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  "

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

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

Example:

\[
\begin{align*}
\frac{7200 \text{ mg. Ca}}{1000 \text{ ml}} \times \frac{28.8 \text{ mg}}{5.0 \text{ g soil}} &= \frac{2880 \text{ mg Ca}}{50 \text{ g soil}} \\
&= 0.288 \text{ mg Ca per g soil}
\end{align*}
\]

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: