

Protocol for HCl extraction of total, inorganic, and organic P from soils

Purpose

To obtain the approximate magnitude of refractory and bioavailable P fractions

Soil preparation

- Samples should be oven-dried prior to analysis (60°C) or air-dried and with an estimate of the percent moisture difference between air-dry and oven-dry soils.
- Grind samples to <125 µm

Reagents

1 N HCl solution – add 85.3 mL concentrated HCl to 1 L D.I. H₂O CAUTION! – always pour acid into water!

Equipment and Supplies

- Muffle furnace
- Shaker box or shaker table
- Analytical balance
- Acid washed glassware and 50 mL centrifuge tubes
- Centrifuge

Procedure (I suggest that all samples be run in duplicate)

1. Preheat muffle furnace to 550°C.
2. Add 0.3 g of dry ground sediment to 125 mL erlenmeyer flask for total P determination.
3. Add 0.3 g of dry ground sediment to 50 mL centrifuge tube for inorganic P determination.
4. Place flasks into the preheated muffle furnace for 2 hours
5. Samples must remain in the furnace for 2 hours AFTER temperatures reach
6. 550°C (when samples are put into the furnace, the temperatures will decrease).
7. Remove flasks from furnace and allow them to return to room temperature.
8. Add 30 mL of 1 N HCl to all Erlenmeyer flasks and centrifuge tubes.
9. Cover Erlenmeyer flasks with Parafilm and screw tops on centrifuge tubes.
10. Place Erlenmeyer flasks and centrifuge tubes in the shaker box (or on a shaker table) at a rate that allows samples to mix thoroughly.
 - a. Shake samples for 16 hours at room temperature.
11. Transfer the samples in the erlenmeyer flasks to 50 mL centrifuge tubes, keeping track of the treatments.
12. Centrifuge all samples for 10 minutes at 8000 rpm.
13. Filter the extractant through 0.45µm filters (Whatman #42).
14. Discard the first 2ml and collect remaining filtrate for analysis.
15. Store samples at room temperature until ready for analysis on LACHAT using molybdate-blue method

References

- Aspila KI, Agemian H & Chau ASY. 1976. A semi-automatic method for the determination of inorganic, organic and total phosphate in sediments. *Analyst* 101:187-197
- Levi, ET, Schlesinger WH. 1999. A comparison of fractionation methods for forms of phosphorus in soils. *Biogeochemistry* 47:25-38.