

METHOD 9080

CATION-EXCHANGE CAPACITY OF SOILS (AMMONIUM ACETATE)

1.0 SCOPE AND APPLICATION

1.1 Method 9080 is used to determine the cation-exchange capacity of soils. The method is not applicable to soils containing appreciable amounts of vermiculite clays, kaolin, halloysite, or other 1:1-type clay minerals. They should be analyzed by the sodium acetate method (Method 9081). That method (9081) is also generally the preferred method for very calcareous soils. For distinctly acid soils, the cation-exchange capacity by summation method (Chapman, p. 900; see Paragraph 10.1) should be employed.

2.0 SUMMARY

2.1 The soil is mixed with an excess of 1 N ammonium acetate solution. This results in an exchange of the ammonium cations for exchangeable cations present in the soil. The excess ammonium is removed, and the amount of exchangeable ammonium is determined.

3.0 INTERFERENCES

3.1 Soils containing appreciable vermiculite clays, kaolin, halloysite, or other 1:1-type clay minerals will often give lower values for exchange capacity. See Paragraph 1.1 above.

3.2 With calcareous soils, the release of calcium carbonate from the soil into the ammonium acetate solution limits the saturation of exchange sites by the ammonium ion. This results in artificially low cation-exchange capacities.

4.0 APPARATUS AND MATERIALS

4.1 Erlenmeyer flask: 500-mL.

4.2 Buchner funnel or equivalent: 55-mm.

4.3 Sieve: 2-mm.

4.4 Aeration apparatus (assembled as in Figure 1):

4.4.1 Kjeldahl flask: 800-mL.

4.4.2 Erlenmeyer flask: 800-mL.

4.4.3 Glass wool filter.

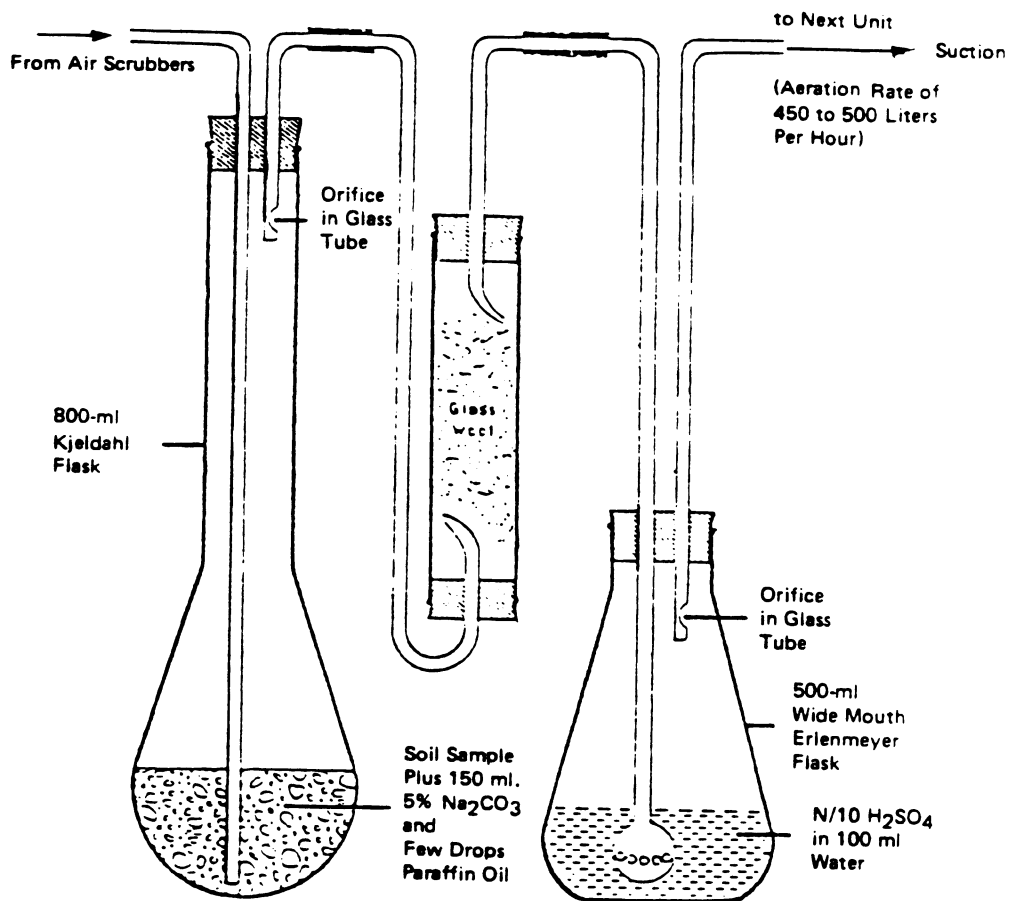


Figure 1. Diagram of aeration unit for determination of absorbed ammonia. Six to twelve such units is a convenient number for routine work; they can be mounted on a portable rack. (Apparatus as modified by Dr. A. P. Vanselow, Dept. of Soils & Plant Nutrition, University of California, Riverside, Calif.).

4.4.4 Glass tubing.

4.4.5 Flow meter.

5.0 REAGENTS

