

# Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment

Terms appearing in the tables are defined in the [Surface Water Ambient Monitoring Program Quality Assurance Program Plan](#), which contains a glossary (Appendix E), as well as a list of abbreviations and acronyms (Appendix F).

**Table 1: Quality Control<sup>1,2</sup>: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment<sup>3</sup>**

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
<b>Tuning<sup>4</sup></b>	Per analytical method	Per analytical method
<b>Calibration</b>	Initial method setup or when the calibration verification fails	<ul style="list-style-type: none"> <li>Correlation coefficient (<math>r^2 &gt; 0.990</math>) 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>4</sup></li> <li>Minimum of 5 points per curve (one of them at or below the RL)</li> </ul>
<b>Calibration Verification</b>	Per 12 hours	<ul style="list-style-type: none"> <li>Expected response or expected concentration <math>\pm 20\%</math></li> <li>RF for SPCCs = initial calibration<sup>4</sup></li> </ul>
<b>Laboratory Blank</b>	Per 20 samples or per analytical batch, whichever is more frequent	<RL for target analytes
<b>Reference Material</b>	Per 20 samples or per analytical batch (preferably blind)	70-130% recovery if certified; otherwise, 50-150% recovery
<b>Matrix Spike</b>	Per 20 samples or per analytical batch, whichever is more frequent	50-150% or based on historical laboratory control limits (average $\pm 3SD$ )
<b>Matrix Spike Duplicate</b>	Per 20 samples or per analytical batch, whichever is more frequent	50-150% or based on historical laboratory control limits (average $\pm 3SD$ ); RPD < 25%
<b>Surrogate</b>	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)
<b>Internal Standard</b>	Included in all samples and all QC samples (as available)	Per laboratory procedure

**Table 1: Quality Control<sup>1,2</sup>: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment<sup>3</sup>  
(continued)**

Field Quality Control	Frequency of Analysis	Measurement Quality Objective
<b>Field Duplicate</b>	5% of total project sample count	Per method
<b>Field Blank, Travel Blank, Equipment Blank</b>	Per method	<RL for target analytes

<sup>1</sup> Unless method specifies more stringent requirements; ELISA results must be assessed against kit requirements

<sup>2</sup> Pyrethroid quality control guidelines are presented in Table 2 immediately below

<sup>3</sup> All detected analytes must be confirmed with a second column, second technique, or mass spectrometry

<sup>4</sup> Mass spectrometry only

**Table 2: Quality Control<sup>1</sup>: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment – Pyrethroids Only**

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
<b>Tuning<sup>2</sup></b>	Per analytical method	Per analytical method
<b>Calibration</b>	Daily, or just prior to analysis; five or more level standards spanning the sample result range <sup>3</sup> , with the lowest standard at or below the RL	$r \geq 0.995$ (or $r^2 \geq 0.995$ , all curve types not forced through origin)
<b>Calibration Verification</b>	Per 10 analytical samples <sup>4</sup>	80-120% <sup>5</sup>
<b>Laboratory Blank</b>	Per 20 samples or per analytical batch, whichever is more frequent	<RL for target analytes
<b>Laboratory Control Sample<sup>6</sup></b>	Per 20 samples or per analytical batch, whichever is more frequent	50-150%
<b>Matrix Spike</b>	Per 20 samples or per analytical batch, whichever is more frequent	50-150%
<b>Matrix Spike Duplicate</b>	Per 20 samples or per analytical batch, whichever is more frequent	50-150%; RPD $\leq$ 35%
<b>Surrogate<sup>7</sup></b>	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)
<b>Internal Standard</b>	Included in all samples and all QC samples (as available)	Per laboratory procedure
<b>Field Quality Control<sup>8</sup></b>	<b>Frequency of Analysis</b>	<b>Measurement Quality Objective</b>
<b>Field Duplicate</b>	5% of total project sample count	RPD $\leq$ 35%

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

<sup>2</sup>Mass spectrometry only

<sup>3</sup>Sample results above the highest standard are to be diluted and re-analyzed.

