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OBJECTIVE

This Standard Operating Procedure (SOP) describes the requirements for the data packages that will be generated as part of the Hudson River Design Support Sediment Sampling and Analysis Program. This SOP applies to the contractor(s) involved in analytical data generation and reporting. All data packages generated for the Hudson River Design Support Sediment Sampling and Analysis Program must be provided in an Adobe Acrobat (.PDF) file format. The laboratory will be notified of the samples that will undergo data validation. For these samples, the laboratory will be required to generate hard copy data packages as well as the Adobe Acrobat (.PDF) file format.

SECTION A-9 DATA PACKAGE DELIVERABLES

The following sections describe in detail the types of data packages designed for the Hudson River Design Support Sediment Sampling and Analysis Program. These details are provided to allow several participating laboratories to produce data packages that are similar in format, order of presentation, and content. The data packages detailed in Section A-9.1 have been developed based on deliverables specified in the US EPA Contract Laboratory Program Statement of Work (CLP SOW). The CLP SOW has additional details concerning data packages that are specific to the CLP analyses. The most recent Statement of Work should be referenced for details concerning CLP-style data packages. Note: the summary forms provided in these data packages should be in similar format and content to the Contract Laboratory Program (CLP) forms listed (as references) next to the form title. These CLP forms references are only provided as guidance on content and format and should be modified by the laboratory to meet specific method requirements. Section A-9.2 provides details concerning specific contents of the data deliverables described in Section A-9.1.

The data package deliverables are categorized into two distinct levels as follows:

Level A - Case Narrative, analytical results, and Chain-of-Custody Records for the sample delivery group (SDG).

Level B - Fully documented data package.

The Level A data package is a basic "results-only" style of data package that includes a cover letter, SDG narrative, field Chain-of-Custody Records, analytical results summaries, and a glossary of qualifier codes. The Level B package resembles the information required by the CLP SOW. This type of package includes all information provided in Level A package but also includes summary forms for quality control procedures and all sample and quality control raw data to support the results reported.

A-9.1 Data Package Contents and Order of Presentation

The laboratory will be required to submit supporting documentation for the reported analytical results. The supporting documentation and the analytical results will be reported in one of two data package delivery categories. The categories are defined below. The data package deliverables must be submitted in the order in which the deliverables appear in the text. The laboratory need not include the documentation for any fraction not required for an SDG.

A-9.1.1 General Format for Level B Deliverables

For some analyses, Level B Sample Data Package deliverables may be requested. A Level A Data Package will also be required with the Level B package as a summary package.

The Level B Sample Data Package shall include data for analyses of all samples in one SDG, including field samples, reanalyses, secondary dilutions, blanks, laboratory control samples, matrix spikes, matrix spike duplicates, and/or laboratory duplicates. The complete Sample Data Package is divided into the units as described below. Units for each analytical fraction have been detailed. If the analysis of that fraction was not required for samples in the SDG, the fraction-specific unit is not a required deliverable. The Sample Data Package must be complete before submission and must be consecutively paginated. The Sample Data Package will be arranged in the following order:

A) Cover Letter/Letter of Transmittal signed by the laboratory manager.

B) Title Page

C) Table of Contents

D) Sample Delivery Group (SDG) Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name; SDG number; GE sample identifications; laboratory sample numbers; and detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing (preparing and analyzing) the samples reported in the data package. A glossary of qualifier codes used in the SDG must also be provided.

The laboratory must also include any technical and administrative problems encountered, corrective actions taken and method of resolution, and an explanation of all flagged edits (i.e., exhibit edits) on quantitation reports.

Additionally, the SDG Narrative must be signed and dated by the laboratory manager.

E) Field and Internal (Laboratory) Chain-of-Custody Records and Sample Receipt Documentation Log

Copies of both the external and internal Chain-of-Custody Records for all samples within the SDG must be included in the deliverables. A description of the condition and temperature of the samples upon laboratory receipt (*i.e.*, custody seal condition, container status) must be provided for each Chain-of-Custody Record/sample cooler.

F) GC/MS Volatile Organic Data.

1. Quality Control (QC) Summary.

a. Surrogate Percent Recovery Summary (modified CLP SOW288 Form II VOA).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOW288 Form III VOA).

c. Laboratory Control Sample Summary (modified CLP SOW288 Form III VOA).

d. Method Blank Summary (modified CLP SOW288 Form IV VOA) -- arranged in chronological order by date of analysis of the blank, by instrument.

e. GC/MS Tuning and Mass Calibration Summary (modified CLP SOW288 Form V VOA) -- arranged in chronological order, by instrument.

f. Internal Standard Area and Retention Time Summary (modified CLP SOW288 Form VIII VOA) -- arranged in chronological order, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for volatile samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Target Compound Results (modified CLP SOW288 Form I VOA).

b. Reconstructed total ion chromatogram (RIC) and quantitation reports.

c. Copies of raw spectra and copies of background-subtracted mass spectra of each target compound identified in the sample and corresponding background-subtracted target compound standard mass spectra.

d. Exhibit work sheet (including example calculations showing how sample results are calculated using the initial calibration and sample responses for at least one sample).

3. Standards Data

a. Initial Calibration Data (modified CLP SOW288 Form VI VOA and associated volatile standard RICs and quantitation reports) -- for all initial calibrations associated with analyses in the SDG, in chronological order, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

b. Continuing Calibration Data (modified CLP SOW288 Form VII VOA and associated volatile standard RICs and quantitation reports) -- for all continuing calibrations associated with analyses in the SDG, in chronological order, by instrument.

4. Raw QC Data

a. For each GC/MS tuning and mass calibration (in chronological order, by instrument):

1. Bromofluorobenzene (BFB) bar graph spectrum.

2. BFB mass listing.

b. Method/Storage Blank Data - in chronological order, by instrument:

i. Target Compound Results (modified CLP SOW288 Form I VOA).

ii. RIC and quantitation reports.

iii. Copies of raw spectra and copies of background-subtracted mass spectra of each target compounds identified in the blank and corresponding background-subtracted target compound standard mass spectra.

c. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOW288 Form I VOA).

ii. RIC and quantitation reports.

d. Matrix Spike Data:

i. Target Compound Results (modified CLP SOW288 Form I VOA).

ii. RIC and quantitation reports.

e. Matrix Spike Duplicate Data:

i. Target Compound Results (modified CLP SOW288 Form I VOA).

ii. RIC and quantitation reports.

