Designation: D7503 – 18

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, health, and environmental 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 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. Reporting of test results in units other than SI shall not be regarded as nonconformance with this test method.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D6026 Practice for Using Significant Digits in Geotechnical Data

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

[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.

*A Summary of Changes section appears at the end of this standard

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.

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 sieve having an opening size of 0.075 mm.

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 *Sieve* having an opening of 2.00 mm.

5.3 *Desiccator,* containing silica gel.

5.4 *Laboratory Balance,* 20 g capacity, ±0.001 g accuracy and precision selected in accordance with Guide D4753.

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).

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.