP-MS	Metals	90	110

Action:

NOTES: For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.
For CCVs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical run.

NOTE: The data validator shall verify the correlation coefficient by calculating it using the standard concentrations and the corresponding instrument response.

1. If the instrument was not calibrated each time the instrument was set up and an ICV standard was not analyzed before field and QC samples, qualify the data as unusable (R). If the instrument was not calibrated with a blank and at least 5 calibration standards, use professional judgment to qualify results that are ≥ Method Detection Limit (MDL) as estimated (J), and non-detects as estimated (UJ). If the calibration curve does not include standards at required concentrations (e.g., a blank and at least one at or below CRQL), use professional judgment to qualify results that are ≥ MDL as estimated (J), and non-detects as estimated (UJ). If there was not at least one calibration standard at or below the CRQL for each analyte, qualify results that are ≥ MDL but < 2x the CRQL as estimated (J), and non-detects as estimated (UJ) and write it in the Contract Problem/Non-Compliance Section of the Data Review Narrative.
2. If the correlation coefficient is < 0.995, percent differences are outside the ±30% limit, or the y-intercept ≥ CRQL, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ). If the correlation coefficient is < 0.990, qualify results that are ≥ MDL as estimated (J) and non-detects as unusable (R).
3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
    a. If the ICV or CCV %R is < 75%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as unusable (R).
    b. If the ICV or CCV %R falls within the range of 75-89%, qualify sample results that are ≥ MDL as estimated low (J-), and qualify non-detects as estimated (UJ).
    c. If the ICV or CCV %R falls within the range of 111-125%, qualify sample results that are ≥ MDL as estimated high (J+).
    d. If the ICV or CCV %R is within the range of 111-125%, non-detects should not be qualified.

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e. If the ICV or CCV %R is > 125%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+). Non-detects should not be qualified.
f. If the %R is > 160%, qualify all results that are ≥ MDL as unusable (R).

4. If the laboratory failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
5. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
6. If calibration criteria are grossly exceeded, note this for CLP Project Officer (CLP PO) action.

NOTE: For critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

Table 4. Calibration Actions for ICP-MS Analysis

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as unusable (R)
Calibration incomplete	Use professional judgment Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)
Not at least one calibration standard at or below the CRQL for each analyte	Qualify results that are ≥ MDL but < 2x the CRQL as estimated (J) Qualify non-detects as estimated (UJ)
Correlation coefficient < 0.995; %D outside ±30%; y-intercept ≥ CRQL	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)
Correlation coefficient < 0.990	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as unusable (R)
ICV/CCV %R < 75%	Qualify results that are ≥ MDL as unusable (R) Qualify all non-detects as unusable (R)
ICV/CCV %R 75-89%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICV/CCV %R 111-125%	Qualify results that are ≥ MDL as estimated high (J+)
ICV/CCV %R > 125%	Qualify results that are ≥ MDL as estimated high (J+)
ICV/CCV %R > 160%	Qualify results that are ≥ MDL as unusable (R)

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Calibration/Preparation Blanks

Action:

NOTES: For ICBs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.
For CCBs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.
For Preparation Blanks that do not meet the technical criteria, apply the action to all samples prepared in the same preparation batch.

NOTES: Convert soil sample result to mg/kg on wet weight basis to compare with the soil preparation result on Form III.
Associated samples are all samples digested with the preparation blank.

1. If the appropriate blanks were not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
    a. Any blank (including Preparation Blanks) reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank (including Preparation Blanks) reported with a negative result, whose value is < (-CRQL) qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
    b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. Specific "method" actions include:
    a. If the absolute value of an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the affected samples were not reanalyzed, report non-detects and results that are ≥ MDL, but ≤ CRQL as CRQL-U. For results that are > CRQL but < Blank Result, report the results at the level of the blank with a "U" qualifier. Use professional judgment to qualify results that are > Blank Result. Note this situation for CLP PO action and record it in the Data Review Narrative.

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b. If the absolute value of the concentration of the Preparation Blank is ≤ CRQL, report non-detects and results that are ≥ MDL but ≤ CRQL as CRQL-U. Use professional judgment to quality results that are > CRQL.

c. If any analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redigested and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redigestion (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redigest and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.

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Table 5. Calibration/Preparation Blank Actions for ICP-MS Analysis

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
ICB/CCB	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < Blank Result	Report at level of Blank Result with a "U"
		> Blank Result	Use professional judgment
ICB/CCB	≤ (-MDL) but ≥ (-CRQL)	≥ MDL, or non-detect	Use professional judgment
ICB/CCB	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)
Preparation Blank	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < 10x the Blank Result	Qualify results as estimated high (J+)
		≥ 10x the Blank Result	No action
Preparation Blank	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
Preparation Blank	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)

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Inductively Coupled Plasma - Interference Check Sample (ICP-ICS)

Action:

NOTE: For an ICS for ICP-MS that does not meet the technical criteria, apply the action to all samples reported from the analytical run.

NOTE: The laboratory should have analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for these reported elements.

1. The raw data may not contain results for interferents. In this case, the reviewer shall use professional judgment to qualify the data. If the data does contain results for interferents, the reviewer should apply the following actions to samples with concentrations of interferents that are comparable to, or greater than, their respective levels in the ICS:
    a. If the ICS %R for an analyte or interferent is > 120% (or greater than the true value + 2x the CRQL, as applicable) and the sample results are non-detects, the data should not be qualified.
    b. If the ICS %R for an analyte or interferent is > 120% (or greater than the true value + 2x the CRQL, as applicable) qualify sample results that are ≥ MDL as estimated high (J+). If the ICS %R (or true value) grossly exceeds the limits, use professional judgment to qualify the data.
    c. If the ICS %R for an analyte or interferent falls within the range of 50-79% (or less than the true value – 2x the CRQL, as applicable) qualify sample results that are ≥ MDL as estimated low (J-).
    d. If the ICS recovery for an analyte falls within the range of 50-79% (or less than the true value – 2x the CRQL, as applicable), the possibility of false negatives exists. Qualify sample non-detects as estimated (UJ).
    e. If the ICSAB %R for an analyte or interferent is < 50%, qualify all sample results that are ≥ MDL and all sample non-detects as unusable (R).
2. If results that are ≥ MDL are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS, qualify sample results that are ≥ MDL as estimated high (J+). Non-detects should not be qualified.
3. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is ≥ MDL, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents, qualify non-detects for the affected analytes as estimated (UJ), and results that are ≥ MDL but < 10x the absolute value of the negative result as estimated low (J-).
4. If the raw data does not contain results for the interferents, note it in the Data Review Narrative.

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5. Actions regarding the interpretation and/or the subsequent qualification of ICP data due to the ICS analytical results can be extremely complex. Use professional judgment to determine the need for the associated sample data to be qualified. The reviewer may need to obtain additional information from the laboratory. All interpretive situations should then be recorded in the Data Review Narrative.
6. If the ICS acceptance criteria are grossly exceeded, note the specifics for CLP PO action.

Table 6. Interference Check Actions for ICP-MS Analysis

Interference Check Sample Results	Action for Samples
ICS %R > 120% (or greater than true value + 2x the CRQL)	Qualify results that are ≥ MDL as estimated high (J+)
ICS %R 50-79% (or less than true value – 2x the CRQL)	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICSAB %R < 50%	Qualify all sample data as unusable (R)
Potential false positives in field samples with interferents	Qualify results that are ≥ MDL as estimated high (J+)
Potential false negatives in field samples with interferents	Qualify results that are ≥ MDL but < 10x the (|negative value|) as estimated low (J-) Qualify non-detects as estimated (UJ)

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Laboratory Control Sample (LCS)

Action:

If the LCS criteria are not met, the laboratory performance and method accuracy are in question. Professional judgment should be used to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data associated with an LCS that does not meet the required criteria.
For an LCS that does not meet the technical criteria, apply the action to all samples in the same preparation batch.

1. **LCS:**
   a. If the LCS %R falls within the range of 40-69%, qualify sample results that are ≥ Method Detection Limit (MDL) as estimated low (J-). If the LCS %R is > 130%, qualify sample results that are ≥ MDL as estimated high (J+).
   b. If the LCS recovery is > 130% and the sample results are non-detects, the data should not be qualified.
   c. If the LCS recovery falls within the range of 40-69%, qualify non-detects as estimated (UJ).
   d. If LCS %R is < 40%, qualify all results that are ≥ MDL as estimated low (J-) and all non-detects as unusable (R).
   e. If the LCS %R is > 150%, qualify all affected data (both detects and non-detects) as unusable (R).
2. If a laboratory fails to analyze an LCS with each SDG, or if a laboratory consistently fails to generate acceptable LCS recoveries, note this for CLP Project Officer (CLP PO) action.
3. Whenever possible, the potential effects on the data due to out-of-control LCS results should be noted in the Data Review Narrative.

Table 7. LCS Actions for ICP-MS Analysis

LCS Result	Action for Samples
%R 40-69%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
%R > 130%	Qualify results that are ≥ MDL as estimated high (J+)
%R < 40%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
%R > 150%	Qualify all results as unusable (R)

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Laboratory Duplicate Sample Analysis

Action:

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to only the field sample used to prepare the duplicate sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

NOTE: Delete "*" from Form IAs.
If one value is > CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL and use this difference to qualify sample results.
If more than one lab duplicate sample was analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for CLP Project Officer (CLP PO) action.
2. If the results from a duplicate analysis for a particular analyte fall outside the control limits for > 5x the CRQL, qualify aqueous sample results that are ≥ CRQL as estimated (J) if the RPD is between 20% - 100% and as unusable (R) if the RPD is > 100%. Qualify soil/sediment sample results that are ≥ CRQL as estimated (J) if the RPD is between 35% - 120% and as unusable (R) if the RPD is > 120%.
3. If the results from a duplicate analysis for a particular analyte fall outside the control limits for ≤ 5x the CRQL, qualify those results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
4. If a field blank or PE sample was used for the duplicate sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
5. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

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Table 8. Duplicate Sample Actions for ICP-MS Analysis

Duplicate Sample Results	Action for Samples
Aqueous: Both original sample and duplicate sample > 5x the CRQL and 20% < RPD < 100%	Qualify those results that are ≥ CRQL as estimated (J)
Aqueous: Both original sample and duplicate sample > 5x the CRQL and RPD ≥ 100%	Qualify those results that are ≥ CRQL as unusable (R)
Soil/Sediment: Both original sample and duplicate sample > 5x the CRQL and 35% < RPD < 120%	Qualify those results that are ≥ CRQL as estimated (J)
Soil/Sediment: Both original sample and duplicate sample > 5x the CRQL and RPD ≥ 120%	Qualify those results that are ≥ CRQL as unusable (R)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL	Qualify those results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ)

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Spike Sample Analysis

Action:

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

NOTE: The final spike concentrations required for the various target analytes are presented in the methods described in the Statement of Work (SOW).

NOTE: When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purpose of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms VA-IN and VB-IN.

NOTES: Not required for Ca, Mg, K, and Na (both matrices); Al and Fe (soil only).
If more than one spiked sample was analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.
Disregard the out of control spike recoveries for analytes whose unspiked concentrations are ≥ 4x the spike added.
Delete "N" from Form IAs.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-digestion spike was not performed, note this for CLP PO action.
4. If the Matrix Spike %R is < 30%, verify that a post-digestion spike was analyzed if required. If the post-digestion spike %R is < 75% or is not performed, qualify sample results that are ≥ MDL as estimated low (J-) and non-detects as unusable (R). If the post-digestion spike %R is ≥ 75%, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
5. If the Matrix Spike %R is 30-74% and the sample results are ≥ MDL, verify that a post-digestion spike was analyzed if required. If the %R for the post-digestion is also < 75% or is not performed, qualify the affected data as estimated low (J-). If the %R for the post-digestion spike is ≥ 75%, qualify the affected data as estimated (J).
6. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).

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7. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.
8. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, verify that a post-digestion spike was analyzed if required. If the %R for the post-digestion spike is also > 125% or is not performed, qualify the affected data as estimated high (J+). If the %R for the post-digestion spike is ≤ 125%, qualify the affected data as estimated (J).
9. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 9. Spike Sample Actions for ICP-MS Analysis

Spike Sample Results	Action for Samples
Matrix Spike %R < 30% Post-digestion spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion Spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ)
Matrix Spike %R > 125% Post-digestion spike %R > 125%	Qualify affected results that are ≥ MDL as estimated high (J+)
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	Qualify affected results that are ≥ MDL as estimated (J)
Matrix Spike %R < 30% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R 30-74% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and non-detects as estimated (UJ)
Matrix Spike %R > 125% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified

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ICP Serial Dilution

Action:

NOTE: For a serial dilution that does not meet the technical criteria, apply the action to only the field sample used to prepare the serial dilution sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

NOTE: Serial dilution analysis is required only when the initial concentration is greater than 50x the MDL.
Delete "E" from Form IAs.

1. If the required %D criteria are not met, qualify affected aqueous results whose raw data are > MDL as estimated (J) if %D is between 10%-100% and qualify as unusable (R) if %D is ≥ 100%. Qualify affected soil/sediment results whose raw data are > MDL as estimated (J) if %D is between 15%-120% and qualify as unusable (R) if %D is ≥ 120%.
2. If evidence of positive or negative interference is found, use professional judgment to qualify the associated sample data. Note the potential effects on the reported data in the Data Review Narrative.
3. It should be noted for Contract Laboratory Program Project Officer (CLP PO) action and in the Data Review Narrative if a field blank or PE sample was used for the serial dilution analysis.

Table 10. Serial Dilution Actions for ICP-MS Analysis

Serial Dilution Result	Action for Samples
Aqueous: Sample concentration > 50x MDL and 10% < %D < 100%	Qualify affected results whose raw data are > MDL as estimated (J)
Aqueous: Sample concentration > 50x MDL and %D ≥ 100%	Qualify affected results whose raw data are > MDL as unusable (R)
Soil/Sediment: Sample concentration > 50x MDL and 15% < %D < 120%	Qualify affected results whose raw data are > MDL as estimated (J)
Soil/Sediment: Sample concentration > 50x MDL and %D ≥ 120%	Qualify affected results whose raw data are > MDL as unusable (R)
Interferences present	Use professional judgment

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ICP-MS Internal Standards

Action:

NOTE: Apply the action to the affected analytes for each sample that does not meet the internal standard criteria.

1. If no internal standards were analyzed with the run, the sample data should be qualified as unusable (R). Record this in the Data Review Narrative and note for CLP Project Officer (CLP PO) action.
2. If less than five of the required internal standards were analyzed with the run, or a target analyte(s) is (are) not associated to an internal standard, the sample data, or analyte data not associated to an internal standard should be qualified as unusable (R). Record this in the Data Review Narrative and note for CLP PO action.
3. If the % Relative Intensities for all internal standards in a sample is within 60-125% of the response in the calibration blank, the sample data should not be qualified.
4. If the %RI for an internal standard in a sample is not within the 60-125% limit, qualify the data for those analytes associated with the internal standard(s) outside the limit as follows:
    a. If the sample was reanalyzed at a two-fold dilution with internal standard %RI within the limits, report the result of the diluted analysis without qualification. If the %RI of the diluted analysis was not within the 60-125% limit, report the results of the original undiluted analyses and qualify the data for all analytes that are ≥ Method Detection Limit (MDL) in the sample associated with the internal standard as estimated (J), and non-detected analytes associated with the internal standard as estimated (UJ).
    b. If the sample was not reanalyzed at a two-fold dilution, the reviewer should use professional judgment to determine the reliability of the data. The reviewer may determine that the results are estimated (J) or unusable (R).

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Table 11. Internal Standard Actions for ICP-MS Analysis

Internal Standard Results	Action for Samples
No internal standards	Qualify all results as unusable (R)
< 5 of the required internal standards	Qualify all results as unusable (R)
Target analyte not associated with internal standard	Qualify all analyte results not associated with an internal standard as unusable (R)
% RI < 60% or > 125%, original sample reanalyzed at 2-fold dilution, and % RI of diluted sample analysis is between 60% and 125%	Do not qualify the data
% RI < 60% or > 125%, original sample reanalyzed at 2-fold dilution, and % RI of diluted sample analysis is outside the 60% to 125% limit	Qualify analytes associated with the failed internal standard that are ≥ MDL as estimated (J) and qualify associated non-detects as estimated (UJ)
Original sample not reanalyzed at 2-fold dilution	Use professional judgment Qualify sample results as estimated (J) or unusable (R)

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Field Duplicates

Action:

NOTES: For field duplicates that do not meet the technical criteria, apply the action to only the field sample and its duplicate. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

Check the Sampling Trip Report for the field duplicate pair.

Substitute MDL for CRQL when MDL > CRQL.

Do not calculate RPD when both values are non-detects.

If one value is > the CRQL and the other value is non-detect, calculate the absolute difference between the value > the CRQL and the MDL, and use this criteria to qualify the results.

1. If a field duplicate pair was collected and analyzed, calculate and report the RPD when the sample and its field duplicate values are both ≥ 5x the CRQL. Calculate and report the absolute difference when at least one value (sample or duplicate) is < 5x the CRQL.
2. When aqueous sample and duplicate values are both ≥ 5x the CRQL, and the RPD is > 20%, qualify the sample and its duplicate as estimated (J).
3. When aqueous sample and/or the duplicate value is < 5x the CRQL, and the absolute difference is > the CRQL, qualify results > the MDL as estimated (J) and non-detects as estimated (UJ).
4. When soil/sediment sample and duplicate values are both ≥ 5x the CRQL, and the RPD is > 50%, qualify the sample and its duplicate as estimated (J).
5. When soil/sediment sample and/or the duplicate value is < 5x the CRQL, and the absolute difference is > 2x the CRQL, qualify results > the MDL as estimated (J) and non-detects as estimated (UJ).

Table 12. Field Duplicate Actions for ICP-MS Analysis

Sample Type	Field Duplicate Result	Action for Samples
Aqueous	Sample and its field duplicate ≥ 5x the CRQL and RPD > 20%	Qualify sample and its duplicate as estimated (J)
	Sample and/or its field duplicate < 5x the CRQL and absolute difference > the CRQL	Qualify results > the MDL as estimated (J) Qualify non-detects as estimated (UJ)
Soil/Sediment Sample	and its field duplicate ≥ 5x the CRQL and RPD > 50%	Qualify sample and its duplicate as estimated (J)
	Sample and/or its field duplicate < 5x the CRQL and absolute difference > 2x the CRQL	Qualify results > the MDL as estimated (J) Qualify non-detects as estimated (UJ)

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Field/Rinsate/Trip Blanks

Action:

NOTE: Designate "Field Blank" as such on Form IA.
Field Blank results previously rejected due to other criteria cannot be used to qualify field samples.
Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.
If the MDL is > the CRQL, substitute CRQL with 2x the MDL.

1. If the appropriate blanks were not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
    a. Any blank reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank reported with a negative result, whose value is < (-CRQL) qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
    b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. If the absolute value of any analyte in a Field/Rinsate/Trip Blank is > the CRQL, then the CRQL shall be raised to the level in the Field/Rinsate/Trip Blank and the associated sample data below this level shall be reported as CRQL-U.
5. Sample results > the Field/Rinsate/Trip Blank value but < 10x the Field/Rinsate/Trip Blank value shall be qualified as estimated (J).
6. Sample results ≥ the MDL but ≤ the CRQL shall be reported at the CRQL value with a "U".

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Table 13. Field/Rinsate/Trip Blank Actions for ICP-MS Analysis

Blank Result	Sample Result	Action for Samples
> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
	> CRQL but < Blank Result	Report at level of Blank Result with a "U"
	> Blank Result but < 10x the Blank Result	Use professional judgment to qualify results as estimated (J)

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Linear Ranges

Action:

1. If any sample result was higher than the high linear range for ICP-MS and the sample was not diluted to obtain the result reported on Form I, qualify the affected results ≥ MDL as estimated (J).

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Percent Solids of Sediments

Action:

1. If the percent solids in sediment for a sample are < 50%, qualify the affected results ≥ MDL as estimated (J) and the non-detects as estimated (UJ).

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Regional Quality Assurance (QA) and Quality Control (QC)

Action:

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Contract Laboratory Program Project Officer (CLP PO) action.

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Overall Assessment

Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.
2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the Sample Delivery Group (SDG) Narrative for Contract Laboratory Program Project Officer (CLP PO) action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.
3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

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Calculations for ICP-MS

Aqueous/Water Sample Concentration by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS):

The concentrations determined in the digestate are to be reported in units of µg/L:

$$C \times \frac{V_f}{V} \times DF$$

Where,

C = Instrument value in µg/L (The average of all replicate integrations).

V_f = Final digestion volume (mL)

V = Initial aliquot amount (mL)

DF = Dilution Factor

Soil/Sediment Sample Concentration by ICP-MS:

The concentrations determined in the digestate are to be reported on the basis of the dry weight of the sample, in units of mg/kg:

$$C \times \frac{V_f}{W \times S} \times \frac{DF}{1000}$$

Where,

C = Instrument value in µg/L (The average of all replicate integrations).

V_f = Final digestion volume (mL)

W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D of ISM01.2 - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, substitute the value of the MDL (µg/L) or CRQL (µg/L) into the "C" term in the equation above. Calculate the adjusted MDL or adjusted CRQL for soil/sediment samples as follows:

$$C \times \frac{W}{S} \times \frac{V_f}{V_i} \times DF$$

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Where,

C = MDL or CRQL (mg/kg)

W_M = Minimum method required aliquot amount (g) (1.00 g or 0.50 g)

W = Initial aliquot amount (g)

V_M = Method required final sample digestion volume (mL) (100 mL)

V_f = Final digestion volume (mL)

S = % Solids/100 (see Exhibit D of ISM01.2 - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

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APPENDIX A: GLOSSARY

Analyte -- The element of interest, ion, or parameter an analysis seeks to determine.
Analytical Services Branch (ASB) -- Directs the Contract Laboratory Program (CLP) from within the Office of Superfund Remediation and Technical Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).
Analytical Sample -- Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB). Note that the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA); Matrix Spike samples; duplicate samples; serial dilution samples, analytical (post-digestion/post-distillation) spike samples; Interference Check Samples (ICSs); Laboratory Control Samples (LCSs); and Preparation Blanks.
Associated Samples -- Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples run under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.
Blank -- A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.
Calibration -- The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentration of acids as used in the sample preparation.
Calibration Blank -- A blank solution containing all of the reagents in the same concentration as those used in the analytical sample preparation. This blank is not subject to the preparation method.
Calibration Curve -- A plot of instrument response versus concentration of standards.
Calibration Standards -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.
Case -- A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).
Continuing Calibration Blank (CCB) -- A reagent water sample that is run 2 hours (ICP-AES, ICP-MS) or every hour (Hg, CN) and designed to detect any carryover contamination.
Contract Compliance Screening (CCS) -- A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under USEPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.
Continuing Calibration Verification (CCV) -- A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be

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represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples. The CCV should have a concentration in the middle of the calibration range and shall be run every 2 hours (ICP-AES, ICP-MS) or every hour (Hg, CN).

Contract Laboratory Program (CLP) -- Supports the USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technical Innovation (OSRTI) of USEPA.

Contract Laboratory Program Project Officer (CLP PO) -- The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.

Contract Required Quantitation Limit (CRQL) -- Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

Duplicate -- A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Field Blank -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

Field Duplicate -- A duplicate sample generated in the field, not in the laboratory.

Holding Time -- The maximum amount of time samples may be held before they are processed.

Contractual -- The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.

Technical -- The maximum amount of time that samples may be held from the collection date until analysis.

Initial Calibration -- Analysis of analytical standards for a series of different specified concentrations to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Initial Calibration Blank (ICB) -- The first blank standard run to confirm the calibration curve.

Initial Calibration Verification (ICV) -- Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when USEPA ICV solutions are not available.

Internal Standard -- A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

Interference Check Sample (ICS) -- Verifies the contract laboratory's ability to overcome interferences typical of those found in samples.

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Laboratory Control Sample (LCS) -- A control sample spiked at known level(s). LCSs are processed using the same sample preparation, reagents, and analytical methods employed for the USEPA samples received.
Matrix -- The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrices are aqueous/water, soil/sediment, wipe, and filter.
Matrix Spike -- Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology (also identified as a pre-distillation/digestion spike).
Method Detection Limit (MDL) -- The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.
Narrative (SDG Narrative) -- Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.
Office of Solid Waste and Emergency Response (OSWER) – The USEPA office that provides policy, guidance, and direction for the USEPA's solid waste and emergency response programs, including Superfund.
Percent Difference (%D) -- As used in this document and the Statement of Work (SOW), is used to compare two values. The difference between the two values divided by one of the values.
Performance Evaluation (PE) Sample -- A sample of known composition provided by USEPA for contractor analysis. Used by USEPA to evaluate Contractor performance.
Post Digestion Spike -- The addition of a known amount of standard after digestion or distillation (also identified as an analytical spike).
Preparation Blank -- An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.
Relative Percent Difference (RPD) -- As used in this document and the Statement of Work (SOW) to compare two values, the RPD is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).
Regional Sample Control Center Coordinator (RSCC) -- In USEPA Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.
Relative Standard Deviation (RSD) -- As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.
Sample -- A single, discrete portion of material to be analyzed, which is contained in single or multiple containers and identified by a unique Sample Number.
Sample Delivery Group (SDG) -- A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
b. Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

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c. Scheduled at the same level of deliverable.

In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG. Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory.

Sample Management Office (SMO) -- A contractor-operated facility operated under the SMO contract, awarded and administered by the USEPA. Provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).

Serial Dilution -- The dilution of a sample by a factor of five. When corrected by the Dilution Factor (DF), the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents [Inductively Coupled Plasma (ICP) only].

Statement of Work (SOW) -- A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

Tune -- Analysis of a solution containing a range of isotope masses to establish Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) mass-scale accuracy, mass resolution, and precision prior to calibration.

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APPENDIX B: INORGANIC DATA EXECUTIVE NARRATIVE TEMPLATE

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 3
DESA/HWSB/HWSS
2890, Woodbridge Avenue, Edison, NJ 08837

EXECUTIVE NARRATIVE

Case No.: **SDG No.:**
Site: **Laboratory:**

QAPP
HWSS #: **Number of Samples:**
Contractor #: **Sampling date:**

SUMMARY:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions.
Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data have been qualified "J" estimated.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings: None

Major Findings: None

Minor Findings: None

COMMENTS:

Validator's Signature: **Date:**
Name:
Affiliation:

Approver's Signature: **Date:**
Name:
Affiliation:

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APPENDIX C: SAMPLE INORGANIC DATA SAMPLE SUMMARY

Case No: 00001	Contract: XYZ1234	SDG No: XY123	Lab Code: ABCD
Sample Number: XY123	Method: ICP_AES	Matrix: FLUFF	MA Number: DEFAULT
Sample Location: SOMEWHERE OUT THERE	pH: 15	Sample Date: 13322059	Sample Time: 24:03:00
% Moisture :		% Solids :	

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable	Validation Level
Aluminum	400	ug/L	1			Yes	S2BVEM
Antimony	40	ug/L	1	U	U	Yes	S2BVEM
Arsenic	40	ug/L	1	U	U	Yes	S2BVEM
Barium	40	ug/L	1	U	U	Yes	S2BVEM
Beryllium	40	ug/L	1	U	U	Yes	S2BVEM
Cadmium	40	ug/L	1	U	U	Yes	S2BVEM
Calcium	400	ug/L	1			Yes	S2BVEM
Chromium	40	ug/L	1	U	U	Yes	S2BVEM
Cobalt	40	ug/L	1	U	U	Yes	S2BVEM
Copper	40	ug/L	1	U	U	Yes	S2BVEM
Iron	40	ug/L	1	J	U	Yes	S2BVEM
Lead	40	ug/L	1	U	U	Yes	S2BVEM
Magnesium	400	ug/L	1			Yes	S2BVEM
Manganese	400	ug/L	1			Yes	S2BVEM
Nickel	40	ug/L	1	U	U	Yes	S2BVEM
Potassium	400	ug/L	1			Yes	S2BVEM
Selenium	40	ug/L	1	U	U	Yes	S2BVEM
Silver	40	ug/L	1	U	U	Yes	S2BVEM
Sodium	400	ug/L	1			Yes	S2BVEM
Thallium	40	ug/L	1	U	U	Yes	S2BVEM
Vanadium	40	ug/L	1	U	U	Yes	S2BVEM
Zinc	40	ug/L	1	U	U	Yes	S2BVEM

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APPENDIX D: ELECTRONIC DATA DELIVERABLE TEMPLATE

DATA_PROVIDER	LAB_MATRIX_CODE	RESULT_UNIT
SYS_SAMPLE_CODE	ANAL_LOCATION	DETECTION_LIMIT_UNIT
SAMPLE_NAME	BASIS	TIC_RETENTION_TIME
SAMPLE_MATRIX_CODE	CONTAINER_ID	RESULT_COMMENT
SAMPLE_TYPE_CODE	DILUTION_FACTOR	QC_ORIGINAL_CONC
SAMPLE_SOURCE	PREP_METHOD	QC_SPIKE_ADDED
PARENT_SAMPLE_CODE	PREP_DATE	QC_SPIKE_MEASURED
SAMPLE_DEL_GROUP	LEACHATE_METHOD	QC_SPIKE_RECOVERY
SAMPLE_DATE	LEACHATE_DATE	QC_DUP_ORIGINAL_CONC
SYS_LOC_CODE	LAB_NAME_CODE	QC_DUP_SPIKE_ADDED
START_DEPTH	QC_LEVEL	QC_DUP_SPIKE_MEASURED
END_DEPTH	LAB_SAMPLE_ID	QC_DUP_SPIKE_RECOVERY
DEPTH_UNIT	PERCENT_MOISTURE	QC_RPD
CHAIN_OF_CUSTODY	SUBSAMPLE_AMOUNT	QC_SPIKE_LCL
SENT_TO_LAB_DATE	SUBSAMPLE_AMOUNT_UNIT	QC_SPIKE_UCL
SAMPLE_RECEIPT_DATE	ANALYST_NAME	QC_RPD_CL
SAMPLER	INSTRUMENT_ID	QC_SPIKE_STATUS
SAMPLING_COMPANY_CODE	COMMENT	QC_DUP_SPIKE_STATUS
SAMPLING_REASON	PRESERVATIVE	QC_RPD_STATUS
SAMPLING_TECHNIQUE	FINAL_VOLUME	BREAK_2
TASK_CODE	FINAL_VOLUME_UNIT	SYS_SAMPLE_CODE
COLLECTION_QUARTER	CAS_RN	LAB_ANL_METHOD_NAME
COMPOSITE_YN	CHEMICAL_NAME	ANALYSIS_DATE
COMPOSITE_DESC	RESULT_VALUE	TOTAL_OR_DISSOLVED
SAMPLE_CLASS	RESULT_ERROR_DELTA	COLUMN_NUMBER
CUSTOM_FIELD_1	RESULT_TYPE_CODE	TEST_TYPE
CUSTOM_FIELD_2	REPORTABLE_RESULT	TEST_BATCH_TYPE
CUSTOM_FIELD_3	DETECT_FLAG	TEST_BATCH_ID
COMMENT	LAB_QUALIFIERS	CASE
BREAK_1	VALIDATOR_QUALIFIERS	CONTRACT_NUM
SYS_SAMPLE_CODE	INTERPRETED_QUALIFIERS	SCRIBE_SAMPLE_ID
LAB_ANL_METHOD_NAME	ORGANIC_YN	SAMPLE_TIME
ANALYSIS_DATE	METHOD_DETECTION_LIMIT	FRACTION
TOTAL_OR_DISSOLVED	REPORTING_DETECTION_LIMIT	PH
COLUMN_NUMBER	QUANTITATION_LIMIT	DATA_VAL_LABEL
TEST_TYPE		

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VALIDATION SOP

Methyl Mercury by CVAFS
USEPA 1630
Rev. 0 07/10

METHYL MERCURY VIA METHOD 1630 VALIDATION SOP REVISION 0

SITE:

DATE/INITIALS:

SDG:

LABORATORY:

Data Completeness and Deliverables	**Y**	**N**	**N/A**
Have any missing deliverables been received and added to the data package?	_____	_____	_____

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If the lab cannot provide them, note the effect on the data review process in the non-compliance section of the data assessment narrative.

Custody Documents and Narratives

Are chains of custody present and complete for all samples?	_____	_____	_____

ACTION: Contact lab for replacement of missing documents.

Do chains of custody or lab narratives indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?	_____	_____	_____

ACTION: If samples were not iced/or melted ice is present in the cooler and cooler temperature was outside the range of 4°C (±2) upon receipt, note in data assessment narrative. If cooler temperature is found to be in excess of 10°C, flag all associated CH_3Hg results as estimated "J."

Holding Times

Have any methyl mercury technical holding times, determined from date of collection to date of preparation and analyses been exceeded (48 hours to preservation, 28 days until analysis)?	_____	_____	_____

NOTE: Properly preserved samples may be stable for 6 months, if kept cool and in the dark. However, data qualification will be performed based on the technical holding time for methyl mercury (preservation within 48 hours, analysis within 28 days).

Table of Holding Time Violations **Y** **N** **N/A**

Sample	Sample Matrix	Date Sampled	Date lab Received	Date Analyzed	Date Digested	Date/Time Preserved
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____	_____

ACTION: If holding times are exceeded, flag all data as estimated "J" for detects and "UJ" for non-detects. If holding times were grossly exceeded (i.e., more than 2x the holding time), flag all positive data as estimated and reject all non-detects as unusable "R".

Sample Preservation

Were the pH values present? _____ _____ _____

Were samples properly preserved per Section 8.5, Method 1630? _____ _____ _____

Samples maybe shipped to the laboratory unpreserved if (1) collected in fluoropolymer bottles (2) filled to the top with no head space (3) Capped tightly and (4) maintained at 0-4°C from the time of collection until preservation. The samples must be acid-preserved within 48 hours of sampling.

NOTE: The pH of all aqueous samples must be tested immediately before aliquotting for processing or direct analysis pH should be < 2.

Preservation samples may be done by adding 5 mL/L of concentrated HCl (to allow both total and methyl Hg determination).

Laboratory Sample Preparation Log

Are all sample preparation bench sheets and logs present? _____ _____ _____

Calibration

Are raw data and summary sheets present for the initial calibration? _____ _____ _____

NOTE: The initial calibration must contain at least 5 points, lowest being at the minimum level (ML) (.06 ng/l) and the analysis of one ethylation blank.

Are the % RSD values for the initial calibration ≤ 15%? _____ _____ _____

	Y	N	N/A
ACTION: Associated sample data for those analytes with % RSD > 15 will be qualified as estimated "J". However, if severe discrepancies exist (i.e., %RSD > 50%) validator may elect to reject associated sample results.			
Do the ethylation blanks contain positive results > 2 pg?	_____	_____	_____
The net concentration recovery (minus ethylation blank) for the lowest standard must be in the range of 65-135% of the expected value.			
ACTION: If the recovery is < 65%, flag associated data as estimated "J" for detects and "R" for non-detects. If the recovery is > 135%, flag associated data "J" for detects.			
Are mis-calculations or transcription errors found?	_____	_____	_____

NOTE: If yes, contact the laboratory.

Quality Control Sample (QCS)

Was a QCS analyzed as an independent check of instrument calibration in the middle of the analytical batch?	_____	_____	_____

NOTE: The laboratory must obtain QCS source from a different source used to produce the standards, CH_3Hg.

ACTION: If < 50% recovery flag all associated data as "J". If > 150% recovery flag all associated positive data as "J".

Initial Precision and Recovery (IPR)

Was an initial precision and accuracy demonstration performed for the appropriate matrix per Section 9.2 of Method 1630 (4 replicates of the IPR solution is used to compute an average percent recovery X and the standard deviations of the percent recoveries for CH_3Hg)?	_____	_____	_____
Were the results of the IPR evaluation acceptable when compared to the acceptance limit listed in Table 2 (69-131%)?	_____	_____	_____

ACTION: If IPR data was not provided by the laboratory, contact the lab to obtain the results of the IPR study.

If the result of the laboratories IPR study does not meet the acceptance criteria for performance test listed in Table 2 of Method 1630, contact the laboratory to initiate remediation of technical difficulties. Further sample analyses should not be performed until the laboratory Satisfactorily demonstrates the IPR performance test.

Ongoing Precision and Recovery (OPR)	**Y**	**N**	**N/A**
Was an OPR standard and an ethylation blank prepared and analyzed prior to each sample batch?	_____	_____	_____
Was an OPR analyzed at the end of an analytical run or at the end of each 12- hour shift?	_____	_____	_____
Circle on each OPR summary report percent recoveries outside control limits (67-133%).			
Is any OPR recovery:			
Less than 10%?	_____	_____	_____
Between 11% and 66%?	_____	_____	_____
Between 134% and 150%?	_____	_____	_____
Greater than 150%?	_____	_____	_____

ACTION: Less than 10%, reject (red-line) all data; between 11% and 66%, flag all associated data as estimated "J"; between 134% and 150%, flag all positive (not flagged with a “U” results as estimated; greater than 150%, reject all positive results.

Blanks

Method Blank

Has a method blank analysis been reported for each analytical batch not to exceed 20 samples?	_____	_____	_____

NOTE: Three method blanks should accompany each analytical batch. The mean blank value should be used to evaluate associated sample results.

Is the method blank summary present?	_____	_____	_____
Upon evaluation of associated method blank results, do all method blanks meet the PQL criteria listed in Table1?	_____	_____	_____

ACTION: If the samples associated with the non-compliant method blank contain positive results for methyl mercury which are < 5x that of the mean method blank value, these sample results should be qualified as “U” non-detected. Non-detect results are not affected nor are sample results that are > 5x that of the contaminated method blank mean.

Trip Blank	**Y**	**N**	**N/A**
Has a trip blank analysis been reported for each sample delivery shipment?	_____	_____	_____
Upon examination of the trip blank, does it contain any positive results ≥ PQL?	_____	_____	_____

ACTION: If the samples associated with the non-compliant trip blank contain positive results for methyl mercury which are < 5x that of the mean trip blank value, these sample results should be qualified as "U" non-detected. Non-detect results are not affected nor are sample results that are > 5x that of the contaminated trip blank.

Field Blank

Has a field blank analysis been reported for each sampling event not to exceed 20 samples?	_____	_____	_____
Has the field blank been analyzed in the proper sequence?	_____	_____	_____

NOTE: The field blank should be analyzed immediately before analyzing samples in the batch.

Upon examination of the field blank does it contain positive results ≥ PQL?	_____	_____	_____

ACTION: If the samples associated with the non-complaint field blank contain positive results for methyl mercury which are < 5x that of the field blank, these sample results should be qualified as "U" non-detected. Non-detect results are not affected nor are sample results that are > 5x that of the contaminated field blank.

Pre-Project Dedicated Equipment Rinsate Blank

Has one dedicated lot check on sample collection tubing and one rinse blank of the filter lot used only in the collection of described metal samples analysis been reported ?	_____	_____	_____
Upon examination of the rinsate blank, does it contain positive results ≥ PQL?	_____	_____	_____

ACTION: If the samples associated with the non-complaint rinsate blank contain positive results for methyl mercury which are < 5x that of the rinsate blank, these sample results should be qualified as "U" non-detected. Non-detect results are not affected nor are sample results that are > 5x that of the contaminated rinsate blank.

Rinsate Blank	**Y**	**N**	**N/A**
Has a rinsate blank analysis been reported for each set of decontaminated field equipment?	_____	_____	_____
Upon examination of the rinsate blank, does it contain positive its ≥ PQL?	_____	_____	_____

ACTION: If the samples associated with the non-complaint rinsate blank contain positive results for methyl mercury which are < 5x that of the rinsate blank, these sample results should be qualified as "U" non-detected. Non-detect results are not affected nor are sample results that are > 5x that of the contaminated rinsate blank.

Compound Quantitation and Reported Detection Limits

Verify at least one detected methyl mercury result in each sample delivery group. Recalculate from the raw data to check for calculation and transcription errors.

Were miscalculation/transcription errors found?	_____	_____	_____

ACTION: If errors are found, contact the lab for explanation/re-submittals.

Are all positive methyl mercury results above the PQL reported with:

three significant figures?	_____	_____	_____
correct units (ng/l)?	_____	_____	_____

Field Duplicates

NOTE: To access the precision and accuracy of the sampling, sample transportation and storage techniques.

The following samples comprise the field duplicate pair: _____

Were field duplicates submitted for methyl mercury analysis at frequency of one per sample delivery group, not to exceed 20?	_____	_____	_____

ACTION: Where both the sample and duplicate values are > 5x the PQL, acceptable sampling and analytical precision is indicated by an RPD for the two field duplicate results of ≤ 20%. Where one or both analytes of the field duplicate pair are < 5x the PQL, satisfactory precision is indicated if the field duplicate results agree within 2x the PQL. If the above criteria are not met for an analyte, qualify all associated sample data for that analyte as estimated "J".

Laboratory Duplicates **Y** **N** **N/A**

Was a laboratory duplicate submitted for methyl mercury analysis? _____ _____ _____

The following sample was duplicated _____

ACTION: Where both the sample and duplicate values are > 5x the PQL, acceptable sampling and analytical precision is indicated by an RPD for the two results of ≤ 20%. Where one or both analytes of the laboratory duplicate pair is ≤ 5 times the PQL, satisfactory precision is indicated if the laboratory duplicate results agree within 2x the PQL. If the above criteria are not met for an analyte, qualify all associated sample data for that analyte as estimated "J".

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The following sample was selected for MS/MSD analysis: _____

Is a matrix spike/matrix spike duplicate summary present? _____ _____ _____

Were matrix spikes analyzed at the required frequency for each of the analytical sample groups (i.e., 10% minimum per analytical batch)? _____ _____ _____

ACTION: If any matrix spike data are missing, call the lab for explanation/re-submittal. If information is not available document the effect in narrative notes.

MS/MSD limits are as follows:

% Recovery	=	65-135%
RPD	=	±35%

Have any matrix spike recoveries failed to meet the criteria listed above? _____ _____ _____

Are any spike recoveries:

Less than 10%? _____ _____ _____

Between 10-64%? _____ _____ _____

Between 136-200%? _____ _____ _____

Greater than 200%? _____ _____ _____

ACTION: If < 10%, reject all associated aqueous data; if between 10-64%, flag all associated aqueous data as estimated "JL" or "UJL", potential low bias; if between 136-200%, flag as estimated "J" all associated aqueous data not flagged with a “U”; if > 200%, reject (red-line) all associated aqueous data not flagged with a “U.”

Have any MS/MSD relative percent difference values failed to meet the acceptance criteria listed above? _____ _____ _____

ACTION: If any relative percent difference value fails to meet the method acceptance criteria, qualify all associated sample results as estimated “J” for detects or “UJ” for non-detects.

Data Validation Qualifiers

Qualifier	Description
J	Estimated value (bias undetermined) – The analyte was positively identified; but the associated numerical value is the approximate concentration of the analyte in the sample.
JH	Estimated value (potential high bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential high bias, of the analyte in the sample.
JL	Estimated value (potential low bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential low bias, of the analyte in the sample.
UJ	Estimated non-detect - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
UJL	Estimated non-detect (potential low bias) – The analyte was not detected and the report sample quantitation limit is biased low.
UJH	Estimated non-detect (potential high bias) – The analyte was not detected and the reported sample quantitation limit is biased high.
M	The analytical result reported was obtained from a sediment sample found to contain between 50 and 90 percent moisture and had no other data qualifiers added during the data validating process.
NJ	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
NJH	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration with a potential high bias, of the analyte concentration.
EMPC	Estimated Maximum Possible Concentration (EMPC).
R	The sample results are rejected. Due to a significant QA/QC problem, the analysis is invalid and provides no information as to whether the analyte is present or not.

VALIDATION SOP

Butyltins

SOP Organotins Prep. 8/05

STANDARD OPERATING PROCEDURE
DATA VALIDATION OF ORGANOTIN ANALYSIS

A. INTRODUCTION

This Standard Operating Procedure (SOP) provides guidance for the validation of organotin analytical data generated from the analysis of water, soils, sediments, or tissue samples. The validation procedure was based upon the EPA Region II data validation SOP for Pesticides/PCBs (EPA 1992)[1]. A copy of the analytical protocol is provided as an appendix to the QAPP.

B. DATA VALIDATION PROCEDURES

1. Holding Times

Criteria: Holding times are established for both extraction and sample analysis. For aqueous, soil and tissue matrices, the extraction must begin within 7 days from sample collection, with analysis within 40 days of extraction.

Actions: The actions are summarized in Table 1. Actions may be taken for either time from sampling to extraction or time from extraction to analysis, but not both. If neither criteria are met, the exceedance which occurs first (i.e., time to extraction, or the criteria which results in additional qualifications or the "R" qualification takes precedence.

Table 1. Summary of Actions for Holding Times

Time from Sampling to Extraction			
Matrix	0-7 days	7-14 days	> 14 days
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects
Time from Extraction to Analysis			
Matrix	0-40 days	40-80 days	> 80 days
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects

2. Initial Calibration

The gas chromotograph undergoes a 5 point initial calibration with all of the targeted analytes. The laboratory may use either a linear regression, or calculate response factors, to ensure proper linear response of the instrument.

Criteria: If linear regression is performed, the laboratory must demonstrate that the regression coefficient ("r") is greater than or equal to 0.995.

If calibration factors are used, the laboratory must demonstrate that the relative standard deviation of the calibration factors is less than 30%.

Actions: The actions are summarized in Table 2.

Table 2. Summary of Actions for Initial Calibration

Initial Calibration using Regression			
Matrix	R ≥ 0.995	0.95 < r < 0.995	R < 0.95
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects
Initial Calibration using Calibration Factors			
Matrix	RSD < 30%	30% < RSD < 80%	RSD > 80%
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects

3. Continuing Calibration

The gas chromatograph undergoes a 1 point daily calibration with all of the targeted analytes to ensure consistency with the initial calibration.

Criteria: If linear regression was used for the initial calibration, then the continuing calibration result calculated from the linear regression must agree within 25% of the nominal concentration.

If calibration factors were used for the initial calibration, the laboratory must demonstrate that the percent difference between the mean calibration factors from the continuing calibration and the average response factor from the initial calibration is less than 30%.

Actions: The actions are summarized in Table 3.

Table 3. Summary of Actions for Continuing Calibration

Calibration using Regression			
Matrix	75 – 125% of nominal value	20 – 74% or 126 – 180% of nominal value	< 20% or > 180% of nominal value
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects
Calibration using Calibration Factors			
Matrix	%D < 30%	30% < %D < 80%	%D > 80%
Sediment, water and tissues	Accept	"J" positives "UJ" non-detects	"J" positives "R" non-detects

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4. Surrogate Recoveries

For soil, water and tissue samples, surrogate is added to the sample extract prior to derivitazation.

Criteria: The laboratory must demonstrate that the surrogate recovery falls between 30 and 160%. If the initial analysis fails this criterion, then the samples must be reprocessed once.

Actions: The actions are summarized in Table 4. Chromatograms should be reviewed prior to taking any action to ensure that there were not interferences or coeluting peaks which resulted in the surrogate recoveries falling outside of the control limit. If it is the Reviewer's professional opinion that the non-compliant surrogate recoveries were due to interferences or co-eluting peaks, no action may be required.

No action is required if the surrogates were diluted out.

Table 4. Summary of Actions for Surrogate Recoveries

	%R < 10%	10 < %R < 30%	30 < %R < 160%	%R > 160%
Positives	"R"	"J"	Accept	"J"
Non-Detects	"R"	"UJ"	Accept	Accept
Note: No action is taken if the surrogates are diluted out.				

5. Method and Field QC Blanks

Method blanks are prepared by the laboratories to identify the potential for laboratory contamination. Field QC blanks are provided by the field team to identify potential contamination by the targeted analytes which may be introduced during field activities.

Criteria: There should be no detected targeted analytes in the method or field QC blanks.

Actions: If the targeted analytes are reported in the method or field QC blanks, and are present at a concentration less than 5 times the blank concentrations in the samples (adjusting for matrix variation and sample size), the reported sample results are potential false-positives and should be qualified with a "U". No action is taken if the sample concentration is greater than 5 times the method or field QC blank results. Likewise, when associated samples are "not detected" for the analyte, no qualification of data is necessary.

6. Data Calculations and Reported Concentrations

The reported concentrations for the targeted analytes in at least one sample in the Sample Delivery Group should be recalculated and verified. If a discrepancy is noted, the laboratory should be contacted to provide a sample calculation. Corrective actions may be required, as appropriate.

If the laboratory employs a confirmation column to verify sample identifications, it is recommended that the Reviewer verify that the concentrations on both columns are comparable. If significant differences are noted which are attributable to interferences or coeluting peaks, the Reviewer should consider reporting the results from the confirmation column.

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7. Sample Moisture Contents

If the sediment samples exhibit moisture contents in excess of 50%, the actions in Table 6 should be taken.

Table 6. Actions for Excessive Moisture Contents in Sediments

	%Moist < 50%	50 < %Moist < 90%	%Moist > 90%
Positives	Accept	"J"	"G"
Non-Detects	Accept	"UJ"	"G"

8. Project Specific Requirements

If the direction of the bias can be determined from the analytical results, then the data reviewer can apply the following data qualifiers:

- Potential high bias: JH or UJ
- Potential low bias: JL or UJL

If the direction of the bias can not be determined, or the assessment of bias is contradicted by the quality control results (e.g., holding time exceedance with high surrogate recoveries), then the overall bias is undetermined, and the "J" or "UJ" qualifiers are appropriate.

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VALIDATION SOP

Chlorinated Herbicides
USEPA Region 2 SOP HW-17
Rev. 3

Validating Chlorinated Herbicides GC, SW-846, Method 8151A

Prepared by: George Karras Date: 8/7/08
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Peer Reviewed by: Russell Arnone Date: 08/11/08
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Linda Mauel Chief HWSS

Approved by: Robert Runyon Date: 8/12/08
Robert Runyon, Chief HWSB

Annual Review
Reviewed by: Russell Arnone Date: 08/11/09

Reviewed by: _____ Date: _____

		YES	NO	NA
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are Traffic Report Forms present for all samples?	[ ]	__	__
	ACTION: If no, contact lab for replacement of missing or illegible copies.			
1.2	Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data?	__	[ ]	__
	ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be qualified as estimated (J). If a soil sample, other than TCLP, contains more than 90% water, all data should be qualified as unusable (R).			
	ACTION: If samples were not iced (4°C) upon receipt at the laboratory, flag all positive results J" and all non-detects "UJ".			
2.0	Holding Times			
2.1	Has the technical holding times, determined from date of sample receipt to date of extraction, been exceeded?	__	[ ]	__
	Note: Samples may be analyzed for herbicide ester and acid. Check Laboratory SDG Narrative.			
	Note: Aqueous samples must be extracted within 7 days. Extracts must be analyzed within 40 days following extraction. Soil/Concentrated Waste samples must be extracted within 14 days and extracts analyzed within 40 days following extraction.			
	ACTION: If technical holding times are exceeded, flag all positive results and non-detects(U)as estimated (J) and document in the narrative that holding times were exceeded. Samples extracted more than 28 days from sample receipt, either on the first analysis or upon re-analysis, flag all positive results as estimate ("J") and non-detects as unusable (R).			

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	YES	NO	NA
3.0 Surrogate Recovery (Form II/Equivalent)			
3.1 Are the Herbicide Surrogate Recovery Summaries (Form II/Equivalent) present for each of the following matrices?			
a. Aqueous	[ ]	__	__
b. Soil/Concentrated Waste	[ ]	__	__
3.2 Are all the samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices?			
a. Aqueous	[ ]	__	__
b. Soil/Concentrated Waste	[ ]	__	__

ACTION: Contact lab for explanation/resubmittals. If missing deliverables are unavailable, document effect in data assessments.

3.3 Were outliers marked correctly with an asterisk?	[ ]	__	__

ACTION: Circle all outliers with red pencil.

Note: Recommend surrogate is 2,4-Dichlorophenylacetic acid (DCAA)

3.4 Did the laboratory provide their developed in-house QC limits/recoveries?	[ ]	__	__

ACTION: If no, use 70 -130% recovery to qualify data

Surrogate Action for Herbicide Analyses

Criteria	Action Detected Target Compounds	Action Non-Detected Target Compounds
% R > 150%	J	No qualification required
70% ≤ % R ≤ 150%	No qualification required	No qualification required
10% ≤ % R ≤ 70%	J	UJ
% R < 10%(Dilution not a factor)	J	R
% R < 10%(Dilution is a factor)	No qualification required	No qualification required

Note: In-house QC limits must be examined for reasonableness, e.g. 10-170% may be appropriate for soil samples but not aqueous samples.

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YES NO NA

Note: Matrix effect is suspected if the LCS data are within QC limits but surrogate data exceeds QC limits.

3.5 Were surrogate retention times (RT) within the window established during the initial 5-point calibration analysis? [ ] __ __

ACTION: If the RT limits are not met, the analysis may be qualified unusable (R) for that sample on the basis of professional judgment.

3.6 Are there any transcription/calculation errors between the raw data and Form II/Equivalent? __ [ ] __

ACTION: If large errors exist, call lab for explanation Or resubmittal. Make any necessary corrections and document effect in the data assessment.

4.0 Laboratory Control Sample

4.1 Are raw data and percent recoveries present for all Laboratory Control samples as required by Method 8000B (section 8.5) [ ] __ __

Verify that QC check samples were extracted and analyzed by the same procedures used for the actual samples.

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation /resubmittals. Make note in the data assessment.

NOTE: For aqueous samples, an additional QC check sample must be prepared and analyzed when any analyte in a matrix spike fails the required acceptance criteria (see section 5.3 below). The additional QC check sample must contain each analyte that failed in the MS analysis.

Note: When the results for matrix spike analysis indicates a problem due to sample matrix effects, the LCS results are used to verify the laboratory can perform the analysis in a clean sample.

4.2 Were Laboratory Control Samples analyzed at the required concentration for all analytes of interest as specified in Method 8000B (sec.8.5)? [ ] __ __

ACTION: If Laboratory Control Samples were not analyzed at the required concentration or the required frequency, make note in the data assessment and use professional judgment to determined the affect on the data.

4.3 Were the LCS recoveries within the laboratory's in-house per cent recoveries (if not available, use 70 - 130%) [ ] __ __

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	YES	NO	NA
4.4 If no, were Laboratory Control Samples re-analyzed?	[ ]	__	__

Note: Corrective action must be taken when one or more of the analytes of interest fail the QC acceptance criteria (Method 8000B, section 8.7.4)

ACTION: If QC check samples were not re-analyzed, or a general system problem is indicated by repeated failure to meet the QC acceptance criteria specified in the method, make note in the data assessment and use professional judgment to determine the effect on the data.

5.0 Matrix Spikes (Form III/Equivalent)

5.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III/Equivalent) present?	[ ]	__	__
5.2 Were matrix spikes analyzed at the required frequency for each of the following matrices?			
a. Aqueous	[ ]	__	__
b. Soil/Concentrated Waste	[ ]	__	__
5.3 Did the laboratory provide their developed in-house QC limits/recoveries?	[ ]	__	__

Note: At a minimum, analysis of at least one matrix spike duplicate unspiked sample or one matrix spike/matrix spike duplicate pair with each batch of up to 20 samples.

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

ACTION: If no, use 70 -130% recovery to qualify data

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgment the data reviewer may use the matrix spike results in conjunction with other QC criteria (e.g. LCS) to determine the need for qualification of the data.

6.0 Blanks (Form IV/Equivalent)

6.1 Is the Method Blank Summary (Form IV) present?	[ ]	__	__

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	YES	NO	NA
6.2 Frequency of Analysis: has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent?	[ ]	__	__

ACTION: If any blank data are missing, take the action specified above in 3.2. If blank data is not available, reject "R" all associated positive data. However, using professional judgment, the data reviewer may substitute field blank data for missing method blank data.

6.3 Has a Herbicide instrument blank been analyzed at the beginning of every analytical sequence of 10 samples?	[ ]	__	__

ACTION: If any blank data are missing, call lab for explanation/resubmittals. If missing deliverables are unavailable, document the effect in data assessments.

6.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for Herbicides?	[ ]	__	__

ACTION: Use professional judgment to determine the effect on the data.

7.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and drilling water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC

7.1 Do any method/instrument/reagent/cleanup blanks have positive results for Herbicides? When applied as described in the table below, the contaminant concentration in the method blank is multiplied by the sample dilution factor and corrected for % moisture when necessary.	__	[ ]	__
7.2 Do any field/rinse blanks have positive results?	__	[ ]	__

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YES NO NA

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. Attach a separate sheet.

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, calibration, or any QC problems.

ACTION: Follow the directions in the table below to qualify analytes due to contamination. Use the largest value in all the associated blanks.

Action Table for Blank Contamination

Blank Type	Blank Result	Sample Result	Action
Method, Field	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank Contamination	Report CRQL value with a U
		≥ CRQL and ≥ blank contamination	No qualification required
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	Gross contamination	Detects	Qualify results as unusable R

NOTE: If gross blank contamination exists (e.g., saturated peaks, "hump-o-grams," "junk" peaks), all affected positive compounds in the associated samples should be qualified as unusable "R", due to interference. Non-detected pesticide target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

7.3 Are there field/rinse/equipment blanks associated with every sample? [ ] __ __

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

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	YES	NO	NA
8.0 Calibration and GC Performance			
8.1 Are the Gas Chromatograms and Data Systems printouts for both columns present for all samples, blanks, QC Check references, and matrix spikes?	[ ]	__	__
ACTION: If no, take action specified in 3.2 above.			
8.2 Are Form VI/Equivalent present and complete for each column and each analytical sequence?	[ ]	__	__
ACTION: If no, take action specified in 3.2 above.			
8.3 Are there any transcription/calculation errors between raw data and Forms VI?	__	[ ]	__
ACTION: If large errors exist, call lab for explanation Or resubmittal, make necessary corrections and document effect in data assessments.			
8.4 Were the retention time windows calculated using the average absolute retention time (at least three measurements ± three times the standard deviation of the absolute retention time, for each standard? (Refer to Method 8000A, section 7.5).	[ ]	__	__
8.5. Was a LCS check standard analyzed prior to environmental samples?	[ ]	__	__
8.5.1 If yes, was the surrogate recovery >50%?	[ ]	__	__
8.5.2 Was the LCS check standard re-extracted/re-analyzed, if surrogate recovery was <50%, or any one analyte was < 40%, or two analytes < 70% ?	[ ]	__	__
Action: If No to any of the above, then qualify positive hits as estimated "J" and non-detects as rejected "R" in the original analysis of all samples in the associated analytical sequence.			
8.6 Do all standard retention times, including each Hericide in each level of Initial Calibration fall within the windows established during the initial calibration analytical sequence? (For Initial Calibration Standards,			

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	YES	NO	NA
Form VI/Equivalent - Herbicides - 1).	[ ]	__	__

ACTION: If no, all samples in the entire analytical sequence are potentially affected. Check to see if the chromatograms contain peaks within an expaned window surrounding the expected retention times. If no peaks are found and the surrogate is visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

8.7 Are the linearity criteria for the Initial Calibration analyses within limits for both columns? (% RSD < 20%)for all analytes).	[ ]	__	__

ACTION: If no, qualify all associated positive results generated during the entire analytical sequence "J" and all non-detects "UJ". When RSD > 90%, flag all non-detect results for that analyte R unusable).

8.8 Are there any transcription/calculation errors between raw data and Form VII - Herbicides-2?	__	[ ]	__

ACTION: If large errors exists, call lab for explanation Or resubmittal, make any necessary corrections and document effect in data assessments.

8.9 Is the resolution between any two adjacent peaks in the QC Reference Check Mixture > 60.0% for both columns? (Form VI-Herbicides-4)	[ ]	__	__

Note: Formula to compute % Resolution - $\% R = \frac{V}{H} \times 100$

V = Depth of the valley between the peaks of interest and Next eluting peak. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.

H = Height of the shorter of the adjacent peaks.

ACTION: If no, positive results for compounds that were not adequately resolved should be qualified

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YES NO NA

"J". Use professional judgment to determine if non-detects which elute in areas affected by co-eluting peaks should be qualified "JN" as presumptive evidence of presence or unusable (R).

8.10 Is Form VII -Continuing Calibration present and complete for each analytical sequence for both columns? [ ] __ __

ACTION: If no, take action as specified in 3.2 above.

8.11 Have all samples been injected within a 24 hr. period beginning with the injection of the first standard? [ ] __ __

ACTION: If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly.

8.12 o all analyte retention times for the Mid-concentration Check standard (Form VII Herb-2) fall within the windows established by the initial calibration sequence? [ ] __ __

ACTION: If no, beginning with the samples which follows the last in-control standard, check to see if the chromatograms contain peaks within an expanded peaks within an expanded window surrounding retention times. If no peaks are found and surrogates are visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

8.13 Are %Diff values for all verification calibration standard compounds < 25.0% [ ] __ __

ACTION: The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyte which failed the criteria.

