

Washington State Department of Ecology

Environmental Assessment Program

**Standard Operating Procedure for Semipermeable Membrane Devices (SPMD) Data Management and Data Reduction.**

**Version 2.0**

Authors - Keith Seiders, Patti Sandvik  
Date - 02/06/2013

Recertification Review – Will Hobbs  
Date -

Unit Approval Debby Sargeant, Toxic Studies Unit Supervisor  
Date

QA Approval - William R. Kammin, Ecology Quality Assurance Officer  
Date

EAP 079

APPROVED: February 11, 2013  
RECERTIFIED: 11/21/2016

Signatures on File

*Please note that the Washington State Department of Ecology's Standard Operating Procedures (SOPs) are adapted from published methods, or developed by in-house technical and administrative experts. Their primary purpose is for internal Ecology use, although sampling and administrative SOPs may have a wider utility. Our SOPs do not supplant official published methods. Distribution of these SOPs does not constitute an endorsement of a particular procedure or method.*

*Any reference to specific equipment, manufacturer, or supplies is for descriptive purposes only and does not constitute an endorsement of a particular product or service by the author or by the Department of Ecology.*

*Although Ecology follows the SOP in most instances, there may be instances in which Ecology uses an alternative methodology, procedure, or process.*

SOP Revision History

Revision Date	Rev number	Summary of changes	Sections	Reviser(s)
12/20/12	1.0	New SOP	all	
8/22/16	2.0	- revised to incorporate contract lab ability to do dialysis - updated location of data repository and Appendix files	all	William O. Hobbs
9/9/2016	2.0	Recertified	all	Bill Kammin
11/21/2016	2.0	Cover page, footer		Bill Kammin

## **TABLE OF CONTENTS**

1.0 Purpose and Scope .....	5
2.0 Applicability .....	5
3.0 Definitions .....	6
4.0 Personnel Qualifications/Responsibilities .....	6
5.0 Equipment and Supplies .....	6
6.0 Summary of Data Reduction Procedure .....	6
6.1 Overview.....	6
6.2 Perform Data Reduction Tasks.....	7
7.0 Records Management .....	14
8.0 Quality Control and Quality Assurance Section.....	15
8.1 Quality Control and Quality Assurance for this SOP consists of a review, or audit, of the SPMD project. ....	15
8.2 Person conducting the audit is assigned by management staff.....	15
8.3 Audit of Data Repository.....	15
8.4 Audit of Data Reduction.....	15
8.5 Report of Audit Findings.....	16
8.6 Audit Follow-up.....	16
9.0 Safety .....	16

## **LIST OF TABLES**

Table 1. Index of Records for Conducting SPMD Projects.....	17
Table 2. Structure of Repository for SPMD Study Data. ....	18
Table 3. Data Reduction Tasks for SPMD Data.....	19

## **1.0 Purpose and Scope**

- 1.1 This document is the Environmental Assessment Program (EAP) Standard Operating Procedure (SOP) for the data management and data reduction tasks for studies using semi-permeable membrane devices (SPMDs) to monitor hydrophobic organic compounds in surface water. SPMDs are described in the parent document “SOP for Conducting Studies Using SPMDs.”
- 1.2 The goal of this SOP is to help staff use and document appropriate data management and data reduction practices when conducting SPMD studies such as: methods, assumptions, decisions, calculations, and reporting. This SOP defines practices in three areas:
  - 1.2.1 Data Reduction: specific steps, tasks, and documentation formats are provided. Described in Section 6 – Summary of Procedure.
  - 1.2.2 Data Management: examples, templates, and a data management structure are provided. Addressed in Section 7 – Recordkeeping
  - 1.2.3 Quality Control: the audit process for the project’s data reduction and data management phases. Described in Section 8 – Quality Control and Quality Assurance.
- 1.3 The analysis and interpretation of SPMD data has used approaches based on various interpretations of early and developing guidance by USGS and others. Due to lack of standard procedures, documentation, and quality control practices, results from SPMDs are challenging to compare to other SPMD studies or to environmental levels of concern. SPMD technology is still considered “experimental” or in research phase: EPA or other organizations have not yet deemed SPMD methodology mature or robust enough to be a “standard” method. While SPMD results are often qualified as “estimates”, the qualification often is disregarded in the use and application of the data, even though SPMD guidance cautions users about use of the results.

## **2.0 Applicability**

- 2.1 This SOP, along with the parent document “SOP for Conducting Studies Using SPMDs” are to be followed whenever Ecology or Ecology-funded studies use SPMDs. Careful planning and documentation are needed in all phases of using SPMDs in order for data to meet project objectives.
- 2.2 The use of SPMDs requires the Project Officers (PO) to coordinate the services of multiple labs for project preparation, QA/QC, and review of results. Project officers also need to conduct a variety of QC procedures that have traditionally been performed within analytical labs. Finally, project officers need to manage large amounts of information in order to analyze, document, model, and report results.

### **3.0 Definitions**

3.1 Refer to the parent document “SOP for Conducting Studies Using SPMDs.”

### **4.0 Personnel Qualifications/Responsibilities**

4.1 Personnel leading or auditing projects that use SPMDs should have prior experience as project managers of studies involving organic contaminants and have a job classification equivalent to an Environmental Specialist 3 or higher. Those using this SOP should be familiar with the parent document “SOP for Conducting Studies Using SPMDs.”

4.2 Personnel conducting data management and data reduction tasks related to SPMDs should be adequately supervised by project leads to ensure appropriate use and management of data.

### **5.0 Equipment and Supplies**

5.1 Equipment for the data management, data reduction, and auditing tasks associated with SPMD projects is limited to the project’s hardcopy and electronic files, including this SOP, the parent SOP, and other related documents. The PO manages all records and makes files available as needed by others.

5.2 Tables 1 and 2 which list the data management and data reduction files are to be used as checklists to guide and document actions taken with project data. These are located at: Y:\SHARED Files\SPMDs\SOP Info\SOP v4 revision 2016.

