Inorganic Analytes in Fresh and Marine Water

A list of analytes included in this category may be found in the associated QAPrPTableReference.

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[1]: Inorganic Analytes 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 for target analyte
Reference Material[2]	Per 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 Duplicate	Per 20 samples or per analytical batch, whichever is more frequent	RPD<25% (n/a if native concentration of either sample<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), unless otherwise specified by method
Field Blank, Equipment Blank	Per method	Blanks<RL for target analyte

[1] Unless method specifies more stringent requirements

[2] Not applicable to selenium speciation

Table 2: Sample Handling: Inorganic Analytes in Fresh and Marine Water

Analyte	Recommended Container[1]	Recommended Preservation[2,3]	Required Holding Time[4]
Hexavalent Chromium (Filtered)	P, G	Cool to ≤6 °C, pH 9.3 – 9.7 within 24 hours	28 days at ≤6 °C[5]
Mercury (Dissolved)	G, PA	Filter and preserve with 0.5% v:v pre-tested 5% BrCl or 12N HCl within 48 hours	90 days at room temperature following acidiciaftion
Mercury (Total)	G, PA	Preserve with 0.5% v:v pre-tested 5% BrCl or 12N HCl within 48 hours	90 days at room temperature following acidification
Methylmercury (Dissolved)[6]	G, PA	Immediately after collection, cool to ≤6 °C in the dark; filter and acidify to 0.5% with pre-tested HCl within 48 hours; if salinity is >0.5 ppt, acidify with H_2SO_4	6 months at to ≤6 °C in the dark following acidification
Methylmercury (Total)[6]	G, PA	Immediately after collection, cool to ≤6 °C in the dark; acidify to 0.5% with pre-tested HCl within 48 hours; if salinity is >0.5 ppt, acidify with H_2SO_4	6 months at to ≤6 °C in the dark following acidification
Selenium Speciation[7]	P	Filter and preserve with 0.4% HCl within 15 minutes of collection; maintain collection temperature as best as possible	6 months
Trace Metals[8] (Dissolved)	P	Filter within 15 minutes of collection; HNO_3 to pH<2 within 48 hours and at least 24 hours prior to analysis	6 months at room temperature following acidification
Trace Metals[8] (Total)	P	HNO_3 to pH<2 within 48 hours and at least 24 hours prior to analysis	6 months at room temperature following acidification

[1] "P" is polyethylene; "G" is glass; "PA" is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic)

[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 (within 15 minutes).

[3] Per 40 CFR 136.3, an aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately.

[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] If the analytical method doesn't include preservation, analysis must occur within 24 hours.

[6] Methylmercury samples may be shipped to the laboratory unpreserved if they are collected in fluoropolymer bottles, filled to the top with no head space, capped tightly, and maintained at ≤6 °C from the time of collection until preservation. The samples must be acid-preserved within 48 hours of sampling.

[7] Including the species selenite, selenate, and selenocyanate

[8] With the exception of mercury, methylmercury, hexavalent chromium, and selenium speciation

Table 3: Recommended Corrective Action: Inorganic Analytes 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 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 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, 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.