

**Determination of Total Phosphorus in Water, Sediments, Soil and Tissue Samples**

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<b>Revision History</b>	<b>Effective Date</b>
Revision # 6	11/23/2015
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The following were editorial changes which have been incorporated the CACHE-NACF SOP-008.

<b>Document</b>	<b>Document Changes</b>	<b>Author (s)</b>
SOP Rev.10	<p>Pg. 3. MDL values were updated in Table 1.</p> <p>Pg. 4. ADM_220711 was included in the Calibration section 3.b. “As part of the calibration evaluation, calculation of the Percent Relative Error (%RE) will be performed for two of the analyses calibration levels (low and midpoint). The Relative Error, at both of these levels, shall meet the criteria of %RE low &lt;20% and %RE mid &lt; %10 and can be calculated using the following equation:</p> $RE(\%) = \left( \frac{ X'i - Xi }{Xi} \right) \times 100$ <p>Where:            Xi = True value for the calibration standard            X'i = Measured concentration of the calibration standard.</p> <p>Pg. 4. ADM_221101B was included in section 3.h. Quality Control (Secondary Standard): “CACHE-NACF purchases certified reference material standards (CRM) or standards with NIST traceability certificates as the Secondary Standards or QC standard. These standards will be prepared according to the instructions provided by the company. Based on the results for each analysis, appropriate dilution factors will be determined so that the results fall within the middle range of the calibration for each analysis. The NIST traceable QC</p>	IL YD MK

standard should be prepared every 90 days.”

Pg. 6. In order to assess method precision, the following statement was included in section 3.i.:

“For method performance evaluation during a long period of time (such as quarter or annual evaluation), precision can be calculated from the Average Deviation of the data set from a quality control standard (i.e. ICV, QC, MS, MSD, CCV) and the Mean of that standard collected during certain time period.”

$$Precision (\%) = \left( \frac{Average\ Deviation}{Mean} \right) \times 100$$

Pgs. 6 and 7. Addition of the following supportive equipment to Section 6.

- a) Mettler Toledo balance XSR104 (serial# C231562564).
- b) Scintillation vials (7 ml, 8 ml and 20 ml)
- e) 300 µl pipette.
- g) Drying Ovens (SN# 20700109, SN# 42372538), Muffle furnaces (SN# 804N0019, SN# 1126290801190117) and Hydrolyzation Oven (SN#151030521).
- h) Vortexer: Fisher brand SN# 210603018 and Corning LSE SN# 0000021111054.

Pg. 9. It was specified that the antimony potassium tartrate is a “trihydrate” in section 8.e.i.1.

Pg. 9. It was specified that the ammonium molybdate is a “tetrahydrate” in sections 8.e.i.2 and 8.e.ii.2, and that the filter used is a 0.8 µm glass fiber filter instead of a 0.7 µm glass fiber filter.

Pg. 9. Correction of DIW volume in section 8.e.i.3, from 900 mL of DIW to 800 mL DIW. Warning for the addition of acid was reworded: “Use caution when making acid solutions. Always add acid to water. The mixing of acid in water may generate heat.”

Pg 9. The recipe of Diluent (8.e.ii.1) was updated using SDS stock instead of solid SDS.

Pg. 10. The following typos were corrected:

- Section 8.e.ii.3: SDS solution (8.e.i) to (8.e.i.5)

Pg. 10. Replacement of the Secondary Standard (QC standard) in sections 8.f.ii for NIST traceable or CRM standard to be used as Secondary Standard (QC Standard) as a result of ADM\_221101B.