### **6.0 Summary of Data Reduction Procedure**

6.1 Overview

6.1.1 The Data Reduction process is outlined in a step by step process shown in the section below. The process is also in a worksheet format as Appendix RR-14, located at: Y:\SHARED Files\SPMD\SOP Info\ SOP Data Redcn & Repositry templates v2 2016.xlsx. Table 3 in this document also shows this table.

6.1.2 The Data Reduction spreadsheet is to be used as a guide, checklist, and place to document data reduction actions: it is not an instructional guide for the use of SPMDs.

6.1.3 Appendix RR-14 is organized into Phases, Step #s, Tasks, and Notes. Each “Phase” is a major task or action which is further divided into discreet “Steps”. Each Step describes a specific SPMD Processing Task. Each SPMD Processing Task has an associated “PO Task” which describes the action to be taken such as compilation of data, calculation, a QC check, and documentation. Fields for Notes are included in the spreadsheet to allow a place to record the date a step was completed and comment on each step as needed. Similar fields allow these notes to be made by the Project Auditor. A final field shows the relevant Appendix or Record associated with each step.

6.1.4 There are likely to be questions, additions, and changes as the data reduction guide is used. Take these issues to the attention of the authors of this SOP or Toxics TCT SPMD Workgroup so they can be addressed and help improve the process. Any changes should be noted so that they can be included in the next revision of the SOP.

6.2 Perform Data Reduction Tasks

6.2.1 Follow the steps below when processing SPMD data. Insert appropriate dates and notes as each step is completed in a copy of Appendix RR-14. Additional steps, worksheets, or documents can be added or referenced, as necessary: the goal being to document all issues that were encountered and how they were addressed. Start with Phase A tasks (Pre-Field Membrane Mfg and Preparation) and proceed to the end of Phase M (Estimate Whole Water Concentrations from Model Outputs and TOC Data). Some steps are completed and documented earlier in the project, such as Phase steps in arranging the correct spiking solutions and shipment of SPMDs before deployment.

6.2.1.1 Phase A. EST Does Pre-Field Membrane Mfg and Preparation

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	EST fabricates SPMDs	get QC data for batches of project membranes/lipids from EST; USGS CERC does analyses
2	MEL or contract lab (CL) prepares spiking solutions: surrogates, matrix spike	document chemicals, conc, vol, which membranes
3	PO starts process to get spiking needs met	follow process to get spiking needs met
4	MEL, CL, or EST prepares spiking solution: PRCs	document chemicals, conc, vol, which membranes
5	Each lab prepared spike sol'n for potential analysis	to be sent along with other QC samples
6	CL prepares spike solutions: EIS and OPR for isotopic dilution method	document
7	EST prepares Blank samples: Field, Day-0, reagent, other	determine #, type, where to use, and analyze or hold
8	EST ships SPMDs to Ecology and QC SPMDs to the CL or MEL	check shipping method, security, and condition of SPMDs

6.2.1.2 Phase B. PO Coordinates Field Deployment, Midcheck, and Retrieval.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	Deploy, Midcheck, Retrieve SPMDs	check completeness of Field Logs, remedy as needed
2	check security and condition of SPMDs	note in Field Logs
3	transcribe field notes to various spreadsheets	check for transcription errors, remedy as needed
4	Ecology ships SPMDs to CL or EST	check shipping method, security, and condition of SPMDs

6.2.1.3 Phase C. PO Checks Tidbit Records.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	download and review Tidbit data:	
2	DO THIS BEFORE EST processes samples;	decide whether to process compromised samples
3	review Tidbit data, determine submergence for sample period	document
4	if submergence interrupted, accept or reject sample	document
5	compare Tidbit data with mean temp measured in field (Field Notes)	document

6.2.1.4 Phase D. EST or contract lab Does Post-Field Processing, Spiking, Extraction, Splitting, Ampulizing.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	PO reviews all spiking info; gives EST or CL permission to process samples	cross-checks all spiking info from all parties for correct spiking; check math
2	cleans SPMDs: samples, QC samples (field and lab)	review Membrane Condition Sheets
3	prepares QC SPMDs per plan (e.g. FrDay0, Spiked Solvent, others)	check Membrane Condition Sheets vs plan
4	spikes sample and QC SPMDs with sol'ns per plan (e.g. EIS, surrogates)	check Membrane Condition Sheets vs plan
5	extraction (dialysis); GPC cleanup, solvent exchanges	check Membrane Condition Sheets vs plan
6	splits extract depending on study needs (e.g. 50:50)	check Membrane Condition Sheets vs plan
7	measure volumes of extracts (request EST, MEL, CL conduct)	check volumes vs assumption of factor to adjusting results by (usually 2x)
8	ampulizes extracts for storage/shipment	check Membrane Condition Sheets vs plan
9	EST ships extracts to MEL	check shipping method, security, and condition of SPMDs

6.2.1.5 Phase E. MEL Conducts Analyses.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	MEL receives extracts	check shipping method, security, and condition of SPMDs
2	MEL splits extracts as needed for in-house analyses	review Case Narrative for proper splitting
3	sample cleanup & analyses	review Case Narrative for appropriate work
4	sample results	review for outliers, spurious values, completeness, other
5	analytical QA/QC results: cal, LCS, surrogates, MS, blanks, etc.	check results vs QC limits in Case Narratives and data tables
6	PO needs to address results of "Membrane Spike": (MEL doesn't treat this as a Matrix Spike)	Report evaluation regarding data usability
7	Case Narrative developed, sent to PO	review Case Narrative
8	Electronic data record developed, sent to PO	review data files vs hardcopy record, resolve differences with MEL

6.2.1.6 Phase F. CL Conducts Analysis.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	CL receives extracts	check shipping method, security, and condition of SPMDs
2	CL may split extract again - project dependent	review Case Narrative for proper splitting
3	PCB congeners: cleanup & analysis	review Case Narrative for appropriate work
4	sample results	review for outliers, spurious values, completeness, other
5	analytical QA/QC results: cal, LCS, surrogates, blanks, etc.	check results vs QC limits in method, Case Narratives
6	Case Narrative developed, sent to PO	review Case Narrative
7	Electronic data record developed, sent to PO	review data files vs hardcopy record, resolve differences with MEL

