SCOPE

The ammonium acetate method (pH 7.0) for cation exchange capacity is suitable for acidic to neutral soils and saline and/or gypsiferous soils. This is also called CEC direct.

PRINCIPLE

Cation exchange capacity (CEC) is defined as the sum total of the exchangeable cations that a soil can absorb. Exchangeable cations are positively charged ions (e.g., Na^{+}, Ca^{2+}, Mg^{2+}, K^{+}) held on or near the negatively charged surfaces of soil particles and which may be replaced by other positively charged ions in the soil solution. Since they are readily exchanged with other cations, they are also readily available to plants. It is normally expressed in cmol/kg (centimoles of charge per kilogram of dry soil) of a soil is a measure of the quantity of negatively charged sites on soil surfaces that can retain positively charged ions (cations) by electrostatic forces. A soil with a higher CEC has a greater capacity to maintain adequate quantities of Ca^{2+}, Mg^{2+} and K^{+} than a soil with a low CEC. A soil with a higher CEC may not necessarily be more fertile because a soil's CEC can also be occupied by acid cations such as H^{+} and Al^{3+}. However, when combined with other measures of soil fertility, CEC is a good indicator of soil quality and productivity.

Cation exchange capacity is used as a measure of fertility, nutrient retention capacity, and the capacity to protect groundwater from cation contamination.

Cation exchange sites are found primarily on clay minerals and organic matter (OM) surfaces. Soil OM will develop a greater CEC at nearly neutral pH than under acidic conditions (pH dependent CEC) thus, the addition of organic material will likely increase a soil's CEC overtime.

If a pH-buffered CEC measurement is needed (e.g. for regulatory and /or soil classification purposes, ammonium acetate buffered at pH 7 is the recommended procedure.

The soil is leached with 1M ammonium acetate solution (pH 7.00) to remove the exchangeable cations from the soil, and the exchange material is saturated with ammonium. After removal of the ammonium present in the soil as acetate, the exchangeable ammonium is determined by distillation.

TEST PRECAUTIONS

The leaching process must not be taken quickly to ensure satisfactory displacement of extractable cations.

It is recommended to purge/wash the distillation unit in every analysis for the optimized efficiency of the unit. This is done by adding 10 mL of 1M NaOH to the distilling tube then fill 3/4 of that tube with distilled water. Place a

receiving flask at the delivery tube to collect the distillate. Check the efficiency using 100 ppm N. Always run blanks and check samples before proceeding to the samples.

For best results of the analysis, samples must be titrated the same day they were distilled.

EQUIPMENT

a) Steam distillation apparatus (Kjeldahl distillation set-up)
b) Balance, precision of 0.0001 g
c) Digital Titrator/Burette
d) Dispenser (1-10 ml calibration)

LABORATORY WARE

a) Leaching Tubes, 100 mL capacity
b) Leaching tubes rack
c) Filter paper pulps
d) Absorbent cotton
e) Volumetric pipettes, 10 mL
f) Graduated cylinder, 25 mL
g) Erlenmeyer flask, 125 mL
h) Volumetric bottle top dispenser, 50 mL
i) Plastic cups, 10 ml
j) Bottle, 100 mL
k) Volumetric flasks, 50 mL and 1L
l) Reagent bottle 500 mL, 2L, 10L
m) Parafilm
n) Aspirator
o) Wash bottle

CHEMICALS AND REAGENTS

a) Ammonium acetate (NH_4OAc, pH 7.0). Dissolve 78.65 g of 98% purity NH_4OAc, dilute to 1L with deionized water. Adjust to pH 7.0 with Acetic Acid or Ammonium Hydroxide.
b) Ethyl alcohol (Analytical Reagent Grade), 95%
c) Replacing solution, 1 M KCl. Dissolve 74.5 g KCl, dilute to 1L with deionized water.
d) Sulfuric acid (H_2SO_4), 0.05 M. Pipet 2.78mL of conc. H_2SO_4 into a one (1) liter volumetric flask and dilute to the mark with distilled water.
e) Sulfuric acid (H_2SO_4), 0.005 M. Measure and transfer 200mL of 0.05M H_2SO_4 into a 2L volumetric flask and dilute up to mark with distilled water.

f) Sodium hydroxide (NaOH), 1 M technical grade. Dissolve 40 g of NaOH flakes, dilute to 1L with distilled water. Cool in water bath.
g) Boric acid (H_3BO_3), 2%. Dissolve 20 g of H_3BO_3, dilute to 1L with distilled water. Heat to hasten dissolution.
h) Mixed indicator. Dissolve 0.075 g Bromocresol green and 0.05 g methyl red up to 100 mL solution with 95% ethanol.
i) Ammonium Sulfate, $(NH_4)_2SO_4$

1000 ppm Nitrogen: Place 4.72g $(NH_4)_2SO_4$ in 1-L volumetric flask then dilute up to mark.
100 ppm Nitrogen: Place an aliquot of 25mL of 1000ppm to 250mL volumetric flask then dilute up to mark.
Place the solutions in a reagent bottle then store in refrigerator when not in use.

HEALTH AND SAFETY

Wear proper protective equipment. Use laboratory coat, closed shoes, gas mask or dust mask and chemical gloves when performing analysis to mitigate the harmful effect of exposure on chemicals.

Observe proper handling when using strong acids, strong bases, boric acid, and mixed indicators. Strong acid must be handled with the fume hood shield down as far as possible but not interfering with the work being done. Always pour acid into water slowly when preparing acid standards. Keep ethanol away to any source of heat or ignition as it is a highly flammable liquid and vapor. Be aware of the nearest shower and eye wash station.

Contact with the chemicals may cause irritation to eyes and skin, harmful if inhaled or absorbed. Repeated exposure can affect fertility of unborn child. Wash exposed skin thoroughly with mild soap and water after handling the chemicals.

PROCEDURE *(Sumner and Miller, 1996)*

Standardization of 0.005 M H_2SO_4 with THAM

1. Weigh 0.050 g of NIST traceable Tris(hydroxymethyl)aminomethane (THAM) in triplicate and transfer to a 250 mL Erlenmeyer flasks.
2. Add 40 mL of CO_2-free (boiled and cooled with cover) water and stopper the flasks.
3. Swirl gently until the reagent dissolves.
4. Add four (4) drops of mixed indicator and titrate with the H_2SO_4 to be standardized, until the solution is pale pink.

$$\text{Molarity of } H_2SO_4 = \frac{\text{weight of THAM, mg}}{\text{Volume of acid, mL} \times 121.14 \times 2}$$