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Standard Test Methods for Detecting Water Soluble Sulfates in Construction Soils¹

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1. Scope

1.1 These methods determine the water soluble sulfate content of cohesive soils used in construction by using the colorimetric technique. Two methods are presented in this standard. Method A is for use in the field and Method B is for use in the laboratory. The colorimetric technique involves measuring the scattering of a light beam through a solution that contains suspended particulate matter. Measurements of sulfate concentrations in construction soils can be used to guide professionals in the selection of appropriate stabilization methods and to assist in assessment of potential deterioration in concrete structures.

NOTE 1—These test methods are partially based on the research conducted by Texas A & M University.

1.2 The field method, Method A, is used as a screening test for the presence of sulfates and their concentration. The laboratory method, Method B, provides better resolution than the field method.

1.3 Ion chromatography is also an acceptable alternative method that can be used to evaluate results, however, it is outside the scope of this standard.

1.4 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026, unless superseded by this test method.

1.5.1 The procedures used to specify how data are collected/recorded and calculated in the standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to

increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of these test methods to consider significant digits used in analysis methods for engineering data.

1.6 *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.*

1.7 *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:*²

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1193 Specification for Reagent Water

D2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)

D2488 Practice for Description and Identification of Soils (Visual-Manual Procedures)

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4220/D4220M Practices for Preserving and Transporting Soil Samples (Withdrawn 2023)³

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 and Data Records in Geotechnical Data

¹ This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.06 on Physical-Chemical Interactions of Soil and Rock.

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² 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 The last approved version of this historical standard is referenced on www.astm.org.

3. Terminology

3.1 Definitions:

3.1.1 For definitions of common technical terms used in this standard, refer to Terminology **D653**.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *colorimetric technique, n*—the process of measuring the light transmission of a liquid and translating it into concentration using a reagent (in this standard, barium chloride) combined with a specimen.

3.2.1.1 *Discussion*—Colorimetry is the measurement of the wavelength and the intensity of electromagnetic radiation in the visible region of the spectrum. Colorimetry can help find the concentration of substances, since the amount and color of the light that is absorbed or transmitted depends on properties of the solution, including the concentration of particles in it.

3.2.2 *filtrate, n*—a substance, usually a solution, that has been passed through a filter.

4. Summary of Test Method

4.1 These test methods are used to determine the average water soluble sulfate concentration in cohesive soils using the colorimetric technique. Method A is performed in the field using a hand-held device, whereas Method B is performed in the laboratory using a benchtop/desktop device under controlled conditions.

4.2 In both methods a sample is dried and processed over specific sieves to obtain three representative specimens. Each specimen is then diluted with distilled or demineralized water to an initial ratio of 1:20 and then vigorously shaken prior to a conditioning period. After conditioning, the soil suspension is filtered and the filtrate is transferred to a cuvette where the barium chloride reagent is added. A minimum of three readings per specimen are obtained and the results are corrected and averaged to yield the average water soluble sulfate concentration in ppm of the sample.

5. Significance and Use

5.1 Where sulfates are suspected, subgrade soils should be tested as an integral part of a geotechnical evaluation because the possibility that sulfate induced heave may occur if calcium containing stabilizers are used to improve the soils and sulfate reactions may also cause deterioration in concrete structures. When planning to treat a soil used in construction with lime, testing the soil for water soluble sulfates prior to treatment becomes very important (**Note 2**).

5.2 When sulfate containing cohesive soils are treated with calcium-based stabilizers for foundation improvements, sulfates and free alumina in natural soils react with calcium and free hydroxide to form crystalline minerals, such as ettringite and thaumasite.⁴ Thaumasite forms when ettringite undergoes changes in the presence of carbonates at low temperatures.⁵ The sulfate minerals expand considerably when they are hydrated.

⁴ Mitchell, J.K., "Practical Problems from Surprising Soil Behavior," ASCE Journal of Geotechnical Engineering, Vol. 112, No. 3, 1986, pp. 259-289.

⁵ Hunter, D., "Lime Induced Heave in Sulfate Bearing Clays," ASCE Journal of Geotechnical Engineering, Vol. 114, No. 2, 1988, pp. 150-167.

NOTE 2—For more information on the effect of treating soils containing water soluble sulfates, refer to the following publication: Little, D.N., Stabilization of Pavement Subgrades and Base Course with Lime, Kendal/Hunt Publishing Co., Dubuque, IA, 1995.