6.2.1.7 Phase G. PO Reviews all Case Narratives.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	review Case Narratives for following items, where applicable	review for proper methods, limits, problems
2	analytical methods	" " "
3	holding times; preservation	" " "
4	calibration and verification and degradation checks	" " "
5	on-going precision and recovery assessments	" " "
6	method blanks	" " "
7	matrix spike/matrix spike duplicates (MS/MSD) recoveries	" " " (note: MEL may not use these data to evaluate analytical performance)
8	qualitative identification	" " "
9	laboratory control samples	" " "
10	surrogate recoveries	" " " (note: MEL may not use these data to evaluate analytical performance)
11	laboratory replicates	" " "
12	internal standards checks	" " "
13	check history of splitting samples/extracts and ensure calculations are correct	" " "
14	MEL reviews CL data and CL Case Narrative	check that case narrative matches data
15	MEL amends data, creates MEL Case Narrative	check that case narrative matches data

6.2.1.8 Phase H. PO Review data sets.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	review electronic data via LIMS or Excel file from MEL lab manager	check completeness; other checks
2	reviews electronic data via CL/MEL spreadsheet	check completeness, other, using checklist for PCB congeners
3	check how PRCs were handled in datasets (surrogates too if applicable)	check whether summed values include result from PRC or surrogate; adjust as needed to exclude added QC analyte
4	evaluates blank contamination and how CL and MEL handled	see Evaluate Blank Contamination below
5	determine how to use MS/MSD & surrogate data in qualifying batch results	document actions and justification
6	address dataset issues to create final dataset for further review	document reviews and actions taken

6.2.1.9 Phase I. PO Evaluates Blanks for Contamination: Field, Process, and Reagent Blanks.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	compile/organize blank results: Field, Day-0, reagent, others	compile/organize
2	determine precision for blanks (e.g. RSD for Field blanks, RSD for Day0D blanks)	document in spreadsheet
3	evaluate all blanks to locate sources and relevance of contamination	describe and document; tables, charts

6.2.1.10 Phase J. PO Determines LOD and LOQ for Each Result Using Results from Field Blanks; Decides Whether to Censor or to Blank Correct.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	determine LOD for each result and add to residue results table	LOD = Mean of Field Blanks + 3 Std Devs of the Mean. Set NDs = RL, or explain approach.
2	determine LOQ for each result and add to residue results table	LOQ = Mean of Field Blanks + 10 Std Devs of the Mean. Set NDs = RL, or explain approach.
3	Select one of two options for handling residue results: censor or blank correct	Note that qualifiers are for residues only - and need be retained for record-keeping
4	1 - Censor: If the reported residue result "R" > LOQ, use the reported value. If R < LOQ, censor as UJ. The value associated with the UJ qualifier will be the LOD if R < LOD; or R if R is > LOD. Do not blank correct.	document in residue data table
5	2 - Blank Correct. If R > LOD, blank correct thus: R minus the mean of field blanks = blank corrected value. Qualify this value as "B1" which is defined as "Analyte detected in sample and method blank. Reported result is blank-corrected" (definition from EIM reference table). If R < LOD, qualify value as U.	ID and flag results that will be blank-corrected; and those that won't
6	consolidate results and document in residue data table and draft report	

## 6.2.1.11

## Phase K. PO Estimates Dissolved Concentrations Using USGS Model.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	Prepare residue results for model	Residue results are usually normalized to 1 membrane - check/adjust SPMD volume as needed
2	Determine if PRC recoveries are within limits of 20% - 80%	document. PRC-initial is the mean of field blanks or mean of Day0- blanks; PRC-final is from individual sample: document which blanks used
3	Define how to estimate SPMD performance: PRCs or temp/flow	document; if using temp/flow, need to obtain values and document justification
4	Select spreadsheet model to use and follow USGS directions (e.g. Version 4, 4-1, 5, 5-1, or more recent edition).	document version used and enter version number at top of spreadsheet model (cell B3 in version 5-1)
5	Verify spreadsheet model by using proven data in a "test run."	test data set to be developed
6	Insert name of project, Field Station ID, and MEL lab ID into model spreadsheet under Project / Site Name (row 31).	Make sure the name of the spreadsheet tab includes the Field Station ID.
7	Define Kows to use	document in spreadsheet
8	Enter PRC data (if used) and select which ones to use based on model instructions	document decisions to use or not use certain PRCs (often based on Uncertainty Factor or % recovery)
9	Define and enter other model inputs as needed	document
10	Input residue data and run model for LOD, LOQ, and samples	for initial PRC value, use mean of field blanks or mean of Day0D blanks; for final PRC value, use sample value: document which blanks used
11	Create file and compile dissolved water concentrations	
12	add qualifier "J" in spreadsheet next to all dissolved values	add qualifier "B1" for results based on blank-corrected residues

6.2.1.12 Phase L. PO Determines Whether and Where to Use Summed or Individual Results for Analyte Groups (e.g. PCBs, DDTs, PBDEs, Chlordanes).

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	determine whether to use summed or individual results	describe reasons for or against using summing values
2	<u>Recommend use of individual results because USGS spreadsheet designed for individual except has option for T-PCBs. NOTE: T-PCBs Kow is an average.</u>	
3	if summing, sum according to TSU guidance	document
4	PCB congeners	document
5	PCB Aroclors	document
6	DDTs	document
7	Chlordanes	document
8	Endosulfans	document
9	PBDEs	document
10	LPAH	document
11	HPAH	document
12	TPAH	document
13	Others? .....add new rows here and list them	document

6.2.1.13 Phase M. PO Estimates Total or Whole Water Concentrations from Model Output and TOC Data.

<u>Step</u>	<u>SPMD Processing Tasks</u>	<u>PO Tasks: QC, Data Reduction, Documentation</u>
1	Determine inputs for estimating Total concentration from Dissolved	
2	TOC	define how this input was determined
3	Koc (organic carbon-water equilibrium partitioning coefficient)	define how this input was determined
4	Mw (mass of water)	define how this input was determined
5	Create spreadsheet to calc Total from Dissolved	no standard format available yet
6	compile Total concentrations in useful format	no standard format available yet

6.2.1.14 Results for dissolved or whole water fractions now ready to use. Ensure all documentation is complete

## **7.0 Records Management**

### 7.1 Records Description

7.1.1 SPMD projects generate many records and data which must be managed effectively to meet project goals and allow other users access to the information. Table 1 is an index of records and indicates which records are used for project planning and which are used for the data reduction and data repository tasks.