<sup>4</sup>Analytical samples include samples only and do not include clean-out or injection blanks.

<sup>5</sup>Limit applies to a mid-level standard; low-level calibration checks near the reporting limit may have a wider range that is project-specific.

<sup>6</sup>Laboratory control samples must be matrix-specific. A clean sediment, roasted sand, or roasted sodium sulfate may be used for sediments.

<sup>7</sup>Laboratory historical limits for surrogate recovery must be submitted to the SWAMP database in the lab result comment section.

<sup>8</sup>A technical group consisting of regional, laboratory, and research representatives determined that field blanks do not add technical value to a pyrethroids data set.

**Table 3: Sample Handling: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment**

Analyte	Recommended Container <sup>2</sup>	Recommended Preservation	Required Holding Time <sup>1</sup>
<b>Diesel Range Organics</b> <b>Organochlorine Pesticides</b> <b>Organophosphate Pesticides</b> <b>Organotins</b> <b>Polynuclear Aromatic Hydrocarbons</b> <b>Surfactants</b> <b>Wastewater Organochlorine Pesticides</b>	G	Cool to ≤6 °C within 24 hours, then freeze to ≤-20 °C	1 year; samples must be extracted within 14 days of collection or thawing and analyzed within 40 days of extraction
<b>Polybrominated Diphenyl Ethers</b> <b>Polychlorinated Biphenyls</b> (as Congeners/Aroclors)	G	Cool to ≤6 °C within 24 hours, then freeze to ≤-20 °C	None
<b>Pyrethroids</b>	G	Short-term storage: ≤6 °C in the dark; long-term storage, or storage of remaining sample: ≤-20 °C in the dark	1 year at ≤-20 °C in the dark; samples must be extracted within 14 days of collection or thawing and analyzed within 40 days of extraction

<sup>1</sup> Each "Required Holding Time" is based on the assumption that the "Recommended Preservation" (or a method-mandated alternative) has been employed. If a "Required Holding Time" for filtration, preservation, preparation, or analysis is not met, the project manager and SWAMP Quality Assurance Officer must be notified. Regardless of preservation technique, data not meeting the "Required Holding Time" will be appropriately flagged in the SWAMP database.

<sup>2</sup> "G" is glass

**Table 4: Recommended Corrective Action: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment<sup>1</sup>**

Laboratory Quality Control	Recommended Corrective Action
<b>Calibration</b>	Recalibrate the instrument. Affected samples and associated quality control must be reanalyzed following successful instrument recalibration.
<b>Calibration Verification</b>	Reanalyze the calibration verification to confirm the result. If the problem continues, halt analysis and investigate the source of the instrument drift. The analyst should determine if the instrument must be recalibrated before the analysis can continue. All of the samples not bracketed by acceptable calibration verification must be reanalyzed.
<b>Laboratory Blank</b>	Reanalyze the blank to confirm the result. Investigate the source of contamination. If the source of the contamination is isolated to the sample preparation, the entire batch of samples, along with the new laboratory blanks and associated QC samples, should be prepared and/or re-extracted and analyzed. If the source of contamination is isolated to the analysis procedures, reanalyze the entire batch of samples. If reanalysis is not possible, the associated sample results must be flagged to indicate the potential presence of the contamination.
<b>Reference Material</b>	Reanalyze the reference material to confirm the result. Compare this to the matrix spike/matrix spike duplicate recovery data. If adverse trends are noted, reprocess all of the samples associated with the batch.
<b>Matrix Spike</b>	The spiking level should be near the midrange of the calibration curve or at a level that does not require sample dilution. Reanalyze the matrix spike to confirm the result. Review the recovery obtained for the matrix spike duplicate. Review the results of the other QC samples (such as reference materials) to determine if other analytical problems are a potential source of the poor spike recovery.
<b>Matrix Spike Duplicate</b>	The spiking level should be near the midrange of the calibration curve or at a level that does not require sample dilution. Reanalyze the matrix spike duplicate to confirm the result. Review the recovery obtained for the matrix spike. Review the results of the other QC samples (such as reference materials) to determine if other analytical problems are a potential source of the poor spike recovery.
<b>Internal Standard</b>	Check the response of the internal standards. If the instrument continues to generate poor results, terminate the analytical run and investigate the cause of the instrument drift.
<b>Surrogate</b>	Analyze as appropriate for the utilized method. Troubleshoot as needed. If no instrument problem is found, samples should be re-extracted and reanalyzed if possible.
Field Quality Control	Recommended Corrective Action
<b>Field Duplicate</b>	Visually inspect the samples to determine if a high RPD between results could be attributed to sample heterogeneity. For duplicate results due to matrix heterogeneity, or where ambient concentrations are below the reporting limit, qualify the results and document the heterogeneity. All failures should be communicated to the project coordinator, who in turn will follow the process detailed in the method.
<b>Field Blank, Travel Blank, Equipment Blank</b>	Investigate the source of contamination. Potential sources of contamination include sampling equipment, protocols, and handling. The laboratory should report evidence of field contamination as soon as possible so corrective actions can be implemented. Samples collected in the presence of field contamination should be flagged.

