

Puget Sound Sediment Reference Material: Requesting and Analyzing the SRM, and Reporting Data

Introduction

The Puget Sound Sediment Reference Material (SRM) has been developed to help assess/evaluate measurement accuracy and monitor laboratory performance when analyzing for chlorinated dioxin, furans, and biphenyl compounds in sediment samples collected from the Puget Sound area. The SRM is currently available free of charge, though recipients must pay shipping costs. This document provides instructions for obtaining, analyzing, and reporting on the SRM. The guidance and procedures are intended to ensure that SRM users:

- Report methods used for analysis
- Report QA/QC procedures used to verify and validate results, and
- Report results that can be included in periodic recalculations of acceptance limits

The Puget Sound SRM has been established for chlorinated dibenzo-p-dioxins / chlorinated dibenzofurans (CDD/CDF), and/or chlorinated biphenyl (CB) congener analysis using high resolution gas chromatography / high resolution mass spectrometry (HRGC/HRMS) methods. This SRM is also suitable for Aroclor analysis using gas chromatography/electron capture detection (GC/ECD) methods.

Request procedure

The Seattle District Corps of Engineers, Washington Department of Ecology and US EPA Region 10 have assigned staff to distribute the Puget Sound SRM in support of agency missions, including regulatory programs. The request procedure is as follows:

- Obtain the electronic Puget Sound SRM Request Form from the appropriate agency involved with the project (see agency contact list below), or from the DMMO website
- Return completed form to agency contact
- Agency contact reviews and certifies/signs the bottom of the form as an “authorized agency requester”, and then forwards the signed form to the EPA Region 10 SRM Manager (Donald Brown) for processing
- Request is processed, typically within a week

Examples of how the request process works:

1. CWA 404 permit applicants would request from and submit the completed form to the Corps of Engineers DMMO contact
2. A CERCLA PRP would submit the request form via EPA
3. The State of Washington's ambient monitoring program would submit the form via Ecology.

The authorized agency contacts are available to help with any questions about the Request Form. Submission of incomplete forms may delay the request processing.

Authorized Agency Contacts:

Seattle District Corps of Engineers – Dredged Material Management Office (DMMO):

David Kendall (206) 764-3768, david.r.kendall@usace.army.mil

David Fox (206) 764-6083, david.f.fox@usace.army.mil

Lauran Warner (206) 764-6550, lauran.c.warner@usace.army.mil

Kelsey van der Elst (206) 764-6945, kelsey.vanderelst@usace.army.mil

Washington Department of Ecology

Laura Inouye (306) 407-6165, lino461@ecy.wa.gov

Tom Gries (360) 407-6327, tgri461@ecy.wa.gov

US Environmental Protection Agency Region 10

Justine Barton (206) 553-6051, barton.justine@epa.gov

Erika Hoffman (360) 753-9540, hoffman.erika@epa.gov

Donald Brown (206) 553-0717, brown.donaldm@epa.gov

Shipping

The Puget Sound SRM is stored at EPA's Quality Assurance Technical Services (QATS) contractor (Shaw Environmental) located in Las Vegas, Nevada. Lab contacts listed on the Request Form should be prepared to confirm shipping details (including UPS or FedEx account number) when contacted by Shaw Environmental. Shaw Environmental will generally ship the SRM within 24 hours of receiving the completed Request Form from the EPA Region 10 SRM Manager. The SRM will arrive with specific instructions on handling

and storage requirements, data reporting requirements, as well as chain of custody paperwork.

When the SRM has been shipped, Shaw Environmental will provide a notification email to the EPA Region 10 SRM Manager, the authorized agency contact (as indicated on the Request Form), and the destination laboratory. The email will include the project name as indicated on the Request Form.

SRM storage requirements

Each amber glass bottle contains approximately 30 grams of the Puget Sound SRM.

The SRM contains compounds that are light sensitive and should be protected from light during storage. Store the SRM at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ until SRM preparation and analysis.

SRM analysis requirements

The SRM is to be analyzed as described in the appropriate methods employed for the analysis of CDD/CDF and/or CB congener analytes using HRGC/HRMS instrumentation and/or Aroclors using GC/ECD instrumentation.

The following analytical methods may be used in the analysis of the SRM:

- SW846 Method 8082A (or current revision), “Polychlorinated Biphenyls by Gas Chromatography”
- SW846 Method 8290A (or current revision), “ Polychlorinated Dibenzo-p-Dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)”
- Method 1613B (or current revision), “Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS
- Method 1668C (or current revision), “Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids and Tissue by HRGC/HRMS”

Data verification/validation

SRM users may be held to different data validation requirements, depending on their program and project circumstances. Data must be validated to EPA Stage 2B but it is strongly recommended that Stage 3 or better validation be conducted. For example, the interagency Dredged Material Management Program (DMMP) strongly recommends third party Stage 4 validation for all TCDD/F data. Any validation narrative must indicate the validation stage used. Data validation stages are described in EPA-540-R-08-005 (see References).