7.1.2 Many of these forms, tables, and records are contained in Excel files as separate sheets. Others may be Word documents or .pdf formats created when scanning hardcopy material, such as EST Membrane Condition Sheets. Table 1 also shows where templates or examples of individual records are located and where these records are to be stored in the data repository. Note that Table 1 in this SOP is the same as Table 1 in the parent document “SOP for Conducting SPMD Studies”.

7.1.3 The use of these records is required in order to standardize the documentation of Ecology’s SPMD projects and help allow comparisons among project results. Descriptions of some records are given in Section 7 of the parent document “SOP for Conducting Studies Using SPMDs”. For others that are not described, their use and function should be self-explanatory as they are viewed. Some flexibility in the use of the templates can be accommodated, especially where modified formats improve the use and efficiency of record-keeping. Other records not yet developed could also be added to the data management process as experience with SPMDs progresses. Questions should be directed to the authors of the SOP or the Toxics TCT SPMD workgroup.

### 7.2 Data Repository

7.2.1 A data repository was created for SPMD projects because Ecology’s Environmental Information Management (EIM) database does not accommodate modeled data and supporting information such as that generated when using SPMDs. All SPMD project-related records in electronic format are kept in a data repository on EAP’s SharePoint site ([http://partnerweb/sites/EAP/passive\\_samplers/default.aspx](http://partnerweb/sites/EAP/passive_samplers/default.aspx)). These files contain all the planning and data-reduction information used during an SPMD project. Table 2 shows the structure of the data repository. Step-by-step instructions for putting SPMD study data into the repository are located in Appendix RR-17.

7.2.2 Most of the required records for SPMD projects are located in Ecology’s shared directory at Y:\SHARED Files\SPMD\SOP Info\ in two Excel files:

7.2.2.1 SOP Plan & Deploy templates Appendix A-N May 2016.xlsx.

7.2.2.2 SOP Data Redcn & Repositry templates v2 2016.xlsx.

7.2.3 Tables 1-2 in this SOP make up a third excel file in the same folder as above:

7.2.3.1 SOP Records and Locations Tbl 1 and 2 May 2016.xlsx.

- 7.2.4 Other documents and records not identified in Table 1 may also be generated during projects, for example: permission to access sampling sites, lists of contact information related to the project, and graphics or photos showing site locations or deployment apparatus. These records should also be retained and added to the Data Repository along with the other project records.
- 7.2.5 Review Section 7 of the parent document “SOP for Conducting Studies Using SPMDs” and manage records as the project proceeds using the templates and examples described above. Recurring or multi-year projects should have data stored in the repository for each calendar year of the project.

## **8.0 Quality Control and Quality Assurance Section.**

- 8.1 The Quality Control and Quality Assurance for this SOP consists of a review, or audit, of the SPMD project. The audit is an independent review of the methods, assumptions, decisions, calculations, documentation, and reporting practices used in the project. The purpose of the audit is to help ensure that the project was conducted using appropriate and defensible practices to support a credible set of data and report product.
- 8.2 The person conducting the audit is assigned by management staff, such as the Unit Supervisor or the QA Officer. The auditor should be a peer experienced with the use of SPMDs. The timing of the audit work should be coordinated with the PO and Unit Supervisor to ensure that the project schedule is considered. The three phases of the audit are described below.
- 8.3 Audit of Data Repository
  - 8.3.1 As an SPMD project nears completion or is completed, the Auditor reviews the records that are in the data repository. Obtain a copy of Tables 1 and 2 and use as checklists to:
    - 8.3.1.1 Record the PO and Auditor’s name and date at the top of Tables 1 and 2.
    - 8.3.1.2 Check the presence/absence and proper location of required records.
    - 8.3.1.3 Check the completeness of the record.
    - 8.3.1.4 Record the status of records, any comments, and date for each of the checks.
  - 8.3.2 The Auditor’s copies of Tables 1 and 2 become part of the Auditor’s Report.
- 8.4 Audit of Data Reduction
  - 8.4.1 Obtain a copy of the PO’s version of Appendix RR-14 (Table 3 in this document).
  - 8.4.2 Conduct a cursory review of the PO’s work on all steps: ensure all steps were completed and contain appropriate documentation.