NOTE 3—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, 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 assure reliable results. Reliable results depend on many factors; Practice **D3740** provides a means of evaluating some of those factors.

6. Interferences

6.1 The readings from the device can be affected if the cuvette is scratched or has dirty or smeared surfaces. Care should be taken to make sure the cuvette surfaces are clean and free of debris and that the filtration has sufficiently removed the larger particulates.

6.2 The presence of aggressive sulfate reducing bacteria could lead to an under reporting of the sulfate content.

7. Apparatus

7.1 *Colorimeter*—A hand held or benchtop/desktop device capable of measuring up to 8,000 ppm.

NOTE 4—A colorimeter is an instrument that compares the amount of light passing through a solution with the amount that can pass through a sample of pure solvent. A colorimeter contains a photocell that is able to detect the amount of light which passes through the solution under evaluation. The more light that hits the photocell, the larger the current it produces, hence showing the absorbance of light. A colorimeter takes 3 wideband readings along the visible light spectrum to obtain a rough estimate of a color sample.

7.2 *Balance*—Balances shall conform to the requirements of Guide **D4753**.

7.2.1 The balance shall have readability without estimation of 0.01 g or better. The capacity of this balance will need to exceed the mass of the representative soil and its container.

7.3 *Drying Oven*—Vented, thermostatically controlled oven capable of maintaining a uniform temperature of $60 \pm 5^\circ\text{C}$ throughout the drying chamber. These requirements typically require the use of a forced-draft oven.

7.4 *Sieves*—4.75-mm (No. 4) and 425- μm (No. 40) sieves used to split and separate the sample, respectively. These sieves are subjected to rough operation and shall not be used for quantitative grain size analysis.

7.5 *Desiccator*—A desiccant containing device of suitable size used to prevent moisture gain during cooling of the oven-dried specimen.

7.6 *Cuvette*—A 10-mL glass vial with alignment markings to hold the filtrate and reagent.

7.7 *Mortar and Rubber-Tipped Pestle*—Apparatus suitable for breaking up aggregations of air-dried soil particles without breaking individual particles.

7.8 *Sample Splitter*—A device capable of adequately splitting the sample.

7.9 *Beakers*—Two, glass or plastic; with a minimum capacity of 500 mL.

7.10 *Graduated Cylinders*—Two, glass or plastic; one each with capacities of 10 mL and 100 mL.

7.11 *Bottles*—Two, wide-mouth round high-density polyethylene bottles; one each with capacities of 250 mL and 500 mL.

7.12 *Pipets*—2 to 10 mL capacity disposable pipets.

7.13 *Permeable Membrane Filter*—150 mm diameter circle filter paper having a pore diameter of 10- μ m (Note 5).

7.14 *Funnel*—A glass or plastic, ~80 mm diameter funnel having a short, wide stem.

7.15 *Plate*—A clean, nonporous, smooth, solid surface that is large enough to hold the representative soil while it air dries. The plate can also be used when the sample is split.

7.16 *Miscellaneous Items*—Items such as a wash/rinse bottle (squirt bottle), latex gloves, pans, brushes, spatulas, delicate task wipes, and band stirring rods may be useful.

NOTE 5—The use of a permeable membrane filter with a pore diameter of 10- μ m is recommended by Puppala et al. to give reproducible results.⁶

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee of Analytical Reagents of the American Chemistry Society where such specifications are available.

8.2 *Test Water*—Distilled or demineralized water is the only permissible test fluid. The use of tap water is not permitted. Specification **D1193** provides information regarding distilled or demineralized water.

8.3 *Barium Chloride (BaCl₂)*—Reagent grade.

8.4 *Standard Sulfate Solution (SO₄)*—Reagent grade, 1000 ppm where 1 mL = 1 mg of SO₄.

9. Hazards

9.1 **Warning**—All soluble barium salts are poisonous, for instance, barium carbonate will dissolve in stomach acid. The antidote to barium poisoning is epsom salts (magnesium sulfate) which immediately precipitates harmless barium sulfate if taken quickly enough.

9.2 Care should be taken to avoid contact with the skin, eyes, and should not be ingested. Use latex gloves when handling barium chloride. Refer to the Safety Data Sheet for more information.

10. Sampling

10.1 *General*—These test methods do not address, in any detail, procurement of the sample. It is assumed the sample is obtained using appropriate methods and is representative of the soil under evaluation. However, the testing agency shall preserve all samples in accordance with Practice **D4220/D4220M** Group B, except if the as-received sample does not

meet those requirements (Note 6). In which case, the water content of the material does not have to be maintained.

