# Surface Water Ambient Monitoring Program Quality Assurance Program Plan: 2008 to 2013 Appendix Revision Summary

In January 2013, the Surface Water Ambient Monitoring Program (SWAMP) Roundtable approved updates to certain quality control (QC), sample handling, and corrective action guidelines appearing in Appendices A, B, and D, respectively, of the 2008 Surface Water Ambient Monitoring Program Quality Assurance Program Plan (QAPrP).

The tables below summarize these updates and include the following information:

- A "2008" row containing language from the 2008 QAPrP tables (available through the <u>SWAMP Quality Assurance Help Desk</u>)
- A "2013" row containing language from the <u>2013 QAPrP tables</u>
- A "Justification" column identifying the rationale behind each update

The tables are divided first by analytical category (e.g., inorganic analytes), then matrix (e.g., fresh and marine water).

This document includes <u>only</u> those guidelines that changed between 2008 and 2013. Unabridged guidelines may be found at the two table links above.

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## **Inorganic Analytes**

#### Inorganic Analytes in Fresh and Marine Water

#### Sample Handling: Inorganic Analytes in Fresh and Marine Water – Hexavalent Chromium (Filtered)

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 °C in the dark	24 hours, must notify lab in advance	Agreement with 40 CFR Section
2013	Cool to ≤6 °C, pH 9.3 – 9.7 within 24 hours	28 days at ≤6 °C¹	136.3

<sup>1</sup>If the analytical method doesn't include preservation, analysis must occur within 24 hours.

#### Sample Handling: Inorganic Analytes in Fresh and Marine Water – Mercury (Dissolved)

Version	Recommended Preservation	Required Holding Time	Justification
2008	Filter within 15 minutes of collection; Cool to 6 ∘C in the dark; Acidify to 0.5% with pre-tested HCl within 48 hours	6 months at room temperature following filtration and/or acidification	Agreement with 40 CFR
2013	Filter and preserve with 0.5% v:v pre- tested 5% BrCl or 12N HCl within 48 hours	90 days at room temperature following acidification	Section 130.5

#### Sample Handling: Inorganic Analytes in Fresh and Marine Water – Mercury (Total)

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 °C in the dark; Acidify to 0.5% with pre-tested HCI within 48 hours	6 months at room temperature following acidification	Agreement with 40 CFR
2013	Preserve with 0.5% v:v pre-tested 5% BrCl or 12N HCl within 48 hours	90 days at room temperature following acidification	Section 136.3

#### Sample Handling: Inorganic Analytes in Fresh and Marine Water – Trace Metals (Dissolved)

Version	Recommended Preservation	Required Holding Time	Justification
2008	Filter within 15 minutes of collection; Cool to ≤6 °C in the dark; Acidify to pH<2 with pre-tested HNO <sub>3</sub> within 48 hours	6 months at room temperature following acidification	Agreement with 40 CFR
2013	Filter within 15 minutes of collection; HNO <sub>3</sub> to pH<2 within 48 hours and at least 24 hours prior to analysis	6 months at room temperature following acidification	Section 130.5

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 ∘C in the dark; Acidify to pH<2 with pre-tested HNO <sub>3</sub> within 48 hours	6 months at room temperature following acidification	Agreement with 40 CFR
2013	HNO₃ to pH<2 within 48 hours and at least 24 hours prior to analysis	6 months at room temperature following acidification	Section 136.3

#### Sample Handling: Inorganic Analytes in Fresh and Marine Water – Trace Metals (Total)

### Inorganic Analytes in Freshwater Tissue and Marine Tissue

#### Sample Handling: Inorganic Analytes in Freshwater Tissue and Marine Tissue

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 ℃ and in the dark	1 year; samples must be analyzed within 14 days of collection or thawing	Agreement with EPA's Guidance for Assessing
2013	Cool to ≤6 ℃ within 24 hours, then freeze to ≤-20 ℃	1 year	Data for Use in Fish Advisories

Inorganic analyte coverage in the QAPrP has been expanded to include selenium speciation in water. The following tables include the associated guidelines.