If %Diff > 25, qualify detected compounds "J" and

non-detects compounds "UJ"

9.0 Analytical Sequence Check (Form VIII)

FOIA_07123_0003552_0608

	YES	NO	NA
9.1 Is Form VIII present and complete for each column and each period of analyses?	[ ]	__	__

ACTION: If no, take action specified in 3.2 above.

9.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses?	[ ]	__	__

ACTION: If no, use professional judgment to determine the severity of the effect on the data and qualify it accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the or calibration was also out of limits.

10.0 Herbicides Identification

10.1 Is Form X complete for every sample in which an Herbicide was detected?	[ ]	__	__

ACTION: If no, take action specified in 3.2 above.

10.2 Are there any transcription/calculation errors between raw data and Form X.	__	[ ]	__

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note errors in data assessment.

10.3 Are retention times (RT) of sample compounds within the established RT windows for both columns?	[ ]	__	__
Was GC/MS confirmation provided instead of confirmation by a second dissimilar column?	[ ]	__	__

Action: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis or by GC/MS. Also qualify as unusable (R) all positive results not meeting RT window unless associated standard compounds show a similar RT shift. The reviewer should use professional judgment to assign an appropriate quantitation limit.

10.4 Is the percent difference (% D) calculated for the positive sample results on the two GC columns < 25.0%?	[ ]	__	__

FOIA_07123_0003552_0609

YES NO NA

ACTION: If the reviewer finds neither column shows interference for the positive hits, Qualify as follows:

% Difference	Qualifier
< 25%	No qualification required
25-50 %	J
50-90 %	JN
> 90 %	R

NOTE: The lower of the two values is reported on Form I. If using professional judgment, the reviewer determines that the higher results was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

10.5 Check chromatograms for false negatives. Were there any false negatives? __ [ ] __

ACTION: Use professional judgment to decide if the compound should be reported.

11.0 Compound Quantitation and Reported Detection Limits

11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found? __ [ ] __

NOTE: The reviewer should use professional judgment to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, the lower of the two values should be reported and qualified as presumptively present at an approximated quantity (NJ). This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate the presence of interferences during the evaluation of the second column confirmation.

11.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture? [ ] __ __

ACTION: If errors are large, call lab for explanation resubmittal, make any necessary corrections and document the effect in data assessments.

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YES NO NA

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted

sample. Specify which Form I is to be used, the draw a red "X" across the entire page of all Form'Is that should not be used, including any in the summary package.

ACTION: Quantitation limits affected by large, off-scale peaks should be qualified as unusable (R). If the interference is on-scale, the reviewer can provide an approximated quantitation limit (UJ) for each affected compound.

11.3 Have all data (Forms and associated chromatograms and quantitation reports) been submitted for original, diluted or re-extraction/re-analysis samples? [ ] __ __

12.0 Chromatogram Quality

12.1 Were baselines stable? [ ] __ __

12.2 Were any electropositive displacement (negative peaks) or unusual peaks seen? __ [ ] __

ACTION: Address comments under System Performance of data assessment. Explain use of professional judgment used to qualify data.

13.0 Field Duplicates

13.1 Were any field duplicates submitted for the Herbicides analysis? [ ] __ __

Note: Check whether SAS Client Request required field duplicates.

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results

FOIA_07123_0003552_0611

YES NO NA

must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

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VALIDATION SOP

Semivolatile Organics

USEPA Region 2 SOP HW-22 Rev.2, 6/01

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to "SW846-Method 8270C" December, 1996. Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. The validation methods and actions discussed in this document are based on the requirements set forth in the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," October 1999. This document covers technical problems specific to each fraction and sample matrix; however, situations may arise where data limitations must be assessed based on the reviewer's professional judgement.

Summary of Method

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are defined on page 4.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance.

Reviewer Qualifications

Data reviewers must possess a working knowledge of SW846 Analytical Method 8270C.

FOIA_07123_0003552_0615

YES NO N/A

DEFINITIONS

Acronyms

BNA - base neutral acid(another name for Semi Volatiles)
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
%D - percent difference
DCB -decachlorobiphenyl
DDD - dichlorodiphenyldichloroethane
DDE - dichlorodiphenylethane
DDT - dichlorodiphenyltrichloroethane
DoC - Date of Collection
GC - gas chromatography
GC/ECD - gas chromatograph/electron capture detector
GC/MS - gas chromatograph/mass spectrometer
GPC - gel permeation chromatography
IS - internal standard
kg - kilogram
µg - microgram
MS - matrix spike
MSD - matrix spike duplicate
l - liter
ml - milliliter
PCB - Polychlorinated biphenyl
PE - performance evaluation
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
RRF - average relative response factor (from initial calibration)
RRT - relative retention time
RSD - relative standard deviation
RT - retention time
RSCC - Regional Sample Control Center
SDG - sample delivery group
SMC - system monitoring compound
SOP - standard operating procedure
SOW - Statement of Work
SVOA - semivolatile organic acid
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TCX -tetrachloro-m-xylene
TIC - tentatively identified compound
TOPO - Task Order Project Officer
TPO - Technical Project Officer
VOA - Volatile organic

FOIA_07123_0003552_0616

YES NO N/A

VTSR - Validated Time of Sample Receipt

Data Qualifiers

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

NJ - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

LAB QUALIFIERS:

D - The positive value is the result of an analysis at a secondary dilution factor.

B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.

E - The concentration of this analyte exceeds the calibration range of the instrument.

A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

X,Y,Z- Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the

FOIA_07123_0003552_0617

YES NO N/A

data user may understand their impact on the data.

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ LAB:

SITE NAME:

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format? [ ] ___

ACTION: If not, note the effect on review of the data in the data assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter present? [ ] ___ ___

2.2 Are case number and SDG number(s) contained in the narrative or cover letter? [ ] ___ ___

II. SEMIVOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples? [ ] ___

ACTION: If no, contact lab for replacement of missing or illegible copies.

FOIA_07123_0003552_0618

	YES	NO	N/A
1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?	___	[ ]	___

FOIA_07123_0003552_0619

YES NO N/A

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, all data should be qualified as unusable (R).

ACTION: If samples were not iced, or if the ice was melted upon arrival at the laboratory and the cooler temperature was elevated (10°C), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any semi volatile technical holding times, determined from date of collection to date of extraction, been exceeded? ___ [ ]

Continuous extraction of water samples for semi volatile analysis must be started within **7** days of the date of collection. Soil/sediment samples must be extracted within 14 days of collection. Extracts must be analyzed within 40 days of the date of extraction.

Table of Holding Time Violations

Sample ID	Sample Matrix	Date Sampled	Date Lab Received	(See Traffic Report) Date Extracted	Date Analyzed
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	
_____	_____	_____	_____	_____	
_____	_____	_____	_____	_____	
_____	_____	_____	_____	_____	
_____	_____	_____	_____	_____	

FOIA_07123_0003552_0620

YES NO N/A

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).

3.0 Surrogate Recovery (Form II)

3.1 Have the semi volatile surrogate recoveries been listed on CLP Surrogate Recovery forms (Form II) for each of the following matrices:

a. Low Water [ ]

b. Low/Med Soil [ ]

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery Summary forms for each matrix:

a. Low Water [ ]

b. Low/Med Soil [ ]

ACTION: If CLP deliverables or equivalent are unavailable, document the effect(s) in data assessments. In some cases the lab may have to be contacted to obtain the data necessary to complete the validation.

FOIA_07123_0003552_0621

	YES	NO	N/A
3.3 Were outliers marked correctly with an asterisk? ACTION: Circle all outliers in red.		[ ]	___
3.4 Were two or more base neutral OR acid surrogate recoveries out of specification for any sample or method blank		___	[ ]
If yes, were samples re-analyzed?		[ ]	
Were method blanks re-analyzed?		[ ]	

ACTION: If all surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet method specifications, for the affected fraction only (i.e. either base-neutral or acid compounds):

1. Flag all positive results as estimated ("J").

2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.

3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any base-neutral or acid surrogate has a recovery of < 10%:

1. Positive results for the fraction with < 10% surrogate recovery are qualified with "J".

2. Non-detects for that fraction should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses.

3.5 Are there any transcription/calculation errors between raw data and Form II?		___	[ ]

ACTION: If large errors exist, call lab for

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YES NO N/A

explanation/resubmittal, make any necessary corrections and document effect in data assessments.

4.0 Matrix Spikes (Form III)

4.1 Have the semivolatile Matrix Spike and Matrix Spike Duplicate or duplicate unspiked Sample recoveries been listed on the CLP Recovery Form (Form III)? [ ] ___

NOTE: This method may not require a Matrix Spike Duplicate. Lab should submit MS/MSD or MS and Duplicate unspiked sample. (see section 8.4.2, page 8270C-22)

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water [ ] ___

b. Low Solid [ ] ___

c. Med Solid [ ] ___

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above. It may be necessary to contact the lab to obtain the required data.

NOTE: If the data has not been reported on CLP forms, then the laboratory must provide the information necessary to evaluate the spike recoveries in the MS and MSD. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

FOIA_07123_0003552_0623

	YES	NO	N/A
The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.			
4.3 Were matrix spikes performed at concentration > 100ug/L ?		[ ]	___
4.4 Were any semivolatile spike recoveries outside QC limits (compare to the values in Table 6, page 8270C-39 and 40) or Lab's in-house generated criteria?		[ ]	___
4.5 Were any RPD's for matrix spike and matrix spike duplicate recoveries outside QC limits?		[ ]	___

ACTION: Circle all outliers with red pencil.

ACTION: No action is taken on MS/MSD data alone. However, using professional judgement, the data reviewer may use the matrix spike / matrix spike duplicate and duplicate unspiked results in conjunction with other QC criteria to determine the need for some qualification of the data.

4.6 Was a LCS analyzed with each analytical batch? (See section 8.4.3, page 8270C-22)		[ ]	___

NOTE: When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

4.7 Were any LCS recovery outside the interim acceptance criteria of 70 - 130% or outside lab's in-house generated limits?		[ ]	___

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present?		[ ]	___

5.2 Frequency of Analysis:

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	YES	NO	N/A
Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch?		[ ]	___
5.3 Has a method blank been analyzed for each GC/MS system used ?		[ ]	___

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, use professional judgement to determine if the associated sample data should be qualified.

5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.

	YES	NO	N/A
Is the chromatographic performance (baseline stability) for each instrument acceptable for the semivolatiles?		[ ]	___

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

	YES	NO	N/A
6.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.		___	[ ]
6.2 Do any field/rinse/ blanks have positive results for target analytes and/or TICs (if required, see paragraph 10 below)?		___	[ ]

ACTION: Prepare a list of the samples associated

FOIA_07123_0003552_0625

YES NO N/A

with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).

For Common Phthalate Esters:	For Other Contaminants:	Action:
Sample conc. > CRDL, but < 10x blank result	Sample conc. > CRDL, but < 5x blank result	Flag sample result with a "U"
Sample conc. is < CRDL & < 10x blank result	Sample conc. < CRDL & < 5x blank result	Report CRDL and qualify with a "U"
Sample conc. > CRDL & > 10x blank result	Sample conc. > CRDL & > 5x blank result	No qualification is necessary

NOTE: Analytes qualified "U" for blank contamination are still considered as hits when qualifying for calibration criteria.

NOTE: If the laboratory did not report TIC analyses, check the project plans to verify whether or not it was required. (see section 7.6.2, page 8270C-19)

6.3 Are there field/rinse/equipment blanks associated

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YES NO N/A

with every sample? [ ] ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Apparatus and Materials

7.1 Did the lab use the proper gas chromatographic column for analysis of semivolatiles by Method 8270C? Check raw data, instrument logs or contact the lab to determine what type of column was used. The method requires the use of 30 m x 0.25 mm ID (or 0.32 mm ID), silicone-coated, fused silica, capillary column. [ ] ___

ACTION: If the specified column, or equivalent, was not used, document the effects in the data assessment. Use professional judgement to determine the acceptability of the data.

8.0 GC/MS Instrument Performance Check

8.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for decafluorotriphenylphosphine (DFTPP)? [ ] ___

NOTE: The performance solution should also contain 4,4-DDT, pentachlorophenol, and benzidine to verify injection port inertness and column performance. The degradation of DDT to DDE and DDD must be less than 20% total and the response of pentachlorophenol and benzidine should be within normal ranges for these compounds (based upon lab experience) and show no peak degradation or tailing before samples are analyzed. (see section 5.5 page 8270C-11).

8.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP

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	YES	NO	N/A
provided for each twelve hour shift?		[ ]	___
8.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?		[ ]	___

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____		
_____	_____		
_____	_____		

ACTION: If the lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable (R).

8.4 Have the ion abundances been normalized to m/z 198?		[ ]	___
8.5 Have the ion abundance criteria been met for each instrument used?		[ ]	___

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, the Region II TOPO must be notified.

8.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.)		___	[ ]

FOIA_07123_0003552_0628

	YES	NO	N/A
8.7 Have the appropriate number of significant figures (two) been reported?		[ ]	___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

	YES	NO	N/A
8.8 Are the spectra of the mass calibration compound acceptable?		[ ]	___

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

9.0 Target Analytes

	YES	NO	N/A
9.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:			
a. Samples and/or fractions as appropriate		[ ]	___
b. Matrix spikes and matrix spike duplicates		[ ]	___
c. Blanks		[ ]	___
D. Lab control samples		[ ]	___
9.2 Has any special cleanup, such as GPC, been performed on all soil/sediment sample extracts (see section 7.2, page 8270C-13)?		[ ]	___

ACTION: If data suggests that extract cleanup was not performed, use professional judgement. Make note in the data assessment narrative.

	YES	NO	N/A
9.3 Are the Reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
a. Samples and/or fractions as appropriate		[ ]	___
b. Matrix spikes and matrix spike duplicates			

FOIA_07123_0003552_0629

	YES	NO	N/A
(Mass spectra not required)		[ ]	
c. Blanks		[ ]	___
D Lab control samples		[ ]	___

ACTION: If any data are missing, take action specified in 3.2 above.

9.4 Is chromatographic performance acceptable with respect to:

Baseline stability?		[ ]	___
Resolution?		[ ]	___
Peak shape?		[ ]	___
Full-scale graph (attenuation)?		[ ]	___
Other:_____		[ ]	___

ACTION: Use professional judgement to determine the acceptability of the data.

9.5 Are the lab-generated standard mass spectra of identified semivolatile compounds present for each sample?		[ ]	___

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the data assessment narrative. If spectra are missing, reject all positive data.

9.6 Is the RRT of each reported compound within ± 0.06 RRT units of the standard RRT in the continuing calibration?		[ ]	___
9.7 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?		[ ]	___

FOIA_07123_0003552_0630

	YES	NO	N/A
9.8 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum?		[ ]	___

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged "N" (Presumptive evidence of the presence of the compound) or changed to not detected ("U") at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

10.0 Tentatively Identified Compounds (TIC)

10.1 If Tentatively Identified Compounds were required for this project, are all Form I's, Part B present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?		[ ]	___
10.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:		[ ]	___
a. Samples and/or fractions as appropriate		[ ]	___
b. Blanks		[ ]	___

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by CAS #.

FOIA_07123_0003552_0631

YES NO N/A

10.3 Are any target compounds from one fraction listed as TIC compounds in another (e.g., an acid compound listed as a base neutral TIC)? ___ [ ]

ACTION: i. Flag with "R" any target compound listed as a TIC.

ii. Make sure all rejected compounds are properly reported in the other fraction.

10.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum? [ ] ___

10.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%? [ ] ___

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate and remove "JN". Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R".

11.0 Compound Quantitation and Reported Detection Limits

NOTE: Average Response Factor from the initial calibration is used for quantitation.

11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found? ___ [ ]

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent

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YES NO N/A

valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two co-eluting peaks to calculate the total concentration).

11.2 Are the method detection limits adjusted to reflect sample dilutions and % moisture in case of soil samples? [ ]

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I (if present) and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red " X" across the entire page of all Form I's that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration? [ ] ___

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

13.0 GC/MS Initial Calibration (Form VI)

13.1 Are the Initial Calibration Forms (Form VI) present and complete for the semivolatile fraction? [ ] ___

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YES NO N/A

ACTION: If any calibration forms or standard raw data are missing, take action specified in 3.2 above.

13.2 Are all **average RRF's** of the four System Performance Check Compounds (SPCCs): N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol and 4-nitrophenol > 0.050? [ ] ___

ACTION: If no:
CONTRACTUAL - Circle all outliers in red. Document in the Data Assessment under contract non compliance.

ACTION: TECHNICAL - For any target analyte, CCC or SPCC with average RRF <0.05

1. "R" all non-detects;

2. "J" all positive results.

13.3 Is the % RSD for each individual Calibration Check Compound (CCC) - Acenaphthene, 1,4-Dichlorobenzene, Hexachlorobutadiene, Diphenylamine, Di-n-octyl phthalate, Fluoranthene, Benzo(a)pyrene, 4-Chloro-3-methylphenol, 2,4-Dichlorophenol, 2-Nitrophenol, Phenol, Pentachlorophenol and 2,4,6-Trichlorophenol less than 30%? [ ]

ACTION: If no:
CONTRACTUAL - Circle all outliers in red. Document in the Data Assessment under contract non compliance.

TECHNICAL - All positive hits for that particular CCC must be qualified "J". If % RSD > 90%, flag all positive results for that analyte "J" and non-detect results for that analyte "R" unusable.

13.4 If % RSD for one or more target analytes exceeds 15%, is the MEAN of the % RSD values for ALL analytes in the calibration less than or equal to 15%? [ ]

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YES NO N/A

ACTION: If yes:
CONTRACTUAL - The initial calibration is valid and the average RF from the initial calibration is used to quantitate sample results.

TECHNICAL - If the % RSD is ≥ 15.0% for any Individual target analyte, qualify positive results for that analyte "J". If % RSD > 90%, flag all positive results for that analyte "J" and non-detect results for that analyte "R" unusable.

13.5 If the MEAN % RSD is greater than 15%, Did the laboratory calculate first or second order regression fit of the calibration curve ? [ ] ___

ACTION: If no:
CONTRACTUAL - The initial calibration is not valid, Document in the Data Assessment under contract non compliance.

TECHNICAL - If % RSD is > 15.0% for any individual target analyte, qualify positive results for that analyte "J". When % RSD > 90%, flag all positive results for that analyte "J" and non-detect results for that analyte "R" unusable.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

13.6 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (Check at least two values but if errors are found, check more.) ___ [ ]

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors in data assessments.

13.7 Do the target compounds for this SDG include

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	YES	NO	N/A
Pesticides?		[ ]	___
13.8 If the pesticide compounds include DDT, was the percent breakdown of DDT to DDD and DDE greater than 20%?		___	[ ]

ACTION: If DDT percent breakdown exceeds 20%:

i. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE results are positive, qualify the quantitation limit for DDT as unusable, "R".

ii. Qualify all positive results for DDD and DDE as presumptively present at an approximate concentration "JN".

14.0 GC/MS Calibration Verification (Form VII)

14.1 Are the Calibration verification Forms (Form VII) present and complete for all compounds of interest?		[ ]	___
14.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument?		[ ]	___

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

ACTION: If any forms are missing or no calibration verification standard has been analyzed within twelve hours of every sample analysis, call lab for explanation/resubmittal. If calibration verification data is not

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YES NO N/A

available, flag all associated sample data as unusable ("R").

14.3 Do any of the SPCCs have an **RRF <0.05**? ___ [ ] ___
If YES, did the lab take corrective action as in section 7.4.4.2, page 8270C-17. [ ] ___

ACTION: If no:
CONTRACTUAL - Circle all outliers in red. Document in the Data Assessment under contract non compliance.

ACTION: TECHNICAL - For any target analyte, SPCC or CCC with RRF <0.05

1. "R" all non-detects;

2. "J" all positive results.

14.4 Do any of the CCCs have a %D between the initial and verification RRF which exceeds 20.0%? ___ [ ] ___
ACTION: If Yes:
CONTRACTUAL - Circle all outliers in red. Document in the Data Assessment under contract non compliance.

TECHNICAL - All positive hits for that particular CCC must be qualified "J" and all non-detects "UJ". When %D > 90%, flag all positive results for that analyte "J"and non-detect results for that analyte "R" unusable.

14.5 Do any target compounds have a % D between the initial and verification RRF which exceeds 20.0%? ___ [ ]

ACTION: If yes:
CONTRACTUAL - Circle all outliers in red.

TECHNICAL - All positive hits for that particular target compound must be qualified "J" and all non-detects "UJ". When %D > 90%, flag all positive results for that analyte "J" and non-detect results for that analyte "R" unusable.

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YES NO N/A

14.6 If %D for one or more target analytes exceeds 20%, Is the MEAN of the %D values for all analytes in The calibration less than or equal to 20%? [ ]

ACTION: If yes:
CONTRACTUAL - The initial calibration is valid and the average RF from the initial calibration is used to quantitate sample results.

If no:
The initial calibration is invalid. Document in the Data Assessment under contract non compliance.

14.7 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or percent difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more). ___ [ ]

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect(s) in the data assessments.

15.0 Internal Standards (Form VIII)

15.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to + 100%) for each continuing calibration? [ ] ___ ___

ACTION: List each outlying internal standard below.

Sample ID	IS #	Area	LowerLimit	Upper Limit

(Attach additional sheets if necessary.)

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YES NO N/A

ACTION: i. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.

ii. Non-detects associated with IS > 100% should not be qualified.

iii. If the IS area is below the lower limit (<50%), qualify all associated non-detects (U-values) "J". If extremely low area counts are reported (<25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable (R).

15.2 Are the retention times of all internal standards within 30 seconds of the associated calibration standard? [ ] ___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

16.0 Field Duplicates

16.1 Were any field duplicates submitted for semivolatile analysis? [ ] ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

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VALIDATION SOP

Semivolatile Organics SIM

USEPA Region 2 SOP HW-35, Revision 2, 3/13

Hazardous Waste Support Section
SOP NO. HW-35 Revision 2
Semivolatile Data Validation

Approvals:

Russell Arnone — 03/28/13
Chemist, Hazardous Waste Support Section — Date

Philip Cocuzza — 3/28/13
Chief, Hazardous Waste Support Section — Date

Jon Gabry — 3/26/13
Chief, Hazardous Waste Support Branch — Date

Approving Official Annual Review — Date

NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (hereafter referred to as USEPA) and other governmental employees. They do not constitute rule making by USEPA, and may not be relied upon to create a substantive or procedural right enforceable by any other person. The Government may take action that is at variance with the policies and procedures in this manual.

The guidance for data validation set forth in the quality assurance project plan (QAPP) for the project associated with the data in question will always take precedence over the data validation guidance listed herein.

Validators should note that their professional judgment supersedes any guidance listed in this document.

Government contractors to the USEPA using this document to validate data should not hesitate to contact their Contracting Officer Representative with any questions regarding data validation or data package completeness.

This document can be obtained from the USEPA's Region 2 Quality Assurance website at:

http://www.epa.gov/region2/qa/documents.htm

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ACRONYMS

%D	Percent Difference
%RSD	Percent Relative Standard Deviation
ARO	Aroclor
ASB	Analytical Services Branch
BFB	Bromofluorobenzene
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CF	Calibration Factor
CLP	Contract Laboratory Program
CLP PO	Contract Laboratory Program Project Officer
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
DART	Data Assessment Rapid Transmittal
DAT	Data Assessment Tool
DCB	Decachlorobiphenyl
DFTPP	Decafluorotriphenylphosphine
DMC	Deuterated Monitoring Compound
DQA	Data Quality Assessment
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EDM	EXES Data Manager
ESAT	Environmental Services Assistance Team
EXES	Electronic Data eXchange and Evaluation System
GC	Gas Chromatograph
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPC	Gel Permeation Chromatography
HWSS	Hazardous Waste Support Section
INDA	Individual Standard Mixture A
INDB	Individual Standard Mixture B
INDC	Individual Standard Mixture C
LCS	Laboratory Control Sample
MS	Matrix Spike
MSD	Matrix Spike Duplicate
OSRTI	Office of Superfund Remediation and Technology Innovation
PCBs	Polychlorinated Biphenyls
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QC	Quality Control
RAS	Routine Analytical Services

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RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference
RRF	Relative Response Factor
$\overline{RRF}$	Mean Relative Response Factor
RRT	Relative Retention Time
RSCC	Regional Sample Control Center Coordinator
RSD	Relative Standard Deviation
RT	Retention Time
SAP	Sampling and Analysis Plan
SCP	Single Component Pesticide
SDG	Sample Delivery Group
SIM	Selected Ion Monitoring
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TCL	Target Compound List
TCLP	Toxicity Characteristics Leachate Procedure
TCX	Tetrachloro-m-xylene
TIC	Tentatively Identified Compound
TOPO	Task Order Project Officer
TR/COC	Traffic Report/Chain of Custody Record
USEPA	United States Environmental Protection Agency
UV	Ultraviolet
VTSR	Validated Time of Sample Receipt

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INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the validity of analytical data generated through the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Multi-Media, Multi-Concentration Organics Analysis (SOM01.2), and any future editorial revisions of SOM01.2, hereinafter referred to as the SOM01.2 SOW. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process.

The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and QC criteria established in the SOW, and (b) the validity and extent of bias of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to site-specific technical requirements, the need to facilitate the progress of specific projects, and availability for re-sampling.

The reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as **professional judgment**, should also be used to determine the ultimate validity of data, especially in those cases where all data does not meet specific technical criteria.

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DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.

DATA PACKAGE INSPECTION

For data obtained through the Contract Laboratory Program (CLP), the EXES Data Manager (EDM) is a useful tool in the data review process. For more information about EDM, please refer to the following Sample Management Office (SMO) website:

https://epasmoweb.fedcsc.com/help/guides/Submit%20and%20Inspect%20Data%20Quick%20Guide%20%28EXES%29.pdf

EDM will identify any missing and/or incorrect information in the data package. The CLP laboratory may submit a reconciliation package for any missing items or to correct data.
If there are any concerns regarding the data package, contact the CLP Project Officer (CLP PO) from the Region where the samples were taken. For personnel contact information, please refer to the following CLP website:

http://www.epa.gov/superfund/programs/clp/contacts.htm

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HWSS DATA VALIDATION PROCESS

After downloading the data package from EDM, the data validator will use the recommendations in this SOP as well as their own professional judgment to validate the data.

The data will be saved in the following location, under the appropriate case number folder:

G:\DESADIV\HWSS\DATA VALIDATION

The file naming conventions will consist of

A. case number i.e., 12345
B. SDG name i.e., BXY12
C. level of validation performed i.e., S3VE

Examples: **12345_BXY12_S3VE.xls**

12345_BXY12_S3VEM.xls

When data validation is completed, the data package is uploaded for the client to download from the HWSS data delivery website:

https://epaqpx.rtp.epa.gov/hwssclpdeliverables

The completed data package includes the Executive Narrative (see Appendix B for template), the Sample Summary Report (see Appendix C for example), and the Electronic Data Deliverable (EDD) (see Appendix D for a list of the column headers included in this document).

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PRELIMINARY REVIEW

This document is for the review of analytical data generated through the SOM01.2 SOW and any future editorial revisions of SOM01.2 for USEPA Region 2. To use this document effectively, the reviewer should have an understanding of the analytical method and a general overview of the Sample Delivery Group (SDG) or sample Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the sample data package [e.g., Modified Analysis requests, Traffic Report/Chain of Custody (TR/COC) documentation, SDG Narratives, etc.].

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The criteria for data validation outlined in the QAPP supersede this Standard Operating Procedure. The reviewer should contact the appropriate Regional Contract Laboratory Program Project Officer (CLP PO) to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

The SDGs or Cases routinely have unique samples that require special attention from the reviewer. These include field blanks and trip blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The sampling records (e.g., TR/COC records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken,
2. The Case number,
3. The complete list of samples with information on:
    a. Sample matrix;
    b. Field blanks (i.e., equipment blanks or rinsate blanks) and trip blanks;
    c. Field duplicates;
    d. Field spikes;
    e. QC audit samples;
    f. Shipping dates;
    g. Preservatives; and
    h. Laboratories involved.

The TR/COC documentation includes sample descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

The laboratory's SDG Narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, preservation, and unusual events should be documented in the SDG Narrative. The reviewer should also inspect any email or telephone/communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office (SMO), and USEPA Region 2.

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Preservation

Action:

1. Qualify aqueous sample results using preservation and technical holding time information as follows (see Table 1):
    a. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed within the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    b. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], no qualification of the data is necessary.
    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.
2. Qualify non-aqueous sample results using preservation and technical holding time information as follows (see Table 1):
    a. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed within the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    b. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed outside the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], no qualification of the data is necessary.
    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding time [14 days from sample collection for

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extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

3. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the Data Review Narrative.
4. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
5. If technical holding times are grossly exceeded, use professional judgment to qualify the data.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, when technical holding times are exceeded.

Table 1. Holding Time Actions for Semivolatile Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤ 7 days (for extraction) ≤ 40 days (for analysis)	J	UJ
	No	> 7 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes	≤ 7 days (for extraction) ≤ 40 days (for analysis)	No qualification	
	Yes	> 7 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes/No Grossly Exceeded		Use professional judgment	
Non-Aqueous	No	≤ 14 days (for extraction) ≤ 40 days (for analysis)	J	UJ
	No	> 14 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes	≤ 14 days (for extraction) ≤ 40 days (for analysis)	No qualification	
	Yes	> 14 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes/No Grossly Exceeded		Use professional judgment	

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Gas Chromatograph/Mass Spectrometer (GC/MS) Instrument Performance Check

Action:

NOTES: These requirements do not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.
All mass spectrometer instrument conditions must be identical to those used during the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the method specifications are contrary to the Quality Assurance (QA) objectives, and are therefore unacceptable.

NOTES: No data should be qualified based on DFTPP failure. Instances of this should be noted in the narrative.
All ion abundance ratios must be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be up to 100% that of m/z 198.
The requirement to analyze the instrument performance check solution is optional when analysis of Polycyclic Aromatic Hydrocarbon (PAH)/pentachlorophenol is to be performed by the Selected Ion Monitoring (SIM) technique.

1. If samples are analyzed without a preceding valid instrument performance check or are analyzed 12 hours after the Instrument Performance Check, qualify all data in those samples as unusable (R).
2. If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.
3. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, the reviewer must use professional judgment to assess the data. Notify the laboratory's Contract Laboratory Program Project Officer (CLP PO).
4. If ion abundance criteria are not met, use professional judgment to determine to what extent the data may be utilized. Guidelines to aid in the application of professional judgment in evaluating ion abundance criteria are discussed as follows:
    a. Some of the most critical factors in the DFTPP criteria are the non-instrument specific requirements that are also not unduly affected by the location of the spectrum on the chromatographic profile. The m/z ratios for 198/199 and 442/443 are critical. These ratios are based on the natural abundances of carbon 12 and carbon 13 and should always be met. Similarly, the relative abundances for m/z 68, 70, 197, and 441 indicate the condition of the instrument and the suitability of the resolution adjustment. Note that all of the foregoing abundances relate to adjacent ions; they are relatively insensitive to differences in instrument design and position of the spectrum on the chromatographic profile.
    b. For the ions at m/z 51, 127, and 275, the actual relative abundance is not as critical. For instance, if m/z 275 has 80.0% relative abundance (criteria: 10.0-60.0%) and other criteria are met, the deficiency is minor.

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c. The relative abundance of m/z 365 is an indicator of suitable instrument zero adjustment. If relative abundance for m/z 365 is zero, minimum detection limits may be affected. On the other hand, if m/z 365 is present, but less than the 0.75% minimum abundance criteria, the deficiency is not as serious.

5. Note, in the Data Review Narrative, decisions to use analytical data associated with DFTPP instrument performance checks not meeting contract requirements.
6. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in Semivolatiles Organic Analysis, Section II.D.5 of the SOM1.2 NFG, obtain additional information on the instrument performance checks. If the techniques employed are found to be at variance with the contract requirements, the performance and procedures of the laboratory may merit evaluation. Note, for CLP PO action, concerns or questions regarding laboratory performance. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than from the DFTPP peak), note this for CLP PO action.
7. Use professional judgment to determine whether associated data should be qualified based on the spectrum of the mass calibration compound.

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Initial Calibration

Table 2. Semivolatile Target Compounds Exhibiting Poor Response

Compounds	
2,2'-Oxybis-(1-chloropropane)	Benzaldehyde
4-Chloroaniline	4-Nitroaniline
Hexachlorobutadiene	4,6-Dinitro-2-methylphenol
Hexachlorocyclopentadiene	N-Nitrosodiphenylamine
2-Nitroaniline	3-3'-Dichlorobenzidine
3-Nitroaniline	1,1'-Biphenyl
2,4-Dinitrophenol	Dimethylphthalate
4-Nitrophenol	Diethylphthalate
Acetophenone	1,2,4,5-Tetrachlorobenzene
Caprolactam	Carbazole
Atrazine	Butylbenzylphthalate
Di-n-butylphthalate	Di-n-octylphthalate
Bis(2-ethylhexyl)phthalate	

Action:

NOTES: The twenty-five (25) poor performing compounds are listed in Table 2 above. The relative response factor (RRF) for these compounds must be greater than or equal to 0.010. The RRF for all other semivolatile target compounds must be ≥ 0.050. Analytes previously qualified as non-detected (U) for blank contamination are still treated as "hits" when qualifying for initial calibration criteria.
If analysis by Selected Ion Monitoring (SIM) technique is requested for PAHs/pentachlorophenols, calibration standards are analyzed at 0.10, 0.20, 0.40, 0.80, and 1.0 ng/µL for each target compound of interest and the associated DMCs. Pentachlorophenol will require only a four-point initial calibration at 0.20, 0.40, 0.80, and 1.0 ng/µL

1. Qualify all semivolatile target compounds, including the compounds exhibiting poor response listed in Table 2, using the following criteria (see Table 3):
    a. If any semivolatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds exhibiting poor response listed in Table 2, and 0.050 for all other semivolatile compounds), use professional judgment for detects, based on mass spectral identification, to qualify the data as estimated (J).
    b. If any semivolatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds exhibiting poor response listed in Table 2, and 0.050 for all other semivolatile compounds), qualify non-detected compounds as unusable (R).

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c. If any of the semivolatile target compounds listed in Table 2 has %RSD greater than 40.0%, qualify detects as estimated (J), and non-detected compounds using professional judgment (see Action 2).
d. For all other semivolatile target compounds, if %RSD is greater than 20.0%, qualify detects as estimated (J), and non-detected compounds using professional judgment (see Action 2).
e. If the semivolatile target compounds meet the acceptance criteria for RRF and the %RSD, no qualification of the data is necessary.
f. No qualification of the data is necessary on the DMC RRF and %RSD data alone. Use professional judgment and follow the guidelines in Action 2 to evaluate the DMC RRF and %RSD data in conjunction with the DMC recoveries to determine the need for qualification of data.

2. At the reviewer's discretion, and based on the project-specific Data Quality Objectives (DQOs), a more in-depth review may be considered using the following guidelines:
   a. If any semivolatile target compound has a %RSD greater than the maximum criterion (40.0% for the compounds listed in Table 2, and 20.0% for all other semivolatile compounds), and if eliminating either the high or the low-point of the curve does not restore the %RSD to less than or equal to the required maximum:
      i. Qualify detects for that compound(s) as estimated (J).
      ii. Qualify non-detected semivolatile target compounds using professional judgment.
   b. If the high-point of the curve is outside of the linearity criteria (e.g., due to saturation):
      i. Qualify detects outside of the linear portion of the curve as estimated (J).
      ii. No qualifiers are required for detects in the linear portion of the curve.
      iii. No qualifiers are required for semivolatile target compounds that were not detected.
   c. If the low-point of the curve is outside of the linearity criteria:
      i. Qualify low-level detects in the area of non-linearity as estimated (J).
      ii. No qualifiers are required for detects in the linear portion of the curve.
      iii. For non-detected semivolatile compounds, use the lowest point of the linear portion of the curve to determine the new quantitation limit.
3. If the laboratory has failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
4. Document in the Data Assessment Report the analytes that fail %RSD and/or RRF criteria.
5. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if calibration criteria are grossly exceeded.

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Table 3. Initial Calibration Actions for Semivolatiles Analyses

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RRF < 0.010 (target compounds listed in Table 2) RRF < 0.050 (all other target compounds)	J (based on mass spectral identification)	R
RRF ≥ 0.010 (target compounds listed in Table 2) RRF ≥ 0.050 (all other target compounds)	No qualification	
% RSD ≤ 40.0 (target compounds listed in Table 2) % RSD ≤ 20.0 (all other target compounds)	No qualification	
% RSD > 40.0 (target compounds listed in Table 2) % RSD > 20.0 (all other target compounds)	J	Use professional judgment

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Continuing Calibration Verification (CCV)

Action:

NOTES: Verify that the CCV was run at the required frequency (an opening and closing CCV must be run within 12-hour period) and the CCV was compared to the correct initial calibration. If the mid-point standard from the initial calibration is used as an opening CCV, verify that the result (RRF) of the mid-point standard was compared to the average RRF from the correct initial calibration.
The closing CCV used to bracket the end of a 12-hour analytical sequence may be used as the opening CCV for the new 12-hour analytical sequence, provided that all the technical acceptance criteria are met for an opening CCV (see Table 4 below). If the closing CCV does not meet the technical acceptance criteria for an opening CCV, then a DFTPP tune followed by an opening CCV is required and the next 12-hour time period begins with the DFTPP tune.
All DMCs must meet RRF ≥ 0.010. No qualification of the data is necessary on the DMCs RRF and %RSD/%D data alone. However, use professional judgment to evaluate the DMC and %RSD/%D data in conjunction with the DMC recoveries to determine the need of qualification the data.

1. If a CCV (opening and closing) was not run at the appropriate frequency, qualify data using professional judgment.
2. Qualify all semivolatile target compounds, including the compounds exhibiting poor response listed in Table 2 using the following criteria:
    a. For an opening CCV, if any semivolatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds exhibiting poor response, and 0.050 for all other semivolatile compounds), use professional judgment for detects, based on mass spectral identification, to qualify detects as estimated (J) and qualify non-detected compounds as unusable (R).
    b. For a closing CCV, if any semivolatile target compound has an RRF value less than 0.010, use professional judgment for detects based on mass spectral identification to qualify detects as estimated (J), and qualify non-detected compounds as unusable (R).
    c. For an opening CCV, if the Percent Difference value for any of the semivolatile target compounds listed in Table 2 is outside the ± 40.0% criterion, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
    d. For an opening CCV, if the Percent Difference value for any other semivolatile target compound is outside the ± 25.0% criterion, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
    e. For a closing CCV, if the Percent Difference value for any semivolatile target compound is outside the ± 50.0% criterion, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
    f. If the semivolatile target compounds meet the acceptable criteria for RRF and the Percent Difference, no qualification of the data is necessary.

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g. No qualification of the data is necessary on the DMC RRF and the Percent Difference data alone. Use professional judgment to evaluate the DMC RRF and Percent Difference data in conjunction with the DMC recoveries to determine the need for qualification of data.

3. If the laboratory has failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
4. Document in the Data Assessment under Contract Problems/Non-Compliance if more than two of the required analytes failed the acceptance criteria in Table 4.
5. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if calibration criteria are grossly exceeded.

Table 4. Continuing Calibration Verification (CCV) Actions for Semivolatile Analyses

Criteria for Opening CCV	Criteria for Closing CCV	Action	
		Detected Associated Compounds	Non-Detected Associated Compounds
RRF < 0.010 (semivolatile target compounds listed in Table 2) RRF < 0.050 (all other semivolatile target compounds)	RRF < 0.010 (all semivolatile target compounds)	J (based on mass spectral identification)	R
RRF ≥ 0.010 (semivolatile target compounds listed in Table 2) RRF ≥ 0.050 (all other semivolatile target compounds)	RRF ≥ 0.010 (all semivolatile target compounds)	No qualification	
%D > 40.0 or < -40.0 (semivolatile target compounds listed in Table 2) %D > 25.0 or < -25.0 (all other semivolatile target compounds)	%D > 50.0 or < -50.0 (all semivolatile target compounds)	J	UJ
%D ≤ 40.0 and ≥ -40.0 (semivolatile target compounds listed in Table 2) %D ≤ 25.0 and ≥ -25.0 (all other semivolatile target compounds)	%D ≤ 50.0 and ≥ -50.0 (all semivolatile target compounds)	No qualification	

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Blanks

Action:

NOTES: The concentration of non-target compounds in all blanks must be less than or equal to 10 µg/L.
The concentration of each target compound found in the method or field blanks must be less than its CRQL listed in the method, except bis(2-ethylhexyl)phthalate, which must be less than 5x its respective CRQL in low-level samples.
Data concerning the field blanks are not evaluated as part of the CCS process. If field blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

NOTES: "Water blanks, "drill blanks", and "distilled water blanks" are validated like any other sample and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.
All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for system monitoring compounds, instrument performance criteria, and spectral or calibration QC problems.
Samples taken from a drinking water tap do not have associated field blanks.
When applied as described in Table 5 below, the contaminant concentration in the blank is multiplied by the sample dilution factor.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one of the same type of blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.

1. If a semivolatile compound is found in a method blank, but not found in the sample, no qualification of the data is necessary (see Table 5).
2. If the method or field blanks contain a semivolatile Target Compound List (TCL) compound(s) at a concentration less than the CRQL (less than 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples) and:
    a. the sample concentration is less than the CRQL (less than 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples), report the CRQL value with a "U".
    b. the sample concentration is greater than or equal to the CRQL (greater than or equal to 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples), no qualification is required.
3. If the method or field blanks contain a semivolatile TCL compound(s) at a concentration greater than the CRQL (greater than 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples) and:

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   a. the sample concentration is less than the CRQL (less than 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples), report the CRQL value with a "U".
   b. the sample concentration is greater than or equal to the CRQL (greater than or equal to 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples), and less than or equal to the blank concentration, report the concentration of the compound in the sample at the same concentration found in the blank and qualify with a "U".
   c. the sample concentration is greater than or equal to the CRQL (greater than or equal to 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples) and greater than the blank concentration, no qualification is required.
4. If the method or field blanks contain a semivolatile TCL compound(s) at a concentration equal to the CRQL (equal to 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples) and:
   a. the sample concentration is less than or equal to the CRQL (less than or equal to 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples), report the CRQL value with a "U".
   b. the sample concentration is greater than the CRQL (greater than 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples), no qualification is required.
5. If gross contamination exists (i.e., saturated peaks by GC/MS) in the method or field blanks, raise the CRQL to the level of the blank contamination and report the associated sample data below this level as CRQL-U. Non-detected semivolatile target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.
6. If contaminants are found in the field blanks, the following is recommended:
   a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank.
      i. If the analyte was present at a comparable level in the method blank, the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.
      ii. If the analyte was not present in the method blank, the source of contamination may be in the storage area, in the field, or during sample transport. Consider all associated samples for possible cross-contamination.
7. Tentatively Identified Compounds (TICs) should only be considered if requested.
   a. For TICs, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank (TIC concentration < 5xblank concentration), qualify the sample data as unusable (R).
8. If method blank data are unavailable, the reviewer may use professional judgment or substitute field blank data for missing method blank data.

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NOTE: There may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, they should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result.

Table 5. Blank Actions for Semivolatiles Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field	Detects	Not detected	No qualification required
	< CRQL *	< CRQL*	Report CRQL value with a U
		≥ CRQL*	No qualification required
	> CRQL *	< CRQL*	Report CRQL value with a U
		≥ CRQL* and ≤ blank concentration	Report blank value for sample concentration with a U
		≥ CRQL* and > blank concentration	No qualification required
	= CRQL*	≤ CRQL*	Report CRQL value with a U
		> CRQL*	No qualification required
	Gross contamination	Detects	Report blank value for sample concentration with a U

* 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level samples.

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Deuterated Monitoring Compounds (DMCs)

Table 6. Semivolatile Deuterated Monitoring Compounds (DMCs) and Recovery Limits

DMC	Recovery Limits (%) for Water Samples	Recovery Limits (%) for Soil/Sediment Samples
Phenol-d_5	39 – 106	17 – 103
Bis-(2-chloroethyl) ether-d_8	40 – 105	12 – 98
2-Chlorophenol-d_4	41 – 106	13 – 101
4-Methylphenol-d_8	25 – 111	8 – 100
Nitrobenzene-d_5	43 – 108	16 – 103
2-Nitrophenol-d_4	40 – 108	16 – 104
2,4-Dichlorophenol-d_3	37 – 105	23 – 104
4-Chloroaniline-d_4	1 – 145	1 – 145
Dimethylphthalate-d_6	47 – 114	43 – 111
Acenaphthylene-d_8	41 – 107	20 – 97
4-Nitrophenol-d_4	33 – 116	16 – 166
Fluorene-d_{10}	42 – 111	40 – 108
4,6-Dinitro-2-methylphenol-d_2	22 – 104	1 – 121
Anthracene-d_{10}	44 – 110	22 – 98
Pyrene-d_{10}	52 – 119	51 – 120
Benzo(a)pyrene-d_{12}	32 – 121	43 – 111
Fluoranthene-d_{10} (SIM)	50 – 150	50 – 150
2-Methylnaphthalene-d_{10} (SIM)	50 – 150	50 – 150

Action:

NOTES: Recoveries for DMCs in semivolatile samples and blanks must be within the limits specified in Table 6.
The recovery limits for any of the compounds listed in Table 6 may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

NOTE: Up to four (4) DMCs per sample may fail to meet the recovery limits, but all recoveries must be > 0%. As per SOM01.2, any sample which has more than 4 DMCs outside the limits must be reanalyzed.

Table 8 lists the semivolatile DMCs and their associated target compounds. If **any** DMC recovery in the semivolatiles fraction is out of specification, qualify the data considering the existence of interference in the raw data (see Table 7). Considerations include, but are not limited to:

1. For any recovery greater than the upper acceptance limit:
    a. Qualify detected associated semivolatile target compounds as estimated high (J+).
    b. Do not qualify non-detected associated semivolatile target compounds.
2. For any recovery less than the lower acceptance limit:

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   a. Qualify detected associated semivolatile target compounds as estimated low (J-).
   b. Qualify non-detected associated semivolatile target compounds as estimated (UJ).
3. For any recovery within acceptance limits, no qualification of the data is necessary.
4. In the special case of a blank analysis having DMCs out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable DMC recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows some use of the affected data, note analytical problems for Contract Laboratory Program Project Officer (CLP PO) action.
5. If more than four DMCs are outside of the recovery limits for semivolatiles analysis and the sample was not reanalyzed, note under Contract Problems/Non-Compliance.

Table 7. Deuterated Monitoring Compound (DMC) Recovery Actions for Semivolatiles Analyses

Criteria	Action	
	Detected Associated Compounds	**Non-detected Associated Compounds**
%R > Upper Acceptance Limit	J+	No qualification
%R < Lower Acceptance Limit	J-	UJ
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	

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Table 8. Semivolatile Deuterated Monitoring Compounds (DMCs) and the Associated Target Compounds

Phenol-d_5	**2-Chlorophenol-d_4**	**2-Nitrophenol-d_4**
Benzaldehyde Phenol	2-Chlorophenol	Isophorone 2-Nitrophenol
bis(2-Chloroethyl) ether-d_8	**4-Methylphenol-d_8**	**4-Chloroaniline-d_4**
bis-(2-Chloroethyl) ether 2,2'-oxybis(1-Chloropropane) bis(2-Chloroethoxy) methane	2-Methylphenol 4-Methylphenol 2,4-Dimethylphenol	4-Chloroaniline Hexachlorocyclopentadiene 3,3'-Dichlorobenzidine
Nitrobenzene-d_5	**2,4-Dichlorophenol-d_3**	**Dimethylphthalate-d_6**
Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene 2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosodiphenylamine	2,4-Dichlorophenol Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol 2,3,4,6-Tetrachlorophenol	Caprolactam 1,1'-Biphenyl Dimethylphthalate Diethylphthalate Di-n-butylphthalate Butylbenzylphthalate bis(2-ethylhexyl)phthalate Di-n-octylphthalate
Fluorene-d_{10}	**Anthracene-d_{10}**	**Pyrene-d_{10}**
Dibenzofuran Fluorene 4-Chlorophenyl-phenylether 4-Bromophenyl-phenylether Carbazole	Hexachlorobenzene Atrazine Phenanthrene Anthracene	Fluoranthene Pyrene Benzo(a)anthracene Chrysene
Acenaphthylene-d_8	**4-Nitrophenol-d_4**	**Benzo (a) pyrene-d_{12}**
Naphthalene 2-Methylnaphthalene 2-Chloronaphthalene Acenaphthylene Acenaphthene	2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol 4-Nitrophenol 4-Nitroaniline	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
4,6-Dinitro-2-methylphenol-d_2	**Fluoranthene-d_{10} (SIM)**	**2-Methylnapthalene-d_{10} (SIM)**
4,6-Dinitro-2-methylphenol	Fluoranthene Pyrene Benzo(a)anthracene Chrysene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene	Napthalene 2-Methylnapthalene Acenapthylene Acenaphthene Fluorene Pentachlorophenol Phenathrene Anthracene

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Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

Action:

NOTES: Data for MS and MSDs will not be present unless requested by the Region. Notify the Contract Laboratory Program Project Officer (CLP PO) if a field blank was used for the MS and MSD.

NOTE: For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

1. No qualification of the data is necessary on MS and MSD data alone. However, using professional judgment, the validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

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Internal Standards

Action:

1. If an internal standard area count for a sample or blank is greater than 200.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration) (see Table 9):
    a. Qualify detects for compounds quantitated using that internal standard as estimated low (J-).
    b. Do not qualify non-detected associated compounds.
2. If an internal standard area count for a sample or blank is less than 50.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration):
    a. Qualify detects for compounds quantitated using that internal standard as estimated high (J+).
    b. Qualify non-detected associated compounds as unusable (R).
3. If an internal standard area count for a sample or blank is greater than or equal to 50.0%, and less than or equal to 200% of the area for the associated standard opening CCV or mid-point standard from initial calibration, no qualification of the data is necessary.
4. Absolute RTs of internal standards should not vary dramatically between samples and the associated 12-hour calibration standard (opening CCV or mid-point standard from the initial calibration). If an internal standard RT varies by more than 30.0 seconds: Examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Detects should not need to be qualified as unusable (R) if the mass spectral criteria are met.
5. If an internal standard RT varies by less than or equal to 30.0 seconds, no qualification of the data is necessary.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.

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Table 9. Internal Standard Actions for Semivolatiles Analyses

Criteria	Action	
	Detected Associated Compounds*	**Non-detected Associated Compounds***
Area counts > 200% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J-	No qualification
Area counts < 50% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J+	R
Area counts ≥ 50% but ≤ 200% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	No qualification	
RT difference > 30.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from initial calibration)	R	
RT difference ≤ 30.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from initial calibration)	No qualification	

* For semivolatile compounds associated to each internal standard, see Table 2 - Semivolatile Target Compounds and Deuterated Monitoring Compounds with Corresponding Internal Standards for Quantitation in SOM01.2, Exhibit D, available at: http://www.epa.gov/superfund/programs/clp/som1.htm

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Standards Data

Action:

If missing deliverables are unavailable, document the effect in the Data Assessment.

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Target Compound Identification

Criteria:

1. The Relative Retention Times (RRTs) of reported compounds must be within ±0.06 RRT units of the standard RRT [opening Continuing Calibration Verification (CCV) or mid-point standard from the initial calibration].
2. Mass spectra of the sample compound and a current laboratory-generated standard [i.e., the mass spectrum from the associated calibration standard (opening CCV or mid-point standard from initial calibration)] must match according to the following criteria:
    a. All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.
    b. The relative intensities of these ions must agree within ±20% between the standard and sample spectra (e.g., for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%).
    c. Ions present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum, must be evaluated by a reviewer experienced in mass spectral interpretation.

Action:

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, qualify all such data as unusable (R).
2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.
3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Contract Laboratory Program Project Officer (CLP PO) action, the necessity for numerous or significant changes.

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Tentatively Identified Compounds (TICs)

Action:

NOTE: Tentatively identified compounds should only be evaluated when requested by a party from outside of the Hazardous Waste Support Section (HWSS).

1. Qualify all TIC results for which there is presumptive evidence of a match (e.g. greater than or equal to 85% match) as tentatively identified (NJ), with approximated concentrations. TICs labeled "unknown" are qualified as estimated (J).
2. General actions related to the review of TIC results are as follows:
    a. If it is determined that a tentative identification of a non-target compound is unacceptable, change the tentative identification to "unknown" or another appropriate identification, and qualify the result as estimated (J).
    b. If all contractually-required peaks were not library searched and quantitated, the Region's designated representative may request these data from the laboratory.
3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. If there is more than one possible match, report the result as "either compound X or compound Y". If there is a lack of isomer specificity, change the TIC result to a nonspecific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).
4. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).
5. Target compounds from other fractions and suspected laboratory contaminants should be marked as "non-reportable".
6. Other Case factors may influence TIC judgments. If a sample TIC match is poor, but other samples have a TIC with a valid library match, similar RRT, and the same ions, infer identification information from the other sample TIC results.
7. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.
8. Note, for Contract Laboratory Program Project Officer (CLP PO) action, failure to properly evaluate and report TICs

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Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

Action:

1. When a sample is analyzed at more than one dilution, the lowest CRQLs are used unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample. Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original Form I and substituting the data from the diluted sample.
2. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.
3. For non-aqueous samples, if the percent moisture is less than 70.0%, no qualification of the data is necessary. If the percent moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as estimated (J) and non-detects as approximated (UJ). If the percent moisture is greater than or equal to 90.0%, qualify detects as estimated (J) and non-detects as unusable (R) (see Table 10).
4. Note, for Contract Laboratory Program Project Officer (CLP PO) action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.

Table 10. Percent Moisture Actions for Semivolatiles Analysis For Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
% Moisture < 70.0	No qualification	
70.0 < % Moisture < 90.0	J	UJ
% Moisture > 90.0	J	R

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Field Duplicates

Action:

NOTE: In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.

Identify which samples within the data package are field duplicates. Estimate the relative percent difference (RPD) between the values for each compound. Use professional judgment to note large RPDs (> 50%) in the narrative.

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System Performance

Action:

Use professional judgment to qualify the data if it is determined that system performance has degraded during sample analyses. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any degradation of system performance which significantly affected the data.

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Overall Assessment of Data

Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

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APPENDIX A: GLOSSARY

Analyte -- The element of interest, ion, or parameter an analysis seeks to determine.
Analytical Services Branch (ASB) -- Directs the Contract Laboratory Program (CLP) from within the Office of Superfund Remediation and Technical Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).
Analytical Sample -- Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB). Note that the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA); Matrix Spike samples; duplicate samples; serial dilution samples, analytical (post-digestion/post-distillation) spike samples; Interference Check Samples (ICSs); Laboratory Control Samples (LCSs); and Preparation Blanks.
Associated Samples -- Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples run under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.
Blank -- A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.
Calibration -- The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentration of acids as used in the sample preparation.
Calibration Blank -- A blank solution containing all of the reagents in the same concentration as those used in the analytical sample preparation. This blank is not subject to the preparation method.
Calibration Curve -- A plot of instrument response versus concentration of standards.
Calibration Standards -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.
Case -- A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).
Contract Compliance Screening (CCS) -- A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under USEPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.
Continuing Calibration Verification (CCV) -- A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples.

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Contract Laboratory Program (CLP) -- Supports the USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technical Innovation (OSRTI) of USEPA.
Contract Laboratory Program Project Officer (CLP PO) -- The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.
Contract Required Quantitation Limit (CRQL) -- Minimum level of quantitation acceptable under the contract Statement of Work (SOW).
Duplicate -- A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.
Field Blank -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.
Field Duplicate -- A duplicate sample generated in the field, not in the laboratory.
Holding Time -- The maximum amount of time samples may be held before they are processed.
Contractual -- The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.
Technical -- The maximum amount of time that samples may be held from the collection date until analysis.
Initial Calibration -- Analysis of analytical standards for a series of different specified concentrations to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.
Initial Calibration Verification (ICV) -- Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when USEPA ICV solutions are not available.
Internal Standard -- A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.
Matrix -- The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrices are aqueous/water, soil/sediment, wipe, and filter.
Matrix Spike -- Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology (also identified as a pre-distillation/digestion spike).
Method Detection Limit (MDL) -- The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

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Narrative (SDG Narrative) -- Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.
Office of Solid Waste and Emergency Response (OSWER) – The USEPA office that provides policy, guidance, and direction for the USEPA's solid waste and emergency response programs, including Superfund.
Percent Difference (%D) -- As used in this document and the Statement of Work (SOW), is used to compare two values. The difference between the two values divided by one of the values.
Performance Evaluation (PE) Sample -- A sample of known composition provided by USEPA for contractor analysis. Used by USEPA to evaluate Contractor performance.
Preparation Blank -- An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.
Relative Percent Difference (RPD) -- As used in this document and the Statement of Work (SOW) to compare two values, the RPD is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).
Regional Sample Control Center Coordinator (RSCC) -- In USEPA Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.
Relative Standard Deviation (RSD) -- As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.
Sample -- A single, discrete portion of material to be analyzed, which is contained in single or multiple containers and identified by a unique Sample Number.
Sample Delivery Group (SDG) -- A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
b. Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
c. Scheduled at the same level of deliverable.

