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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) CACHÉ-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