#### Sample Handling: Inorganic Analytes in Fresh and Marine Water – Selenium Speciation

Analyte	Recommended Container <sup>1</sup>	Recommended Preservation <sup>2,3</sup>	Required Holding Time <sup>4</sup>
Selenium Speciation <sup>7</sup>	Р	Filter and preserve with 0.4% HCl within 15 minutes of collection; maintain collection temperature as best as possible	6 months

#### Quality Control<sup>1</sup>: Inorganic Analytes in Fresh and Marine Water – Selenium Speciation

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Calibration Standard Per analytical method or manufacturer's specifications		Per analytical method or manufacturer's specifications
Calibration Verification	Per 10 analytical runs	80-120% recovery
Laboratory Blank	Per 20 samples or per analytical batch, whichever is more frequent	<rl analyte<="" for="" target="" th=""></rl>
Reference Material2Per 20 samples or per analytical batch, whichever is more frequent		75-125% recovery (70-130% for MMHg)
Matrix Spike	Per 20 samples or per analytical batch, whichever is more frequent	75-125% recovery (70-130% for MMHg)
Matrix Spike Duplicate	Per 20 samples or per analytical batch, whichever is more frequent	75-125% recovery (70-130% for MMHg); RPD<25%

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Laboratory Duplicate         Per 20 samples or per analytical batch, whichever is more frequent		RPD<25% (n/a if native concentration of either sample <rl)< th=""></rl)<>
Internal Standard	Accompanying every analytical run when method appropriate	60-125% recovery
Field Quality Control	Frequency of Analysis	Measurement Quality Objective
Field Duplicate         5% of total project sample count		RPD<25% (n/a if native concentration of either sample <rl), by="" method<="" otherwise="" specified="" th="" unless=""></rl),>
Field Blank, Equipment Blank	Per method	Blanks <rl analyte<="" for="" target="" th=""></rl>

<sup>1</sup> Unless method specifies more stringent requirements <sup>2</sup> Not applicable to selenium speciation

### **Organic Compounds**

For <u>all</u> organic analyses, the following updates have been approved:

- SWAMP's laboratory duplicate requirement will be discontinued. Because precision is assessed by matrix spike and matrix spike duplicate analyses, the laboratory duplicate is considered an unnecessary expense that does not add technical value to the dataset.
- All mass spectrometry analyses will include a method-specific tuning step

Category-specific updates appear in the following tables.

### Semi-Volatile Organic Compounds in Fresh and Marine Water

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications	
		<ul> <li>Correlation coefficient (r<sup>2</sup> &gt;0.990) for linear and non-linear curves</li> </ul>	
		<ul> <li>If RSD&lt;15%, average RF may be used to quantitate; otherwise use equation of the curve</li> </ul>	Agreement with EPA Method
2013	Initial method setup or when the calibration verification fails	<ul> <li>First- or second-order curves only (not forced through the origin)</li> </ul>	SW-846 Method 8270
		<ul> <li>Refer to SW-846 methods for SPCC and CCC criteria<sup>1</sup></li> </ul>	
		<ul> <li>Minimum of 5 points per curve (one of them at or below the RL)</li> </ul>	

#### Quality Control: Semi-Volatile Organic Compounds in Fresh and Marine Water – Calibration Standard

<sup>1</sup>Mass spectrometry only

#### Quality Control: Semi-Volatile Organic Compounds in Fresh and Marine Water – Reference Material

Version	Frequency of Analysis	Justification
2008	Method Validation: as many as required to assess accuracy and precision of method before routine analysis of samples; Routine Accuracy Assessment: per 20 samples or per analytical batch (preferably blind)	Removal of method validation language, which is beyond the scope of these tables
2013	Per 20 samples or per analytical batch, whichever is more frequent	

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per method	Per method	Developed from focus group meetings and communication
2013	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)	with other technical experts; approval from WPCL and QAT

#### Quality Control: Semi-Volatile Organic Compounds in Fresh and Marine Water – Surrogate

#### Quality Control: Semi-Volatile Organic Compounds in Fresh and Marine Water – Internal Standard

Version	Frequency of Analysis	Justification
2008	Per method	Agreement with EPA Method 625, EPA Method
2013	Included in all samples and all QC samples (as available)	1625, and SW-846 Method 8270

#### Semi-Volatile Organic Compounds in Freshwater Sediment and Marine Sediment

# Quality Control: Semi-Volatile Organic Compounds in Freshwater Sediment and Marine Sediment – Calibration

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications	
2013		<ul> <li>Correlation coefficient (r<sup>2</sup> &gt;0.990) for linear and non-linear curves</li> </ul>	
	Initial method setup or when the calibration verification fails	<ul> <li>If RSD&lt;15%, average RF may be used to quantitate; otherwise use equation of the curve</li> </ul>	Agreement with SW-846
		<ul> <li>First- or second-order curves only (not forced through the origin)</li> </ul>	Method 8270
		Refer to SW-846 methods for SPCC and CCC criteria <sup>1</sup>	
		<ul> <li>Minimum of 5 points per curve (one of them at or below the RL)</li> </ul>	