- 8.4.3 Select and indicate the specific steps to review in more detail by color-coding the Step # in the 2<sup>nd</sup> column of Appendix RR-14. The review must include 2-5 steps within each phase of the Data Reduction process.
- 8.4.4 Select which steps to review after consulting with the PO. The Auditor should also check with the Unit Supervisor and QA Officer who may also specify certain steps to be reviewed
- 8.4.5 For each step selected, independently review the PO's work on the task shown in the 4<sup>th</sup> field ("PO tasks in: Checks, Data Reduction, Documentation"). This may involve checking the calculations that were done by the PO or reviewing the decisions and assumptions made.
- 8.4.6 Insert comments in the field designated for auditor notes when the review of each step is completed. Note the date when the review of each selected step was completed. Save the file in the Data Repository.
- 8.5 Report of Audit Findings
  - 8.5.1 Complete the Audit Report within 15 working days after all project material becomes available.
  - 8.5.2 The Audit Report consists of:
    - 8.5.2.1 Complete checklists for Tables 1 and 2 (Index of Records, Structure of Repository) with dates and notes inserted as appropriate.
    - 8.5.2.2 Completed Table 3 (Appendix RR-14) with dates and notes inserted as appropriate.
    - 8.5.2.3 A summary of main findings and importance of each as appropriate, and recommended actions to resolve any problems (Word document).
  - 8.5.3 Send the Audit Report to the PO, Unit Manager, and QA Officer.
  - 8.5.4 Review the Audit Report with the PO and agree on a timeframe for addressing items noted during the audit. Ideally, resolution of issues should occur within 10 working days when possible.
  - 8.5.5 For issues where the PO and Auditor differ, the Auditor and PO jointly forward the issue to the Toxics TCT workgroup, Unit Manager and QA Officer, for consideration and help in resolving the issue within 10 working days.
- 8.6 Audit Follow-up
  - 8.6.1 The Auditor checks with the PO within 10 working days of distributing the Audit Report and determines the status of resolving the findings of the audit.
  - 8.6.2 When the Audit Report is completed, the PO files it in the Data Repository.
- 9.0 Safety**
  - 9.1 Use general safety practices for working in the office.

Table 1. Index of Records for Conducting SPMD Projects.

Table 1. Index of Records for Conducting SPMD Projects.									
PO Name: _____				Auditor Name: _____			version: 8/29/12		
orig order	Use for Plan + Deploy	Req'd for Data Redcn + Reopstry	Record Description: Template in file or sheet format	SOP Apdx #	Location for records and templates	Repository folder	Auditor: present, absence (P/A)	Auditor Note	Auditor: Date Done
1	X		Index of Records for SPMD Studies	Table 1 (in SOP doc)	j	see Audit Report below			
2	X		Structure of Repository for SPMD Study Data	Table 2 (in SOP doc)	j	see Audit Report below			
3	X		Types of Chemicals Sampled by SPMDs	A	a				
4	X		Field Checklist for SPMD Projects	B	a				
5	X		Process for Meeting Spiking Needs	C-1	a				
6	X		Spiking Solution Worksheet	C-2	a				
7	X		Analytical Methods for SPMD Projects	D	a				
8	X		Types and Characteristics of Quality Control Samples	E	a				
9	X		Major Tasks and Timeline for SPMD Projects	F-1	a				
10	X		Contract Lab Process and Timeline	F-2	a				
11	X		Bi-Pod Boom Assembly	G (in SOP doc)	b				
12	X	X	Field Logs for SPMD Projects	H	a	Field Data			
13	X		Shipping Instructions and Responsibilities	I	a				
14	X	X	Master Sample and Analysis Plan	J	a	Planning Documents			
15	X	X	Samples, Spikes, Splits, and Analyses Plan	K	a	Planning Documents			
16	X	X	Spike Solution Standard Certificate from MEL	L	a	Planning Documents			
17	X	X	Draft Spiking Instructions from MEL	M	a	Planning Documents			
18	X	X	EST Services Summary	N	a	Planning Documents			
19	X	X	Request for Quote - for EST	Other	c	Planning Documents			
20	X	X	Request for Qualifications and Quote - for Contract Labs	Other	c	Planning Documents			
21	X	X	Request for Laboratory Analyses - for Contract Labs	Other	c	Planning Documents			
22	X	X	Project QAPP	Other	d	Planning Documents			
23	X	X	Lab Analysis Required (LAR) Form	Other	e	Planning Documents			
24		X	TidBit Download Instructions	RR-1.1	f	Field Data			
25		X	Deployment Temperature Summary	RR-1.2	f	Field Data			
26		X	Deployment Time Summary	RR-1.3	f	Field Data			
27		X	Streamflow During Deployment	RR-1.4	f	Field Data			
28		X	SPMD Air Exposure Times	RR-2	f	Field Data			
29		X	Ancillary data (e.g. TOC, TSS, flow)	RR-3	f	Lab Results			
30		X	PRC Recoveries	RR-4	f	Data Reduction & Modeling			
31		X	SPMD residue and ancillary results, original EDD format; includes CL work	RR-5	f	Lab Results			
32		X	SPMD residue results - compiled by PO after QA/QC reviews done; includes CL work; Excel format	RR-6	f	Data Reduction & Modeling			
33		X	Log Kow's Used in USGS Model.	RR-7	f	Data Reduction & Modeling			
34		X	USGS model spreadsheet for each sample	RR-8	f	Data Reduction & Modeling			
35		X	Water Concentration Estimates (Dissolved, Whole)	RR-9	f	Data Reduction & Modeling			
36		X	Field Replicate data	RR-10	f	Data Reduction & Modeling			
37		X	Sample site description	RR-11	f	Project General Info			
38		X	Project Summary Form	RR-12	f	Project General Info			
39		X	EST Membrane Condition Sheets	RR-13	h	Case Narratives			
40		X	Data Reduction checklist (PO's initial copy)	RR-14	f	Data Reduction & Modeling			
41		X	Checklist for Reviewing Contract Lab Data Packages	RR-15	f	Lab Results			
42		X	Calculation of Total Water Concentration.	RR-16	f	Data Reduction & Modeling			
43		X	Instructions for Loading SPMD Data into Repository	RR-17	Sharepoint	Project-specific library			
44		X	MEL Case Narratives, for CL too; includes hardcopy results	Other	g	Case Narratives			
45		X	Project Final Report	Other	d	Project General Info			
46		X	Any other information pertaining to the study	Other	i	in appropriate folder			
47		X	Auditor's copy: Index of Records for SPMD Studies	Table 1 (in SOP doc)	j	Audit Report			
48		X	Auditor's copy: Structure of Repository for SPMD Studies	Table 2 (in SOP doc)	j	Audit Report			
49		X	Auditor's copy: Data Reduction checklist (RR-14)	Copy of RR-14	from PO	Audit Report			
50		X	Auditor's Report and Recommendations	Other	Auditor	Audit Report			
<b>Codes for Location of Records and Templates</b>									
a	Y:\SHARED Files\SPMD\SOP Info\SOP Plan & Deploy templates Appendix A-N May 2016.xlsx								
b	Y:\SHARED Files\SPMDs\SOP Info\SOP v4 revision 2016								
c	Y:\SHARED Files\SPMD\SOP Info\								
d	PO: Word doc, filename may vary								
e	PO: Excel table or MEL hardcopy, filename may vary								
f	Y:\SHARED Files\SPMD\SOP Info\SOP Data Redcn & Repository templates v2 2016.xlsx								
g	PO file: PDF files from MEL								
h	PO file: scan EST documents								
i	PO file: other								
j	Y:\SHARED Files\SPMD\SOP Info\SOP Records and Locations Tbl 1 and 2 May 2016.xlsx								

Table 2. Structure of Repository for SPMD Study Data.