In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG. Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory.
Sample Management Office (SMO) -- A contractor-operated facility operated under the SMO contract, awarded and administered by the USEPA. Provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).
Statement of Work (SOW) -- A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

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APPENDIX B: ORGANIC DATA EXECUTIVE NARRATIVE TEMPLATE

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
DESA/HWSB/HWSS
2890, Woodbridge Avenue, Edison, NJ 08837

EXECUTIVE NARRATIVE

Case No. :
Site:
Number of Samples:
Analysis:

SDG No.:
Laboratory:
Sampling dates:

QAPP
HWSS #:
Contractor Document #:

SUMMARY:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.
Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated.
Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

Major Findings:

Minor Findings:

COMMENT:

Reviewer Name(s):

Approver's Signature: Date:

Name:

Affiliation: USEPA/R2/HWSB/HWSS

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SAMPLE

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APPENDIX C: SAMPLE ORGANIC DATA SAMPLE SUMMARY

Case No:	00001	Contract:	XY1234	SDG No:	XY123	Lab Code:	00001
Sample Number:	XY123	Method:	BNA	Matrix:	Water	MA Number:	
Sample Location:	SOMEWHERE OUT THERE	pH:	8.4	Sample Date:	13322059	Sample Time:	10:09:00
% Moisture :				% Solids :			

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable	Validation Level
Benzaldehyde	5.0	ug/L	1.0	U	U	Yes	S3VEM
Phenol	5.0	ug/L	1.0	U	U	Yes	S3VEM
Bis(2-Chloroethyl)ether	5.0	ug/L	1.0	U	U	Yes	S3VEM
2-Chlorophenol	5.0	ug/L	1.0	U	U	Yes	S3VEM
2-Methylphenol	5.0	ug/L	1.0	U	U	Yes	S3VEM
2,2'-Oxybis(1-chloropropane)	5.0	ug/L	1.0	U	U	Yes	S3VEM
Acetophenone	5.0	ug/L	1.0	U	U	Yes	S3VEM
4-Methylphenol	5.0	ug/L	1.0	U	U	Yes	S3VEM
N-Nitroso-di-n-propylamine	5.0	ug/L	1.0	U	U	Yes	S3VEM
Hexachloroethane	5.0	ug/L	1.0	U	U	Yes	S3VEM
Nitrobenzene	5.0	ug/L	1.0	U	U	Yes	S3VEM
Isophorone	5.0	ug/L	1.0	U	U	Yes	S3VEM
2-Nitrophenol	5.0	ug/L	1.0	U	U	Yes	S3VEM
2,4-Dimethylphenol	5.0	ug/L	1.0	U	U	Yes	S3VEM
Bis(2-chloroethoxy)methane	5.0	ug/L	1.0	U	U	Yes	S3VEM
2,4-Dichlorophenol	5.0	ug/L	1.0	U	U	Yes	S3VEM
Naphthalene	5.0	ug/L	1.0	U	U	Yes	S3VEM
4-Chloroaniline	5.0	ug/L	1.0	U	U	Yes	S3VEM
Hexachlorobutadiene	5.0	ug/L	1.0	U	U	Yes	S3VEM
Caprolactam	5.0	ug/L	1.0	U	U	Yes	S3VEM
4-Chloro-3-methylphenol	5.0	ug/L	1.0	U	U	Yes	S3VEM
2-Methylnaphthalene	5.0	ug/L	1.0	U	U	Yes	S3VEM
Hexachlorocyclopentadiene	5.0	ug/L	1.0	U	U	Yes	S3VEM
2,4,6-Trichlorophenol	5.0	ug/L	1.0	U	U	Yes	S3VEM
2,4,5-Trichlorophenol	5.0	ug/L	1.0	U	U	Yes	S3VEM
1,1'-Biphenyl	5.0	ug/L	1.0	U	U	Yes	S3VEM
2-Chloronaphthalene	5.0	ug/L	1.0	U	U	Yes	S3VEM

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APPENDIX D: ELECTRONIC DATA DELIVERABLE TEMPLATE

DATA_PROVIDER	LAB_MATRIX_CODE	RESULT_UNIT
SYS_SAMPLE_CODE	ANAL_LOCATION	DETECTION_LIMIT_UNIT
SAMPLE_NAME	BASIS	TIC_RETENTION_TIME
SAMPLE_MATRIX_CODE	CONTAINER_ID	RESULT_COMMENT
SAMPLE_TYPE_CODE	DILUTION_FACTOR	QC_ORIGINAL_CONC
SAMPLE_SOURCE	PREP_METHOD	QC_SPIKE_ADDED
PARENT_SAMPLE_CODE	PREP_DATE	QC_SPIKE_MEASURED
SAMPLE_DEL_GROUP	LEACHATE_METHOD	QC_SPIKE_RECOVERY
SAMPLE_DATE	LEACHATE_DATE	QC_DUP_ORIGINAL_CONC
SYS_LOC_CODE	LAB_NAME_CODE	QC_DUP_SPIKE_ADDED
START_DEPTH	QC_LEVEL	QC_DUP_SPIKE_MEASURED
END_DEPTH	LAB_SAMPLE_ID	QC_DUP_SPIKE_RECOVERY
DEPTH_UNIT	PERCENT_MOISTURE	QC_RPD
CHAIN_OF_CUSTODY	SUBSAMPLE_AMOUNT	QC_SPIKE_LCL
SENT_TO_LAB_DATE	SUBSAMPLE_AMOUNT_UNIT	QC_SPIKE_UCL
SAMPLE_RECEIPT_DATE	ANALYST_NAME	QC_RPD_CL
SAMPLER	INSTRUMENT_ID	QC_SPIKE_STATUS
SAMPLING_COMPANY_CODE	COMMENT	QC_DUP_SPIKE_STATUS
SAMPLING_REASON	PRESERVATIVE	QC_RPD_STATUS
SAMPLING_TECHNIQUE	FINAL_VOLUME	BREAK_2
TASK_CODE	FINAL_VOLUME_UNIT	SYS_SAMPLE_CODE
COLLECTION_QUARTER	CAS_RN	LAB_ANL_METHOD_NAME
COMPOSITE_YN	CHEMICAL_NAME	ANALYSIS_DATE
COMPOSITE_DESC	RESULT_VALUE	TOTAL_OR_DISSOLVED
SAMPLE_CLASS	RESULT_ERROR_DELTA	COLUMN_NUMBER
CUSTOM_FIELD_1	RESULT_TYPE_CODE	TEST_TYPE
CUSTOM_FIELD_2	REPORTABLE_RESULT	TEST_BATCH_TYPE
CUSTOM_FIELD_3	DETECT_FLAG	TEST_BATCH_ID
COMMENT	LAB_QUALIFIERS	CASE
BREAK_1	VALIDATOR_QUALIFIERS	CONTRACT_NUM
SYS_SAMPLE_CODE	INTERPRETED_QUALIFIERS	SCRIBE_SAMPLE_ID
LAB_ANL_METHOD_NAME	ORGANIC_YN	SAMPLE_TIME
ANALYSIS_DATE	METHOD_DETECTION_LIMIT	FRACTION
TOTAL_OR_DISSOLVED	REPORTING_DETECTION_LIMIT	PH
COLUMN_NUMBER	QUANTITATION_LIMIT	DATA_VAL_LABEL
TEST_TYPE		

FOIA_07123_0003552_0681

EPA

Hazardous Waste Support Branch

Request for Standard Operating Procedure (SOP) Change

Information

Initiator Name: Elizabeth Melenbrink Date of Initiation: 8 May 2013

Department Name: HWSS SOP Number: HW-35 Revision Number: 2

SOP Title: Semivolatile Data Validation

☐ Major Revision ☒ Minor Revision

Change(s) (Use attachment if necessary):

Add to the section **HWSS DATA VALIDATION PROCESS**:

All data is initially marked as "reportable" (Y) in EDM before validation is begun. Sometimes, due to dilutions, re-analyses, or SIM/scan runs all being performed, there will be multiple results for a single analyte from a single sample. The following criteria and professional judgment are used to determine which result should be reported:

The analysis with the lower CRQL
The analysis with the better QC results
The analysis with the higher result

The analyte values and their respective CRQLs are then transferred into a single sample run. The runs that are not to be used are updated as "not reportable" or (N) in EDM.

Reason(s) for change(s):

These circumstances were not addressed in this version of the SOP.

Approval

Philip Cocuzzo	Philip Cocuzzo	5/14/13
Section Chief/Team Leader	*Signature*	*Date*
Jon Gabry	[illegible]	5/13/13
HWSB Branch Chief	*Signature*	*Date*

Effective Date

Date: 5/20/13

EPA

Hazardous Waste Support Branch

Request for Standard Operating Procedure (SOP) Change

Information

Initiator Name: Elizabeth Melenbrink Date of Initiation: 8 May 2013

Department Name: HWSS SOP Number: HW-35 Revision Number: 2

SOP Title: Semivolatile Data Validation

☐ Major Revision ☒ Minor Revision

Change(s) (Use attachment if necessary):

Add to the section **Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)**:

Results between the MDL and CRQL should be qualified as estimated (J).
Results less than the MDL should be reported at the CRQL and qualified (U). MDLs themselves are not reported.

Reason(s) for change(s):

These circumstances were not addressed in this version of the SOP.

Approval

Philip Cuccuzza	Philip Cuccuzza	5/14/13
Section Chief/Team Leader	*Signature*	*Date*
Jon Gabry	[illegible]	5/13/13
HWSB Branch Chief	*Signature*	*Date*

Effective Date

Date: 5/20/13

VALIDATION SOP

Aroclor PCBs
USEPA Region 2 SOP HW-37
Rev. 1

SOP NO. HW-37/Aroclor
Validation of Data
USEPA Contract Laboratory Program
Statement of Work for Organic Analysis of Low/Medium Concentration of Aroclor Organic Compounds SOM01.2

Prepared by: George Karras Date: 8/13/07
George Karras, Chemist
Hazardous Waste Support Section

Peer Reviewed by: Russell Arnone Date: 10/3/07
Russell Arnone, Chemist
Hazardous Waste Support Section

Concurred by: Linda M. Mauel Date: 10/9/07
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: Robert Runyon Date: 10/10/07
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the method in the "USEPA Contract Laboratory Program Statement of Work for Organics Analysis Multi-Media, Multi-Concentration, SOM01.1, May 2005". The validation procedures and actions discussed in this document are based on the requirements set forth in the "USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, January 2005". This document attempts to cover technical problems specific to low/Medium concentration of Aroclor compounds. Situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

In addition to technical requirements, contractual requirements may also be covered in this document. While it is important that instances of contract non-compliance be addressed in the Data Assessment, the technical criteria are always used to qualify the analytical data.

Summary

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are as follows:

Data Qualifiers

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

JN - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate

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concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Lab Qualifiers:

D - The positive value is the result of an analysis at a secondary dilution factor.

B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.

E - The concentration of this analyte exceeds the calibration range of the instrument.

P - Aroclor target analytes when the % Difference between the analyte concentrations obtained from the two dissimilar GC columns is greater than 25%.

C - This flag applies to Aroclors results when the identification has been confirmed by GC/MS analysis.

S - Single point calibration.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance.

Reviewer Qualifications:

Data reviewers must possess a working knowledge of the USEPA Statement of Work SOM01.2 and National Functional Guidelines mentioned above.

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YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:____________________ **LAB:**______________________________

SITE NAME:______________________ **SDG No(s).:**________________________

1.0 Chain of Custody and Sampling Trip Reports

1.1 Are the Traffic Reports/Chain-of-Custody Records present for all samples? [ ] ___ ___

ACTION: If no, contact RSCC, or the TOPO to obtain replacement of missing or illegible copies from the lab.

1.2 Is the Sampling Trip Report present for all samples? [ ] ___ ___

ACTION: If no, contact either RSCC or ask the TOPO to obtain the necessary information from the prime contractor.

2.0 Data Completeness and Deliverables

2.1 Have any missing deliverables been received and added to the data package? ___ [ ] ___

ACTION: Contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the lab. If lab cannot provide them, note the effect on the review of the data package in the Contract

YES NO N/A

Problems/Non-compliance section of the Data Assessment.

2.2 Was SMO/CLASS CCS checklist included with the package? [ ] ___ ___

2.3 Are there any discrepancies between the Traffic Reports/Chain-of-Custody Records, and Sampling Trip Report? ___ [ ] ___

ACTION: If yes, contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the laboratory.

3.0 Cover Letter SDG Narrative

3.1 Is the SDG Narrative or Cover Letter Present? [ ] ___ ___

3.2 Are case number, SDG number and contract number contained in the SDG Narrative or cover letter (see SOW, Exhibit B, section 2.5.1)? EPA sample numbers in the SDG, detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing the samples? Corrective action taken? [ ] ___ ___

3.3 Does the Narrative contain the following information SOM01.1, page B-12, section 2.5.1)? column used, storage of samples, case#, SDG#, analytical problems, and discrepancies between field and lab weights. [ ] ___ ___

3.5 Did the contractor record the temperature of the cooler on the Form DC-1, Item 9 - Cooler Temperature, and in the SDG Narrative? [ ] ___ ___

3.6 Does the Case Narrative contain the "verbatim" statement (page B-12, section 2.5.1 of the SOM)? [ ] ___ ___

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	YES	NO	N/A

ACTION: If "No", to any question in this section, contact the TOPO to obtain necessary resubmittals. If unavailable, document under the Contract Problems/ Non-Compliance section of the Data Assessment.

4.0 Data Validation Checklist

4.1 Check the package for the following (see SOM reporting requirements, section 2.1, page B-10):

	YES	NO	N/A
a. Is the package paginated in ascending order starting from the SDG narrative?	[ ]	___	___
b. Are all forms and copies legible?	[ ]	___	___
c. Assembled in the order set forth in the SOW?	[ ]	___	___
d. All Aroclor Data present?	[ ]	___	

PART A: Low/Medium Aroclor Analyses

1.0 Sample Conditions/Problems

	YES	NO	N/A
1.1 Do the Traffic Reports/Chain-of-Custody Records, Sampling Trip Report or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?	___	[ ]	___

ACTION: If samples were not iced or the ice was melted upon arrival at the laboratory and the temperature of the cooler was > 10° C, then flag all positive results with a "J" and all non-detects "UJ".

2.0 Holding Times

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YES NO N/A

2.1 Have any Aroclor technical holding times, determined from date of collection to date of analysis, been exceeded? ___ [ ] ___

2.2 Preservation: Aqueous and Non-aqueous samples must be cooled at 4°C ± 2°C.

ACTION: Qualify sample results according to the following table.

Holding Time Actions for Low/Medium Aroclor Analyses

Matrix	**Preserved**	**Criteria**	**Action**	
			Detected Associated Compounds	**Non-Detected Associated Compounds**
Aqueous	No	≤ 7 days (extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 7 days (extraction) > 40 days (analysis)	J	UJ
	Yes	≤ 7 days (extraction) ≤ 40 days (analysis)	No qualification	
	Yes	> 7 days (extraction) > 40 days (analysis)	J	UJ
	Yes/No	> 28 Days (extraction)	J	R
Non-aqueous	No	≤ 14 days (extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes	≤ 14 days (extraction) ≤ 40 days (analysis)	No qualification	
	Yes	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes/No	> 28 Days (extraction)	J	R

* Only if cooler temperature exceeds 10°C (see ACTION in Section 1.1 above). No action required if temperature ≤ 10°C.

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YES NO N/A

3.0 Surrogate Recovery (Form II ARO-1, Form II ARO-2, Form VIII ARO)

3.1 Are the Aroclor Recovery Summary Forms present? [ ] ___ ___

ACTION: Contact the TOPO to obtain an explanation/resubmittal from the lab. If missing deliverables are unavailable, document the effect in the Data Assessment.

3.2 Were the two surrogates, tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) added to all samples, MS/MSD, LCS, blanks including standards? [ ] ___ ___

ACTION: If no, use professional judgment in qualifying data as missing surrogate analyte may not directly apply to target analytes.

3.3 Were outliers marked with an asterisk on Form II? [ ] ___ ___

ACTION: Circle all outliers with a red pencil.

If yes, were effected samples re-analyzed? [ ] ___ ___

3.4 The RTs of the surrogates in each mid-point Aroclor standards used for continuing calibration verification, all samples, including MS/MSD, LCS and all blanks must be within the calculated RT window. TCX must be within ± 0.05 minutes and DCB must be within ± 0.10 minutes of the mean retention time (RT) determined from the initial calibration and tabulated in Form VIII Pest.

Were any outliers marked with an asterisk on Form VIII ARO? ___ [ ] ___

ACTION: Circle all outliers with a red pencil. If any Surrogate is outside the required limits, qualify their associated target compounds (See Table below) as follows:

Surrogate Compound Recovery Action for Aroclors

Criteria	Action	
	Detected Target Compounds	**Non-Detected Target Compounds**
%R > 200%	J	No qualification
150% < %R ≤ 200%	J	No qualification
30% ≤ %R ≤ 150%	No qualification	
10% ≤ %R < 30%	J	UJ

YES NO N/A

%R < 10% (sample dilution not a factor)	J	R
%R < 10% (sample dilution is a factor)	J	Use Professional Judgement
RT out of RT window	Use professional judgment	
RT within RT window	No qualification	

Note: Blank analysis having surrogates out of specification:

The reviewer must give special consideration to the validity of associated samples. Basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence.

ACTION: Note in the Data Assessment under Contract Problems/ Non-Compliance if the Lab did not perform reanalysis and reviewer's judgment regarding blank problem.

3.5 Are there any transcription/calculation errors between raw data and Form IIs? ___ [ ] ___

ACTION: If large errors exist, ask the TOPO to obtain an explanation/resubmittal from the lab, make any necessary corrections and note errors in the data assessment.

Note: Surrogate recovery limits criteria and qualification apply to samples diluted 5X and less. For samples diluted greater than 5X, recovery criteria does not apply Because it is assumed surrogate is diluted below the quantitation range.

4.0 Matrix Spike/Matrix Spike Duplicate Recovery (Form III)

Note: Data for MS/MSD will not be present unless requested.

4.1 Are the MS/MSD Recovery Forms (Form III ARO) present? [ ] ___ ___

4.2 Was the MS/MSD analyzed at the required frequency (once per SDG, or every 20 samples, whichever is more frequent)? [ ] ___ ___

ACTION: If any MS/MSD data are missing, take action as specified in section 3.1 above.

ACTION: No action is taken on MS/MSD data alone. However, using professional judgement, the validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. If Any MS/MSD % recovery or RPD is out of specification, qualify data to include the consideration of the existence of interference in the raw

data. Consideration include, but not limited to the following "Action":

Matrix Spike/Matrix Spike Duplicate Action for Aroclor

Criteria	Action	
	Detected Spike Compounds	Non-detected Spike Compounds
%R or RPD > Upper Acceptance Limit	J	No qualification
20% ≤ %R < Lower Acceptance Limit	J	UJ
%R < 20%	Use professional judgment	
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualification	

Note: If it can be determined that the results of the MS/MSD affects only the sample spiked, limit qualification to only this sample. However, use professional judgment when it is determined through the MS/MSD results that the laboratory is having systematic problem in the analysis of one or more analytes that affect all associated samples.

5.0 Blanks (Form IV)

5.1 Is the Aroclor Method Blank Summary (Form IV ARO) present for aqueous and soil samples? [ ] ___ ___

5.2 Frequency of Analysis: For the analysis of AROCLOR, has a method blank been analyzed for each SDG or every 20 samples, whichever is more frequent? [ ] ___ ___

ACTION: If any blank data are missing, take action as specified above in section 3.1. If blank data is not available, reject "R" all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

5.3 A separate Form IV should be present if part of an extraction batch required sulfur removal. In such cases some samples will be listed on two blank summary forms - once under the method blank, and once under the sulfur clean-up blank (PCBLK). Was this additional blank raw data and Form IV submitted when required? [ ] ___ ___

ACTION: If Form IV sulfur clean-up blank is missing, take action as specified in section 3.1 above.

		YES	NO	N/A
5.4	Has a Aroclor instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence (minimum contract requirement)?	[ ]	___	___

ACTION: If any blank data are missing, take action specified in Section 3.1.

5.5	Was the correct identification scheme used for all Aroclor blanks? (See page B-39, section 3.3.7.3 of SOM01.1 for further information)	[ ]	___	___

ACTION: Contact the TOPO to obtain resubmittals or make the required corrections on the forms. Document in the Data Assessment under Contract Problems/Non-Compliance all corrections made by the validator.

5.6	<u>Chromatography</u>: Review the blank raw data chromatogram, quant. Reports and data system printout. Is the chromatographic performance (baseline stability) acceptable for each instrument?	[ ]	___	___

ACTION: Use professional judgement to determine the effect on the data.

5.7	Are all detected hits for target compounds in method, and field blanks less than the CRQL?	[ ]	___	___

ACTION: IF no, an explanation and laboratory's corrective actions must be addressed in the case SDG narrative. Contact TOPO to request from Lab. revised narrative and make a note in the Contract Problems/Non-Compliance section of the Data Assessment.

6.0 <u>Contamination</u>

NOTE: "Water blanks", "drill blanks", and distilled water blanks" are validated like any other sample, and are <u>not</u> used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1	Do any method/reagent or cleanup blanks contain positive hits for target Aroclor compounds with values greater than the CRQL for that analyte?	___	[ ]	___

Note: The concentration of each target compound in the instrument blank must be less than the CRQL for that analyte.

ACTION: Make note in data assessment under Contract Problems/Non-Compliance if any blank contains hit above the CRQLs.

6.2	Do any instrument blanks contain positive Aroclor results with values greater than CRQLs?	___	[ ]	___

YES NO N/A

ACTION: Take the action specified in section 6.1.

6.3 Do any field/rinse blanks have positive Aroclor results? ___ [ ] ___

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems.

ACTION: Follow the directions in the table below to qualify results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated, all associated sample data should be qualified unusable (R).

Blank Action for Aroclor Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field, Sulfur Cleanup, Instrument	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report concentration of sample with a U
		≥ CRQL and $ blank contamination	No qualification required
	Gross contamination	Detects	Qualify results as unusable R

NOTE: Analytes qualified "U" for blank contamination are treated as "hits" when qualifying for calibration criteria.
Note: When applied as described in the table above, the contaminant concentration in the blank are multiplied by the sample dilution factor.

6.4 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

YES NO N/A

ACTION: Note in data assessment if there's no associated field/rinse/equipment blank.

Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 Aroclor Initial and Continuing Calibration

7.1 Are the following Forms, chromatograms and data system printouts present?

a.) Form VI ARO-1/Aroclor Initial Calibration (Multipoint) [ ] ___ ___

b.) Form VI ARO-2/Aroclor Initial Calibration (Multipoint) [ ] ___ ___

c.) Form VI ARO-3/Aroclor Initial Calibration(Singlepoint) [ ] ___ ___

d.) Form VII ARO/Aroclor Calibration Verification [ ] ___ ___

e.) Form VIII ARO/Aroclor Analytical Sequence [ ] ___ ___

f.) Form X ARO/Identification Summary for Multicomponent Analysis [ ] ___ ___

7.2 **Initial Calibration**

7.2.1 Was the following contract required initial calibration sequence provided by the laboratory? [ ] ___ ___

Initial Calibration Sequence	
1.	Aroclor 1221 CS3 (400ng/ml)
2.	Aroclor 1232 CS3 (400 ng/ml)
3.	Aroclor 1242 CS3 (400 ng/ml)
4.	Aroclor 1248 CS3 (400 ng/ml)
5.	Aroclor 1254 CS3 (400 ng/ml)
6.	Aroclor 1262 CS3 (400 ng/ml)
7.	Aroclor 1268 CS3 (400 ng/ml)
8.	Aroclor1016/1260 (100 ng/ml) CS1
9.	Aroclor1016/1260 (200 ng/ml) CS1
10.	Aroclor1016/1260 (400 ng/ml) CS1
11.	Aroclor1016/1260 (800 ng/ml) CS1

12.	Aroclor1016/1260 (1600 ng/ml) CS1
13.	Instrument Blank

ACTION: If initial calibration is not performed or not performed in the proper sequence, notify the TOPO and make a note in the data assessment.

7.3 Are there any transcription/calculation errors between raw data and the Forms? ___ [ ] ___

ACTION: If large errors exist, take action specified in section 3.1 above.

7.4 Mean Retention Time (RT) and RT Window

Were the following mean RT and RT window met: [ ] ___ ___

a.) The mean RT of each of the three to five major peaks were determined from the five-point initial calibration for all Aroclors

b.) RT window was calculated as ± 0.07 for each of the three to five major peaks and ± 0.05 and ± 0.10 for the surrogates tetrachloro-m-xylene and decachlorobiphenyl, respectively.

ACTION: If no, follow the action as specified in section 3.1.

7.5 Was at least one chromatogram from each of the Aroclor standards yield peaks that give deflection between 50-100% of full scale? [ ] ___ ___

ACTION: IF no, take action as specified in section 3.1.

7.6 Was the mean Calibration Factor (CF) calculated for the three to five major peaks of each Aroclor, as well as for the surrogates, over the initial calibration range? [ ] ___ ___

7.7 Were the Percent Relative Standard Deviation (%RSD) of the Calibration Factor for the three to five major peaks < 20% of each of the Aroclor compounds and surrogates? [ ] ___ ___

ACTION: If no, take action as specified in the following Table.

Initial Calibration Action for Aroclor Analyses

Criteria	Action

YES NO N/A

	Detected Associated Compounds	Non-Detected Associated Compounds
Initial calibration is not performed or not performed in proper sequence	Use Professional Judgment and notify Contract Lab Program (CLP) Project Officer	
%RSD exceeds allowable limits *	J	UJ
%RSD within allowable limits *	No qualification	

* %RSD < 20.0% for Aroclors and surrogates (tetrachloro-m-xylene and decachlorobiphenyl.

7.8 **Continuing Calibration Verification (CCV) (Form VII)**

Were the Absolute Retention Time (RT) for each Aroclor and surrogate in the mid-point concentration (CS3) of the Standard used for CCV must be within the RT window determined from the initial calibration?

7.9 For opening CCV, or closing CCV that is used as an opening CCV for the next 12-hour period, the Percent Difference (%D) between the CF of each of the three to five peaks used to identify an Aroclor and surrogates in the mid-point concentration (CS3) of the Aroclor standards and the CF from the initial calibration must be within ±15.0%.

7.10 For a closing CCV, the %D between the CF of each of the three to five peaks used to identify an Aroclor and surrogates in the mid-point concentration (CS3) of the Aroclor standards and the CF from the initial calibration must be within ±50.0%.

7.11 No more than 14 hours may elapse from the injection of the instrument Blank that begins an analytical sequence (opening CCV) and the injection of the last mid-point concentration (CS3) of the Aroclor standards that ends an analytical sequence (closing CCV).

7.12 No more than 12 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV and the injection of the last sample or blank that is part of the same analytical sequence.

Were sections 7.8 to 7.12 met? [ ] ___ ___

ACTION: If no, use the following table to qualify Aroclor data:

Continuing Calibration Verification (CCV) Action for Aroclor Analyses

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Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
RT out of RT Window	Use professional Judgment *	
Percent Difference not within limits ± 15% as specified in section 7.9 above	J	UJ
Percent Difference not within limits ± 50% as specified in section 7.10 above	J	UJ
Time elapsed is greater than acceptable limits as specified in section 7.11 & 7.12 above	R	
Percent Difference, time elapsed and RT are within acceptable limits	No qualification	

* For <u>non-detected</u> target compounds in the affected samples, check to see if the sample chromatogram contain any peak that are close to the expected RT window of the Aroclor of interest.

If no peaks are present, consider the non-detected values to be valid and no qualification of the data is necessary.

If any peaks are present close to the expected RT window of the Aroclor of interest, qualify the non-detected values as presumptively present "N".

For <u>detected compounds</u> in the affected samples, if the peaks are within the RT window, no qualification of the data is necessary. If the peaks are close to the expected RT window of the Aroclors of interest, the reviewer may take additional effort to determine if sample peaks represent the compound of interest.

For example, the reviewer can examine the data package for the presence of three or more standards containing the Aroclor of interest that were run within the analytical sequence during which the sample was analyzed. If three or more such standards are present, the RT window can be re-evaluated using the mean RT of the standards.

If the peaks in the affected sample fall within the revised window, qualify the detected Aroclor as "JN".

If the reviewer cannot do anything with the data to resolve the problem of concern, qualify all non-detects as unuseable "R".

8.0 <u>Analytical Sequence Check (Form VIII-ARO)</u>

	YES	NO	N/A
8.1 Is Form VIII-Pest present and complete for each column and each period of analyses?	[ ]	___	___

ACTION: If no, take action as specified in section 3.1

	YES	NO	N/A
8.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses, and all standards analyzed at the required frequency for each GC/ECD instrument used?	[ ]	___	___

ACTION: If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly. Generally, the effect is negligible unless the sequence was grossly altered and/or the calibration was out of QC limits.

	YES	NO	N/A
8.3 Are the surrogate retention time (RT) from the initial calibration for TCX and DCB provided on Form VIII-Pest?	[ ]	___	___

ACTION: If no, take action as specified in section 3.1

	YES	NO	N/A
8.4 Was the asterisk (*) applied to the RT of any blanks, samples, standards, MS/MSD, and LCS that did not meet the QC Limits of ± 0.05 minutes for TCX (tetrachloro-m-xylene) and ± 0.10 minutes for DCB (decachlorobiphenyl)?	[ ]	___	___

ACTION: If any data are missing, take action specified in 3.1 above.

If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly. Document in the data assessment under Contract Problems/Non-Compliance.

9.0 **<u>Sulfuric Acid and Gel Permeation Chromatography (GPC) Cleanup Procedures</u>**

	YES	NO	N/A
9.1 Was sulfuric acid added to all extracts?	[ ]	___	___

Note: Sulfuric acid cleanup is <u>mandatory</u> for <u>all</u> extracts

ACTION: If no, take action specified in section 3.1

9.2 <u>Gel Permeation Chromatography (GPC</u>

GPC is an optional cleanup procedure for both aqueous and non-aqueous samples that contain high molecular weight compounds that interfere with Aroclor analysis.

YES NO N/A

9.3 If GPC cleanup was performed on samples, GPC calibration is acceptable if the two UV traces meet the following requirements.

a. Peaks must be observed and should be symmetrical for all compounds in the calibration solution.

b. Corn oil and phthalate peaks should exhibit greater than 85% resolution.

c. The phthalate and Methoxychlor peaks should exhibit greater than 85% resolution.

d. Methoxychlor and perylene peaks should exhibit greater than 85% resolution.

e. Perylene and sulfur peaks must be saturated and should exhibit greater than 90% baseline resolution.

f. The RT shift is less than 5% between UV traces for bis(2-ethylhexylphthalate and perylene.

9.4 Were all above criteria met? [ ] ___ ___

ACTION: If no, examine the raw data for the presence of high molecular weight contaminants. Examine the subsequent sample data for unusual peaks and use professional judgment in qualifying the data.

10.0 Laboratory Control Samples (LCSs)

10.1 LCSs provide information on the accuracy of the analytical method and laboratory performance.

Aroclor Laboratory Control Sample Recovery - Aqueous and Non-Aqueous

Compound	% Recovery QC Limits
Aroclor 1016	50 - 150
Aroclor 1260	50 - 150
Tetrachloro-m-xylene (surrogate)	30 - 150
Decachlorobiphenyl (surrogate)	30 - 150

10.2 Were the above recoveries met? [ ] ___ ___

ACTION: If no, qualify the sample data as follows:

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Criteria	**ACTION**	
	Detected Associated Compound	**Non-Detected Associated Compound**
%R> Upper Acceptance Limit	J	No qualification
%R< Lower Acceptance Limit	J	R
Lower Acceptance Limit < %R < Upper Acceptance Limit	No qualification	

11.0 <u>Aroclor Identification (Form X ARO/Identification Summary for Multicomponent Analysis</u>

11.1 Is Form X (ARO) complete for every sample in which Aroclor was detected? [ ] ___ ___

ACTION: Take action as specified in section 3.1 above.

11.2 The identification of a Multi component Aroclor by GC method is based primarily on RT data and pattern recognition. Were the following requirements met: [ ] ___ ___

a.) A Minimum of 3 major peaks were selected for each Aroclor. If more than one Aroclor is observed in a sample, a peak common to other Aroclor(s) must not be used to quantitate other Aroclor. Lab must choose different peaks to quantitate each Aroclor.

b.) If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

c.) The Retention Time (RT) of both of the surrogates and reported target compounds must be within the calculated RT window of both columns.

STANDARD OPERATING PROCEDURE . . . . .

USEPA Region II Date: August 2007
Method: CLP/SOW, SOM01.2/Aroclor SOP HW-37/Aroclor, Revision 1
S))))))

STANDARD OPERATING PROCEDURE . . . . .

USEPA Region II Date: August 2007
Method: CLP/SOW, SOM01.2/Aroclor SOP HW-37/Aroclor, Revision 1
S))))))

ACTION: If errors exist, take action as specified in section 3.1 above.

ACTION: When a sample is required to be diluted, the lowest CRQL is used (unless a QC exceedance dictates the use of the higher CRQL from the diluted sample). Replace concentration which exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with the result from the diluted sample. Specify which Form I to use. Use a red pencil and draw a red "X" across the entire page of all Form I's that should not be used, including those in the data summary package.

At the top or bottom of the Forms, write with red pencil, "DO Not Use".

Note: If the sample dilution factor (DF) is greater than 10, an additional 10 times more <u>concentrated</u> than the diluted sample extract must be analyzed and reported with the sample data. If the DF is less or equal to 10, but greater than 1, the results of the original undiluted analysis must also be reported (see SOM01.1/section 10.3.3.4/page D-44/ARO).

ACTION: IF the above requirement was not met, contact the TOPO to obtain an explanation/resubmittal from the lab and make a note in the Data Assessment under Contract Problems/Non-Compliance section.

13.3 For non-aqueous samples, were the percent moisture < 70%? [ ] ___ ___

Action: If the % moisture ≥ 70.0% and < 90.0%, qualify detects as "J" and non-detects as approximated "UJ" If the % Moisture ≥ 90%, qualify detects as "J" and non-detects as "R"

14.0 <u>Field Duplicates</u>

14.1 Were any field duplicates submitted for Aroclor analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between duplicate results must be addressed in the reviewer narrative. If large differences exist,

contact the TOPO to confirm identification of field duplicates with the sampler.

Definitions

ARO - Aroclor
CCS - contract compliance screening
CF - Calibration Factor
CLASS - Contract Laboratory Analytical Services Support
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
GC/ECD - Gas Chromatography/Electron Capture Detector
kg - kilogram
:g - microgram
R - liter
mR - milliliter
QC - quality control
RAS - Routine Analytical Services
RPD - Relative Percent Difference
RRF - Relative Response Factor
RRF - Average Relative Response Factor (from initial calibration)
RRT - Relative Retention Time
RSD - Relative Standard Deviation
RT - Retention Time
RSCC - Regional Sample Control Center
SDG - Sample Delivery Group
SOP - standard operating procedure
SOW - Statement of Work
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TIC - Tentatively Identified Compound
TPO - Technical Project Officer
VTSR - Validated Time of Sample Receipt
TOPO - Task Order Project Officer

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References

1. USEPA Contract Laboratory Program of Work for Organic Analysis Multi-Media, Multi-Concentration, SOW/CLP/SOM01.1, October 2004
2. National Functional Guidelines for Superfund Organic Methods Data Review January 2005

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VALIDATION SOP

Pesticides

USEPA Region 2 SOP HW-36, Rev. 4, 5/13

Hazardous Waste Support Section
SOP NO. HW-36 Revision 4
Pesticide Data Validation

Approvals:

Russell Arnone — 05/14/13
Chemist, Hazardous Waste Support Section — Date

Philip Cocuzza — 5/14/13
Chief, Hazardous Waste Support Section — Date

Jon Gabry — 5/13/13
Chief, Hazardous Waste Support Branch — Date

Approving Official Annual Review — Date

NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (hereafter referred to as USEPA) and other governmental employees. They do not constitute rule making by USEPA, and may not be relied upon to create a substantive or procedural right enforceable by any other person. The Government may take action that is at variance with the policies and procedures in this manual.

The guidance for data validation set forth in the quality assurance project plan (QAPP) for the project associated with the data in question will always take precedence over the data validation guidance listed herein.

Validators should note that their professional judgment supersedes any guidance listed in this document.

Government contractors to the USEPA using this document to validate data should not hesitate to contact their Contracting Officer Representative with any questions regarding data validation or data package completeness.

This document can be obtained from the USEPA's Region 2 Quality Assurance website at:

http://www.epa.gov/region2/qa/documents.htm

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ACRONYMS

%D	Percent Difference
%RSD	Percent Relative Standard Deviation
ARO	Aroclor
ASB	Analytical Services Branch
BFB	Bromofluorobenzene
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CF	Calibration Factor
CLP	Contract Laboratory Program
CLP PO	Contract Laboratory Program Project Officer
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
DART	Data Assessment Rapid Transmittal
DAT	Data Assessment Tool
DCB	Decachlorobiphenyl
DFTPP	Decafluorotriphenylphosphine
DMC	Deuterated Monitoring Compound
DQA	Data Quality Assessment
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EDM	EXES Data Manager
ESAT	Environmental Services Assistance Team
EXES	Electronic Data eXchange and Evaluation System
GC	Gas Chromatograph
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPC	Gel Permeation Chromatography
HWSS	Hazardous Waste Support Section
INDA	Individual Standard Mixture A
INDB	Individual Standard Mixture B
INDC	Individual Standard Mixture C
LCS	Laboratory Control Sample
MS	Matrix Spike
MSD	Matrix Spike Duplicate
OSRTI	Office of Superfund Remediation and Technology Innovation
PCBs	Polychlorinated Biphenyls
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QC	Quality Control
RAS	Routine Analytical Services

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RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference
RRF	Relative Response Factor
$\overline{RRF}$	Mean Relative Response Factor
RRT	Relative Retention Time
RSCC	Regional Sample Control Center Coordinator
RSD	Relative Standard Deviation
RT	Retention Time
SAP	Sampling and Analysis Plan
SCP	Single Component Pesticide
SDG	Sample Delivery Group
SIM	Selected Ion Monitoring
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TCL	Target Compound List
TCLP	Toxicity Characteristics Leachate Procedure
TCX	Tetrachloro-m-xylene
TIC	Tentatively Identified Compound
TOPO	Task Order Project Officer
TR/COC	Traffic Report/Chain of Custody Record
USEPA	United States Environmental Protection Agency
UV	Ultraviolet
VTSR	Validated Time of Sample Receipt

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INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the validity of analytical data generated through the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Multi-Media, Multi-Concentration Organics Analysis (SOM01.2), and any future editorial revisions of SOM01.2, hereinafter referred to as the SOM01.2 SOW. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process.

The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and QC criteria established in the SOW, and (b) the validity and extent of bias of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to site-specific technical requirements, the need to facilitate the progress of specific projects, and availability for re-sampling.

The reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as **professional judgment**, should also be used to determine the ultimate validity of data, especially in those cases where all data does not meet specific technical criteria.

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DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
C	This qualifier applies to results when the identification has been confirmed by Gas Chromatograph/Mass Spectrometer (GC/MS)
X	This qualifier applies to results when GC/MS analysis was attempted but unsuccessful

DATA PACKAGE INSPECTION

For data obtained through the Contract Laboratory Program (CLP), the EXES Data Manager (EDM) is a useful tool in the data review process. For more information about EDM, please refer to the following Sample Management Office (SMO) website:

https://epasmoweb.fedcsc.com/help/guides/Submit%20and%20Inspect%20Data%20Quick%20Guide%20%28EXES%29.pdf

EDM will identify any missing and/or incorrect information in the data package. The CLP laboratory may submit a reconciliation package for any missing items or to correct data.
If there are any concerns regarding the data package, contact the CLP Project Officer (CLP PO) from the Region where the samples were taken. For personnel contact information, please refer to the following CLP website:

http://www.epa.gov/superfund/programs/clp/contacts.htm

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HWSS DATA VALIDATION PROCESS

After downloading the data package from EDM, the data validator will use the recommendations in this SOP as well as their own professional judgment to validate the data.

All data is initially marked as "reportable" (Y) in EDM before validation is begun. Sometimes, due to dilutions, re-analyses, or SIM/scan runs all being performed, there will be multiple results for a single analyte from a single sample. The following criteria and professional judgment are used to determine which result should be reported:

The analysis with the lower CRQL
The analysis with the better QC results
The analysis with the higher result

The analyte values and their respective CRQLs are then transferred into a single sample run. The runs that are not to be used are updated as "not reportable" or (N) in EDM.

The data will be saved in the following location, under the appropriate case number folder:

G:\DESADIV\HWSS\DATA VALIDATION

The file naming conventions will consist of

A. case number i.e., 12345
B. SDG name i.e., BXY12
C. level of validation performed i.e., S3VE

Examples: **12345_BXY12_S3VE.xls**

12345_BXY12_S3VEM.xls

When data validation is completed, the data package is uploaded for the client to download from the HWSS data delivery website:

https://epaqpx.rtp.epa.gov/hwssclpdeliverables

The completed data package includes the Executive Narrative (see Appendix B for template), the Sample Summary Report (see Appendix C for example), and the Electronic Data Deliverable (EDD) (see Appendix D for a list of the column headers included in this document).

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PRELIMINARY REVIEW

This document is for the review of analytical data generated through the SOM01.2 SOW and any future editorial revisions of SOM01.2 for USEPA Region 2. To use this document effectively, the reviewer should have an understanding of the analytical method and a general overview of the Sample Delivery Group (SDG) or sample Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the sample data package [e.g., Modified Analysis requests, Traffic Report/Chain of Custody (TR/COC) documentation, SDG Narratives, etc.].

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The criteria for data validation outlined in the QAPP supersede this Standard Operating Procedure. The reviewer should contact the appropriate Regional Contract Laboratory Program Project Officer (CLP PO) to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

The SDGs or Cases routinely have unique samples that require special attention from the reviewer. These include field blanks and trip blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The sampling records (e.g., TR/COC records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken,
2. The Case number,
3. The complete list of samples with information on:
    a. Sample matrix;
    b. Field blanks (i.e., equipment blanks or rinsate blanks) and trip blanks;
    c. Field duplicates;
    d. Field spikes;
    e. QC audit samples;
    f. Shipping dates;
    g. Preservatives; and
    h. Laboratories involved.

The TR/COC documentation includes sample descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

The laboratory's SDG Narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, preservation, and unusual events should be documented in the SDG Narrative. The reviewer should also inspect any email or telephone/communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office (SMO), and USEPA Region 2.

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Preservation

Action:

1. Qualify aqueous sample results using preservation and technical holding time information as follows (see Table 1):
    a. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed within the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    b. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], no qualification of the data is necessary.
    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.
2. Qualify non-aqueous sample results using preservation and technical holding time information as follows (see Table 1):
    a. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed within the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    b. If there is no evidence that the samples were properly preserved (T = 4°C ± 2°C), and the samples were extracted or analyzed outside the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ).
    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], no qualification of the data is necessary.
    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding time [14 days from sample collection for

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extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

3. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the Data Review Narrative.
4. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
5. If technical holding times are grossly exceeded, use professional judgment to qualify the data.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, when technical holding times are exceeded.

Table 1. Holding Time Actions for Pesticide Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤ 7 days (for extraction) ≤ 40 days (for analysis)	J	UJ
	No	> 7 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes	≤ 7 days (for extraction) ≤ 40 days (for analysis)	No qualification	
	Yes	> 7 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes/No Grossly Exceeded		Use professional judgment	
Non-Aqueous	No	≤ 14 days (for extraction) ≤ 40 days (for analysis)	J	UJ
	No	> 14 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes	≤ 14 days (for extraction) ≤ 40 days (for analysis)	No qualification	
	Yes	> 14 days (for extraction) > 40 days (for analysis)	J	UJ
	Yes/No Grossly Exceeded		Use professional judgment	

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Gas Chromatograph with Electron Capture Detector (GC/ECD) Instrument Performance Check

Action:

1. Resolution Check Mixture
    a. The resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 80.0% for all analytes for the primary column and greater than or equal to 50.0% for the confirmation column in order to use one Individual Standard Mixture (C). The resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60.0% if two Individual Standard Mixtures (A and B) are to be used. If resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may also be questionable if coelution exists.
        i. Qualify detects for target compounds that were not adequately resolved as tentatively identified (NJ) (see Table 2).
        ii. Qualify non-detected compounds as unusable (R).
2. PEM
    a. If PEM analysis is not performed at the required frequency (see Pesticides Organic Analysis, Section II.C.2.a of SOM01.2 NFG), qualify all associated sample and blank results as unusable (R).
    b. The resolution between any two adjacent peaks in the initial calibration and continuing calibration verification PEMs must be greater than or equal to 90% on each GC column. If PEM resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may be questionable if coelution exists.
        i. Qualify detects as tentatively identified (NJ).
        ii. Qualify non-detects as unusable (R).
    c. If 4,4'-DDT breakdown is greater than 20.0%:
        i. Qualify detects for 4,4'-DDT as estimated (J).
        ii. Qualify detects for 4,4'-DDD and/or 4,4'-DDE as estimated (J).
        iii. If 4,4'-DDT was not detected, but 4,4'-DDD and/or 4,4'-DDE were detected, qualify non-detects for 4,4'-DDT as unusable (R), and qualify detects for 4,4'-DDD and/or 4,4'-DDE as presumptively present at an approximated quantity (NJ).
    d. If Endrin breakdown is greater than 20.0%:
        i. Qualify detects for Endrin as estimated (J).
        ii. Qualify detects for Endrin aldehyde and/or Endrin ketone as estimated (J).
        iii. If Endrin was not detected, but Endrin aldehyde and/or Endrin ketone were detected, qualify the non-detects for Endrin as unusable (R), and qualify detects for Endrin aldehyde and/or Endrin ketone as presumptively present at an approximated quantity (NJ).

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   e. If the combined 4,4'-DDT and Endrin breakdown is greater than 30.0%, the reviewer should consider the degree of individual breakdown of 4,4'-DDT and Endrin and apply qualifiers as described in this section.

3. Mid-point Individual Standard Mixtures (A and B) or (C)
   a. If mid-point Individual Standard Mixture analysis is not performed at the required frequency (see Pesticides Organic Analysis, Sections II.C.3.b and II.C.3.c of SOM01.2 NFG), qualify all associated sample and blank results as unusable (R).
   b. The resolution between any two adjacent peaks in the mid-point concentration of Individual Standard Mixtures (A and B) in the initial calibration and continuing calibration verification must be greater than or equal to 90.0% on each column. The resolution between any two adjacent peaks in the mid-point concentration of Individual Standard Mixture (C) in the initial calibration and continuing calibration verification must be greater than or equal to 80.0% for the primary column and greater than or equal to 50.0% for the secondary column. If mid-point Individual Standard Mixtures (A and B) or (C) resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may be questionable if coelution exists.
      i. Qualify detected target compounds that were not adequately resolved tentatively identified (NJ).
      ii. Qualify non-detects as unusable (R).
4. Note in the Data Review Narrative the potential effects on the sample data resulting from the instrument performance check criteria. Notify the Contract Laboratory Program Project Officer (CLP PO) if the laboratory has repeatedly failed to comply with the requirements for linearity, resolution, or 4,4'-DDT/Endrin breakdown.

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Table 2. Gas Chromatograph with Electron Capture Detector (GC/ECD) Instrument Performance Check Actions

Criteria [Individual Standard Mixtures (A and B)]	Criteria (Individual Standard Mixture C)	Action
Resolution Check Mixture % Resolution <60.0%	Resolution Check Mixture % Resolution <80.0% (primary column) % Resolution <50.0% (secondary column)	Detects: NJ Non-detects: R
PEM % Resolution <90.0%		Detects: NJ Non-detects: R
PEM: 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected		Detects for 4,4'-DDT: J Detects for 4,4'-DDD: J Detects for 4,4'-DDE: J
PEM: 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected		Non-detects for 4,4'- DDT: R Detects for 4,4'-DDD: NJ Detects for 4,4'-DDE: NJ
PEM: Endrin % Breakdown >20.0% and Endrin is detected		Detects for Endrin: J Detects for Endrin aldehyde: J Detects for Endrin ketone: J
PEM: Endrin % Breakdown >20.0% and Endrin is not detected		Non-detects for Endrin: R Detects for Endrin aldehyde: NJ Detects for Endrin ketone: NJ
PEM: Combined % Breakdown >30%		Apply qualifiers as described above considering degree of individual breakdown.
Mid-point Individual Standard Mixtures (A and B) % Resolution <90.0%	Mid-point Individual Standard Mixture (C) % Resolution <80.0% (primary column) Mid-point Individual Standard Mixture (C) % Resolution <50.0% (secondary column)	Detects: NJ Non-detects: R
PEM analysis not performed at the required frequency (see Pesticides, Section II.C.2.a of SOM01.2 NFG)		All results: R
Mid-point Individual Standard Mixtures analysis not performed at the required frequency (see Pesticides, Sections II.C.3.b.1 and II.C.3.c.1 of SOM01.2 NFG)		All results: R

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Initial Calibration

Table 3. Concentration Levels of Calibration Standards

Compound	Concentration (ng/mL)				
	CS1	CS2	CS3	CS4	CS5
alpha-BHC	5.0	10	20	40	80
gamma-BHC	5.0	10	20	40	80
Heptachlor	5.0	10	20	40	80
Endosulfan I	5.0	10	20	40	80
Dieldrin	10	20	40	80	160
Endrin	10	20	40	80	160
4,4'-DDD	10	20	40	80	160
4,4'-DDT	10	20	40	80	160
Methoxychlor	50	100	200	400	800
beta-BHC	5.0	10	20	40	80
delta-BHC	5.0	10	20	40	80
Aldrin	5.0	10	20	40	80
Heptachlor-epoxide	5.0	10	20	40	80
4,4'-DDE	10	20	40	80	160
Endosulfan II	10	20	40	80	160
Endosulfan sulfate	10	20	40	80	160
Endrin ketone	10	20	40	80	160
Endrin aldehyde	10	20	40	80	160
alpha-Chlordane	5.0	10	20	40	80
gamma-Chlordane	5.0	10	20	40	80
Tetrachloro-m-xylene	5.0	10	20	40	80
Decachlorobiphenyl	10	20	40	80	160
Toxaphene	500	1000	2000	4000	8000

Action:

NOTES: At least one chromatogram from each of the Individual Standard Mixtures (A and B) or (C) must yield peaks that give recorder deflections between 50-100% of full scale.
Either peak area or peak height may be used to calculate the Calibration Factors (CFs) that are, in turn, used to calculate %RSD. However, the type of peak measurement used to calculate each CF for a given compound must be consistent. For example, if peak area is used to calculate the low-point CF for Endrin, the mid-point and high-point CFs for Endrin must also be calculated using peak area.

1. If the proper initial calibration sequence is not performed, or the steps of the initial calibration are not followed in the proper sequence, use professional judgment to evaluate

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the effect on the data and notify the Contract Laboratory Program Project Officer (CLP PO) (see Table 4). This is especially critical for the low-level standards and non-detects.

2. If RT Windows are not calculated correctly, recalculate the windows and use the corrected values for all evaluations.
3. If the chromatogram display (recorder deflection) criteria are not met, use professional judgment to evaluate the effect on the data.
4. If the standard concentrations listed in Table 3 are not used, use professional judgment to evaluate the effect on the data and notify the CLP PO. This is especially critical for the low-level standards and non-detects.
5. The Percent Relative Standard Deviation (%RSD) of the CFs for each of the single component target compounds must be less than or equal to 20.0%, except for alpha-BHC and delta-BHC. The %RSD of the CFs for alpha-BHC and delta-BHC must be less than or equal to 25.0%. The %RSD of the CFs for the two surrogates (tetrachloro-m-xylene and decachlorobiphenyl) must be less than or equal to 30.0%. If the %RSD criteria are not met, qualify detects as estimated (J) and use professional judgment to qualify non-detected target compounds.
6. If the %RSD criteria are within allowable limits, no qualification of the data is necessary.
7. At the reviewer's discretion, and based on the project-specific data quality objectives, consider a more in-depth review using the following guidelines:
    a. If any pesticide target compound has a %RSD greater than the maximum criterion, and if eliminating either the high or the low-point of the curve does not restore the %RSD to less than or equal to the required maximum:
        i. Qualify detects for that compound(s) as estimated (J).
        ii. Qualify non-detected pesticide target compounds using professional judgment.
    b. If the high-point of the curve is outside of the linearity criteria (e.g., due to saturation):
        i. No qualifiers are required for detects in the linear portion of the curve.
        ii. Qualify detects outside of the linear portion of the curve as estimated (J).
        iii. No qualifiers are required for pesticide target compounds that were not detected.
    c. If the low-point of the curve is outside of the linearity criteria:
        i. No qualifiers are required for detects in the linear portion of the curve.
        ii. Qualify low-level detects in the area of non-linearity as estimated (J).
        iii. For non-detected pesticide compounds, use the lowest point of the linear portion of the curve to determine the new quantitation limit.
8. Note in the Data Review Narrative potential effects on the sample data due to problems with calibration. Notify the CLP PO if the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, RT, or resolution.
9. Qualify data for Toxaphene sharing the same RT Window with any Single Component Pesticide (SCP) in any Individual Standard Mixture using professional judgment.

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Table 4. Initial Calibration Actions for Pesticide Analyses

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
Initial calibration is not performed or not performed in the proper sequence	Use professional judgment and notify CLP PO	
%RSD exceeds allowable limits*	J	Use professional judgment
%RSD within allowable limits*	No qualification	

* %RSD < __20.0% for single component target compounds except alpha-BHC and delta-BHC.
%RSD ≤ 25.0% for alpha-BHC and delta-BHC.
%RSD ≤ 30.0% for Toxaphene peaks.
%RSD ≤ 30.0% for surrogates (tetrachloro-m-xylene and decachlorobiphenyl).

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Continuing Calibration Verification (CCV)

Action:

1. The RT Windows are used in qualitative identification. If the standards do not fall within the RT Windows, carefully evaluate the associated sample results (see Table 5). All samples injected after the last in-control standard are potentially affected.
    a. For non-detected target compounds in the affected samples, check to see if the sample chromatograms contain any peaks that are close to the expected RT Window of the pesticide of interest.
        i. If no peaks are present, consider non-detected values to be valid and no qualification of the data is necessary.
        ii. If any peaks are present close to the expected RT Window of the pesticide of interest, use professional judgment to qualify the non-detects as presumptively present (NJ).
    b. For detected compounds in the affected samples, if the peaks are within the RT Window, no qualification of the data is necessary. However, if the peaks are close to the expected RT Window of the pesticide of interest, the reviewer may take additional effort to determine if sample peaks represent the compounds of interest.
    For example, the reviewer can examine the data package for the presence of three or more standards containing the pesticide of interest that were run within the analytical sequence during which the sample was analyzed. If three or more such standards are present, the RT Window can be re-evaluated using the Mean Retention Times (RTs) of the standards.
        i. If the peaks in the affected sample fall within the revised window, qualify detects as tentatively identified (NJ).
        ii. If the reviewer cannot do anything with the data to resolve the problem of concern, qualify all non-detects as unusable (R).
2. For the PEM, if the Percent Difference is not within ±25.0%, qualify associated detects as estimated (J) and non-detects as estimated (UJ).
3. For the Calibration Verification Standard (CS3), if the Percent Difference is not within ±20.0%, qualify associated detects as estimated (J) and non-detects as estimated (UJ).
4. If more than 14 hours has elapsed from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of either a PEM or mid-point concentration of the Individual Standard Mixtures (A and B) or (C), qualify all data as unusable (R).
5. If more than 12 hours has elapsed from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of the last sample or blank that is part of the same analytical sequence, qualify all data as unusable (R).
6. If more than 72 hours has elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene Calibration Verification Standard (CS3), qualify all data as unusable (R).

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7. If the Percent Difference, time elapsed, and RTs are within acceptable limits, no qualification of the data is necessary.
8. Note in the Data Review Narrative potential effects on the sample data due to problems with calibration.

Table 5. Continuing Calibration Verification (CCV) Actions for Pesticide Analyses

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RT out of RT window	Use professional judgment	
%D not within limits*	J	UJ
Time elapsed is greater than acceptable limits**	R	
%D, time elapsed, and RT are within acceptable limits	No qualification	

* See Actions 2 and 3
** See Actions 4, 5, and 6

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Blanks

Action:

NOTES: The concentration of non-target compounds in all blanks must be less than or equal to 10 µg/L.
The concentration of each target compound found in the method or field blanks must be less than its CRQL listed in the method.
Data concerning the field blanks are not evaluated as part of the CCS process. If field blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

NOTES: "Water blanks, "drill blanks", and "distilled water blanks" are validated like any other sample and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.
All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for system monitoring compounds, instrument performance criteria, and spectral or calibration QC problems.
Analytes qualified "U" for blank contamination are treated as "hits" when qualifying for calibration criteria.
Samples taken from a drinking water tap do not have associated field blanks.
When applied as described in Table 6 below, the contaminant concentration in the blank is multiplied by the sample dilution factor.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one of the same type of blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.

1. If a target SCP or Toxaphene is found in a method blank, but not found in the sample, no qualification of the data is necessary (see Table 6).
2. If a target SCP or Toxaphene concentration in a method or field blank is less than the CRQL and:
    a. the sample concentration is less than the CRQL, report the CRQL value with a "U".
    b. the sample concentration is greater than or equal to the CRQL, no qualification is required.
3. If a target SCP or Toxaphene concentration in a method or field blank is greater than the CRQL and:
    a. the sample concentration is less than the CRQL, report the CRQL value with a "U".

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   b. the sample concentration is greater than or equal to the CRQL, and less than or equal to the blank concentration, report the concentration of the compound in the sample at the same concentration found in the blank and qualify with a "U".
   c. the sample concentration is greater than or equal to the CRQL and greater than the blank concentration, no qualification is required.
4. If a target SCP or Toxaphene concentration in a method or field blank is equal to the CRQL and:
   a. the sample concentration is less than or equal to the CRQL, report the CRQL value with a "U".
   b. the sample concentration is greater than the CRQL, no qualification is required.
5. If gross contamination exists (i.e., saturated peaks, "hump-o-grams", "junk" peaks), raise the CRQL to the level of the blank contamination and report the associated sample data below this level as CRQL-U. Non-detected pesticide target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.
6. If contaminants are found in the field blanks, the following is recommended:
   a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank.
      i. If the analyte was present at a comparable level in the method blank, the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.
      ii. If the analyte was not present in the method blank, the source of contamination may be in the storage area, in the field, or during sample transport. Consider all associated samples for possible cross-contamination.
7. If method blank data are unavailable, the reviewer may use professional judgment or substitute field blank data for missing method blank data.

NOTE: There may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, they should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result.

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Table 6. Blank Actions for Pesticide Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Sulfur Cleanup, Instrument, Field	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and ≤ blank concentration	Report blank value for sample concentration with a U
		≥ CRQL and > blank concentration	No qualification required
	= CRQL	≤ CRQL	Report CRQL value with a U
		> CRQL	No qualification required
	Gross contamination	Detects	Report blank value for sample concentration with a U

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Surrogate Spikes

Action:

If either surrogate spike recovery is outside the acceptance limits, the reviewer must consider the existence of coelution and interference in the raw data and use professional judgment to qualify data, as surrogate recovery problems may not directly apply to target analytes.

1. For any surrogate recovery greater than 150% (see Table 7):
    a. Qualify detected target compounds as biased high (J+).
    b. Do not qualify non-detected target compounds.
2. If both surrogate recoveries are greater than or equal to 30%, and less than or equal to 150%, no qualification of the data is necessary.
3. For any surrogate recovery greater than or equal to 10%, and less than 30%:
    a. Qualify detected target compounds as biased low (J-).
    b. Qualify non-detected target compounds as approximated (UJ).
4. For any surrogate recovery less than 10%, the reviewer should examine the sample chromatogram to assess the qualitative validity of the analysis. If low surrogate recoveries are from sample dilution, professional judgment should be used to determine if the resulting data should be qualified. If sample dilution is not a factor:
    a. Qualify detected target compounds as biased low (J-).
    b. Qualify non-detected target compounds as unusable (R).
5. In the special case of a blank analysis with surrogates out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. Note, for Contract Laboratory Program Project Officer (CLP PO) action, analytical problems even if this judgment allows some use of the affected data.
6. If surrogate RTs in PEMs, Individual Standard Mixtures, samples, and blanks are outside of the RT Windows, the reviewer must use professional judgment to qualify data.
7. If surrogate RTs are within RT windows, no qualification of the data is necessary.
8. If the two surrogates were not added to all samples, MS/MSDs, standards, LCSs, and blanks, use professional judgment in qualifying data as missing surrogate analyte may not directly apply to target analytes.

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Table 7. Surrogate Actions for Pesticide Analyses

Criteria	Action*	
	Detected Target Compounds	**Non-detected Target Compounds**
%R > 150%	J+	No qualification
30% < %R < 150%	No qualification	
10% < %R < 30%	J-	UJ
%R < 10% (sample dilution not a factor)	J-	R
%R < 10% (sample dilution is a factor)	Use professional judgment	
RT out of RT window	Use professional judgment	
RT within RT window	No qualification	

* Use professional judgment in qualifying data, as surrogate recovery problems may not directly apply to target analytes.

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Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

Action:

NOTES: Data for MS and MSDs will not be present unless requested by the Region. Notify the Contract Laboratory Program Project Officer (CLP PO) if a field blank was used for the MS and MSD, unless designated as such by the Region.

NOTE: For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

1. No qualification of the data is necessary on MS and MSD data alone. However, using professional judgment, the validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

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Laboratory Control Samples (LCSs)

Table 10. Pesticides Laboratory Control Sample (LCS) Spike Compounds and Recovery Limits

LCS Spike Compound	Recovery Limits (%)
gamma-BHC	50 – 120
Heptachlor epoxide	50 – 150
Dieldrin	30 – 130
4,4'-DDE	50 – 150
Endrin	50 – 120
Endosulfan sulfate	50 – 120
gamma-Chlordane	30 – 130
Tetrachloro-m-xylene (surrogate)	30 – 150
Decachlorobiphenyl (surrogate)	30 – 150

Action:

NOTES: The recovery limits for any of the compounds in the LCS may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.
All samples prepared and analyzed with an LCS that does not meet the technical acceptance criteria in the method will require re-extraction and re-analysis.

If the LCS criteria are not met, laboratory performance and method accuracy are in question. Use professional judgment to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data for which the associated LCS does not meet the required criteria.

1. If the LCS recovery criteria are not met, use the LCS results to qualify sample data for the specific compounds that are included in the LCS solution (see Table 11).
    a. If the LCS recovery exceeds the upper acceptance limit, qualify detected target compounds as estimated (J). Do not qualify non-detected target compounds.
    b. If the LCS recovery is less than the lower acceptance limit, qualify detected target compounds as estimated (J) and non-detects as unusable (R).
    c. Use professional judgment to qualify data for compounds other than those compounds that are included in the LCS.
    d. Use professional judgment to qualify non-LCS compounds. Take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in the performance of the LCS compound to the non-LCS compound.
2. If the LCS recovery is within allowable limits, no qualification of the data is necessary.
3. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if a laboratory fails to analyze an LCS with each Sample Delivery Group (SDG), or if the reviewer has knowledge that a laboratory consistently fails to generate acceptable LCS recoveries.

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Table 11. Laboratory Control Sample (LCS) Recovery Actions

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
%R > Upper Acceptance Limit	J	No qualification
%R < Lower Acceptance Limit	J	R
Lower Acceptance Limit < %R < Upper Acceptance Limit	No qualification	

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Florisil Cartridge Performance Check

Action:

NOTE: Florisil cartridge cleanup is mandatory for all extracts.

1. If the Florisil Cartridge Performance Check criteria are not met, examine the raw data for the presence of polar interferences and use professional judgment in qualifying the data as follows (see Table 12):
   a. If the Percent Recovery is greater than 120% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected compounds as estimated (J). Do not qualify non-detected target compounds.
   b. If the Percent Recovery is greater than or equal to 80% and less than or equal to 120% for all the pesticide target compounds, no qualification of the data is necessary.
   c. If the Percent Recovery is greater than or equal to 10% and less than 80% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected target compounds as estimated (J) and non-detected target compounds as approximated (UJ).
   d. If the Percent Recovery is less than 10% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected compounds as estimated (J) and qualify non-detected target compounds as unusable (R).
   e. If the Percent Recovery of 2,4,5-trichlorophenol in the Florisil Cartridge Performance Check is greater than or equal to 5%, use professional judgment to qualify detected and non-detected target compounds, considering interference on the sample chromatogram.
2. Note in the Data Review Narrative potential effects on the sample data resulting from the Florisil Cartridge Performance Check analysis not yielding acceptable results.