<sup>1</sup>Mass spectrometry only

# Quality Control: Semi-Volatile Organic Compounds in Freshwater Sediment and Marine Sediment – Internal Standard

Version	Frequency of Analysis	Justification
2008	Per method	Agreement with CN/ 946 Method 9270
2013	Included in all samples and all QC samples (as available)	Agreement with SW-846 Method 8270

# Quality Control: Semi-Volatile Organic Compounds in Freshwater Sediment and Marine Sediment – Reference Material

Version	Frequency of Analysis	Justification
2008	Method Validation: as many as required to assess accuracy and precision of method before routine analysis of samples; Routine Accuracy Assessment: per 20 samples or per analytical batch (preferably blind)	Removal of method validation language, which is beyond the scope of these tables
2013	Per 20 samples or per analytical batch (preferably blind)	

# Quality Control: Semi-Volatile Organic Compounds in Freshwater Sediment and Marine Sediment – Surrogate

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per method	Per method	Developed from focus group meetings and communication
2013	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)	with other technical experts; approval from WPCL and QAT

### Synthetic Organic Compounds in Fresh and Marine Water

#### Quality Control: Synthetic Organic Compounds in Fresh and Marine Water – Calibration

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications	
2013	Initial method setup or when the calibration verification fails	<ul> <li>Correlation coefficient (r<sup>2</sup> &gt;0.990) for linear and non-linear curves</li> <li>If RSD&lt;15%, average RF may be used to quantitate; otherwise use equation of the curve</li> <li>First- or second-order curves only (not forced through the origin)</li> <li>Refer to SW-846 methods for SPCC and CCC criteria<sup>1</sup></li> <li>Minimum of 5 points per curve (one of them at or below the RL)</li> </ul>	Agreement with EPA Method 625, EPA Method 1625, and SW-846 Method 8270

<sup>1</sup>Mass spectrometry only

#### Quality Control: Synthetic Organic Compounds in Fresh and Marine Water – Internal Standard

Version	Frequency of Analysis	Justification
2008	Per method	Agreement with EPA Method 625, EPA
2013	Included in all samples and all QC samples (as available)	Method 1625, and SW-846 Method 8270

#### Quality Control: Synthetic Organic Compounds in Fresh and Marine Water – Reference Material

Version	Frequency of Analysis	Justification
2008	Method Validation: as many as required to assess accuracy and precision of method before routine analysis of samples; Routine Accuracy Assessment: per 20 samples or per analytical batch (preferably blind)	Removal of method validation language, which is beyond the scope of these tables
2013	Per 20 samples or per analytical batch (preferably blind)	

#### Quality Control: Synthetic Organic Compounds in Fresh and Marine Water – Surrogate

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per method	Per method	Developed from focus group meetings and communication with other
2013	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)	technical experts; approval from WPCL and QAT

#### Sample Handling: Synthetic Organic Compounds in Fresh and Marine Water - Glyphosate

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 $^{\circ}$ C in the dark.	6 months at -20 ℃; Samples must be analyzed within 7 days of collection or thawing	
2013	Cool to ≤6 ℃; store in the dark; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if residual chlorine is present; freeze to ≤-20 ℃	18 months (14 days if unfrozen)	Agreement with EPA Method 547

#### Sample Handling: Synthetic Organic Compounds in Fresh and Marine Water – Phenols

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 $^{\circ}$ C in the dark.	Samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.	Agreement with 40 CFR
2013	Cool to ≤6 ℃; 0.008% Na₂S₂O₃if residual chlorine is present	7 days until extraction, 40 days after extraction	Section 136.3

# Sample Handling: Synthetic Organic Compounds in Fresh and Marine Water – Polychlorinated Biphenyls (as Congeners/Aroclors)

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 $^{\circ}$ C in the dark.	Samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.	Agreement with 40 CFR Section
2013	Cool to ≤6 ℃	1 year until extraction, 1 year after extraction	130.3

# Sample Handling: Synthetic Organic Compounds in Fresh and Marine Water – Polynuclear Aromatic Hydrocarbons

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 $^{\circ}$ C in the dark.	Samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.	Agreement with 40 CFR Section
2013	Cool to ≤6 ℃; store in the dark; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if residual chlorine is present	7 days until extraction, 40 days after extraction	136.3

### Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment

# Quality Control: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment – Calibration Standard

Version	Frequency of Analysis	Measurement Quality Objective Justification	
2008	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications	
2013	Initial method setup or when the calibration verification fails	<ul> <li>Correlation coefficient (r<sup>2</sup> &gt;0.990) for linear and non-linear curves</li> <li>If RSD&lt;15%, average RF may be used to quantitate; otherwise use equation of the curve</li> <li>First- or second-order curves only (not forced through the origin)</li> <li>Refer to SW-846 methods for SPCC and CCC criteria<sup>1</sup></li> <li>Minimum of 5 points per curve (one of them at or below the RL)</li> </ul>	Agreement with SW-846 Method 8270

<sup>1</sup>Mass spectrometry only

# Quality Control: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment – Calibration Verification

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per 10 analytical runs	85-115% recovery	
2013	Per 12 hours	<ul> <li>Expected response or expected concentration ±20%</li> <li>RF for SPCCs=initial calibration<sup>1</sup></li> </ul>	part 6000 and the SW-846 8000 series

<sup>1</sup>Mass spectrometry only

# Quality Control: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment – Internal Standard

Version	Frequency of Analysis	Justification	
2008	Per method	A second with CM/ 040 Mathed 0270	
2013	Included in all samples and all QC samples (as available)	Agreement with SW -646 Method 6270	

# Quality Control: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment – Reference Material

Version	Frequency of Analysis	Justification
2008	Method Validation: as many as required to assess accuracy and precision of method before routine analysis of samples; Routine Accuracy Assessment: per 20 samples or per analytical batch (preferably blind)	Removal of method validation language, which is beyond the scope of these tables
2013	Per 20 samples or per analytical batch (preferably blind)	

#### Quality Control: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment - Surrogate

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per method	Per method	Developed from focus group meetings and
2013	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)	experts; approval from WPCL and QAT

# Sample Handling: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment – Polybrominated Diphenyl Ethers and Polychlorinated Biphenyls (as Congeners/Aroclors)

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 °C in the dark.	1 year at -20 °C; Samples must be extracted within 14 days of collection or thawing and analyzed within 40 days of extraction	Agreement of SW-846 Chapter
2013	Cool to ≤6 ℃ within 24 hours, then freeze to ≤- 20 ℃	None	4 (Table 4-1)

### Synthetic Organic Compounds in Freshwater Tissue and Marine Tissue

# Quality Control: Synthetic Organic Compounds in Freshwater Tissue and Marine Tissue – Calibration Standard

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications	
2013	Initial method setup or when the calibration verification fails	<ul> <li>Correlation coefficient (r<sup>2</sup> &gt;0.990) for linear and non-linear curves</li> <li>If RSD&lt;15%, average RF may be used to quantitate; otherwise use equation of the curve</li> <li>First- or second-order curves only (not forced through the origin)</li> <li>Refer to SW-846 methods for SPCC and CCC criteria<sup>1</sup></li> <li>Minimum of 5 points per curve (one of them at or below the RI )</li> </ul>	Agreement with SW-846 Method 8270

<sup>1</sup> Mass spectrometry only

#### Quality Control: Synthetic Organic Compounds in Freshwater Tissue and Marine Tissue – Reference Material

Version	Frequency of Analysis	Justification
2008	Method Validation: as many as required to assess accuracy and precision of method before routine analysis of samples; Routine Accuracy Assessment: per 20 samples or per analytical batch (preferably blind)	Removal of method validation language, which is beyond the scope of these tables
2013	Per 20 samples or per analytical batch (preferably blind)	

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per method	Per method	Developed from focus group meetings
2013	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)	technical experts; approval from WPCL and QAT

#### Quality Control: Synthetic Organic Compounds in Freshwater Tissue and Marine Tissue – Surrogate

#### Quality Control: Synthetic Organic Compounds in Freshwater Tissue and Marine Tissue – Internal Standard

Version	Frequency of Analysis	Justification
2008	Per method	Agrooment with CM/ 046 Method 0270
2013	Included in all samples and all QC samples (as available)	Agreement with SW-846 Method 8270

# Sample Handling: Synthetic Organic Compounds in Freshwater and Marine Tissue – Polybrominated Diphenyl Ethers and Polychlorinated Biphenyls (as Congeners/Aroclors)

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 °C	1 year at -20 °C; Samples must be extracted within 14 days of collection or thawing and analyzed within 40 days of extraction	Agreement of SW-846 Chapter 4 (Table 4-1)
2013	Cool to ≤6 ℃ within 24 hours, then freeze to ≤-20 ℃	None	

