84-016 TOTAL PHOSPHORUS acid digestion

1. Application

1.1 Total phosphorus is determined spectrophotometrically in solutions obtained by dissolving the soil sample with acid or alkali. For some soils, acid digestion is reported to result in slightly low P values, due perhaps to undissolved mineral grains containing P. However, for most soils, acid digestion is suitable and it is more convenient than alkali fusion.

2. Apparatus

2.1 Perchloric acid fume hood.

2.2 Spectrophotometer.

2.3 100 mL tall form teflon beakers.

2.4. 100 mL and 50 mL volumetric flasks.

2.5 Repipet dispensing bottles (accuracy 1%, reproducibility 0.1%).

2.6 Hot plate.

3. Reagents

3.1 Concentrated nitric acid HNO_3.

3.2 Perchloric acid $HClO_4$ 60%.

3.3 0.5N HCl (41 mL concentrated HCl per liter).

3.4 5N H_2SO_4 (139 mL concentrated H_2SO_4 per liter).

3.5 p-nitrophenol.

3.6 Stock solution A: Dissolve 12 g of ammonium molybdate in 250 mL of distilled water. In 100 mL of distilled water dissolve 0.2908 g antimony potassium tartrate. Add both of these solutions to 1000 mL of 5N H_2SO_4. Make to 2000 mL with distilled water and store in a dark pyrex bottle in a cool compartment.

3.7 Solution B: Dissolve 1.056 g of ascorbic acid in 200 mL of solution A and mix thoroughly. Prepare this solution B daily as required, it is stable for only 24 hours.

3.8 Certified atomic absorption standards $\pm$1%. Alternatively the P standard may be prepared as follows: Dilute 0.4393 g of oven dry KH_2PO_4 to 1 liter in a volumetric flask with distilled water. The concentration is 100 µg/mL.

4. Procedure

4.1 Digestion

4.1.1 Accurately weigh 1.000 g of soil (80 mesh or finer) into a 100 mL tall form teflon beaker. A reagent blank should be run through the digestion process.

4.1.2 Add 20 mL concentrated HNO_3, cover with a watch glass and heat (130°C) to oxidize organic matter.
NOTE: The following step must be done in a perchloric acid fumehood.

4.1.3 Add 10 mL of 60% $HClO_4$ and digest at 200°C until dense white fumes appear, use a little extra $HClO_4$ to wash down the sides of the beaker as necessary, continue heating for an additional 10-15 minutes, cool the solution and dilute to about 25 mL with warm distilled water.

4.1.4 Filter through a Whatman No. 41 filter paper into a 100 mL volumetric flask. Wash the residue with 0.5N HCl. The residue may be discarded or saved for further analysis.

4.1.5 Make the digestions to volume (100 mL) with 0.5N HCl.

4.2 Colorimetric determination of P.

4.2.1 Pipette aliquots of the digests containing up to 1 µg/mL P into 50 mL volumetric flasks, add distilled water to make 35 mL.

4.2.2 If necessary acidify (use 5N H_2SO_4) to pH 5.0 with p-nitrophenol (colorless in acidic, yellow in alkaline solution). This will generally not be necessary for soil extracts since the P is dissolved in 0.5N HCl.

4.2.3 Add 10 mL of reagent B and add distilled water to bring to a final volume of 50 mL, mix, allow 10 minutes for color development and read at 690 or 880 mµ (there are two absorption maxima, the one at 880 is a little more intense).

4.2.4 Prepare a standard curve by making up a range of P standards with concentrations of 0-1 µg/mL in the same manner as above.

5. Calculations

5.1 $$\text{Total P\%} = \frac{\mu\text{g/mL P in final sol'n}}{\text{sample weight (mg)} \times 1000} \times \frac{100}{\text{aliquot (mL)}} \times 100$$

$$= \frac{\mu\text{g P in final sol'n} \times 10}{\text{sample wt. (mg)} \times \text{aliquot (mL)}}$$

6. Precision

6.1 Insufficient data available.

7. References

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7.4 Syers, J.K., Williams, J.D.H., Tyner, E.H. and Walker, T.W. 1969. Primary and secondary origin of "non-extractable" soil inorganic phosphorus. Soil Sci. Soc. Am. Proc. 35, 635-636.

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Notes