Table 2. Structure of Repository for SPMD Study Data.							version: 8/29/12
Folder Level 1	Folder Level 2	File Level	File Type	Auditor: present, absence (P/A)	Auditor Note	Auditor: Completion Date	
<b>* Project Name</b>							
<b>* 1 Project General Info</b>							
		RR-11 Sample site descriptions	xlsx				
		RR-12 Project summary	xlsx				
		Other: Report	pdf, docx				
<b>* 2 Planning Documents</b>							
		Other: Project QAPP (+addendums)	pdf, docx				
		J. Master Sample+Analysis Plan	xlsx				
		K. Table of Sample-Spike-Split-Analyses	xlsx				
		L. Spike Solution Standard Certificate	pdf				
		M. Draft Spiking Instructions from MEL	pdf				
		N. EST Services Summary	xlsx				
		Other: Request for Quote - for EST	pdf, docx				
		Other: Request for Qualifications and Quote - for Contract Labs	pdf, docx				
		Other: Request for Laboratory Analyses - for Contract Labs	pdf, docx				
		Other: Lab Analysis Required (LAR) Form	pdf, xlsx				
<b>* 3 Field Data</b>							
		H. Field Logs	pdf				
		RR-1.1 Tidbit instructions	xlsx				
		RR-1.2 Deployment Temperature	xlsx				
		RR-1.3 Deployment Time	xlsx				
		RR-1.4 Streamflow	xlsx				
		RR-2 SPMD air exposure times	xlsx				
		Field & Membrane Notes (optional compilation of notes for effective cross referencing field issues, spiking, and other membrane handling issues)	xlsx				
<b>* 4 Case Narratives</b>							
		RR-13 Membrane Condition Sheets	pdf				
		Other: Case Narratives	pdf				
<b>* 5 Lab Results</b>							
		RR-3 Ancillary data	xlsx				
		RR-5 SPMD Residue and Ancillary Results (original EDD format)	xlsx				
		RR-15 PCB congener data review	pdf, xlsx				
<b>*6 Data Reduction and Modeling</b>							
		RR-4 PRC recovery	xlsx				
		RR-6 SPMD Residues-compiled	xlsx				
		RR-7 Log Kow's	xlsx				
		RR-8 USGS model used	xlsx				
		RR-9 Water Concentration Estimates	xlsx				
		RR-10 Field Replicate evaluation	xlsx				
		RR-14 Data reduction steps	xlsx				
		RR-16 Total Water Concentration Estimates	xlsx				
<b>*7 Audit Report</b>							
		Table 1. Auditor's Index of Records	pdf, xlsx				
		Table 2. Auditor's Copy of Repository Structure	pdf, xlsx				
		Copy of PO RR-14 Data Reduction Tasks for SPMD Data	pdf, xlsx				
		Other: Auditor's Report and Recommendations	pdf, docx				
Notes:							
* All folder names begin with the "Project Year_Project Name" (e.g. 2007-08_Potholes)							
Repository for SPMD data is located at EAP Sharepoint site.							

Table 3. Data Reduction Tasks for SPMD Data.