Data reporting

Guidance for Distribution & Reporting 5-29-2012

Individual laboratories typically provide all project data and validation reports to their clients. The client/project proponent is responsible for ensuring that all information relative to the SRM, including associated QA data, is sent to the original agency requester. For DMMP projects, submittal of the complete validated data package to the DMMO contact fulfills this requirement.

For SRM data meeting established QA requirements, the agency contact will submit the validated electronic data deliverable/data summary sheets (or the equivalent) and validation reports relevant to the SRM to the EPA Region 10 SRM Manager. Changes made by the data validator (e.g. modification of data qualifiers) must be clearly indicated on the data sheets. SRM data not meeting established QA requirements will not be forwarded to Shaw Environmental, however, Shaw Environmental will be notified of the QA failure for their records. Minimum SRM data package deliverables are as follows:

1. Validation Report
 - Confirmation of data verification or third party validation
 - Include validation report if SRM results are affected

2. SRM Sample Data Summary Report including the following information:
 - Identification and quantitation of target analytes including dilution and reanalysis
 - CAS numbers
 - Name of Laboratory
 - Project Number
 - Sample ID number (bottle bar code)
 - Agency Sample number
 - Laboratory Sample number
 - Date SRM received
 - Date and time of analysis
 - For Aroclor data labs must provide reporting limits (RLs) and method detection limits (MDLs)
 - For CB congener and dioxin/furan data labs must provide reporting limits (RLs) and estimated detection limits (EDLs)
 - Laboratory Qualifiers and Definitions
 - Validation Qualifiers

Storage and use of previously opened SRM is not recommended. However, it is requested that any additional data results derived from use of the SRM be submitted to the EPA Region 10 SRM manager.

Performance / Acceptance Limits

The acceptance limits presented below are guidance values based on the original laboratory round robin associated with the development of the SRM. The implications associated with not meeting these acceptance limits will be determined by data reviewers on a case by case basis, based on the goals of their program/project. For upcoming dredging year 2013, the DMMP will review results on a case by case basis and will consider the values advisory.

PCB Aroclors. A twelve-lab round robin testing of the SRM (including commercial and CLP labs), was used to calculate an acceptance limit for Aroclor 1260. The average Aroclor 1260 concentration found during the round robin was 108 ug/kg. The acceptance limit is set at the 95% confidence interval.

- **Aroclor 1260:**

warning low 41 ug/kg

warning high 180 ug/kg

CDD/CDF. A ten-lab round robin testing of the SRM (including commercial and CLP labs) was used to calculate an acceptance limit of +/- 50% action low and action high for each congener as follows:

Acceptance Limits Source	Analyte	CAS No.	Avg. Conc. ng/Kg	Action Low -50%	Action High +50%
+/- 50 Percent	1,2,3,4,6,7,8-HpCDD	35822-46-9	90.6	45.3	136
	1,2,3,4,6,7,8-HpCDF	67562-39-4	18.7	9.36	28.1
	1,2,3,4,7,8,9-HpCDF	55673-89-7	1.63	0.815	2.44
	1,2,3,4,7,8-HxCDD	39227-28-6	1.59	0.797	2.39
	1,2,3,4,7,8-HxCDF	70648-26-9	3.02	1.51	4.53
	1,2,3,6,7,8-HxCDD	67653-85-7	3.88	1.94	5.82
	1,2,3,6,7,8-HxCDF	57117-44-9	1.09	0.545	1.64
	1,2,3,7,8,9-HxCDD	19408-74-3	3.04	1.52	4.55
	1,2,3,7,8,9-HxCDF	72918-21-9	0.51 1	0.255	0.77
	1,2,3,7,8-PeCDD	40321-76-4	1.08	0.542	1.63
	1,2,3,7,8-PeCDF	57117-41-6	1.23	0.613	1.84
	2,3,4,6,7,8-HxCDF	60851-34-5	1.83	0.917	2.75
	2,3,4,7,8-PeCDF	57117-31-4	1.07	0.533	1.60
	2,3,7,8-TCDD	1746-01-6	1.05	0.525	1.57
	2,3,7,8-TCDF	51207-31-9	1.11	0.557	1.67
	OCDD	3268-87-9	811	406	121 7
OCDF	39001-02-0	58.4	29.2	87.6	

CB Congeners. These values have not yet been reviewed and no acceptance limits have been determined to date.

Recalculation of Acceptance Limits

Shaw Environmental will store the SRM, conduct stability testing, and maintain the SRM database used to recalculate acceptance limits. Depending on the quantity of high quality data received, recalculation may occur annually. It is anticipated that the next recalculation will occur after 30 new data points have been received.

References

Revised Supplemental Information on Polychlorinated Dioxins and Furans (PCDD/F) For Use in Preparing a Quality Assurance Project Plan (QAPP), dated November 8, 2010. EPA-540-R-08-005 Guidance For Labeling Externally Validated Laboratory Analytical Data for Superfund Use, dated January 13, 2009

Attachments

SRM Request Form (this form may be requested and returned in an electronic-fillable form from/to agency contacts listed above). This form is also available on the Seattle District DMMO website.