Table 12. Florisil Cartridge Performance Check Actions

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
%R > 120% (pesticide target compounds)	J	No qualification
80% ≤ %R ≤ 120%	No qualification	
10% ≤ %R <80% (pesticide target compounds)	J	UJ
%R < 10% (pesticide target compounds)	J	R
%R > 5% (2,4,5-trichlorophenol)	Use professional judgment*	

* Check sample chromatogram for interferences.

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Gel Permeation Chromatography (GPC) Performance Check

Action:

NOTE: GPC cleanup is mandatory for all soil samples.

1. If GPC criteria are not met, examine the raw data for the presence of high molecular weight contaminants; examine subsequent sample data for unusual peaks; and use professional judgment in qualifying the data. Notify the Contract Laboratory Program Project Officer (CLP PO) if the laboratory chooses to analyze samples under unacceptable GPC criteria.
2. If the Percent Recovery is less than 10% for the pesticide compounds and surrogates during the GPC calibration check, the non-detected target compounds may be suspect. Qualify detected compounds as estimated (J) (see Table 13). Qualify all non-detected target compounds as unusable (R).
3. If the Percent Recovery is greater than or equal to 10% and is less than 80% for any of the pesticide target compounds in the GPC calibration, qualify detected target compounds as estimated (J) and non-detected target compounds as approximated (UJ).
4. If the Percent Recovery is greater than or equal to 80% and less than or equal to 110% for all the pesticide target compounds, no qualification of the data is necessary.
5. If high recoveries (i.e., greater than 110%) were obtained for the pesticides and surrogates during the GPC calibration check, qualify detected compounds as estimated (J). Do not qualify non-detected target compounds.
6. Note in the Data Review Narrative potential effects on the sample data resulting from the GPC cleanup analyses not yielding acceptable results.

Table 13. Gel Permeation Chromatography (GPC) Performance Check Actions

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
%R < 10% (pesticide target compounds)	J	R
10% ≤ %R < 80%	J	UJ
80% ≤ %R ≤ 110%	No qualification	
%R > 110% (pesticide target compounds)	J	No qualification

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Target Compound Identification

Criteria:

1. The Retention Times (RTs) of both of the surrogates and reported target compounds in each sample must be within the calculated RT Windows on both columns. Tetrachloro-m-xylene (TCX) must be within ±0.05 minutes of the Mean RT (RT) determined from the initial calibration and Decachlorobiphenyl (DCB) must be within ±0.10 minutes of the RT determined from the initial calibration.
2. The Percent Difference (%D) for the detected mean concentrations of a pesticide target compound between the two Gas Chromatograph (GC) columns must be within the inclusive range of ±25.0.
3. When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract must use the same scaling factor as was used for the low-point standard of the initial calibration associated with those analyses.
4. Chromatograms must display Single Component Pesticides (SCPs) detected in the sample and the largest peak of any multi-component analyte detected in the sample at less than full scale.
5. If an extract must be diluted, chromatograms must display SCPs peaks between 10-100% of full scale, and multi-component analytes between 25-100% of full scale.
6. For any sample, the baseline of the chromatogram must return to below 50% of full scale before the elution time of alpha-BHC, and also return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of DCB.
7. If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

Action:

1. If the qualitative criteria for both columns were not met, all target compounds that are reported as detected should be considered non-detected. The reviewer should use professional judgment to assign an appropriate quantitation limit using the following guidance:
   a. If the detected target compound peak was sufficiently outside the pesticide RT Window, the reported values may be a false positive and should be replaced with the sample Contract Required Quantitation Limits (CRQL) value.
   b. If the detected target compound peak poses an interference with potential detection of another target peak, the reported value should be considered and qualified as unusable (R).
2. If the data reviewer identifies a peak in both GC column analyses that falls within the appropriate RT Windows, but was reported as a non-detect, the compound may be a false negative. Use professional judgment to decide if the compound

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should be included. Note in the Data Review Narrative all conclusions made regarding target compound identification.

3. If the Toxaphene peak RT windows determined from the calibration overlap with SCPs or chromatographic interferences, use professional judgment to qualify the data.
4. If target compounds were detected on both GC columns, and the Percent Difference between the two results is greater than 25.0%, consider the potential for coelution and use professional judgment to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, use professional judgment to determine how best to report, and if necessary, qualify the data according to the guidelines in Table 14 below.
5. If Toxaphene exhibits a marginal pattern-matching quality, use professional judgment to establish whether the differences are due to environmental "weathering" (i.e., degradation of the earlier eluting peaks relative to the later eluting peaks). If the presence of Toxaphene is strongly suggested, report results as presumptively present (N).

Table 14. Action on Qualifying Positive Pesticide Result

Percent Differences	Qualifier
0% – 25%	No qualification
26% – 200%	Professional judgment
101% – 200% (interferences detected)*	JN
> 50% (pesticide value < CRQL)**	U
> 200%	R

* When interferences are detected on either column, qualify the data as "JN".
** When the pesticide value is below CRQL and %D > 50%, raise the value to CRQL and qualify "U" undetected.

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Gas Chromatograph/Mass Spectrometer (GC/MS) Confirmation

Action:

NOTE: This confirmation is not usually provided by the laboratory. In cases where it is provided, use professional judgment to determine if data qualified with "C" can be salvaged if it was previously qualified as unusable (R).

1. If the quantitative criteria for both columns were met (≥ 5.0 ng/µL for SCPs and ≥ 125 ng/µL for Toxaphene), determine whether GC/MS confirmation was performed. If it was performed, qualify the data using the following guidance (see Table 15):
    a. If GC/MS confirmation was not required because the quantitative criteria for both columns was not met, but it was still performed, use professional judgment when evaluating the data to decide whether the detect should be qualified with "C".
    b. If GC/MS confirmation was performed, but unsuccessful for a target compound detected by GC/ECD analysis, qualify those detects as "X".

Table 15. Gas Chromatograph/Mass Spectrometer (GC/MS) Confirmation Actions

Criteria	Action
SCP/Toxaphene was confirmed by GC/MS	Detects C
SCP/Toxaphene was not confirmed by GC/MS	Detects X

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Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

Action:

1. When a sample is analyzed at more than one dilution, the lowest CRQLs are used unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample. Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original Form I and substituting the data from the diluted sample.
2. Results between the MDL and CRQL should be qualified as estimated (J).
3. Results less than the MDL should be reported at the CRQL and qualified (U). MDLs themselves are not reported.
4. For non-aqueous samples, if the percent moisture is less than 70.0%, no qualification of the data is necessary. If the percent moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as estimated (J) and non-detects as approximated (UJ). If the percent moisture is greater than or equal to 90.0%, qualify detects as estimated (J) and non-detects as unusable (R) (see Table 16).
5. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.

Table 16. Percent Moisture Actions for Pesticide Analysis For Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
% Moisture < 70.0	No qualification	
70.0 < % Moisture < 90.0	J	UJ
% Moisture > 90.0	J	R

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Field Duplicates

Action:

NOTE: In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.

Identify which samples within the data package are field duplicates. Estimate the relative percent difference (RPD) between the values for each compound. Use professional judgment to note large RPDs (> 50%) in the narrative.

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Overall Assessment of Data

Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

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APPENDIX A: GLOSSARY

Analyte -- The element of interest, ion, or parameter an analysis seeks to determine.
Analytical Services Branch (ASB) -- Directs the Contract Laboratory Program (CLP) from within the Office of Superfund Remediation and Technical Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).
Analytical Sample -- Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB). Note that the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA); Matrix Spike samples; duplicate samples; serial dilution samples, analytical (post-digestion/post-distillation) spike samples; Interference Check Samples (ICSs); Laboratory Control Samples (LCSs); and Preparation Blanks.
Associated Samples -- Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples run under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.
Blank -- A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.
Calibration -- The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentration of acids as used in the sample preparation.
Calibration Blank -- A blank solution containing all of the reagents in the same concentration as those used in the analytical sample preparation. This blank is not subject to the preparation method.
Calibration Curve -- A plot of instrument response versus concentration of standards.
Calibration Standards -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.
Case -- A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).
Contract Compliance Screening (CCS) -- A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under USEPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.
Continuing Calibration Verification (CCV) -- A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples.

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Contract Laboratory Program (CLP) -- Supports the USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technical Innovation (OSRTI) of USEPA.
Contract Laboratory Program Project Officer (CLP PO) -- The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.
Contract Required Quantitation Limit (CRQL) -- Minimum level of quantitation acceptable under the contract Statement of Work (SOW).
Duplicate -- A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.
Field Blank -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.
Field Duplicate -- A duplicate sample generated in the field, not in the laboratory.
Holding Time -- The maximum amount of time samples may be held before they are processed.
Contractual -- The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.
Technical -- The maximum amount of time that samples may be held from the collection date until analysis.
Initial Calibration -- Analysis of analytical standards for a series of different specified concentrations to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.
Initial Calibration Verification (ICV) -- Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when USEPA ICV solutions are not available.
Internal Standard -- A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.
Matrix -- The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrices are aqueous/water, soil/sediment, wipe, and filter.
Matrix Spike -- Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology (also identified as a pre-distillation/digestion spike).
Method Detection Limit (MDL) -- The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

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Narrative (SDG Narrative) -- Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.
Office of Solid Waste and Emergency Response (OSWER) – The USEPA office that provides policy, guidance, and direction for the USEPA's solid waste and emergency response programs, including Superfund.
Percent Difference (%D) -- As used in this document and the Statement of Work (SOW), is used to compare two values. The difference between the two values divided by one of the values.
Performance Evaluation (PE) Sample -- A sample of known composition provided by USEPA for contractor analysis. Used by USEPA to evaluate Contractor performance.
Preparation Blank -- An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.
Relative Percent Difference (RPD) -- As used in this document and the Statement of Work (SOW) to compare two values, the RPD is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).
Regional Sample Control Center Coordinator (RSCC) -- In USEPA Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.
Relative Standard Deviation (RSD) -- As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.
Sample -- A single, discrete portion of material to be analyzed, which is contained in single or multiple containers and identified by a unique Sample Number.
Sample Delivery Group (SDG) -- A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
b. Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
c. Scheduled at the same level of deliverable.

In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG. Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory.
Sample Management Office (SMO) -- A contractor-operated facility operated under the SMO contract, awarded and administered by the USEPA. Provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).
Statement of Work (SOW) -- A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

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APPENDIX B: ORGANIC DATA EXECUTIVE NARRATIVE TEMPLATE

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
DESA/HWSB/HWSS
2890, Woodbridge Avenue, Edison, NJ 08837

EXECUTIVE NARRATIVE

Case No. :
Site:
Number of Samples:
Analysis:

SDG No.:
Laboratory:
Sampling dates:

QAPP
HWSS #:
Contractor Document #:

SUMMARY:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.
Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated.
Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

Major Findings:

Minor Findings:

COMMENT:

Reviewer Name(s):

Approver's Signature: Date:

Name:

Affiliation: USEPA/R2/HWSB/HWSS

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SAMPLE

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APPENDIX C: SAMPLE ORGANIC DATA SAMPLE SUMMARY

Case No: 00001	Contract: XY1234	SDG No: XY123	Lab Code: 00001
Sample Number: BA1X5	Method: Pest	Matrix: Soil	MA Number:
Sample Location:	pH: 8.2	Sample Date: 13322059	Sample Time: 13:08:00
% Moisture : 56.85		% Solids :	

Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable	Validation Level
2,4,5-T	3.9	ug/kg	1.0	U	U	Yes	S3VEM
2,4,5-TP (Silvex)	3.9	ug/kg	1.0	U	U	Yes	S3VEM
2,4-D	39	ug/kg	1.0	U	U	Yes	S3VEM
2,4-DB	39	ug/kg	1.0	U	U	Yes	S3VEM
Dalapon	97	ug/kg	1.0	U	U	Yes	S3VEM
Dicamba	3.9	ug/kg	1.0	U	U	Yes	S3VEM
Dichloroprop	39	ug/kg	1.0	U	U	Yes	S3VEM
Dinoseb	19	ug/kg	1.0	U	U	Yes	S3VEM
MCPA	390	ug/kg	1.0	U	UJ	Yes	S3VEM
MCPP	390	ug/kg	1.0	U	UJ	Yes	S3VEM
Pentachlorophenol	3.9	ug/kg	1.0	U	U	Yes	S3VEM
4-Nitrophenol	39	ug/kg	1.0	U	U	Yes	S3VEM

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APPENDIX D: ELECTRONIC DATA DELIVERABLE TEMPLATE

DATA_PROVIDER	LAB_MATRIX_CODE	RESULT_UNIT
SYS_SAMPLE_CODE	ANAL_LOCATION	DETECTION_LIMIT_UNIT
SAMPLE_NAME	BASIS	TIC_RETENTION_TIME
SAMPLE_MATRIX_CODE	CONTAINER_ID	RESULT_COMMENT
SAMPLE_TYPE_CODE	DILUTION_FACTOR	QC_ORIGINAL_CONC
SAMPLE_SOURCE	PREP_METHOD	QC_SPIKE_ADDED
PARENT_SAMPLE_CODE	PREP_DATE	QC_SPIKE_MEASURED
SAMPLE_DEL_GROUP	LEACHATE_METHOD	QC_SPIKE_RECOVERY
SAMPLE_DATE	LEACHATE_DATE	QC_DUP_ORIGINAL_CONC
SYS_LOC_CODE	LAB_NAME_CODE	QC_DUP_SPIKE_ADDED
START_DEPTH	QC_LEVEL	QC_DUP_SPIKE_MEASURED
END_DEPTH	LAB_SAMPLE_ID	QC_DUP_SPIKE_RECOVERY
DEPTH_UNIT	PERCENT_MOISTURE	QC_RPD
CHAIN_OF_CUSTODY	SUBSAMPLE_AMOUNT	QC_SPIKE_LCL
SENT_TO_LAB_DATE	SUBSAMPLE_AMOUNT_UNIT	QC_SPIKE_UCL
SAMPLE_RECEIPT_DATE	ANALYST_NAME	QC_RPD_CL
SAMPLER	INSTRUMENT_ID	QC_SPIKE_STATUS
SAMPLING_COMPANY_CODE	COMMENT	QC_DUP_SPIKE_STATUS
SAMPLING_REASON	PRESERVATIVE	QC_RPD_STATUS
SAMPLING_TECHNIQUE	FINAL_VOLUME	BREAK_2
TASK_CODE	FINAL_VOLUME_UNIT	SYS_SAMPLE_CODE
COLLECTION_QUARTER	CAS_RN	LAB_ANL_METHOD_NAME
COMPOSITE_YN	CHEMICAL_NAME	ANALYSIS_DATE
COMPOSITE_DESC	RESULT_VALUE	TOTAL_OR_DISSOLVED
SAMPLE_CLASS	RESULT_ERROR_DELTA	COLUMN_NUMBER
CUSTOM_FIELD_1	RESULT_TYPE_CODE	TEST_TYPE
CUSTOM_FIELD_2	REPORTABLE_RESULT	TEST_BATCH_TYPE
CUSTOM_FIELD_3	DETECT_FLAG	TEST_BATCH_ID
COMMENT	LAB_QUALIFIERS	CASE
BREAK_1	VALIDATOR_QUALIFIERS	CONTRACT_NUM
SYS_SAMPLE_CODE	INTERPRETED_QUALIFIERS	SCRIBE_SAMPLE_ID
LAB_ANL_METHOD_NAME	ORGANIC_YN	SAMPLE_TIME
ANALYSIS_DATE	METHOD_DETECTION_LIMIT	FRACTION
TOTAL_OR_DISSOLVED	REPORTING_DETECTION_LIMIT	PH
COLUMN_NUMBER	QUANTITATION_LIMIT	DATA_VAL_LABEL
TEST_TYPE		

FOIA_07123_0003552_0753

VALIDATION SOP

Volatile Organics

USEPA Region 2 SOP HW-24
Rev.1, 6/99

SOP NO. HW-24
Revision 1
June 1999

STANDARD OPERATING PROCEDURE FOR THE VALIDATION OF ORGANIC DATA ACQUIRED USING SW-846 METHOD 8260B (Rev 2, Dec 1996)

VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) : CAPILLARY COLUMN TECHNIQUE

FOIA_07123_0003552_0755

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the USEPA SW-846, Method 8260B. The validation methods and actions discussed in this document are based on the requirements set forth in USEPA SW-846, Chapter Two, Rev 3, December 1996; Method 8000B, Rev 2, December 1996; Method 8260B, Rev 2, December 1996; and "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," February, 1994. This document covers technical as well as method specific problems; however situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

Summary

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are defined on page 25.

The reviewer must prepare a detailed data assessment to be submitted along with the complete SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data, and contract non-compliance.

FOIA_07123_0003552_0758

YES NO N/A

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ LAB:

SITE NAME:

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format or CLP Forms Equivalent? [ ] ___
ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative, signed release, or cover letter present? [ ]

2.2 Are case number and SDG number(s) contained in the narrative or cover letter? [ ] ___

II. VOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Reports, Chain of Custodies, or signed releases from the field samplers present for all samples? [ ] ___
ACTION: If no, contact the laboratory/sampling team for replacement of missing or illegible copies.

1.2 Is a sampling trip report present (if required)? [ ] ___

1.3 Sample Conditions/Problems

1.3.1 Do the Traffic Reports, Chain of Custodies, or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data? ___ [ ]

FOIA_07123_0003552_0759

YES NO N/A

ACTION: If all the VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, flag all positive results "J" and all non-detects "R".

ACTION: If samples were not iced or if the ice was melted upon receipt at the laboratory and the temperature of the cooler was elevated (>10°C), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any volatile holding times, determined from date of collection to date of analysis, been exceeded?

The holding time for aqueous samples is 14 days.

The holding time for soils is 10 days.

NOTE: If unpreserved, aqueous samples maintained at 4°C for aromatic hydrocarbons analysis must be analyzed within 7 days. If preserved with acid to a pH<2 and stored at 4°C, then aqueous samples must be analyzed within 14 days from time of collection. If uncertain about preservation, contact the laboratory/sampling team to determine whether or not samples were preserved.

ACTION: If holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis,

the reviewer must use professional judgement

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YES NO N/A

to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that

___ [ ]

non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).

3.0 Surrogate Recovery (CLP Form II Equivalent)

3.1 Have the volatile surrogate recoveries been listed on Surrogate Recovery forms for each of the following matrices:

a. Wate

FOIA_07123_0003552_0761

	YES	NO	N/A
b. Soil			
3.2 If so, are all the samples listed on the appropriate Surrogate Recovery forms for each matrix:	[ ]	___	
a. Water	[ ]	___	
b. Soil			
ACTION: If large errors exist, deliverables are unavailable or information is missing,	[ ]	___	
document the effect(s) in Data Assessments and contact the laboratory/project	[ ]	___	
officer/appropriate official for an explanation/resubmittal, make any necessary corrections and document effect in the Data Assessment.			
3.3 Were outliers marked correctly with an asterisk?			
ACTION: Circle all outliers with a red pencil.			
3.4 Were one or more volatile surrogate recoveries out of specification for any sample or method blank (Surrogate recovery is 80-120% for aqueous and 70-130% for soil/sediment samples)?	[ ]	___	
NOTE: Laboratory may use in-house performance criteria (as per SW-846, 8000B-43, Sect. 8.6 & 8.7).	___	[ ]	
If yes, were samples reanalyzed?			
Were method blanks reanalyzed?			
ACTION: If all surrogate recoveries are > 10% but 1 or more compounds do not meet method specifications:	[ ]	___	
	[ ]	___	

1. Flag all positive results as estimated ("J").

2. Flag t

FOIA_07123_0003552_0762

YES NO N/A

he lower acceptance limit.

3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any surrogate has a recovery of < 10%:

1. Positive results are qualified with ("J").
2. Non-detects for that should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and reported data?

ACTION: If large errors exist, take action as specified in section 3.2 above.

4.0 Laboratory Control Samples/Matrix Spikes (CLP Form III Equivalent) ___ [ ]

4.1 Have the volatile Laboratory Control Samples (LCS) recoveries been listed on the laboratory reporting form?

NOTE: If the data has not been reported, then contact the laboratory/project officer to obtain the information necessary to evaluate the spike recoveries in the MS, MSD, and LCS. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes. [ ] ___

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

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YES NO N/A

NOTE: The LCS spike is spiked with the same analytes at the same concentrations as the matrix spike (as per SW-846, 8000B-40, Sect. 8.5) if different, make note in Data Assessment.

1. If 25% of the LCS recoveries were < lower in-

4.2 Were Laboratory Control Samples analyzed at the required frequency for each of the following matrices:

a. Water

b. Soil

c. Med Soil

2. If two or more LCS recoveries were < 10%

ACTION: If any LCS data are missing, take the action specified in section 3.2 above.

4.3 How many LCS volatile spike recoveries are outside QC limits?

Water	Soil			
		[ ]	___	
____ out of ____	____ out of	[ ]	___	___
		[ ]	___	

ACTION: Circle all outliers with a red pencil.

4.4 Were one or more of the volatile LCS recoveries outside of the in-house laboratory recovery criteria for spiked analytes? If none are present, then use 70-130% recovery as per SW-846, 8000B-41, Sect. 8.5.4.

ACTION:
1. If the recovery is > upper in-house limit (or 130%), only positive values for the affected compound(s) are flagged "J".
2. If the recovery is < lower in-house limit (or 70%), flag positive values for the affected compound(s) "J" and non-detects "R". ___ [ ] ___

All analytes in associated sample results are quali

FOIA_07123_0003552_0764

YES NO N/A

4.5 Have the volatile Matrix Spike(MS)/Matrix Spike Duplicate (MSD) recoveries been listed on the laboratory reporting form? out of ____

ACTION: Circ

4.6 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Water

b. Soil

c. Med Soil

4.9 Were one or more of the volatile MS/MSD recoveries

NOTE: The laboratory should use one matrix spike and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If the sample is not expected to contain target analytes, a MS/MSD should be analyzed (SW-846, 8260B-25, Sect. 8.4.2)

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above. [ ] ___

4.7 How many MS/MSD volatile spike recoveries are outside QC limits?

[ ] ___

Water Soil

[ ] ___

____ out of ____ ____ out of

[ ] ___

4.8 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water Soil

FOIA_07123_0003552_0765

YES NO N/A

___ [ ]

NOTE: If any individual % recovery in the MS (or MSD) falls outside the designated range for recovery the reviewer should determine if there is a matrix effect. A matrix effect is indicated if the LCS data are within limits but the MS data exceeds the limits.

NOTE: MS/MSD criteria apply only to the original sample, its dilutions, and the associated MS/MSD samples.

1. If the recovery is > upper in-house limit/130%, only positive values for the affected compound(s) are flagged "J".
2. If the recovery is < lower in-house limit/70%, flag positive values for the affected compound(s) "J" and non-detects "UJ".
3. If two or more MS/MSD recoveries were < 10% qualify all positive results "J" and all non-detects "R".

5.0 Blank (CLP Form IV Equivalent)

5.1 Is the Method Blank Summary form present?

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported for samples of similar matrix, or concentration level, and for

FOIA_07123_0003552_0766

	YES	NO	N/A
each extraction batch?	[ ]	___	
5.3 Has a method blank been analyzed for each GC/MS system used ?			
ACTION: If any method blank data are missing, take action as specified in section 3.2. If not available, use professional judgement to determine if the associated sample data should be qualified.	[ ]	___	
5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.	[ ]	___	
Is the chromatographic performance (baseline stability) for each instrument acceptable for the volatiles? ACTION: Use professional judgement to determine the effect on the data.	[ ]	___	

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and

FOIA_07123_0003552_0767

YES NO N/A

ot used to qualify the data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.

6.2 Do any trip/field/rinse/ blanks have positive results for target analytes and/or TICs? [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (May attach a separate sheet.) ___ [ ]

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field Blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

FOIA_07123_0003552_0768

YES NO N/A

trip/field/rinse/equipment blank.

	Sample conc > CRQL but < 10x blank value	Sample conc < CRQL & <10x blank value	Sample conc > CRQL & >10x blank	
Methylene Chloride Acetone Toluene 2-Butanone	Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification needed	
	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL value & > 5x blank	
Other contam-inants	Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification needed	

NOTE: Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

NOTE: The reporting of TIC compounds may or may not be required.

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" unusable.

6.3 Are there trip/field/rinse/equipment blanks associated with every sample?

ACTION: For low level samples, note in Data Assessment that there is no associated

FOIA_07123_0003552_0769

	YES	NO	N/A
h high concentrations, use professional judgement on qualification of these values and make note in Data Assessment. Exception: samples taken from a drinking water tap do not have associated field blanks.	[ ]	___	

7.0 GC/MS Apparatus and Materials

7.1 Did the lab use the proper gas chromatographic column(s) for analysis of volatiles by Method 8260B? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used.

FOIA_07123_0003552_0770

YES NO N/A

For the analysis of volatiles, the method requires requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL(Supelco) or equivalent column. (see SW-846, page 8260B-7, section 4.9.2) [ ]

ACTION: If the specified column, or equivalent, was not used, document the effects in the Data Assessment. Use professional judgement to determine the acceptability of the data.

8.0 GC/MS Instrument Performance Check (CLP Form V Equivalent)

8.1 Are the GC/MS Instrument Performance Check forms present for Bromofluorobenzene (BFB), and do these forms list the associated samples with date/time analyzed?

8.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift? [ ] ___

8.3 Has an instrument performance check solution (BFB) been analyzed for every twelve hours of sample analysis per instrument?(see Table 4, SW-846, page 8260B-36) [ ] ___

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available. [ ] ___

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	
_____	_____	_____	
_____	_____	_____	

ACTION: If the laboratory/project officer/appropriate official cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If

FOIA_07123_0003552_0771

YES NO N/A

mass assignment is in error, flag all associated sample data as unusa

8.4 Have the ion abundances been normalized to m/z 95? [ ] ___

8.5 Have the ion abundance criteria been met for each instrument used? [ ] ___

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, take action as specified in section 3.2.

8.6 Are there any transcription/calculation errors between mass lists and reported values? (Check at least two values but if errors are found, check more.) ___ [ ]

8.7 Have the appropriate number of significant figures (two) been reported? [ ] ___

ACTION: If large errors exist, take action as specified in section 3.2.

8.8 Are the spectra of the mass calibration compound acceptable? [ ] ___

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

9.0 Target Analytes (CLP Form I Equivalent)

9.1 Are the Organic Analysis reporting forms present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate [ ] ___
b. Matrix spikes and matrix spike duplicates
c. Blanks [ ] ___

d. Laboratory Control Samples [ ] ___

[ ] ___

9.2 Are the Reconstructed Ion Chromatograms, mass spec

FOIA_07123_0003552_0772

	YES	NO	N/A
tra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?	[ ]	___	
a. Samples and/or fractions as appropriate	[ ]	___	
b. Matrix spikes and matrix spike duplicates (Mass spectra not required)	[ ]	___	
c. Blanks	[ ]	___	
d. Laboratory Control Samples			
ACTION: If any data are missing, take action specified in 3.2 above.			
9.3 Are the response factors shown in the Quant Report?	[ ]	___	
9.4 Is chromatographic performance acceptable with respect to:			
Baseline stability?	[ ]	___	
Resolution?	[ ]	___	
Peak shape?	[ ]	___	
Full-scale graph (attenuation)?	[ ]	___	
Other:_____	[ ]	___	
ACTION: Use professional judgement to determine the acceptability of the data.	[ ]	___	
9.5 Are the lab-generated standard mass spectra of identified volatile compounds present for each sample?			
ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the Data Assessment. If spectra are missing, reject all positive data.	[ ]		
9.6 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing			calibratio
	[ ]		
9.7 Are all ions present in the standard mass			spec

FOIA_07123_0003552_0773

YES NO N/A

trum at a a. Sampl
relative intensity greater than 10% (of the most b. Blanl
abundant ion) also present in the sample mass spectrum?

9.8 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding [ ] ___ relative intensities in the reference spectrum?

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged ("N") - Presumptive evidence of the presence of the compound) or changed to non detected ("U") at the calculated detection limit. In order to be positively ident-ified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.

ACTION: When sample carry-over is a possibility, if instrument cross-contamination has affected any positive compound identification.

10.0 Tentatively Identified Compounds (TIC) (CLP Form I/TIC Equivalent)

10.1 If Tentatively Identified Compound were required for this project, are all Tentatively Identified Compound reporting forms present; and do listed TICs include scan number or retention time, estimated concentration and a qualifier? [ ]

NOTE: Add "N" qualifier to all TICs which have CAS number, if missing.

NOTE: Have the project officer/appropriate official check the project plan to determine if lab was required to identify non-target analytes (SW-846, page 8260B-23, Sect. 7.6.2).

10.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

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YES NO N/A

[ ] ___

[ ] ___

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by a CAS#.

NOTE: If TICs are present in the associated blanks take action as specified in section 6.2 above.

10.3 Are any priority pollutants listed as TIC compounds (i.e., an BNA compound listed as a VOA TIC)?

ACTION: 1. Flag with "R" any target compound listed as a TIC.

2. Make sure all rejected compounds are properly reported if they are target compounds.

10.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

10.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R". (Common lab contaminants: CO_2(M/E 44), Siloxanes (M/E 73), Hexane, Aldol Condensation Products, Solvent Preservatives, and related byproducts).

11.0 Compound Quantitation and Reported Detection Limits

11.1 Are there any transcription/calculation errors in organic analysis reporting form results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and average initial RRF/

FOIA_07123_0003552_0775

YES NO N/A

CF were used to calculate organic analysis reporting form result. Were any errors found?

___ [ ]

___ [ ]

[ ]

[ ] ___

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

11.2 Are the method CRQL's adjusted

FOIA_07123_0003552_0776

YES NO N/A

to reflect sample
dilutions and, for soils, sample moisture?

ACTION: If errors are large, take action as specified in section 3.2 above. [ ] ___

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original reporting form (if present) and substituting the data from the analysis of the diluted sample. Specify which organic analysis reporting form is to be used, then draw a red "X" across the entire page of all reporting forms that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant Reports) present for initial and continuing calibration?

ACTION: If any calibration standard data are missing, take action specified in section 3.2 above. [ ] ___

13.0 GC/MS Initial Calibration (CLP Form VI Equivalent)

13.1 Are the Initial Calibration reporting forms present and complete for the volatile fraction? [ ] ___

ACTION: If any calibration forms or standard raw data are missing, take action specified in section 3.2 above.

FOIA_07123_0003552_0777

YES NO N/A

13.2 Are all average RRFs > 0.050? analyte "J"

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list. If individual RRF values reported are below the listed values document in the Data Assessment. NOTE: The a

[ ] ___

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

ACTION: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF < 0.05, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

13.3 Are response factors stable over the concentration range of the calibration. The % relative standard deviation (%RSD) ≤ 15.0% as per SW-846, 8260B-17 Sect. 7.3.6.2.

NOTE: (Method Requirement) For the following CCC compounds, the %RSD values must be ≤ 30.0%. If %RSD values reported are > 30.0% document in the Data Assessment. [ ] ___

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

ACTION: Circle all outliers with a red pencil.

ACTION: If the % RSD is > 15.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, qualify all positive results for that

FOIA_07123_0003552_0778

YES NO N/A

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

13.4 Was the % RSD determined using RRF or CF?

If no, what method was used to determine the linearity of the initial calibration? Document any effects to the case in the Data Assessment.

13.5 Are there any transcription/calculation errors in the reporting of RRF or % RSD? (Check at least two values but if errors are found, check more.)

ACTION: Circle errors with a red pencil.

ACTION: If errors are large, take action as specified in section 3.2 above.

14.0 GC/MS Calibration Verification (CLP Form VII Equivalent)

14.1 Are the Calibration Verification reporting forms present and complete for all compounds of interest?

14.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument?

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

ACTION: If any forms are missing or no calibration verification standard has been analyzed twelve hours prior to sample analysis, take

action as specified in section 3.2 above.

FOIA_07123_0003552_0779

	YES	NO	N/A
f calibration verification data are not available, flag all associated sample data as unusable ("R").	[ ]	___	
14.3 Was the % D determined from the calibration verification determined using RRF or CF?			
If no, what method was used to determine the calibration verification? Document any effects to the case in the			
	[ ]	___	
	___	[ ]	
	[ ]	___	
Data Assessment.			
14.4 Do any volatile compounds have a % D (difference or drift) between the initial			

FOIA_07123_0003552_0780

YES NO N/A

and continuing RRF or CF which exceeds 20% (SW-846, page 8260B-19, section 7.4.5.2).

NOTE: (Method Requirement) For the following CCC compounds, the %D values must be ≤ 20.0%. If %D values reported are > 20.0% document in the Data Assessment. ___ [ ]

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

ACTION: Circle all outliers with a red pencil.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated, "J". When %D is above 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

14.5 Do any volatile compounds have a RRF < 0.05?

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list for each calibration verification. If average RRF values reported are below the listed values document in the data assessment. ___ [ ]

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

ACTION: Circle all outliers with a red pencil.

ACTION: If RF

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YES NO N/A

NOTE: The above data qualification action applies regardless of method requirements.

14.6 Are there any transcription/calculation errors in the reporting of %D between initial and continuing RRFs/CFs?
(Check at least two values but if errors are found, check more).

ACTION: Circle errors with a red pencil.

ACTION: If errors are large, take action as specified in section 3.2 above.

15.0 Internal Standards (CLP Form VIII Equivalent)

15.1 Are the internal standard areas on the internal standard reporting forms of every sample and blank within the upper and lower limits (-50% to + 100%) for each initial mid-point calibration (SW-846, 8260B-20, Sect. 7.4.7)?

ACTION: If errors are large or information is missing, take action as specified in section 3.2 above.

ACTION: List each outlying internal standard below.

Sample ID	IS #	Area Lower Limit	Upper Limit
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

(Attach additional sheets if necessary.)

ACTION: 1. If the internal standard area count is outside the upper or lower

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YES NO N/A

itated with this internal standard.

2. Do not qualify non-detects when the associated IS are counts area > + 100%.

3. If the IS area is below the lower limit

___ [ ]

[ ] ___

< - 50%), qualify all associated non-detects

4. If extremely low area counts are reported (<

etects as unusable "R" and positive results

15.2 Are the retention times of

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YES NO N/A

all internal standards within 30 seconds of the associated initial mid-point calibration standard (SW-846, 8260B-20, Sect. 7.4.6)?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

duplicates and calculate the relative percent difference.

16.0 Field Duplicates

16.1 Were any field duplicates submitted for volatile analysis?

ACTION: Compare the reported results for field

ACTION: Any gross variation between field duplicate results must be addressed in the Data Assessment. However, if large differences exist, take action specified in section 3.2 above.

[ ] ___

[ ] ___

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DEFINITIONS

Acronyms:

BFB - bromofluorobenzene
BNA - base neutral acid
CCC - calibration check compound
CF - calibration factor
CLP - contract laboratory program
CRQL - contract required quantitation limit
% D - percent difference or percent drift
GC/MS - gas chromatography/mass spectroscopy
IS - internal standard
l - liter
LCS - laboratory control sample
Kg - kilograms
m - meter
mm - millimeter
MS - matrix spike
MSD - matrix spike duplicate
m/z - mass to charge ratio
QC - quality control
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
RRT - relative retention time
RSD - relative standard deviation
RT - retention time
SDG - sample delivery group
SOP - standard operating procedure
SPCC - system performance check compound
TIC - tentatively identified compound
TCLP - toxicity characteristic leach procedure
ug - micrograms
VOA - volatile organic acid

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DEFINITIONS

Data Qualified Definitions:

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".

NJ - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

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VALIDATION SOP

Cyanide
USEPA Region 2 SOP HW-2
Rev. 13

Validation of Metals for the Contract Laboratory Program (CLP) based on SOW ILMO5.3 (SOP Revision 13)

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Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

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I.0 Scope

I.I This Standard Operating Procedure (SOP) applies to the evaluation of Routine Analytical Services (RAS) inorganic data generated in accordance with the EPA Contract Laboratory Program (CLP) protocols.

1.2 This Region 2 inorganic data validation SOP is used to determine the usability of analytical data generated from water and soil/sediment samples collected from Superfund sites in EPA Region 2.

1.3 Data should be generated and validated in accordance with the site specific Project Quality Objectives (PQOs) developed prior to the sample collection event. This SOP can be customized to validate the data according to the site specific PQOs. If the site specific DQOs are not available, this SOP must be used in its entirety.

1.4 This SOP is based, for the most part, upon analytical and quality assurance requirements specified in the Statement of Work SOW-ILM05.3, as well as in the final (October 2004) of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. The SOP Checklist, Appendix A.1, provides guidance in conducting the data validation. The result of the use of this SOP is a **Total Review** of the data: **Technical plus Contract - Compliance Review.**

2.0 Contract Compliance Review

This type of review is the first step in data validation which is carried out to ensure that the CLP laboratory has analyzed the environmental samples in accordance with the Statement of Work (SOW), and provided a data package which is both complete and compliant. This means that laboratory's procedures were performed exactly as specified in the CLP Statement of Works (SOW) and the data package contains all the deliverables including the information required under the contract.

2.1 Completeness

The data validator must check the entire data package to ensure that all deliverables required under the CLP contract are present and legible. In addition, copies of the Contract Compliance Screening (CCS) report, re-submittal from the laboratory, and Regional documentation should also be present in the data package. In Region 2, the data package completeness check is currently performed by the Regional Sample Control Coordinator (RSCC)for each Sample Delivery Group (SDG). The data package is not released to the data validator until all the required deliverables are received
from the laboratory.

2.2 Compliance

The data validator must check to ensure that all steps from sample receipt through sample preparation, analysis, data calculation and reporting are documented, and the information/data required under the contract is present in the appropriate reporting Forms and laboratory logs.

2.3 Contract Compliance Screening (CCS)

This screening step essentially checks the data package for the Completeness and Compliance requirements, and is performed by the Sample Management Office (SMO) currently operated by Computer Sciences Corporation (CSC), an EPA contractor. The CCS Report outlines the incomplete and non-compliant items as "Defects" in the data package, and is sent to the laboratory which is required to

provide additional or missing information/data required under the contract. The CCS Report for each SDG is transmitted electronically by the SMO to the Regional office. The CCS Report is intended to aid the data validator in locating any problems, both corrected and uncorrected. The incorrect original deliverable(s)of the data package must be replaced by the re-submittal(s)received from the laboratory in response to the CCS Report. The data validation should, however, be carried out even if the CCS Report is not available.

Web-based CCS is available for CLP laboratories to check their data prior to its delivery to EPA.

3.0 **Technical Review**

Technical review of the RAS data is carried out on the complete and compliant data to ensure its **validity** (i.e., data is of known quality and scientifically valid) and **usability** (i.e., data set is sufficiently complete and of sufficient quality to support a decision or an action described in the specific objectives of a data collection activity). The technical review process provides information on analytical limitations of data, if any, based on specific Quality Assurance/Quality Control (QA/QC) criteria. This is accomplished by performing an in-depth review of both the field deliverables which document the field sampling activities, and the laboratory analytical data deliverables which document the laboratory activities carried out to generate the reported data. Essentially, the validator shall first ensure that the data package is complete and compliant. The validator shall then evaluate data/information on all these deliverables (Final data sheets, Forms for QC analyses Chain-of-Custody/Traffic Report Forms, raw data, etc.) against the QA/QC acceptance criteria specified in the SOP "Checklist" (Appendix A.1). The validator must answer each question in the " Checklist" and take an appropriate action as required under "Action" to qualify the data. As a result of the technical review, the data validator may qualify some of the data as **rejected** or as **estimated.** The data validator shall write a **Data Review Narrative** documenting the qualified data and the reason(s) for the qualification.

3.1 If the **raw data** necessary to support the reported results are not provided, the data validation must not be performed. The laboratory must be contacted to obtain missing raw data.

3.2 If batch quality control analyses are performed on samples other than **site specific samples**, data must not be validated
or at best be considered as estimated. The data user must be notified of this action.

3.3 **QA/QC Acceptance Criteria**

In order that reviews be consistent among reviewers, QA/QC protocol (stated in Appendix A.I) should be strictly adhered to. If a lab provides more than one set of QC analyses or more than one particular QC analysis for an SDG, the validator shall use the worst QC analysis to evaluate the SDG data. Professional judgement should only be used in the rare instances not addressed in the "Checklist".

3.4 **Data Validation Flags**

Three types of data validation flags (J, R & U) are used in Region 2 to qualify the data.

3.4.1 **Flag "R" indicates Rejected Data**

Sample results determined to be unacceptable must preferably be lined over and flagged " R" with a red pencil only on the Inorganic Analysis Data Sheets (CLP Form I's). Data rejected on the basis of an unacceptable QC analysis should be excluded from further review or consideration. Data are rejected when associated QC analysis results exceed the expanded control limits of the QC criteria. The rejected data are known to contain significant errors based on documented information. The data user **must not** use the rejected data to make environmental decisions.

3.4.2 **Flag "J" indicates Estimated Data**

Sample results determined to be estimated must be flagged "J" with a red pencil only on the CLP Form I's. Data are flagged (J) when a QC analysis falls outside the primary acceptance limits. The qualified "J" data are not excluded from further review or consideration. However, only one flag (J) is applied to a sample result even though several associated QC analyses may fail. The "J" data may be biased high or low.

3.4.3 **Flg "U" indicates Non-Detects**

Sample results ≥ MDL associated with a contaminated blank are flagged "U" with a red pencil only on Form I's.

4.0 **Contractual Qualifiers**

The CLP laboratory applies contractual qualifiers on all Form I'S and the QC Forms when QC analyses are outside the control limits. These qualifiers are not applied on the Lotus or XLS spreadsheets with the exception of U and J. The contractual qualifiers and their meanings are as follows:

N : This qualifier indicates the lack of accuracy in the reported result, and is applied when matrix spiked sample recovery is outside the control limits.

E : This qualifier indicates the presence of interference, and is applied when the ICP serial dilution analysis is outside the control limits.

* : This qualifier indicates the lack of precision, and is applied to sample results on Form I's and Form VI when the Lab Duplicate analysis is outside the control limits.

U : This is a concentration qualifier that laboratory applies to a non-detected result which is essentially less than the Method Detection Limit(MDL). A non-detected result of an analysis is indicated by the Contract Required Quantitation Limit (CRQL) of that analyze suffixed with "U".

J : This is a concentration qualifier that the laboratory applies to a positive result below the CRQL(i.e.,≥MDL but <CRQL).

NOTE: The laboratory qualifiers are crossed out and replaced with the appropriate data validation qualifiers (J, R or U) by the data validator.

4.0 Rounding Rule

The data reviewer must follow the standard practice to round off percent recoveries on the QC reporting forms.

5.0 Data Review Narrative (Appendix A.2)

The data review narrative should be written using the format of Appendix A.2. The narrative should indicate the QC analyses outside the acceptance limits and the actions taken to qualify the associated data. The narrative should be prepared on a Personal Computer or a typewriter. If hand-written, under no circumstances should a pencil be used to write the narrative. The Data Review Narrative should be written in four (4) Sections: (i)Data Case Description, (ii)Complete SDG File (CSF) Audit Section, (iii) Technical Review Section, and (iv) Contract-Problems/Non-Compliance Section.

5.1 Data Case Description Section

The data validator must briefly describe the data case in this Section, outlining important information such as the number of samples, their matrix, sampling date(s), analysis (TAL metals, mercury or cyanide), samples used for QC analyses, Field Blank(s), Field Duplicates, etc.

5.2 Complete SDG File (CSF) Audit Section

The data validator must perform an audit on each SDG in the data package to ensure that all SDG-specific documents (sampling, samples shipping and receiving, telephone contact logs, etc.) are present in the data case. The audit shall also discover any discrepancy in the deliverables. In Region 2, this audit is currently performed by the ESAT data validator and its findings reported under “Comments” on a CSF inventory checklist. The validator informs the CLP Project Officer (PO) of the missing or additional information/deliverable required for data validation. The PO then contacts the lab for the desired deliverable/information. The findings of the CSF audit are reported in the CSF Section of the Data Review Narrative (Appendix A.2).

5.3 Technical Review Section

The data validator shall report in this Section only the rejected (R) and estimated data (J) and the data rendered non-detects (U) as a result of technical review. It is imperative that the data reviewer highlights (i) QC analysis criteria applied to reject (R) or flag (J, U) the data, (ii) Samples rejected (R) or flagged (J, U), and (iii) the QC analysis out of control limits. The rest of the data that are not qualified (rejected or estimated) are not reported in this Section, and should be considered **fully useable.**

5.4 Contract-Problems/Non-Compliance Section

All the CLP non-compliant items detected during data review must be reported in this Section.

6.0 Computer-Aided Data Review and Evaluation (CADRE)

CADRE is a computer program that performs semi-automated Quality Assurance (QA) and Quality Control (QC) checks of results from the chemical analysis of soil and water samples according to the CLP protocols. After the CADRE data qualification is complete, a Lotus 1,2,3 spreadsheet or an XLS spreadsheet with data validation qualifiers (R,J,U) is generated for each SDG. Currently, Sample Management Office (SMO) performs this task using Data Assessment Tool (DAT), a software-driven process, and forwards to the Regions the customized electronic spreadsheets (Lotus 1,2,3 or XLS spreadsheet) and QC reports via the DART (Data Assessment Rapid Transmittal) system. Manual data validation is performed in conjunction with electronic data validation which can only be done by a trained and experienced data validator. The manual data review complements CADRE's findings to complete an assessment of data quality in a shorter time than by a solely manual process. The data validator must review the XLS or Lotus 1,2,3 spreadsheet against Form I's to ensure that the same results on Form I's and the Spreadsheet are qualified with the same data validation qualifiers. The spreadsheet for each SDG is provided with the Data Review Narrative.

7.0 Performance Evaluation Sample(PES)Based Data Validation Strategy

7.1 Scope and Summary

This strategy offers the use of Performance Evaluation Samples (PES) in the data validation process as a means of ensuring the quality of the CLP data while significantly reducing the validation time. The single blind PES provided by EPA (or any other reputable firm) is analyzed with samples of each matrix in a Sample Delivery Group (SDG). A software program (e.g.,PEAC TOOLS, SPS Web or equivalent)is used to determine whether or not the PES results fall within the previously statistically determined acceptance limits ("Action Low" and "Action High")for the Contaminants of Concern (**COC**). The PES results falling within the Action Limits are considered as acceptable results and may be designated as "Passed" analytes, and results of the analytes falling outside the Action Limits are considered as unacceptable and may be designated as "Failed" analytes. In either case ("Passed" Analytes or "Failed" analytes), the associated data is validated according to the Region 2 data validation SOP HW-2 in conjunction with the latest version of the WinCadre QC reports. The following strategy (procedure) is used:

7.2 "Passed" COC

If the COC in an SDG are within statistically generated Action Limits, the data validation is conducted according
to QC analyses indicated by check marks (☐/)in the "Review COC For" column of the Table I. The SDG samples are validated using the Region 2 data validation SOP in

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conjunction with the latest version of the WinCADRE QC reports. The validation flags (J, R, U) are applied on Form I's as well on the CADRE Lotus 1,2,3 or XLS spreadsheet. Corrections, if needed, are
then made on the Lotus or XLS spreadsheet to ensure that all results on Form I's carry the same data validation and concentration flags as are on the Lotus or XLS Spreadsheet.

7.3 **"Failed" COC**

If the COC in an SDG are not within the statistically generated Action Limits, the data validation is conducted according to the data validation SOP QC Criteria indicated by check marks (☐/)in the "Review COC For" column of Table II. The SDG samples are validated using the Region 2 data validation SOP in conjunction with the latest version of the WinCADRE QC reports. The data validation flags (J,R,U) are applied on Form I's as well on the CADRE Lotus 1,2,3 or XLS Spreadsheet. Corrections, if needed, are then made on the Lotus or XLS spreadsheet to ensure that all results on Form I's carry the same data validation and concentration flags as are on the Lotus or XLS Spreadsheet.

7.4 **COC "Not Evaluated"**

Acceptance limits for the analytes not present/spiked in the PE sample are not provided on the PES Scoring Evaluation Report. Such analytes will be marked as "Not Evaluated" in the PES Evaluation Column. These analytes will be validated much the same way as the "Failed Analytes".

The failed analytes and the analytes not present/spiked in the PE sample require data validation according to the QC criteria specified in Table II, and are identified by the TOPO in the TDF for the Case/SDG.

Table I

Passed PES - All Contaminants of Concern are within the limits
(Action Low ≤ PES Result ≤ Action High)

QC Criteria	Review COC for
Holding Time & Preservation	☐ /
Initial Calibration	
Initial Calibration Verification	
CRQL Standard	☐ /
Blanks-Initial & Continuing	
Preparation Blank	
ICP Interference Check Sample	
Pre- Digestion/Distillation Matrix Spike	
Post Digestion Spike	
Laboratory Duplicate	
Field Duplicates Comparison	☐ /
Lab Control Sample	
ICP Serial Dilution	
Field Blank Contamination	☐ /
Percent Solids	☐ /
Transcription/Computation Check	
Raw Data	
Total vs. Dissolved Concentrations Comparison	☐ /

- The CSF (Complete SDG File) audit will be completed before the PES validation strategy is applied.
- Comparison of the Lotus or XLS Spreadsheet must be after the PES validation strategy is applied. The Contract
- Compliance can be checked after the PES validation strategy is applied.

Table II
Failed PES - Contaminants of Concern are not within the limits
(PES Result ≤ Action Low, PES Result ≥ Action High **OR** The Limits Not Established)

QC Criteria	Review COC for
Holding Time & Preservation	☐ /
Initial Calibration	
Initial Calibration Verification	
CRQL Standard	☐ /
Blanks-Initial & Continuing	
Preparation Blank	☐ /
ICP Interference Check Sample	
Pre- Digestion/Distillation Matrix Spike	☐ /
Post Digestion Spike	
Laboratory Duplicate	☐ /
Field Duplicates Comparison	☐ /
Lab Control Sample	☐ /
ICP Serial Dilution	☐ /
Field Blank Contamination	☐ /
Percent Solids	☐ /
Transcription/Computation Check	☐ /
Raw Data	
Total vs. Dissolved Concentrations Comparison	☐ /

- The CSF (Complete SDG File) audit will be completed before the PES validation strategy is applied.
- Comparison of the Lotus or XLS Spreadsheet must be after the PES validation strategy is applied.
- The Contract Compliance can be checked after the PES validation strategy is applied.

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8.0 **Sampling Trip Report**

The sampler prepares a Sampling Trip Report for each sampling event and sends it to the RSCC. The report provides details of all activities performed for each sampling event on the Superfund site. It also lists the field QC samples such as Field Duplicates, Field/Rinse Blanks, sampling time and date for each sample, and samples associated with each field/rinse blank. The validator must use this information to evaluate the Field Duplicate pairs as well as the samples associated with contaminated Field/Rinse Blanks.

9.0 **Telephone Record Log (Appendix A.3)**

A Telephone Record Log (Appendix A.3) must be written by the data validator when a deliverable is missing or a clarification is needed about a lab procedure. The data validator should outline a basic profile of the Case on the Telephone Record Log Form, clearly indicating the reason(s) for inquiry and forward this Form to CLP PO/TOPO who will contact the lab to receive the missing document or information. The original Telephone Record Log is kept in the data package and a copy attached to the Data Review Narrative.

10.0 **Request for Re-Analysis (Appendix A.6)**

Data validator must note all items of contract non-compliance in the Data Review Narrative. If holding times and sample storage times have not been exceeded, the Project Officer (PO) may request re-analysis if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-Analysis Request/Approval Record" form (Appendix A.4).

11.0 **CLP Data Assessment Summary Form (Appendix A.7)**

Fill in the total number of analytes performed by different methods and the number of analytes rejected (R) or flagged (J) as estimated due to corresponding quality control criteria. Place an "X" in boxes wherever analyses were not performed, or criteria do not apply.

12.0 **Data Review Log:**

It is recommended that the data validator maintain a log of the reviews completed to document:

a. Case number
b. SDG # (s)
c. number of samples
d. matrix of samples
e. contract laboratory
f. site name
g. start-date of the data case review
h. completion-date of the data case review
i. actual hours spent
j. reviewer's signature

13.0 **Record of Communication** -
This is a Regional document prepared and provided by the RSCC for each data package. The ROC indicates the Case #, site name, samples and sample matrix and the laboratory name. The presence of a ROC in a data package is an indication that the package has been reviewed by the RSCC for completeness and is ready for data validation.

14.0 **Forwarded Paperwork**
Upon completion of review, the following are to be forwarded to EPA for final review:

a. Data package
b. Completed data assessment checklist (Appendix A.1,original)
c. Original and a copy of completed data review narrative Appendix A.2)
d. CLASS Contract Compliance Screening (CCS) report
e. Telephone Record Log (Appendix A.3)
f. Field Duplicates Form (Appendix A.4)
g. Total/Dissolved Concentrations Form (Appendix A.5)
h. CLP Re-analysis Request/Approval Record Form (Appendix A.6)
i. Data Assessment Summary Form (Appendix A.7)
j. CADRE Spreadsheet on a computer diskette.

ACRONYMS

AA Atomic Absorption
AOC Analytical Operations/Data Quality Center
CADRE Computer-Aided Data Review and Evaluation
CCB Continuing Calibration Blank
CCS Contract Compliance Screening
CCV Continuing Calibration Verification
CLP Contract Laboratory Program
CO Contracting Officer
COC Contaminants of Concern
CRI CRQL Check Standard
CRQL Contract Required Quantitation Limit
CSF Complete SDG File
CVAA Cold Vapor AA
DART Data Assessment Rapid Transmittal
DAT Data Assessment Tool
DF Dilution Factor
DQO Data Quality Objective
ICB Initial Calibration Blank
ICP Inductively Coupled Plasma
ICP-AES Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS Inductively Coupled Plasma - Mass Spectrometry
ICS Interference Check Sample
ICV Initial Calibration Verification
LCS Laboratory Control Sample
LRS Linear Range Sample
MDL Method Detection Limit
NIST National Institute of Standards and Technology
OERR Office of Emergency and Remedial Response
OSWER Office of Solid Waste and Emergency Response
PB Preparation Blank
PE Performance Evaluation
%D Percent Difference
%R Percent Recovery
%RI Percent Relative Intensity
%RSD Percent Relative Standard Deviation
%S Percent Solids
PO Project Officer
QA Quality Assurance
QAPP Quality Assurance Project Plan
QC Quality Control
RPD Relative Percent Difference
RSCC Regional Sample Control Center

SDG Sample Delivery Group
SMO Sample Management Office
SOP Standard Operating Procedure
SOW Statement of Work
TAL Target Analyze List
TR/COC Traffic Report/Chain of Custody Documentation

Inorganic Target Analyze List And Contract Required Quantitation Limits (CRQLs)

Analyze	CAS Number	ICP-AES CRQL Water Ug/L	ICP-AES CRQL Soil mg/kg	ICP-MS CRQL Water Ug/L
Aluminum	7429-90-5	200	20	---
Antimony	7440-36-0	60	6	2
Arsenic	7440-38-2	10	1	1
Barium	7440-39-3	200	20	10
Beryllium	7440-41-7	5	0.5	1
Cadmium	7440-43-9	5	0.5	1
Calcium	7440-70-2	5000	500	-----
Chromium	7440-47-3	10	1	2
Cobalt	7440-48-4	50	5	1
Copper	7440-50-8	25	2.5	2
Iron	7439-89-6	100	10	----
Lead	7439-92-1	10	1	1
Magnesium	7439-95-4	5000	500	-----
Manganese	7439-96-5	15	1.5	1
Mercury	7439-97-6	0.2	0.1	---
Nickel	7440-02-0	40	4	1
Potassium	7440-09-7	5000	500	-----
Selenium	7782-49-2	35	3.5	5
Silver	7440-22-4	10	1	1
Sodium	7440-23-5	5000	500	-----
Thallium	7440-28-0	25	2.5	1
Vanadium	7440-62-2	50	5	1
Zinc	7440-66-6	60	6	2
Cyanide	57-12-5	10	2.5	----

Site:

Case #:

SDG #:

Samples: Soil Water

	YES	NO	N/A
A.I.I **Contract Compliance Screening Report**			
Present?	[___]	___	___
ACTION: If no, contact RSCC/PO.			
A.I.2 **Record of Communication (from RSCC)**			
Present?	[___]	___	___
ACTION: If no, request from the RSCC.			
A.1.3 **Sampling Trip Report**			
Present and complete?	[___]	___	___
ACTION: If no, contact RSCC/PO.			
A.I.4 **Chain of Custody/Sample Traffic Report**			
Present?	[___]	___	___
Legible?	[___]	___	___
Signature of sample custodian present?	[___]	___	___
ACTION: If no, contact RSCC/WAM/PO.			
A.I.5 **Cover Page**			
Present?	[___]	___	___
Is the Cover Page properly filled in and the verbatim signed by the lab manager or the manager's designee?	[___]	___	___
Do the sample identification numbers on the Cover Page agree with sample Identification numbers on:			
(a) Traffic Report Sheet?	[___]	___	___

	YES	NO	N/A
(b) Form I's?	[___]	___	___
Is the number of samples on the Cover Page the same as the number of samples on the Traffic Report sheet and the Regional Record of Communication (ROC) for the data Case?	[___]	___	___

ACTION:
If no for any of the above, prepare Telephone Record Log and contact RSCC/PO for re-submittal of the corrected Cover Page from the laboratory.

A.1.6 **SDG Narrative, DC-1 & DC-2 Form**

Is the SDG Narrative present?	[___]	___	___
Is Sample Log-In Sheet(Form DC-1) present and complete?	[___]	___	___
Is Complete SDG Inventory Sheet(Form DC-2) present and complete?	[___]	___	___

ACTION:
If no, write in the Contract-Problems/ Non-Compliance Section of the Data Review Narrative.

A.1.7 **Form I to XV**

A.1.7.1 Are all the Form I through Form XV labeled with:

Laboratory Name?	[___]	___	___
Laboratory Code?	[___]	___	___
RAS/Non-RAS Case No.?	[___]	___	___
SDG No.?	[___]	___	___

	YES	NO	N/A
Contract No.?	[___]	___	___

ACTION:
If no for any of the above, note under Contract Problem/Non-Compliance Section of the "Data Review Narrative" and contact PO for corrected Form(s) from the laboratory.

A.1.7.2 After comparing values on Forms I-IX against the raw data, do any computation/ transcription errors exceed 10% of the reported values on the Forms for:

(a) all analytes analyzed by ICP-AES?	___	[_]	___
(b) all analytes analyzed by ICP-MS?	_	[___]	_
(c) Mercury?	__	[_]	_____
(d) Cyanide?	_	[_]	_

ACTION:
If yes, prepare Telephone Record Log and contact CLP PO/TOPO for the corrected data from the laboratory.

A.1.8 Raw Data

Data shall not be validated without the hard/electronic copies of the associated raw data for samples and QC samples.

A.1.8.1 Digestion/Distillation Log

Digestion Log for ICP-AES (Form XII)present?	[___]	___	___
Digestion Log for ICP-MS (Form XII) present?	[___]	___	___
Digestion Log for mercury (Form XII) present?	[___]	___	___
Distillation Log for cyanide (Form XII) present?	[___]	___	___

Are pH values for metals and

	YES	NO	N/A
cyanide reported for each aqueous sample?	[___]	_	_
Are percent solids calculations present for soils/sediments?	[_]	___	___
Are preparation dates present on the sample preparation logs/bench sheets?	[___]	_	_

NOTE:
Digestion/Distillation log must include weights, volumes, and dilutions used to obtain the reported results.

A.1.8.2 Is the analytical instrument real-time printouts present for:

ICP-AES?	[___]	_	_
ICP-MS?	[___]	___	___
Mercury?	[_]	_	_
Cyanide?	[___]	___	___

Are all laboratory bench sheets and instrument raw data printouts necessary to support all sample analyses and QC operations:

Legible?	[_]	_	_
Properly labeled?	[_]	_	_

Are all field samples, QC samples and field QC samples present on:

Digestion/Distillation log?	[___]	___	___
Instrument Printouts?	[_]	_	_

ACTION:
If no for any of the above questions in Section A.1.8.1 and Section A.1.8.2, write Telephone Record Log and contact TOPO/PO for re-submittal from the laboratory.

YES NO N/A

A.1.9 **Technical Holding Times**: (Aqueous and soil samples)
(Examine sample Traffic Reports and digestion/distillation logs to determine the holding time from the sample collection date to the sample preparation date.)

A.1.9.1 Cyanide distillation(14 days)exceeded? _ [_] _

Mercury analysis(28 days) exceeded? _ [___] _

Other Metals analysis(180 days)exceeded? ___ ___ [_] _

ACTION:
If yes, reject (R) and red-line non-detects and flag as estimated (J)results ≥ MDL even if sample(s) was preserved properly.

