



**Protocol for Acid Persulfate Digest for Soil Total Phosphorus**

*Equipment*

- 1) 15 ml screw-cap pyrex culture tubes (teflon-lined caps)
- 2) 1000 ul pipettor (blue tips)
- 3) 5000 ul pipettor (white tips)
- 4) 25 ml plastic scintillation vials
- 5) 125 ml Nalgene screw-cap mixing jars
- 6) Mettler 4-place balance
- 7) Weighing paper
- 8) Autoclave

*Reagents*

- 1) Deionized water (diH<sub>2</sub>O)
- 2) Potassium Persulfate K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (e.g. Fisher P282-100)
- 3) 5.5M Sulfuric acid:  
Slowly add 308.0 ml conc. H<sub>2</sub>SO<sub>4</sub> to 500 ml diH<sub>2</sub>O in 1000 ml volumetric (gets hot!)  
Allow to cool  
Bring to volume  
Cap with parafilm, invert to mix and allow to stand 30 min.  
Recheck volume
- 4) 8.0M Sulfuric acid:  
Slowly add 440.8 ml conc. H<sub>2</sub>SO<sub>4</sub> to 500 ml diH<sub>2</sub>O in 1000 ml volumetric (gets hot!)  
Allow to cool  
Bring to volume  
Cap with parafilm, invert to mix and allow to stand 30 min.  
Recheck volume
- 5) Potassium sulfate/sulfuric acid solution:  
700 ml diH<sub>2</sub>O  
Add 7.36 g K<sub>2</sub>SO<sub>4</sub> and mix  
Add 25.0 ml 8.0M H<sub>2</sub>SO<sub>4</sub> and mix  
Bring to volume  
Cap with parafilm, invert to mix and allow to stand 30 min.  
Recheck volume
- 6) N.B.S. soil standards, e.g. NBS 2710 Montana agricultural soil

*Procedure*

Day 1

- 1) Dry soil/sediment sample 60°C for 24 hours
- 2) Grind in Spex mill to correspond to NIST soil standard texture
- 3) Weigh approx. .0250 g (~25 mg) ground sample; record weight
- 4) Transfer sample to digest tube
- 5) Pipette 500 ul diH<sub>2</sub>O into digest tube
- 6) Pipette 500 ul 5.5M H<sub>2</sub>SO<sub>4</sub> into digest tube (BEWARE of etching the pipettor; clean well!)
- 7) Weigh 0.200 g K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and add to digest tube
- 8) Cap tube tightly and autoclave 1 hour (liquid cycle)
- 9) After cooling, rinse digest tube contents into scintillation vial using 15 ml diH<sub>2</sub>O
- 10) Cap vial, mix and allow particulates to settle overnight.
- 11) NOTE: it is necessary also to prepare digested reagent blanks by above process.



Day 2

- 1) Take 5.0 ml of clear supernatant solution from scint. vial; mix with 10.0 ml acid matrix (K<sub>2</sub>SO<sub>4</sub>-H<sub>2</sub>SO<sub>4</sub> solution)
- 2) Analyze by Alpchem continuous-flow colorimetry: ortho-PO<sub>4</sub> manifold, acid matrix (K<sub>2</sub>SO<sub>4</sub>-H<sub>2</sub>SO<sub>4</sub> solution) as standard matrix and wash solution.
- 3) Alternatively to the manual dilution in 1), run scintillation vial contents directly on Alpchem using 1:3 dilution loop.
- 3) Calculate dry sample content from measured dilute extract values, taking reagent blank absorption into account of this analyzes as non-zero.
- 4) As recovery check, digest known NBS agricultural soil standards in parallel to unknowns and analyze as above.

*Post-analysis calculations*

Calculations for deriving soil solid-sample P content from a diluted extract:

Sample material: NBS 2710 Montana agricultural soil

Known P content: 0.106% TP = .106 parts P in 100 parts soil  
= 0.0265 mg P per 0.0250 g soil

Weight extracted: 0.0250 g = 25.00 mg

Digest liquid volume: 0.5000 ml diH<sub>2</sub>O (500.0 ul)  
0.5000 ml 5.5M H<sub>2</sub>SO<sub>4</sub> (500.0 ul)

15.00 ml diH<sub>2</sub>O (2-place repipettor)

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16.00 ml digest liquid

Concentration of digest liquid

.025 g soil in 16.00 ml digest liquid

= 0.0265 mg P in 16.00 ml digest liquid

= 1.656 mg/L P in undiluted digest liquid

Predicted concentration of diluted Alpchem sample

5.000 ml (5000 ul) sample digest liquid added to 10.00 ml acid matrix

initial volume = 5.000 ml original digest liquid

final volume = 15.0 ml diluted liquid sample

$$\text{dilution factor} = \frac{\text{final volume}}{\text{initial volume}} = \frac{15.00}{5.000} = \frac{3}{1} = 3:1$$

$$\frac{1.656 \text{ mg P}}{1 \text{ L extract}} * \frac{1}{3} = \frac{1.656 \text{ mg P}}{3 \text{ L Alp sample}}$$

$$= 0.5521 \text{ mg/l in digest}$$



Reverse calculation from Alpkem determined value

P conc. of undiluted digest liquid =  
(Alp determined P conc. of diluted sample) \* dilution factor

$$\frac{0.5521 \text{ mg Alp P}}{1 \text{ L diluted sample}} * \frac{3}{1}$$

= 1.656 mg/l P in undiluted digest liquid  
= 0.0265 mg P in 16.00 ml undiluted digest liquid

Change of state, liquid extract to solid material

Since 16.00 ml of undiluted extract should contain all the P from 0.0250 g (25.00 mg) of soil, a direct substitution is possible:

0.0265 mg P in 16.00 ml undiluted digest liquid  
= 0.0265 mg P in 0.0250 g (25.00 mg) soil

N.B. Weight of analyte (P) divided by actual weight (i.e. slightly more or less than 0.5g) of dry material extracted gives proportion (weight to weight) of analyte in the dry material:

$$\frac{A \text{ mg P}}{B \text{ g soil}} = \frac{X \text{ mg P}}{1.0 \text{ g soil}}$$

$$\frac{A}{B} \text{ mg per g} = X \text{ mg P per g soil}$$

= 1.060 mg P in 1.000 g soil  
= 0.106% TP in Montana soil

#### References

Nelson, N. S. 1987. An acid-persulfate digestion procedure for determination of phosphorus in sediments. Commun. in Soil Sci. Plant Anal. v.18 no.4 p.359-69.