Appendix RR-14. Data Reduction Tasks for SPMD Data.				8-29-12 version					
PO Name: _____ Auditor Name: _____									
Phase	Step #	SPMD Processing Tasks	PO tasks in: QC Checks, Data Reduction, Documentation	PO Note	PO Complete Date	Auditor Note	Auditor Complete Date	Relevant Appendix or Record	
<b>A</b>		<b>EST Does Pre-Field Membrane Mfg and Preparation</b>							
A	1	EST fabricates SPMDs	get QC data for batches of project membranes/lipids from EST; USGS CERC does analyses					other doc	
A	2	MEL prepares spiking solutions: surrogates, matrix spike	document chemicals, conc, vol, which membranes					K, L, M	
A	3	PO starts process to get spiking needs met	follow process to get spiking needs met					C-1, C-2	
A	4	MEL, CL, or EST prepares spiking solution: PRCs	document chemicals, conc, vol, which membranes					K, L, M	
A	5	Each lab prepared spike sol'n for potential analysis	to be sent along with other QC samples					K, L, M	
A	6	CL prepares spike solutions: EIS and OPR for isotopic dilution method	document					K, L, M	
A	7	EST prepares Blank samples: Field, Day0D, reagent, other	determine #, type, where to use, and analyze or hold					K, L, M	
A	8	EST ships SPMDs to Ecology	check shipping method, security, and condition of SPMDs					shipping docs	
<b>B</b>		<b>PO Coordinates Field Deployment, Midcheck, and Retrieval</b>							
B	1	Deploy, Midcheck, Retrieve SPMDs	check completeness of Field Logs, remedy as needed					H, other	
B	2	check security and condition of SPMDs	note in Field Logs					H, other	
B	3	transcribe field notes to various spreadsheets	check for transcription errors, remedy as needed					H, other	
B	4	Ecology ships SPMDs to EST	check shipping method, security, and condition of SPMDs					LAR form, shipng docs	
<b>C</b>		<b>PO Checks Tidbit Records</b>							
C	1	download and review Tidbit data:							
C	2	DO THIS BEFORE EST processes samples (don't process useless samples)							
C	3	review Tidbit data, determine submergence for sample period	document					RR-1.123, RR-34	
C	4	if submergence interrupted, accept or reject sample	document					RR-1.123, RR-34	
C	5	compare Tidbit data with mean temp measured in field (Field Notes)	document					RR-1.123, RR-34	
<b>D</b>		<b>EST Does Post-Field Processing, Spiking, Extraction, Splitting, Ampulizing</b>							
D	1	PO reviews all spiking info; gives EST permission to process samples	cross-checks all spiking info from all parties for correct spiking; check math					other (email)	
D	2	cleans SPMDs: samples, QC samples (field and lab)	review ESTs' Membrane Condition Sheets					K, RR-13	
D	3	prepares QC SPMDs per plan (e.g. FrDay0, Spiked Solvent, others)	check Membrane Condition Sheets vs plan					K, RR-13	
D	4	spikes sample and QC SPMDs with sol'ns per plan (e.g. EIS, surrogates)	check Membrane Condition Sheets vs plan					K, RR-13	
D	5	extraction (dialysis); GPC cleanup, solvent exchanges	check Membrane Condition Sheets vs plan					K, RR-13	
D	6	splits extract depending on study needs (e.g. 50:50)	check Membrane Condition Sheets vs plan					K, RR-13	
D	7	measure volumes of extracts (request EST, MEL, CL conduct)	check volumes vs assumption of factor to adjusting results by (usually 2x)					other: data from labs	
D	8	ampulizes extracts for storage/shipment	check Membrane Condition Sheets vs plan					K, RR-13	
D	9	EST ships extracts to Ecology	check shipping method, security, and condition of SPMDs					LAR form, shipng docs	
<b>E</b>		<b>MEL Conducts Analyses</b>							
E	1	MEL recieves extracts	check shipping method, security, and condition of SPMDs					LAR form, shipng docs	
E	2	MEL ships designated extract ampules to CL	check shipping method, security, and condition of SPMDs					LAR form, shipng docs	
E	3	MEL splits extracts as needed for in-house analyses	review Case Narrative for proper splitting					K, L, M	
E	4	sample cleanup & analyses	review Case Narrative for appropriate work					D (specific to project)	
E	5	sample results	review for outliers, spurious values, completeness, other					other	
E	6	analytical QA/QC results: cal, LCS, surrogates, MS, blanks, etc. PO needs to address results of "Membrane Spike": (MEL doesn't treat this as a Matrix Spike)	check results vs QC limits in Case Narratives and data tables Report evaluation regarding data usability					Case Narratives Project Report: QA section	
E	7	Case Narrative developed, sent to PO	review Case Narrative					Case Narratives	
E	8	Electronic data record developed, sent to PO	review data files vs hardcopy record, resolve differences with MEL					Case Narratives, other	

Table 3, continued.

Appendix RR-14. Data Reduction Tasks for SPMD Data (continued).									
PO Name: _____ Auditor Name: _____									
Phase	Step #	SPMD Processing Tasks	PO tasks in: QC Checks, Data Reduction, Documentation	PO Note	PO Complete Date	Auditor Note	Auditor Complete Date	Relevant Appendix or Record	
<b>F</b>		<b>CL Conducts Analysis</b>							
F	1	CL receives extracts	check shipping method, security, and condition of SPMDs					LAR form, shipping docs	
F	2	CL may split extract again - project dependent	review Case Narrative for proper splitting					K, L, M	
F	3	PCB congeners: cleanup & analysis	review Case Narrative for appropriate work					Case Narratives	
F	4	sample results	review for outliers, spurious values, completeness, other					other	
F	5	analytical QA/QC results: cal, LCS, surrogates, blanks, etc.	check results vs QC limits in method, Case Narratives					Case Narratives, other	
F	6	Case Narrative developed, sent to PO	review Case Narrative					Case Narratives	
F	7	Electronic data record developed, sent to PO	review data files vs hardcopy record, resolve differences with MEL					Case Narratives, other	
<b>G</b>		<b>PO Reviews all Case Narratives</b>							
G	1	review Case Narratives for following items, where applicable	review for proper methods, limits, problems					Project Report: QA section	
G	2	analytical methods	" " "					" "	
G	3	holding times; preservation	" " "					" "	
G	4	calibration and verification and degradation checks	" " "					" "	
G	5	on-going precision and recovery assessments	" " "					" "	
G	6	method blanks	" " "					" "	
G	7	matrix spike/matrix spike duplicates (MS/MSD) recoveries	" " " (note: MEL may not use these data to evaluate analytical performance)					" "	
G	8	qualitative identification	" " "					" "	
G	9	laboratory control samples	" " "					" "	
G	10	surrogate recoveries	" " " (note: MEL may not use these data to evaluate analytical performance)					" "	
G	11	laboratory replicates	" " "					" "	
G	12	internal standards checks	" " "					" "	
G	13	check history of splitting samples/extracts and ensure calculations are correct	" " "					" "	
G	14	MEL reviews CL data and CL Case Narrative	check that CN matches data					" "	
G	15	MEL amends data, creates MEL Case Narrative	check that CN matches data					" "	
<b>H</b>		<b>PO Review data sets</b>							
H	1	review electronic data via LIMS or Excel file from MEL lab manager	check completeness; other checks					Case Narratives and EDDs	
H	2	reviews electronic data via CL/MEL spreadsheet	check completeness, other, using checklist for PCB congeners					Case Narratives, EDD, RR-15	
H	3	check how PRCs were handled in datasets (surrogates too if applicable)	check whether summed values include result from PRC or surrogate; adjust as needed to exclude added QC analyte					Case Narratives, EDD, RR-15	
H	4	evaluates blank contamination and how CL and MEL handled	see Evaluate Blank Contamination below					Case Narratives, EDD, RR-15	
H	5	determine how to use MS/MSD & surrogate data in qualifying batch results	document actions and justification					Project Report: QA section	
H	6	address dataset issues to create final dataset for further review	document reviews and actions taken					Case Narratives, EDD, RR-15	
<b>I</b>		<b>PO Evaluates Blanks for Contamination: Field, Process, and Reagent Blanks</b>							
I	1	compile/organize blank results: Field, Day0D, reagent, others	compile/organize					Lab results; need template	
I	2	determine precision for blanks (e.g. RSD for Field blanks, RSD for Day0D blanks)	document in spreadsheet					Lab results; need template	
I	3	evaluate all blanks to locate sources and relevance of contamination	describe and document; tables, charts					Lab results; need template	
<b>J</b>		<b>PO Determines LOD and LOQ for Each Result Using Results from Field Blanks; Decides Whether to Censor or to Blank Correct</b>							
J	1	determine LOD for each result and add to residue results table	LOD = Mean of Field Blanks + 3 Std Devs of the Mean. Set NDs = RL, or explain approach.					RR-6	
J	2	determine LOQ for each result and add to residue results table	LOQ = Mean of Field Blanks + 10 Std Devs of the Mean. Set NDs = RL, or explain approach.					RR-6	
J	3	Select one of two options for handling residue results: censor or blank correct	Note that qualifiers are for residues only - and need be retained for record-keeping					RR-6	
J	4	1 - Censor: If the reported residue result "R" > LOQ, use the reported value. If R < LOQ, censor as UJ. The value associated with the UJ qualifier will be the LOD if R < LOD; or R if R is > LOD. Do not blank correct.	document in residue data table					RR-6	
J	5	2 - Blank Correct. If R > LOD, blank correct thus: R minus the mean of field blanks = blank corrected value. Qualify this value as "B1" which is defined as "Analyte detected in sample and method blank. Reported result is blank-corrected" (definition from EIM reference table). If R < LOD, qualify value as U.	ID and flag results that will be blank-corrected; and those that won't					RR-6	
J	6	consolidate results and document in residue data table and draft report						RR-6	