G) GC/MS Semivolatile Organic Data

1. QC Summary

a. Surrogate Percent Recovery Summary (modified CLP SOW288 Form II SV).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOW288 Form III SV).

c. Laboratory Control Sample Summary (modified CLP SOW288 Form III SV).

d. Method Blank Summary (modified CLP SOW288 Form IV SV) -- arranged in chronological order by date of analysis of the blank, by instrument.

e. GC/MS Tuning and Mass Calibration Summary (modified CLP SOW288 Form V SV) -- arranged in chronological order, by instrument.

f. Internal Standard Area and Retention Time Summary (modified CLP SOW288 Form VIII SV-1, SV-2) -- arranged in chronological order, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries, followed by the raw data for semivolatile samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Target Compound Results (modified CLP SOW288 Form I SV-1, SV-2).

b. RIC and quantitation report.

c. Copies of raw spectra and copies of background-subtracted mass spectra of each target compound identified in the sample and corresponding background-subtracted target compound standard mass spectra.

d. UV traces from Gel Permeation Chromatography (GPC) chromatograms cleanup (if performed).

e. Exhibit work sheet (including example calculations showing how sample results are calculated using the initial calibration and sample responses for at least one sample).

3. Standards Data

   a. Initial Calibration Data (modified CLP SOW288 Form VI SV-1, SV-2 and associated semivolatile standard RICs and quantitation reports) -- for all initial calibrations associated with analyses in the SDG, in chronological order, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

   b. Continuing Calibration Data (modified CLP SOW288 Form VII SV-1, SV-2 and associated semivolatile standard RICs and quantitation reports) -- for all continuing calibrations associated with analyses in the SDG, in chronological order, by instrument.

4. Raw QC Data

   a. For each GC/MS tuning and mass calibration (in chronological order, by instrument):

      i. Decafluorotriphenylphosphine (DFTPP) bar graph spectrum.

      ii. DFTPP mass listing.

   b. Blank Data -- in chronological order, by instrument:

      i. Target Compound Results (modified CLP SOW288 Form I SV-1, SV-2).

ii. RIC and quantitation reports.

iii. Copies of raw spectra and copies of background-subtracted mass spectra of each target compounds identified in the blank and corresponding background-subtracted target compound standard mass spectra.

c. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOW288 Form I SV-1, SV-2).

ii. RIC and quantitation reports.

d. Matrix Spike Data:

i. Target Compound Results (modified CLP SOW288 Form I SV-1, SV-2).

ii. RIC and quantitation reports.

e. Matrix Spike Duplicate Data

i. Target Compound Results (modified CLP SOW288 Form I SV-1, SV-2).

ii. RIC and quantitation reports.

H) GC Organochlorine Pesticide Data

1. QC Summary

a. Surrogate Percent Recovery Summary (modified CLP SOW288 Form II PEST).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOW288 Form III PEST).

c. Laboratory Control Sample Summary (modified CLP SOW288 Form III PEST).

d. Method Blank Summary (modified CLP SOW288 Form IV PEST) -- arranged in chronological order by date of analysis of the blank, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for organochlorine pesticide samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary (modified CLP SOW288 Form I PEST).

b. Copies of organochlorine pesticide chromatograms.

c. Copies of organochlorine pesticide chromatograms from second GC column confirmation (if performed).

d. GC integration reports or data system printouts.

e. Exhibit work sheet (including example calculation showing how sample results are calculated using initial calibration standard and sample responses for at least one sample).

f. UV traces from GPC cleanup (if performed).

g. If organochlorine pesticides are confirmed by GC/MS, the laboratory must submit copies of raw spectra and copies of background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. For multi-component pesticides confirmed by GC/MS, the laboratory will submit mass spectra of three major peaks of multi-component compounds from samples and standards.

3. Standards Data

a. Analytical Sequence Form -- in chronological order, by GC column, by instrument for all samples and quality control analyses.

b. Initial Calibration Data (Initial Calibration Summary Form [inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.], organochlorine pesticide standard chromatograms, and integration reports) -- for each initial calibration associated with SDG in chronological order, by GC column, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

c. Continuing Calibration Data (Continuing Calibration Summary Form [inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.], organochlorine pesticide standard chromatograms, and integration reports) -- for each continuing calibration associated with SDG in chronological order, by GC column, by instrument following the associated initial calibrations.

d. 4,4′-DDT and Endrin Breakdown Data (Percent Breakdown Summary Form, organochlorine pesticide chromatograms and integration reports) -- for each standard associated with SDG in chronological order by GC column, by instrument.

4. Raw QC Data

a. Blank Data -- in chronological order, by instrument:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

b. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

c. Matrix Spike Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

d. Matrix Spike Duplicate Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. Organochlorine pesticide chromatograms and integration reports.

e. UV traces from GPC cleanup (if performed).

i. UV traces for the initial calibration standards and blanks. Compound names shall be written over the peaks or printed over the peaks, or retention times shall be written over the peaks, and a separate table listing compounds and retention times shall be provided.

ii. Chromatographs and data system reports for all standards used to quantify compounds in the GPC blanks.

iii. Chromatographs and data system reports for the GPC calibration check solution and all standards used to quantify compounds in the GPC calibration check solution.

f. Raw Florisil® data, arranged in chronological order.

i. Chromatographs and data system reports for the analysis of the Florisil® cartridge performance check.

ii. Chromatographs and data system reports for the standards used to quantify compounds in the Florisil® cartridge performance check analysis (*i.e.*, INDA, INDB, and the 2,4,5-trichlorophenol standards).

I) GC Polychlorinated Biphenyl (PCB) Data

1. QC Summary

a. Surrogate Percent Recovery Summary (modified CLP SOW288 Form II PEST).

b. Matrix Spike/Matrix Spike Duplicate Summary (modified CLP SOW288 Form III PEST).

c. Laboratory Control Sample Summary (modified CLP SOW288 Form III PEST).

d. Method Blank Summary (modified CLP SOW288 Form IV PEST) -- arranged in chronological order by date of analysis of the blank, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for PCB samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary (modified CLP SOW288 Form I PEST).

b. Copies of PCB chromatograms.

c. Copies of PCB chromatograms from second GC column confirmation (if performed).

d. GC integration reports or data system printouts. The integration reports or data system printouts must include all peaks not just the peaks corresponding to the target analytes.

e. Exhibit work sheets (including example calibration showing how sample results are calculated using initial calibration and sample responses for at least one sample).

f. UV traces from GPC (if performed).

g. If PCBs are confirmed by GC/MS, then the laboratory must submit copies of raw spectra and background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. The laboratory will submit mass spectra of three major peaks of multi-component compounds from samples and standards for each PCB result confirmed by GC/MS.

