Standard Test Method for
Measuring the Exchange Complex and Cation Exchange Capacity of Inorganic Fine-Grained Soils[1]

This standard is issued under the fixed designation D7503; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes the procedures for measuring the soluble and bound cations as well as the cation exchange capacity (CEC) of fine-grained inorganic soils. Clay minerals in fine-grained soils carry a negative surface charge that is balanced by bound cations near the mineral surface. These bound cations can be exchanged by other cations in the pore water, which are referred to as soluble cations. The cation exchange capacity is a measure of the negative surface charge on the mineral surface. The CEC generally is satisfied by calcium (Ca), sodium (Na), magnesium (Mg), and potassium (K), although other cations may be present depending on the environment in which the soil exists. This test method was developed from concepts described previously in Lavkulich (1981) (**1**)[2] and Rhoades (1982) (**2**). In soils with appreciable gypsum or calcite, dissolution of these minerals will release Ca in solution that may affect the measurement.

1.2 In this test method, the soluble salts from the mineral surface are washed off with de-ionized water and then the concentration of soluble salts within the extract is measured. The bound cations of the clay are measured by using a solution containing an index ion that forces the existing cations in the bound layer into solution. The total concentrations of bound and soluble cations in this solution are measured. The CEC is measured by displacing the index ion with another salt solution and measuring the amount of the displaced index ion.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.4 All observed and calculated values shall conform to the guide for significant digits and rounding established in Practice D6026. The procedures in Practice D6026 that are used to specify how data are collected, recorded, and calculated are regarded as the industry standard. In addition, they are representative of the significant digits that should generally be retained. The procedures do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the objectives of the user. Increasing or reducing the significant digits of reported data to be commensurate with these considerations is common practice. Consideration of the significant digits to be used in analysis methods for engineering design is beyond the scope of this standard.

1.5 The values stated in SI units are to be regarded as the standard, unless other units are specifically given.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D653 Terminology Relating to Soil, Rock, and Contained Fluids
D1193 Specification for Reagent Water
D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass
D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
D6026 Practice for Using Significant Digits in Geotechnical Data
E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Terminology

3.1 For definitions of other terms used in this standard, see Terminology D653.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *acid wash, n*—the process of initially rinsing equipment with tap water, followed by a rinse with 10 % HNO_3 solution, and then finally rinsing 3 times with DI water.

3.2.2 *bound cations (BC), n*—cations that are adsorbed (bound) to mineral surfaces that may be exchanged.

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[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.04 on Hydrologic Properties and Hydraulic Barriers.

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[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.3 *cation exchange capacity (CEC), n*—the total negative charge on mineral surface to be satisfied by bound cations.

3.2.4 *exchange complex, n*—the collection of bound cations satisfying the CEC.

3.2.5 *fine-grained soils, n*—any soil with more than 50 % passing the No. 200 US standard sieve.

3.2.6 *inorganic soils, n*—any soil with a loss of ignition (LOI) less than 1 %.

3.2.7 *soluble cations (SC), n*—cations in the soil that are not bound to the mineral surface.

4. Significance and Use

4.1 Fine-grained soils are used in waste containment systems as barriers to flow and contaminant transport. Liquids contained by these barriers can contain ions that may interact with the mineral surfaces in fine-grained soils.

4.2 The liquid passing through the pores of fine-grained soil can interact with the mineral surface, and affect the physical and chemical characteristics of the soil. This method can be used as part of an evaluation of these interactions.

NOTE 1—The quality of the result produced by this standard depends on the competence of the personnel performing the test and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing, sampling, inspection, etc. Users of this standard are cautioned that compliance with Practice D3740 does not in itself ensure reliable results. Reliable results depend on many factors. Practice D3740 provides a means of evaluating some of these factors.

5. Apparatus

5.1 *Drying Oven,* capable of maintaining a uniform temperature of 105 ± 5 °C that meets the requirements of Specification E145.

5.2 *No. 10 U.S. Standard Sieve*

5.3 *Desiccator,* containing silica gel.

5.4 *Laboratory Balance,* 20 g capacity, ±0.001 g accuracy and precision.

5.5 *Weighing Paper,* or small weighing dish.

5.6 *End Over End Shaker,* capable of 30 rpm.

5.7 *Capped Containers* should tightly fit in the end over end shaker holding compartment with capacities larger than 40 mL.