NOTE:
In addition to qualifying the data, a list of all samples and analytes which exceeded the holding times must be prepared. Report for each sample the number of days that were exceeded. (Subtract the sample collection date from the sample preparation date). Attach this list to the data review narrative.

A.1.9.2 Is pH of aqueous samples for:

Metals Analysis ≤ 2? [___] ___ ___

Cyanide Analysis ≥ 12? [_] _ _

ACTION:
If no for any of the above, flag non-detects as "R" and detects as "J".

A.1.9.3 Is the cooler temperature ≤ 10 C°? [_] _ _

ACTION:
If cooler temperature is >10 ^{0}C , flag non-detects as "UJ" and detects as "J".

A.1.10 **Final Data Correctness - Form I**

A.1.10.1 Are Form I's for all samples

	YES	NO	N/A
present and complete?	[___]	___	___

ACTION:
If no, prepare Telephone Record Log and contact CLP PO/TOPO for submittal from the laboratory.

A.1.10.2 Verify there are no calculation and transcription errors in the results reported on Form I's. Circle on each Form I all results that are incorrect.

Is the calculation error less than 10% of the correct result?	[___]	_	_
Are results on Form I's reported in correct units (ug/L for aqueous and MG/KG for soils)?	[___]	___	___
Are results on Form I'S reported by correct significant figures?	[___]	___	__
Are soil sample results on Form I's corrected for percent solids?	[___]	___	___
Are all "less than MDL" values reported by the CRQLs and coded with "U"?	[___]	___	___
Are values less than the CRQLs but greater than or equal to the MDLs flagged with "J"?	[___]	___	___
Are appropriate contractual quality control and Method qualifiers used?	[___]	____	___

ACTION:
If no for any of the above questions, prepare Telephone Record Log, and contact CLP PO/TOPO for corrected data.

A.1.10.3 Do EPA sample identification numbers and the corresponding laboratory sample identification numbers match on the Cover Page, Form I's and in the raw data? [___] ___ ___

Was a brief physical description

	YES	NO	N/A
of the samples before and after digestion given on the Form I's?	[___]	___	___
Was any sample result outside the mercury/cyanide calibration range or the ICP-AES/ICP-MS linear range diluted and noted on the Form I?	[___]	___	___

ACTION:
If no for any of the above, note under the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.11 **Initial Calibration**

A.1.11.1 Is a record of at least 2 point (A blank and a standard)calibration present for ICP-AES analysis?	[___]	___	___
Is a record of at least 2 point (a blank and a standard)calibration present for ICP-MS analysis?	[___]	___	___
Is a record of at least 5 point calibration (a blank & 4 standards)present for Hg analysis?	[___]	___	___
Is a record of at least 4 point calibration (a blank & 4 standards)present for cyanide?	[___]	___	___

ACTION:
If incomplete or no initial calibration was performed, reject (R) and red-line the associated data (detects & non-detects).

Is one initial calibration standard at the CRQL level for cyanide and mercury?	[___]	___	___

ACTION:
If no, write in the Contract Problem/ Non-Compliance Section of the Data Review Narrative.

A.1.11.2 Is the curve correlation coefficient $\geq$ 0.995 for:			

		YES	NO	N/A
	Mercury Analysis?	[___]	___	___
	Cyanide Analysis?	[___]	___	___
	ICP-AES(more than 2 point Calib.)?	[___]	___	___
	ICP-MS (more than 2 point calib.)?	[___]	___	___

ACTION:
If no, qualify the associated sample results ≥ MDL as estimated "J" and non-detects as "UJ".

NOTE:
The correlation coefficient shall be calculated by the data validator using standard concentrations and the corresponding instrument response (e.g. absorbance, peak area, peak height, etc.).

A.1.12 **Initial and Continuing Calibration Verification- Form IIA**

A.1.12.1	Present and complete for every metal and cyanide?	[___]	___	___
	Present and complete for ICP-AES and ICP-MS when both these methods were used for the same analyte?	[___]	___	___

ACTION:
If no for any of the above, prepare a Telephone Record Log and contact PO/TOPO for re-submittal from the laboratory.

A.1.12.2	Was a Continuing Calibration Verification performed every 10 samples or every 2 hours whichever is more frequent?	[___]	___	___

ACTION:
If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative.

A.1.12.3	Was an ICV or a mid-range standard distilled and analyzed with each batch of cyanide samples?	[___]	___	___

		YES	NO	N/A
	ACTION: If no for any of the above, write in the Contract-Problem/Non-Compliance Section of the Data Review Narrative and qualify results ≥ MDL as estimated (J).			
A.1.12.2	Circle on each Form IIA all percent recoveries that are outside the contract windows.			
	Are ICV/CCVs within control limits for:			
	Metals - 90-110%R?	[___]	___	___
	Hg - 80-120%R?	[___]	___	___
	Cyanide - 85-115%R?	[___]	___	___

ACTION:
If no, qualify all samples between a previous technically acceptable CCV standard and a subsequent technically acceptable CCV standard as follows as follows:

Qualify as estimated (J) all detects and non-detects, if the ICV/CCV %R is between 75-89%(65-79% for Hg; 70-84% for CN). Qualify only positive results(≥ MDL) as "J" if the ICV/CCV %R is between 111-125%(121-135% for Hg;116-130% for CN). Reject (R) and red-line only
detects if the recovery is greater than 125% (135% for Hg; 130% for CN). Reject (R) and red-line all associated results (hits and non-detects)if the recovery is less than 75%(65% for Hg;70% for CN).

NOTE:
For ICV that does not fall within the acceptance limits, qualify all samples reported from the analytical run.

A.1.12.3	Was the distilled ICV or mid-range standard for cyanide within acceptance limits (85-115%)?	[___]	___	___

ACTION:
If no, Qualify all cyanide results ≥ MDL as "J".

A.1.13 **CRQL Standard Analysis - Form IIB**

A.1.13.1 For each ICP-AES run, was a CRI

	YES	NO	N/A
(CRQL or MDL when MDL > CRQL) standard analyzed? **(Note:CRI is not required for Al, Ba, Ca, Fe, Mg, Na and K.)**	[___]	___	___
For each ICP-MS run, was a CRI (CRQL or MDL when MDL > CRQL) standard analyzed for each mass/isotope used for the analysis?	[___]	___	___ ___
For each mercury run, was a CRQL standard analyzed?	[___]	___	___
For each cyanide run, was a CRQL standard analyzed?	[___]	___	___

ACTION:
If no for any of the above, write this deficiency in the Contract Problems/ Non-Compliance Section of the Data Review Narrative, inform CLP PO and flag results in the affected ranges (detects <2xCRQL)as J and non-detects UJ.

The affected ranges are:
ICP-AES Analysis - *True Value ± CRQL
ICP-MS Analysis - *True Value ± CRQL
Mercury Analysis - *True Value ± CRQL
Cyanide Analysis - *True Value ± CRQL
*** True value of the CRQL Standard**

A.1.13.2	Was a CRQL standard analyzed after the ICV/ICB, before the final CCV/CCB and once every 20 analytical samples in the analytical run for each analysis?	[___]	___	___

ACTION:
If no, write in the Contract Problem/ Non-Compliance Section of the "Data Review Narrative".

A.1.13.3 Circle on each Form IIB all percent recoveries that are outside the acceptance windows.

	YES	NO	N/A
Is the CRQL standard within control limits for:			
Metals(ICP-AES/ICP-MS)- 70 - 130%?	[___]	___	___
Mercury- 70 - 130%?	[___]	___	___
Cyanide - 70 - 130%?	[___]	___	___

ACTION:
If no, flag detects <2xCRQL as "J" and non-detects as "UJ" if the CRQL standard recovery is between 50-69%. Flag(J) only detects <2xCRQL if the recovery is between 131% and ≤180%. If the recovery is less than 150%, reject(R) and red-line non-detects and detects < 2xCRQL, and flag (J) detects between 2xCRQL and ICV/CCV. Reject and red-line only detects <2xCRQL and flag (J)detects ≥ 2xCRQL but < ICV/CCV if the recovery is > 180%.

NOTE:
1. Qualify all field samples analyzed between a previous technically acceptable analysis of the CRQL standard and a subsequent acceptable analysis of the CRQL standard
2. Flag (J) or reject (R) only the final sample results on Form I's when **Sample raw data** are within the affected ranges and the CRQL standard is outside the acceptance windows.
3. The samples and the CRQL standard must be analyzed in the same analytical run.

A.1.14 Initial and Continuing Calibration Blanks - Form III

	YES	NO	N/A
A.1.14.1 Present and complete for all the instruments used for the metals and cyanide analyses?	[___]	___	___
Was an initial Calibration Blank analyzed after ICV?	[___]	___	___
Was a continuing Calibration Blank analyzed after every CCV and every 10 samples or every 2 hours, whichever is more frequent?	[___]	___	___

Were the ICB & CCB values ≥ MDL but < CRQL reported on Form III and flagged "J" by

		YES	NO	N/A
	using MDLs from direct analysis(Preparation Method "NP1")? (Check Form III against the raw data)	[___]	___	___
	ACTION: If no, inform CLP PO/TOPO and make a note in the Contract-Problems/Non-Compliance Section of the "Data Review Narrative".			
A.1.14.2	Circle with red pencil on each Form III all Calib. Blank values that are: ≥ MDL but ≤ CRQL; > CRQL			
A.1.14.2.1	When MDL < CRQL, is any Calib. Blank value ≥ MDL but ≤ CRQL?	___	[___]	___
	ACTION: If yes, change sample results ≥ MDL but ≤ CRQL to the CRQL with a "U". Do not qualify non-detects.			
A.1.14.2.2	When MDL < CRQL, is any Calib. Blank value > CRQL?	___	[___]	___
	ACTION: If yes, reject (R) and red line the associated sample results > CRQL but <ICB/CCB Blank Result. Flag as "J" detects > ICB/CCB blank value but < 10xICB/CCB value. Change the sample results ≥ MDL but ≤ the CRQL to CRQL with a "U".			
A.1.14.2.3	Is any Calibration Blank value below the negative CRQL?	___	[___]	___
	ACTION: If yes, flag (J) as estimated all associated sample results ≥ CRQL but <10xCRQL.			

NOTE:

1. For ICB that does not meet the technical QC Criteria, apply the action to all samples

	YES	NO	N/A

reported from the analytical run.
2. For CCBs that do not meet the technical QC criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.,

A.1.15 **Preparation Blank - FORM III**

NOTE:The Preparation Blank for mercury is the same as the calibration blank.

A.1.15.1 Was one Preparation Blank prepared with and analyzed for:

Each Sample Delivery Group (SDG)?	[___]	___	___
Each batch of the SDG samples digested/distilled?	[___]	___	___
Each matrix type?	[___]	___	___
All instruments used for metals and cyanide analyses?	[___]	___	___

ACTION:
If no for any of the above, flag as estimated (J) all the associated positive data <10xMDL for which the Preparation Blank was not analyzed.

NOTE:
If only one blank was analyzed for more than 20 samples, then the first 20 samples analyzed are not estimated(J),but all additional samples must be qualified (J).

A.1.15.2 Circle with red pencil on each Form III all Prep. Blank values that are:

$\geq$ MDL but $\leq$ CRQL, and

> CRQL

A.1.15.2.1 When MDL < CRQL, is any preparation blank value $\geq$ MDL but $\leq$ CRQL? ___ [___] ___

ACTION:
If yes, change sample result $\geq$ MDL

		YES	NO	N/A
	but ≤ CRQL to CRQL with a "U".			
A.1.15.2.2	When the MDL ≤ CRQL, is any Preparation Blank value greater than its CRQL?	___	[___]	___
	If yes, is the Prep. Blank value greater than the value of the associated Field Blank collected and analyzed with the SDG samples?	___	[___]	___
	If yes, is the lowest concentration of that analyte in the associated samples less than 10 times the Preparation Blank value?	___	[___]	___

ACTION:
If yes, reject (R) and red-line all associated sample results greater than the CRQL but less than the Prep.Blank value. Flag as "J" detects > Prep. Blank value but <10xPrep.Blank. If the sample result ≥ MDL but ≤ CRQL, replace it with CRQL-U.

If the Prep. Blank value is less than the same analyte value in the Field Blank, do not qualify the sample results due to the Prep. Blank criteria.

NOTE:
Convert soil sample result to mg/Kg on wet weight basis to compare with the soil Prep. Blank result on Form III.

A.1.15.2.3	Is the Prep. Blank concentration below the negative CRQL?	___	[___]	___

ACTION:
If yes, flag (J) all associated sample results less than 10xCRQL. Qualify non-detects as estimated (UJ).

A.1.15.2.4	When the MDL is greater than the CRQL, is the preparation blank concentration on Form III greater than two times the MDL?	___	[___]	___

ACTION:

		YES	NO	N/A
	If yes, reject (R) and red-line all positive sample results with sample raw data less than 10 times the Preparation Blank value.			

A.1.16 **ICP-AES/ICP-MS Interference Check Sample (ICS)- Form IV**

NOTE: Not required for CN, Hg, Al, Ca, Fe and Mg.

A.1.16.1	Present and complete?	[___]	___	___
	Was ICS analyzed at the beginning and end of each analytical run, and once for every 20 analytical samples?	[___]	___	___
	Was ICS analyzed at the beginning of the ICP-MS analytical run?	[___]	___	___

ACTION:
If no, flag as estimated (J) all sample results.

A.1.16.2 **ICP-AES Method**

A.1.16.2.1	**ICSA Solution:** For ICP-AES, are the ICSA "Found" analyte values within the control limits ± of CRQL of the true/established mean value?	[___]	___	___
	If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSA Solution on Form IV?	[___]	___	___

ACTION:
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated only sample results ≥MDL

	YES	NO	N/A
for which the ICSA "Found" value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA "Found" value is less than (True value-CRQL), flag non-detects as "UJ" and detects as "J".			
A.1.16.2.3 **ICSAB Solution** For ICP-AES, are all analyte results in ICSAB within the control limits of 80-120 of the true/established mean value?	[___]	___	___
If no for any of the above, is the sample concentration of Al, Ca, Fe, or Mg in the same units (ug/L or MG/KG) greater than or equal to its respective concentration in the ICSAB Solution on Form IV?	[___]	___	___

ACTION:
If yes, apply the following action to all samples analyzed between a previous technically acceptable analysis of the ICS and a subsequent technically acceptable analysis of the ICS in the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79%, qualify sample results ≥ MDL as "J" and non-detects as "UJ". Reject (R) and red-line all sample results (detects & non-detects) for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%, reject (R) and red-line only positive results.

A.1.16.3 **ICP-MS Method**

	YES	NO	N/A
A.1.16.3.1 **ICSA Solution:** For ICP-MS, are the ICSA "Found" analyte values within the control limits of ±CRQL of the true/established mean value?	[___]	___	___

ACTION:
If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated only sample results ≥ MDL if the ICSA "Found" value is greater than (True value+CRQL). Do not qualify non-detects. If the ICSA "Found" value is less than (True value-CRQL), flag the associated sample detects as "J" and non-detects as "UJ".

		YES	NO	N/A
A.1.16.3.3	**ICSAB Solution** For ICP-MS, are all analyte results in ICSAB within the control limits of 80-120% of the true/established mean value, whichever is greater?	[___]	___	___

ACTION:
If no, apply the following action to all samples reported from the analytical run:

Flag (J) as estimated those associated sample results ≥ MDL for which the ICSAB analyte recovery is greater than 120% but ≤ 150%. If the ICSAB recovery falls within 50-79% flag (J) as estimated the associated sample results ≥ MDL. Reject (R) and red-line those all sample detects and non-detects for which the ICSAB analyte recovery is less than 50%. If the recovery is above 150%,reject (R) and red-line only detects (≥ MDL).

A.1.17 **Spiked Sample Recovery: Pre-Digestion/Pre-Distillation)-Form V A**

Note: Not required for Ca,Mg,K,and Na(both matrices);Al and Fe (soil only)

A.1.17.1 Was Matrix Spike analysis performed:

	YES	NO	N/A
For each matrix type?	[___]	___	___
For each SDG?	[___]	___	___
On one of the SDG samples?	[___]	___	___
For each concentration range (i.e.,low, med., high)?	[___]	___	___
For each analytical Method (ICP-AES,ICP-MS, Hg, CN)used?	[___]	___	___
Was a spiked sample prepared and analyzed with the SDG samples?	[___]	___	___

ACTION:
If no for any of the above, flag as estimated(J)all the positive data for which a spiked sample was not analyzed.

NOTE:
If more than one spiked sample were analyzed for one SDG, then qualify the associated data based on the worst spiked sample analysis.

		YES	NO	N/A
A.1.17.2	Was a field blank or PE sample used for the spiked sample analysis?	___	[___]	___
	ACTION: If yes, flag (J) as estimated positive data of the associated SDG samples for which field blank or PE sample was used for the spiked sample analysis.			
A.1.17.3	Circle on each Form VA all spike recoveries that are outside the control limits (75-125%) that have sample concentrations less than four times the added spike concentrations.			
	Are all recoveries within the control limits when sample concentrations are less than or equal to four times the spike concentrations?	[___]	___	___
	NOTE: Disregard the out of control spike recoveries for analytes whose concentrations are greater than or equal to four times the spike added.			
	Are results outside the control limits (75-125%)flagged with Lab Qualifier "N" on Form I's and Form VA?	[___]	___	___
	ACTION: If no for any of the above, write in the Contract - Problems/Non-Compliance Section of the Data Review Narrative.			
A.1.17.4	**Aqueous**			
	Are any spike recoveries:			
	(a) less than 30%?	___	[___]	___
	(b) between 30-74%?	___	[___]	___
	(c) between 126-150%?	___	[___]	___
	(d) greater than 150%?	___	[___]	___
	ACTION: If the matrix spike recovery is less than 30%,reject (R) and red-line all associated aqueous data (detects & non-detects). If between 30-74%, qualify all associated aqueous data ≥ MDL as "J" and non-detects			

	YES	NO	N/A
as "UJ". If between 126-150%, flag (J) all data ≥ MDL as "J". If greater than 150%, reject (R) and red-line all associated data ≥ MDL.			
(**NOTE**:Replace "N" with "J", "R" as appropriate.)			

A.1.17.5 **Soil/Sediment**

Are any spike recoveries:

(a) less than 10%?	___	[___]	___
(b) between 10-74%?	___	[___]	___
(c) between 126-200%?	___	[___]	___
(d) greater than 200%?	___	[___]	___

ACTION:
If yes for any of the above, proceed as follows:

If the matrix spike recovery is less than 10%,reject (R) and red-line all associated data (detects & non-detects); if between 10-74%,qualify all associated data ≥ MDL as "J" and non-detects as "UJ"; if between 126-200%, flag (J) all associated data ≥ MDL as "J" If greater than 200%, reject (R) and red-line all associated data ≥ MDL.
(**NOTE**:Replace "N" with "J" or "R" as appropriate.)

A.1.18 **Lab Duplicates) - Form VI**

A.1.18.1 Was the lab duplicate analysis performed:

For each SDG?	[___]	___	___
On one of the SDG samples?	[___]	___	___
For each matrix type?	[___]	___	___
For each concentration range (low or med.)?	[___]	___	___
For each analytical Method (ICP-AES/ICP-MS,Hg,CN)Used?	[___]	___	___
Was a lab duplicate prepared and analyzed with the SDG samples?	[___]	___	___

		YES	NO	N/A
	ACTION: If no for any of the above, flag (J) as estimated all the SDG sample results (detects & non-detects) for which the lab duplicate analysis was not performed.			
	NOTE: If more than one lab duplicate sample were analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.			
A.1.18.2	Was a Field Blank or PE sample used for the Lab Duplicate analysis?	___	[___]	___
	ACTION: If yes, flag as estimated (J) all SDG sample results (hits & non-detects) for which Field Blank or PE sample was used for duplicate analysis.			
A.1.18.3	Circle on each Form VI all values that are:			
	RPD > 20%, or			
	Absolute Difference > CRQL			
	Are all values within control limits (RPD ≤ 20% or absolute difference ≤ ±CRQL)?	[___]	___	___
	If no, are all results outside the control limits flagged with an "*" (Lab Qualifier) on Form VI and on all Form I's?	[___]	___	___
	ACTION: If no, write in the Contract-Problems/ Non-Compliance Section of the Data Review Narrative.			
	NOTE: The laboratory is not required to report on Form VI the RPD when both values are non-detects.			
A.1.18.4	**Aqueous**			
A.1.18.4.1	When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),			

	YES	NO	N/A
is any RPD > 20% but < 100%?	___	[___]	___
is any RPD ≥ 100%?	___	[___]	___

ACTION:
If the RPD is > 20% but < 100%, flag (J) as estimated the associated sample data ≥ CRQL. If the RPD is ≥ 100%, reject (R) and red-line the associated sample data ≥ CRQL.

(**NOTE:** Replace "*" with "J" or "R" as appropriate.)

A.1.18.4.2 When the sample and/or duplicate value <5xCRQL (substitute MDL for CRQL when MDL >CRQL), is the absolute difference between sample and duplicate values:

> ± CRQL?	___	[___]	___
> ± 2xCRQL?	___	[___]	___

ACTION:
If the absolute difference is > CRQL, flag as estimated all the associated sample results ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the absolute difference is > 2xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but < 5xCRQL.

NOTE:
1. Replace "*" with "J", "UJ" or "R" as appropriate.)
2. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

A.1.18.5 **Soil/Sediment**

A.1.18.5.1 When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 35% but < 120%?	___	[___]	___
is any RPD ≥ 120%?	___	[___]	___

ACTION:
If the RPD is ≥ 35% and < 120%, flag (J) as estimated the associated sample

		YES	NO	N/A
	data ≥ CRQL. If the RPD is ≥ 120%, reject (R)and red-line the associated sample data ≥ CRQL.			
A.1.18.5.2	When the sample and/or duplicate value <5xCRQL(substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and duplicate:			
	> ± 2 x CRQL?	___	[___]	___
	> ± 4 x CRQL	___	[___]	___

ACTION:
If the absolute difference is > 2 x CRQL, flag all the associated sample results ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the absolute difference is > 4xCRQL, reject (R) and red-line all the associated non-detects and detects ≥ MDL but <5xCRQL.

NOTE:
1. Replace "*" with "J", "UJ" or "R" as appropriate.)
2. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this difference to qualify sample results.

A.1.19 Field Duplicates

Aqueous Field Duplicates

A.1.19.1	Was an aqueous Field Duplicate pair collected and analyzed? (Check Sampling Trip Report)	[___]	___	____

ACTION:
If yes, prepare a Form (Appendix A.4) for each aqueous Field Duplicate pair. Report the sample and Field Duplicate results on Appendix A.4 from their respective Form I's. Calculate and report RPD on Appendix A.4 when sample and its Field Duplicate values are both > 5xCRQL. Calculate and report the absolute difference on Appendix A.4 when at least one value (sample or duplicate) is <5xCRQL. Evaluate the aqueous Field Duplicate analysis in accordance with the

	YES	NO	N/A
QC criteria stated in Sections A.1.19.2 and A.1.19.3.			

NOTE:
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and use this the criteria to qualify the results.

A.1.19.2 Circle all values on the Form (Appendix A.4) for Field Duplicates that have:

RPD ≥ 20% or

Difference > ± CRQL

When sample and duplicate values are both ≥5xCRQL (substitute MDL for CRQL when MDL > CRQL),

is any RPD ≥ 20%?	___	[___]	___
is any RPD ≥ 100%?	___	[___]	___

ACTION:
If the RPD is >20% but < 100%, flag (J) only the associated sample and its Field Duplicate results ≥ CRQL. If the RPD is ≥ 100%, reject(R) and red-line only the associated sample and its Field Duplicate result ≥ CRQL.

A.1.19.3 When the sample and/or duplicate value(s) <5xCRQL (substitute MDL for CRQL when MDL >CRQL), is the absolute difference between sample and duplicate:

> ± CRQL?	___	[___]	___
> ± 2 x CRQL?	___	[___]	___

ACTION:
If the absolute difference is > CRQL, flag detects ≥ MDL but < 5xCRQL as "J" and non-detects as "UJ". If the difference is > 2xCRQL,reject (R) and red-line non-detects

		YES	NO	N/A
	and results ≥ MDL but <5xCRQL of the sample and its Field Duplicate.			

Soil/Sediment Field Duplicates

A.1.19.4	Was a soil field duplicate pair collected and analyzed? (Check Sampling Trip Report)	[___]	___	___

ACTION:
If yes, for each soil Field Duplicate pair proceed as follows:

Prepare Appendix A.4 for each Field Duplicate pair. Report on Appendix A.4 all sample and its Field Duplicate results in MG/KG from their respective Form I's. Calculate and report RPD when sample and its duplicate values are both greater than 5xCRQL. Calculate and report the absolute difference when at least one value (sample or duplicate)is < 5xCRQL. Evaluate the Field Duplicate analysis in accordance with the QC Criteria stated in Sections A.1.19.5 and A.1.19.6.

NOTE:
1. Do not transfer "*" from Form I's to Appendix A.4.
2. Do not calculate RPD when both values are non-detects.
3. Substitute MDL for CRQL when MDL > CRQL.
4. If one value is >CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL, and apply the criteria to qualify the results.

A.1.19.5	Circle on each Appendix A.4 all values that have: RPD ≥ 35%, or Difference > ± 2xCRQL When sample and duplicate values are both ≥ 5xCRQL (substitute MDL for CRQL when MDL > CRQL),			
	is any RPD ≥ 35% but < 120%?	___	[___]	___
	is any RPD ≥ 120%?	___	[___]	___

ACTION:
If the RPD is ≥ 35% but < 120%,

		YES	NO	N/A
	flag only the associated sample and its Field Duplicate results ≥ CRQL as "J". If the RPD is ≥ 120%, reject (R) and red-line only the sample and its Field Duplicate results ≥ CRQL.			
A.1.19.6	When the sample and/or duplicate value(s) <5xCRQL (substitute MDL for CRQL when MDL > CRQL), is the absolute difference between sample and Field Duplicate:			
	> ± 2 x CRQL?	___	[___]	___
	> ± 4 x CRQL?	___	[___]	___
	ACTION: If the absolute difference is > 2xCRQL, flag Sample and its Field Duplicate resuts ≥ MDL but <5xCRQL as "J" and non-detects as "UJ". If the difference is >4xCRQL, reject(R) and red-line non-detects and detects ≥ MDL but <5xCRQL of the sample and its Field Duplicate.			
A.1.20	**Laboratory Control Sample (LCS)- Form VII**			
A.1.20.1	Was one LCS prepared and analyzed for:			
	Each SDG?	[___]	___	___
	Each matrix type?	[___]	___	___
	Each batch samples digested/distilled?	[___]	___	___
	For each Method(ICP-AES,ICP-MS,Hg,CN) used?	[___]	___	___
	Was an LCS prepared and analyzed with the samples?	[___]	___	___
	ACTION: If no for any of the above, prepare Telephone Record Log and contact CLP PO or TOPO for submittal of the LCS results. Flag (J) as estimated all the data for which an LCS was not analyzed.			
	NOTE: If only one LCS was analyzed for			

	YES	NO	N/A
more than 20 samples, then the first 20 samples analyzed are not flagged(J), but all additional samples must be qualified (J).			

A.1.20.2 **Aqueous LCS**

Circle on each Form VII the LCS percent recoveries outside control limits 80-120%.

NOTE: 1.Use digested ICV as LCS for aqueous mercury
2.Use distilled ICV as LCS for aqueous cyanide

Is any LCS recovery:

Less than 50%?	___	[___]	___
Between 50% and 79%?	___	[___]	___
Between 121% and 150%?	___	[___]	___
Greater than 150%?	___	[___]	___

ACTION:
If the LCS recovery is less than 50%, reject (R) and red-line all associated sample data (detects & non-detects); for a recovery between 50-79%, flag detects as "J" all non-detects as "UJ". if the LCS recovery is between 121-150%, flag only detects as "J". if the recovery is greater than 150%, reject (R) and red-line all detects.

A.1.20.3 **Solid LCS**

If an analyte's MDL is equal to or greater than the true value of LCS, disregard the "Action" below for that analyte even though the LCS is out of control limits.

Is the LCS "Found" value greater than the Upper Control Limit reported on Form VII?	___	[___]	___

ACTION:

		YES	NO	N/A
	If yes, flag (J) all the associated detects ≥ MDL as estimated (J).			
	Is the LCS "Found" value lower than the Lower Control Limit reported on Form VII?	___	[___]	___
	ACTION: If yes, flag detects as "J" and non-dectes as "UJ".			
A.1.21	**ICP-AES/ICP-MS Serial Dilution - Form VIII**			
	NOTE: Serial dilution analysis is required only when the initial concentration is equal to or greater than 50 x MDL.			
A.1.21.1	Was a Serial Dilution analysis performed:			
	For each SDG?	[___]	___	___
	On one of the SDG samples?	[___]	___	___
	For each matrix type?	[___]	___	___
	For each concentration range (low or med.)?	[___]	___	___
	Was a Serial Dilution sample analyzed with the SDG samples?	[___]	___	___
	ACTION: If no for any of the above, flag as estimated (J) detects ≥ MDL of all the SDG samples for which the ICP Serial Dilution Analysis was not performed.			
A.1.21.2	Was a Field Blank or PE sample used for the Serial Dilution Analysis?	___	[___]	___
	ACTION: If yes, flag as estimated (J) detects ≥ MDL of all the SDG samples			
A.1.21.3	Circle on Form VIII the Percent Differences (%D) between sample results and its dilution results that are outside the control limits ± 10%			

		YES	NO	N/A
	when initial concentrations ≥ 50 x MDLs.			
	Are results outside the control limits flagged with an "E"(Lab Qualifier) on Form VIII and all Form I's?	[___]	___	___
	ACTION: If no, write in the Contract-Problem/ Non-Compliance Section of the Data Review Narrative.			
A.1.21.4	Are any %D values:			
	> 10%?	___	[___]	___
	≥ 100%?	___	[___]	___
	ACTION: If the Percent Difference (%D) is greater than 10%, flag (J) as estimated all associated samples whose **raw data** ≥ MDL; if the %D is ≥ 100%, reject (R) and red-line all associated samples with **raw data** ≥ MDL.			
	(**NOTE**:Replace "E" with "J" or "R" as appropriate.)			
A.1.22	**Total/Dissolved or Inorganic/Total Analytes**			
A.1.22.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s)?	___	[___]	___
	Were any analyses performed for inorganic as well as total analytes on the same sample(s)?	___	[___]	___
	ACTION: If yes, prepare a Form (Appendix A.5) to compare the differences between dissolved (or inorganic)and total analyte concentrations. Compute each difference on Appendix A.5 as a percent of the total analyte only when both of the following conditions are fulfilled:			
	(1) The dissolved(or inorganic)concentration is greater than total concentration, and (2) greater than or equal to 5xMDL.			
A.1.22.2	Is any dissolved (or inorganic) concentration greater than its total concentration by more than 20%?	___	[___]	___

		YES	NO	N/A
A.1.22.3	Is any dissolved(or inorganic) concentration greater than its total concentration by more than 50%?	___	[___]	___

ACTION:
If the percent difference is greater than 20%, flag (J) both dissolved/inorganic and total concentrations as estimated. If the difference is more than 50%, reject (R) and red-line both the values.

A.1.23 **Field Blank - Form I**
NOTE: **Designate "Field Blank" as such on Form I**

		YES	NO	N/A
A.1.23.1	Was a Field/Rinsate Bank collected and analyzed with the SDG samples?	[___]	___	___
	If yes, is any Field/Rinsate Blank absolute value of an analyte on Form I greater than its CRQL(or 2xMDL when MDL>CRQL)?	___	[___]	___
	If yes, circle the Field Blank value on Form I that is greater than the CRQL,(or 2 x MDL when MDL > CRQL).			
	Is any Field Blank value greater than CRQL also greater than the Preparation Blank value?	___	[___]	___
	If yes, is the Field Blank value (> CRQL and > the prep. blank value) already rejected due to other QC criteria?	[___]	___	___

ACTION:
If the Field Blank value was not rejected, reject all associated sample data (except the Field Blank results)greater than the CRQL but less than the Field Blank value. Reject on Form I's the soil sample results whose raw values in ug/L in the instrument printout are greater than the CRQL but less than the Field Blank value in ug/L. Flag as "J" detects between the Field Blank value and 10xField Blank value. If the sample result ≥ MDL but ≤ CRQL, replace it with CRQL-U.

If the Field Blank value is less than the

	YES	NO	N/A
Prep.Blank value, do not qualify the sample results due to the Field Blank criteria.			

NOTE:
1. Field Blank result previously rejected due to other criteria cannot be used to qualify field samples.
2. Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.

A.1.24 **Verification of Instrumental Parameters - Form IX, XA, XB, XI**

A.1.24.1 Is verification report present for:

Method Detection Limits (Form IX-Annually)?	[___]	___	___
ICP-AES Interelement Correction Factors (Form XA & XB -Quarterly)?	[___]	___	___
ICP-AES & ICP-MS Linear Ranges (Form XI-Quarterly)?	[___]	___	___

ACTION:
If no, contact CLP PO/TOPO for submittal from the laboratory.

A.1.24.2 **Method Detection Limits - Form IX**

A.1.24.2.1 Are MDLs present on Form IX for:

All the analytes?	[___]	___	___
All the instruments used?	[___]	___	___
Digested and undigested samples and Calib.Blanks?	[___]	___	___
ICP-AES and ICP-MS when both instruments are used for the same analyte?	[___]	___	___

ACTION:
If no for any of the above, prepare Telephone Record Log and contact CLP PO/TOPO for submittal of the MDLs from the laboratory. Report to CLP PO and write in the Contract Problems/ Non-Compliance Section of the Data Review Narrative if the MDL concentration is not less than ½ CRQL.

		YES	NO	N/A
A.1.24.2.2	Is MDL greater than the CRQL for any analyte?	___	[___]	___
	If yes,is the analyte concentration on Form I greater than 5 x MDL for the sample analyzed on the instrument whose MDL exceeds CRQL?	[___]	___	___
	ACTION: If no, flag as estimated (J) all values less than five times MDL for the analyte whose MDL exceeds the CRQL.			
A.1.24.3	**Linear Ranges - Form XI**			
A.1.24.3.1	Was any sample result higher than the high linear range for ICP-AES or ICP-MS?	___	[___]	___
	Was any sample result higher than the highest calibration standard for mercury or cyanide?	___	[___]	___
	If yes for any of the above, was the sample diluted to obtain the result reported on Form I?	[___]	___	___
	ACTION: If no, flag (J) as estimated the affected detects ($\geq$ MDL) reported on Form I.			
A.1.25	**ICP-MS Tune Analysis - Form XIV**			
A.1.25.1	Was the ICP-MS instrument tuned prior to calibration?	[___]	___	___
	ACTION: If no, reject (R) and red-line all sample data for which tuning was not performed.			
A.1.25.2	Was the tuning solution analyzed or scanned at least five times consecutively?	[___]	___	___
	Were all the required isotopes spanning the analytical range present in the tuning solution?	[___]	___	___
	Was the mass resolution within			

		YES	NO	N/A
	0.1 amu for each isotope in the tuning solution?	[___]	___	___
	Was %RSD less than 5% for each isotope of each analyte in the tuning solution?	[___]	___	___
	ACTION: If no for any of the above, qualify all results ≥ MDL associated with that Tune as estimated "J", and all non-detects associated with that Tune as "UJ".			
A.1.26	**ICP-MS Internal Standards - Form XV**			
A.1.26.1	Were the Internal Standards added to all the samples and all QC samples and calibration standards (except the Tuning Solution)?	[___]	___	___
	Were all the target analyte masses bracketed by the masses of the five internal standards?	[___]	___	___
	ACTION: If none of the Internal Standards was added to the samples, reject (R) and red-line all the associated sample data (detects & non-detects). If internal standards were used but did not cover all the analyte masses, reject (R) and red-line only the analyte results not bracketed by the internal standard masses.			
A.1.26.2	Was the intensity of an Internal Standard in each sample within 60-125% of the intensity of the same Internal Standard in the calibration blank?	[___]	___	___
	If no, was the original sample diluted two fold, Internal Standard added and the sample re-analyzed?	[___]	___	___
	Was the %RI for the two fold diluted sample within the acceptance limits (60-125%)?	[___]	___	___
	ACTION: If no for any of the above, flag detects as "J" and non-detects "UJ" of all the analytes with atomic masses between the atomic mass of the internal standard lighter			

than the affected internal standard, and the atomic mass of the internal standard heavier than the affected internal standard.

A.1.27 **Percent Solids of Sediments**

A.1.27.1 Are percent solids in sediment(s):

< 50%? ___ [___] ___

ACTION:
If yes, qualify as estimated (J) all detects and non-detects of a sample that has percent solids less than 50%(i.e.,moisture content greater than 50%).

NOTE:
Flag(J) only the sample results that were not previously flagged due to other QC criteria.

Inorganic Data Review Narrative

Case# _____ **Site:** _____ **Matrix: Soil** ____

SDG# _____ **Lab:** _____ **Water** ____

Sampling Team: _____ **Reviewer:** _____ **Other** _____

A.2.1 **Data Validation Flags**:
The following flags may have been applied in red by the data validator and must be considered by the data user.

J - This flag indicates the result qualified as **estimated**

R and Red-Line - A red-line drawn through a sample result indicates **unusable** value. The red-lined data are known to contain significant errors based on documented information and must not be used by the data user.

U - This data validation qualifier is applied to sample results ≥ MDL when associated blank is contaminated

Fully Usable Data - The results that do not carry "J" or "red-line" are fully **usable**.

A.2.2 **Laboratory Qualifiers**:
The CLP laboratory applies a contractual qualifier on all

Form I'S and the QC Form when a QC analysis is outside the control limits. These qualifiers are not applied on the Lotus or XLS spreadsheets. These qualifiers and their meanings are as follows:

N: This qualifier indicates the lack of accuracy in the reported result, and is applied when matrix spiked sample recovery is outside the control limits.

E: This qualifier indicates the the presence of interference, and is applied when the ICP serial dilution is outside the control limits.

*****: This qualifier indicate the lack of precision , and is pplied on Fom I'S and Form VI when the Lab Duplicate analysis is outside the control limits.

U: This is a concentration qualifier that laboratory applies to a non-detected result which is essentially less than the Method Detection Limit(MDL). A non-detected result of an analyte is indicated by the Contract Required Quantitation Limit (CRQL) of that analyte suffixed with "U".

J: This is also a concentration qualifier that laboratory applies to a positive result below the CRQL.

NOTE: The laboratory qualifiers are crossed out and replaced with the appropriate data validation qualifiers (J, R or U) by the data validator.

A.2.3.1 **Data Case Description**:

A.2.3.2 **CSF Audit**:

A.2.3.3 **Technical Review**:

A.2.3.4 **Contract-Problem/Non-Compliance:**

HWSS Reviewer: _____ Date:_____
Signature

Contractor Reviewer: _____ Date:_____
Signature

Verified by: _____ Date:_____
Signature

CASE #
SDG #

Contract Laboratory Program
REGION II/LABORATORY COMMUNICATION SYSTEM

Telephone Record Log

Date of Call: _____

ESAT Reviewer/Date: _____

Type of Analysis: ___Inorganic_____

Laboratory Name: _____

Lab Contact: _____

Call Initiated By: ___Laboratory _X_Region II

Inquiry made in reference to data for the following sample number(s):

Summary of Questions/Issues Discussed:

Summary of Resolution:

_____
Signature **Date:** _____

FIELD DUPLICATES

Sample No. **Field Duplicate No.** **Sample Matrix:**

Lab Code: **Case No. :** **SDG No.:**

% Solids Sample: **% Solids Duplicate:**

Concentration Units (ug/l or mg/kg dry weight):

	Action Limit	Sample Concentration	C	Duplicate Concentration	C	RPD	Difference	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									

					YES	NO	N/A		
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Total/Dissolved Concentrations

Lab Code **Case No.** **SDG No.** **Sample Matrix: Water**

Concentration: ug/L

ANALYTE	TOTAL	C	DISSOLVED	C	DIFFERENCE	Q	M
ALUMINUM							
ARSENIC							
BARIUM							
BERYLLIUM							
CADMIUM							
CALCIUM							
CHROMIUM							
COBALT							
COPPER							
IRON							
LEAD							
MAGNESIUM							
MAGNESE							
MERCURY							
NICKEL							

POTASSIUM							
SELENIUM							
SILVER							
SODIUM							
THALLIUM							
VANADIUM							
ZINC							
CYANIDE							

CONTRACT LABORATORY PROGRAM
CLP RAS RE-ANALYSIS REQUEST/APPROVAL RECORD

SECTION A (TO BE COMPLETED BY REGIONAL SENDING OFFICIAL)

Initiated By:

Name, Affiliation, Phone Number

_____

Case Number:_____
! OLM
! OLC
! ILM

Details of Re-Analysis Request:

! Laboratory Name /Contract Number: _____

! Affected Sample Number(s) and Fraction(s): _____

! Reason for Re-Analysis: _____

! Contract Statement of Work Citation*: _____

_____

! Comments:_____

! * PROVIDE SOW CITATION THAT SUPPORTS THIS REQUEST

RE-ANALYSIS Billable ( ) Not Billable ( )

! Approved By: _____ Date: _____

Authorized Regional Sending CLP PO Signature

SECTION B (TO BE COMPLETED BY SMO)

Name of SMO Contact _____ Date:_____

FOIA_07123_0003552_0844

Date of Laboratory Notification (Verbal): _____

Re-analysis Start Date: _____ Data Due Date: _____

_____

Return completed form to:
Sample Management Office (SMO)

Distribtion: (1) CLP PO Copy (2) Regional Sending Official Copy (3) SMO File Copy (4) Laboratory Copy
Final 9/3/99

CLP DATA ASSESSMENT SUMMARY FORM (INORGANICS)

Type of Review: _____ Date:_____ Case# _____SDG#___

Site: _____ Lab Name: _____

Reviewer's Initials:_____ Number of Samples:_____

Analytes Rejected (R) Due to Exceeding Review Criteria

	Holding Time	CRQL Std	Blanks	ICS	Spike Recovery	Dup. Lab.	Dup. Field	LCS	ICP Serial Dilution	% Solids	Internal Std. ICP-MS	Tuning ICP-MS	Total Analytes	Rejection %
ICP-AES														
ICP-MS														
Mercury														
Cyanide														
Total														

Analytes Flagged (J) as Estimated Due to Exceeding Review Criteria

FOIA_07123_0003552_0845

	Holding Time	CRQL Std	Blanks	ICS	Spike Recovery	Dup. Lab.	Dup. Field	LCS	ICP Serial Dilution	% Solids	Internal Std. ICP-MS	Tuning ICP-MS	Total Analytes	Rejection %
ICP-AES														
ICP-MS														
Mercury														
Cyanide														
Total														

VALIDATION SOP

TOC (Solid/Liquid)

EDS SOP: TOC-01
Rev. 2 07/10

TOTAL ORGANIC CARBON (TOC) METHOD VALIDATION CHECKLIST
Environmental Data Services
Lloyd Kahn (soil) & USEPA 9060 (liquid)

SITE:

DATE:

SDG:

Note: This procedure is appropriate for validation of the following analyses: Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC) and Particulate Organic Carbon (POC)

	Y	N	N/A
Data Completeness and Deliverables			
Have any missing deliverables been received and added to the data package?	_____	_____	_____
ACTION: Call lab for explanation/resubmittal of any missing deliverables. If the lab cannot provide them, note the effect on review of the data in the non-compliance section of the data assessment narrative.			
Validation TOC Data			
The data validation criteria outlined below applies to Total Organic Carbon data obtained via method Lloyd Kahn for soil samples and/or USEPA 9060 for liquid samples.			
Custody Documents and Narratives			
Are chains of custody present and complete for all samples?	_____	_____	_____
ACTION: Contact lab for replacement of missing documents.			
Do chains of custody or lab narratives indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?	_____	_____	_____

FOIA_07123_0003552_0848

	Y	N	N/A
Preservation Requirements			
Is the cooler temperature for soil and liquid samples received at the laboratory below $4^{o}C \pm 2$?	_____	_____	_____
Is the liquid sample acidified with H_2SO_4 to a pH of ≤ 2?	_____	_____	_____

ACTION: If samples were not iced or if the ice was melted upon arrival at the laboratory and the temperature of the cooler was elevated ($>10^{o}C$), or liquid samples not acidified, flag all positive results as estimated "J" and all non-detects "UJ".

Holding Times			
Have any TOC technical holding times, determined from date of collection to date of analyses been exceeded?	_____	_____	_____

NOTE: Liquid and soil samples must be analyzed within 14 days of sample collection.

Table of Holding Time Violations

Sample	Sample Matrix	Date Sampled	Date Lab Received	Date Analyzed
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

ACTION: If holding times are exceeded, flag all data as estimated ("J" for detects and "UJ" for non-detects). If holding times were grossly exceeded (i.e., more than 2x the holding time), flag all positive data as estimated and reject all non-detects as unusable ("R").

FOIA_07123_0003552_0849

	Y	N	N/A
Matrix Spike			
The following sample was spiked _____			
Is a matrix spike/matrix spike duplicate summary present?	_____	_____	_____
Were matrix spikes analyzed at the required frequency for each of the following matrixes:			
a. low water?	_____	_____	_____
b. low soil?	_____	_____	_____
c. medium soil?	_____	_____	_____

ACTION: If any matrix spike data are missing, call the lab for explanation/re-submittal. If information is not available, document the effect in narrative notes.

Does the matrix spike/matrix spike duplicate for the liquid and soil samples fall within:

% Recovery = 50 – 150%
RPD = $\leq$ 35%

How many matrix spike recoveries are outside QC limits?	_____	_____	_____

water _____ out of _____

soil _____ out of _____

ACTION: Do not qualify associated sample results on the basis of the MS/MSD data alone. Use the MS/MSD results in conjunction with other QC criteria to determine the need for qualification of associated data. If the MS and MSD both have less than 10 percent recovery for an analyte, reject non-detect results for that analyte and qualify positive results for that analyte as estimated for the sample used for the MS/MSD analysis. If the MS and MSD both have greater than 200 percent recovery for an analyte, reject detected results for that analyte and qualify non-detect results for that analyte as estimated for the sample used for the MS/MSD analysis. Use professional judgment in applying this criterion to other samples.

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	Y	N	N/A
Laboratory Control Sample			
Were LCS samples evaluated with each batch of 20 samples or less and were observed percent recoveries within the acceptance limits of (50-150%)?	_____	_____	_____

ACTION:

- Contact the lab for an explanation/re-submittal.
- If the % Recovery for the LCS is < 10%, flag all associated sample data "R", for non-detects and "J" for detects.
- If the LCS % Recovery is between 11% - 49%, flag all associated sample data as "J" for both non-detects and detects.
- If the LCS % Recovery is > 150%, flag all associated positive sample data as "J".
- Document in Data Assessment Narrative.

Blanks			
Has a method blank analysis been reported per twenty samples of a similar matrix or concentration level, and for each extraction batch?	_____	_____	_____
Are there field/rinse/equipment blanks associated with every sample?	_____	_____	_____
Is the method blank summary present?	_____	_____	_____
Upon examination of laboratory and field blank data, do any blanks contain positive results ≥ project quantitation limit (PQL)?	_____	_____	_____

ACTION: If yes, qualify associated results as follows:

If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag sample result as non-detect ("U"). If the sample result is reported as detected at a concentration less than the reporting limit and less than 5 times the blank concentration, qualify the sample result as non-detectable at the PQL. For liquid blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank. The equivalent concentration is determined by assuming that all of the analyte present in the blank aliquot analyzed is present in the sample aliquot analyzed.

The reviewer should note that the blank analyses may not involve the same weights, volumes or dilution factors as the associated samples. These factors must be taken into consideration when applying the 5X criteria such that a comparison of the total contaminants is actually made.

Prepare a list of samples affected.

	Y	N	N/A
Calibration			
Are raw data and summary sheets present for both initial and continuing calibrations?	_____	_____	_____
Check calibration factors and % RSD values back to raw data for 10% of data received. Are miscalculations or transcription errors found?	_____	_____	_____
NOTE: If yes, contact the laboratory.			
Are the % RSD values for the 5 level initial calibration less than or equal to 20% or correlation coefficient greater than 0.995?	_____	_____	_____
ACTION: Associated sample data for those analytes with % RSD > 20 will be qualified as estimated.			
Were initial and continuing calibration verifications evaluated (one for every ten samples)?	_____	_____	_____
Are the % D values between the true and measured concentration values for the continuing calibrations ± 20?	_____	_____	_____
ACTION: If no, data following the last in-control standard to the next-in-control standard are potentially affected. Associated detected sample data will be qualified as estimated and associated non-detected sample data will be qualified as estimated if low bias is determined to be present.			
Compound Quantitation and Reported Detection Limits			
Check data for one or more detected target analytes per sample delivery group. Recalculate from the raw data to check for calculation and transcription errors.			
Were miscalculation/transcription errors found?	_____	_____	_____
Are the PQL's adjusted to reflect sample dilutions and percent moisture for solid samples?	_____	_____	_____
ACTION: If errors are found 100% of the data will be evaluated. Contact the lab for the explanation/resubmittals.			
Field Duplicates			
Were field duplicates submitted for TOC analysis?	_____	_____	_____
ACTION: Where both the sample duplicate values are greater than 5 times the PQL, acceptable sampling and analytical precision is indicated by an RPD for the two field duplicate results of less than or equal to 20 percent for liquid samples and 50 percent for soil samples. Where one or both analytes of the field duplicate pair are less than 5 times the PQL, satisfactory precision is indicated if the field duplicate results agree within 2 times the PQL. If the above criteria are not met for an analyte, qualify all associated sample data for that analyte as estimated ("J").			

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Data Validation Qualifiers

Qualifier	Description
J	Estimated value (bias undetermined) – The analyte was positively identified; but the associated numerical value is the approximate concentration of the analyte in the sample.
JH	Estimated value (potential high bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential high bias, of the analyte in the sample.
JL	Estimated value (potential low bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential low bias, of the analyte in the sample.
UJ	Estimated non-detect - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
UJL	Estimated non-detect (potential low bias) – The analyte was not detected and the report sample quantitation limit is biased low.
UJH	Estimated non-detect (potential high bias) – The analyte was not detected and the reported sample quantitation limit is biased high.
M	The analytical result reported was obtained from a sediment sample found to contain between 50 and 90 percent moisture and had no other data qualifiers added during the data validating process.
NJ	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
NJH	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration with a potential high bias, of the analyte concentration.
EMPC	Estimated Maximum Possible Concentration (EMPC).
R	The sample results are rejected. Due to a significant QA/QC problem, the analysis is invalid and provides no information as to whether the analyte is present or not.

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VALIDATION SOP

TEPH

EDS SOP: TEPH-01, Rev.3, 07/07

TEPH METHOD VALIDATION CHECKLIST

SITE:

DATE:

SDG:

	Y	N	N/A
Data Completeness and Deliverables			
Have any missing deliverables been received and added to the data package?	_____	_____	_____
ACTION: Call lab for explanation/resubmittal of any missing deliverables. If the lab cannot provide them, note the effect on review of the data in the non-compliance section of the data assessment narrative.			
Custody Documents and Narratives			
Are chains of custody present and complete for all samples?	_____	_____	_____
ACTION: Contact lab for replacement of missing documents.			
Do chains of custody or lab narratives indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?	_____	_____	_____
ACTION: If any sample analyzed as a soil other than a TCLP, contains 50-90% water, flag all data as estimated. If the soil sample, other than TCLP, contains more than 90% water, all data would be flagged as unusable.			
ACTION: If samples were not iced upon receipt, flag all positive results as estimated, an all non-detects "UJ".			
Holding Times			
Have any TEPH technical holding times, determined from date of collection to date of extraction been exceeded?	_____	_____	_____

NOTE: Water and Soil sample must be extracted within 7 days of sample collection. Extracts must be analyzed within forty days of extraction.

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Table of Holding Time Violations

Sample	Sample Matrix	Date Sampled	Date Lab Received	Date Analyzed	Date Extracted
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

ACTION: If holding times are exceeded, flag all data as estimated ("J" for detects and "UJ" for non-detects). If holding times were grossly exceeded (i.e., more than 2x the holding time), flag all positive data as estimated and reject all non-detects as unusable ("R").

Surrogate Recovery

	Y	N	N/A
Are surrogate recoveries summarized within the report?	_____	_____	_____

ACTION: If no, contact the lab for explanation/re-submittals. If re-deliverables are not available, document in the narrative notes. (Surrogate % R limits 60-120%).

Were outliers marked as such?	_____	_____	_____

ACTION: Circle outliers in red.

Were surrogate recoveries outside of specifications for any sample or method blank?	_____	_____	_____

ACTION: If any surrogate recoveries are >10% but do not meet project requirements.

1. Flag all positive results as estimated.
2. Flag all non-detects as estimated detection limits when recoveries are less than the lower limit.
3. If recoveries are above the upper limit, do not flag non-detects.
4. If any surrogate recovery is less than 10%:
   a. qualify positive results as estimated.
   b. non-detects for that sample should be qualified as unusable.

   Professional judgment is used to qualify data that have method blank surrogate recoveries outside acceptance limits in both the original and re-analysis.

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	Y	N	N/A
Are surrogate standard retention times within the retention time windows established during the initial calibration?	_____	_____	_____

ACTION: If the retention time limits are not met, the analysis may be qualified unusable (R) for the affected sample on the basis of professional judgement.

Are there any transcription/calculation errors between the surrogate recoveries listed and raw data?	_____	_____	_____

Matrix Spike

Is a matrix spike/matrix spike duplicate summary present?	_____	_____	_____
Were matrix spikes analyzed at the required frequency for each of the following matrixes:			
a. low water?	_____	_____	_____
b. low soil?	_____	_____	_____
c. medium soil?	_____	_____	_____

ACTION: If ay matrix spike data are missing, call the lab for explanation/re-submittal. If information is not available, document the effect in narrative notes.

% Recovery = 70 – 130
RPD = 50%

How many matrix spike recoveries are outside QC limits?	_____	_____	_____

water _____ out of _____

soil _____ out of _____

ACTION: Do not qualify associated sample results on the basis of the MS/MSD data alone. Use the MS/MSD results in conjunction with other QC criteria to determine the need for qualification of associated data. If the MS and MSD both have less than 10 percent recovery for an analyte, reject non-detect results for that analyte and qualify positive results for that analyte as estimated for the sample used for the MS/MSD analysis. If the MS and MSD both have greater than 200 percent recovery for an analyte, reject detected results for that analyte and qualify non-detect results for that analyte as estimated for the sample used for the MS/MSD analysis. Use professional judgment in applying this criterion to other samples.

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	Y	N	N/A
Laboratory Control Sample			
Were LCS samples evaluated with each batch of 20 samples or less and were observed percent recoveries within the laboratory defined limits of (70-120%)?	_____	_____	_____
ACTION: Document in Data Assessment Narrative.			
Blanks			
Has a method blank analysis been reported per twenty samples of a similar matrix or concentration level, and for each extraction batch?	_____	_____	_____
Upon examination of laboratory and field blank data, do any blanks contain positive results?	_____	_____	_____
ACTION: If yes, qualify associated results as follows:			
If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag sample result as non-detect ("U"). If the sample result is reported as detected at a concentration less than the reporting limit and less than 5 times the blank concentration, qualify the sample result as non-detectable at the laboratory reporting limit. For aqueous blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank. The equivalent concentration is determined by assuming that all of the analyte present in the blank aliquot analyzed is present in the sample aliquot analyzed.			
Prepare a list of sample effected.			
Are there field/rinse/equipment blanks associated with every sample?	_____	_____	_____
Calibration			
Are raw data and summary sheets present for both initial and continuing calibrations?	_____	_____	_____
Are the % RSD values for the initial calibration less than or equal to 20% or correlation coefficient greater than 0.995?	_____	_____	_____
ACTION: Associated sample data for those analytes with % RSD > 20 will be qualified as estimated.			

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	Y	N	N/A
Are the % D values between the true and measured concentration values for the continuing calibrations ± 20?	_____	_____	_____

ACTION: If no, data following the last in-control standard to the next-in-control standard are potentially affected. Associated detected sample data will be qualified as estimated and associated non-detected sample data will be qualified as estimated if low bias is determined to be present.

Check calibration factors and % RSD values back to raw data for 10% of data received.

Are miss-calculations or transcription errors found?	_____	_____	_____

NOTE: If yes, contact the laboratory.

Compound Quantitation and Reported Detection Limits

Check data for one or more detected target analytes per sample for ten percent of the data packages. Recalculate from the raw data to check for calculation and transcription errors.

Were miscalculation/transcription errors found?	_____	_____	_____

ACTION: If errors are found 100% of the data will be evaluated. Contact the lab for the explanation/resubmittals.

Field Duplicates

Were field duplicates submitted for TEPH analysis?	_____	_____	_____

ACTION: Where both the sample duplicate values are greater than 5 times the SQL, acceptable sampling and analytical precision is indicated by an RPD for the two field duplicate results of less than or equal to 100 percent. Where one or both analytes of the field duplicate pair are less than 5 times the SQL, satisfactory precision is indicated if the field duplicate results agree within 2 times the SQL. If the above criteria are not met for an analyte, qualify all associated sample data for that analyte as estimated ("J").

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Data Validation Qualifiers

Qualifier	Description
J	Estimated value (bias undetermined) – The analyte was positively identified; but the associated numerical value is the approximate concentration of the analyte in the sample.
JH	Estimated value (potential high bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential high bias, of the analyte in the sample.
JL	Estimated value (potential low bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential low bias, of the analyte in the sample.
UJ	Estimated non-detect - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
UJL	Estimated non-detect (potential low bias) – The analyte was not detected and the report sample quantitation limit is biased low.
UJH	Estimated non-detect (potential high bias) – The analyte was not detected and the reported sample quantitation limit is biased high.
M	The analytical result reported was obtained from a sediment sample found to contain between 50 and 90 percent moisture and had no other data qualifiers added during the data validating process.
NJ	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
NJH	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration with a potential high bias, of the analyte concentration.
EMPC	Estimated Maximum Possible Concentration (EMPC).
R	The sample results are rejected. Due to a significant QA/QC problem, the analysis is invalid and provides no information as to whether the analyte is present or not.

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VALIDATION SOP

Hexavalent Chromium

NJDEP SOP for Analytical Data Validation of Hexavalent Chromium, 5.A.10, Rev.2, 8/05

State of New Jersey

Richard J. Codey
Acting Governor

Department of Environmental Protection

Bradley M. Campbell
Commissioner

DIVISION OF REMEDIATION MANAGEMENT & RESPONSE

STANDARD OPERATING PROCEDURE

TITLE: Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium

REVISION No.: 2 **ISSUE DATE:** **SOP NO.: 5.A.10**

APPROVED: Barry Frasco
Dr. Barry R. Frasco, Assistant Director
Hazardous Site Science Element

August 15, 2005
(Date)

Greg Toffoli
Greg Toffoli, Section Chief
Office of Data Quality

August 15, 2005
(Date)

PURPOSE/SCOPE:

This document is the Standard Operating Procedure (SOP) for laboratory data evaluation and validation of Hexavalent Chromium analyzed in accordance with Update III (July 1998) USEPA SW-846 Method 3060A, USEPA SW-846 Method 7196A and USEPA SW-846 Method 7199.

ORIGINATING ORGANIZATIONAL UNIT (S): **ODQ**

OTHER ORGANIZATIONAL UNIT (S) AFFECTED: **ALL DRMR, DRS & DSW&HW**

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DIVISION OF REMEDIATION MANAGEMENT & RESPONSE

STANDARD OPERATING PROCEDURE

TITLE: Standard Operating Procedure (SOP) for Analytical Data Validation of Hexavalent Chromium

REVISION No.: 2 **ISSUE DATE:** **SOP NO.: 5.A.10**

APPROVED: _____ _____

Dr. Barry R. Frasco, Assistant Director (Date)
Hazardous Site Science Element

_____ _____

Greg Toffoli, Section Chief (Date)
Office of Data Quality

PURPOSE/SCOPE:

This document is the Standard Operating Procedure (SOP) for laboratory data evaluation and validation of Hexavalent Chromium analyzed in accordance with Update III (July 1998) USEPA SW-846 Method 3060A, USEPA SW-846 Method 7196A and USEPA SW-846 Method 7199.

ORIGINATING ORGANIZATIONAL UNIT (S): **ODQ**

OTHER ORGANIZATIONAL UNIT (S) AFFECTED: **ALL DRMR, DRS & DSW&HW**

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I. PURPOSE AND SCOPE:

This document is the Standard Operating Procedure (SOP) for laboratory data evaluation and validation of Hexavalent Chromium analyzed in accordance with USEPA SW-846 Methods 3060A, 7196A and 7199.