3. Standards Data

a. Analytical Sequence Form -- in chronological order, by GC column, by instrument for all samples and quality control analyses.

b. Initial Calibration Data -- Initial Calibration Summary Form (inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.), PCB standard chromatograms, and integration reports for each initial calibration associated with SDG in chronological order, by GC column, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

c. Continuing Calibration Data -- Continuing Calibration Summary Form (inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.), PCB standard chromatograms, and integration reports for each continuing calibration associated with SDG in chronological order, by GC column, by instrument following the associated initial calibration.

4. Raw QC data

a. Blank Data -- in chronological order, by instrument:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. PCB chromatograms and integration reports.

b. Laboratory Control Sample Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. PCB chromatograms and integration reports.

c. Matrix Spike Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. PCB chromatograms and integration reports.

d. Matrix Spike Duplicate Data:

i. Target Compound Results (modified CLP SOW288 Form I PEST).

ii. PCB chromatograms and integration reports.

e. UV traces from GPC cleanup (if performed).

i. UV traces for the initial calibration standards and blanks. Compound names shall be written or printed over the peaks, or retention times shall be written over the peaks, and a

separate table listing compounds and retention times shall be provided.

ii. Chromatographs and data system reports for all standards used to quantify compounds in the GPC blanks.

iii. Chromatographs and data system reports for the GPC calibration check solution and all standards used to quantify compounds in the GPC calibration check solution (or used to assess the Aroclor pattern).

f. Raw Florisil® data, arranged in chronological order:

i. Chromatographs and data system reports for the analysis of the Florisil® cartridge performance check.

ii. Chromatographs and data system reports for the standards used to quantify compounds in the Florisil® cartridge performance check analysis (*i.e.*, INDA, INDB, and the 2,4,5-trichlorophenol standards).

J) GC Herbicide Data

1. QC Summary

a. Surrogate Percent Recovery Summary ("CLP SOW288-like" Form II PEST).

b. Matrix Spike/Matrix Spike Duplicate Summary ("CLP SOW288-like" Form III PEST).

c. Laboratory Control Sample Summary ("CLP SOW288-like" Form III PEST).

d. Method Blank Summary ("CLP SOW288-like" Form IV PEST) -- arranged in chronological order by date of analysis of the blank, by instrument.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for herbicide samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary ("CLP SOW288-like" Form I PEST).

b. Copies of herbicide chromatograms.

c. Copies of herbicide chromatograms from second GC column confirmation (if performed).

d. GC integration reports or data system printouts.

e. Exhibit work sheets (including example calculation showing how sample results are calculated using initial calibration and sample responses for at least one sample).

f. UV traces from GPC (if performed).

g. If herbicides are confirmed by GC/MS, the laboratory must submit copies of raw spectra and copies of background-subtracted mass spectra of target compounds that are identified in the sample and corresponding background-subtracted target compound standard mass spectra.

3. Standards Data

a. Analytical Sequence Form -- in chronological order, by GC column, by instrument for all samples and quality control analyses.

b. Initial Calibration Data (Initial Calibration Summary Form [inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.], herbicide standard chromatograms, and integration reports) -- for each initial calibration associated with SDG in chronological order, by GC column, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

c. Continuing Calibration Data (Continuing Calibration Summary Form [inclusive of retention time windows, calibration factors, %RSDs, %Ds, etc.], herbicide standard chromatograms, and integration reports) -- for each continuing calibration associated with SDG in chronological order, by GC column, by instrument following the associated initial calibrations.

4. Raw QC Data

a. Blank Data -- in chronological order, by instrument:

i. Target Compound Results ("CLP SOW288-like" Form I PEST).

ii. Herbicide chromatograms and integration reports.

b. Laboratory Control Sample Data:

i. Target Compound Results ("CLP SOW288-like" Form I PEST).

ii. Herbicide chromatograms and integration reports.

c. Matrix Spike Data:

i. Target Compound Results ("CLP SOW288-like" Form I PEST).

ii. Herbicide chromatograms and integration reports.

d. Matrix Spike Duplicate Data:

i. Target Compound Results ("CLP SOW288-like" Form I PEST).

ii. Herbicide chromatograms and integration reports.

e. UV traces from GPC cleanup (if performed).

i. UV traces for the initial calibration standards and blanks. Compound names shall be written or printed over the peaks, or retention times shall be written over the peaks and a separate table listing compounds and retention times shall be provided.

ii. Chromatographs and data system reports for all standards used to quantify compounds in the GPC blanks.

iii. Chromatographs and data system reports for the GPC calibration check solution and all standards used to quantify compounds in the GPC calibration check solution (or used to assess the Aroclor pattern).

K) GC/MS Dioxin/Furan Data

1. Quality Control (QC) Summary

a. Matrix Spike/Matrix Spike Duplicate Summary.

b. Ongoing Precision and Recovery (ORP) Summary.

c. Method Blank Analysis Summary.

2. Sample Data

Sample data shall be arranged in packets consisting of the Analytical Results Summaries followed by the raw data for dioxin/furan samples. These sample packets should then be placed in increasing alphanumeric order by GE sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary.

For each sample including peak retention times, ion ratios, reported concentrations, Estimated Detection Limit (EDL) designation, and internal standard recoveries.

b. Calculation of Toxicity Equivalence.

c. Dioxin/Furan Review Worksheet and Quantitation Report. The quantitation reports must include all information required to reproduce reported positive results and EDL results.

d. Extracted Ion Current Profile (EICP) Chromatograms.

e. Second Column Confirmation Data (if necessary; will include A-9.1.1.K, Section 2, items a, b, c, and d).

f. Exhibit work sheets (including example calibration showing how sample results are calculated using initial calibration and sample responses for at least one sample. The calculations should cover positive results and EDL results).

3. Standards Data

a. Mass spectrometer performance standard data for each calibration associated with the SDG, in chronological order by GC column, by instrument.

b. Window-defining mix and isotope ratio data for each calibration associated with the SDG, in chronological order by GC column, by instrument. The retention time windows must be summarized for reference.

c. Isomer Specificity Test Standard Summary and raw data in chronological order by GC column, by instrument.

d. Initial Calibration Data (Initial Calibration Summary Form, quantitation report, and EICP Chromatograms) for each initial calibration associated with the SDG, in chronological order by GC column, by instrument. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

e. Continuing Calibration Data (Continuing Calibration Summary Form, quantitation report, and EICP Chromatograms) for each continuing calibration associated with the SDG, in chronological order, by GC column, by instrument.

