Standard Operating Procedure
Dissolved Phosphorus and Nitrogen in Water and
USGS Persulfate Digestion for Total Phosphorus and Nitrogen in Water

Soil Environmental Chemistry Program, The Ohio State University

Version 2

Project/Client

Sample Description

Batches

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1.0 Scope of Method

1.1 1.1 Persulfate digestion for determination of total nitrogen (organic +NH_4 + NO_2 + NO_3 N) and total phosphorus (dissolved + sediment bound P) in runoff water and soil/sediment extracts.

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2.0 Definitions

2.1 Duplicate Samples: A duplicate test involves splitting a sample into two sub-samples and processing each through the same sample preparation procedure in order to determine the level of homogenization and therefore representativeness of the water sample collected.

2.2 Pre-digestion Spike: A duplicate sample is spiked prior to digestion with KNO_3 and dihydrogen phosphate (KH_2PO_4) in order to evaluate of the sample matrix on digestion recovery of NO_3 and PO_4.

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 2.4 Control Sample: A water sample of known N concentration prepared by adding approximately 0.01g a soil with known total N (by dry combustion).

3.0 Equipment and Supplies

3.1 Autoclave

3.2 Sodium Hydroxide

3.3 ACS grade KNO_3

3.4 ACS grade KH_2PO_4

3.5 2-10 ml pipette

3.6 100-1000ul Pipette

3.7 ≥18 MΩ deionized water (DI)

3.8 ACS grade potassium persulfate

3.9 20x125 mm culture tube with caps

3.14 2mL IC autosampler vials.

4.0 Procedure

4.1 Prepare Digestion solution. Record in appendix A.
4.1a Make 1.5M NaOH, transfer to a screw top bottle, and date (good for 6 mo).
4.1b. Make 0.15M NaOH, 0.15M $K_2S_2O_8$ digestion solution with 1.5M NaOH stock solution and ACS grade potassium persulfate (good for 24 hours).

4.2 Check pipettes for accuracy with 5 aliquots of DI water and record in appendix:
4.2a 10.0 ± 0.2ml
4.2b 0.250 ± 0.0025ml
4.2c 5.0 ± 0.05ml

4.3 Prepare control sample (1g/L) by weighing 0.01g±0.002 directly into culture tube. Pipette 10.0 ± 0.2ml of DI water into culture tube. Record exact mass of soil to four decimal places in analysis sheet.

4.4 Mix bulk water sample by overhead stirring.

4.5 Pipette 10.0 ± 0.2ml of sample into culture tube.
4.a Rinse stirring paddles with DI water in between each sample.

4.6 Add 0.250 mL ± 0.005mL of 200 mg/L NO_3-N to pre-digestion spike sample in culture tube.

4.8 Add 5mL ± 0.01ml of digestion solution to culture tubes.

4.9 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 ± 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 ±20%. One sample duplicate must be run with every microwave batch.

$$RPD = 100 \times \frac{(S - D)}{}$$

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 ≥ 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 total N, bottles should be moved to downstairs storage until data is uploaded into access for the project.

6.3 Once total N samples have been run successfully on Lachat, the digestions should be disposed of.

7.0 Reporting

7.1 Worksheets

7.1a Fill in appendix for pipettes used during the course of this SOP.
7.1b Provide masses and volumes used to prepare digestion solution.
7.1c Flag data where appropriate according to codes in appendix.

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Appendix

Batches

Digestion solution Preparation:

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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

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

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

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Pass/ Fail	Flag	Measurement	QA/QC Check[1]	Frequency	Acceptance Criteria	Corrective Action
	i	Method	LCS	1/batch	±20% or w/in 95% PI	Check microwave function and re-digest batch.
	ii	Sample prep	Blank	1/batch	Below MDL or samples >10x	Check ICP for carryover and dish washing procedures re-digest batch.
	iii	Reproducibility	Duplicate	1/batch	RPD ±20%	Check microwave function and re-digest batch.
	Iv	Method/ Matrix affects	Pre-Digest Spike	1/batch	±25%	Check microwave function and ICP for signs of matrix affects. Re-digest batch if ICP is acceptable.