II. AUTHORITY:

This document was prepared and revised under the authority of the Assistant Director of the DRMR-HSSE and the Section Chief, ODQ. This revision, maintenance and use of this document is a work output under the NJDEP Quality Assurance Program Plan. The QA Program Plan was prepared by the NJDEP Office of Quality Assurance and in part by DRMR-HSSE-ODQ, and was approved by the USEPA Region II.

III. REFERENCE:

This document was prepared based on materials contained in the document entitled: USEPA Office of Solid Waste and Emergency Response Update III (July 1998) - "Test Methods for Evaluating Solid Waste", USEPA SW-846.

IV. RESPONSIBILITY:

The Assistant Director of DRMR HSSE is responsible for the final review and approval of this document. The Section Chief of ODQ is responsible for the annual review of this document. The Supervisor of ODQ is responsible for the preparation of any revision to this document as well as maintaining ODQ staff compliance with this document.

V. POLICY:

The actions contained in this document are the policy of DRMR-HSSE-ODQ and are derived based on requirements contained in the referenced USEPA SW-846 Methods 3060A, 7196A and 7199.

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VI. PROCEDURE:

(A) Introduction

This document is designed to offer guidance in laboratory data evaluation and validation. In some aspects, it is equivalent to a Standard Operating Procedure (SOP); in other more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples.

Those areas where specific SOPs are possible are primarily areas in which definitive performance requirements are established. These requirements are concerned with specifications that are not sample dependent; they specify performance requirements on matters that should be fully under a laboratory's control. These specific areas include laboratory preparation blanks, calibration standards, calibration check standards, and laboratory control standards. Failure to meet these performance requirements warrants that corrective action is taken by the laboratory.

At times, there may be an urgent need to use data that do not meet all QA/QC requirements. Any decision to utilize data for which non-sample specific criteria have not been met is strictly to facilitate the progress of projects requiring the availability of the data and such decisions should be clearly noted in the data validation report. Use of this data does not constitute acceptance of the data in terms of method compliance nor does it release the laboratory from the obligation to perform as per the analytical method. A laboratory submitting data, which are out of specification, may be required to re-run or resubmit data. The only exception to this is in the area of requirements for individual sample analysis; if the nature of the sample itself limits the attainment of specifications, appropriate allowances must be made. An overriding concern of the DRMR-ODQ is to prevent non-sample specific data validation requirements from adversely affecting overall data validation activities. There is ultimately no justification for noncompliance on requirements for performance relative to such areas as blanks, calibration and performance verification standards. Ideally, data validation activities should only be concerned with subjects requiring professional judgment on individual sample results.

With these concepts in mind, this document is designed to permit structured data review. To this end, the document is arranged in order, with the most objective, straightforward validation elements given first.

It will be the data reviewer's responsibility to notify the assigned NJDEP Technical Coordinator and Case Manager concerning problems and shortcomings in regards to laboratory data via a Data Validation Report and a Target Analyte Summary List. If mandatory actions are required, they should be specifically noted in a Data Validation Report. This report should also be used to note overall deficiencies requiring attention as well as comments on general laboratory performance and any discernible trends in the quality of data.

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(B) Data Package Deliverables

Data generated using USEPA SW-846, Update III Hexavalent Chromium methods must be delivered to NJDEP in the regulatory format as defined in the currently active Professional Laboratory Services Contract and the Technical Requirements for Site Remediation N.J.A.C. 7:26E. Data delivered in the "Reduced Regulatory Format" can only be reviewed against the requirements of this SOP and cannot be validated.

(C) Preliminary Review

In order to use this document effectively, the reviewer should have a general overview of the data deliverable package at hand. The exact number of samples, their assigned laboratory and field identification numbers, their matrix, and concentration level, the identity of any field QC samples (blanks, duplicates, spikes, splits), sampling dates and the name of laboratory performing sample analyses are essential information. Background information on the site is helpful but at times, it is very difficult to locate. The NJDEP Technical Coordinator or the Case Manager is the best sources for answers or further direction.

The chain-of-custody record provides sample descriptions and the date and time of sampling. Sampling procedures are addressed by NJDEP "Field Sampling Procedures Manual" requirements. Any discrepancies found by the reviewer must be noted on the data validation report. The non-conformance summary that is submitted by the laboratory is another source of general information. Notable problems with matrices, insufficient sample size for analysis or reanalysis, sample temperature and preservation and unusual events should be found here.

(D) Data Validation

1. Sample Holding Times

A. Objective

The objective is to ascertain the validity of results, based on the holding time of the sample from time of collection to time of analysis. From the standpoint of laboratory performance, the time of analysis is needed to determine compliance with the USEPA SW-846, Hexavalent Chromium methods.

B. Requirements

The following holding time requirements were established by the USEPA SW-846, Hexavalent Chromium methods.

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1) Non Aqueous Samples for Hexavalent Chromium: thirty (30) days from time of sampling to digestion. In addition, the alkaline digestate has been shown to be stable for up to 168 hours after digestion.

2) Aqueous Samples for Hexavalent Chromium: 24 hours from time of sampling to analysis.

3) The non-aqueous samples should arrive at four degrees centigrade.

C. Evaluation Procedure

1) Actual holding times are established by comparing the sampling dates and times on the chain of custody with the dates and times of analysis found in the laboratory data. Exceeding the holding time for a sample may result in a loss of the Hexavalent Chromium. This occurs through any number of mechanisms, such as deposition on the sample container walls or chemical activity. Therefore, from a usability standpoint, when holding time violations occur, the results that are most severely called into question are those which fall below or close to the detection limit or a cleanup level.

2) Sample temperature can be found on the chain of custody.

D. Action

1) For nonaqueous samples, if the holding time is greater than thirty days but less than or equal to thirty-two days, then the sample concentration and the nondetects are qualified and flagged the data with a "J" or "UJ" respectively. If the holding time exceeds thirty-two days, then all sample results are rejected "R".

2) For aqueous samples, if the holding time is greater than 24 hours but less than or equal to 48 hours, then the sample concentration and the nondetects are qualified and flagged the data with a "J" or "UJ" respectively. If the holding time exceeds 48 hours, then all sample results are rejected "R".

3) If sample receiving temperature is greater than 6 degrees centigrade, than qualify all associated samples.

2. Instrument Calibration Curve

A. Objective

The objective in establishing compliance requirements for satisfactory instrument calibration is to ensure that the instrument is capable of producing acceptable quantitative data.

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B. Requirements

1) The instrument must be calibrated daily (once every 24 hours) or each time the instrument is set up, whichever is more frequent.

2) The instrument standardization date and time must be included in the raw data.

3) A calibration blank and at least four (4) standards in graduated amounts and in the appropriate range (0.10 to 2.0 mg/L) are recommended in establishing the analytical curve for Method 7196A.

4) A calibration blank and at least three (3) standards bracketing the anticipated concentration range of the samples are recommended in establishing the analytical curve for Method 7199.

5) The calibration curve must have a correlation coefficient of 0.995 or greater for Method 7196A.

6) The calibration curve must have a correlation coefficient of 0.999 or greater for Method 7199.

C. Evaluation Procedure

1) By checking the raw data, verify that the instrument was calibrated at the proper frequency.

2) Verify that the correct number of standards and calibration blanks was used.

3) Verify that the date and time of sample analysis was provided.

4) Verify that the correlation coefficient for the calibration curve was greater than or equal to 0.995 for Method 7196A.

5) Verify that the correlation coefficient for the calibration curve was greater than or equal to 0.999 for Method 7199.

6) Verify that if the correlation coefficient was less than the 0.995/0.999, the laboratory analyzed a new calibration curve.

D. Action

1) If there are inconsistent time(s), date(s) or instrument IDs on the raw data sheets and reporting forms, the laboratory must be contacted and all inconsistencies must

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be resolved. If the reviewer is unable to resolve any discrepancies with the laboratory, then the reviewer must determine on a case by case basis whether to accept, qualify "J", or reject "R" the data in question.

2) If the instrument calibration was not performed or calibrated daily, then all associated data are rejected "R".

3) If the correct number of instrument calibration standards was not analyzed then all associated data are qualified "J".

5) If the correlation coefficient for the calibration curve was less than 0.995 for Method 7196, then all associated data are rejected "R".

6) If the correlation coefficient for the calibration curve was less than 0.999 for Method 7199, then all associated data are rejected "R".

3. Calibration Check Standard (CCS)

A. Objective

The CCS documents satisfactory instrument performance (calibration accuracy) during each analysis run.

B. Requirements

1) The CCS analyses must be performed at a minimum frequency of once every 10 samples during an analysis run. The CCS should be analyzed at the beginning of the run and after the last analytical sample. For Method 7199, the calibration curve must be verified by analyzing a Quality Control Sample (QCS).

2) The CCS should be at or near the mid range level of the calibration curve.

3) The same concentration for the CCS must be used throughout the sample analyses.

4) The CCS and the QCS results must fall within the control limits of 90-110% of the established value.

5) The CCS must be an independently prepared standard from a different source than that used for the initial calibration.

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C. Evaluation Procedure

1) Review the supporting raw data to verify that the CCS/QCS was performed at the proper frequency.

2) Verify that the CCS/QCS was independently prepared and the standard used was at or near the mid range level of the curve.

3) Verify that the same CCS concentration was used throughout the analyses.

4) Verify that the standard used for performing the CCS/QCS met the acceptance criteria for 90-110%.

D. Action

1) If there are inconsistent time(s), date(s) or instrument IDs on the raw data sheets and reporting forms, the laboratory must be contacted and all inconsistencies must be resolved. If the reviewer is unable to resolve any discrepancies with the laboratory, then the reviewer must determine on a case by case basis whether to accept, qualify, or reject the data questioned.

2) If the CCS/QCS analysis was not performed, then all associated sample data are rejected “R”.

3) If the CCS analysis was not performed at the correct frequency, then all associated sample data are qualified "J".

4) If the CCS was not at or near the mid range level of the curve, then all associated sample data are qualified "J".

5) If the CCS concentration was not the same throughout the analysis, then qualify "J" all associated sample data if the concentration was within the calibration range and reject "R" all associated sample data if the concentration was not within the calibration range.

6) If the CCS/QCS analysis was performed but did not meet the percent recovery requirements, use the following guidelines:

   a) If the CCS/QCS falls outside the acceptance windows but within the ranges of 80-89% or 111-120%, flag the positive hit data of associated samples as estimated (J). In the data validation report, give an indication to the data end user as to the bias of the results (i.e., if the CCS for an analyte is 115%, then it could be stated that the reported results for that analyte should be biased high.)

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b) If an analyte is not detected in a sample and the associated CCS/QCS result is greater than 110% but less than or equal to 120%, then the analytical sample determination is acceptable.

c) If the analyte is not detected in a sample and the associated CCS/QCS result is less than 90% but greater than or equal to 80%, then the detection limit may be biased low. In the data validation report, note that the detection limit for that sample may be elevated and flag the data for these samples as estimated (UJ).

d) If the CCS/QCS result is less than 80% or greater than 120%, this is indicative of severe analytical deficiencies and the data are rejected as unusable (R).

7) For a given CCS, the actions described in item 6) will affect the samples that are analyzed between the two acceptable CCSs that bracket the unacceptable CCS.

4. Calibration Blanks

A. Objective

Calibration Blanks are analyzed in order to establish that the instrument has no contamination or drifting problems, and to ensure that the instrument is capable of producing acceptable quantitative data.

B. Requirements

1) A Calibration Blank must be used in establishing the analytical curve.

2) The absolute value of the calibration blank should not exceed the Method Detection Limit (MDL).

3) A Calibration Blank must be analyzed before the initial instrument's calibration standards and after each CCS.

C. Evaluation Procedure

1) Review the supporting raw data to verify that the Calibration Blank analysis was performed and at the proper sequence.

2) Verify that the Calibration Blank absolute value was less than the MDL.

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D. Action

1) If there are inconsistent time(s), date(s) or instrument IDs on reporting forms and raw data sheets, the laboratory must be contacted and all inconsistencies resolved. If the reviewer is unable to resolve any discrepancies with the laboratory, then the reviewer must determine on a case by case basis whether to accept, qualify "J", or reject "R" the data in question.

2) If the absolute value of the Calibration Blank exceeds the MDL, then highly qualify "J" the data in all associated analytical samples.

3) If no Calibration Blank was analyzed before the initial instrument's calibration standards was run, then all associated sample data are rejected "R".

5. Preparation/Reagent Blanks and Field Blanks

A. Objective

The assessment of results regarding blank analyses is for determining the existence and magnitude of contamination problems. The criteria for the evaluation of blanks apply to all blanks, including, but not limited to preparation/reagent blanks and field blanks. The responsibility for action in the case of unsuitable blank results depends upon the circumstances and the origin of the blank. If problems with any blank exist, all associated data must be carefully evaluated to determine whether there is an inherent variability in the data set or the problem is an isolated occurrence not affecting other data.

B. Requirements

1) The laboratory preparation/reagent blank is an in-house blank the laboratory is responsible for reporting.

2) At least one preparation/reagent blank, consisting of deionized distilled water, processed through each sample preparation and analysis procedure must be prepared and analyzed with every Sample Delivery Group (SDG), or for each batch of samples digested, whichever is more frequent. (Exception: If only soil samples were analyzed, an aqueous preparation blank is not required for the associated field blank.)

3) The preparation/reagent blank concentration must be less than the method detection limit or one-tenth the regulatory limit or action level, whichever is greater or the entire batch must be redigested.

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4) The minimum field blank requirement is as follows. There should be at least one field blank/matrix/per sampling date.

5) It should be noted that inorganic analysis for trip blanks is not required unless specifically requested by NJDEP on a site by site basis.

C. Evaluation Procedure

1) Review the results for the preparation/reagent blank(s) (raw data, strip charts, printer tapes, bench sheets, etc.) in order to verify that results were accurately reported.

2) Verify that the proper number of preparation/reagent blanks was analyzed.

3) Verify that the proper number of field blanks was analyzed, as per the request of the data end user.

4) Verify that the sample concentration is not corrected for any preparation/reagent blank, trip blank or field blank values.

5) Verify that the preparation/reagent blank concentration was less than the method detection limit or one-tenth the regulatory limit or action level, whichever is greater.

D. Action

1) If there are inconsistent time(s), date(s) or instrument IDs on reporting forms and raw data sheets, the laboratory must be contacted and all inconsistencies must be resolved. If the reviewer is unable to resolve any discrepancies with the laboratory, then the reviewer must determine on a case by case basis whether to accept, qualify "J", or reject "R" the data.

2) If any samples are not associated with a preparation/reagent blank, all data from the affected samples are rejected "R". (An aqueous reagent blank for the field blank is not required if only soil samples were analyzed).

3) If no field blanks were requested for analyses or if the incorrect number was collected and analyzed, note it in the data validation report.

4) If the preparation/reagent blank or field blank results were subtracted from associated sample results, add the applicable blank results to the sample results before proceeding to steps 6) and 7) below. The laboratory must be notified of the data reporting error and a revised data report package must be submitted.

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5) If the sample concentration of Hexavalent Chromium is greater than ten (10) times the preparation/reagent blank and the blank was less than the MDL or one-tenth the regulatory limit, then no qualifications are necessary. If the preparation/reagent blank was greater than the MDL or one-tenth the regulator limit and the samples were not redigested, then all associated sample results are rejected “R”.

6) When the sample concentration of Hexavalent Chromium is less than or equal to ten (10) times the preparation/reagent blank, then the following actions are to be taken.

   a) If the concentration of Hexavalent Chromium in a sample is less than or equal to three (3) times the concentration of Hexavalent Chromium in the associated preparation/reagent blank, the presence of Hexavalent Chromium in the sample is negated due to laboratory contamination, as indicated by the preparation/reagent blank. The "B" qualifier must be reported with the analytical result.

   b) If the concentration of Hexavalent Chromium in a sample is greater than three (3) times the concentration of Hexavalent Chromium in the associated preparation/reagent blank, the presence of Hexavalent Chromium in the sample is considered "real". The "B" qualifier must be reported with the analytical result. The concentration must also be reported with the "J" qualifier and is quantitatively qualified due to preparation/reagent blank contamination.

   c) If the value of Hexavalent Chromium in the preparation/reagent blank is negative, then all positive values found in a field sample will be quantitatively qualified because of the possibility of a negative drift in the instrument and may be biased low.

   d) If the value of Hexavalent Chromium in the preparation/reagent blank is negative, then all non-detected values found in a field sample are reported "UJ" because of the possibility of a negative drift in the instrument and may give rise to a false negative (ND).

7) When the sample concentration of Hexavalent Chromium is less than or equal to ten (10) times the field blank concentration, then the following actions are to be taken.

   a) If the concentration of Hexavalent Chromium in a sample is less than or equal to three (3) times the concentration of Hexavalent Chromium in the associated field blank, then the presence of Hexavalent Chromium in the

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sample is negated due to introduced contamination, as indicated by the field blank.

b) If the concentration of Hexavalent Chromium in a sample is greater than three (3) times the concentration of Hexavalent Chromium in the associated field blank, then the presence of Hexavalent Chromium in the sample is considered "real". The concentration must be reported with the "J" qualifier and is quantitatively qualified due to field blank contamination.

6. Predigestion Spike Sample Analyses (non aqueous samples only)

A. Objective

The soluble and insoluble spiked samples analyses are designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. The insoluble spike is used to evaluate the dissolution during the digestion process.

B. Requirements

1) At least two spiked sample (soluble and insoluble) analyses must be performed on each group of samples of a similar matrix type (e.g., sediment and soil) and concentration (e.g., low and medium) for each digestion batch of samples or for each 20 samples received, whichever is more frequent.

2) If the spike analyses are performed on the same sample that was also chosen for the duplicate sample analysis, spike calculations must be performed using the results of the original sample analysis.

3) The analyte spikes must be added before sample digestion and the spike recovery must be within the control limits of 75-125%.

4) The soluble spike concentration should be at 40 mg Cr(VI)/Kg or at twice the sample concentration whichever is greater.

5) The insoluble spike concentration should be around 400 to 800 mg Cr(VI)/Kg.

C. Evaluation Procedure

1) Verify that the proper number of spikes and the proper spike concentrations were used.

2) If the spike analyses are performed on the same sample that was chosen for

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duplicate analysis, verify that the laboratory used the original sample results for calculations.

3) Spot check the raw data to verify that results were correctly calculated and reported on the spike analysis form. Predigestion spike percent recoveries (% R) are calculated as follows:

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where: SSR = Spike sample results
SR = Sample results
SA = Spike added

4) Verify that the spike recoveries fall within the control limits of 75-125%.

5) Verify that if spike recoveries fell outside the control limits of 75-125% that the entire batch was rehomogenized/redigested/reanalyzed.

6) Verify that a field sample and not a field blank was used for predigestion spike analyses as per method requirements.

D. Action

1) If there are inconsistent time(s), date(s) or instrument IDs on reporting forms and raw data sheets, the laboratory must be contacted and all inconsistencies resolved. If the reviewer is unable to resolve any discrepancies with the laboratory, then the reviewer must determine on a case by case basis whether to accept, qualify "J", or reject "R" the data in question.

2) If no spike analyses were performed for non-aqueous samples (i.e., either soil and/or sediment samples) or if a field blank was used, then all associated sample data are rejected "R".

3) If the frequency of the spike analysis exceeded 1 in 20 samples but was within 1 in 25, qualify "J" the data from samples 21-25.

4) If the frequency of a spike analysis exceeded 1 in 25 samples, reject "R" all sample data that follow the 25th sample.

5) If either spike recovery was outside the QC limit and the laboratory failed to rehomogenize/redigest/reanalyze, then all associated sample data are rejected "R".

6) If the wrong spike concentration was used, then qualify "J" hexavalent chromium in

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all associated sample data.

7) If the reported sample concentration for hexavalent chromium is greater than four times the concentration of the spike, no further action needs to be taken and the sample data are acceptable for use.

8) The following guidelines are recommended for use in evaluating data usability when the spike recoveries do not fall within control limits and the reported sample concentration is less than four times the concentration of the spike.

   a) If the spike recovery is > 125% and the reported sample results are less than the MDL, this data are acceptable for use.

   b) If the spike recovery is > 125% but ≤ 150% and the reported sample levels are greater than the MDL, flag the data as estimated "J" and indicate in the data validation report as to the potential high bias in the results.

   c) If the spike recovery is ≥ 50% but < 75% and the reported sample levels are greater than the MDL, flag the data as estimated "J" and indicate in the data validation report as to the potential low bias of the results.

   d) If an analyte is not detected and the spike recovery is ≥ 50% but < 75%, the detection limit may be biased low. In the data validation report, note that the detection limit reported by the laboratory for that sample may be biased low. Flag the data for these samples as estimated "UJ".

   e) For sample spike recovery results that fall < 50%, the data should be reported as unusable "R". This is indicative of severe analytical deficiency and the reviewer should state it in the narrative.

   f) If the spike recovery is > 150%, and the reported sample results > MDL, the data should be reported as unusable "R". This is indicative of severe analytical deficiency and the reviewer should state it in the narrative.

   g) The laboratory should be contacted and they should consider redigesting the spike sample with a higher matrix spike level.

7. Post-Digestion Spike Sample (PDS)

A. Objective

The PDS analysis is designed to verify that neither a reducing condition nor a chemical interference is affecting the analysis.

B. Requirements

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1) At least one PDS analysis must be performed on each group of samples of a similar matrix type (e.g., water and soil) for each batch of samples or for each 20 samples received, whichever is more frequent.

2) As per method requirements, samples identified as field or preparation/reagent blanks cannot be used for PDS analysis.

3) If the PDS analysis is performed on the same sample that was also chosen for the duplicate sample analysis, PDS spike recovery calculations must be performed using the result of the original sample analysis.

4) The aqueous sample chosen for PDS should be spiked at twice the sample concentration or at 30ug/L whichever is greater.

5) The soil sample chosen for PDS should be spiked at 40 mg/Kg or twice the sample concentration, whichever is greater.

6) The PDS spike recovery must be within the control limits of 85-115%.

7) If the PDS recovery is less than 85% for method 7196A, then reanalyze the PDS to determine if the low spike recovery is due to a reducing agent.

8) When the sample concentration is less than the MDL, use 0 for the sample results only for calculating the % recovery.

C. Evaluation Procedure

1) Verify that the PDS analysis was performed at the proper frequency (1 for every 20 samples) and that the proper PDS concentration was used.

2) Verify that a field sample and not a field blank or preparation/ reagent blank was used for PDS analysis as per method requirements.

3) Spot-check the raw data to verify that PDS recovery result was correctly calculated and reported. PDS percent recoveries (% R) are calculated as follows:

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where: SSR = PDS sample result
SR = Sample result
SA = Spike added

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4) Verify that the PDS recovery results fall within the specified limits of 85-115%.

5) Verify that the PDS was reanalyzed if the recovery was less than 85% for method 7196A.

6) Verify that when the addition of a PDS to a sample extends the concentration beyond the calibration range, a dilution was performed.

D. Action

1) If there are inconsistent time(s), date(s) or instrument IDs on reporting forms or raw data sheets, the laboratory must be contacted and all inconsistencies resolved. If the reviewer is unable to resolve any of the discrepancies, then the reviewer must determine on a case by case basis whether to accept, qualify "J", or reject "R" the data in question.

2) If no PDS analysis was performed for either soil and/or aqueous samples, all associated sample data are rejected "R".

3) If a field blank or preparation/ reagent blank was used for PDS analysis, then reject "R" all associated sample data.

5) If the frequency of the PDS analysis exceeded 1 in 20 samples but was within 1 in 25 qualify "J" the data from samples 21-25.

5) If the frequency of a PDS analysis exceeded 1 in 25 samples, reject "R" all sample data that follow the 25th sample.

6) If the wrong PDS concentration was used, qualify "J", Hexavalent Chromium in all associated sample data.

7) The following guidelines are recommended for use in evaluating data usability when the PDS recoveries do not fall within 85-115% limits:

   a) If the PDS recovery is > 115% and the reported sample results are less than the MDL, the data are acceptable for use.

   b) If the PDS recovery is > 115% and the reported sample concentrations are greater than the MDL, then flag data as estimated "J" and give an indication in the data validation report as to the potential high bias in the results.

   c) If the PDS recovery is less than 85%, the following actions are taken:

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i) If no reanalysis was performed, and the reported sample levels are greater than the IDL, then the data are qualified "J". In the data validation report, give an indication as to the low bias of the results.

ii) If no reanalysis was performed, and the reported sample levels are less than the IDL then the detection limit is qualified "UJ". In the data validation report, note that the detection limit reported by the laboratory for that sample may be biased low.

iii) If the laboratory reanalyzes the aliquot and the recovery is within 85-115% recovery limits, no action is needed.

iv) If the reanalysis is still outside the (recovery) limits, then qualify "J" all associated samples.

8) If the laboratory failed to make a dilution to any PDS that exceeded the calibration range, qualify " J" all associated samples.

8. Duplicate Sample Analysis

A. Objective

The duplicate sample analysis is used to evaluate the precision of the method for Hexavalent Chromium. The data reviewer can use the results of the duplicate analysis as an indicator of the precision of the sample results.

B. Requirements

1) One duplicate sample must be analyzed from each group of samples of a similar matrix type (i.e., water, and soil) and for each batch of samples or for each 20 samples received, whichever is more frequent.

2) As per method requirements, samples identified as field blanks or preparation/reagent blanks cannot be used for duplicate sample analysis.

3) Duplicate results must be reported on duplicate form in ug/L for aqueous samples and mg/Kg dry weight basis for solid samples.

4) A control limit of $\leq$20 % Relative Percent Different (RPD) shall be used if both the original and the duplicate results are greater than or equal to four times the laboratory reporting limit.
The RPD for Hexavalent Chromium is calculated as follows:

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$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where: RPD = Relative Percent Difference
S = First sample value (original)
D = Second sample value (duplicate)

5) A control limit of ± the reporting limit shall be used when either the original or the duplicate sample is less than four times the laboratory reporting limit.

C. Evaluation Procedure

1) Verify that duplicate samples were analyzed for each matrix type and at the proper frequency.

2) Spot-check the raw data to verify that the results have been correctly reported on the duplicate form.

3) Verify that a field sample was used for duplicate analysis as per method requirements.

4) Verify that the correct control limits were used.

D. Action

1) If there are inconsistent time(s), date(s), or instrument IDs on reporting forms and raw data sheets, the laboratory must be contacted and all inconsistencies resolved. If the reviewer is unable to resolve any discrepancies with the laboratory, then the reviewer must determine on a case by case basis whether to accept, qualify "J", or reject "R" the data in question.

2) If no duplicate sample was analyzed for either soil and/or aqueous matrices, then all associated sample data are rejected "R".

3) If a field blank or preparation/reagent blank was used for duplicate analysis, then all associated sample data are rejected “R”.

4) If the frequency of the duplicate analysis exceeded 1 in 20 samples but was within 1 in 25, qualify "J" the data from samples 21-25.

5) If the frequency of a duplicate analysis exceeded 1 in 25 samples, reject "R" all sample data that follow the 25th sample.

6) If the duplicate sample analyses results for Hexavalent Chromium fall outside the

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control windows of 20% RPD or ± the reporting limit, whichever is appropriate, the results in all associated samples of the same matrix type should be flagged as estimated "J".

9. Laboratory Control Sample Analysis (non aqueous samples only)

A. Objective

The laboratory control sample analysis (LCS) is designed to serve as a monitor of the efficiency of the digestion procedure. The inability of the laboratory to successfully analyze a known LCS is indicative of an analytical problem related to digestion/sample preparation procedures and/or instrument operations.

B. Requirements

1) One LCS must be analyzed for every SDG of non-aqueous samples received or for each batch of samples digested whichever is more frequent. Results for each Hexavalent Chromium should be reported on the LCS form.

2) The LCS must be prepared by the laboratory.

3) The LCS percent recovery (%R) control limit is 80% -120%.

C. Evaluation

1) Verify that the LCS was analyzed and at the proper frequency.

2) Review the LCS form and verify that the results fall within the specified control limits.

3) Spot check the raw data (printouts, strip chart, and bench sheets) to verify the reported recoveries on the LCS form.

4) Verify that if the LCS recovery fell outside the control limits of 80% -120% that the LCS was reanalyzed.

D. Action

1) If there are inconsistent time(s), date(s) or instrument IDs on reporting forms and raw data sheets, the laboratory must be contacted and all inconsistencies resolved. If the reviewer is unable to resolve any discrepancies with the laboratory, then the reviewer must determine on a case by case basis whether to accept, qualify "J", or reject "R" the data.

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2) If the frequency of a LCS analysis exceeded 1 in 20 samples, qualify "J" the data from the 21st sample on.

3) If the LCS was not within control limit and the laboratory failed to reanalyze the LCS, then reject "R" all associated samples.

4) If the LCS was not within control limit, and the laboratory reanalyzed and the LCS is still outside the control limit then qualify "J" all associated samples.

10. Sample Result Verification

A. Objective

The sample results verification process checks the correctness of the data acquisition, computation, transcription and the validity of the calibration curve construction.

B. Requirements

1) It is implicit within the USEPA SW-846 Final Update III document that all required data reduction, reporting and documentation be performed and presented in such a manner so as to ensure the data package is both complete as well as free of computational and/or transcription errors.

2) Percent solids determinations are required for all non-aqueous samples. Sample dry weight corrections are made using the percent solids results.

3) USEPA SW-846 methods 3060A, 7196A & 7199 require pH adjustments for each sample and their final readings recorded in a laboratory notebook.

   a) Method 3060A-pHs of all analytical solutions for non-aqueous samples analyzed by Method 7196A must be adjusted within a range of 7.0 - 8.0.

   b) Method 3060A-pHs of all analytical solutions for non-aqueous samples analyzed by Method 7199 must be adjusted within a range of 9.0 - 9.5.

   c) Method 7196A - pHs of all analytical solutions must be adjusted within a range of 1.5 - 2.5.

4) USEPA SW-846 Method 3060A requires that the digestion solution temperature range of 90 - 95 degrees centigrade must be maintain for at least 60 minutes. The laboratory should monitor and record the temperature in the laboratory notebook for one sample.

5) For Method 7199, each sample should be injected twice and the relative standard

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deviation of the duplicates should be $\leq$ 20 % RPD.

C. Evaluation Procedure

1) Examine the raw data for any anomalies (i.e., negative absorbance, omissions, etc.).

2) Verify that there were no computational errors in sample concentration by re-calculating the results for Hexavalent Chromium in a percentage of samples.

3) Calculations

   a) For aqueous samples, calculate the Hexavalent Chromium results as follows:

      Hexavalent Chromium in mg/L = A x E

      Where: A = concentration from the calibration curve in mg/L.
      E = Dilution (if necessary)

   b) For solid samples, when concentrations are reported as mg/Kg on a dry basis use the following formula.

      $$\text{mg Hexavalent Chromium/Kg sample} = \frac{A \times B \times E}{C \times D}$$

      Where: A = concentration from the calibration curve in mg/L.
      B = Final digested volume in liters.
      C = Wet weight of sample in kilograms.
      D = % Solids/100
      E = Dilution (if necessary)

4) Verify that there were no transcription errors by checking the raw data versus the analytical result summary sheet.

5) a) Verify that percent solids analysis for all non-aqueous samples was performed.

   b) Verify the percent solids determinations by spot checking the laboratory results using the following formula.

      $$\% \text{ Solids} = \frac{\text{Sample dry weight}}{\text{Sample wet weight}} \times 100$$

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6) Verify that the laboratory has provided pH readings for Methods 3060A, 7196A and/or 7199.

7) Verify that the pH reading(s) were within the specified range(s) for each sample.

6) Verify that the sample digestion solution temperature readings were provided, and were within a temperature range of 90 – 95 degrees centigrade.

9) Verify that for Method 7199, each sample was injected twice and the relative standard deviation of the duplicates was $\leq$ 20 % RPD.

D. Action

1) If any raw data anomalies were found, the reviewer should use judgement on how the sample data would be affected.

2) If differences are identified between the laboratory reported result and the reviewer calculated result, the following actions should be taken:

   a) If the laboratory reported result is within 10% of the reviewer calculated result and the difference could be attributed to rounding, no corrective action is required.

   b) If the laboratory reported result differs by 10% from the reviewer calculated result, but not attributable to rounding, try to determine the source(s) of error. If this cannot be determined, the laboratory should be contacted about the sample result discrepancy. If an error is confirmed, request submission of corrected data sheets from the laboratory. Summarize all actions taken in the Data Validation Report.

3) Transcription errors that affect the data shall be noted in Data Validation Report. Also, the laboratory shall be contacted and the submission of corrected data sheets shall be requested.

4) If the percent solids data were not provided then note the deficiency in the data validation report. The results are qualified and possibly biased low.

5) If the pH readings are not provided, then contact the laboratory. The data are conditionally rejected pending satisfactory verification of the pH results.

6) If the laboratory failed to record the pH data in a laboratory notebook, then the

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following actions are taken:

a) The data are qualified with an unknown bias if the positive results exceed the clean-up action level.

b) The data are suspect if the positive results are below the clean-up action level.

c) The "non-detected" data are rejected because the possibility of false NDs exists.

7) If the sample digestion solution temperature readings are not provided, then contact the laboratory. The data are conditionally rejected pending satisfactory verification of the digestion solution temperature.

8) If the laboratory failed to record the sample digestion solution temperature in a laboratory notebook, then the following actions are taken:

a) The data are qualified with an unknown bias if the positive results exceed the clean-up action level.

b) The data are suspect if the positive results are below the clean-up action level.

c) The "non-detected" data are rejected because the possibility of false NDs exists.

7) For Method 7199, if the samples were not injected twice, then qualify the affected sample.

8) For Method 7199, if the sample was injected twice but relative standard deviation of the duplicates was > 20 % RPD, then qualify the affected sample.

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APPENDIX I - GLOSSARY OF TERMS

ABSORBANCE - a measure of decrease in incident light passing through a sample into the detector. It is defined mathematically as:

$$A = \frac{I\ (solvent)}{I\ (solution)} = \log \frac{Io}{I}$$

Where, I = radiation intensity

ALIQUOT- a measured portion of a field sample taken for analysis.

ANALYSIS DATE/TIME - the date and military time (24-hour clock) of the injection of the sample, standard, or blank into the analysis system.

ANALYTE - the element or ion an analysis seeks to determine; the element of interest.

AUTOZERO - zeroing the instrument at the proper wavelength. It is equivalent to running a standard blank with the absorbance set at zero.

AVERAGE INTENSITY - the average of two different injections (exposures).

BACKGROUND CORRECTION - a technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

BATCH - the basic unit for analytical quality control is the analytical batch. The analytical batch is defined as 20 samples or less which are analyzed together with the same method sequence and the same lots of reagents and the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition (e.g. groundwater, sludge, ash, etc.).

CALIBRATION - the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of reagents as used in the sample preparation.

CALIBRATION BLANK - a volume of digestion solution/distilled water.

CALIBRATION CHECK STANDARD - analytical standard run every 1O analytical samples to verify the calibration of the analytical system.

COEFFICIENT OF VARIATION (CV) - the standard deviation as a percent of the arithmetic mean.

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CONTROL LIMITS - a range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

CORRELATION COEFFICIENT - a number (r) which indicates the degree of dependence between two variables (concentration - absorbance). The more
dependent they are the closer the value to one. Determined based on the least squares line.

DAY - unless otherwise specified, day shall mean calendar day.

DIGESTION LOG - an official record of the sample preparation (digestion).

DUPLICATE - a second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

FIELD BLANK – an analytical sample design to assess specific sources of field contamination.

HOLDING TIME - the elapsed time expressed in days from the date of sample collection until the date of its analysis.

Holding time = (sample analysis date - sampling date)

INDEPENDENT STANDARD - a laboratory-prepared standard solution that is composed of Hexavalent Chromium from a different source other than that used in the standards for the initial calibration.

INJECTION - introduction of the analytical sample into the instrument excitation system for the purpose of measuring absorbance, emission or concentration of an analyte. May also be referred to as exposure.

INSTRUMENT DETECTION LIMIT (IDL) - determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x IDL on three nonconsecutive days with seven consecutive measurements per day.

INTERFERENTS - substances which affect the analysis for the element of interest.

MATRIX - the predominant material of which the sample to be analyzed is composed.

MATRIX SPIKE - aliquot of a sample (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

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METHOD DETECTION LIMIT (MDL) - The minimum concentration of a substance that can be measured and reported with 99 % confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. The procedures for determining the MDL are located in Chapter 1 of SW-846.

PREPARATION BLANK (reagent blank, method blank) - an analytical control that contains distilled, deionized water and reagents, which is carried through the entire analytical procedure (digested and analyzed). An aqueous method blank is treated with the same reagents as a sample with a water matrix; A solid method blank is treated with the same reagents as a soil sample.

PROTOCOL - a compilation of the procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control.

ROUNDING RULES - if the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

RUN - a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the USEPA, SW-846.

SOIL - synonymous with soil/sediment or sediment as used herein.

SPECTROMETER - An instrument with an entrance slit, a dispersing device, and one or more exit slits, in which measurements are made at selected wavelengths within the spectral range, or by scanning over the range.

STOCK SOLUTION - a standard solution which can be diluted to derive other standards.

WET WEIGHT - the weight of a sample aliquot including moisture (undried).

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VALIDATION SOP

pH (solid/Liquid), Total Phosphorus, Total Kjeidhal Nitrogen, AVS/SEM and Ammonia

USEPA Contract Laboratory Program National Functional guidelines for Inorganic Superfund Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 01/10

Office of Superfund Remediation and Technology Innovation (OSRTI)
United States Environmental Protection Agency (USEPA)
Washington, DC 20460

OSWER 9240.1-51
USEPA-540-R-10-011

January 2010

USEPA Contract Laboratory Program

National Functional Guidelines

for Inorganic Superfund Data Review

Final

FOIA_07123_0003552_0892

NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (hereafter referred to as USEPA) and other governmental employees. They do not constitute rule making by USEPA, and may not be relied upon to create a substantive or procedural right enforceable by any other person. The Government may take action that is at variance with the policies and procedures in this manual.

This document can be obtained from the USEPA's Contract Laboratory Program (CLP) Web site at:
http://www.epa.gov/superfund/programs/clp/guidance.htm

January 2010

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ACRONYMS

AA	Atomic Absorption
ASB	Analytical Services Branch
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
CO	Contracting Officer
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
CVAA	Cold Vapor AA
DAT	Data Assessment Tool
DF	Dilution Factor
DQO	Data Quality Objective
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICS	Interference Check Sample
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
MDL	Method Detection Limit
NIST	National Institute of Standards and Technology
OSRTI	Office of Superfund Remediation and Technology Innovation
OSWER	Office of Solid Waste and Emergency Response
PE	Performance Evaluation
%D	Percent Difference
%R	Percent Recovery
%RI	Percent Relative Intensity
%RSD	Percent Relative Standard Deviation
%S	Percent Solids
PO	Project Officer
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center Coordinator
SDG	Sample Delivery Group
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TAL	Target Analyte List
TR/COC	Traffic Report/Chain of Custody Documentation
USEPA	United States Environmental Protection Agency

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TARGET ANALYTE LIST

Al	Aluminum
Sb	Antimony
As	Arsenic
Ba	Barium
Be	Beryllium
Cd	Cadmium
Ca	Calcium
Cr	Chromium
Co	Cobalt
Cu	Copper
CN	Cyanide
Fe	Iron
Pb	Lead
Mg	Magnesium
Mn	Manganese
Hg	Mercury
Ni	Nickel
K	Potassium
Se	Selenium
Ag	Silver
Na	Sodium
Tl	Thallium
V	Vanadium
Zn	Zinc

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INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the usability of analytical data generated through the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) ISM01.X Inorganic Superfund Methods (Multi-Media, Multi-Concentration), hereinafter referred to as the ISM01.2 SOW, and any future editorial revisions of ISM01.2. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process. It should not be used to establish specific contract compliance; use of this document to evaluate data generated under Inorganic SOWs other than ISM01.2 is cautioned. Definitive guidance is provided where performance should be fully under a laboratory's control [e.g., blanks, calibration verification standards, Interference Check Samples (ICSs), Quality Control (QC) audit samples, and instrument performance checks (tuning)], while general guidance is provided for evaluating subjective data that is affected by site conditions.

The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and QC criteria established in the SOW, and (b) the usability and extent of bias of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to site-specific technical requirements, the need to facilitate the progress of specific projects, and availability for re-sampling. To make judgments at this level requires the reviewer to have a complete understanding of the intended use of the data. The reviewer is strongly encouraged to establish a dialogue with the user prior to and after data review, to discuss usability issues and to answer questions regarding the review. It should also be understood that in all cases, data which do not meet specified criteria are never to be fully acceptable without qualification.

The reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as professional judgment, should also be used to determine the ultimate usability of data, especially in those cases where all data does not meet specific technical criteria. The reviewer should also be aware that minor modifications to some of the analytical methods may be made through the "Request for Quote (RFQ) for Modified Analysis" to meet site-specific requirements, and that these modifications could affect certain validation criteria such as Contract Required Quantitation Limits (CRQLs) and Target Analyte Lists (TALs). A copy of any modification request made to the analytical method should be included in the data package by the laboratory.

Please visit the CLP Web site at http://www.epa.gov/superfund/programs/clp/index.htm for more information on how to obtain service through the CLP.

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DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process. If the Regions choose to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review.

U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

DATA PACKAGE INSPECTION

For data obtained through the Contract Laboratory Program (CLP), the Data Assessment Tool (DAT) report is a useful tool in the data review process. The DAT report incorporates Contract Compliance Screening (CCS) and National Functional evaluation results, and is transmitted electronically via email. For more information about DAT, please refer to the following CLP Web site:

http://www.epa.gov/superfund/programs/clp/data_assessment.htm

The DAT report will identify any missing and/or incorrect information in the data package. The CLP laboratory may submit a reconciliation package for any missing items or to correct data.

If there are any concerns regarding the data package, contact the CLP Project Officer (CLP PO) from the Region where the samples were taken. For personnel contact information, please refer to the following CLP Web site:

http://www.epa.gov/superfund/programs/clp/contacts.htm

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PRELIMINARY REVIEW

This document is for the review of analytical data generated through the ISM01.2 SOW and any future editorial revisions of ISM01.2. To use this document effectively, the reviewer should have an understanding of the analytical method and a general overview of the Sample Delivery Group (SDG) or sample Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the sample data package [e.g., Modified Analysis requests, Traffic Report/Chain of Custody (TR/COC) documentation, SDG Narratives, etc.].

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The reviewer should contact the appropriate Regional Contract Laboratory Program Project Officer (CLP PO) to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

The SDGs or Cases routinely have unique samples that require special attention from the reviewer. These include field blanks and trip blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The sampling records (e.g., TR/COC records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken, and
2. The complete list of samples with information on:
    a. Sample matrix;
    b. Field blanks*;
    c. Field duplicates*;
    d. Field spikes*;
    e. PE samples*;
    f. Shipping dates;
    g. Preservatives;
    h. Types of analysis; and
    i. Laboratories involved.

    * If applicable.

The TR/COC documentation includes sample descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

The laboratory's SDG Narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, preservation, and unusual events should be documented in the SDG Narrative. The reviewer should also inspect any email or telephone/communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office (SMO), and the USEPA Region.

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DATA REVIEW NARRATIVE

A Data Review Narrative, including the Inorganic Data Review Summary form (see Appendix B), must accompany the laboratory data forwarded to the intended data recipient (client) or user to promote communication. A copy of the Data Review Narrative should be submitted to the CLP PO assigned oversight responsibility for the laboratory producing the data.

The Data Review Narrative should include comments that clearly identify the problems associated with a Case or SDG and state the limitations of the data. Documentation should also include the CLP Sample Number, analytical method, extent of the problem, and assigned qualifiers.

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ICP-AES DATA REVIEW

The inorganic data requirements for Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) to be reviewed during validation are listed below:

I. Preservation and Holding Times 7
II. Calibration 9
III. Blanks 12
IV. Inductively Coupled Plasma - Interference Check Sample (ICP-ICS) 15
V. Laboratory Control Sample (LCS) 18
VI. Duplicate Sample Analysis 20
VII. Spike Sample Analysis 22
VIII. ICP Serial Dilution 25
IX. Regional Quality Assurance (QA) and Quality Control (QC) 27
X. Overall Assessment 28
Calculations for ICP-AES 30

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An Example Analytical Sequence for ICP-AES

S0
S
S
S
S
S
ICV
ICB
ICSA
ICSAB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

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Preservation and Holding Times

A. Review Items:

Form IA-IN, Form IB-IN, Form XII-IN, Form XIII-IN, Traffic Report/Chain of Custody (TR/COC) documentation, Form DC-1, raw data, and the Sample Delivery Group (SDG) Narrative checking for: pH; cooler temperature; holding time; and other sample conditions.

B. Objective:

The objective is to ascertain the validity of the analytical results based on the sample condition, and the holding time of the sample from the date of collection to the date of analysis.

C. Criteria:

1. The technical holding time criteria for aqueous/water metal samples is 180 days, preserved (with nitric acid) to pH ≤ 2. The addition of nitric acid to adjust the pH is only required for aqueous/water samples.
2. The technical holding time criteria for soil/sediment metal samples is 180 days, based on the technical holding time criteria for aqueous/water samples.
3. The technical holding time criteria for wipe and air filter samples is 180 days, based on the technical holding time criteria for aqueous/water samples.
4. Aqueous/water and soil/sediment samples shall be maintained at 4□C (±2□C) until preparation. Wipe and air filter samples may be maintained at room temperature until preparation.

D. Evaluation:

Technical holding times are established by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form XIII-IN and the raw data. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. Verify that the analysis dates on the Form XIIIs and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication that there were problems with the samples, the integrity of the samples may be compromised and professional judgment should be used to evaluate the effect of the problem on the sample results.

E. Action:

NOTE: Apply the action to each sample for which the preservation or holding time criteria was not met.

1. If the pH of aqueous/water metal samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH of the sample to ≤ 2 at the time of sample receipt. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of the metal(s) of interest. Qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-), and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data, based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-), and qualify non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment, wipe, and filter samples, it is left to the discretion of the data reviewer whether to apply aqueous/water holding time criteria to soil/sediment, wipe, or filter samples. If they are applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.
5. When holding times are grossly exceeded, note it for Contract Laboratory Program Project Officer (CLP PO) action.

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Table 1. Technical Holding Time Actions for ICP-AES Analysis

Preservation & Holding Time Results	Action for Samples
Aqueous/water metals samples received with pH > 2 and pH not adjusted	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Aqueous/water and soil/sediment metals samples not maintained at 4□C (±2□C)	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Aqueous/water Metals > 180 day	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Soil/sediment, wipe, filter Metals > 180 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

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Calibration

A. Review Items:

Form II-IN (Part A), Form XI-IN, Form XIII-IN, Form XVI-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective:

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for the metals on the Inorganic Target Analyte List (TAL). Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

C. Criteria:

1. Initial Calibration

The instruments shall be successfully calibrated each time the instrument is set up and after Continuing Calibration Verification (CCV) failure. The calibration date and time shall be included in the raw data.

a. A blank and at least five calibration standards shall be used to establish each analytical curve. At least one of these standards shall be at or below the CRQL. All measurements shall be within the instrument working range where the interelement correction factors are valid. A minimum of three replicate exposures are required for standardization, all Quality Control (QC), and sample analyses. The average result of all the multiple exposures for the standardization, QC, and sample analyses shall be used. The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The curve must have a correlation coefficient ≥ 0.995. The percent differences calculated for all of the non-zero standards must be within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

2. Initial and Continuing Calibration Verification (ICV and CCV)

The acceptance criteria for the ICVs and CCVs are presented in Table 2:

Table 2. Acceptance Criteria for ICVs and CCVs,

Analytical Method	Inorganic Analytes	ICV/CCV Low Limit (% of True Value)	ICV/CCV High Limit (% of True Value)
ICP-AES	Metals	90	110

a. Initial Calibration Verification (ICV)

1) Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s). If the ICV %R falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.

2) Only if the ICV is not available from USEPA, analyses shall be conducted using a certified solution of the analytes from an independent commercial standard source, at a concentration level other than that used for instrument calibration, but within the calibrated range.

3) The ICV solution shall be run at each analytical wavelength used for analysis.

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b. Continuing Calibration Verification (CCV)

1) To ensure accuracy during the course of each analytical run, the CCV shall be analyzed and reported for each wavelength used for the analysis of each analyte.
2) The CCV standard shall be analyzed at a frequency of every two hours during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample.
3) The analyte concentration(s) in the CCV standard(s) shall be different than the concentration used for the ICV, and at a concentration equivalent to the mid level of their respective calibration curves.
4) The same CCV standard solution shall be used throughout the analysis runs for a Sample Delivery Group (SDG).
5) The CCV shall be analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

D. Evaluation:

1. Verify that the instrument was calibrated each time the instrument was set up, utilizing a blank and at least five calibration standards, one of which was at or below the CRQL.
2. Confirm that the measurements were within the working range, and were the average result of at least three replicate exposures.
3. Verify that the ICV and CCV standards were analyzed for each analyte at the proper frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.
4. Recalculate one or more of the ICV and CCV %R using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form IIA-IN.

$$\%R = \frac{\text{Found (value)}}{\text{True (value)}} \times 100$$

Where,

Found (value) = Concentration (in µg/L) of each analyte <u>measured</u> in the analysis of the ICV or CCV solution

True (value) = Concentration (in µg/L) of each analyte in the ICV or CCV source

E. Action:

NOTES: For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

For CCVs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical run.

1. If the instrument was not calibrated each time the instrument was set up, qualify the data as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank and at least one at or below CRQL), use professional judgment to qualify results that are ≥ Method Detection Limit (MDL) as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).
2. If the correlation coefficient is <0.995, percent differences are outside the ±30% limit, or the y-intercept ≥CRQL, qualify sample results that are ≥MDL as estimated (J) and non-detects as estimated (UJ).

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3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
   a. If the ICV or CCV %R is < 75%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as estimated low (J-) or unusable (R).
   b. If the ICV or CCV %R falls within the range of 75-89%, qualify sample results that are ≥ MDL as estimated low (J-), and qualify non-detects as estimated (UJ).
   c. If the ICV or CCV %R falls within the range of 111-125%, qualify sample results that are ≥ MDL as estimated high (J+).
   d. If the ICV or CCV %R is within the range of 111-125%, non-detects should not be qualified.
   e. If the ICV or CCV %R is > 125%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+) or unusable (R). Non-detects should not be qualified.
   f. If the %R is > 160%, qualify all results that are ≥ MDL as unusable (R).
4. If the laboratory failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
5. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
6. If calibration criteria are grossly exceeded, note this for CLP Project Officer (CLP PO) action.

NOTE: For truly critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

Table 3. Calibration Actions for ICP-AES Analysis

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as unusable (R)
Calibration incomplete	Use professional judgment Qualify results that are ≥ MDL as estimated (J) or unusable (R) Qualify non-detects as estimated (UJ) or unusable (R)
Correlation coefficient <0.995; %D outside ±30%; y-intercept ≥CRQL	Qualify results that are ≥MDL as estimated (J) Qualify non-detects as estimated (UJ)
ICV/CCV %R < 75%	Qualify results that are ≥ MDL as estimated low (J-) or unusable (R) Qualify all non-detects as unusable (R)
ICV/CCV %R 75-89%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICV/CCV %R 111-125%	Qualify results that are ≥ MDL as estimated high (J+)
ICV/CCV %R > 125%	Qualify results that are ≥ MDL as estimated high (J+) or unusable (R)
ICV/CCV %R > 160%	Qualify results that are ≥ MDL as unusable (R)

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Blanks

A. Review Items:

Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective:

The objective of blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, calibration blanks, field blanks, etc.). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blank(s).
2. The Initial Calibration Blank (ICB) shall be analyzed after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) during the initial calibration of the instrument (see Section II.C.1).
3. A Continuing Calibration Blank (CCB) shall be analyzed at each wavelength used for the analysis, immediately after every Continuing Calibration Verification (CCV). The CCB shall be analyzed at a frequency of every two hours during the run. The CCB shall be analyzed at the beginning of the run, and again after the last CCV that was analyzed after the last analytical sample of the run. The CCB result (absolute value) shall not exceed the Contract Required Quantitation Limit (CRQL) of each analyte for which analysis is performed.
4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. The Preparation Blank consists of reagent water or a clean wipe or filter processed through the appropriate sample preparation and analysis procedure.
5. If any analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10 times (10x) the Preparation Blank concentration. Otherwise, all samples associated with that Preparation Blank with the analyte's concentration < 10x the Preparation Blank concentration, and > CRQL, should be redigested and reanalyzed for that analyte (except for an identified field blank). The laboratory is not to correct the sample concentration for the blank value.
6. If the concentration of the Preparation Blank for a certain analyte is < (-CRQL) all samples reported < 10x the CRQL (associated with that analyte in that blank), should be redigested and reanalyzed.

D. Evaluation:

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the proper frequency and location during the run, and Preparation Blanks were prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. Review the results reported on the Blank Summary (Form III-IN), as well as the raw data for all blanks, and verify that the results were accurately reported.
3. Evaluate all of the associated blanks for the presence of target analytes. Verify that if target analytes were present in a Preparation Blank or if a concentration was < (-CRQL) the affected samples were redigested and reanalyzed. Verify that if target analytes were present in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

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E. Action:

NOTES: For ICBs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

For CCBs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.

For Preparation Blanks that do not meet the technical criteria, apply the action to all samples prepared in the same preparation batch.

1. If the appropriate blanks were not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
   a. Any blank (including Preparation Blanks) reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank (including Preparation Blanks) reported with a negative result, whose value is < (-CRQL) qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
   b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. Specific "method" actions include:
   a. If the absolute value of an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the affected samples were not reanalyzed, report non-detects and results that are ≥ MDL, but ≤ CRQL as CRQL-U. For results that are > CRQL but < Blank Result, use professional judgment to qualify the data as unusable (R) or to report the results at the level of the blank with a "U" qualifier. Use professional judgment to qualify results that are > Blank Result. Note this situation for CLP PO action and record it in the Data Review Narrative.
   b. If the absolute value of the concentration of the Preparation Blank is ≤ CRQL, report non-detects and results that are ≥ MDL but ≤ CRQL as CRQL-U. Use professional judgment to quality results that are > CRQL.
   c. If any analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redigested and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redigestion (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redigest and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.

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Table 4. Blank Actions for ICP-AES Analysis

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
ICB/CCB	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < Blank Result	Report at level of Blank Result with a "U" or qualify data as unusable (R)
		> Blank Result	Use professional judgment
ICB/CCB	≤ (-MDL) but ≥ (-CRQL)	≥ MDL, or non-detect	Use professional judgment
ICB/CCB	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)
Preparation Blank	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < 10x the Blank Result	Use professional judgment to qualify results as unusable (R) or estimated high (J+)
		≥ 10x the Blank Result	No action
Preparation Blank	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
Preparation Blank	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)

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Inductively Coupled Plasma - Interference Check Sample (ICP-ICS)

A. Review Items:

Form IVA-IN, Form XIII-IN, instrument printouts, and raw data.

B. Objective:

The Inductively Coupled Plasma (ICP) - Interference Check Sample (ICS) verifies the analytical instrument's ability to overcome interferences typical of those found in samples.

NOTE: The laboratory should have analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for these reported elements.

C. Criteria:

1. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A, for all wavelengths used for each analyte reported by Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES).
2. An ICS must be run at the beginning of each sample analysis run. The ICS is not to be run prior to the Initial Calibration Verification (ICV), and is to be immediately followed by a Continuing Calibration Verification (CCV), which will be followed by a Continuing Calibration Blank (CCB).
3. Results for the analysis of ICS Solution A must fall within the control limits of ± Contract Required Quantitation Limit (CRQL), or ± 20% of the true value (whichever is greater) for the analytes and interferents.
4. Results for the analysis of ICS Solution AB must fall within the control limits of ± CRQL, or ± 20% of the true value (whichever is greater) for the analytes and interferents included in the solution.
5. If the value of an ICS result exceeds ± CRQL, or ± 20% of true value (whichever is greater) criteria, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the new calibration then reverified, and the affected samples reanalyzed.
6. The ICS should be obtained from USEPA if available, and analyzed according to the instructions supplied with the solutions. The ICS may be prepared with the interferents at 2x the level specified in the Statement of Work (SOW) if high levels of interferents are found in the field samples. If the ICS is not available from USEPA, an independent ICS solution shall be prepared with the interferent and analyte concentrations at the levels specified in the method.

D. Evaluation:

1. Verify using the raw data (ICP instrument printout) that the ICS was analyzed at the proper frequency and location during the analytical run.
2. Evaluate the ICS raw data for results with an absolute value that is ≥ Method Detection Limit (MDL) for those analytes which are not present in the ICS solution.
3. Recalculate using the raw data and the following equation, one or more of the analyte Percent Recoveries (%R), and verify that the recalculated value agrees with the laboratory-reported values on Form IV-IN.

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$$\%R = \frac{\text{Found (value)}}{\text{True (value)}} \times 100$$

Where,

Found (value) = Concentration (in µg/L) of each analyte interferent measured in the analysis of ICS Solution A or ICS Solution AB

True (value) = Concentration (in µg/L) of each analyte or interferent in ICS Solution A or ICS Solution AB

4. If the value of an ICS result exceeds the ± CRQL, or ± 20% of true value (whichever is greater) criteria, and the laboratory failed to terminate the analysis, and take the appropriate corrective action, note this for Contract Laboratory Program Project Officer (CLP PO) action and record in the Data Review Narrative. Use professional judgment to assess the data.

E. Action:

NOTE: For an ICS that does not meet the technical criteria, apply the action to all samples analyzed in the analytical run.

1. The raw data should, but may not, contain results for interferents. If not, the reviewer shall use professional judgment to qualify the data. If the data does contain results for interferents, the reviewer should apply the following actions to samples with concentrations of interferents that are comparable to, or greater than, their respective levels in the ICS:
   a. If the ICS %R for an analyte or interferent is > 120% (or greater than the true value + CRQL, as applicable) and the sample results are non-detects, the data should not be qualified.
   b. If the ICS %R for an analyte or interferent is > 120% (or greater than the true value + CRQL, as applicable) qualify sample results that are ≥ MDL as estimated high (J+). If the ICS %R (or true value) grossly exceeds the limits, use professional judgment to qualify the data.
   c. If the ICS %R for an analyte or interferent falls within the range of 50-79% (or less than the true value - CRQL, as applicable) qualify sample results that are ≥ MDL as estimated low (J-).
   d. If the ICS recovery for an analyte falls within the range of 50-79% (or less than the true value - CRQL, as applicable), the possibility of false negatives exists. Qualify sample non-detects as estimated (UJ).
   e. If the ICSAB %R for an analyte or interferent is < 50%, qualify all sample results that are ≥ MDL as estimated low (J-) and all sample non-detects as unusable (R).
2. If results that are ≥ MDL are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS, qualify sample results that are ≥ MDL as estimated high (J+). Non-detects should not be qualified.
3. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is ≥ MDL, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents, qualify non-detects for the affected analytes as estimated (UJ), and results that are ≥ MDL but < 10x the absolute value of the negative result as estimated low (J-).

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4. In general, ICP-AES sample data can be accepted if the concentrations of Al, Ca, Fe, and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. If these elements are present at concentrations greater than the level in the ICS, or other elements are present in the sample at > 10 mg/L, the reviewer should investigate the possibility of other interference effects as given in the ICP-AES method or as indicated by the laboratory's interelement correction factors reported on Forms XA-IN and XB-IN for that particular instrument. The analyte concentration equivalents presented in the method should be considered only as estimated values since the exact value of any analytical system is instrument-specific. Therefore, estimate the concentration produced by an interfering element. If the estimateis > 2x the CRQL, and also > 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).
5. If the raw data does not contain results for the interferents, note it in the Data Review Narrative.
6. Actions regarding the interpretation and/or the subsequent qualification of ICP data due to the ICS analytical results can be extremely complex. Use professional judgment to determine the need for the associated sample data to be qualified. The reviewer may need to obtain additional information from the laboratory. All interpretive situations should then be recorded in the Data Review Narrative.
7. If the ICS acceptance criteria are grossly exceeded, note the specifics for CLP PO action.

Table 5. Interference Check Actions for ICP-AES Analysis

Interference Check Sample Results	Action for Samples
ICS %R > 120% (or greater than true value + CRQL)	Qualify results that are ≥ MDL as estimated high (J+)
ICS %R 50-79% (or less than true value - CRQL)	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICSAB %R < 50%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Potential false positives in field samples with interferents	Qualify results that are ≥ MDL as estimated high (J+)
Potential false negatives in field samples with interferents	Qualify results that are ≥ MDL but < 10x(|negative value|) as estimated low (J-) Qualify non-detects as estimated (UJ)

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Laboratory Control Sample (LCS)

A. Review Items:

Form VII-IN, Form XII-IN, preparation logs, instrument printouts, and raw data.

B. Objective:

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation.