4. Raw QC Data

a. Blank Data -- in chronological order, by instrument:

i. Analytical Results Summary.

For each blank including peak retention times, ion ratios, reported concentrations, EDL designation, and internal standard recoveries.

ii. Dioxin/Furan Review Worksheet and Quantitation Report.

iii. EICP Chromatograms.

b. OPR Standard Data:

i. Analytical Results Summary.

For each OPR standard including peak retention times, ion ratios, reported concentrations, EDL designation, and internal standard recoveries.

ii. Dioxin/Furan Review Worksheet and Quantitation Report.

iii. EICP Chromatograms.

c. Matrix Spike Data:

i. Analytical Results Summary.

For each matrix spike including peak retention times, ion ratios, reported concentrations, EDL designation, and internal standard recoveries.

ii. Dioxin/Furan Review Worksheet and Quantitation Report.

iii. EICP Chromatograms.

d. Matrix Spike Duplicate Data:

i. Analytical Results Summary.

For each matrix spike duplicate including peak retention times, ion ratios, reported concentrations, EDL designation, and internal standard recoveries.

ii. Dioxin/Furan Review Worksheet and Quantitation Report.

iii. EICP Chromatograms.

5. GC/MS Instrument Run Logs.

L) Inorganic Data for ICP or ICP/MS

1. Cover Page for the Inorganic Analyses Data Package.

2. Sample Results Summaries (modified CLP SOW390 Form I-INs) -- for all samples in the SDG, arranged in increasing alphanumeric order by GE sample identification.

3. Quality Control and Quarterly Verification of Instrument Parameters Summaries:

a. Initial and Continuing Calibration Verification summaries (modified CLP SOW390 Form II [PART 1]-INs).

b. Detection Limit Standards summaries (if performed, modified CLP SOW390 Form II [PART 2]-INs).

c. Blanks summaries (modified CLP SOW390 Form III-INs).

d. ICP Interference Check Sample summaries (modified CLP SOW390 Form IV-INs).

e. Matrix Spike/Matrix Spike Duplicate Sample Recovery summary (modified CLP SOW390 Form V [PART 1]-IN).

f. Post-Digest Spike Sample Recovery forms (modified CLP SOW390 Form V [PART 2]-IN).

g. Duplicates summary (modified CLP SOW390 Form VI-IN).

h. Laboratory Control Sample summary (modified CLP SOW390 Form VII-IN)

i. Method of Standard Addition Results summary (modified CLP SOW390 Form VIII-IN).

j. ICP Serial Dilution summary (modified CLP SOW390 Form IX-IN).

k. Method Detection Limits (MDL) and Reporting Limits (modified CLP SOW390 Form X-IN).

l. ICP Interelement Correction Factors (if performed, modified CLP SOW390 Form XI [PART 1]-IN).

m. ICP Linear Ranges (if performed, modified CLP SOW390 Form XII-INs).

n. Preparation Logs (modified CLP SOW390 Form XIII-INs).

o. Analytical Run Logs (modified CLP SOW390 Form XIV-INs).

4. ICP/MS Data Package will also include the following additional forms. The forms for ICP analysis listed A-9.1.1.K Sections 1-3 are also required using the SOW1091-LCIN protocol.

a. Linear Range Standard Summary (if performed, modified CLP For IV-LCIN).

b. ICP and ICP/MS Interference Check Sample (modified CLP Form VI-LCIN).

c. ICP/MS Tuning and Response Factor Criteria (modified CLP Form XIV-LCIN).

d. ICP/MS Internal Standards Summary (modified Form XV-LCIN).

5. Raw Data

For each reported value, the contracted laboratories will provide all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, and all sample analysis results. This statement does not apply to the Quarterly Verifications Parameters submitted as part of each data package (Section A-9.1.1.L, items 3k-3m). Raw data must contain all instrument readouts used for the sample results. Each exposure or instrumental reading must be provided, including those readouts that may fall below the MDL. All AA and ICP instruments must provide a legible hard copy of the direct real-time instrument readout (*i.e.,* strip charts, printer tapes, etc.). A photocopy of the instrument's direct sequential readout must be included. A hard copy of the instrument's direct instrument readout for cyanide must be included if the instrumentation has the capability.

The order of raw data in the data package shall be ICP-AES, ICP/MS, flame AA, furnace AA, mercury, and cyanide. All flame and furnace AA data will be grouped by element.

M) Wet Chemistry/Conventionals Data

The wet chemistry data will be arranged in the following order by individual parameter requested for the samples in the SDG.

1. Analytical Results Summaries -- for all samples in the SDG, arranged in increasing alphanumeric order by GE sample identification.

2. Quality Control Summaries

   a. Initial and Continuing Calibration Verification summaries.

   b. Blanks summaries.

   c. Spike Sample/Spike Duplicate Recovery summary.

   d. Duplicates summary.

   e. Laboratory Control Sample summary.

   f. Analytical Run Logs for instrumental analyses.

3. Raw Data

   For each reported value, the contracted laboratories will provide all raw data (instrument printouts or logbook pages) used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. Raw data must contain all instrument readouts/logbooks pages used for the sample results. Each exposure or instrumental reading must be provided, including those readouts/logbook pages that may fall below the quantitation limit. A photocopy of the instrument's direct sequential readout must be included if the instrumentation has the capability.

P) Preparation Logs

1. TCLP Extraction Logs (if TCLP extraction was performed).

2. Volatile Extraction Logs (if medium-level volatile analyses were performed).

3. Semivolatile Extraction Logs.

4. Organochlorine Pesticide/PCB Extraction Logs.

5. Herbicide Extraction Logs.

6. Dioxin/Furan Extraction Logs.

7. Metals Digestion Logs.

8. Wet Chemistry Preparation Logs (by parameter).

A-9.1.3 General Format for Level A Deliverables

A Level A Data Package will be prepared concurrently with each complete Sample Data Package prepared for quality assurance review. The Level A Data Package shall contain data for all samples in one SDG. All Level A Data Packages will be arranged in the following order:

A) Cover Letter/Letter of Transmittal

B) SDG Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name, SDG number, GE sample identifications, laboratory sample numbers, and detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing (preparing and analyzing) the samples reported in the data package. A glossary of qualifier codes used in the SDG must also be provided.

The laboratory must also include any technical and administrative problems encountered, and corrective actions taken. An explanation of all flagged edits (*i.e.,* exhibit edits) on quantitation reports must be included in the SDG Narrative.

