

**Standard Operating Procedure**  
**Suspended Solids – Dissolved and Total Phosphorus in Runoff**  
**Followed by Inductively Coupled Plasma (ICP) Spectrometry analysis**  
Soil Environmental Chemistry Program, The Ohio State University  
Version 11

Project/Client

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Sample Description

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### **1.0 Scope of Method**

- 1.1 This method is an autoclave method for the determination of total phosphorus (dissolved + sediment bound P) in Runoff water. The samples are brought to 121°C and held for 60 minutes in the presence of sodium persulfate and concentrated H<sub>2</sub>SO<sub>4</sub>.
- 1.2 0.45um filtration for determination of dissolved N and P.

### **2.0 Definitions**

- 2.1 Duplicate Samples: A duplicate test involves splitting a sample two sub-samples and processing each through the same sample preparation procedure in order to determine the precision of the method.
- 2.2 Pre-digestion Spike: A duplicate sample is spiked prior to digestion with dihydrogen potassium phosphate (KH<sub>2</sub>PO<sub>4</sub>) in order to evaluate of the sample matrix on digestion recovery of P.
- 2.3 Preparation Blank: The Preparation Blank is a sample that contains only the reagents used in the extraction procedure. The preparation blanks is processed through the same preparation procedures as the samples and therefore gives an indication of any contamination picked up during the sample preparation process.
- 2.4 Control Sample: A water sample of known N and P concentration prepared by adding approximately 0.01g a soil with known total N (by dry combustion) and total P (by US EPA 3051a) to 10 mL of deionized water.
- 2.5 ICP-AES: Inductively Coupled Plasma-Atomic Emission Spectrometry.

### **3.0 Equipment and Supplies**

- 3.1 Autoclave
- 3.2 Trace metal grade H<sub>2</sub>SO<sub>4</sub>
- 3.3 2-10 ml pipette
- 3.4 100-1000ul Pipette
- 3.5 ≥18 MΩ deionized water (DI).
- 3.6 ACS grade sodium persulfate
- 3.7 16x100 mm culture tube with caps
- 3.9 0.45um nylon syringe filters

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3.10 syringes

#### **4.0 Water sample collection from ISCO bottles**

4.1 Shake ISCO sampler bottle vigorously to dislodge sediment from bottom of bottle.

4.2 Insert overhead stirring and sampling apparatus into sample. Turn on motor and allow stirring to create a homogeneous suspension.

4.3 Pull approximately a 100mL sample into 125mL bottle using the apparatus.

4.4 Rinse out apparatus between each sample.

#### **5.0 Procedure**

5.1 Prepare Digestion solution. Good for 1 month.

4.1a Make 0.4M  $\text{Na}_2\text{S}_2\text{O}_8$  digestion solution.

5.2 Check pipettes for accuracy with 5 aliquots of DI water and record in Wdrive>SEC Lab> Pindex water>P Index water samples.xls

5.2a  $10.0 \pm 0.2\text{ml}$

5.2b  $0.250 \pm 0.0025\text{ml}$

5.2c  $1.0 \pm 0.05\text{ml}$

5.3 Prepare control sample (1g/L) by weighing  $0.01\text{g} \pm 0.002$  directly into culture tube. Pipette  $10.0 \pm 0.2\text{ml}$  of DI water into culture tube. Record exact mass of soil to four decimal places in water sample tracking sheet (Wdrive>SEC Lab> Pindex water>P Index water samples.xls)

5.5 Mix bulk water sub-samples by overhead stirring.

5.6 Pipette  $10.0 \pm 0.2\text{ml}$  of sample into culture tube.

4a Rinse stirring paddles with DI water in between each sample.

4b Rinse pipette tip with DI water between each sample.

5.7 While samples are stirred, 0.45um nylon syringe filter  $10.0 \pm 2$  of sample into labeled falcon tube. After filtration, add 1-2 drops of concentrated trace metal grade HCl.

Note: Digestions batches can be completed before filtering for dissolved. However, do not process more than 3 batches of digestions before returning to samples to filter for dissolved.

5.8 Add  $0.250 \text{ mL} \pm 0.005\text{mL}$  of 200 mg/L P solution to pre-digestion spike sample in culture tube.

5.9 Add  $1\text{mL} \pm 0.01\text{ml}$  of digestion solution to culture tubes.

5.10 Add  $0.250 \text{ mL} \pm 0.005\text{mL}$  of concentrated  $\text{H}_2\text{SO}_4$ .

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- 5.11 Autoclave culture tubes for 60 min at 121 C (approx. 12-15 psi) under the liquid setting.  
4.9a Verify that the samples are held at 121 C for 60 mins.

### **5.0 Quality Control**

- 5.1 Laboratory Control Sample (LCS): The laboratory control sample must fall within  $\pm 20\%$  of the known value. The laboratory control sample must be run with each batch of digestions.
- 5.2 Sample Duplicates: The relative percent difference (RPD) must be no more than  $\pm 20\%$ . One sample duplicate must be run with every microwave batch.

$$RPD = 100 \times \frac{(S - D)}{\text{Avg. (S,D)}}$$

- 5.3 Preparation Blank: If any analyte concentration is above the detection limit in the preparation blank, the lowest concentration of the analyte reported in associated samples must be  $\geq 10$  times the preparation blank concentration. A preparation blank must be performed with each batch of microwave digests.
- 5.4 Pre-digestion Spike: Spike recoveries must fall within the limits of 75-125%. At least one spike analyses (matrix spikes) shall be performed on each batch of digests

### **6.0 Sample Storage and Disposal**

- 6.1 Once 100 mL sub-samples are processed for dissolved and total, bottles should be moved to downstairs storage until the final dataset for the calendar year is submitted (usually Feb or March of the following year)
- 6.2 Acidified filtered samples should be moved to downstairs storage indefinitely.
- 6.3 Once total P samples have been run on ICP, the digestions should be disposed of.

**Standard Operating Procedure**  
**Persulfate Digestion for Total Phosphorus in Runoff**  
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Appendix

Pipette Calibration  
Verification

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

Volume	g DI	g DI	g DI	g DI	g DI	date	initials

spike preparation

Pre-  
digestion

