Conventional Parameters in Fresh and Marine Water

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

Table 1: Quality Control: Conventional Parameters in Fresh and Marine 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
Matrix Spike	Per 20 samples or per analytical batch, whichever is more frequent (n/a for chlorophyll a and pheophytin a)	80-120% recovery
Matrix Spike Duplicate	Per 20 samples or per analytical batch, whichever is more frequent (n/a for chlorophyll a and pheophytin 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 ²	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>

¹ Unless method specifies more stringent requirements

² Field duplicate relative percent differences are not calculated for chlorophyll a analyses for bioassessment

Table 2: Sample Handling: Conventional Parameters in Fresh and Marine Water

Analyte	Recommended Container ¹	Recommended Preservation ^{2,3}	Required Holding Time ⁴
Alkalinity (as CaCO₃) ⁵	Р	Cool to ≤6 °C	14 days
Biochemical Oxygen Demand	Р	Cool to ≤6 °C; add 1 g FAS crystals per liter if residual chlorine is present	48 hours
Chemical Oxygen Demand (Titrametric)	G	Cool to ≤6 °C; H₂SO₄ to pH<2	28 days; biologically active samples should be tested as soon as possible
Chloride	Р	None required	28 days
Chlorophyll a Pheophytin a	Per method	Centrifuge or filter as soon as possible after collection; if processing must be delayed, keep samples on ice or at ≤6 °C; store in the dark	Samples must be frozen or analyzed within 4 hours of collection; filters can be stored frozen for 28 days
Cyanide (Total)	Р	Cool to ≤6 °C; NaOH to pH>10; add 0.6 g C ₆ H ₈ O ₆ if residual chlorine is present	14 days
Fluoride	Р	None required	28 days
Hardness (as CaCO₃)	Р	Cool to ≤6 °C; HNO₃ or H₂SO₄ to pH<2	6 months
Oil and Grease	G	Cool to ≤6 °C; HNO₃ or H₂SO₄ to pH<2	28 days
Organic Carbon (Dissolved)	G	Filter and preserve to pH<2 within 48 hours of collection; cool to ≤6 °C	28 days
Organic Carbon (Total)	G	Cool to ≤6 ∘C; acidify to pH<2 with HCl, H₃PO₄, or H₂SO₄ within 2 hrs	28 days
Perchlorate	P, G	Protect from temperature extremes	28 days
Phenols ⁶	G	Cool to ≤6 °C; H ₂ SO ₄ to pH<2	28 days
Silica	Р	Cool to ≤6 °C; HNO ₃ to pH<2	28 days; 6 months if acidified
Specific Conductance	Р	Cool to ≤6 °C; if analysis is not completed within 24 hours of sample collection, sample should be filtered through a 0.45 micron filter and stored at ≤6 °C	28 days
Sulfate	Р	Cool to ≤6 °C	28 days
Turbidity	Р	Cool to ≤6 °C	48 hours

¹ "P" is polyethylene; "G" is glass

² Per the draft *National Coastal Assessment Quality Assurance Project Plan* (August 2009), marine waters in plastic containers may be ultra-frozen to ≤-50 °C for a maximum of six months.

³ 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).

⁴ 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.

⁵ Marine samples for alkalinity (as CaCO₃) may be cooled to ≤6 °C for a maximum of 24 hours.

⁶ This table applies to phenols analysis using colorimetry. Guidelines for the chromatographic analysis of phenols are located in *Synthetic Organic Compounds in Water* Table 4: *Sample Handling.*

Table 3: Recommended Corrective Action: Conventional Parameters in Fresh and Marine Water

Laboratory Quality Control	Recommended Corrective Action	
Calibration Standard	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 of 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 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 of 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.	
Laboratory Duplicate	Reanalyze the duplicate samples to confirm the results. Visually inspect the samples to determine if a high RPD between the 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.	
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.	
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.	