Additionally, the SDG Narrative must be signed and dated by the laboratory manager.

C) Field and Internal (Laboratory) Chain-of-Custody Records and Sample Receipt Documentation Log

Copies of both the external and internal Chain-of-Custody Records for all samples within the SDG must be included in the deliverables. A description of the condition and temperature of the samples upon laboratory receipt (*i.e.*, custody seal condition, container status) must be provided for each Chain-of-Custody Record/sample cooler.

D) Analytical Results Summaries, grouped by fraction, and submitted in the same order of fractions as the Level B Deliverables.

A-9.2 Deliverables Reporting Requirements for GC/MS Volatile and Semivolatile Organic Analyses

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e*., recovery ranges, relative percent difference limits, *etc*.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any recoveries that are outside of the acceptance criteria. The raw data associated with the samples, blanks, and standards must clearly identify the GE sample identifier, the laboratory sample number, the instrument, the laboratory file number for the analysis, and the peak areas/heights and retention times that correspond to the compounds of interest observed in all analyses reported. If the requirement of a summary form is not applicable to a particular sample, standard, or blank, the requirement should still appear on the form; however, no entry will be necessary on the form for that sample, standard, or blank.

A) 1. An analysis summary of the results for all target compounds for all sample analyses, matrix spike analyses, matrix spike duplicate analyses, laboratory control sample analyses, and method/storage blank analyses must be supplied. The summary must include an entry for each target compound, date(s) and time(s) of analysis, GE sample identification, laboratory sample number, date of sample collection, sample matrix, sample weight, sample percent solids, heated or unheated purge, column type(s), column internal diameter, dilution factor, solid extract volume, solid aliquot volume,

concentration units, and sample results. For semivolatile analyses, date of sample extraction, final extract volume, injection volume, and an indication of whether the GPC cleanup was performed (yes/no) is also required. If positive results below the lowest calibration standard are reported, they must be flagged as estimated ("J") on the analysis summary. "Not-detected" results will be represented by the GE required quantitation limit and a "U" flag. If a compound was detected in a sample as well as in the method blank associated with the sample, the result must be flagged with a "B" on the summary form. Additionally, if a dilution is performed on a sample because a target compound is above the calibration range, then the positive result for the particular compound should be flagged with a "D." If the compound is still above the calibration rage after a dilution is performed on the sample, then the compound should be flagged with an "E."

2. The raw data for the sample analyses, method blank analyses, and storage blank analyses by GC/MS methodologies will include the RICs, mass spectra for all target compounds identified, and quantitation reports for the target compounds and surrogates. The raw data for the matrix spike and matrix spike duplicate analyses will include the RIC and quantitation report for the target compounds. These are required only for Level B Deliverables.

B) A surrogate percent recoveries summary for all of the reported analyses (samples, blanks, *etc*.). The surrogate recovery forms should be segregated by method (*i.e*., high-level solid samples separate from low-level solid samples). The summary form should also include the surrogate recovery limits and the laboratory should flag the compounds that do not meet the recovery limits, on the summary form.

C) A matrix spike/matrix spike duplicate concentration and percent recovery/relative percent difference summary for each matrix spike/matrix spike duplicate pair analyzed. The matrix spike/matrix spike duplicate summary form will indicate the GE identification of the unspiked sample, the MS/MSD sample, the spike concentrations, the matrix, and the concentrations of the compounds present in the unspiked sample and the MS/MSD sample. The summary form should also include the MS/MSD recovery criteria and RPD criterion. The laboratory should flag the compounds that do not meet the criteria. A similar form for the LCS must be included with the deliverables.

D) A method/storage blank summary form for each method/storage blank which identifies the samples associated with each method/storage blank. The date of analysis, time of analysis, file number, and matrix of the method/storage blank must also be reported on the summary form. Storage blanks are only required for volatiles analysis.

E) 1. A GC/MS tuning summary which summarizes the percent abundances for the mass ions of interest and the acceptance criteria for the mass ions. Additionally, the summary must include a list of the sample and QC sample analyses (sample names, file numbers, and dates and times of analysis) associated with the GC/MS tune. The summary should indicate the instrument identification, date and time of analysis, column type, diameter of the column, and the type of purge (heated or unheated for volatiles) used to analyze the samples.

2. The raw data for the GC/MS tuning summary, consisting of a summary of the mass ion abundances and a mass spectral representation of the tuning peak.

F) 1. For the internal standard calibration method, an initial calibration summary for each initial calibration performed, summarizing all of the relative response factors for each calibration standard, the average relative response factor, and the relative standard deviation among the relative response factors. If a calibration curve equation is utilized, the laboratory must summarize the curve equation and the coefficient of determination. Additionally, the summary should indicate the compounds that must meet a minimum relative response factor or a maximum relative standard deviation criterion and the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the file identifications of the analyses, the dates and times of calibration commencement and completion, column type, diameter of the column, and the type of purge (heated or unheated for volatiles) used to analyze the samples.

2. The raw data for the initial calibration, consisting of the reconstructed ion chromatogram and the raw quantitation report for each calibration standard. This is a requirement for the Level B Deliverables only.

G) 1. For the internal standard calibration method, a continuing calibration summary for each continuing calibration standard analyzed, summarizing the average relative response factors of the initial calibration associated with the continuing calibration standard, the relative response factors of the continuing calibration standard, and the percent differences between the

average relative response factors of the initial calibration and the relative response factors of the continuing calibration. If calibration curve equations are utilized the laboratory must summarize the true concentration, observed concentration, and the percent drift. Additionally, the summary must indicate the compounds that are subject to a minimum relative response factor criterion, the compounds that are subject to a maximum percent difference criterion, and the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the date of the initial calibration, the date and time of analysis, column type, diameter of the column, and the type of purge (heated or unheated for volatiles) used to analyze the samples.

2. The raw data for the continuing calibration, consisting of the reconstructed ion chromatogram and the raw quantitation report for each calibration standard. This is a requirement only for the Level B Deliverables.

H) An internal standard area counts summary, containing a summary of the area counts and retention times for the internal standards for a continuing calibration. The summary must indicate the acceptance windows for the internal standard retention times and area counts. This summary must supply a comparison of the continuing calibration internal standards to the mid-level initial calibration internal standards. Additionally, the summary must include a listing of the internal standard retention times and area counts for all of the samples, method blanks, matrix spikes, and matrix spike duplicates associated with the continuing calibration standard.