10.2 The sample can be from a variety of sources. Typically, samples for water soluble sulfate testing are obtained in the following forms: large bags or buckets, small bags, jar samples, or tube samples.

10.3 Samples should be obtained at the specified sampling frequency. For Method A, the sample mass must be greater than 300 g and for Method B, the sample mass must be greater than 1500 g in order to obtain representative specimens for testing.

NOTE 6—To reduce the potential for growth of sulfate reducing bacteria it is recommended for sulfate testing to be carried out within 28 days of sampling and for the samples to be stored at or below 40°C.

11. Preparation of Test Specimens

11.1 *Preparation for Method A (Field):*

11.1.1 Obtain from the sample approximately 300 g of representative soil.

11.1.2 Place the representative soil on the plate or other smooth, nonporous surface and allow it to air dry to a constant mass.

11.1.3 Process the entire representative soil over the 425- μ m (No. 40) sieve being careful to dislodge material adhering to the aggregate particles and avoid breaking down the natural size of the particles.

11.1.4 Split the representative soil passing the 425- μ m (No. 40) sieve using the sample splitter and obtain three 10 \pm 0.1 g specimens. Determine and record the mass of each specimen to the nearest 0.1 g.

11.2 *Preparation for Method B (Laboratory):*

11.2.1 Dry the entire sample in the 60 \pm 5°C drying oven to a constant mass. Then, remove from the oven and place the sample in the desiccator. Allow the sample to cool to 25 \pm 3°C.

11.2.2 Remove the sample from the desiccator and process the entire sample over the 4.75-mm (No. 4) sieve being careful to dislodge material adhering to the aggregate particles and avoid breaking down the natural size of the particles.

11.2.3 Split the processed sample passing the 4.75-mm (No. 4) sieve to obtain approximately 1,500 g of representative soil.

11.2.4 Process the 1,500 g representative soil using a rubber tipped pestle over the 425- μ m (No. 40) sieve being careful to dislodge material adhering to the aggregate particles and avoid breaking down the natural size of the particles.

11.2.5 Split the representative soil passing the 425- μ m (No. 40) sieve using the sample splitter and obtain three 20 \pm 0.1 g specimens. Determine and record the mass of each specimen to the nearest 0.1 g.

12. Calibration/Preparation of Apparatus

12.1 *Colorimeter*—Calibrate the colorimeter following the manufacturer's instructions and generate the correction curve as applicable with the guidance below (Note 7).

12.1.1 Develop the calibration correction curve for each colorimeter used in testing as detailed in **Annex A1**.

⁶ Puppala, A.J., Vinyant, C., Kruzic, A.P., and Perrin, L., "Evaluation of a Modified Soluble Sulfate Determination Method for Fine-Grained Cohesive Soils," *Geotechnical Testing Journal*, GTJODJ, Vol. 25, No. 1, 2002, pp. 85-94.

12.1.2 Perform the calibration of the colorimeter and develop correction curves, as applicable, at the interval specified in [A1.2.8](#).

NOTE 7—Some colorimeters may have a user defined correction curve stored automatically to correct the reading. When using this feature, follow the manufacturer's instructions to produce the correction curve and the corrected reading.

13. Procedure

13.1 Test Method A: Field Determination:

13.1.1 Place the 10 ± 0.1 g soil specimen in a 250 mL bottle and add 200 mL of test water using the 100 mL graduated cylinder to provide an initial dilution ratio of 1:20.

13.1.2 Close and shake the bottle vigorously by hand for approximately 1 min to mix the soil and test water. Then, allow it to sit/condition for a minimum of 12 h.

13.1.3 After a minimum of 12 h of conditioning has elapsed, prepare to filter the suspension by placing the permeable membrane filter inside the funnel. Insert the stem of the funnel into a beaker. Pour the suspension through the permeable membrane filter and capture the filtrate in the beaker.

13.1.4 Pour the filtrate from the beaker back into the bottle and shake vigorously for 1 min. Then, pour the suspension through the permeable membrane filter back into the beaker. Repeat the filtration process until a clear filtrate is attained. Proceed to [13.3](#).

13.2 Test Method B: Laboratory Determination:

13.2.1 Place the 20 ± 0.1 g soil specimen in a 500 mL bottle and add 400 mL of test water using the 100 mL graduated cylinder to provide an initial dilution ratio of 1:20.