C. Criteria:

1. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples.
   a. One LCS shall be prepared and analyzed for every group of aqueous/water or soil/sediment samples in a Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. The LCS shall be spiked such that the final digestate contains each analyte at two times the CRQL for the associated matrix.
   b. All LCS Percent Recoveries (%R) must fall within the control limits of 70-130%, except for Sb and Ag which must fall within the control limits of 50-150%. If the %R for the aqueous/water and soil/sediment LCS falls outside of the control limits, the analysis should be terminated, the problem corrected, and the samples prepared with that LCS redigested and reanalyzed.
   c. One LCS shall be prepared and analyzed for each group of wipe or filter samples in an SDG, or with each batch of wipe or filter samples digested, whichever is more frequent. The wipe or filter LCS shall be spiked such that the final digestate contains each analyte at two times the CRQL for the associated matrix.
   d. All wipe or filter sample LCS %R shall fall within the control limits of 70-130%.

D. Evaluation:

1. Verify using Form VII-IN, Form XII-IN, and raw data that the appropriate number of required LCSs were prepared and analyzed for the SDG.
2. Evaluate Form VII-IN and verify that all results for each analyte fall within the established control limits.
   a. Check the raw data to verify that the %Rs on Form VII-IN were accurately transcribed. Recalculate one or more of the reported %Rs using the following equation:

$$\%R = \frac{\text{Found (value)}}{\text{True (value)}} \times 100$$

Where,

Found (value) = Concentration of each analyte (in µg/L, mg/kg, or µg) <u>measured</u> in the analysis of the LCS

True (value) = Concentration of each analyte (in µg/L, mg/kg, or µg) in the LCS

3. Verify that the LCS was prepared at the same time as the associated samples using the same procedures.

E. Action:

If the LCS criteria are not met, the laboratory performance and method accuracy are in question. Professional judgment should be used to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data associated with an LCS that does not meet the required criteria.

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For an LCS that does not meet the technical criteria, apply the action to all samples in the same preparation batch.

1. **Aqueous/Water and Soil/Sediment LCS:**
   a. If the LCS %R falls within the range of 40-69% (20-49% for Ag and Sb), qualify sample results that are ≥ Method Detection Limit (MDL) as estimated low (J-). If the LCS %R is > 130% (150% for Ag and Sb), qualify sample results that are ≥ MDL as estimated high (J+).
   b. If the LCS recovery is > 130% (150% for Ag and Sb) and the sample results are non-detects, the data should not be qualified.
   c. If the LCS recovery falls within the range of 40-69% (20-49% for Ag and Sb), qualify non-detects as estimated (UJ).
   d. If LCS %R is < 40% (<20% for Ag and Sb), qualify all results that are ≥ MDL as estimated low (J-) and all non-detects as unusable (R).
   e. If the LCS %R is > 150% (170% for Ag and Sb), qualify all affected data (both detects and non-detects) as unusable (R).
2. **Wipe/Filter LCS:**
   a. If the LCS %R is in the range of 40-69%, qualify sample results that are ≥ MDL as estimated low (J-) and qualify non-detects as estimated (UJ).
   b. If the LCS %R is < 40%, qualify all results that are ≥ MDL as estimated low (J-) and all non-detects as unusable (R).
   c. If the LCS %R is > 130% and the sample results are non-detects, do not qualify the data.
   d. If the LCS %R is > 130%, qualify all results ≥ MDL as estimated high (J+).
3. If a laboratory fails to analyze an LCS with each SDG, or if a laboratory consistently fails to generate acceptable LCS recoveries, note this for CLP Project Officer (CLP PO) action.
4. Whenever possible, the potential effects on the data due to out-of-control LCS results should be noted in the Data Review Narrative.

Table 6. LCS Actions for ICP-AES Analysis

LCS Result	Action for Samples
Aqueous/Water and Soil/Sediment %R 40-69% (20-49% Ag, Sb)	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
Aqueous/Water and Soil/Sediment %R > 130% (150% Ag, Sb)	Qualify results that are ≥ MDL as estimated high (J+)
Aqueous/Water and Soil/Sediment %R < 40% (<20% Ag, Sb)	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Aqueous/Water and Soil/Sediment %R > 150% (>170% Ag, Sb)	Qualify all results as unusable (R)
Wipe/Filter %R 40-69%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
Wipe/Filter %R > 130%	Qualify results that are ≥ MDL as estimated high (J+)
Wipe/Filter %R < 40%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

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Duplicate Sample Analysis

A. Review Items:

Cover Page, Form VI-IN, Form XII-IN, instrument printouts, and raw data.

B. Objective:

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analyses are also performed to generate data that determines the long-term precision of the analytical method on various matrices. Non-homogenous samples can impact the apparent method precision. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.
2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each Sample Delivery Group (SDG). Duplicates are not required for wipe or filter samples. Duplicates cannot be averaged for reporting on Form I-IN. Additional duplicate sample analyses may be required by USEPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.
3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL).
4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form VI-IN. If both samples are non-detects, the RPD is not calculated for Form VI-IN.

NOTE: The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

D. Evaluation:

1. Verify from the Cover Page, Form XII-IN, and the raw data that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.
2. Evaluate Form VI-IN and the raw data to verify that all duplicate results for each analyte and method fall within the established control limits.
3. Verify that a field blank or PE sample was not used for duplicate analysis.
4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form VI-IN:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = Relative Percent Difference

S = Sample Result (original)

D = Duplicate Result

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E. Action:

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix if the reviewer considers the samples to be sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data when determining similarity, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions]. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the duplicate, and thus that only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for CLP Project Officer (CLP PO) action.
2. If the results from a duplicate analysis for a particular analyte fall outside the appropriate control limits, qualify sample results that are ≥ Method Detection Limit (MDL) as estimated (J) and non-detects as estimated (UJ).
3. If a field blank or PE sample was used for the duplicate sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
4. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 7. Duplicate Sample Actions for ICP-AES Analysis

Duplicate Sample Results	Action for Samples
Both original sample and duplicate sample > 5x the CRQL and RPD > 20%*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)

* The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

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Spike Sample Analysis

A. Review Items:

Cover Page, Form V-IN (Part A & B), Form XII-IN, instrument printouts, and raw data.

B. Objective:

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. Non-homogenous samples can impact the apparent method recovery. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three. If the spike is added to the sample before the digestion (e.g., prior to the addition of other reagents), it is referred to as a spiked sample, pre-digestion spike, or Matrix Spike. If the spike is added to the sample after the completion of the digestion procedures, it is referred to as a post-digestion spike, or analytical spike.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for spiked sample analysis.
2. At least one spiked sample (pre-digestion) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each Sample Delivery Group (SDG). Matrix Spikes are not required for wipe or filter samples.
3. When the Matrix Spike recovery falls outside of the control limits and the sample result is < four times (4x) the spike added, a post-digestion spike shall be performed for those analytes that do not meet the specified criteria. An aliquot of the remaining unspiked sample shall be spiked at 2x the indigenous level or 2x the Contract Required Quantitation Limit (CRQL), whichever is greater.

NOTE: Post-digestion spikes are not required for Ag.

4. The spike Percent Recovery (%R) shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.
5. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for the purpose of determining %R.

NOTE: The final spike concentrations required for the various target analytes are presented in the methods described in the Statement of Work (SOW).

D. Evaluation:

1. Verify using the Cover Page, Form VA-IN, Form XII-IN, and raw data, that the appropriate number of required spiked samples were prepared and analyzed for the SDG.
2. Verify that a field blank or PE sample was not used for the spiked sample analysis.
3. Evaluate Form VA-IN and the raw data to verify that all pre-digestion spiked sample results for each required analyte fall within the established control limits. If not, verify that a post-digestion/post-distillation spike was prepared and analyzed.
4. Recalculate using the raw data, one or more of the %R using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms V (A & B)-IN:

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$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

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

NOTE: When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purpose of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms VA-IN and VB-IN.

E. Action:

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the Matrix Spike, and thus that only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-digestion spike was not performed, note this for CLP PO action.
4. If the Matrix Spike %R is < 30%, verify that a post-digestion spike was analyzed (if required). If the post-digestion spike %R is < 75% or is not performed, qualify sample results that are ≥MDL as estimated low (J-) and non-detects as unusable (R). If the post-digestion spike %R is ≥ 75%, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
5. If the Matrix Spike %R is 30-74% and the sample results are ≥ MDL, verify that a post-digestion spike was analyzed (if required). If the %R for the post-digestion is also < 75% or is not performed, qualify the affected data as estimated low (J-). If the %R for the post-digestion spike is ≥ 75%, qualify the affected data as estimated (J).
6. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).
7. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.

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8. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, verify that a post-digestion spike was analyzed if required. If the %R for the post-digestion spike is also > 125% or is not performed, qualify the affected data as estimated high (J+). If the %R for the post-digestion spike is ≤ 125%, qualify the affected data as estimated (J).
9. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 8. Spike Sample Actions for ICP-AES Analysis

Spike Sample Results	Action for Samples
Matrix Spike %R < 30% Post-digestion spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion Spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ)
Matrix Spike %R > 125% Post-digestion spike %R > 125%	Qualify affected results that are ≥ MDL as estimated high (J+)
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	Qualify affected results that are ≥ MDL as estimated (J)
Matrix Spike %R < 30% No post-digestion spike performed (e.g., not required for Ag)	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R 30-74% No post-digestion spike performed (e.g., not required for Ag)	Qualify affected results that are ≥ MDL as estimated low (J-) and non-detects as estimated (UJ)
Matrix Spike %R > 125% No post-digestion spike performed (e.g., not required for Ag)	Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified

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ICP Serial Dilution

A. Review Items:

Form I-IN, Form VIII-IN, instrument printouts, and raw data.

B. Objective:

The serial dilution of samples quantitated by Inductively Coupled Plasma - AtomicEmission Spectroscopy (ICP-AES) determines whether or not significant physical or chemical interferences exist due to sample matrix.

C. Criteria:

1. An ICP Serial Dilution analysis shall be performed on a sample from each group of samples with a similar matrix type (e.g., water, soil, wipe, or filter) or for each Sample Delivery Group (SDG), whichever is more frequent.
2. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for the ICP Serial Dilution analysis.
3. If the analyte concentration is sufficiently high [concentration in the original sample is > 50 times (50x) the Method Detection Limit (MDL)], the Percent Difference (%D) between the original determination and the serial dilution analysis (a five-fold dilution) after correction for dilution shall be less than 10.

NOTE: The above criteria are **method requirements** for serial dilution samples, regardless of the sample matrix type. However, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., %D < 15) to be assessed against serial dilution soil samples.

D. Evaluation:

1. Verify that a field blank or PE sample was not used for the serial dilution analysis.
2. Check the raw data and recalculate the %D using the following equation. Verify that the serial dilution analysis results, and the calculated %D results agree with the values reported by the laboratory on Form VIII-IN:

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

Where,

I = Initial Sample Result

S = Serial Dilution Result

3. Check the raw data for any evidence of positive or negative interference (results from the diluted sample which are significantly different than the original sample), possibly due to high levels of dissolved solids in the sample, ionization effects, etc.

E. Action:

NOTE: For a serial dilution that does not meet the technical criteria, apply the action to all samples of the same matrix if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar

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to the serial dilution sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for serial dilution, and thus that only the field sample used to prepare the serial dilution sample should be qualified.

1. If the required %D criteria are not met, qualify all affected results that are ≥ MDL as estimated (J) and all affected non-detects as estimated (UJ).
2. If evidence of positive or negative interference is found, use professional judgment to qualify the associated sample data. Note the potential effects on the reported data in the Data Review Narrative.
3. It should be noted for Contract Laboratory Program Project Officer (CLP PO) action and in the Data Review Narrative if a field blank or PE sample was used for the serial dilution analysis.

Table 9. Serial Dilution Actions for ICP-AES Analysis

Serial Dilution Result	Action for Samples
Sample concentration > 50x MDL and %D > 10*	Qualify affected results that are ≥ MDL as estimated (J) Qualify affected non-detects as estimated (UJ)
Interferences present	Use professional judgment

* The above criteria are **method requirements** for serial dilution samples, regardless of the sample matrix type. However, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., %D < 15) to be assessed against serial dilution soil samples.

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Regional Quality Assurance (QA) and Quality Control (QC)

A. Review Items:

Form I-IN, instrument printouts, and raw data.

B. Objective:

Regional QA/QC samples refer to any QA and/or QC sample initiated by the Region, including field duplicate samples, Performance Evaluation (PE) samples, blind spikes and blind blanks. The use of these QA/QC samples is highly recommended (e.g., the use of field duplicates can provide information on sampling precision and homogeneity).

C. Criteria:

Criteria are determined by each Region.

D. Evaluation:

Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Compare results for PE samples to the acceptance criteria for the specific PE samples if possible.

Calculate the Relative Percent Difference (RPD) between field duplicates and provide this information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Contract Laboratory Program Project Officer (CLP PO) action.

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Overall Assessment

A. Review Items:

Entire sample data package, data review results, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data (including any confirmation data).

B. Objective:

1. The objective is to ensure that the reported sample quantitation results are accurate. It is appropriate for the data reviewer to make professional judgments and express concerns, as well as to comment on the validity of the overall data for a Case. This is particularly appropriate when there are several Quality Control (QC) criteria that are outside of the specification parameters. The additive nature of QC factors that fall outside of specification parameters is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user of data quality and data limitations to assist that user to avoid inappropriate use of the data, while not precluding any consideration of the data. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the acceptance or performance criteria were provided. The Inorganic Review Summary (see Appendix B) and supplementary documentation must be included with the review.

C. Criteria:

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method.

D. Evaluation:

Examine the raw data to verify that correct calculations of the sample results were reported by the laboratory. Digestion logs, instrument printouts, etc., should be compared to the reported sample results recorded on the appropriate Inorganic Summary Forms (Form I-IN through Form XIII-IN).

1. Evaluate any technical problems not previously addressed.
2. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).
3. Verify that appropriate methods and amounts were used in preparing the samples for analysis. If reduced volumes were used, verify that the laboratory had received Contract Laboratory Program Project Officer (CLP PO) approval for the use of the reduced volume.
4. Verify that there are no transcription or reduction errors [e.g., dilutions, Percent Solids (%S), sample weights, etc.] on one or more samples.
5. Verify that results fall within the calibrated range(s) of the Inductively Coupled Plasma (ICP) instrument(s).
6. If appropriate information is available, the reviewer may assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the Standard Operating Procedure(s) (SOPs), and communication with the user concerning the intended use and desired quality of these data.

E. Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.

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2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the Sample Delivery Group (SDG) Narrative for Contract Laboratory Program Project Officer (CLP PO) action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.
3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

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Calculations for ICP-AES

Aqueous/Water Samples by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES):

The concentrations determined in the digestate are to be reported in units of µg/L:

$$\text{Concentration (ug/L)} = C \times \frac{V_f}{V} \times DF$$

Where,

C = Instrument value in µg/L (The average of all replicate exposures.)

V_f = Final digestion volume (mL)

V = Initial aliquot amount (mL)

DF = Dilution Factor

Soil/Sediment Samples by ICP-AES:

The concentrations determined in the digestate are to be reported on the basis of the dry weight of the sample, in units of mg/kg:

$$\text{Concentration (drywt.)(mg/kg)} = C \times \frac{V_f}{W \times S} \times DF/1000$$

Where,

C = Instrument value in µg/L (The average of all replicate exposures).

V_f = Final digestion volume (mL)

W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, substitute the value of the MDL (µg/L) or CRQL (µg/L) into the "C" term in the equation above.

Calculate the adjusted MDL or adjusted CRQL for soil/sediment samples as follows:

$$\text{Adjusted Concentration (mg/kg)} = C \times \frac{W_M}{W \text{ x } S} \times \frac{V_f}{V_M} \times DF$$

Where,

C = MDL or CRQL (mg/kg)

W_M = Minimum method required aliquot amount (g) (1.00 g or 0.50 g)

W = Initial aliquot amount (g)

V_M = Method required final sample digestion volume (mL) (100 mL or 50 mL)

V_f = Final digestion volume (mL)

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

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Wipe/Filter Mass:

$$\text{Mass } (\mu g) = C \times V_f \times DF / 1000$$

Where,

C = Instrument value in µg/L (The average of all replicate exposures).

V_f = Final digestion volume (mL)

DF = Dilution Factor

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ICP-MS DATA REVIEW

The inorganic data requirements for Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) to be reviewed during validation are listed below:

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An Example Analytical Sequence for ICP-MS

Tune
S0
S
S
S
S
S
ICV
ICB
ICSA
ICSAB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

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I. Preservation and Holding Times

A. Review Items:

Form IA-IN, Form IB-IN, Form XII-IN, Form XIII-IN, Traffic Report/Chain of Custody (TR/COC) documentation, Form DC-1, raw data, and the Sample Delivery Group (SDG) Narrative checking for: pH; cooler temperature; holding time; and other sample conditions.

B. Objective:

The objective is to determine the validity of the analytical results based on the sample condition, and the holding time of the sample from the date of collection to the date of analysis.

C. Criteria:

1. The technical holding time criteria for aqueous/water metal samples is 180 days, preserved (with nitric acid) to pH ≤ 2. The addition of nitric acid to adjust the pH is only required for aqueous/water samples.
2. The technical holding time criteria for soil/sediment metal samples is 180 days, based on the technical holding time criteria for aqueous/water samples.
3. Samples shall be maintained at 4□C (±2□C) until preparation and analysis.

D. Evaluation:

Technical holding times are established by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form XIII-IN, and the raw data. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. Verify that the analysis dates on the Form XIIIs and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. Review the SDG Narrative to determine if the aqueous/water samples were received in glass containers. If there is an indication that there were problems with the samples, the integrity of the samples may be compromised and professional judgment should be used to evaluate the effect of the problem on the sample results.

E. Action:

NOTE: Apply the action to each sample for which the preservation or holding time criteria was not met.

1. If the pH of aqueous/water metals samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH to ≤2 at the time of sample receipt. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of the metal(s) of interest. Qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-), and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-), and qualify non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment samples, it is left to the discretion of the data reviewer whether to apply 180 day holding time criteria to soil/sediment samples. If it is applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.
5. When holding times are grossly exceeded, note this for Contract Laboratory Program Project Officer (CLP PO) action.

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Table 10. Technical Holding Time Actions for ICP-MS Analysis

Preservation & Holding Time Results	Action for Samples
Aqueous/water metals samples received with pH > 2 and pH not adjusted	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Metals samples not maintained at 4□C (±2□C)	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Aqueous/water Metals > 180 day	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Soil/sediment Metals > 180 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

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ICP-MS Tune Analysis

A. Review Items:

Form XIV-IN, instrument printouts, and raw data.

B. Objective:

The Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) tune serves as an initial demonstration of instrument stability and precision.

C. Criteria:

1. Prior to calibration, the laboratory shall analyze or scan the ICP-MS tuning solution at least five times (5x) consecutively. The tuning solution contains 100 µg/L of Be, Mg, Co, In, and Pb. The solution shall contain all required isotopes of the above elements. The laboratory shall make any adjustments necessary to bring peak width within the instrument manufacturer's specifications and adjust mass resolution to within 0.1 u over the range of 6-210 u.
2. The Percent Relative Standard Deviation (%RSD) of the absolute signals for all analytes in the tuning solution must be < 5%.

D. Evaluation:

1. Verify using the raw data and Form XIV-IN that the appropriate number of analyses or scans of the ICP-MS tuning solution were performed, and that the appropriate analytes were present in the solution.
2. Verify using the raw data and Form XIV-IN that the mass calibration falls within the limits for each isotope of each analyte.
3. Verify using the raw data and Form XIV-IN that the %RSD is < 5% for each isotope of each analyte.
4. Check the raw data to verify that the reported average mass and %RSD on Form XIV-IN was accurately calculated. Recalculate one or more of the average masses and %RSDs for an isotope using the following equations:

$$\text{Mean} = \frac{\sum x}{n}$$

Where,

x = Mass from analysis

n = Number of analyses

$$\text{Percent Relative Standard Deviation} = \frac{\sigma_{n-1} \times 100}{\bar{x}}$$

Where,

$\bar{x}$ = Mean

σ_{n-1} = Standard Deviation

E. Action:

NOTE: For ICP-MS tunes that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

1. If the ICP-MS instrument was not tuned prior to calibration, the sample data should be qualified as unusable (R).

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2. If the tuning solution was not analyzed or scanned at least 5x consecutively or the tuning solution does not contain the required analytes spanning the analytical range, the reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be recorded in the Data Review Narrative and noted for Contract Laboratory Program Project Officer (CLP PO) action.
3. If the resolution of the mass calibration is not within 0.1 u for any isotope in the tuning solution, qualify all analyte results that are ≥ Method Detection Limit (MDL) associated with that isotope as estimated (J), and all non-detects associated with that isotope as estimated (UJ). The situation should be recorded in the Data Review Narrative and noted for CLP PO action.
4. If the %RSD exceeds 5% for any isotope in the tuning solution, qualify all sample results that are ≥ MDL associated with that tune as estimated (J), and all non-detects associated with that tune as estimated (UJ). The situation should be recorded in the Data Review Narrative and noted for CLP PO action.

Table 11. ICP-MS Tune Actions for ICP-MS Analysis

ICP-MS Tune Results	Action for Samples
Tune not performed	Qualify all results as unusable (R)
Tune not performed properly	Use professional judgment
Resolution of mass calibration not within 0.1 u	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)
%RSD > 5%	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)

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Calibration

A. Review Items:

Form II-IN (Part A), Form XI-IN, Form XIII-IN, Form XVI-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective:

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for the metals on the Inorganic Target Analyte List (TAL). Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continual basis.

C. Criteria:

1. Initial Calibration

The instruments shall be successfully calibrated each time the instrument is set up and after Continuing Calibration Verification (CCV) failure. The calibration date and time shall be included in the raw data.

a. A blank and at least five calibration standards shall be used to establish each analytical curve. At least one standard shall be at or below the CRQL. All measurements shall be within the instrument working range. A minimum of three replicate scans are required for standardization and all Quality Control (QC) and sample analyses. The average result of all the multiple scans for the standardization, QC, and sample analyses shall be used. The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The curve must have a correlation coefficient of ≥ 0.995. The calculated percent differences for all of the non-zero standards must be within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

2. Initial and Continuing Calibration Verification (ICV and CCV)

The acceptance criteria for the ICVs and CCVs are presented in Table 12:

Table 12. Acceptance Criteria for ICV and CCV Standards

Analytical Method	Inorganic Analytes	ICV/CCV Low Limit (% of True Value)	ICV/CCV High Limit (% of True Value)
ICP-MS	Metals	90	110

a. Initial Calibration Verification (ICV)

1) Immediately after each ICP-MS system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s). If the ICV Percent Recovery (%R) falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.

2) Only if the ICV is not available from USEPA, analyses shall be conducted using a certified solution of the analytes from an independent commercial standard source, at a concentration level other than that used for instrument calibration, but within the calibrated range.

3) The ICV solution shall be run at each analytical mass used for analysis.

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b. Continuing Calibration Verification (CCV)

1) To ensure accuracy during the course of each analytical run, the CCV shall be analyzed and reported for each mass used for the analysis of each analyte.
2) The CCV standard shall be analyzed at a frequency of every two hours during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample.
3) The analyte concentration(s) in the CCV standard(s) shall be different than the concentration used for the ICV, and at a concentration equivalent to the mid level of their respective calibration curves.
4) The same CCV standard solution shall be used throughout the analysis runs for a Sample Delivery Group (SDG).
5) The CCV shall be analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant calibration verification reanalyzed.

D. Evaluation:

1. Verify that the instrument was calibrated each time the instrument was set up, utilizing a blank and at least five calibration standards, one of which was at or below the CRQL.
2. Confirm that the measurements were within the working range, and were the average result of at least three replicate exposures.
3. Verify that the ICV and CCV standards were analyzed for each analyte at the proper frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.
4. Recalculate one or more of the ICV and CCV %Rs using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form IIA-IN.

$$\%R = \frac{\text{Found (value)}}{\text{True (value)}} \times 100$$

Where,

Found (value) = Concentration (in µg/L) of each analyte <u>measured</u> in the analysis of the ICV or CCV solution

True (value) = Concentration (in µg/L) of each analyte in the ICV or CCV source

E. Action:

NOTE: For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

For CCVs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical run.

1. If the instrument was not calibrated each time the instrument was set up, qualify the data as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank and at least one at or below CRQL), use professional judgment to qualify results that are ≥ MDL as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).
2. If the correlation coefficient is <0.995, percent differences are outside the ±30% limit, or the y-intercept ≥CRQL, qualify sample results that are ≥MDL as estimated (J) and non-detects as estimated (UJ).

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3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
   a. If the ICV or CCV %R is < 75%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as estimated low (J-) or unusable (R).
   b. If the ICV or CCV %R falls within the range of 75-89%, qualify sample results that are ≥MDL as estimated low (J-), and qualify non-detects as estimated (UJ).
   c. If the ICV or CCV %R falls within the range of 111-125%, qualify sample results that are ≥ MDL as estimated high (J+).
   d. If the ICV or CCV %R falls within the range of 111-125%, non-detects should not be qualified.
   e. If the ICV or CCV %R is > 125%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+) or unusable (R). Non-detects should not be qualified.
   f. If the %R is > 160%, qualify all results that are ≥ MDL as unusable (R).
4. If the laboratory failed to provide adequate calibration information, the USEPA Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
5. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
6. If calibration criteria are grossly exceeded, note this for Contract Laboratory Program Project Officer (CLP PO) action.

NOTE: For truly critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

Table 13. Calibration Actions for ICP-MS Analysis

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as unusable (R)
Calibration incomplete	Use professional judgment Qualify results that are ≥ MDL as estimated (J) or unusable (R) Qualify non-detects as estimated (UJ) or unusable (R)
Correlation coefficient <0.995; %D outside ±30%; y-intercept ≥CRQL	Qualify results that are ≥MDL as estimated (J) Qualify non-detects as estimated (UJ)
ICV/CCV %R < 75%	Qualify results that are ≥ MDL as estimated low (J-) or unusable (R) Qualify all non-detects as unusable (R)
ICV/CCV %R 75-89%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICV/CCV %R 111-125%	Qualify results that are ≥ MDL as estimated (J)
ICV/CCV %R > 125%	Qualify results that are ≥ MDL as estimated high (J+) or unusable (R)
ICV/CCV %R > 160%	Qualify results that are ≥ MDL as unusable (R)

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Blanks

A. Review Items:

Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective:

The objective of blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, calibration blanks, field blanks, etc.). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blank(s).
2. The Initial Calibration Blank (ICB) shall be analyzed after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) during the initial calibration of the instrument (see Section III.C.1).
3. A Continuing Calibration Blank (CCB) shall be analyzed at each mass used for the analysis, immediately after every Continuing Calibration Verification (CCV). The CCB shall be analyzed at a frequency of every two hours during the run. The CCB shall be analyzed at the beginning of the run, and again after the last CCV that was analyzed after the last analytical sample of the run. The CCB result (absolute value) shall not exceed the Contract Required Quantitation Limit (CRQL) of each analyte for which analysis is performed.
4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.
5. If any analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10 times (10x) the Preparation Blank concentration. Otherwise, all samples associated with that Preparation Blank with the analyte's concentration < 10x the Preparation Blank concentration, and > CRQL, should be redigested and reanalyzed for that analyte (except for an identified field blank). The laboratory is not to correct the sample concentration for the blank value.
6. If the concentration of the Preparation Blank for a certain analyte is < (-CRQL), all samples reported < 10x the CRQL (associated with that analyte in that blank), should be redigested and reanalyzed.

D. Evaluation:

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the proper frequency and location during the run, and Preparation Blanks were prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. Review the results reported on the Blank Summary (Form III-IN), as well as the raw data for all blanks, and verify that the results were accurately reported.
3. Evaluate all of the associated blanks for the presence of target analytes. Verify that if target analytes were present in a Preparation Blank or if a concentration was < (-CRQL), the affected samples were redigested and reanalyzed. Verify that if target analytes were present in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

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E. Action:

NOTES: For ICBs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

For CCBs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.

For Preparation Blanks that do not meet the technical criteria, apply the action to all samples prepared in the same preparation batch.

1. If the appropriate blanks were not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
   a. Any blank (including Preparation Blanks) reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank (including Preparation Blanks) reported with a negative result, whose value is < (-CRQL), qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
   b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. Specific "method" actions include:
   a. If the absolute value of an ICB or a CCB result > CRQL, the analysis should be terminated. If the analysis was not terminated and the affected samples were not reanalyzed, report non-detect and results that are ≥ MDL but ≤ CRQL as CRQL-U. For results that are > CRQL but < Blank Result, use professional judgment to qualify the data as unusable or to report the results at the level of the blank with a "U" qualifier. Use professional judgment to qualify results that are > Blank Result. Note this situation for CLP PO action and record it in the Data Review Narrative.
   b. If the absolute value of the concentration of the Preparation Blank is ≤ CRQL, report non-detects and results that are ≥ MDL but ≤ CRQL as CRQL-U. Use professional judgment to qualify results that are > CRQL.
   c. If any analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of that analyte in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redigested and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redigestion (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redigest and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.

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Table 14. Blank Actions for ICP-MS Analysis

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
ICB/CCB	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < Blank Result	Report at level of Blank Result with a "U" or qualify data as unusable (R)
		> Blank Result	Use professional judgment
ICB/CCB	≤ (-MDL), but ≥ (-CRQL)	≥ MDL, or non-detect	Use professional judgment
ICB/CCB	< (-CRQL)	< 10x CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)
Preparation Blank	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < 10x the Blank Result	Qualify results as unusable (R) or estimated high (J+)
		≥ 10x the Blank Result	No action
Preparation Blank	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
Preparation Blank	< (-CRQL)	< 10x CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)

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Inductively Coupled Plasma - Interference Check Sample (ICP-ICS)

A. Review Items:

Form IVA-IN, Form IVB-IN, Form XIII-IN, instrument printouts, and raw data.

B. Objective:

The Inductively Coupled Plasma-Interference Check Sample (ICP-ICS) verifies the analytical instrument's ability to overcome isobaric interferences typical of those found in samples.

C. Criteria:

1. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A, for all masses used for each analyte or interferent reported by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS).
2. An ICS must be run at the beginning of each analysis run. The ICS is not to be run prior to the Initial Calibration Verification (ICV), and shall be immediately followed by a Continuing Calibration Verification/ContinuingCalibration Blank (CCV/CCB).
3. Results for the ICP-MS analysis of the ICS Solution A shall fall within the control limits of ± 2x the CRQL, or ± 20% of the true value (whichever is greater) for the analytes included in the solution.
4. Results for the ICP-MS analysis of the ICS Solution AB must fall within the control limits of ± 2x the CRQL, or ± 20% of the true value (whichever is greater) for the analytes included in the solution.
5. If the value of an ICS result exceeds ± 2x the CRQL, or ± 20% of true value (whichever is greater) criteria, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the new calibration then reverified, and all analytical samples analyzed since the last compliant ICS reanalyzed.
6. The ICS should be obtained from USEPA, if available, and analyzed according to the instructions supplied with the solutions. If the ICS is not available from USEPA, an independent ICS solution shall be prepared with the interferent and analyte concentrations at the levels specified in the method.

D. Evaluation:

1. Verify using the raw data (ICP instrumental printout) that the ICS was analyzed at the proper frequency and location during the analytical run.
2. Evaluate the ICS raw data for results with an absolute value that is ≥ Method Detection Limit (MDL) for those analytes that are not present in the ICS solution.
3. Recalculate using the raw data and the following equation, one or more of the analyte Percent Recoveries (%R), and verify that the recalculated value agrees with the laboratory-reported values on Form IV-IN.

$$\%R = \frac{\text{Found (value)}}{\text{True (value)}} \times 100$$

Where,

Found (value) = Concentration (in µg/L) of each analyte interferent measured in the analysis of ICS Solution A or ICS Solution AB

True (value) = Concentration (in µg/L) of each analyte or interferent in ICS Solution A or ICS Solution AB

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4. If the value of an ICS result exceeds ± 2x the CRQL, or ± 20% of true value (whichever is greater) criteria, and the laboratory failed to terminate the analysis and take the appropriate corrective action, note this for Contract Laboratory Program Project Officer (CLP PO) action and record in the Data Review Narrative. Use professional judgment to assess the data.

E. Action:

NOTE: For an ICS for ICP-MS that does not meet the technical criteria, apply the action to all samples reported from the analytical run.

1. The raw data may not contain results for interferents. In this case, the reviewer shall use professional judgment to qualify the data. If the data does contain results for interferents, the reviewer should apply the following actions to samples with concentrations of interferents that are comparable to, or greater than, their respective levels in the ICS:
   a. the ICS %R for an analyte is > 120% (or greater than the true value + 2x the CRQL as applicable) and the sample results are non-detects, the data should not be qualified.
   b. If the ICS %R for an analyte is > 120% (or greater than the true value + 2x the CRQL as applicable) qualify sample results that are ≥ MDL as estimated high (J+). If the ICS %R (or true value) grossly exceeds the limits, use professional judgment to qualify the data.
   c. If the ICS %R for an analyte falls within the range of 50-79% (or less than the true value - 2x the CRQL as applicable) qualify sample results that are ≥ MDL as estimated low (J-).
   d. If the ICS recovery for an analyte falls within the range of 50-79% (or less than the true value - 2x the CRQL as applicable), the possibility of false negatives exists. Qualify sample non-detects as estimated (UJ).
   e. If the ICSAB %R for an analyte or interferent is < 50%, qualify all sample results that are ≥ MDL and all sample non-detects as unusable (R).
2. If results that are ≥ MDL are observed for analytes which are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS, qualify sample results that are ≥ MDL as estimated high (J+). Non-detects should not be qualified.
3. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is ≥ MDL, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents, qualify non-detects for the affected analytes as estimated (UJ), and results that are ≥ MDL but < 10x the absolute value of the negative result as estimated low (J-).
4. If the raw data does not contain results for the interferents, note this in the Data Review Narrative.
5. Actions regarding the interpretation and/or the subsequent qualification of ICP data due to the ICS analytical results can be extremely complex. Use professional judgment to determine the need for the associated sample data to be qualified. The reviewer may need to obtain additional information from the laboratory. All interpretive situations should then be recorded in the Data Review Narrative.
6. If the ICS acceptance criteria are grossly exceeded, note the specifics for CLP PO action.

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Table 15. Interference Check Actions for ICP-MS Analysis

Interference Check Sample Results	Action for Samples
ICS %R > 120% (or > true value + 2x the CRQL)	Qualify results that are ≥ MDL as estimated high (J+)
ICS %R 50-79% (or < true value - 2x the CRQL)	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICSAB %R < 50%	Qualify all sample data as unusable (R)
Potential false positives in field samples with interferents	Qualify results that are ≥ MDL as estimated high (J+)
Potential false negatives in field samples with interferents	Qualify results that are ≥ MDL but < 10x(|negative value|) as estimated low (J-) Qualify non-detects as estimated (UJ)

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Laboratory Control Sample (LCS)

A. Review Items:

Form VII-IN, Form XII-IN, preparation logs, instrument printouts, and raw data.

B. Objective:

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation.

C. Criteria:

1. LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples.
   a. LCS shall be prepared and analyzed for every group of aqueous/water or soil/sediment samples in a Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. The aqueous/water and soil/sediment LCS shall be spiked such that the final digestate contains each analyte at two times the CRQL for the associated matrix.
   b. All LCS Percent Recoveries (%R) must fall within the control limits of 70-130%. If the %R for the LCS falls outside of the control limits, the analysis should be terminated, the problem corrected, and the samples prepared with that LCS redigested and reanalyzed.

D. Evaluation:

1. Verify using Form VII-IN, Form XII-IN, and raw data that the appropriate number of required LCSs were prepared and analyzed for the SDG.
   a. Check the LCSs documentation from the supplier and verify that the LCSs identification and the control limits for it are listed on the Form VII-IN match those of the specific LCSs used for the analysis.
   b. Evaluate Form VII-IN and verify that all results for each analyte fall within the established control limits.
2. Check the raw data to verify that the %Rs on Form VII-IN were accurately transcribed. Recalculate one or more of the reported %Rs using the following equation:

$$\%R = \frac{\text{Found (value)}}{\text{True (value)}} \times 100$$

Where,

Found (value) = Concentration of each analyte (in µg/L or mg/kg) <u>measured</u> in the analysis of the LCS

True (value) = Concentration of each analyte (in µg/L or mg/kg) in the LCS

3. Verify that the LCS was prepared at the same time as the associated samples using the same procedures.

E. Action:

If the LCS criteria are not met, the laboratory performance and method accuracy are in question. Professional judgment should be used to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data associated with an LCS that does not meet the required criteria.

For an LCS that does not meet the technical criteria, apply the action to all samples in the same preparation batch.

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1. **LCS:**
   a. If the LCS %R falls within the range of 40-69%, qualify sample results that are ≥ Method Detection Limit (MDL) as estimated low (J-). If the LCS %R is > 130%, qualify sample results that are ≥ MDL as estimated high (J+).
   b. If the LCS recovery is > 130% and the sample results are non-detects, the data should not be qualified.
   c. If the LCS recovery falls within the range of 40-69%, qualify non-detects as estimated (UJ).
   d. If LCS %R is < 40%, qualify all results that are ≥ MDL as estimated low (J-) and all non-detects as unusable (R).
   e. If the LCS %R is > 150%, qualify all affected data (both detects and non-detects) as unusable (R).
2. If a laboratory fails to analyze an LCS with each SDG, or if a laboratory consistently fails to generate acceptable LCS recoveries, note this for Contract Laboratory Program Project Officer (CLP PO) action
3. Whenever possible, the potential effects on the data due to out-of-control LCS results should be noted in the Data Review Narrative.

Table 16. LCS Actions for ICP-MS Analysis

LCS Result	Action for Samples
Aqueous/Water and Soil/Sediment %R 40-69%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
Aqueous/Water and Soil/Sediment %R > 130%	Qualify results that are ≥ MDL as estimated high (J+)
Aqueous/Water and Soil/Sediment %R < 40%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Aqueous/Water and Soil/Sediment %R > 150%	Qualify all results as unusable (R)

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Duplicate Sample Analysis

A. Review Items:

Cover Page, Form VI-IN, Form XII-IN, instrument printouts, and raw data.

B. Objective:

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analyses are also performed to generate data that determines the long-term precision of the analytical method on various matrices. Non-homogenous samples can impact the apparent method precision. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.
2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each Sample Delivery Group (SDG). Duplicates cannot be averaged for reporting on Form I-IN. Additional duplicate sample analyses may be required by USEPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.
3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL).
4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form VI-IN. If both samples are non-detects, the RPD is not calculated for Form VI-IN.

NOTE: The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

D. Evaluation:

1. Verify from the Cover Page, Form XII-IN, and the raw data that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.
2. Evaluate Form VI-IN and the raw data to verify that all duplicate results for each analyte and method fall within the established control limits.
3. Verify that a field blank or PE sample was not used for duplicate analysis.
4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form VI-IN:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = Relative Percent Difference

S = Sample Result (original)

D = Duplicate Result

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E. Action:

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples to be sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, when determining similarity, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions]. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the duplicate, and thus only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples were not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If the results from a duplicate analysis for a particular analyte fall outside the appropriate control limits, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
3. If a field blank or PE sample was used for the duplicate sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
4. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 17. Duplicate Sample Actions for ICP-MS Analysis

Duplicate Sample Results	Action for Samples
Both original sample and duplicate sample > 5x the CRQL and RPD> 20%*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)

* The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

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Spike Sample Analysis

A. Review Items:

Cover Page, Form V-IN (Parts A & B), Form XII-IN, instrument printouts, and raw data.

B. Objective:

The spike sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. Non-homogenous samples can impact the apparent method recovery. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three. If the spike is added to the sample before the digestion (e.g., prior to the addition of other reagents), it is referred to as a spiked sample, pre-digestion spike, or Matrix Spike. If the spike is added to the sample after the completion of the digestion procedures, it is referred to as a post-digestion spike, or analytical spike.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for spiked sample analysis.
2. At least one spiked sample (pre-digestion) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each Sample Delivery Group (SDG).
3. When the matrix spike recovery falls outside of the control limits and the sample result is < four times (4x) the spike added, a post-digestion spike shall be performed for those analytes that do not meet the specified criteria. An aliquot of the remaining unspiked sample shall be spiked at 2x the indigenous level or 2x the Contract Required Quantitation Limit (CRQL), whichever is greater.
4. The spike Percent Recovery (%R) shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.
5. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for the purpose of determining %R.

NOTE: The final spike concentrations required for the various target analytes are presented in the methods described in the Statement of Work (SOW).

D. Evaluation:

1. Verify using the Cover Page, Form VA-IN, Form XII-IN, and raw data that the appropriate number of required spiked samples were prepared and analyzed for the SDG.
2. Verify that a field blank or PE sample was not used for the spiked sample analysis.
3. Evaluate Form VA-IN and the raw data to verify that all pre-digestion spiked sample results for each required analyte fall within the established control limits. If not, verify that a post-digestion spike was prepared and analyzed.
4. Recalculate using the raw data, one or more of the %R using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms V (A & B)-IN:

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$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

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

NOTE: When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purposes of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms VA-IN and VB-IN.

E. Action:

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the Matrix Spike, and thus that only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for CLP Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-digestion spike was not performed, note this for CLP PO action.
4. If the Matrix Spike %R is < 30%, verify that a post-digestion spike was analyzed if required. If the post-digestion spike %R is < 75% or is not performed, qualify sample results that are ≥ MDL as estimated low (J-) and non-detects as unusable (R). If the post-digestion spike %R is ≥ 75%, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
5. If the Matrix Spike %R is 30-74% and the sample results are ≥ MDL, verify that a post-digestion spike was analyzed, if required. If the %R for the post-digestion spike is also < 75% or is not performed, qualify the affected data as estimated low (J-). If the %R for the post-digestion spike is ≥ 75%, qualify the affected data as estimated (J).
6. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).
7. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.

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8. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, verify that a post-digestion spike was analyzed, if required. If the %R for the post-digestion spike is also > 125% or is not performed, qualify the affected data as estimated high (J+). If the %R for the post-digestion spike is ≤ 125%, qualify the affected data as estimated (J).
9. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 18. Spike Sample Actions for ICP-MS Analysis

Spike Sample Results	Action for Samples
Matrix Spike %R < 30% Post-digestion spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) affected non-detects as estimated (UJ)
Matrix Spike %R > 125% Post-digestion spike %R > 125%	Qualify affected results that are ≥ MDL as estimated high (J+)
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	Qualify affected results that are ≥ MDL as estimated (J)
Matrix Spike %R < 30% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R 30-74% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R > 125% No post-digestion spike performed	Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified

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ICP Serial Dilution

A. Review Items:

Form I-IN, Form VIII-IN, instrument printouts, and raw data.

B. Objective:

The serial dilution of samples quantitated by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) determines whether or not significant physical or chemical interferences exist due to sample matrix.

C. Criteria:

1. An ICP Serial Dilution analysis shall be performed on a sample from each group of samples with a similar matrix type (e.g., water or soil) or for each Sample Delivery Group (SDG), whichever is more frequent.
2. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for the ICP serial dilution analysis.
3. If the analyte concentration is sufficiently high [concentration in the original sample is > 50 times (50x) the Method Detection Limit (MDL)], the Percent Difference (%D) between the original determination and the serial dilution analysis (a five-fold dilution) after correction shall be less than 10.

NOTE: The above criteria are **method requirements** for serial dilution samples, regardless of the sample matrix type. However, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., %D < 15) to be assessed against serial dilution soil samples.

D. Evaluation:

1. Verify that a field blank or PE sample was not used for the serial dilution analysis.
2. Check the raw data and recalculate the %D using the following equation. Verify that the serial dilution analysis results, and the calculated %D results agree with the values reported by the laboratory on Form VIII-IN:

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

Where,

I = Initial Sample Result

S = Serial Dilution Result

3. Check the raw data for any evidence of positive or negative interference (results from the diluted sample which are significantly different than the original sample), possibly due to high levels of dissolved solids in the sample, ionization effects, etc.

E. Action:

NOTE: For a serial dilution that does not meet the technical criteria, apply the action to all samples of the same matrix if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar

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to the serial dilution sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for serial dilution, and thus only the field sample used to prepare the serial dilution sample should be qualified.

1. If the required %D criteria are not met, qualify all affected results that are ≥ MDL as estimated (J) and all affected non-detects as estimated (UJ).
2. If evidence of positive or negative interference is found, use professional judgment to qualify the associated sample data. Note the potential effects on the reported data in the Data Review Narrative.
3. It should be noted for CLP Project Officer (CLP PO) action and in the Data Review Narrative if a field blank or PE sample was used for the serial dilution analysis.

Table 19. Serial Dilution Actions for ICP-MS Analysis

Serial Dilution Result	Action for Samples
Sample concentration > 50x MDL and %D > 10*	Qualify affected results that are ≥ MDL as estimated (J) Qualify affected non-detects as estimated (UJ)
Interferences present	Use professional judgment

* The above criteria are **method requirements** for serial dilution samples, regardless of the sample matrix type. However, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., %D < 15) to be assessed against serial dilution soil samples.

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ICP-MS Internal Standards

A. Review Items:

Form XIII-IN, Form XV-IN, Form XVII-IN, instrument printouts, and raw data.

B. Objective:

The analysis of Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) internal standards determines the existence and magnitude of instrument drift and physical interferences. The criteria for evaluation of internal standard results apply to all analytical and Quality Control (QC) samples analyzed during the run, beginning with the calibration.

C. Criteria:

1. All samples analyzed during a run, with the exception of the ICP-MS tune, shall contain internal standards. A minimum of five internal standards from the following list shall be added to each sample: Li (the Li^6 isotope); Sc; Y; Rh; Tb; Ho; Lu; and Bi. If the laboratory uses lithium as an internal standard, the laboratory shall use an Li^6-enriched standard. The laboratory shall monitor the same internal standards throughout the entire analytical run and shall assign each analyte to at least one internal standard.
2. The intensity of the internal standard response in a sample is monitored and compared to the intensity of the response for that internal standard in the calibration blank. The Percent Relative Intensity (%RI) in the sample shall fall within 60-125% of the response in the calibration blank.
3. If the %RI of the response in the sample falls outside of these limits, the laboratory shall reanalyze the original sample at a two-fold dilution with internal standard added.

D. Evaluation:

1. Verify using Forms XV-IN, XVII-IN, and the raw data that a minimum of five internal standards from the specified list were used for the analysis, that the same internal standards were monitored for the entire run, and that each analyte was associated to at least one internal standard.
2. Verify using Form XV-IN and the raw data that these internal standards were added to each sample in the run, including calibrations, samples, and QC samples (except tune).
3. Verify using Form XV-IN that the %RI between an internal standard in a sample and the internal standard in the calibration blank was reported for each sample.
4. Verify using Form XIII-IN, Form XV-IN, and the raw data that if the %RI for a sample was outside the limits (60-125%), the sample was reanalyzed of a 2x dilution with internal standard added.

E. Action:

NOTE: Apply the action to the affected analytes for each sample that does not meet the internal standard criteria.

1. If no internal standards were analyzed with the run, the sample data should be qualified as unusable (R). Record this in the Data Review Narrative and note for CLP Project Officer (CLP PO) action.
2. If less than five of the required internal standards were analyzed with the run, or a target analyte(s) is (are) not associated to an internal standard, the sample data, or analyte data not associated to an internal standard should be qualified as unusable (R). Record this in the Data Review Narrative and note for CLP PO action.
3. If the %RIs for all internal standards in a sample is within the 60-125% limit, the sample data should not be qualified.
4. If the %RI for an internal standard in a sample is not within the 60-125% limit, qualify the data for those analytes associated with the internal standard(s) outside the limit as follows:

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a. If the sample was reanalyzed at a two-fold dilution with internal standard %RI within the limits, report the result of the diluted analysis without qualification. If the %RI of the diluted analysis was not within the 60-125% limit, report the results of the original undiluted analyses and qualify the data for all analytes that are ≥ Method Detection Limit (MDL) in the sample associated with the internal standard as estimated (J), and non-detected analytes associated with the internal standard as estimated (UJ).

b. If the sample was not reanalyzed at a two-fold dilution, the reviewer should use professional judgment to determine the reliability of the data. The reviewer may determine that the results are estimated (J) or unusable (R).

Table 20. Internal Standard Actions for ICP-MS Analysis

Internal Standard Results	Action for Samples
No internal standards	Qualify all results as unusable (R)
< 5 of the required internal standards	Qualify all analyte results not as unusable (R)
Target analyte not associated with internal standard	Qualify all analyte results not associated with internal standard as unusable (R)
%RI < 60% or > 125%, and original sample reanalyzed at 2-fold dilution	If %RI of diluted sample analysis 60-125%, do not qualify the data If the %RI of the diluted sample analysis is outside the 60-125% limit, qualify results that are ≥ MDL as estimated (J) and qualify non-detects as estimated (UJ)
Original sample not reanalyzed at 2-fold dilution	Use professional judgment Qualify sample results as estimated (J) or unusable (R)

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Regional Quality Assurance (QA) and Quality Control (QC)

A. Review Items:

Form I-IN, instrument printouts, and raw data.

B. Objective:

Regional QA/QC samples refer to any QA and/or QC samples initiated by the Region, including field duplicate samples, Performance Evaluation (PE) samples, blind spikes, and blind blanks. The use of these QA/QC samples is highly recommended (e.g., the use of field duplicate samples can provide information on sampling precision and sample homogeneity).

C. Criteria:

Criteria are determined by each Region.

D. Evaluation:

Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Compare results for PE samples to the acceptance criteria for the specific PE samples if possible.

Calculate the Relative Percent Difference (RPD) between field duplicates and provide this information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Contract Laboratory Program Project Officer (CLP PO) action.

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Overall Assessment

A. Review Items:

Entire sample data package, data review results, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data (including any confirmation data).

B. Objective:

The objective is to ensure that the reported sample quantitation results are accurate. It is appropriate for the data reviewer to make professional judgments and express concerns, as well as to comment on the validity of the overall data for a Case. This is particularly appropriate when there are several Quality Control (QC) criteria that are outside of the specification parameters. The additive nature of QC factors that fall outside of specification parameters is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user of data quality and data limitations to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the acceptance or performance criteria were provided. The Inorganic Review Summary (see Appendix B) and supplementary documentation must be included with the review.

C. Criteria:

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method.

D. Evaluation:

Examine the raw data to verify that the correct calculation of the sample results was reported by the laboratory. Digestion logs, instrument printouts, etc., should be compared to the reported sample results recorded on the appropriate Inorganic Summary Forms (Form I-IN through Form XV-IN).

1. Evaluate any technical problems not previously addressed.
2. Examine the raw data for any anomalies (e.g., baseline shifts, negative response, mass dependent drift, omissions, illegibility, etc.).
3. Verify appropriate methods and volumes were used in preparing the samples for analysis. If reduced volumes were used, verify that the laboratory had received Contract Laboratory Program Project Officer (CLP PO) approval for the use of the reduced volume.
4. Verify that there are no transcription or reduction errors [e.g., dilutions, Percent Solids (%S), sample weights, etc.] on one or more samples.
5. Verify that results fall within the calibrated range(s) of the Inductively Coupled Plasma (ICP) instrument(s) (Form XI).
6. If appropriate information is available, the reviewer may assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the Standard Operating Procedure(s) (SOPs), and communication with the user concerning the intended use and desired quality of these data.

E. Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.

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2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the SDG Narrative for CLP PO action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.
3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

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Calculations for ICP-MS

Aqueous/Water Sample Concentration by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS):

The concentrations determined in the digestate are to be reported in units of µg/L:

$$\text{Concentration (ug/L)} = C \times \frac{V_f}{V} \times DF$$

Where,

C	=	Instrument value in µg/L (The average of all replicate integrations).
V_f	=	Final digestion volume (mL)
V	=	Initial Aliquot Amount (mL)
DF	=	Dilution Factor

Soil/Sediment Sample Concentration by ICP-MS:

The concentrations determined in the digestate are to be reported on the basis of the dry weight of the sample, in units of mg/kg:

$$\text{Concentration (dry wt.)(mg/kg)} = C \text{ x } \frac{V_f}{W \times S} \times DF / 1000$$

Where,

C	=	Instrument value in µg/L (The average of all replicate integrations).
V_f	=	Final digestion volume (mL)
W	=	Initial aliquot amount (g)
S	=	% Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
DF	=	Dilution Factor

Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, substitute the value of the MDL (µg/L) or CRQL (µg/L) into the "C" term in the equation above.

Calculate the adjusted MDL or adjusted CRQL for soil/sediment samples as follows:

$$\text{Adjusted Concentration (mg/kg)} = C \times \frac{W_M}{W \text{ x } S} \times \frac{V_f}{V_M} \times DF$$

Where,

C	=	MDL or CRQL (mg/kg)
W_M	=	Minimum method required aliquot amount (g) (1.00g or 0.50g)
W	=	Initial aliquot amount (g)
V_M	=	Method required final sample digestion volume (mL) (100 mL)
V_f	=	Final digestion volume (mL)
S	=	% Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
DF	=	Dilution Factor

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MERCURY DATA REVIEW

The inorganic data requirements for mercury data review to be reviewed during validation are listed below:

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II. Calibration **66**

III. Blanks **69**

IV. Duplicate Sample Analysis **72**

V. Spike Sample Analysis **74**

VI. Regional Quality Assurance (QA) and Quality Control (QC) **76**

VII. Overall Assessment **77**

Calculations for Mercury **79**

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An Example Analytical Sequence for Mercury

S0
S0.2
S1.0
S2.0
S5.0
S10.0
ICV
ICB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

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I. Preservation and Holding Times

A. Review Items:

Form IA-IN, Form IB-IN, Form XII-IN, Form XIII-IN, Traffic Report/Chain of Custody (TR/COC) documentation, Form DC-1, raw data, and the Sample Delivery Group (SDG) Narrative checking for: pH; cooler temperature; holding time; and other sample conditions.

B. Objective:

The objective is to ascertain the validity of the analytical results based on the sample condition, and the holding time of the sample from the date of collection to the date of analysis.

C. Criteria:

1. The technical holding time criteria for aqueous/water mercury samples is 28 days; preserved (with nitric acid) to pH ≤ 2. The addition of nitric acid to adjust the pH is only required for aqueous/water samples.
2. The technical holding time criteria for soil/sediment mercury samples is 28 days, based on the technical holding time criteria for aqueous/water samples.
3. Mercury samples shall be maintained at 4□C (±2□C) until preparation and analysis to allow for re-preparation.
4. Samples and standards shall be analyzed with 48 hours of preparation.

D. Evaluation:

Technical holding times are established by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form XIII-IN, and the raw data. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. Verify that the analysis dates on the Form XIIIs and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication that there were problems with the samples, the integrity of the samples may be compromised and professional judgment should be used to evaluate the effect of the problem on the sample results.

E. Action:

NOTE: Apply the action to each sample for which the preservation or holding time criteria was not met.

1. If the pH of aqueous/water metals samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH to ≤2 at the time of sample receipt. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of the metal(s) of interest. Qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-), and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-), and qualify non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment samples, it is left to the discretion of the data reviewer whether to apply aqueous/water holding time criteria to soil/sediment samples. If they are applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.
5. When holding times are grossly exceeded, note it for Contract Laboratory Program Project Officer (CLP PO) action.

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6. When shipping or storage temperatures grossly exceed the requirements, the loss of volatile mercury compounds or metallic mercury is possible. The expected bias would be low. Use professional judgment to qualify the samples and note for CLP PO action.

Table 21. Technical Holding Time Actions for Mercury Analysis

Preservation & Holding Time Results	Action for Samples
Aqueous/water metals samples received with pH > 2 and pH not adjusted	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Samples not maintained at 4□C (±2□C)	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding time exceeded: Aqueous/water Samples > 28 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical Holding Time exceeded: Soil/sediment Metals > 28 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

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Calibration

A. Review Items:

Form II-IN (Part A), Form XI-IN, Form XIII-IN, Form XVI-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective:

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for mercury. Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

C. Criteria:

1. Initial Calibration

The instruments shall be successfully calibrated daily (or once every 24 hours), and each time the instrument is set up. The calibration date and time shall be included in the raw data. The calibration curve shall be prepared by the same method used to prepare the samples for analysis. The curve shall be prepared with the samples that will be analyzed using this calibration curve.

a. Cold Vapor Mercury Analysis

1) A blank and at least five calibration standards shall be employed to establish the analytical curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curves for mercury shall possess a correlation coefficient of ≥ 0.995 to ensure the linearity over the calibrated range. The percent differences calculated for all of the non-zero standards must fall within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL. All sample results shall be reported from an analysis within the calibrated range.

2. Initial and Continuing Calibration Verification (ICV and CCV)

The acceptance criteria for the ICVs and CCVs are presented in Table 22. These standards shall be prepared by the same method used to prepare the samples for analysis.

Table 22. Acceptance Criteria for ICVs and CCVs

Analytical Method	Inorganic Analyte	ICV/CCV Low Limit (% of True Value)	ICV/CCV High Limit (% of True Value)
Cold Vapor AA	Mercury	85	115

a. Initial Calibration Verification (ICV)

1) Immediately after each Atomic Absorption (AA) system has been calibrated, the accuracy of the initial calibration must be verified and documented for mercury by the analysis of an ICV solution(s). If the ICV %R falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.

2) If the ICV is not available from USEPA, or where a certified solution of the analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration level other than that used for instrument calibration, but within the calibrated range.

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b. Continuing Calibration Verification (CCV)

1) To ensure accuracy during the course of each analytical run, the CCV shall be analyzed and reported.
2) The CCV standard shall be analyzed at a frequency of every hour during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample.
3) The analyte concentration in the CCV standard shall be different than the concentration used for the ICV, and shall be at the mid level of the calibration curve.
4) The same CCV standard solution shall be used throughout the analysis runs for a Sample Delivery Group (SDG).
5) The CCV shall be analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

D. Evaluation:

1. Verify that the instrument was calibrated daily (once every 24 hours) and each time the instrument was set up, utilizing a blank and at least five calibration standards. Confirm that at least one of the calibration standards was analyzed at or below the CRQL. Confirm that calibration standards and samples were prepared at the same time.
2. Confirm that the measurements were within the documented working range of the calibration standards.
3. Verify that the ICV and CCV standards were analyzed for mercury at the proper frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.
4. Recalculate one or more of the ICV or CCV %R using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form IIA-IN.

$$\%R = \frac{\text{Found (value)}}{\text{True (value)}} \times 100$$

Where,

Found (value) = Concentration (in µg/L) of mercury <u>measured</u> in the analysis of the ICV or CCV solution

True (value) = Concentration (in µg/L) of mercury in the ICV or CCV source

E. Action:

NOTES: For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

For CCVs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical run.

1. If the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank, and at least one standard at or below the CRQL), or if the instrument was not calibrated with standards prepared at the same time as the samples, use professional judgment to qualify results that are ≥ Method Detection Limit (MDL) as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).

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2. If the correlation coefficient is < 0.995, percent differences are outside the ±30% limit, or the y-intercept is >CRQL, qualify sample results that are ≥ MDL as estimated (J), and non-detects as estimated (UJ). Depending on the degree of the deviation from linearity, further qualification of the data may be required depending on the professional judgment of the reviewer [e.g., unusable data (R)].
3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
   a. If the ICV or CCV %R is < 70%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as estimated low (J-) or unusable (R).
   b. If the ICV or CCV %R falls within the range of 70-84%, qualify sample results that are ≥ MDL as estimated low (J-) and qualify non-detects as estimated (UJ).
   c. If the ICV or CCV %R falls within the range of 116-130%, qualify sample results that are ≥ MDL as estimated high (J+).
   d. If the ICV or CCV %R falls within the range of 116-130%, non-detects should not be qualified.
   e. If the ICV or CCV %R is > 130%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+) or unusable (R). Non-detects should not be qualified.
   f. If the %R is > 165%, qualify all results that are ≥ MDL as unusable (R).
4. If the laboratory failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
5. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
6. If calibration criteria are grossly exceeded, note this for CLP Project Officer (CLP PO) action.