I) A copy of all of the extraction log information for semivolatiles is required. At a minimum, the extraction information must include the date the extraction was

started, the date the extraction was completed, the initial sample weight or volume, final extraction volume, laboratory sample number, the amount and concentration of surrogate spike added, and the amount and concentration of matrix spike solution added. Additionally, the extraction log should indicate if a cleanup procedure was performed on the sample. If a medium-level extraction was performed for the volatiles analysis, all extraction logs for this analysis will be required. For volatile organics analyses that require weighing sample aliquots in the field, copies of the field measurement documentation will be included in this section.

A-9.3 Deliverables Reporting Requirements for Organochlorine Pesticide, PCB, and Herbicide Analysis

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (i.e., recovery ranges, relative percent difference limits, etc.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any recoveries that are outside of the acceptance criteria. The raw data associated with the samples, blanks, and standards must clearly identify the GE sample identification, the laboratory sample number, the instrument, the laboratory file number for the analysis, and the peak areas/heights and retention times that correspond to the compounds of interest observed in all analyses reported. If the requirement of a summary form is not applicable to a particular sample, standard or blank, the requirement should still appear on the form; however, no entry will be necessary on the form for that requirement.

A) 1. An analysis summary of the concentrations of all target compounds for all sample analyses, matrix spike analyses, matrix spike duplicate analyses, and blank analyses. The blank analyses must consist of all of the extraction

(method) blank analyses, injection blank analyses, and any blanks associated with cleanup procedures. The summary must include dates and times of analysis, GE sample identifications, laboratory sample numbers, dates of sample collection, date of sample receipt, dates of sample extraction, sample matrices, sample weights or volumes, sample percent solids, column types, column internal diameters, dilution factors, initial extract volumes/weights, final extract volumes, concentration units, the type of cleanup performed, and sample results. If positive results below the lowest calibration standard are reported, they must be flagged as estimated ("J") on the analysis summary. "Not-detected" results will be represented by the GE required quantitation limit and a "U" flag. If a compound was detected in a sample as well as in the method blank associated with the sample, the result must be flagged with a "B" on the summary form. Additionally, if a dilution is performed on a sample because a target compound is above the calibration range then the positive result for the particular compound should be flagged with a "D." If the compound is still above the calibration range after a dilution is performed on the sample, then the positive result for the compound should be flagged with an "E."

2. The raw data for the sample analyses, matrix spike analyses, matrix spike duplicate analyses, and blank analyses, consisting of the chromatograms indicating the surrogate peaks and target compound peaks and quantitation reports for the target compounds and surrogates. This is a requirement only for the Level B Deliverables.

B) A surrogate percent recovery summary for all of the reported analyses (samples, blanks, *etc*.). The surrogate recovery forms should be segregated by matrix and

method (*i.e.*, medium-level solid samples separate from low-level solid samples). The summary form should also include the surrogate recovery limits and the laboratory should flag the compounds that do not meet the recovery limits on the summary form.

C) A matrix spike/matrix spike duplicate concentration and percent recovery/relative percent difference summary for each matrix spike/matrix spike duplicate pair analyzed. The matrix spike/matrix spike duplicate summary form will indicate the GE identification of the unspiked sample, the MS/MSD sample, the spike concentrations, the matrix, and the concentrations of the compounds present in the unspiked sample and the MS/MSD sample. The summary form should also include the MS/MSD recovery criteria and RPD criterion. The laboratory should flag the compounds that do not meet the criteria. A similar form for the LCS should be included with the deliverables.

D) A method blank summary form for each method blank, identifying the samples associated with each method blank. The date, time, lab file number, and matrix of the method blank must also be reported on the summary form.

E) Initial Calibration Data: A summary of the initial calibration retention times, mean retention time, and a retention time window for all target compounds and surrogates must be provided for all initial calibrations. A second summary of the initial calibration standard calibration factors, average calibration factors, and relative standard deviations for all target compounds and surrogates must also be provided for all initial calibrations. If a calibration curve equations is utilized the laboratory must supply the curve equation and the coefficient of determination. Both summaries should include the SDG number, instrument identification, GC column

type and diameter, date(s) of analysis, the concentration level for each initial calibration standard (as a multiplication factor of the low calibration standard), and the acceptance limit for the relative standard deviation. Copies of the pesticide, herbicide, and PCB standard chromatograms and integration reports associated with summaries should immediately follow the summary (only for the Level B Deliverables). Each initial calibration associated with the SDG must be presented in chronological order, by GC column and by instrument.

F) Continuing Calibration Data: A summary of the observed retention times, calculated compound concentrations, true concentrations, percent differences, and retention time window from the initial calibration (or from the daily retention time window update) must be provided for all continuing calibration standards. If calibration curve equations are utilized the laboratory must summarize the true concentration, observed concentration, and the percent drift. The summary should list the SDG number, GC column type and diameter, date and time of analysis, laboratory sample number, initial calibration dates, and acceptance limits. Copies of the pesticide, herbicide, and PCB standard chromatograms and integration reports associated with summaries should immediately follow the summary (only for the Level B Deliverable). Each continuing calibration associated with an SDG must be presented in chronological order, by GC column and by instrument.

G) 4,4′-DDT and Endrin Breakdown Data (organochlorine pesticides only): A summary of the observed 4,4′-DDT, endrin, and combined percent breakdowns must be presented for each breakdown check performed. (Alternatively, if this data is obtained from a continuing calibration standard rather than a specific breakdown standard, this information may be reported on the associated continuing calibration

summary form.) The summary should list the SDG number, GC column type and diameter, date and time of analysis, laboratory sample number, initial calibration dates, and acceptance limits. Copies of the pesticide/PCB standard chromatograms and integration reports associated with summaries should immediately follow the summary (only for the Level B Deliverables). Each breakdown summary associated with an SDG must be presented in chronological order, by GC column and by instrument.

H) A summary of the analytical sequence for each column and instrument used for the analysis of the project samples. The summary must contain the GC column number, the internal diameter of the column, initial calibration dates associated with the sequence, the instrument identification, the mean retention time(s) for the surrogate(s) utilized, a listing of the GE sample names, the laboratory sample numbers, dates and times of analysis, and the retention times for the surrogate(s). The summary should also indicate the retention time window for all surrogates used and any surrogate retention times that do not meet the acceptance criterion. The summary must contain all of the analyses for the samples, blanks, initial calibration standards, and continuing calibration standards associated with the sequence. All sequences will begin with an initial calibration and will terminate with a continuing calibration or breakdown check standard that meets all acceptance criteria.