13.2.2 Close and shake the bottle vigorously by hand for approximately 1 min to mix the soil and test water. Then, allow it to sit/condition for a minimum of 12 h.

13.2.3 After a minimum of 12 h has elapsed, prepare to filter the suspension by placing the permeable membrane filter inside the funnel. Insert the stem of the funnel into a beaker. Pour the suspension through the permeable membrane filter and capture the filtrate in the beaker.

13.2.4 Pour the filtrate from the beaker back into the bottle and shake vigorously for 1 min. Then, pour the suspension through the permeable membrane filter back into the beaker. Repeat this process until a clear filtrate is attained. Proceed to [13.3](#).

13.3 Use a clean, disposable pipet to measure and transfer 10 mL of filtrate from the beaker to the 10 mL graduated cylinder.

13.4 Prior to transferring the filtrate from the graduated cylinder to the cuvette, make sure the cuvette is clean and free from any residue or scratches. Wearing latex gloves while handling the cuvette aids in preventing the transfer of oils from the skin to the cuvette surfaces. If the cuvettes are to be reused, make sure they have been double rinsed with distilled water prior to reuse.

13.5 Transfer the 10 mL of filtrate from the graduated cylinder into the cuvette. Cap the cuvette and wipe the outside of the cuvette clean with a delicate task wipe to remove dirt,

fingerprints, or other substances that will obstruct or obscure a light beam from passing through the glass and filtrate.

13.6 Turn the colorimeter on and if the device has parameter settings, make sure it is set for sulfate testing. [Fig. 1](#) is an example of a typical bench/desktop colorimeter device, however colorimeters come in various styles.

13.7 Prepare a blank sample by placing 10 mL of test water in a cuvette. Cap the cuvette and make sure the cuvette is clean as described in [13.5](#).

13.8 Place the blank sample in the correct orientation into the colorimeter and zero the meter per manufacturer's instructions. Once the zero has been determined and accepted, remove the blank sample from the colorimeter.

13.9 Remove the cap from the cuvette containing the specimen and carefully add 0.02 g of BaCl_2 to the cuvette. Use latex gloves when handling BaCl_2 . Do not allow the BaCl_2 to come into contact with bare skin.

13.10 Mix the BaCl_2 thoroughly until it is completely dissolved and dispersed in the cuvette. Cap the cuvette and wipe it clean as described in [13.5](#). Place the cuvette in the correct orientation in the colorimeter and test the specimen according to the manufacturer's instructions.

13.11 Take and record a minimum of three readings per specimen and average the results.

13.12 *Device Indications*—If the device indicates in some manner the result is below the measuring range limit, record the sulfate concentration as <100 ppm. If the device indicates in some manner the result exceeds the measuring range limit, dilute the specimen further using the following:

13.12.1 Measure and transfer 5 mL of filtrate into the 10 mL graduated cylinder and add 5.0 mL of test water using a clean pipet. The dilution ratio is now 1:40; which corresponds to a maximum testing range of 8,000 ppm. Record the dilution ratio. Repeat steps [13.4](#) through [13.11](#).

