Nutrients 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 Control¹: Nutrients 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	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>

¹ Unless method specifies more stringent requirements

Table 2: Sample Handling: Nutrients in Fresh and Marine Water

Analyte	Recommended Container ¹	Recommended Preservation ²	Required Holding Time ³
Ammonia (as N)	Р	Cool to ≤6 °C; samples may be preserved with 2 mL of H ₂ SO ₄ per L	48 hours; 28 days if acidified
Kjeldahl Nitrogen (Total)	Р	Cool to ≤6 °C; H ₂ SO ₄ to pH<2	7 days; 28 days if acidified
Nitrate (as N)	Р	Cool to ≤6 °C	48 hours (unless calculated from nitrate + nitrite (as N) and nitrite (as N) analyses)
Nitrate + Nitrite (as N)	Р	Cool to ≤6 °C; H ₂ SO ₄ to pH<2	48 hours; 28 days if acidified
Nitrite (as N)	Р	Cool to ≤6 °C	48 hours
Nitrogen (Total)	Р	Cool to ≤6 °C; H ₂ SO ₄ to pH <2	28 days
Orthophosphate (Dissolved, as P; Soluble Reactive Phosphorus)	Р	Filter within 15 minutes of collection ⁴ ; cool to ≤6 °C	48 hours
Orthophosphate (Total, as P)	Р	Cool to ≤6 °C	48 hours
Phosphorus (Dissolved, as P)	Р	Filter within 15 minutes of collection; cool to ≤6 °C; H₂SO₄ to pH <2	28 days
Phosphorus (Elemental)	G	Cool to ≤6 °C	48 hours
Phosphorus (Total, as P)	Р	Cool to ≤6 °C; H ₂ SO ₄ to pH <2	28 days

¹ "P" is polyethylene; "G" is glass

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 Per 40 CFR 136.3, the immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus (i.e., that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection (i.e., within 15 minutes of collection).

Table 3: Recommended Corrective Action: Nutrients 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.	
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.	