I) When a GPC cleanup procedure is required for the samples, a summary for each check standard associated with the GPC calibration. The summary must contain the GPC column identification, the calibration date of the GPC column, the GC column(s) used for the analysis of the standard, the GC column internal diameter, the theoretical concentrations of the compounds in the GPC standard, the observed concentrations of the GPC standard, the percent recovery for each compound in the

GPC standard, the GE sample identification, laboratory sample number, and the date(s) of analysis for all samples associated with the GPC standard. The limits for each compound in the GPC standard should be listed on the summary form. The laboratory should flag any compound if the percent recovery was not within the control limits.

J) When a Florisil® cartridge cleanup procedure is required for the samples, a summary for each check standard associated with a Florisil® cartridge lot. The summary must contain the Florisil® cartridge lot number, the date of analysis of the Florisil® cartridge check standard, the GC column(s) used for the analysis of the standard, the GC column internal diameter(s), the theoretical concentrations of the compounds in the Florisil® cartridge check standard, the observed concentrations of the Florisil® cartridge check standard, the percent recovery for each compound in the Florisil® cartridge check standard, the GE sample identifications, the laboratory sample number, and the date(s) of analysis for all samples in the data deliverable associated with each lot of Florisil® cartridges.

K) Second column confirmation may be performed for all pesticide, PCB, and herbicide analyses when there is a positive result reported for a project sample. When the laboratory performs a dual column quantitative analysis for organochlorine pesticides, PCBs, and herbicides, a summary of the identified compounds and observed concentrations for the two columns utilized for sample analyses is required. The summary must contain the GE sample identification, the laboratory sample number, the dates and times of analysis, the instruments used for analysis, the GC columns, the GC column internal diameters, the retention time windows for each peak used to quantitate the compound, the observed retention time for each

peak used to quantitate the compound, the calculated concentration for each peak used, the mean concentration for each column for each compound identified, and the percent difference between the mean concentrations calculated for each column.

If the percent difference between the results for the analyte from the two GC columns is greater than 40% for the analysis, then the higher of the two values is reported and flagged with a "P." Finally, the "C" flag is used when the identification of a pesticide result is confirmed by GC/MS.

A-9.4 Deliverables Reporting Requirements for Dioxin/Furan Analyses

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e*., recovery ranges, relative percent difference limits, *etc*.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any recoveries that are outside of the acceptance criteria. The raw data associated with the samples, blanks, and standards must clearly identify the GE sample identifier, the laboratory sample number, the instrument, the laboratory file number for the analysis, and the peak areas/heights and retention times that correspond to the compounds of interest observed in all analyses reported. The raw data must provide all information necessary to reproduce all reported positive and EDL results. If the requirement of a summary form is not applicable to a particular sample, standard, or blank, the requirement should still appear on the form; however, no entry will be necessary on the form for that requirement.

A) 1. An analysis summary of the results for all target compounds for all sample analyses, second column confirmation analyses, matrix spike analyses, ORP

standard analyses, and method blank analyses must be supplied. The summary must include an entry for each target 2,3,7,8-substituted compound and total homologue concentrations, date(s) and time(s) of analysis, GE sample identification, laboratory sample number, date of sample collection, date of sample preparation, sample matrix, sample weight, sample percent solids, column type(s), column internal diameter(s), dilution factor, concentrated extract volume, concentration units, peak retention times, isotope ratios, and sample results. If positive results below the lowest calibration standard are reported, they must be flagged as estimated ("J") on the analysis summary. "Not-detected" results will be represented by the EDL and a "U" flag. If a compound was detected in a sample as well as in the method blank associated with the sample, the result must be flagged with a "B" on the summary form. Additionally, if a dilution is performed on a sample because a target compound is above the calibration range, then the positive result for the particular compound should be flagged with a "D". If the compound is still above the calibration rage after a dilution is performed on the sample, the positive result for the compound should be flagged with an "E".

2. The raw data for the sample analyses and method blank analyses by GC/MS methodologies, consisting of the EICP, quantitation reports for the target compounds, the associated areas or height for each peak within the established retention time window, and all other information required to reproduce all reported positive and EDL results. The raw data for the matrix spike and matrix spike duplicate analyses will include the EICP chromatogram and quantitation report for the target compounds.

B) A matrix spike concentration and percent recovery summary for each matrix spike analyzed is required. The matrix spike summary form will indicate the GE identification of the unspiked sample, the sample, the matrix, and the concentrations of the compounds present in the unspiked and spiked sample. The summary form should also include the MS recovery criteria. The laboratory should mark the compounds that do not meet the specified criteria. A similar form for the OPR standard should be included with the deliverables.

C) A method blank summary form for each method blank that identifies the samples associated with each method blank. The date of extraction, date of analysis, time of analysis, lab file number, sample weight, and matrix of the method blank must also be reported on the summary form.

D) A mass spectrometer performance summary for each mass spectrometer performance standard analyzed should identify the sample number, lab file identification, date and time of analysis, instrument identification, GC column identification, and static resolving power.

E) A window defining mix summary form for each window defining analysis should identify the sample number, lab file identification, date and time of analysis, instrument identification, and GC column identification. This form should include the retention time of the first eluting and last eluting isomer for each congener group.

F) An isomer specificity test standard summary should identify the sample number, file number, instrument ID, date and time of analysis, the GC column and instrument identification, and the percent valley determination between $^{13}C_{12}$-2,3,7,8-TCDD and $^{13}C_{12}$-1,2,3,4-TCDD. In addition, if second column confirmation is required,

percent valley for 2,3,7,8-TCDD and the closest isomers should be calculated and reported.

G) A summary of the analytical sequence for each column and instrument used for the analysis of the project samples. The summary must contain the GC column number, the internal diameter of the column, initial calibration dates associated with the sequence, the instrument identification, a listing of the GE sample names, the laboratory sample numbers, and dates and times of analysis. The summary must contain all of the analyses for the samples, blanks, initial calibration standards, and the continuing calibration standards associated with the sequence.

H) 1. An initial calibration summary for each initial calibration performed, summarizing all of the relative response factors for each calibration standard, the average relative response factor, and the relative standard deviation among the relative response factors. If calibration curve equations are utilized, the laboratory must supply the curve equation and coefficient of determination. Additionally, the summary should indicate maximum relative standard deviation and minimum relative response factor criteria as well as the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the dates and times of calibration commencement and completion, column type, and diameter of the column.

2. The raw data for the initial calibration, consisting of the EICPs and the raw quantitation report for each calibration standard.

