Synthetic Organic Compounds in Fresh and Marine Water

Table 1: Quality Control[1, 2]: Synthetic Organic Compounds in Fresh and Marine Water[3]

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Tuning[4]	Per analytical method	Per analytical method
Calibration	Initial method setup or when the calibration verification fails	• Correlation coefficient (r^2 >0.990) for linear and non-linear curves • If RSD<15%, average RF may be used to quantitate; otherwise use equation of the curve • First- or second-order curves only (not forced through the origin) • Refer to SW-846 methods for SPCC and CCC criteria[4] • Minimum of 5 points per curve (one of them at or below the RL)
Calibration Verification	Per 12 hours	• Expected response or expected concentration ±20% • RF for SPCCs=initial calibration[4]
Laboratory Blank	Per 20 samples or per analytical batch, whichever is more frequent	<RL for target analytes
Reference Material	Per 20 samples or per analytical batch (preferably blind)	70-130% recovery if certified; otherwise, 50-150% recovery
Matrix Spike	Per 20 samples or per analytical batch, whichever is more frequent	50-150% or based on historical laboratory control limits (average±3SD)
Matrix Spike Duplicate	Per 20 samples or per analytical batch, whichever is more frequent	50-150% or based on historical laboratory control limits (average±3SD); RPD<25%
Surrogate	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)
Internal Standard	Included in all samples and all QC samples (as available)	Per laboratory procedure

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

[1] Unless method specifies more stringent requirements; ELISA results must be assessed against kit requirements.

[2] Pyrethroids quality control guidelines are presented in Table 2 immediately below.

[3] All detected analytes must be confirmed with a second column, second technique, or mass spectrometry.

[4] Mass spectrometry only

Table 2: Quality Control[1]: Synthetic Organic Compounds in Whole Water - Pyrethroids Only

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

Field Quality Control[8]	Frequency of Analysis	Measurement Quality Objective
Field Duplicate	5% of total project sample count	RPD ≤ 35%

[1] Unless project specifies more stringent requirements

[2] Mass spectrometry only

[3] Sample results above the highest standard are to be diluted and re-analyzed.

[4] Analytical samples include samples only and do not include clean-out or injection blanks.

[5] Limit applies to a mid-level standard; low-level calibration checks near the reporting limit may have a wider range that is project - specific

[6] Laboratory control samples must be matrix specific. A clean sediment, roasted sand, or roasted sodium sulfate may be used for sediments.

[7] Laboratory historical limits for surrogate recovery must be submitted to the SWAMP database in the lab result comment section.

[8] A technical group consisting of regional, laboratory, and research representatives determined that field blanks do not provide technical value to a pyrethroids dataset.

Table 3: Sample Handling: Synthetic Organic Compounds in Fresh and Marine Water[1]

Matrix	Recommended Container	Recommended Preservation[2]	Required Holding Time[3]
Carbamate Pesticides **Organochlorine Pesticides** **Organophosphate Pesticides** **Wastewater Organochlorine Pesticides**	Glass	Cool to ≤6 °C; pH 5-9	7 days until extraction, 40 days after extraction
Diesel Range Organics **Triazine Pesticides**	Glass	Cool to ≤6 °C	7 days until extraction, 40 days after extraction
Glyphosate	Glass	Cool to ≤6 °C; store in the dark; 0.008% $Na_2S_2O_3$ if residual chlorine is present; freeze to ≤-20 °C	18 months (14 days if unfrozen)
Phenols[4]	Glass	Cool to ≤6 °C; 0.008% $Na_2S_2O_3$ if residual chlorine is present	7 days until extraction, 40 days after extraction
Polychlorinated Biphenyls (as Congeners/Aroclors)	Glass	Cool to ≤6 °C	1 year until extraction, 1 year after extraction
Polynuclear Aromatic Hydrocarbons	Glass	Cool to ≤6 °C; store in the dark; 0.008% $Na_2S_2O_3$ if residual chlorine is present	7 days until extraction, 40 days after extraction
Pyrethroids	Glass	Cool ≤ 6 °C 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
Surfactants	Glass	Cool to ≤6 °C, store in the dark	7 days until extraction, 40 days after extraction

[1] Pyrethroids information applies to a whole water matrix.

[2] Per 40 CFR 136.3, aqueous samples must be preserved at ≤6 °C and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

[3] 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.

[4] This table applies to phenols analysis using gas chromatography. Guidelines for the colorimetric analysis of phenols are located in *Conventional Parameters in Water* Table 2*: Sample Handling.*

Table 4: Recommended Corrective Action: Synthetic Organic Compounds in Fresh and Marine Water[1]

Laboratory Quality Control	Recommended Corrective Action
Calibration	Recalibrate the instrument. Affected samples and associated quality control must be reanalyzed following successful instrument recalibration.
Calibration Verification	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 the samples not bracketed by acceptable calibration verification must be reanalyzed.
Laboratory Blank	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.
Reference Material	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 the samples associated with the batch.
Matrix Spike	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.
Matrix Spike Duplicate	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.
Internal Standard	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.
Surrogate	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.

[1] Pyrethroids corrective actions are presented in Table 5 immediately below

Field Quality Control	Recommended Corrective Action
Field Duplicate	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.
Field Blank, Travel Blank, Equipment Blank	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.

Table 5: Recommended Corrective Action: Synthetic Organic Compounds in Whole Water – Pyrethroids Only

Laboratory Quality Control	Recommended Corrective Action
Calibration	Affected samples and associated quality control must be reanalyzed following successful instrument recalibration.
Calibration Verification	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.
Laboratory Blank	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.
Laboratory Control Sample	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.
Matrix Spike	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.
Matrix Spike Duplicate	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 to investigate matrix interference. If matrix interference is suspected and LCS recoveries are acceptable, the matrix spike duplicate result must be flagged.
Surrogate	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.
Internal Standard	Analyze as appropriate per method. Troubleshoot as appropriate. If, after trouble-shooting, 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
Field Duplicate	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.

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).