



Protocol for Double Acid Extraction for Metals and Available P

This extraction procedure was developed for use with acid, sandy soils found in the southeastern U.S., having less than 5% organic matter

Equipment

- 1) 125 ml screw-cap Nalgene extraction bottles
- 2) mechanical shaker
- 3) Whatman #1 filter paper (15 cm dia.)
- 4) glass funnels (8 cm.) and funnel racks
- 5) scintillation vials with plastic (not metal-lined) caps
- 6) 2-liter volumetric flask
- 7) automatic pipetter, variable volume (1-10 ml)

Reagents

- 1) concentrated HCl
- 2) concentrated H₂SO₄
- 3) fresh distilled deionized water
- 4) Quality-control samples: NBS 2709, 2710, 2711 standard soils

Double Acid Extraction Solution

Place ~1 L deionized H₂O and a stir bar in the 2 L volumetric flask on stir plate. Add 8 ml concentrated HCl and 1.4 ml concentrated H₂SO₄ and mix. Remove stir bar and bring to full volume with deionized H₂O. Cap with doubled parafilm and invert several times to mix. Let stand ~30 min. and re-check volume. The resulting extract solution is 0.05N HCl and 0.025N H₂SO₄.

Procedure

- 1) Label extraction bottles and scintillation vials to correspond to original sample containers.
- 2) Scoop 5 g (alternatively, 4 cubic cc. volume) of sample material into an extraction bottle
- 3) Add 20 ml extraction reagent
- 4) Place all extraction bottles on rack of mechanical shaker and shake at 250 cycles per minute for 5 minutes.
- 5) Pour the extract solution in each bottle through a funnel lined with filter paper; capture the filtrate in labelled scintillation vials. Cap and refrigerate.
- 6) Analyze filtrate for metals by atomic absorption spectrophotometry; for extracted orthophosphate by Alpkem (colorimetric, automated Murphy-Riley)

Post-analysis calculations:

- 1) Expected extract content of NBS 2711 standards (for choosing suitable calibrant ranges):

NBS certified content:

Ca 2.88% (+/- 0.08%) by weight
K 2.45% (+/- 0.08%) " "
Mg 1.05% (+/- 0.03%) " "
P 0.086% (+/- 0.007%) " "

5.0 g sample contains:

Ca 0.144 g = 144 mg in 20 ml extract = 7200 mg/liter
K 0.123 g = 123 mg " " " " = 6150 "
Mg 0.053 g = 53 mg " " " " = 2650 "
P 0.004 g = 4 mg " " " " = 200 "



2) Back-calculation of dry soil content from determined extract values:

$$\text{Soil content} = \frac{\text{(determined analyte mg.)}}{\text{1 liter extract sol'n}} \times \frac{\text{(volume of extract sol'n)}}{\text{(weight of soil extracted)}}$$

Example:

$$\frac{(7200 \text{ mg. Ca})}{1000 \text{ ml}} \times \frac{(20 \text{ ml sol'n})}{(5.0 \text{ g soil})} = \frac{28.8 \text{ mg Ca}}{1 \text{ g soil}} = \frac{.0288 \text{ mg Ca}}{1 \text{ mg soil}} = 2.88 \% \text{ Ca by weight}$$

If any dilution of the extract solution is necessary to obtain samples within the analytical range of the instruments, this dilution must also be accounted for. The dilution factor df is

$$\text{df} = \frac{\text{final volume after dilution}}{\text{initial volume before dilution}}$$

and the colorimetrically determined content of the diluted sample must be multiplied by the inverse of the df. (The Alpkem driver software does this automatically when using a dilution loop to apply the same dilution to an entire sample run.)

References:

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