5.8 *500 mL Filtering Flask,* connectable to low-pressure vacuum line, acid washed (See Fig. 1).

5.9 *Flexible Tubing,* appropriate size to connect filtering flask to the low-pressure vacuum line (See Fig. 1).

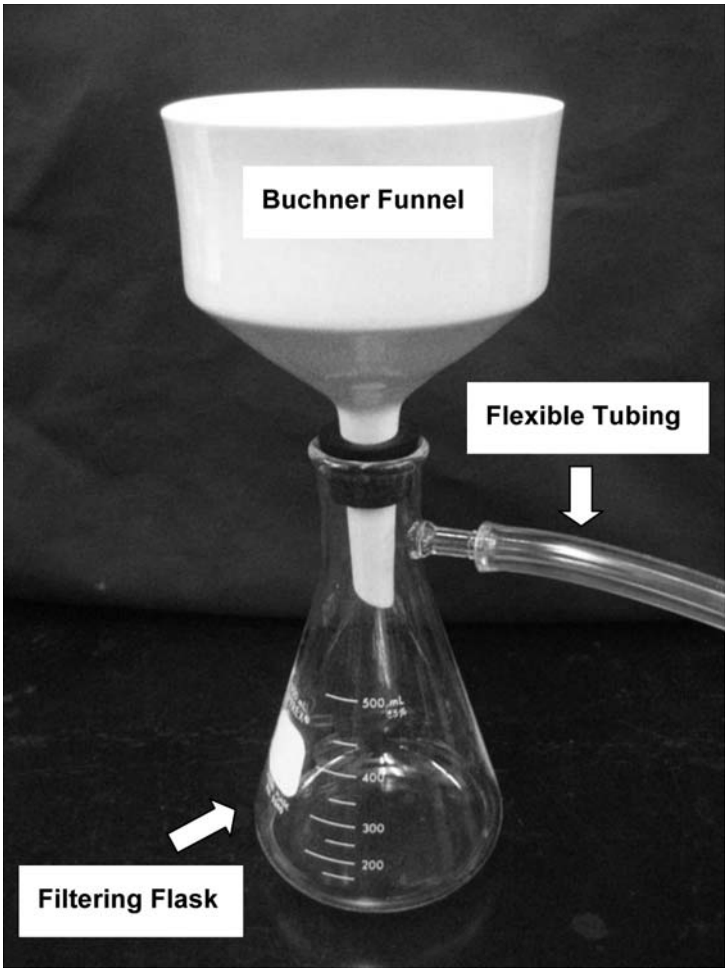

FIG. 1 Experimental Setup for Vacuum Filtration

5.10 *Buchner Funnel,* 55 mm or 90 mm diameter, acid washed (See Fig. 1).

5.11 *Wash Bottle,* for dispensing solutions, new or acid washed.

5.12 *Graduated Cylinder,* for measuring solution portions, acid washed.

5.13 *2.5 µm Ashless Filter Paper* that covers the surface of Buchner funnel.

5.14 *250 mL Volumetric Flasks,* class A flask for precision and accuracy.

6. Reagents

6.1 *Reagent Water:* Use only ASTM Type II water as defined in Specification D1193.

6.2 *Ammonium Acetate, 1M:* Dissolve 77.08 g of 99.9 % pure NH_4OAc in Type II DI water (See Specification D1193) and fill to volume in a 1000 mL volumetric flask. Adjust the pH of the solution to 7 with ammonium hydroxide or acetic acid. Approximately 1 L of NH_4OAc is needed per 6 samples.

6.3 *Isopropanol:* Reagent grade.

6.4 *Potassium Chloride, 1M:* Dissolve 74.6 g of 99 % pure KCl in Type II DI water and fill to volume in a 1000 mL volumetric flask. Approximately 1 L of KCl is needed per 6 samples.

6.5 *Ammonium Sulfate:* Dry 238 mg of ACS Certified $(NH_4)_2SO_4$ for 4 h at 40 °C. Make a 200 mg/L stock solution by dissolving the dried compound in 100 mL of Type II DI water and fill to volume in a 250 mL volumetric flask. Prepare calibration standards by diluting the stock solution into concentrations of 10, 20, 40, 50, and 80 mg/L.

6.6 *Ca, Mg, K, and Na:* Use ICP-grade or AA-grade element standards in an HNO_3 matrix to prepare quality control spikes in a NH_4OAc matrix.