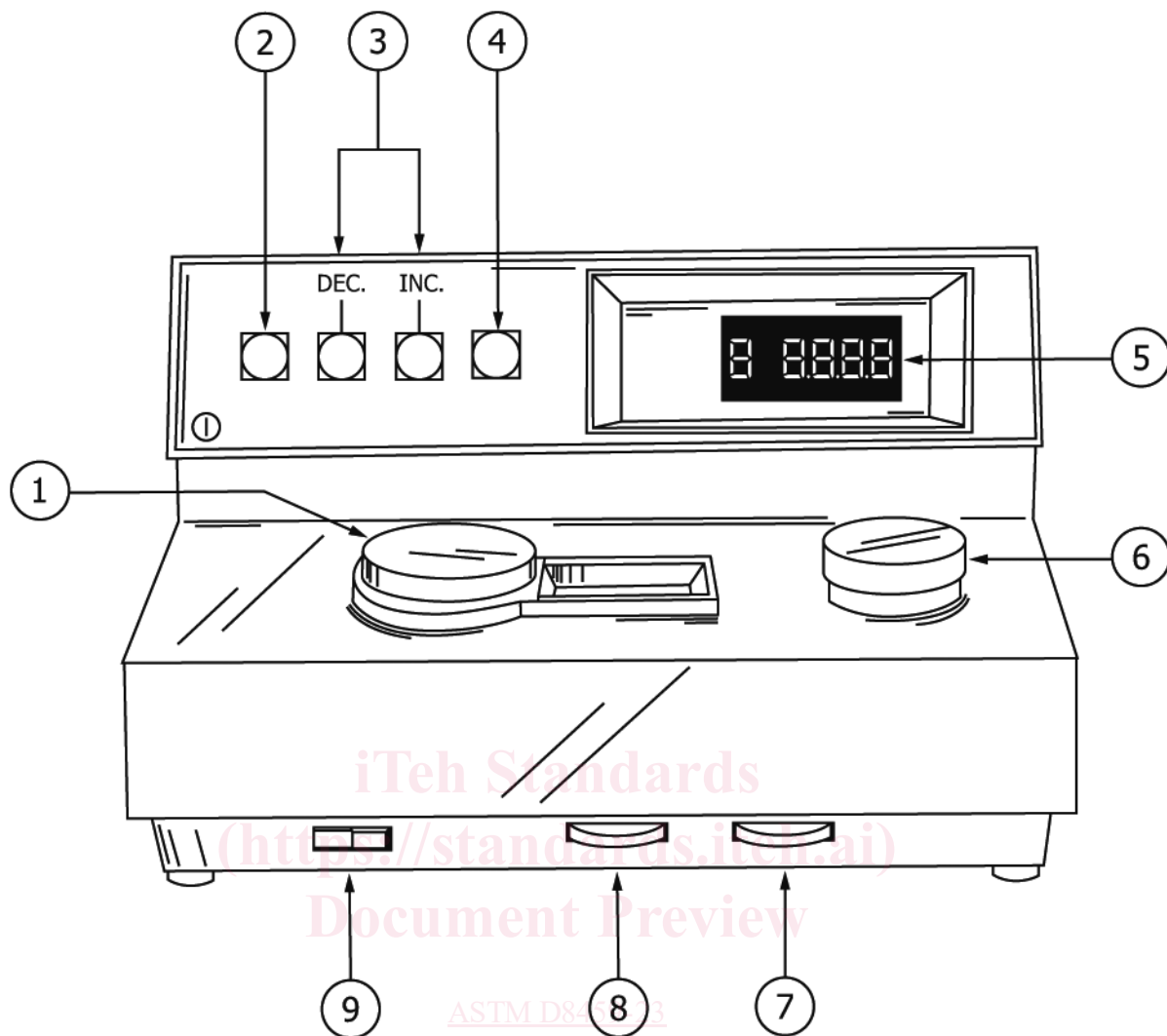
13.12.2 If the device again indicates it exceeds the measuring range limit, the soil has more than 8,000 ppm of water soluble sulfate content and additional dilution(s) is necessary. Continue to dilute the specimen and repeat steps [13.4](#) through [13.11](#) until values are obtained. See [X2.2](#) for more information regarding dilution ratios ([Note 8](#)).

NOTE 8—If the average sulfate concentration is greater than 3,000 ppm, the soil is not suitable for lime, cement, or any calcium based treatment without employing special construction methods that are beyond the scope of this standard.

14. Calculation

14.1 Correct the average reading of each specimen using the calibration curve determined in [Section 12](#) by locating the reading of the specimen on the bottom of the scale, follow a vertical line to the curve, and then following a horizontal line to the vertical scale ([Note 7](#)). The corresponding value on the vertical scale is the corrected average reading.

14.2 Calculate the average corrected reading for the sample, R_{ac} , by averaging the specimens' corrected readings.



1. Wave length selection
2. Printer button
3. Concentration factor adjustment
4. UV mode selector (Deuterium lamp)
5. Readout display
6. Specimen compartment
7. Zero control (100 % T)
8. Sensitivity switch
9. Power switch

FIG. 1 Schematic of a Typical Bench/Desktop Colorimeter

14.3 Calculate the water soluble sulfate concentration, WSS_{avg} , to the nearest whole number in ppm using the following equation:

$$WSS_{avg} = R_{ac} \times D \quad (1)$$

where:

- WSS_{avg} = water soluble sulfate concentration, nearest whole ppm
- R_{ac} = average corrected reading of the sample, nearest whole ppm
- D = final dilution

15. Report: Test Data Sheet(s)/Form(s)

15.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.5 and Practice D6026.

15.2 Record as a minimum the following general information (data):

15.2.1 Identification of the soil being tested, such as the project identification, boring number, sample number, sample location, collection methods, and depth.