DATE: SEPT 2004

Exchangeable Cations (Ca^{++}, Mg^{++}, K^{+}, Na^{+})

1. Application

This procedure covers the extraction and analysis of exchangeable cations (Ca^{++}, Mg^{++}, K^{+}, and Na^{+}) in soil.

2. Summary of methods

Exchangeable cations are extracted from the soil using an extracting solution (1 N NH_4OAc) at pH 7.0. The extracted solution is then analyzed by AA (atomic absorption) for the soil cations.

3. Safety

Each chemical compound should be treated as a potential health hazard. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material handling data sheets should be made available to all personnel involved in the chemical analysis.

4. Interferences

Ca, Mg, K and Na are partially ionized in the nitrous oxide–air acetylene or air acetylene flame of AA. To suppress ionization, cesium nitrate or chloride solution is added to give a final concentration of 1000 ppm in all solutions including the standards and blank. The purest available cesium compound must be used to avoid potassium contamination.

5. Apparatus and Materials

5.1 Soil scoop calibrated to hold 1.5 g of light-colored silt loam soils.
5.2 Erlenmeyer flasks (50 ml).
5.3 Constant suction pipette apparatus (15 ml).
5.4 Time-controlled oscillating shaker (Eberbach) set at 160 excursions per minute.
5.5 Filter paper (9 cm Whatman No. 2 or equivalent).
5.6 Acid washed filter paper (9 cm Whatman No. 2 or equivalent).
5.7 Funnel tubes (15 ml)
5.8 Disposable plastic test tubes (13x100).
5.9 Atomic absorption spectrophotometer (AA), (Varian SpectrAA 220 FS with SIPS pump unit and auto sampler SPS-5).

6. Reagents

6.1 Extracting solution (1 N NH_4OAc; add 57 ml glacial acetic acid to 800 ml of deionized water in a volumetric flask. Mix thoroughly and slowly add 67 ml of concentrated NH_4OH. Mix and cool to room temperature. Adjust the pH of the solution to 7.0 by adding acetic acid or NH_4OH and dilute to 1 liter).
6.2 10,000 ppm cesium chloride solution (12.67g cesium chloride [ultra configuration grade] in 1 liter of 1% HNO_3).
6.3 10,000 ppm Ca stock solution
6.4 10,000 ppm Mg stock solution
6.5 10,000 ppm K stock solution
6.6 10,000 ppm Na stock solution
6.7 40 ppm Ca/Mg bulk solution (2 ml each 10,000 ppm Ca and 10,000 ppm Mg stock solution diluted to 500 ml with 1 N NH_4OAc)
6.8 15 ppm Exchangeable K bulk solution (.75 ml 10,000 ppm K stock solution diluted to 500 ml with 1 N NH_4OAc)
6.9 15 ppm Na bulk solution (.75 ml 10,000 ppm Na stock solution diluted to 500 ml with 1 N NH_4OAc)

7. Methods

7.1 Place a 1.5 g scoop of soil into a 50-ml Erlenmeyer flask.
7.2 Add 15 ml of extracting solution (1 N NH_4OAc, pH 7.0) by constant suction pipette.
7.3 Shake the suspension on an oscillating shaker for 15 minutes.
7.4 Filter through Whatman No. 2 filter paper into 15-ml funnel tubes. Acid washed filter papers should be used for Na extrations.
7.5 Determine Ca, Mg, K and Na in the filtered extract via AA spectrophotometry, using a bulk standard containing 40 ppm of Ca / Mg (run simultaneously); 15 ppm of K; or 15 ppm of Na respectively; which is diluted by the AA to make as many standards as the user specifies.

Note: Ca, Mg determinations are made using a nitrous oxide–acetylene flame. To suppress ionization for all of these elements, cesium chloride solution is added to all samples, blanks and standards to give a final concentration of 1000 ppm using the SIPS pump unit.

8. Calculations

Any necessary weight to volume dilutions are performed by computer during analysis, (in this case ppm in soil x 10).

9. Quality Control

9.1 Laboratory Reagent Blank (LRB) – At least one LRB is analyzed with each batch of samples to assess contamination from the laboratory environment. Contamination from the laboratory or reagents is suspected if the LRB values exceed the detection limit of the method. Corrective action must be taken before proceeding.

9.2 Standard soil – One or more standard soils of known extractable Ca, Mg, and content is analyzed with each batch of samples to check instrument calibration and procedural accuracy.

10. Reporting

Results are reported as ppm of exchangeable Ca, Mg, K and Na in soil. If the results are to be used to estimate cation exchange capacity (CEC), convert ppm of each cation to meq/100g of soil, then sum the values for the four cations:

$$\text{Estimated CEC} = \left[\frac{\text{Ca (ppm)}}{200} + \frac{\text{Mg (ppm)}}{122} + \frac{\text{K (ppm)}}{391}\right] \times \frac{\text{(soil + tare) – tare*}}{4.25}$$

$$\left[\frac{\text{(soil + tare) - tare*}}{4.25}\right] = \text{Soil density}$$

if soil density is not given:

$$\text{Estimated CEC} = \left[\frac{\text{Ca (ppm)}}{200} + \frac{\text{Mg (ppm)}}{122} + \frac{\text{K (ppm)}}{391}\right] \times \frac{\text{5-g scoop}}{\text{default wt*}}$$

default wt* = 5 for texture code 2 (medium + fine) and 4 (red) soils
wt = 6.25 for texture code 1 soils (sands)
wt = 3.5 for texture code 3 soils (mucks)

11. References

11.1 Thomas, G.W. 1982. Exchangeable cations. pp. 159-165. *In* A.L. Page et al. (eds.), Methods of Soil Analysis, Part 2, 2nd ed. Agron. Monogr. 9. ASA and SSSA, Madison, WI.

11.2 Warncke, D., and J.R. Brown. 1982. Potassium and other basic cations. pp. 31-33.*In* J.R. Brown (ed.), Recommended Chemical Soil Test Procedures for the North Central Region. (Revised.) Missouri Agr. Exp. Sta. SB1001. Columbia, MO.