	<p>Pg. 11. The following statement was added to Section 10.b. (Calibration and Standardization): “Any sample result that falls outside the Linear Dynamic Range (LDR, 13.a.iv) will be diluted and re-analyzed.”</p> <p>Pg. 12. Temperature Indicating Liquid (OMEGALAQ) is no longer used to confirm if the furnace reaches 550 °C since the lab now verifies the furnace temperature with a NIST traceable thermometer.</p> <p>Pg. 14. The following typos were corrected:</p> <ul style="list-style-type: none"> <li>• Section 12.ii: QC sample (8.f.ii) to QC sample (8.h) one per 20 samples.</li> <li>• Section 12.ii: CRM sample (8.f.v) to CRM sample (8.f.iv)</li> </ul> <p>Pg. 15. Linear Calibration range in section 13.a.iii was rephrased to “The analysis of the calibration curves is performed to ensure that the resulting curve is linear and within the acceptance criteria”.</p> <p>Pg. 15. In order to assess method uncertainty, the following statement was included in section 13.a.vi.:</p> <p>“For method performance evaluation during a long period of time (such as quarter or annual evaluation), uncertainty (such as quarter or annual evaluation) is calculated from 2 times of the Standard Deviation of the data set from a quality control standard (i.e., ICV, QC, CCV) and the Mean of that standard collected during certain time period.”</p> $Uncertainty (\%) = \left( \frac{2 \times Standard\ Deviation}{Mean} \right) \times 100$ <p>Pg. 16. Description for the Data Qualifier “J” in section 13.a.ix.3 was modified from “Estimated Value, qualifier has to be accompanied by detailed explanation. Value outside the established criteria” to “Value outside the established criteria”.</p> <p>Pg. 17. Addition of the 2016 NELAC Standard as a reference in Section 18.</p>	
ADM_220711	Calculation of Percent Relative Error (%RE) for two of the analysis calibration’s points	IL
ADM_221101B	Quality Control Standard or Secondary Standard used for the analysis will be purchased from a NIST traceable provider and prepared according to the provider’s instructions.	IL
SOP Rev.9	Pg.3, MDL definition is updated to “The method detection limit (MDL) is defined as the minimum measured concentration of a substance that can be reported with 99% confidence that the measured	IL YD MK

	<p>concentration is distinguishable from method blank results”.</p> <p>Pg.3, MDL values were updated to current ones (released 07/01/21) in table 1.</p> <p>Pg.5, Percent recovery ranges for Matrix Spike were updated to 80-120%.</p> <p>Pg.9. SDS solution volume (1.7 mL) replaced the 0.3 grams of SDS</p> <p>Pg.15, Additional sentence was added to clarify that the LDR is determined annually based on the quarterly collected data.</p>	
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Method Summary of  
CACHÉ-NACF SOP-008, Rev. 8  
Prepared for Battelle NEON program

**Determination of Total Phosphorus  
in Water, Sediments, Soil and Tissue Samples**

Prepared by  
Center for Aquatic Chemistry and Environment - Nutrient Analysis Core Facility  
(CACHÉ-NACF)

## 1) Summary of Method

- a) CACHE-NACF SOP-008 follows the EPA method 365.1, Rev. 2.0 (1993) but for the sample preparation procedure CACHÉ-NACF does not use the typical ammonium persulfate digestion. Instead, CACHÉ-NACF uses a modification of the sample preparation methods described by Solórzano and Sharp (*Determination of total dissolved phosphorus and particulate phosphorus in natural waters*. Limnol. Oceanogr, 25(4), 1980, 754-758). CACHÉ-NACF SOP-008P describes the use of the standard ammonium persulfate digestion procedure for waters.
- b) Total phosphorus (TP) is determined in water, soil, sediment, and tissue samples by oxidizing and hydrolyzing all of the phosphorus-containing compounds to soluble reactive phosphate.
- c) Soluble reactive phosphate is then determined by reacting phosphate with molybdenum (VI) and antimony (III) in an acid medium to form a phosphoantimonymolybdenum complex. This complex is then reduced with ascorbic acid to form a blue colored dye. The intensity of the color is measured at 880nm photometrically.

## 2) Sample Storage

- a) Unfiltered sample bottles for TP analysis are stored in refrigerators designated for sample storage only. Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples are maintained at 2-6 °C and may be held for up to 28 days.

## 3) Water sample preparation

Sample preparation follows method described in Solórzano and Sharp (*Determination of total dissolved phosphorus and particulate phosphorus in natural waters*. Limnol. Oceanogr, 25(4), 1980, 754-758) with minor modifications.

## 4) Analysis

Every analytical batch (20 samples) include:

- S6 as SYNC or primer to mark the start of analysis, two carryover (CO), reagent blanks (RB), calibration curve standards in **decreasing order of concentration**, MB, ICV, QC, UMS(D) and MS(D).
- First set of 10 samples
- Samples are bracketed by CCVs. Each bracket includes no more than 10 samples plus one analytical replicate at the end of each bracket. RB and CCVs are run every 10 samples to monitor baseline and intra-run calibration drifts.
- Second set of 10 samples
- If running multiple batches of samples, a set of quality control samples including MB, QC, UMS(D), MS(D) are required for every 20 samples