5.1 Ammonium acetate (NH_4OAc), 1 N: Dilute 114 mL of glacial acetic acid (99.5%) with water to a volume of approximately 1 liter. Then add 138 mL of concentrated ammonium hydroxide (NH_4OH) and add water to obtain a volume of about 1,980 mL. Check the pH of the resulting solution, add more NH_4OH , as needed, to obtain a pH of 7, and dilute the solution to a volume of 2 liters with water.

5.2 Isopropyl alcohol: 99%.

5.3 Ammonium chloride (NH_4Cl), 1 N: Dissolve 53.49 g of NH_4Cl in Type II water, adjust the pH to 7.0 with NH_4OH , and dilute to 1 L.

5.4 Ammonium chloride (NH_4Cl), 0.25 N: Dissolve 13.37 g of NH_4Cl in Type II water, adjust the pH to 7.0 with NH_4OH , and dilute to 1 L.

5.5 Ammonium oxalate ($(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$), 10%: Add 90 mL of Type II water to 10 g of ammonium oxalate ($(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$) and mix well.

5.6 Dilute ammonium hydroxide (NH_4OH): Add 1 volume of concentrated NH_4OH to an equal volume of water.

5.7 Silver nitrate (AgNO_3), 0.10 N: Dissolve 15.39 g of AgNO_3 in Type II water, mix well, and dilute to 1 L.

5.8 Reagents for aeration option:

5.8.1 Sodium carbonate solution (Na_2CO_3), 5%: Add 95 mL of Type II water to 5 g of Na_2CO_3 and mix well.

5.8.2 Paraffin oil.

5.8.3 Sulfuric acid (H_2SO_4), 0.1 N standard: Add 2.8 mL concentrated H_2SO_4 to Type II water and dilute to 1 L. Standardize against a base of known concentration.

5.8.4 Sodium hydroxide (NaOH), 0.1 N standard: Dissolve 4.0 g NaOH in Type II water and dilute to 1 L. Standardize against an acid of known concentration.

5.8.5 Methyl red indicator, 0.1%: Dissolve 0.1 g in 99.9 mL of 95% ethanol and mix well.

5.9 Reagents for distillation option:

5.9.1 **Sodium chloride**, NaCl (acidified), 10%: Dissolve 100 g of NaCl (ammonium-free) in 900 mL of Type II water; mix well. Add approximately 0.42 mL of concentrated HCl to make the solution approximately 0.005 N.

5.9.2 **Sodium hydroxide** (NaOH), 1 N: Dissolve 40 g of NaOH in Type II water and dilute to 1 L.

