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 (diH2O)
2) Potassium Persulfate K2S2O8 (e.g. Fisher P282-100)
3) 5.5M Sulfuric acid:
   Slowly add 308.0 ml conc. H2SO4 to 500 ml diH20 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. H2SO4 to 500 ml diH20 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 diH20
   Add 7.36 g K2SO4 and mix
   Add 25.0 ml 8.0M H2SO4 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 60OC 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 diH2O into digest tube
6) Pipette 500 ul 5.5M H2SO4 into digest tube (BEWARE of etching the pipettor; clean well!)
7) Weigh 0.200 g K2S2O8 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 diH2O
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 (K2SO4-H2SO4 solution)
2) Analyze by Alpkem continuous-flow colorimetry: ortho-PO4 manifold, acid matrix (K2SO4-H2SO4 solution) as standard matrix and wash solution.
3) Alternatively to the manual dilution in 1), run scintillation vial contents directly on Alpkem 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 diH2O (500.0 ul)
                       0.5000 ml 5.5M H2SO4 (500.0 ul)
                       15.00 ml diH2O (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 Alpkem 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

\[
\text{dilution factor} = \frac{15.00}{5.000} = 3:1
\]
\[
\frac{1.656 \text{ mg P}}{1 \text{ L extract}} \times 3 = \frac{4.968 \text{ mg P}}{3 \text{ L Alp sample}}
\]
\[
= 0.5521 \text{ mg/l in digest}
\]
Reverse calculation from Alpkem determined value

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

\[
\frac{0.5521 \text{ mg Alp P}}{1 \text{ L diluted sample}} \times \frac{3}{1} = 1.656 \text{ mg/l P in undiluted digest liquid}
\]

= 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:

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\frac{0.0265 \text{ mg P in 16.00 ml undiluted digest liquid}}{0.0250 \text{ 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:

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

\[
A \quad \text{--- mg per g} = \quad X \text{ mg P per g soil}
B
\]

= 1.060 mg P in 1.000 g soil

= 0.106% TP in Montana soil

References