<sup>1</sup> Pyrethroid-related corrective actions are presented in Table 5 immediately below.

**Table 5: Recommended Corrective Action: Synthetic Organic Compounds in Freshwater Sediment and Marine Sediment - Pyrethroids Only**

Laboratory Quality Control	Recommended Corrective Action
<b>Calibration Standard</b>	Affected samples and associated quality control must be reanalyzed following successful instrument recalibration.
<b>Calibration Verification</b>	Initial calibration is analyzed immediately after calibration and should be from a source different than the calibration curve. Bracketing continuing calibration standards are used every ten sample runs for quantitation per method protocol. The analysis must be halted, the problem investigated, and the instrument recalibrated. All samples after the last acceptable continuing calibration verification must be reanalyzed.
<b>Laboratory Blank</b>	The sample analysis must be halted, the source of the contamination investigated, the samples along with a new laboratory blank prepared and/or re-extracted, and the sample batch and fresh laboratory blank reanalyzed. If reanalysis is not possible due to sample volume, flag associated samples.
<b>Laboratory Control Sample</b>	The LCS is analyzed in the same manner as an environmental sample and the spike recovery demonstrates the accuracy of the method. Affected samples and associated quality control must be reanalyzed following LCS troubleshooting and resolution. After troubleshooting, compare to matrix spike/matrix spike duplicate recovery data. If adverse trends are noted, reprocess all samples associated with the batch.
<b>Matrix Spike</b>	The spiking level should be near the midrange of the calibration curve or at a level that does not require sample dilution. Appropriately spiked results should be compared to the matrix spike duplicate to investigate matrix interference. If matrix interference is suspected, the matrix spike result must be flagged. Appropriately spiked results should be compared to the matrix spike duplicate to investigate matrix interference. If matrix interference is suspected and LCS recoveries are acceptable, the matrix spike and matrix spike duplicate results must be flagged.
<b>Matrix Spike Duplicate</b>	The spiking level should be should be near the midrange of the calibration curve or at a level that does not require sample dilution. Appropriately spiked results should be compared to the matrix spike to investigate matrix interference. If matrix interference is suspected and LCS recoveries are acceptable, the matrix spike duplicate result must be flagged.
<b>Surrogate</b>	Analyze as appropriate per method. Trouble shoot as appropriate, if no instrument problem is found samples should be re-extracted and re-analyzed if possible.
<b>Internal Standard</b>	Analyze as appropriate per method. Troubleshoot as appropriate. If, after troubleshooting, the responses of the internal standards remain unacceptable, the analysis must be terminated and the cause of drift investigated.
Field Quality Control	Recommended Corrective Action
<b>Field Duplicate</b>	For duplicates with a heterogeneous matrix or ambient levels below the reporting limit, failed results may be flagged. All failures should be communicated to the project coordinator, who in turn will follow the process detailed in the method.