I) 1. A continuing calibration summary for each continuing calibration standard analyzed, summarizing the average relative response factors of the initial calibration associated with the continuing calibration standard, the relative response factors of the continuing calibration standard, and the percent differences between the average relative response factors of the initial calibration and the relative response factors of the continuing calibration, and the isotope ratios and retention times. If calibration curve equations are utilized the laboratory must summarize the true concentration, observed concentration, and the percent drift. Additionally, the summary must indicate the compounds that are subject to a minimum relative response factor criterion, the compounds that are subject to a maximum percent difference criterion, and the compounds that did not meet the acceptance criteria. The summary should indicate the instrument identification, the date of the initial calibration, the date and time of analysis, column type, and diameter of the column.

2. The raw data for the continuing calibration, consisting of the EICPs and the raw quantitation report for each calibration standard.

A-9.5 Deliverables Reporting Requirements for Inorganic Analyses

The laboratory will be required to submit the following information as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e.,* recovery ranges, relative percent difference limits, *etc*.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any quality control results that are outside the acceptance criteria. All instrument raw data printouts for the points discussed below must be provided in an orderly

fashion. This applies to all required QA/QC measurements, and instrument standardization, as well as sample analysis results. Additionally, all associated extraction, digestion, and distillation logs must be supplied. The order of the raw data in the data package shall be ICP-AES, ICP/MS, flame AA (if performed), furnace AA (if performed), and mercury. All flame and furnace AA data shall be grouped by element. All raw data shall be grouped by analysis date for all analytical results.

A) 1. A sample reference list for all samples present in an SDG. This reference list must summarize and correlate the laboratory sample number, the GE designated sample identification, and any laboratory code (*i.e.*, truncation of GE designated sample number by the laboratory) for each sample in an SDG.

2. A Table of Contents listing page numbers associated with information such as:

a. Methodology Summary

b. Case Narrative

c. Sample Results

d. Quality Control Data

e. Verification of Instrument Parameters

f. Preparation and Analysis Logs

g. Raw Data, including but not limited to:

i. ICP-AES, ICP/MS, Flame AA, GFAA, and Mercury Data

ii. Digestion Logs

iii. Confirmation Data

h. Chain-of-Custody Records

B) Analysis summaries of the concentrations of all target analytes for all sample analyses. The summary must include the GE designated sample number, the laboratory sample number, date of sample collection, date of sample receipt, sample matrix, sample percent solids, concentration units, sample results, data qualifier codes, analysis method codes, description of sample before and after analysis, and any comments relating to the sample.

C) A summary of the initial and continuing calibration verifications for each calibration performed. This summary will include the concentrations observed as well as the true value of the analyte in the initial and continuing calibrations. A percent recovery will be summarized based on the observed and true values for each analyte.

D) A summary of the Detection Limit (DL) standard analyses for both Atomic Absorption (AA) and Inductively Coupled Plasma (ICP) analyses. This summary will include the concentrations observed as well as the true value of the analyte in the DL standard. A percent recovery will be summarized based on the observed and true values for each analyte.

E) A summary of the initial and continuing laboratory blank analyses for each calibration performed. This summary will include the concentrations (positive or negative) observed of any analyte in the initial and continuing blank analyses at values greater than the MDL. The summary should also include the concentrations of any analyte observed in the laboratory preparation blank associated with each calibration sequence performed by the laboratory.

F) A summary of the ICP interference check sample analysis for each analytical sequence performed. This form will summarize the true and found values (positive, negative, or zero) of all analytes present in Solutions A and AB of the ICP interference check sample analysis. This form will also summarize the percent recoveries of the analytes/interferences present in the standards.

G) A summary of the pre-digestion matrix spike analysis. This form will summarize the percent recovery control limit for each analyte. Also, the sample result, the spike sample result, and the spike-added amount must be summarized on this form for all parameters analyzed. The laboratory-calculated percent recovery as well as the laboratory qualifier stating whether the calculated percent recovery was within control limits must also be summarized on this form.

H) A summary of the post-digestion matrix spike analysis. This form will require the same information described in item G.

I) A summary of the laboratory duplicate analysis. This form will summarize the percent differences observed between the sample and laboratory duplicate analyses. The appropriate control limits must be specified by the laboratory, and a summary of

the sample and laboratory duplicate analyses must be provided. The percent solids for the sample and the duplicate sample should be included on the summary form.

J) A summary of the Laboratory Control Sample (LCS) analysis. This form will summarize the percent recovery, control limits, and true and found values for the solid sample analyses.

K) A summary of any required Method of Standard Additions (MSA) determinations. This form will summarize the concentrations and absorbencies of all samples and analytes that require analysis by MSA. The correlation coefficient for the MSA analysis will be calculated and summarized on this form. Also, the sample concentration determined from the MSA determination will be summarized on this form.

L) A summary of the ICP Serial Dilution analyses performed by the laboratory. This summary will show the result of the initial sample analysis (in aqueous units, as observed from the raw data), the result of the five-fold serial dilution analysis, and the percent difference between the two analyses.

M) The summaries necessary for the verification of instrument parameters. These include an Method Detection Limit and Reporting Limit Summary, an ICP Interelement Correction Factor Summary (if performed) for each ICP used for analysis, and an ICP Linear Range Summary (if performed) for each ICP used for analysis.

N) The analysis log summaries. These include a Sample Preparation log that provides the sample identification; the preparation date; the sample weight (in grams) used;

and the digestion volume (in mL) used and an Analysis Run Log that provides the instrument identification, the sample identification, any dilution factors employed in the analysis, the date and time of analysis, the method of analysis, and the parameters analyzed. Additionally, the GFAA post-digestion analytical spike sample recoveries are listed on the Analysis Run Log.

A-9.6 Deliverables Reporting Requirements for Wet Chemistry/Conventionals Analysis

The laboratory will be required to submit the information detailed in Sections A-9.5 A) -C), A-9.5, E) and A-9.5, G) - J) and A-9.5-N as support documentation for the reported analytical results. The quality control summary forms must include the acceptance criteria (*i.e*., recovery ranges, relative percent difference limits, *etc*.) and spike-added amounts (where applicable). Additionally, the quality control summary forms must indicate any quality control results that are outside the acceptance criteria. All instrument raw data printouts for the points discussed in the above mentioned sections must be provided in an orderly fashion. This applies to all required QA/QC measurements, and instrument standardization, as well as sample analysis results. Additionally, a direct sequential readout must be included if the instrument has the capability.