### Volatile Organic Compounds in Freshwater Sediment and Marine Sediment

Quality Control: Volatile Organic Compounds in Freshwater Sediment and Marine Sediment - Calibratic	'n
Standard	

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications	
		<ul> <li>Correlation coefficient (r<sup>2</sup> &gt;0.990) for linear and non-linear curves</li> </ul>	
2013	<ul> <li>Initial method setup or when the calibration verification fails</li> </ul>	<ul> <li>If RSD&lt;15%, average RF may be used to quantitate; otherwise use equation of the curve</li> </ul>	Agroomont with SW/ 946
		<ul> <li>First- or second-order curves only (not forced through the origin)</li> </ul>	Method 8260
		Refer to SW-846 methods for SPCC and CCC criteria <sup>1</sup>	
		<ul> <li>Minimum of 5 points per curve (one of them at or below the RL)</li> </ul>	

<sup>1</sup>Mass spectrometry only

# Quality Control: Volatile Organic Compounds in Freshwater Sediment and Marine Sediment – Internal Standard

Version	Frequency of Analysis	Justification
2008	Per method	Agroament with SW/ 946 Method 9260
2013	Included in all samples and all QC samples (as available)	- Agreement with SVV-846 Method 8260

#### Quality Control: Volatile Organic Compounds in Freshwater Sediment and Marine Sediment – Reference Material

Version	Frequency of Analysis	Justification
2008	Method Validation: as many as required to assess accuracy and precision of method before routine analysis of samples; Routine Accuracy Assessment: per 20 samples or per analytical batch (preferably blind)	Removal of method validation language, which is beyond the scope of these tables
2013	Per 20 samples or per analytical batch, whichever is more frequent	

#### Quality Control: Volatile Organic Compounds in Freshwater Sediment and Marine Sediment – Surrogate

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per method	Per method	Developed from focus group meetings and communication with other
2013	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)	technical experts; approval from WPCL and QAT

In addition to the table updates above, new pyrethroids guidelines have been approved.

#### Quality Control: Pyrethroids in Whole Water and Sediment – Calibration

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications	Developed from focus group mostings
2013	Daily, or just prior to analysis; five or more standards spanning the sample result range <sup>1</sup> , with the lowest standard at or below the RL	r ≥0.995 (or r <sup>2</sup> ≥0.995, all curve types not forced through origin)	and communication with other technical experts; approval from WPCL and QAT

<sup>1</sup>Sample results above the highest standard are to be diluted and re-analyzed.

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per 10 analytical runs	85-115% recovery	Developed from focus group meetings and communication with other
2013	Per 10 analytical samples <sup>1</sup>	80-120% <sup>2</sup>	technical experts; approval from WPCL and QAT

#### Quality Control: Pyrethroids in Whole Water and Sediment - Calibration Verification

<sup>1</sup>Analytical samples include samples only and do not include clean-out or injection blanks.

<sup>2</sup>Limit applies to a mid-level standard; low-level calibration checks near the RL may have a wider range that is project specific

#### Quality Control: Pyrethroids in Whole Water and Sediment – Reference Material/Laboratory Control Sample

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008 (Reference Material) Method Validation: as many as required to assess accuracy and precision of method before routine analysis of samples; Routine Accuracy Assessment: per 20 samples or per analytical batch (preferably blind)		70-130% recovery if certified; otherwise, 50- 150% recovery	Developed from focus group meetings and communication with other technical experts;
2013     Per 20 samples or per analytical batch,       Control     whichever is more frequent       Sample)     Sample		50-150%	approval from WPCL and QAT

#### Quality Control: Pyrethroids in Whole Water and Sediment - Surrogate

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per method	Per method	Developed from focus group meetings and communication with other
2013	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)	technical experts; approval from WPCL and QAT

#### Quality Control: Pyrethroids in Whole Water and Sediment - Internal Standard

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per method	Per method	Developed from focus group meetings and communication
2013	Included in all samples and all QC samples (as available)	Per laboratory procedure	with other technical experts; approval from WPCL and QAT

### Sample Handling: Pyrethroids in Whole Water

Version	Required Preservation	Required Holding Time	Justification
2008	Cool to 6 $^{\circ}$ C in the dark.	Samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.	Developed from focus group
2013	Cool ≤ 6 ℃ in the dark; samples must be extracted or preserved according to laboratory procedures with suitable preservative or extraction solvent within 72 hours of collection	7 days until extraction, 40 days after extraction	meetings and communication with other technical experts; approval from WPCL and QAT