Table 3, continued.

Appendix RR-14. Data Reduction Tasks for SPMD Data (continued).								
PO Name: _____		Auditor Name: _____						
Phase	Step #	SPMD Processing Tasks	PO tasks in: QC Checks, Data Reduction, Documentation	PO Note	PO Complete Date	Auditor Note	Auditor Complete Date	Relevant Appendix or Record
<b>K</b>		<b>PO Estimates Dissolved Concentrations Using USGS Model</b>						
K	1	Prepare residue results for model	Residue results are usually normalized to 1 membrane - check/adjust SPMD volume as needed					other spreadsheet
K	2	Determine if PRC recoveries are within limits of 20% - 80%	document. PRC-initial is the mean of field blanks or mean of Day0D blanks; PRC-final is from individual sample: document which blanks used					RR-4
K	3	Define how to estimate SPMD performance: PRCs or temp/flow	document; if using temp/flow, need to obtain values and document justification					RR-1, -4
K	4	Select spreadsheet model to use and follow USGS directions (e.g. Version 4, 4-1, 5, 5-1, or more recent edition).	document version used and enter version number at top of spreadsheet model (cell B3 in version 5-1)					RR-8
K	5	verify spreadsheet model by using proven data in a "test run."	test data set to be developed					other
K	6	Insert name of project, Field Station ID, and MEL lab ID into model spreadsheet under Project / Site Name (row 31).	Make sure the name of the spreadsheet tab includes the Field Station ID.					RR-8
K	7	Define Kows to use	document in spreadsheet					RR-7
K	8	Enter PRC data (if used) and select which ones to use based on model instructions	document decisions to use or not use certain PRCs (often based on Uncertainty Factor or % recovery)					RR-8
K	9	Define and enter other model inputs as needed	document					RR-8
K	10	DELETE K9 - not needed						
K	11	Input residue data and run model for LOD, LOQ, and samples	for initial PRC value, use mean of field blanks or mean of Day0D blanks; for final PRC value, use sample value: document which blanks used					RR-8
K	12	Create file and compile dissolved water concentrations						RR-9
K	13	add qualifier "J" in spreadsheet next to all dissolved values	add qualifier "B1" for results based on blank-corrected residues					RR-9
<b>L</b>		<b>PO Determines Whether and Where to Use Summed or Individual Results for Analyte Groups (e.g. PCBs, DDTs, PBDEs, Chlordanes)</b>						
L	1	determine whether to use summed or individual results	describe reasons for or against using summing values					
L	2	<u>Recommend use of individual results because USGS spreadsheet designed for individual except has option for T-PCBs. NOTE: T-PCBs Kow is an average.</u>						
L	3	if summing, sum according to TSU guidance	document					See TCTT documents
L	4	PCB congeners	document					PO spreadsheet
L	5	PCB Aroclors	document					PO spreadsheet
L	6	DDTs	document					PO spreadsheet
L	7	Chlordanes	document					PO spreadsheet
L	8	Endosulfans	document					PO spreadsheet
L	9	PBDEs	document					PO spreadsheet
L	10	LPAH	document					PO spreadsheet
L	11	HPAH	document					PO spreadsheet
L	12	TPAH	document					PO spreadsheet
L	13	Others? .....add new rows here and list them	document					PO spreadsheet
<b>M</b>		<b>PO Estimates Total or Whole Water Concentrations from Model Output and TOC Data</b>						
M	1	Determine inputs for estimating Total concentration from Dissolved						RR-1, -3, -9,
M	2	TOC	define how this input was determined					RR-3
M	3	Koc (organic carbon-water equilibrium partitioning coefficient)	define how this input was determined					RR-16
M	4	Mw (mass of water)	define how this input was determined					RR-16
M	5	Create spreadsheet to calc Total from Dissolved	no standard format available yet					RR-16
M	6	compile Total concentrations in useful format	no standard format available yet					RR-9
		<b>Results for dissolved or whole water fractions now ready to use</b>	<b>ensure all documentation is complete</b>					

This Table 3 is Appendix RR-14 located at Y:\SHARED Files\SPMDs\SOP Info\ SOP Data Redcn & Repository templates v2 2016.xlsx.