5.9.3 **Boric acid** (H_3BO_3), 2% solution: Dissolve 20 g H_3BO_3 in 980 mL Type II water and mix well.

5.9.4 **Standard sulfuric acid** (H_2SO_4), 0.1 N: See Step 5.8.3.

5.9.5 **Bromocresol green-methyl red mixed indicator**: Triturate 0.1 g of bromocresol green with 2 mL 0.1 N NaOH in an agate mortar and add 95% ethyl alcohol to obtain a total volume of 100 mL. Triturate 0.1 g of methyl red with a few mL of 95% ethyl alcohol in an agate mortar. Add 3 mL of 0.1 N NaOH and dilute the solution to a volume of 100 mL with 95% ethyl alcohol. Mix 75 mL of the bromocresol green solution with 25 mL of the methyl red solution and dilute the mixture to 200 mL with 95% ethyl alcohol.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

7.0 PROCEDURE

7.1 Sieve a sample aliquot of the soil through a 2-mm screen and allow the sieved soil to air dry (at a temperature of $\leq 60^\circ C$). Place 10 g of the air-dried soil in a 500-mL Erlenmeyer flask and add 250 mL of neutral, 1 N NH_4OAc . (Use 25 g of soil if the exchange capacity is very low, e.g., 3-5 meq per 100 g.) Shake the flask thoroughly and allow it to stand overnight.

7.2 Filter the soil with light suction using a 55-mm Buchner funnel or equivalent. Do not allow the soil to become dry and cracked.

7.3 Leach the soil with the neutral NH_4OAc reagent until no test for calcium can be obtained in the effluent solution. (For the calcium test, add a few drops each of 1 N NH_4Cl and 10% ammonium oxalate, dilute NH_4OH to 10 mL of the leachate in a test tube, and heat the solution to near the boiling point. The presence of calcium is indicated by a white precipitate or turbidity.)

7.4 Then leach the soil four times with neutral 1 N NH_4Cl and once with 0.25 N NH_4Cl .

7.5 Wash out the electrolyte with 150 to 200 mL of 99% isopropyl alcohol. When the test for chloride in the leachate (use 0.10 AgNO₃) becomes negligible, allow the soil to drain thoroughly.

7.6 Determine the adsorbed NH₄ either by the aeration method (Paragraph 7.7) or by the acid-NaCl method (Paragraph 7.8).

7.7 Aeration method:

7.7.1 Place an excess of 0.1 N standard H₂SO₄ in the 500-mL Erlenmeyer flask on the aeration apparatus (50 mL is an ample quantity for most soils) and add 10 drops of methyl red indicator and enough distilled water to make the total volume about 100 mL.

7.7.2 Attach the flask to the apparatus. Then transfer the ammonium-saturated sample of soil (from Paragraph 7.5) quantitatively to the 800-mL Kjeldahl flask located in the flow line just before the Erlenmeyer flask with the standard acid. Use a rubber policeman and a stream of distilled water from a wash bottle, as needed, to complete the transfer.

7.7.3 Add 150 mL Na₂CO₃ solution and a few drops of paraffin oil and attach the flask to the apparatus.

7.7.4 Apply suction to the outflow end of the apparatus and adjust the rate of flow to 450 to 500 liters of air per hr. Continue the aeration for 17 hr.

7.7.5 Shut off the suction and remove the flask. Titrate the residual acid in the absorption solutions with standard 0.1 N NaOH from the original red color through orange to yellow at the end point. From the titration values obtained with the soil and blank solutions, calculate the content of adsorbed ammonium in milligram equivalents per 100 g soil.

7.8 Acid-NaCl method:

7.8.1 Leach the ammonium-saturated soil from Paragraph 7.5 with 10% acidified NaCl until 225 mL have passed through the sample. Add small portions at a time, allowing each portion to pass through the sample before adding the next portion.