NOTE: For truly critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

Table 23. Calibration Actions for Mercury Analysis

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as unusable (R)
Calibration incomplete	Use professional judgment Qualify results that are ≥ MDL as estimated (J) or unusable (R) Qualify non-detects as estimated (UJ) or unusable (R)
Correlation coefficient <0.995, %D outside ±30%, y-intercept ≥CRQL	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)
ICV/CCV %R < 70%	Qualify results that are ≥ MDL as estimated low (J-) or unusable (R) Qualify all non-detects as unusable (R)
ICV/CCV %R 70-84%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICV/CCV %R 116-130%	Qualify results that are ≥ MDL as estimated (J)
ICV/CCV %R > 130%	Qualify results that are ≥ MDL as estimated high (J+) or unusable (R)
ICV/CCV %R > 165%	Qualify results that are ≥ MDL as unusable (R)

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Blanks

A. Review Items:

Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective:

The objective of blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, calibration blanks, field blanks, etc.). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blank(s).
2. The Initial Calibration Blank (ICB) shall be analyzed after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) during the initial calibration of the instrument (see Section II.C.1). The ICB shall be prepared by the same method used to prepare the samples for analysis.
3. A Continuing Calibration Blank (CCB) shall be analyzed immediately after every Continuing Calibration Verification (CCV). The CCB shall be prepared by the same method used to prepare the samples for analysis. The CCB shall be analyzed at a frequency of every hour during the run. The CCB shall be analyzed at the beginning of the run, and again after the last CCV that was analyzed after the last analytical sample of the run. The CCB result (absolute value) shall not exceed the Contract Required Quantitation Limit (CRQL) for mercury.
4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.
5. If the mercury concentration in the Preparation Blank is > CRQL, the lowest concentration of mercury in the associated samples must be ≥ 10 times (10x) the Preparation Blank concentration. Otherwise, all samples associated with that Preparation Blank with a mercury concentration < 10x the Preparation Blank concentration, and > CRQL, should be redigested and reanalyzed (except for an identified field blank). The laboratory is not to correct the sample concentration for the blank value.
6. If the concentration of the Preparation Blank for mercury is < (-CRQL), all samples reported < 10x the CRQL (associated with that analyte in that blank), should be redigested and reanalyzed.

D. Evaluation:

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the proper frequency and location during the run, and Preparation Blanks are prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. Review the results reported on the Blank Summary (Form III-IN), as well as the raw data for all blanks, and verify that the results are accurately reported.
3. Evaluate all of the associated blanks for the presence of mercury. Verify that if mercury was present in a Preparation Blank or if a concentration was < (-CRQL), the affected samples were redigested and reanalyzed. Verify that if mercury was present in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

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E. Action:

NOTES: For ICBs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

For CCBs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.

For Preparation Blanks that do not meet the technical criteria, apply the action to all samples prepared in the same preparation batch.

1. If the appropriate blanks are not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
   a. Any blank (including Preparation Blanks) reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank (including Preparation Blanks) reported with a negative result, whose value is < (-CRQL), qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
   b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. Specific "method" actions include:
   a. If the absolute value of an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the affected samples are not reanalyzed, report non-detects and results that are ≥ MDL but ≤ CRQL as CRQL-U. For results that are > CRQL but < Blank Result, use professional judgment to qualify the data as unusable (R), or to report the results at the level of the blank with a "U" qualifier. Use professional judgment to qualify results that are > Blank Result. Note this situation for CLP PO action and record it in the Data Review Narrative.
   b. If the absolute value of the concentration of the Preparation Blank is ≤ CRQL, report non-detect and results that are ≥ MDL but ≤ CRQL as CRQL-U. Use professional judgment to qualify results that are > CRQL.
   c. If the mercury concentration in the Preparation Blank is > CRQL, the lowest concentration of mercury in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redigested and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redigestion (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redigest and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.

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Table 24. Blank Actions for Mercury Analysis

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	Absolute value is ≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL	Use professional judgment
ICB/CCB	Absolute value is > CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < Blank Result	Report at level of Blank Result with a "U" or qualify data as unusable (R)
		> Blank Result	Use professional judgment
ICB/CCB	≤ (-MDL), but ≥ (-CRQL)	≥ MDL, or non-detect	Use professional judgment
ICB/CCB	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)
Preparation Blank	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a "U"
		> CRQL but < 10x the Blank Result	Qualify results as unusable (R) or estimated high (J+)
		≥ 10x the Blank Result	No action
Preparation Blank	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL with a "U"
		> CRQL	Use professional judgment
Preparation Blank	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)

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Duplicate Sample Analysis

A. Review Items:

Cover Page, Form VI-IN, Form XII-IN, instrument printouts, and raw data.

B. Objective:

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analyses are also performed to generate data that determines the long-term precision of the analytical method on various matrices. Non-homogenous samples can impact the apparent method precision. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.
2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each Sample Delivery Group (SDG). Duplicates cannot be averaged for reporting on Form I-IN. Additional duplicate sample analyses may be required by USEPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.
3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL).
4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form VI-IN. If both samples are non-detects, the RPD is not calculated for Form VI-IN.

NOTE: The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

D. Evaluation:

1. Verify from the Cover Page, Form XII-IN, and the raw data that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.
2. Evaluate Form VI-IN and the raw data to verify that all mercury duplicate results for each method fall within the established control limits.
3. Verify that a field blank or PE sample was not used for duplicate analysis.
4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form VI-IN:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = Relative Percent Difference

S = Sample Result (original)

D = Duplicate Result

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E. Action:

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the duplicate, and thus that only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If the results from a duplicate analysis for mercury fall outside the appropriate control limits, qualify sample results that are ≥ Method Detection Limit (MDL) as estimated (J) and non-detects as estimated (UJ).
3. If a field blank or PE sample was used for the duplicate sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
4. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 25. Duplicate Sample Actions for Mercury Analysis

Duplicate Sample Results	Action for Samples
Both original sample and duplicate sample > 5x the CRQL and RPD> 20%*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)

* The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

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Spike Sample Analysis

A. Review Items:

Cover Page, Form V-IN (Parts A & B), Form XII-IN, instrument printouts, and raw data.

B. Objective:

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. Non-homogenous samples can impact the apparent method recovery. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three. If the spike is added to the sample before the digestion (e.g., prior to the addition of other reagents), it is referred to as a spiked sample, pre-digestion spike, or Matrix Spike.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for spiked sample analysis.
2. At least one spiked sample (pre-digestion) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each Sample Delivery Group (SDG).
3. The spike Percent Recovery (%R) shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥ four times (4x) the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.
4. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for the purpose of determining %R.

NOTE: The final spike concentrations required for mercury are presented in the method described in the Statement of Work (SOW).

D. Evaluation:

1. Verify using the Cover Page, Form VA-IN, Form XII-IN, and raw data that the appropriate number of required spiked samples were prepared and analyzed for the SDG.
2. Verify that a field blank or PE sample was not used for the spiked sample analysis.
3. Evaluate Form VA-IN and the raw data to verify that all Matrix Spike sample results for mercury fall within the established control limits.
4. Recalculate using the raw data, one or more of the %R using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms V (A & B)-IN:

$$\%\text{Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

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

NOTE: When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purposes of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms VA-IN and VB-IN.

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E. Action:

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the Matrix Spike, and thus that only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for CLP Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike %R is < 30%, qualify affected results that are ≥ MDL as estimated low (J-). Qualify affected non-detects as unusable (R).
4. If the Matrix Spike %R falls within the range of 30-74% and the sample results are ≥ MDL, qualify the affected data as estimated low (J-).
5. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).
6. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.
7. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, qualify the affected data as estimated high (J+).
8. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Table 26. Spike Sample Actions for Mercury Analysis

Spike Sample Results	Action for Samples
Matrix Spike %R < 30%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R 30-74%	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R > 125%	Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified

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Regional Quality Assurance (QA) and Quality Control (QC)

A. Review Items:

Form I-IN, instrument printouts, and raw data.

B. Objective:

Regional QA/QC samples refer to any QA and/or QC sample initiated by the Region, including field duplicates, Performance Evaluation (PE) samples, blind spikes, and blind blanks. The use of these QA/QC samples is highly recommended (e.g., the use of field duplicates can provide information on sampling precision and homogeneity).

C. Criteria:

Criteria are determined by the Region.

D. Evaluation:

Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Compare results for PE samples with the acceptance criteria for the specific PE samples if possible.

Calculate the Relative Percent Difference (RPD) between field duplicates and provide his information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Contract Laboratory Program Project Officer (CLP PO) action.

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Overall Assessment

A. Review Items:

Entire data package, data review results, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data (including any confirmation data).

B. Objective:

The objective is to ensure that the reported sample quantitation results are accurate. It is appropriate for the data reviewer to make professional judgments and express concerns, as well as to comment on the validity of the overall data for a Case. This is particularly appropriate when there are several Quality Control (QC) criteria that are outside of the specification parameters. The additive nature of QC factors that fall outside of specification parameters is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user concerning data quality and data limitations to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the acceptance or performance criteria are provided. The Inorganic Review Summary (see Appendix B) and supplementary documentation must be included with the review.

C. Criteria:

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method.

D. Evaluation:

Examine the raw data to verify that the correct calculation of the sample results was reported by the laboratory. Digestion logs, instrument printouts, etc., should be compared to the reported sample results recorded on the appropriate Inorganic Summary Forms (Form I-IN through Form XV-IN).

1. Evaluate any technical problems not previously addressed.
2. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).
3. Verify that the appropriate methods and amounts were used to prepare samples and standards for analysis. If reduced volumes are used, verify that the laboratory had received Contract Laboratory Program Project Officer (CLP PO) approval for the use of the reduced volume.
4. Verify that there are no transcription or reduction errors [e.g., dilutions, Percent Solids (%S), sample weights, etc.] on one or more samples.
5. Verify that results fall within the calibrated range for mercury.
6. If appropriate information is available, the reviewer may assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the Standard Operating Procedure(s) (SOPs), and communication with the user concerning the intended use and desired quality of these data.

E. Action:

1. Use professional judgment to determine if there is any need to qualify data which are not qualified based on the QC criteria previously discussed.
2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the SDG Narrative for CLP PO action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.

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3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

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Calculations for Mercury

Aqueous/Water Samples:

$$\text{Hg Concentration (ug/L)} = C \times DF$$

Where,

C = Instrument value in µg/L from the calibration curve

DF = Dilution Factor of the original sample

Soil/Sediment Samples:

$$\text{Hg Concentration (mg/kg)} = C \times \frac{1}{W \times S} \times DF \times 0.1$$

Where,

C = Instrument value in µg/L from the calibration curve

W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, multiply the value of the MDL (µg/L) or CRQL (µg/L) by the Dilution Factor (DF). Calculate the adjusted MDL or adjusted CRQL for soil/sediment samples as follows:

$$\text{Adjusted Concentration (mg/kg)} = C \times \frac{W_m}{W \times S} \times DF$$

Where,

C = MDL or CRQL (mg/kg)

W_m = Method required minimum sample weight (g) (0.50 g)

W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

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CYANIDE DATA REVIEW

The inorganic data requirements for cyanide data review to be reviewed during validation are listed below:

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An Example Analytical Sequence for Cyanide

S0
S10
S50
S100
S200
S400
ICV
ICB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

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I. Preservation and Holding Times

A. Review Items:

Form IA-IN, Form IB-IN, Form XII-IN, Form XIII-IN, Traffic Report/Chain of Custody (TR/COC) documentation, Form DC-1, raw data, and the Sample Delivery Group (SDG) Narrative checking for: pH; cooler temperature; holding time; and other sample conditions.

B. Objective:

The objective is to ascertain the validity of the analytical results based on the sample condition, and the holding time of the sample from the date of collection to the date of analysis.

C. Criteria:

1. The technical holding time criteria for aqueous/water cyanide samples is 14 days; oxidizing agents removed, then preserved (with sodium hydroxide) to pH ≥ 12. The addition of sodium hydroxide to adjust the pH is only required for aqueous/water samples.
2. The technical holding time criteria for soil/sediment cyanide samples is 14 days, based on the technical holding time criteria for aqueous/water samples.
3. Samples shall be maintained at 4□C (±2□C) until preparation and analysis.

D. Evaluation:

Technical holding times are established by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form XIII-IN, and the raw data. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. Verify that the analysis dates on the Form XIIIs and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication that there are problems with the samples, the integrity of the samples may be compromised and professional judgment should be used to evaluate the effect of the problem on the sample results. For aqueous/water cyanide samples, the reviewer should look for evidence that the samples were tested for the presence of sulfides or oxidizing agents, and whether the appropriate preservation steps were taken.

E. Action:

NOTE: Apply the action to each sample for which the preservation or holding time criteria were not met.

1. If oxidizing agents are detected in aqueous/water cyanide samples at the time of sample preparation, qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-) and non-detects as unusable (R). If sulfides are detected in aqueous/water cyanide samples at the time of sample preparation and there is no evidence that the laboratory removed the sulfides (using precipitation and filtration), qualify results that are ≥ MDL as estimated (J) and non-detects as unusable (R). If the pH of aqueous/water cyanide samples is < 12 at the time of sample receipt, use professional judgment to qualify the samples based on the pH of the sample. Qualify results that are ≥ MDL as estimated low (J-) and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples are properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-) and non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment samples, it is left to the discretion of the data reviewer whether to apply aqueous/water holding time criteria to soil/sediment samples. If they are applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.

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5. When holding times are grossly exceeded, note it for Contract Laboratory Program Project Officer (CLP PO) action.

Table 27. Technical Holding Time Actions for Cyanide Analysis

Preservation & Holding Time Results	Action for Samples
Aqueous/water cyanide samples received with oxidizing agents present.	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Aqueous/water cyanide samples received with sulfides present, and sulfides are not removed	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as unusable (R)
Aqueous/water cyanide samples received with pH < 12	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)
Technical holding time exceeded: Cyanide > 14 days	Use professional judgment Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as unusable (R)

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Calibration

A. Review Items:

Form II-IN (Part A), Form XI-IN, Form XIII-IN, Form XVI-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

B. Objective:

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for cyanide. Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

C. Criteria:

1. Initial Calibration

   The instruments shall be successfully calibrated daily (or once every 24 hours), and each time the instrument is set up. The calibration date and time shall be included in the raw data.

   a. A blank and at least five calibration standards shall be employed to establish the analytical curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curve for cyanide shall possess a correlation coefficient of ≥ 0.995 to ensure the linearity over the calibrated range. The percent differences calculated for all of the non-zero standards must be within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

   b. All sample results shall be reported from an analysis within the calibrated range.

   c. All standards shall be distilled.

2. Initial and Continuing Calibration Verification (ICV and CCV)

   The acceptance criteria for the ICVs and CCVs are presented in Table 28:

Table 28. Acceptance Criteria for ICVs and CCVs

Analytical Method	Inorganic Analyte	ICV/CCV Low Limit (% of True Value)	ICV/CCV High Limit (% of True Value)
Colorimetric	Cyanide	85	115

   a. Initial Calibration Verification (ICV)

      1) Immediately after each cyanide colorimetric system has been calibrated, the accuracy of the initial calibration must be verified and documented for cyanide by the analysis of an ICV solution(s). If the ICV %R falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.

      2) If the ICV is not available from USEPA, or where a certified solution of the analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration level other than that used for instrument calibration, but within the calibrated range.

      3) For cyanide analysis, the ICV standard solution shall be distilled.

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b. Continuing Calibration Verification (CCV)

1) To ensure accuracy during the course of each analytical run, the CCV shall be analyzed and reported.
2) The CCV standard shall be analyzed at a frequency of every hour during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample.
3) The analyte concentration in the CCV standard shall be different from the concentration used for the ICV, and shall be at the mid level of the calibration curve.
4) The same CCV standard solution shall be used throughout the analysis runs for a Sample Delivery Group (SDG). The CCV shall be distilled.
5) The CCV shall be processed and analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

D. Evaluation:

1. Verify that the instrument was calibrated daily (once every 24 hours) and each time the instrument was set up, utilizing a blank and at least five calibration standards. Confirm that at least one of the calibration standards was analyzed at or below the CRQL.
2. Check the distillation log and verify that the calibration standards, the ICV and the CCVs were distilled and analyzed.
3. Verify that the ICV and CCV standards were analyzed for cyanide at the proper frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.
4. Recalculate one or more of the ICV or CCV %R using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form IIA -IN.

$$\%R = \frac{\text{Found (value)}}{\text{True (value)}} \times 100$$

Where,

Found (value) = Concentration (in µg/L) of cyanide measured in the analysis of the ICV or CCV solution

True (value) = Concentration (in µg/L) of cyanide in the ICV or CCV source

E. Action:

NOTES: For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

For CCVs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical run.

1. If the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank and a standard at or below the CRQL), use professional judgment to qualify results that are ≥ Method Detection Limit (MDL) as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).
2. If the correlation coefficient is < 0.995, percent differences outside the ±30% limit, or y-intercept ≥CRQL, qualify sample results that are ≥ MDL as estimated (J), and non-detects as estimated (UJ).

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3. If the standards, the ICV, or the CCVs are not distilled for cyanide, qualify sample results that are ≥ MDL as estimated (J).
4. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
   a. If the ICV or CCV %R is < 70%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as estimated low (J-) or unusable (R).
   b. If the ICV or CCV %R falls within the range of 70-84%, qualify sample results that are ≥ MDL as estimated low (J-), qualify non-detects as estimated (UJ).
   c. If the ICV or CCV %R falls within the range of 116-130%, qualify sample results that are ≥ MDL as estimated high (J+).
   d. If the ICV or CCV %R is within the range of 116-130%, non-detects should not be qualified.
   e. If the ICV or CCV %R is > 130%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+) or unusable (R). Non-detects should not be qualified.
   f. If the %R is > 165%, qualify all results that are ≥ MDL as unusable (R).
5. If the laboratory failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
6. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
7. If calibration criteria are grossly exceeded, note this for Contract Laboratory Program Project Officer (CLP PO) action.

NOTE: For truly critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

Table 29. Calibration Actions for Cyanide Analysis

Calibration Result	Action for Samples
Calibration not performed	Qualify all results as unusable (R)
Calibration incomplete	Use professional judgment Qualify results that are ≥ MDL as estimated (J) or unusable (R) Qualify non-detects as estimated (UJ) or unusable (R)
Correlation coefficient < 0.995; %D outside ±30%, y-intercept ≥CRQL	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated (UJ)
Standards and QC not distilled	Qualify results that are ≥ MDL as estimated (J)
ICV/CCV %R < 70%	Qualify results that are ≥ MDL as estimated low (J-) or unusable (R) Qualify all non-detects as unusable (R)
ICV/CCV %R 70-84%	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
ICV/CCV %R 116-130%	Qualify results that are ≥ MDL as estimated (J)
ICV/CCV %R > 130%	Qualify results that are ≥ MDL as estimated high (J+) or unusable (R)
ICV/CCV %R > 165%	Qualify results that are ≥ MDL as unusable (R)

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Blanks

A. Review Items:

Form I-IN, Form III-IN, Form XII-IN, Form XIII-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

B. Objective:

The objective of blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, calibration blanks, field blanks, etc.). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blank(s).
2. The Initial Calibration Blank (ICB) shall be analyzed after the analytical standards, but not before analysis of the Initial Calibration Verification (ICV) during the initial calibration of the instrument (see Section II.C.1).
3. A Continuing Calibration Blank (CCB) shall be analyzed immediately after every Continuing Calibration Verification (CCV). The CCB shall be analyzed at a frequency of every hour during the run. The CCB shall be analyzed at the beginning of the run, and again after the last CCV that was analyzed after the last analytical sample of the run. The CCB result (absolute value) shall not exceed the Contract Required Quantitation Limit (CRQL) of cyanide.
4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every Sample Delivery Group (SDG), or with each batch of samples distilled, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.
5. If the cyanide concentration in the Preparation Blank is > CRQL, the lowest concentration of cyanide in the associated samples must be ≥ 10 times (10x) the Preparation Blank concentration. Otherwise, all samples associated with that Preparation Blank with a cyanide concentration < 10x the Preparation Blank concentration, and > CRQL, should be redistilled and reanalyzed cyanide (except for an identified field blank). The laboratory is not to correct the sample concentration for the blank value.
6. If the concentration of the Preparation Blank for cyanide is < (-CRQL), all samples reported < 10x the CRQL (associated with that blank), should be redistilled and reanalyzed.

D. Evaluation:

1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the proper frequency and location during the run, and Preparation Blanks are prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
2. Review the results reported on the Blank Summary (Form III-IN), as well as the raw data for all blanks, and verify that the results were accurately reported.
3. Evaluate all of the associated blanks for the presence of cyanide. Verify that if cyanide was present in a Preparation Blank or if a concentration was < (-CRQL), the affected samples were redistilled and reanalyzed. Verify that if cyanide was present in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

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E. Action:

NOTES: For ICBs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.

For CCBs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.

For Preparation Blanks that do not meet the technical criteria, apply the action to all samples prepared in the same preparation batch.

1. If the appropriate blanks are not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
   a. Any blank (including Preparation Blanks) reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank (including Preparation Blanks) reported with a negative result, whose value is < (-CRQL) qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
   b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. Specific "method" actions include:
   a. If the absolute value of an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the affected samples are not reanalyzed, report non-detects and results that are ≥ MDL but ≤ CRQL as CRQL-U. For results that are > CRQL but < Blank Result, use professional judgment to qualify the data as unusable (R), or report the results at the level of the blank with a "U" qualifier. Use professional judgment to qualify results that are > Blank Result. Note this situation for CLP PO action and record it in the Data Review Narrative.
   b. If the absolute value of the concentration of the Preparation Blank is ≤ CRQL, no correction of the sample results should be performed, report non-detects and results ≥ MDL but ≤ CRQL as CRQL-U. Use professional judgment to qualify results that are > CRQL.
   c. If the cyanide concentration in the Preparation Blank is > CRQL, the lowest concentration of cyanide in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redistilled and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redistillation (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redistill and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.

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Table 30. Blank Actions for Cyanide Analysis

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB	Absolute value is ≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a ”U”
		> CRQL	Use professional judgment
ICB/CCB	Absolute value is > CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a “U”
		> CRQL but < Blank Result	Report at level of Blank Result with a “U” or qualify data as unusable (R)
		> Blank Result	Use professional judgment
ICB/CCB	≤ (-MDL), but ≥ (-CRQL)	≥ MDL, or non-detects	Use professional judgment
ICB/CCB	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)
Preparation Blank	> CRQL	≥ MDL but ≤ CRQL	Report CRQL value with a “U”
		> CRQL but < 10x the Blank Result	Qualify results as unusable (R) or estimated high (J+)
		≥ 10x the Blank Result	No action
Preparation Blank	≥ MDL but ≤ CRQL	Non-detect	No action
		≥ MDL but ≤ CRQL	Report CRQL value with a “U”
		> CRQL	Use professional judgment
Preparation Blank	< (-CRQL)	< 10x the CRQL	Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ)

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Duplicate Sample Analysis

A. Review Items:

Cover Page, Form VI-IN, Form XII-IN, instrument printouts, and raw data.

B. Objective:

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. Duplicate analyses are also performed to generate data that determines the long-term precision of the analytical method on various matrices. Non-homogenous samples can impact the apparent method precision. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.
2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each Sample Delivery Group (SDG). Duplicates cannot be averaged for reporting on Form I-IN. Additional duplicate sample analyses may be required by USEPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.
3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL).
4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form VI-IN. If both samples are non-detects, the RPD is not calculated for Form VI-IN.

NOTE: The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

D. Evaluation:

1. Verify from the Cover Page, Form XII-IN, and the raw data that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.
2. Evaluate Form VI-IN and the raw data to verify that all cyanide duplicate results for each method fall within the established control limits.
3. Verify that a field blank or PE sample was not used for duplicate analysis.
4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form VI-IN:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where,

RPD = Relative Percent Difference

S = Sample result (original)

D = Duplicate result

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E. Action:

NOTE: For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the duplicate, and thus that only the field sample used to prepare the duplicate sample should be qualified.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If the results from a duplicate analysis for cyanide fall outside the appropriate control limits, qualify sample results that are ≥ Method Detection Limit (MDL) as estimated (J) and non-detects as estimated (UJ).
3. If a field blank or PE sample was used for the duplicate sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
4. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 31. Duplicate Sample Actions for Cyanide Analysis

Duplicate Sample Results	Action for Samples
Both original sample and duplicate sample > 5x the CRQL and RPD> 20%*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)

* The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

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Spike Sample Analysis

A. Review Items:

Cover Page, Form V-IN (Parts A & B), Form XII-IN, instrument printouts, and raw data.

B. Objective:

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. Non-homogenous samples can impact the apparent method recovery. However, aqueous/water samples are generally homogenous and most soil/sediment samples are homogenous within a factor of two or three. If the spike is added to the sample prior to any distillation steps (e.g., cyanide), it is referred to as a spiked sample, pre-distillation spike, or Matrix Spike. If the spike is added to the sample after the completion of the distillation procedures, it is referred to as a post-distillation spike, or analytical spike.

C. Criteria:

1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for spiked sample analysis.
2. At least one spiked sample (pre-distillation) shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each Sample Delivery Group (SDG).
3. When the pre-distillation spike recovery falls outside of the control limits and the sample result is < four times (4x) the spike added, a post-distillation spike shall be performed An aliquot of the remaining unspiked sample shall be spiked at 2x the indigenous level or 2x the Contract Required Quantitation Limit (CRQL), whichever is greater.
4. The spike Percent Recovery (%R) shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.
5. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for the purpose of determining %R.

NOTE: The final spike concentrations required for cyanide are presented in the method described in the Statement of Work (SOW).

D. Evaluation:

1. Verify using the Cover Page, Form VA-IN, Form XII-IN, and raw data that the appropriate number of required spiked samples were prepared and analyzed for the SDG.
2. Verify that a field blank or PE sample was not used for the spiked sample analysis.
3. Evaluate Form VA-IN and the raw data to verify that all pre-distillation spiked sample results fall within the established control limits. If not, verify that a post-distillation spike was prepared and analyzed.
4. Recalculate using the raw data, one or more of the %R using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Forms V (A & B)-IN:

$$\%\text{Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where,

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

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NOTE: When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purposes of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Form V (A & B)-IN.

E. Action:

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix, if the reviewer considers the samples sufficiently similar. The reviewer will need to exercise professional judgment in determining sample similarity. The reviewer should make use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSSs), Total Dissolved Solids (TDSs), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions], in determining similarity. The reviewer should also use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. The reviewer may determine that only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified. Or, the reviewer may determine that no samples are sufficiently similar to the sample used for the Matrix Spike, and thus that only the field sample used to prepare the Matrix Spike sample should be qualified.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for CLP Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-distillation spike was not performed, note this for CLP PO action.
4. If the Matrix Spike %R is < 30%, verify that a post-distillation spike was analyzed if required. If the post-distillation spike %R is < 75% or is not performed, qualify sample results that are ≥ MDL as estimated low (J-) and non-detects as unusable (R). If the post-distillation spike %R is ≥ 75%, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
5. If the Matrix Spike %R falls within the range of 30-74% and the sample results are ≥ MDL, verify that a post-distillation spike was analyzed if required. If the %R for the post-distillation spike is also < 75% or not performed, qualify the affected data as estimated low (J-). If the %R for the post-distillation spike is ≥ 75%, qualify the affected data as estimated (J).
6. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).
7. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.
8. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, verify that a post-distillation spike was analyzed if required. If the %R for the post-distillation spike is also > 125% or is not performed, qualify the affected data as estimated high (J+). If the %R for the post-distillation spike is ≤ 125%, qualify the affected data as estimated (J).
9. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

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Table 32. Spike Sample Actions for Cyanide Analysis

Spike Sample Results	Action for Samples
Matrix Spike %R < 30% Post-distillation spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) Qualify affected non-detects as unusable (R)
Matrix Spike %R < 30% Post-distillation spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) Qualify affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-distillation spike %R < 75%	Qualify affected results that are ≥ MDL as estimated low (J-) Qualify affected non-detects as estimated (UJ)
Matrix Spike %R 30-74% Post-distillation spike %R ≥ 75%	Qualify affected results that are ≥ MDL as estimated (J) Qualify affected non-detects as estimated (UJ)
Matrix Spike %R > 125% Post-distillation spike %R > 125%	Qualify affected results that are ≥ MDL as estimated high (J+)
Matrix Spike %R > 125% Post-distillation spike %R ≤ 125%	Qualify affected results that are ≥ MDL as estimated (J)
Matrix Spike %R < 30% No post-distillation spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R)
Matrix Spike %R 30-74% No post-distillation spike performed	Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ)
Matrix Spike %R > 125% No post-distillation spike performed	Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified

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Regional Quality Assurance (QA) and Quality Control (QC)

A. Review Items:

Form I-IN, instrument printouts, and raw data.

B. Objective:

Regional QA/QC samples refer to any QA and/or QC sample initiated by the Region, including field duplicate samples, Performance Evaluation (PE) sample, blind spikes, and blind blanks. The use of these QA/QC sample sis highly recommended (e.g., the use of field duplicates can provide information on sampling precision and homogeneity).

C. Criteria:

Criteria are determined by each Region.

D. Evaluation:

Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Compare results for PE samples to the acceptance criteria for the specific PE samples if possible.

Calculate the Relative Percent Difference (RPD) between field duplicates and provide this information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Contract Laboratory Program Project Officer (CLP PO) action.

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Overall Assessment

A. Review Items:

Entire data package, data review results, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data (including any confirmation data).

B. Objective:

The objective is to ensure that the reported sample quantitation results are accurate. It is appropriate for the data reviewer to make professional judgments and express concerns, as well as to comment on the validity of the overall data for a Case. This is particularly appropriate when there are several Quality Control (QC) criteria that are outside of the specification parameters. The additive nature of QC factors that fall outside of specification parameters is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user concerning data quality and data limitations to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The datareviewer would be greatly assisted in this endeavor if the acceptance or performance criteria were provided. The Inorganic Review Summary (see Appendix B) and supplementary documentation must be included with the review.

C. Criteria:

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method.

D. Evaluation:

Examine the raw data to verify that the correct calculation of the sample results was reported by the laboratory. Distillation logs, instrument printouts, etc., should be compared to the reported sample results recorded on the appropriate Inorganic Summary Forms (Form I-IN through Form XVI-IN).

1. Evaluate any technical problems not previously addressed.
2. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).
3. Verify that the appropriate methods and amounts were used to prepare samples for analysis. If reduced volumes were used, verify that the laboratory had received Contract Laboratory Program Project Officer (CLP PO) approval for the use of the reduced volume.
4. Verify that there were no transcription or reduction errors [e.g., dilutions, Percent Solids (%S), sample weights, etc.] on one or more samples.
5. Verify that results fall within the calibrated range for cyanide.
6. If appropriate information is available, the reviewer may assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the Standard Operating Procedure(s) (SOPs), and communication with user concerning the intended use and desired quality of these data.

E. Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.
2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the SDG Narrative for CLP PO action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.

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3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

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Calculations for Cyanide

Aqueous/Water Sample Concentration:

$$\text{CN Concentration (ug/L)} = C \times \frac{V_f}{V} \times DF$$

Where,

C	=	Instrument response in µg/L CN from the calibration curve
V_f	=	Final prepared (absorbing solution) volume (mL)
V	=	Initial aliquot amount (mL)
DF	=	Dilution Factor

Soil/Sediment Sample Concentration:

$$\text{CN Concentration (mg/kg)} = C \times \frac{V_f}{W \times S} \times (1/1000) \times DF$$

Where,

C	=	Instrument response in µg/L CN from the calibration curve
V_f	=	Final prepared (absorbing solution) volume (mL)
W	=	Initial aliquot amount (g)
S	=	% Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6)
DF	=	Dilution Factor

Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:

To calculate the adjusted aqueous/water MDL or adjusted aqueous/water CRQL, follow the instructions in Exhibit D - Data Analysis and Calculations, Section 11.1.1.

The adjusted soil/sediment MDL or adjusted soil/sediment CRQL for all methods shall be calculated as follows:

$$\text{Adjusted Concentration (mg/kg)} = C \times \frac{W_M}{W \times S} \times DF$$

Where,

C	=	MDL or CRQL (mg/kg)
W_M	=	Minimum method required aliquot amount (1.00 g for Midi or 0.50 g for Micro)
W	=	Initial aliquot amount (g)
S	=	% Solids/100 (see Exhibit D - Introduction to Analytical Methods, Section 1.6).
DF	=	Dilution Factor

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APPENDIX A: GLOSSARY

Analyte -- The element of interest, ion, or parameter an analysis seeks to determine.

Analytical Services Branch (ASB) -- Directs the Contract Laboratory Program (CLP) from within the Office of Superfund Remediation and Technical Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).

Analytical Sample -- Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB). Note that the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA); Matrix Spike samples; duplicate samples; serial dilution samples, analytical (post-digestion/post-distillation) spike samples; Interference Check Samples (ICSs); Laboratory Control Samples (LCSs); and Preparation Blanks.

Associated Samples -- Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples run under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.

Blank -- A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

Calibration -- The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

Calibration Blank -- A blank solution containing all of the reagents in the same concentration as those used in the analytical sample preparation. This blank is not subject to the preparation method.

Calibration Curve -- A plot of instrument response versus concentration of standards.

Calibration Standards -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

Case -- A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

Continuing Calibration Blank (CCB) -- A reagent water sample that is run 2 hours (ICP-AES, ICP-MS) or every hour (Hg, CN) and designed to detect any carryover contamination.

Contract Compliance Screening (CCS) -- A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under USEPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.

Continuing Calibration Verification (CCV) -- A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples. The CCV should have a concentration in the middle of the calibration range and shall be run every 2 hours (ICP-AES, ICP-MS) or every hour (Hg, CN).

Contract Laboratory Program (CLP) -- Supports the USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technical Innovation (OSRTI) of USEPA.

Contract Laboratory Program Project Officer (CLP PO) -- The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.

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Contract Required Quantitation Limit (CRQL) -- Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

Duplicate -- A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Field Blank -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

Field Duplicate -- A duplicate sample generated in the field, not in the laboratory.

Holding Time -- The maximum amount of time samples may be held before they are processed.

Contractual -- The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.

Technical -- The maximum amount of time that samples may be held from the collection date until analysis.

Initial Calibration -- Analysis of analytical standards for a series of different specified concentrations to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Initial Calibration Blank (ICB) -- The first blank standard run to confirm the calibration curve.

Initial Calibration Verification (ICV) -- Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when USEPA ICV solutions are not available.

Internal Standard -- A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

Interference Check Sample (ICS) -- Verifies the contract laboratory's ability to overcome interferences typical of those found in samples.

Laboratory Control Sample (LCS) -- A control sample spiked at known level(s). LCSs are processed using the same sample preparation, reagents, and analytical methods employed for the USEPA samples received.

Matrix -- The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrices are aqueous/water, soil/sediment, wipe, and filter.

Matrix Spike -- Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology (also identified asa pre-distillation/digestionspike).

Method Detection Limit (MDL) -- The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

Narrative (SDG Narrative) -- Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Office of Solid Waste and Emergency Response (OSWER) – The USEPA office that provides policy, guidance, and direction for the USEPA's solid waste and emergency response programs, including Superfund.

Percent Difference (%D) -- As used in this document and the Statement of Work (SOW), is used to compare two values. The difference between the two values divided by one of the values.

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Performance Evaluation (PE) Sample -- A sample of known composition provided by USEPA for contractor analysis. Used by USEPA to evaluate Contractor performance.

Post Digestion Spike -- The addition of a known amount of standard after digestion or distillation (also identified as an analytical spike).

Preparation Blank -- An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

Relative Percent Difference (RPD) -- As used in this document and the Statement of Work (SOW) to compare two values, the RPD is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

Regional Sample Control Center Coordinator (RSCC) -- In USEPA Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.

Relative Standard Deviation (RSD) -- As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.

Sample -- A single, discrete portion of material to be analyzed, which is contained in single or multiple containers and identified by a unique Sample Number.

Sample Delivery Group (SDG) -- A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
b. Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
c. Scheduled at the same level of deliverable.

In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG. Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory.

Sample Management Office (SMO) -- A contractor-operated facility operated under the SMO contract, awarded and administered by the USEPA. Provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).

Serial Dilution -- The dilution of a sample by a factor of five. When corrected by the Dilution Factor (DF), the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents [Inductively Coupled Plasma (ICP) only].

Statement of Work (SOW) -- A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

Tune -- Analysis of a solution containing a range of isotope masses to establish Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) mass-scale accuracy, mass resolution, and precision prior to calibration.

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APPENDIX B:
INORGANIC DATA REVIEW SUMMARY

CASE NO. _____ SITE _____

LABORATORY _____ NO. OF SAMPLES/MATRIX _____

MOD. REF. NO. _____ SDG NO. _____

SOW NO. _____ REGION _____

REVIEWER NAME _____ COMPLETION DATE _____

CLP PO: ACTION _____ FYI _____

REVIEW CRITERIA	**METHOD/ANALYTE**			
	ICP-AES	ICP-MS	Mercury	Cyanide
1. Preservation/Holding Time	_____	_____	_____	_____
2. Calibration	_____	_____	_____	_____
3. Blanks	_____	_____	_____	_____
4. Interference Check Sample	_____	_____		
5. Laboratory Control Sample	_____	_____		
6. Duplicate Sample Analysis	_____	_____	_____	_____
7. Spike Sample Analysis	_____	_____	_____	_____
8. ICP Serial Dilution	_____	_____		
9. ICP-MS Tune Analysis	_____			
10. ICP-MS Internal Standards	_____			
11. Field Duplicates	_____	_____	_____	_____
12. Overall Assessment	_____	_____	_____	_____

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VALIDATION SOP

% Moisture (solid/tissue) and Grain Size (solids)

EDS SOP V-14, Rev.2, 2/10 - Verification/Validation Geotechnical Data

Environmental Data Services
SOP V-14
Verification/Validation
Geotechnical

Site: _____ Project: _____
Collection Date(s): _____ SDG: _____
Verifier/Validator: _____ Date(s): _____

DIRECTIONS: Complete the checklist questions and proceed with the corresponding "Action," when necessary. Attachment 1 shall be completed to tally the number of samples exhibiting non-conformances (outliers).

Geotechnical Parameters (circle those that apply):

Grain Size	Organic Content
Specific Gravity	Water Content
Atterberg Limits	Bulk Density/In-Place Density
Consolidation	Paint Filter
Unconsolidated undrained shear strength	
Consolidated undrained shear strength	

	Y	N	N/A
I. Data Completeness and Deliverables			
1. Were all project specified deliverables received? Refer to QAPP worksheet #29 for required deliverables.	___	___	___
2. Is there evidence of proper communication procedures by laboratory and/or field personnel?	___	___	___
Action: If "no", contact the laboratory for submittal of missing or illegible information.			
II. Chain-of-Custody (COC)			
1. Are all field and laboratory COCs present and complete for all samples?	___	___	___
2. Were the project required analytical groups and corresponding analytes reported as requested on the field COCs?	___	___	___
3. Examine chain-of-custody records against the project procedural requirements specified in the QAPP. Were all project requirements met?	___	___	___
Action: If "no", contact the laboratory or field personnel as appropriate for submittal of missing or illegible information.			
III. Sample Handling and Receipt			
1. Were samples received at the appropriate temperature (0-10°C)? Record cooler temps: _____ °C	___	___	___
2. Were any deviations from project required sample handling, receipt, or storage documented?	___	___	___
Action: If "no", contact the laboratory or field personnel for submittal of missing or illegible information. Document the non-conformance and associated samples along with potential data limitations and attach to this checklist.			

Environmental Data Services
SOP V-14
Verification/Validation
Geotechnical

	Y	N	N/A
IV. Final Data / EDD			
1. Was a Case Narrative including the following present for each analysis group?			
☐ Identification of QC samples	___	___	___
☐ Communication logs	___	___	___
☐ Corrective action reports	___	___	___
☐ Documentation of corrective action results	___	___	___
☐ Documentation of laboratory method deviations	___	___	___
☐ Signatures for laboratory sign-off	___	___	___
2. Were results reported for all geotechnical analytes specified in the QAPP?	___	___	___
3. Perform a 10% transcription check, i.e., from laboratory instrument to form. Was the accuracy of transcription verified?	___	___	___
4. Compare results on Form 1s to EDD. Is the EDD free of transcription errors?	___	___	___
Action: If "no", contact the laboratory or field personnel for submittal of missing or illegible information. Document the non-conformance and associated samples along with potential data limitations and attach to this checklist.			
V. Sampling and Analytical Methods and Procedures			
1. Were required sampling and field measurement methods used and were any deviations noted (see QAPP Worksheet #21)?	___	___	___
2. Were required analytical methods used and were any deviations noted (see QAPP Worksheet #23) for?			
☐ Grain Size	___	___	___
☐ Specific Gravity	___	___	___
☐ Atterberg Limits	___	___	___
☐ Organic Content	___	___	___
☐ Water Content	___	___	___
☐ Bulk Density/In-Place Density	___	___	___
☐ Paint Filter	___	___	___
☐ Consolidation	___	___	___
☐ Unconsolidated undrained shear strength	___	___	___
☐ Consolidated undrained shear strength	___	___	___
Action: If "no", contact the laboratory or field personnel for submittal of missing or illegible information. Document the non-conformance and associated samples along with potential data limitations and attach to this checklist.			

	Y	N	N/A
VI. Field Duplicate			
1. Were the % RPD between sample and field duplicate within the project specified limits of <100% for?			
☐ Grain Size	___	___	___
☐ Specific Gravity	___	___	___
☐ Atterberg Limits	___	___	___
☐ Organic Content	___	___	___
☐ Water Content	___	___	___
☐ Bulk Density/In-Place Density	___	___	___
☐ Paint Filter	___	___	___
☐ Consolidation	___	___	___
☐ Unconsolidated undrained shear strength	___	___	___
☐ Consolidated undrained shear strength	___	___	___
Action: Document non-conformance and associated samples along with potential data limitations and attach to this checklist.			

VALIDATION SOP

Total Sulfide

EDS SOP: V-12 Rev.0, 1/09

Environmental Data Services
SOP V-12
Verification/Validation
Sulfide

Site: _____ Project: _____
Collection Date(s): _____ SDG: _____
Verifier/Validator: _____ Date(s): _____

DIRECTIONS: Complete the checklist questions and proceed with the corresponding "Action," when necessary. Attachment 1 shall be completed to tally the number of samples exhibiting non-conformances (outliers).

	Y	N	N/A
I. Data Completeness and Deliverables			
1. Were all project specified deliverables received? Refer to QAPP worksheets #29-2 thru 29-4 for required deliverables.	___	___	___
2. Is there evidence of proper communication procedures by laboratory and/or field personnel?	___	___	___
Action: If "no", contact the laboratory for submittal of missing or illegible information.			
II. Chain-of-Custody (COC)			
1. Are all field and laboratory COCs present and complete for all samples?	___	___	___
2. Were the project required analytical groups and corresponding analytes reported as requested on the field COCs?	___	___	___
3. Examine chain-of-custody records against the project procedural requirements specified in the QAPP. Were all project requirements met?	___	___	___
Action: If "no", contact the laboratory or field personnel as appropriate for submittal of missing or illegible information.			
III. Sample Handling and Receipt			
1. Were samples received at the appropriate temperature (0-10°C)? Record cooler temps: _____ °C	___	___	___
2. Were samples received with proper preservation?	___	___	___
3. Were any deviations from project required sample handling, receipt, or storage documented?	___	___	___
Action: If "no", contact the laboratory or field personnel for submittal of missing or illegible information. Document the non-conformance and associated samples along with potential data limitations and attach to this checklist.			

Environmental Data Services
SOP V-12
Verification/Validation
Sulfide

	Y	N	N/A
IV. Final Data / EDD			
1. Was a Case Narrative including the following present for each analysis group?			
☐ Identification of QC samples	___	___	___
☐ Communication logs	___	___	___
☐ Corrective action reports	___	___	___
☐ Documentation of corrective action results	___	___	___
☐ Documentation of laboratory method deviations	___	___	___
☐ Signatures for laboratory sign-off	___	___	___
2. Were results reported for all analytes specified in the QAPP for each analysis group (see applicable table in worksheet #15)?	___	___	___
3. Were project specified quantitation limits achieved (see applicable table in worksheet #15)?	___	___	___
4. Were project specified laboratory data qualifiers used (see worksheet #29-3)?	___	___	___
5. Perform a 10% transcription check, i.e., from laboratory instrument to form. Was the accuracy of transcription verified?	___	___	___
6. Compare results on Form 1s to EDD. Is the EDD free of transcription errors?	___	___	___
Action: If "no", contact the laboratory or field personnel for submittal of missing or illegible information. Document the non-conformance and associated samples along with potential data limitations and attach to this checklist.			
V. Holding Time			
1. Were solid and liquid samples analyzed within 7 days of collection?	___	___	___
2. If holding times were not met, were deviations documented, appropriate notifications made and approval to proceed obtained?	___	___	___
	___	___	___
Action: If "no", contact the laboratory or field personnel for submittal of missing or illegible information. Document the non-conformance and associated samples along with potential data limitations and attach to this checklist.			
VI. Sampling and Analytical Methods and Procedures			
1. Were required sampling and field measurement methods used and were any deviations noted (see QAPP Worksheet #21)?	___	___	___
2. Were required analytical methods used and were any deviations noted (see QAPP Worksheet #23-1)?	___	___	___
Action: If "no", contact the laboratory or field personnel for submittal of missing or illegible information. Document the non-conformance and associated samples along with potential data limitations and attach to this checklist.			

Environmental Data Services
SOP V-12
Verification/Validation
Sulfide

	Y	N	N/A
VII. Blanks			
1. Were the preparation (extraction) blanks associated with the samples free of contamination (i.e., no target compound > PQL)?	___	___	___
2. Were the rinsate blanks associated with the samples free of contamination (i.e., no target compound > PQL)?	___	___	___
Action: Document non-conformance and associated samples along with potential data limitations and attach to this checklist.			
VIII. Matrix Spike (MS)			
1. Were the matrix spike recoveries within the project specified limits or 75-125% for Sediment?	___	___	___
Action: Document non-conformance and associated samples along with potential data limitations and attach to this checklist.			
IX. Lab Duplicates			
1. Were the % RPD (% Reproducibility) between laboratory duplicates within the project specified limit of 35% when target in both original and lab duplicate samples are >5x PQL or concentrations differ by <2x PQL when detects are <5x PQL for Sediment?	___	___	___
Action: Document non-conformance and associated samples along with potential data limitations and attach to this checklist.			
X. Laboratory Control Sample (LCS)			
1. Were the LCS recoveries within the project specified limits described below:			
Liquid 80-120%	___	___	___
Sediment certified acceptance range noted on Form VII	___	___	___
Action: Document non-conformance and associated samples along with potential data limitations and attach to this checklist.			
XI. Field Duplicate			
1. Were the % RPD between sample and field duplicate within the project specified limits, i.e.,			
RPD <100% when target in both field duplicate samples are >5x PQL?	___	___	___
or			
Concentrations differ by <2x PQL when detects are <5x PQL?	___	___	___
Action: Document non-conformance and associated samples along with potential data limitations and attach to this checklist.			

Appendix O

SOP No. 8 – Crab and Clam Tissue Processing

Standard Operating Procedure No. 8

Crab and Clam Tissue Processing

October 2013

Revision 0

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2. Procedures **3**

2.1 Equipment List 4

2.2 Equipment Decontamination and Calibration Procedures 5

2.3 Crab Tissue Processing Procedures 5

2.4 Clam Tissue Processing Procedures 8

4. Documentation **10**

5. References **10**

1. Purpose and Scope

The purpose of this document is to define the standard operating procedure (SOP) for processing blue crab (*Callinectes sapidus*) and clam (various species) tissue samples from the Newark Bay Study Area for analysis. Crab and clam samples will be analyzed for constituents as outlined in the Crab and Clam Sampling and Analysis Quality Assurance Project Plan (Crab/Clam QAPP).

This SOP may change depending upon equipment limitations or limitations imposed by the procedure. Substantive modification to this SOP will be approved in advance by Tierra Solutions Inc.'s (Tierra's) Facility Coordinator and the United States Environmental Protection Agency Remedial Project Manager. The ultimate procedure employed will be documented in the laboratory notebook.

Other SOPs will be utilized with this procedure, include:

- SOP No. 3 – Decontamination.
- SOP No. 4 – Management and Disposal of Residuals
- SOP No. 5 - Containers, Preservation, Handling, and Tracking of Samples for Analysis
- SOP No. 10 - Documentation of Field Activities
- SOP No 11 – Data Management

In instances where this SOP will be utilized, a hard copy or electronic version will be available at the point of use.

2. Procedures

Field-collected organisms will be preserved on wet ice within a properly labeled, sealed, and documented (i.e., with chain-of-custody form(s)) container, and will be received from the field processing facility via express overnight courier. Upon receipt, the laboratory representative will place all clams and crabs in frozen storage. For clams, the freezing process will allow tissue expansion, which will cause dehingment of the shell and allow for safer shucking procedures upon defrosting.

It is anticipated that all tissue processing will be conducted at the end of the collection period. Coordination between field and laboratory staff regarding verification of samples received and completeness (i.e., full number of target clams or crabs per

sample) will allow for proper compositing of tissue samples received over multiple sample shipments. Further decisions for sample compositing of hepatopancreas tissue from crab samples will be based on initial tissue processing results. A sufficient number of crabs will be utilized to meet the analytical sample volumes for each tissue type specified in Worksheet No. 10 of the Crab/Clam QAPP. Hepatopancreas tissue samples will likely be composited using crabs from more than one location within a zone (i.e., Newark Bay North).

At each location, preference should be given to compositing equal numbers of male and female blue crabs of similar relative size, as practicable. Clam and crab tissue samples will be processed in a timely manner to meet the specified holding times detailed within the QAPP.

Sample batches will be coordinated with the laboratory for defrosting to allow laboratory technician and contractor biologist/ecologist time to process samples during an eight hour work day. This will alleviate any concerns for partial processing or any unnecessary freezing and thawing cycles that would potentially compromise the tissue samples.

The following procedures will be used to process crab and clam samples.

2.1 Equipment List

The following equipment list contains materials that may be needed to perform the procedures outlined in this SOP. Not all equipment listed below may be necessary for a specific activity. Additional equipment not listed here may be required dependent on field conditions.

- ☐ personal protective equipment and other safety equipment, as required by the Health and Safety/ContingencyPlan, Rev. 1 (Tierra 2007)
- ☐ stainless steel shell-cracker
- ☐ stainless steel spoon or knife
- ☐ stainless steel strainer
- ☐ glass plate or petri dish
- ☐ glass or stainless steel work tray

- electronic balance with calibration weights (checked for accuracy at least daily, and documented in accordance with laboratory protocols)
- labeled sample jars
- laboratory logbook or individual bench sheet forms.

2.2 Equipment Decontamination and Calibration Procedures

1. Personal protective equipment will be donned and procedures outlined in SOP #3 will be reviewed prior to performing equipment decontamination.
2. Prior to start of tissue processing for each individual tissue composite sample, all laboratory glassware and instruments will be cleaned according to SOP #3.
3. Steps 1 and 2 will be repeated at the end of the processing for each tissue composite sample or less frequently if the inventory of properly cleaned laboratory glassware and instruments are still available.
4. Laboratory electronic balance capable of reading to 0.1 grams will be used to measure the processed crab and clam tissue.
5. At the start of each sample processing day, the designated laboratory electronic balance will be calibrated using certified sample weights supplied by the manufacturer. The target calibration weight mass will be recorded in the laboratory logbook. The electronic balance will be tared to zero and the calibrated weight will be measured using the electronic balance. The mass will be recorded in the laboratory logbook and if within the specified tolerances set forth in the QAPP or by the manufacturer, then no adjustments will be made. If the measured weight falls outside of the tolerances, then a calibration adjustment will be made to the electronic balance following the manufacturer's specifications and/or procedures.

2.3 Crab Tissue Processing Procedures

Tissue processing for crabs will follow the following procedures:

1. Personal protective equipment will be donned and procedures will be reviewed prior to performing crab tissue processing.
2. Individually bagged crab tissue composite samples will have been thawed from laboratory frozen storage to allow tissue processing to commence.

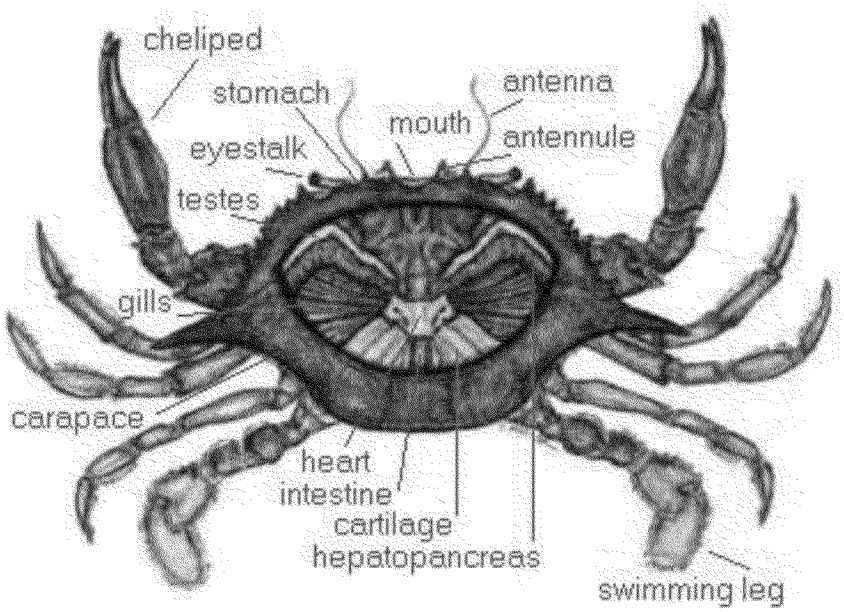
3. Based on updated COCs and received field sample forms, crabs will be sorted and compiled into the specified sample composite per location.

4. Upon sorting, each individual crab will be rinsed with de-ionized water to remove any remaining field sediment or vegetation. Rinsed individuals will be placed on a clean and labeled glass or stainless steel work tray.

5. Place appropriate labeled sample containers used for holding and homogenization of muscle and hepatopancreas tissue in work station. Have each sample container pre-tared to allow for accurate weight measurements of processed tissue samples. Record sample container tare weights in laboratory logbook for each sample.

6. Review the anatomy of the blue crab shown below to get familiar with the position of hepatopancreas tissue.

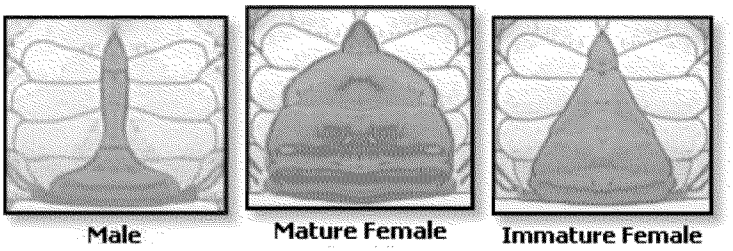
Figure 1. Anatomy of Blue Crab



Graphic courtesy of Virginia Sea Grant

7. Individual crabs will be dissected to obtain separate samples of muscle and hepatopancreas tissue according to the following protocols as practicable.

8. Determine the sex of the blue crab by looking at the abdomen (specifically the apron area highlighted in red) of each individual as shown in the pictures below. Record each individual's sex in laboratory logbook.



Diagrams courtesy of BlueCrab.Info at: http://www.bluecrab.info/identification.html

9. Break off the chelipeds at the carapace and place claws aside for tissue removal. Lift the tail, place fingers into the body cavity of the crab and pull the top carapace off, exposing the internal organs. Use caution during this step to avoid potential disturbance of exposed hepatopancreas tissue.

10. Using a clean, decontaminated stainless steel spoon or knife, remove as much of the hepatopancreas from the upper and lower portions of the carcass as possible, placing the tissue on a decontaminated glass plate or petri dish.

11. Following removal of the hepatopancreas, remove the muscle tissue from the thoracic cavity, claws, legs, and abdomen portions of the crab using a clean, decontaminated stainless steel spoon or knife, placing it on a separate glass plate or petri dish. The edible tissue can be removed from the claws by breaking open the cheliped using a stainless steel cracker and scraping or pulling out all of the muscle tissue. If hepatopancreas tissue had inadvertently fallen or smeared onto muscle tissue, use some de-ionized water to rinse off muscle tissue and place on glass plate or petri dish.

12. Upon completion of the tissue removal of the individual crab, weigh the hepatopancreas and muscle tissue and record each in laboratory logbook.

13. Repeat steps 9 through 12 until the target tissue weight for the sample is achieved. Record the total tissue mass and the total number and sex of each crab processed in the composite tissue sample. For each tissue (muscle and hepatopancreas), a composite sample should have approximately 232 grams. If additional individuals were collected to perform a QA/QC laboratory sample, then approximately 464 grams is required. If upon completion of processing for both muscle and hepatopancreas tissue per composite sample the minimum tissue mass is not met, then immediate contact should be made with the laboratory project manager to allow for communication with the project manager and subsequent decision-making for sample analysis.

14. Place in labeled sample jar on ice until laboratory sample homogenization is performed using the laboratory-specific SOP.

2.4 Clam Tissue Processing Procedures

Tissue processing for clams will follow the following procedures:

1. Personal protective equipment will be donned and procedures will be reviewed prior to performing clam tissue processing.

2. Individually bagged clam tissue composite samples will be thawed from laboratory frozen storage to allow tissue processing to commence. Drain excess liquid from sample bag into waste sink.

3. Based on updated COCs and received field sample forms, clam tissue individuals will be sorted and compiled per sample location to form a composite.

4. Upon sorting, each individual clam will be placed on a clean and labeled glass or stainless steel work tray.

5. Place appropriate labeled sample containers used for holding and homogenization of muscle tissue in work station. Have each sample container pre-tared to allow for accurate weight measurements of processed tissue samples. Record sample container tare weights in laboratory logbook for each sample.

6. Review the anatomy of the soft shell clam, as shown below. All internal tissue will be used for the tissue analysis.

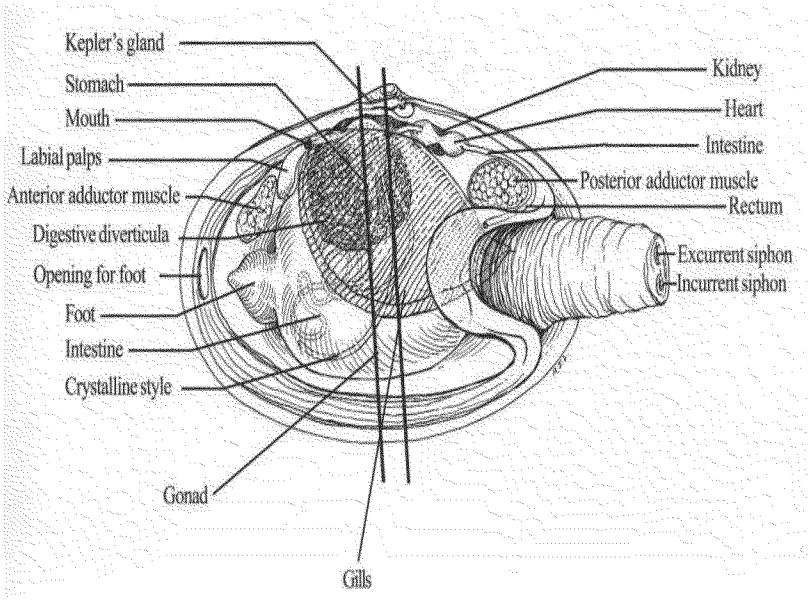


Illustration From: Howard DW, Lewis EJ, Keller BJ, et al. Histological Techniques for Marine Bivalve Mollusks and Crustaceans. NOAA Technical Memorandum NOS NCCOS 5. Washington, DC: National Oceanic and Atmospheric Administration; 2004.

1. Based on freezing and thawing cycle, the clams should be slightly open and possibly dehinged to allow for easy shucking. Using a clean, decontaminated knife, gently pry open the clam and remove the soft tissue from the shell. Place soft tissue on a decontaminated glass plate or petri dish.

2. When all clams from a given location have been processed, combine the soft tissue to form a single composite sample. Place the composite sample in a stainless steel strainer for approximately one minute to allow for excess liquid to decant. After the excess moisture has been removed, dump the contents of the strainer into a tared sample jar and weigh the total mass of the soft tissue and record in laboratory logbook. Each tissue composite sample should have approximately 232 grams. If additional individuals were collected to perform a QA/QC laboratory sample, then approximately 464 grams is required. If upon completion of processing the soft tissue per composite sample the minimum tissue mass is not met, then immediate contact should be made with the laboratory project manager to allow for communication with the project manager and subsequent decision-making for sample analysis.

3. Place in labeled sample jar on ice until laboratory sample homogenization is performed using the laboratory-specific SOP.

3. Quality Assurance

The following quality assurance/quality control procedures will be performed during the sampling events:

- All pertinent field information and COCs will be provided to the sample processing team. The designated contractor biologist/ecologist will review the sample information and confirm that the number of individuals, collection date(s), collection time(s), species, and required tissue analyses match the COCs. If any questions arise, the contractor biologist/ecologist will contact the project manager and laboratory project manager to resolve.

- Laboratory logbook will be checked between individual and composite tissue processing steps to make sure required information is completed per the next section.

4. Documentation

Sample measurements and observations will be noted in the laboratory logbook or on individual bench sheets. Information to be recorded in the laboratory notebook will include, at a minimum:

- responsible person's name
- dates and times of activities (including collection and processing)
- identification and size of each organism (as appropriate)
- sample IDs
- sample location(s)
- tissue weights (both individual and total crab sample and total clam sample).

5. References

Tierra. 2007. Newark Bay Study Area Remedial Investigation Work Plan [Rev. 1]. Volume 2 Health and Safety/Contingency Plan. September.