7. Hazards

7.1 This standard does not address all of the safety concerns associated with its use. The user of this standard is responsible for implementing proper safety precautions and should be aware of any possible health concerns and risks related with the materials and chemicals used while following this standard.

8. Determination of Required Air-Dried Mass of Soil for Analysis

8.1 Air-dry approximately 30 g of soil (12 g of solid is required for testing) according to the procedures described in Test Methods D2216.

8.2 Oven-dry at least 2 g of the air-dry soil to determine the water content following Test Methods D2216.

8.3 Determine total mass of air-dry soil needed to have 2 g of soil solids for determination of soluble cations.

8.4 Determine total mass of air-dry soil needed to have 10 g of soil solids for determination of bound cations.

8.5 Use the oven-dry weight (2 or 10 g) of the soil for all calculations.

NOTE 2—Oven-dried soils should not be used for determining CEC, soluble cations, or bound cations because gypsum ($CaSO_4$•$2H_2O$) is transformed to plaster of paris ($CaSO_4$•½ H_2O) at high temperatures, and plaster of paris is more soluble in water than gypsum.

9. Determination of Soluble Cations

9.1 Use only air-dry soil that passes the No. 10 US Standard Sieve.

9.2 Add mass of air-dry soil corresponding to 2 g of soil solids and 100 mL of Type II DI water to a covered container that fits tightly into the shaker.

9.3 Place the containers in an end-over-end shaker and shake for 1 h at 30 rpm.

9.4 Vacuum filter the mixture in each container using 2.5 µm ashless filter paper.

9.5 Transfer the extract to a 100 mL acid washed volumetric flask, preserve with 1 mL HNO_3, and fill to volume.

9.6 Analyze each extract for cation concentration (in mg/L) using inductively coupled plasma spectrometry, atomic absorption, or another suitable method.

NOTE 3—Low solid to liquid ratios can result in peptization and hydrolysis in some cases. If these reactions are of concern a lower solid to liquid ratio such as 1:2 can be used.

10. Determination of Bound Cations

10.1 Use only air-dry soil that passes the No. 10 US Standard Sieve.

10.2 Prepare a blank sample for analysis by placing 100 mL of DI water in a covered container.

10.3 Prepare quality control samples for analysis by creating a duplicate and a spike and place in a covered container. Add determined mass of air-dried soil corresponding to 10.0 g of soil solids and 40 mL of 1 M NH_4OAc into 100 mL covered container (use a container which tightly fits into the end over end shaker).

10.4 Shake the covered containers for 5 min in an end over end shaker at 30 rpm. Agitate the container to rinse the particles from the side of the container and let the mixture stand for 24 h.

10.5 After 24 h shake the container with the mixture for 15 min at 30 rpm in the end over end shaker.

10.6 Rinse the 500 mL filtering flask and Buchner funnel with NH_4OAc.

10.7 Place the Buchner funnel over the 500 mL filtering flask and line the Buchner funnel with 2.5 µm ashless filter paper (See Fig. 1).

10.8 Transfer the contents of the shaken container to the Buchner funnel.

10.9 Rinse the container and cap into the Buchner funnel using a squirt bottle containing 1 M NH_4OAc.

10.10 Apply low suction to the filtering flask (<10 kPa).

10.11 Wash the soil in the Buchner funnel with four 30 mL portions of 1 M NH_4OAc. Add each 30 mL portion slowly and allow the entire 30 mL portion to drain before adding the next 30 mL portion. Do not allow the soil to dry between additions of NH_4OAc.

10.12 Turn the suction off to the filtering flask after the last washing. Leave the NH_4OAc washed soil in the Buchner funnel; this soil is to be used for determining the cation exchange capacity (CEC).

10.13 Rinse the 250 mL volumetric flask with 1 M NH_4OAc.

10.14 Transfer the filtered aqueous solution into the 250 mL volumetric flask. Preserve the solution to pH of 2 with ICP-grade nitric acid and fill the volumetric flask to volume with NH_4OAc.

10.15 Analyze the cation concentration (in mg/L) in the aqueous solution using inductively coupled plasma spectrometry or atomic absorption.

NOTE 4—Typically Na, Ca, Mg, and K cations satisfy the majority of the CEC. The user may analyze for other cations of concern, which may include exchangeable acidity.