### Sample Handling: Pyrethroids in Sediment

Version	<b>Required Preservation</b>	Required Holding Time	Justification
2008	Cool to 6 °C in the dark	1 year at -20 ℃; Samples must be extracted within 14 days of collection or thawing and analyzed within 40 days of extraction.	Developed from focus group
2013	Short-term storage: ≤6 ℃ in the dark; long-term storage, or storage of remaining sample: ≤-20 ℃ in the dark	1 year at ≤-20 ℃ in the dark; samples must be extracted within 14 days of collection or thawing and analyzed within 40 days of extraction	with other technical experts; approval from WPCL and QAT

# **Conventional Parameters**

### Conventional Parameters in Fresh and Marine Water

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 $^{\circ}$ C and store in the dark	28 days	Agreement with 40 CFR Section 136.3 and Standard Methods
2013	None required	28 days	1060: Collection and Preservation of Samples

#### Sample Handling: Conventional Parameters in Fresh and Marine Water – Chloride

#### Sample Handling: Conventional Parameters in Fresh and Marine Water – Cyanide

Version	Recommended Preservation	Required Holding Time	Justification
2008	Preserve to pH>12 with ~ 2 mL 1:1 NaOH, Add 0.6 g $C_6H_8O_6$ if residual Cl present; Cool to 6 °C and store in the dark	14 days	Agreement with 40 CFR Section 136.3
2013	Cool to ≤6 °C; NaOH to pH>10; add 0.6 g C <sub>6</sub> H <sub>8</sub> O <sub>6</sub> if residual chlorine is present	14 days	

#### Sample Handling: Conventional Parameters in Fresh and Marine Water – Fluoride

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 $^{\circ}$ C and store in the dark	28 days	Agreement with 40 CFR Section 136.3 and Standard Methods
2013	None required	28 days	1060: Collection and Preservation of Samples

#### Sample Handling: Conventional Parameters in Fresh and Marine Water - Organic Carbon (Dissolved)

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 °C and store in the dark.	28 days	Agreement with 40 CFR Section 136.3, Standard Methods 1060:
2013	Filter and preserve to pH<2 within 48 hours of collection; cool to ≤6 ℃	28 days	Collection and Preservation of Samples, and the June 28, 2010 WPCL memo SWAMP QAPrP Organic Carbon Preservation Requirements

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 °C and store in the dark. If analysis is to occur more than two hours after sampling, acidify (pH < 2) with HCl or H2SO4.	28 days	Agreement with 40 CFR Section 136.3, Standard Methods 1060: Collection and Preservation of Samples, and the June 28, 2010
2013	Cool to ≤6 ∘C; acidify to pH<2 with HCl, H <sub>3</sub> PO <sub>4</sub> , or H <sub>2</sub> SO <sub>4</sub> within 2 hrs	28 days	WPCL memo SWAMP QAPrP Organic Carbon Preservation Requirements

#### Sample Handling: Conventional Parameters in Fresh and Marine Water – Organic Carbon (Total)

#### Sample Handling: Conventional Parameters in Fresh and Marine Water – Phenols

Version	Recommended Preservation	Required Holding Time	Justification
2008	Preserve to pH <2 with ~2 mL of concentrated H₂SO4; Cool to 6 °C and store in the dark	Samples must be extracted within 7 days of collection, and analyzed within 28 days of extraction.	Agreement with 40 CFR Section 136.3 and Standard Methods 1060: Collection and Preservation
2013	Cool to ≤6 °C; H₂SO₄ to pH<2	28 days	or Samples

#### Sample Handling: Conventional Parameters in Fresh and Marine Water – Silica

Version	Recommended Preservation	Required Holding Time	Justification
2008	Acidify with (1+1) $HNO_3$ to pH <2.	6 months	Addition of unacidified sample coverage from 40 CFR Section
2013	Cool to ≤6 °C; HNO₃ to pH <2	28 days; 6 months if acidified	136.3 and Standard Methods 1060: Collection and Preservation of Samples

### Conventional Parameters in Freshwater Sediment and Marine Sediment

#### Sample Handling: Conventional Parameters in Freshwater Sediment and Marine Sediment - Grain Size

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 °C in the dark up to 28 days. Do not freeze	Please refer to method	Adherence to the National Coastal Assessment Quality
2013	Wet ice to ≤6 °C in the field, then refrigerate at ≤6 °C	1 year	Assurance Project Plan, as well as the April 27, 2011 SWAMP memo Preservation and Holding Time Guidelines for Grain Size