7.8.2 Transfer the leachate quantitatively to an 800-mL Kjeldahl flask, add 25 mL of 1 N NaOH, and distill 60 mL of the solution into 50 mL of 2% H₃BO₃.

7.8.3 Add 10 drops of bromocresol green-methyl red mixed indicator and titrate the boric acid solution with standard 0.1 N H₂SO₄. The color change is from bluish green through bluish purple to pink at the end point. Run blanks on the reagents. Correct the titration figure for the blanks and calculate the milliequivalents of ammonium in 100 g of soil.

7.8.4 Results should be reported as "determined with ammonium acetate" at pH 7.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 Material of known cation-exchange capacity must be routinely analyzed.

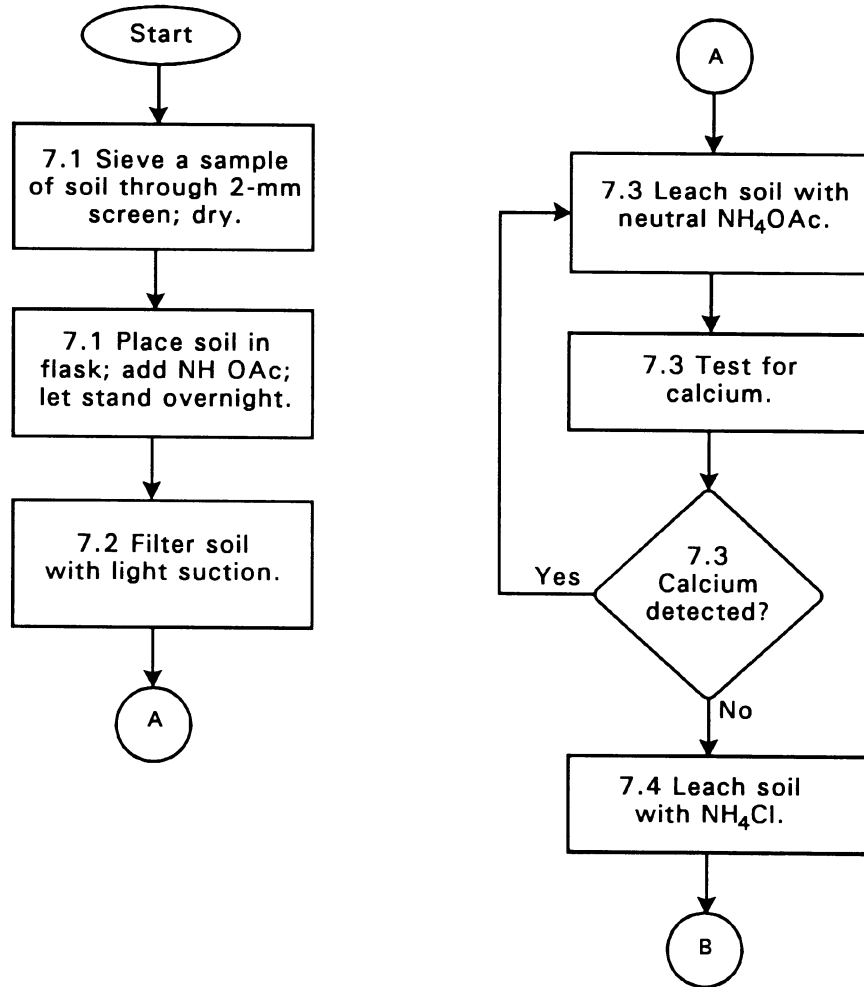
9.0 METHOD PERFORMANCE

9.1 No data provided.

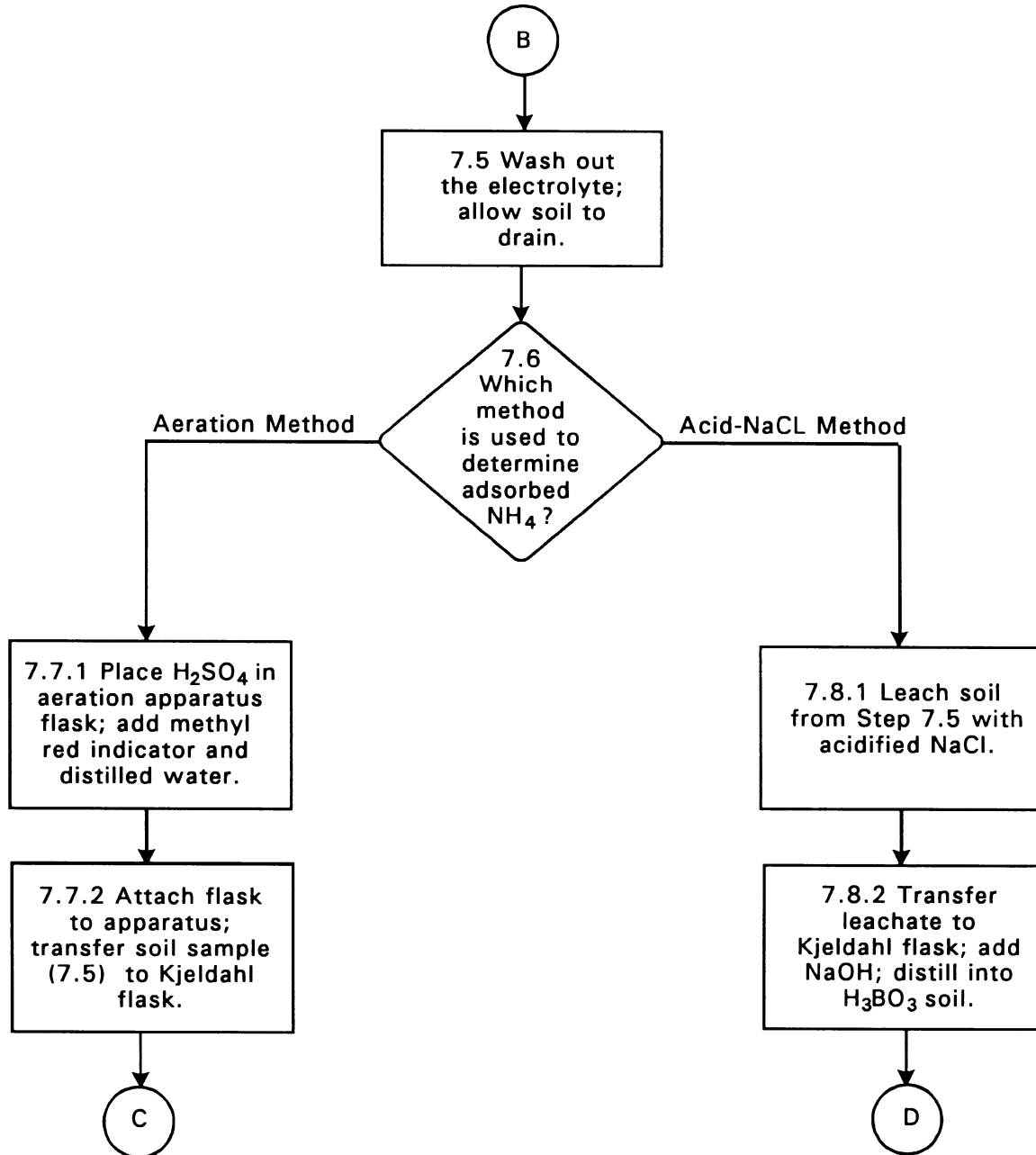
10.0 REFERENCES

1. This method is based on Chapman, H.D., "Cation-exchange Capacity," pp. 891-900, in C.A. Black (ed.), Method of Soil Analysis, Part 2: Chemical and Microbiological Properties, Am. Soc. Agron., Madison, Wisconsin (1965).

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