OBJECTIVE

This Standard Operating Procedure (SOP) describes the requirements for the analytical data packages that will be generated in association with the Stage 2 Delaware River Estuary PCB Total Maximum Daily Loads (TMDL) Project. This SOP applies to the contractor(s) involved in analytical data generation and reporting. Two hard copy data packages as well as an Adobe Acrobat (.PDF) file format must be generated for the Stage 2 Delaware River Estuary PCB TMDL. The laboratory is required to maintain copies of the .pdf and hard copy data packages for 5 years from the time of submission.

DATA PACKAGE DELIVERABLES

The following sections describe in detail the types of data packages designed for the Stage 2 Delaware River Estuary PCB TMDL project. 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 package deliverables are divided into two sections; Section 1.1 and 1.2. These Sections have been developed based on deliverables specified in the US EPA Contract Laboratory Program Statement of Work (CLP SOW). Section 1.1 specifies the data package contents and order of presentation. Section 1.2 provides details concerning specific contents of the data deliverables described in Section 1.1.

The fully documented data package described in this SOP resembles the information required by the CLP SOW. This type of package includes a cover letter, SDG narrative, field Chain-of-Custody Records, analytical results summaries, a glossary of qualifier codes, summary forms for quality control procedures and all sample and quality control raw data to support the results reported.

1.1 Data Package Contents and Order of Presentation

The laboratory must submit supporting documentation for the reported analytical results. Furthermore, the data package deliverables must be submitted in the order in which the deliverables appear in the text.

The Sample Data Package shall include data for analyses of all samples in one SDG, including field samples, second column analyses, re-extractions, reanalyses, secondary dilutions, blanks, ongoing precision and recovery (OPR) standards, matrix spikes, matrix spike duplicates, and/or laboratory duplicates. The complete Sample Data Package is divided into the units as described below. The Sample Data Package must be complete before submission and must be 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 (or indexing system such as tabs or bookmarks and links as in a .pdf file)

D) Sample Delivery Group (SDG) Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name; SDG number; field 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.

The SDG Narrative must be signed and dated by the laboratory manager, project manager or the chemist that reviewed and approved the release of data.

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

Copies of the field 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 Polychlorinated Biphenyl (PCB) Data

1. Quality Control (QC) Summary

   a. Duplicate Precision Summary (if requested).

   b. Ongoing Precision and Recovery (OPR) 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 PCB samples. These sample

packets should then be placed in increasing alphanumeric order by sample identification. The order of each sample packet is as follows:

a. Analytical Results Summary.

For each sample, including compound/peak relative retention times (RRTs), peak co-elution information, ion abundance ratios, reported concentrations, laboratory qualifiers, Estimated Detection Limits (EDLs), internal standard (both extraction and injection) recoveries, and clean-up standard recoveries.

b. Quantitation Report-must be submitted and include all information required to reproduce reported positive results and EDL results.

c. Selected Ion Current Profile (SICP) Chromatograms. (SICP are to be provided in pdf format only. They are not required to be submitted as hardcopies.)

d. Second Column Data (if necessary; will include 1.1.1.F, Section 2, items a, b, and c).

e. Work sheets which include example calculations showing how sample results were calculated using initial calibration and sample responses for at least one sample per data package. The calculations should cover both positive results and EDLs

3. Standards Data

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

b. Multi-point Initial Calibration by Isotope Dilution Data for the toxics/LOC CBs (Initial Calibration Summary Form, quantitation report, and SICP Chromatograms) for each initial calibration associated with the SDG, in chronological order by GC column, by instrument. Note, for the labeled compounds, internal standard calibration is performed using the multi-point initial calibration data. If a curve equation is utilized, the laboratory must provide the curve equation and coefficient of determination.

c. Single-Point Initial Calibration by Internal Standard Data for the native CBs for which a labeled compound is not available (Initial Calibration Summary Form, quantitation report, and SICP Chromatograms) for each initial calibration associated with the SDG, in chronological order by GC column, by instrument.

d. Calibration Verification Data (Calibration Verification Summary Form, quantitation report, and SICP Chromatograms) for each calibration verification associated with the SDG, in chronological order, by GC column, by instrument.

4. Raw QC Data

For the blank, OPR standard, and Laboratory Duplicate provide in chronological order, by instrument:

i. Analytical Results Summary.

Compound/peak RRTs, peak coelution information, ion abundance ratios, reported concentrations, laboratory qualifiers, EDLs, internal standard (both extraction and injection) recoveries, and clean-up standard recoveries. Also provide any imbedded and/or associated calculations or comments imbedded on the chromatograms.

ii. Quantitation Report.

iii. SICP Chromatograms.

5. GC/MS Instrument Run Logs.

6. Extract Clean-up Data (if available)

a. 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. SICP Chromatograms and quantitation reports for the GPC calibration check solution and all standards used to quantify compounds in the GPC calibration check solution.

G) Preparation Logs

1. PCB Extraction and Clean-up Logs.

1.2 Deliverables Reporting Requirements for PCB Analyses

The laboratory will be required to submit the following information as support documentation for the reported analytical results. All documentation shall be provided in hard copy format and as an Adobe Acrobat ..pdf file with the exception of the chromatograms, which shall be provided only in the .pdf format. 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 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 analyses, re-extractions/reanalyses, secondary dilutions, matrix spike analyses, matrix spike duplicate analyses, laboratory duplicate analyses, OPR standard analyses, and method blank analyses must be supplied. The summary must include an entry for each congener concentration, date(s) and time(s) of analysis, 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 relative retention times, co-elution information, 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". A description of qualifier flags for this DRBC project is provided in the description of Data Qualifiers.

2. The raw data for the field sample, second column, re-extraction/reanalysis, secondary dilution, blank, OPR standard, matrix spike, matrix spike duplicate, and/or laboratory duplicate analyses by GC/MS methodologies, consisting of the SICP, 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.

B) A OPR and percent recovery summary for each OPR analyzed is required. The OPR summary form will indicate the concentrations of the compounds present in the spiked sample. The summary form should also include the OPR recovery criteria. The laboratory should mark the compounds that do not meet the specified criteria.

C) A relative percent difference summary for each laboratory duplicate analyzed is required. The laboratory duplicate summary form will indicate the field identification of the parent sample, the sample, the matrix, and the concentrations of the compounds present in the parent and duplicate sample. The summary form should also include the RPD criteria. The laboratory should mark the compounds that do not meet the specified criteria.

D) 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.

E) Mass spectrometer resolution data for the reference standard (PFK or other substance) analyzed to demonstrate mass resolution. Output for each descriptor should identify the lab file identification, date and time of analysis, instrument identification, and exact mass ions monitored.

F) 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 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.

G) 1. An initial calibration summary for each multi-point 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 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. An initial calibration summary for each single-point initial calibration performed, summarizing all of the relative response factors for each compound. The summary should indicate the instrument identification, the dates and times of calibration commencement and completion, column type, and diameter of the column.

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

H) 1. A calibration verification summary for each calibration verification standard analyzed, summarizing the true and found concentrations, and the percent recoveries and the relative response factors of the calibration verification, and the isotope ratios and retention times. Additionally, the summary must indicate the compounds that are subject to recovery criteria, 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 calibration verification, consisting of the SICPs and the raw quantitation report for each calibration standard.