### **Nutrients**

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 °C and store in the dark	28 days	Agreement with 40 CFR Section
2013	Filter within 15 minutes of collection; cool to ≤6 °C; H₂SO₄ to pH <2	28 days	Collection and Preservation of Samples

#### Sample Handling: Nutrients in Fresh and Marine Water – Phosphorus (Dissolved, as P)

#### Sample Handling: Nutrients in Fresh and Marine Water – Phosphorus (Total, as P)

Version	Recommended Preservation	Required Holding Time	Justification
2008	Cool to 6 °C and store in the dark	28 days	Agreement with 40 CFR Section 136.3 and Standard Methods 1060:
2013	Cool to ≤6 °C; H₂SO₄ to pH <2	28 days	Collection and Preservation of Samples

Nutrient coverage in the QAPrP has been expanded to include total nitrogen and elemental phosphorus in water. The following tables include the guidelines for these analytes.

#### Quality Control: Nutrients in Fresh and Marine Water - Nitrogen (Total)

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Calibration Standard	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications
Calibration Verification	Per 10 analytical runs	90-110% recovery
Laboratory Blank	Per 20 samples or per analytical batch, whichever is more frequent	<rl analyte<="" for="" target="" th=""></rl>
Reference Material	Per 20 samples or per analytical batch, whichever is more frequent	90-110% recovery
Matrix Spike	Per 20 samples or per analytical batch, whichever is more frequent	80-120% recovery
Matrix Spike Duplicate	Per 20 samples or per analytical batch, whichever is more frequent	80-120% recovery RPD<25% for duplicates
Laboratory Duplicate	Per 20 samples or per analytical batch, whichever is more frequent	RPD<25% (n/a if native concentration of either sample <rl)< th=""></rl)<>

#### Quality Control: Nutrients in Fresh and Marine Water - Nitrogen (Total - Continued)

Field Quality Control	Frequency of Analysis	Measurement Quality Objective
Field Duplicate	5% of total project sample count	RPD<25% (n/a if native concentration of either sample <rl)< th=""></rl)<>
Field Blank, Travel Blank, Equipment Blank	Per method	<rl analyte<="" for="" target="" th=""></rl>

#### Sample Handling: Nutrients in Fresh and Marine Water – Nitrogen (Total)

Recommended Container	Recommended Preservation	Required Holding Time
Р	Cool to ≤6 °C; H₂SO₄ to pH <2	28 days

#### Quality Control: Nutrients in Fresh and Marine Water – Phosphorus (Elemental)

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Calibration Standard	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications
Calibration Verification	Per 10 analytical runs	90-110% recovery
Laboratory Blank	Per 20 samples or per analytical batch, whichever is more frequent	<rl analyte<="" for="" target="" th=""></rl>
Reference Material	Per 20 samples or per analytical batch, whichever is more frequent	90-110% recovery
Matrix Spike	Per 20 samples or per analytical batch, whichever is more frequent	80-120% recovery
Matrix Spike Duplicate	Per 20 samples or per analytical batch, whichever is more frequent	80-120% recovery RPD<25% for duplicates
Laboratory Duplicate	Per 20 samples or per analytical batch, whichever is more frequent	RPD<25% (n/a if native concentration of either sample <rl)< th=""></rl)<>
Field Quality Control	Frequency of Analysis	Measurement Quality Objective
Field Duplicate	5% of total project sample count	RPD<25% (n/a if native concentration of either sample <rl)< th=""></rl)<>
Field Blank, Travel Blank, Equipment Blank	Per method	<rl analyte<="" for="" target="" th=""></rl>

#### Sample Handling: Nutrients in Fresh and Marine Water - Phosphorus (Elemental)

Recommended Container	Recommended Preservation	Required Holding Time
G	Cool to ≤6 °C; H₂SO₄ to pH <2	48 hours; 28 days if acidified

# **Ancillary Parameters**

### Ancillary Parameters in Freshwater Sediment and Marine Sediment

#### Quality Control: Ancillary Parameters in Freshwater Sediment and Marine Sediment – Laboratory Blank

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	n/a	n/a	Agreement with Standard
2013	One per analytical batch	Per method	Practices