11. Determination of the Cation Exchange Capacity

11.1 Rinse an acid washed 500 mL filtering flask with isopropanol.

11.2 Place the Buchner funnel with the 1 M NH_4 OAc washed sample onto the 500 mL filtering flask (See Fig. 1).

11.3 Apply low suction (<10 kPa) to the filtering flask. Do not allow the soil to dry when suction is applied.

11.4 Wash the soil with three 40 mL portions of isopropanol. Allow each 40 mL portion to drain before adding the next portion. Washing with isopropanol removes residual NH_4OAc.

11.5 Turn off the suction to the filtering flask when free liquid is no longer visible.

11.6 Separate the Buchner funnel from the filtering flask. Discard the isopropanol collected in the 500 mL filtering flask and rinse the flask with Type II DI water three times.

11.7 Return the Buchner funnel containing the isopropanol washed soil to the rinsed filtering flask (See Fig. 1).

11.8 Apply suction to the filtering flask and wash the soil with four 50 mL portions of 1 M KCl solution. Allow each portion of the 1 M KCl solution to drain before adding the next portion. Do not allow the soil to dry between additions of KCl solution.

11.9 Rinse a 250 mL volumetric flask with 1 M KCl.

11.10 Transfer the extract into the 250 mL volumetric flask. Rinse the filtering flask with Type II DI water and transfer the contents into the volumetric flask.

11.11 Fill the volumetric flask to volume with water.

11.12 Analyze the KCl extract for nitrogen concentration (mg/L) using a spectrophotometer.

12. Calculations

12.1 Calculate the concentration of soluble cations as follows:

$$S = C \times \frac{0.100L}{M_o(g)} \times 1000\frac{g}{kg}$$

where:

S = concentration of soluble cations (cmol$^+$/kg) in the soil,
C = concentration of cations (cmol$^+$/L) in the DI water extract from 9.6, and
M_o = oven-dry mass of soil.

12.2 Calculate the concentration of bound cations as follows:

$$M^+ = C \times \frac{0.25L}{M_o(g)} \times 1000\frac{g}{kg} - S$$

where:

M^+ = concentration of adsorbed cation (cmol$^+$/kg) in soil, and
C = concentration of cation (cmol$^+$/L) in the NH_4OAc extract from 10.15.

12.3 Calculate the cation exchange capacity as follows:

$$CEC = N \times \frac{1\,cmol^+}{140mg} \times \frac{0.25L}{M_o(g)} \times 1000\frac{g}{kg}$$

where:

CEC = concentration of cation exchange capacity (cmol$^+$/kg), and
N = concentration of nitrogen (mg/L) from 11.12.

13. Report

13.1 Report the following information:

13.1.1 Source and description of the soil.

13.1.2 Source and description of all chemicals used to make mixtures and solutions.

13.1.3 Dilution factor of aqueous samples prior to chemical analysis.

13.1.4 Concentration of bound cations, concentration of soluble cations, and CEC in units of cmol$^+$/kg.

13.1.5 Any modifications to this standard test method.

14. Precision and Bias

14.1 *Precision*—Test data on precision are not presented due to the nature of the soil or rock, or both materials, tested by this standard. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program. In addition, it is either not feasible or too costly to produce multiple specimens that have uniform physical properties. Any variation observed in the data is just as likely to be due to specimen variation as to operator or laboratory testing variation.

14.1.1 Subcommittee D18.04 is seeking any pertinent data from users of these test methods that might be used to make a limited statement on precision.

14.2 *Bias*—There is no accepted reference value for these test methods; therefore, bias cannot be determined.

15. Keywords

15.1 bentonite; bound cations; cation exchange capacity; cations; CEC; clay; exchange complex; soluble cations

REFERENCES

(1) Lavkulich, L., "Exchangeable Cations and Total Exchange Capacity by the Ammonium Acetate Method at pH 7.0," Soil Sampling and Methods of Analysis (Martin R. Carter, editor), Canadian Society of Soil Science, Ottawa, Ontario, Canada, 173 -175. 1981.

(2) Rhoades, J., "Soluble Salts," Methods of Soil Analysis, Part 2. Chemical and Microbiological Properties, 2nd Edition (A. Page, R. Miller, D. Keeney, editors). Soil Science Society of America, Madison, Wisconsin, USA, 167-180. 1982.

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