#### Sample Handling: Ancillary Parameters in Freshwater Sediment and Marine Sediment

Version	Recommended Preservation	Required Holding Time	Justification
2008	Please refer to the method associated with the target analyte or parameter	Please refer to the method associated with the target analyte or parameter	Agreement with Standard Methods 1060: Collection and
2013	Cool to ≤6 °C	7 days	Preservation of Samples

#### Ancillary Parameters in Freshwater Tissue and Marine Tissue

#### Sample Handling: Ancillary Parameters in Freshwater Tissue and Marine Tissue – Moisture

Version	Recommended Preservation	Required Holding Time	Justification
2008	Please refer to the method associated with the target analyte	Please refer to the method associated with the target analyte	Agreement with Standard Methods 1060: Collection and
2013	Cool to ≤6 ℃	7 days	Preservation of Samples

## **Solid Parameters**

Version	Frequency of Analysis	Measurement Quality Objective	Justification
2008	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications	Agreement with Standard Methods
2013	None	None	Control

#### Quality Control: Solid Parameters in Fresh and Marine Water – Calibration Standard

#### Sample Handling: Solid Parameters in Fresh and Marine Water – Fixed & Volatile Dissolved Solids

Version	Recommended Preservation	Required Holding Time	Justification
2008	Refrigeration or icing to 6°C, to minimize microbiological decomposition of solids is recommended.	24 hours, maximum 7 days	Agreement with 40 CFR Section 136.3 and Standard Methods 1060: Collection and Preservation of
2013	Cool to ≤6 °C	7 days	Samples

#### Sample Handling: Solid Parameters in Fresh and Marine Water – Volatile Suspended Solids

Version	Recommended Preservation	Required Holding Time	Justification
2008	Refrigeration or icing to 6°C, to minimize microbiological decomposition of solids is recommended.	Analysis must begin as soon as possible.	Agreement with 40 CFR Section 136.3 and Standard Methods 1060: Collection and Preservation of
2013	Cool to ≤6 °C	7 days	Samples

Solids coverage in the QAPrP has been expanded to include ash-free dry mass (AFDM) in water. The following tables include the guidelines proposed for AFDM.

#### **Quality Control: Ash-Free Dry Mass in Water**

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Calibration Standard	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications
Calibration Verification	Per 10 analytical runs	80-120% recovery
Laboratory Blank	Per 20 samples or per analytical batch, whichever is more frequent	<rl analyte<="" for="" target="" th=""></rl>
Reference Material	Per 20 samples or per analytical batch, whichever is more frequent	80-120% recovery

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Matrix Spike	Per 20 samples or per analytical batch, whichever is more frequent (chlorophyll a/pheophytin a: n/a)	80-120% recovery
Matrix Spike Duplicate	Per 20 samples or per analytical batch, whichever is more frequent (chlorophyll a/pheophytin a: n/a)	80-120% recovery RPD<25% for duplicates
Laboratory Duplicate	Per 20 samples or per analytical batch, whichever is more frequent (chlorophyll a/pheophytin a: per method)	RPD<25% (n/a if native concentration of either sample <rl)< th=""></rl)<>
Internal Standard	Accompanying every analytical run as method appropriate	Per method
Field Quality Control	Frequency of Analysis	Measurement Quality Objective
Field Duplicate <sup>2</sup>	5% of total project sample count	RPD<25% (n/a if native concentration of either sample <rl)< th=""></rl)<>
Field Blank, Travel Blank, Equipment Blank	Per method	<rl analyte<="" for="" target="" th=""></rl>

<sup>1</sup> Unless method specifies more stringent requirements

<sup>2</sup> Field duplicate RPDs are not calculated for chlorophyll a or AFDM analyses for bioassessment

### Sample Handling: Ash-Free Dry Mass in Water

Recommended Container	Recommended Preservation	Required Holding Time
Glass-fiber filter	Cool to ≤6 °C (foil-wrapped); freeze to ≤-20 °C	28 days

# **Toxicity Testing**

Approved toxicity table updates include:

- Addition of the table: Acute Marine Water Testing: 96-Hour Acute Americamysis bahia Survival Test
- Addition of the table: Acute Freshwater Testing: 96-Hour Survival Hyalella azteca
   Toxicity Test
- Separation of water and sediment tables
- Reordering of test-specific tables
- Retitling of tables for clarification and consistency
- Updates to table footnotes

**Indicator Bacteria** 

[Indicator Bacteria guidelines will be added to this document when they are finalized and approved by the SWAMP Roundtable.]

# **Field Measurements**

[Field measurement guidelines will be added to this document when they are finalized and approved by the SWAMP Roundtable.]