Designation: C1580 – 15

Standard Test Method for Water-Soluble Sulfate in Soil[1]

This standard is issued under the fixed designation C1580; 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 is for the determination of water-soluble sulfate in soils.

1.2 This test method was developed for concentrations of water-soluble sulfate in soils between 0.02 and 3.33 % sulfate by mass.

1.3 This test method does not determine sulfur in any form except as sulfate.

1.4 Some governing bodies regulate the movement of soils from one area to another. It is up to the sampler and laboratory to comply with all regulations.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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 and health practices and to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C125 Terminology Relating to Concrete and Concrete Aggregates
C114 Test Methods for Chemical Analysis of Hydraulic Cement
D1193 Specification for Reagent Water
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology C125.

4. Significance and Use

4.1 This test method can be used to determine if soils could have an adverse reaction with hydraulic cement concrete.

5. Apparatus

5.1 *Photometer*—One of the following, given in order of preference:

5.1.1 Nephelometer or turbidimeter,

5.1.2 Spectrophotometer for use at 420 nm with light path of 4 to 5 cm, and

5.1.3 Filter photometer with a violet filter having a maximum near 420 nm and a light path of 4 to 5 cm. Filter photometers and photometric practices prescribed in this test method shall conform to Practice E60; spectrophotometer practices shall conform to Practice E275.

5.2 *Stopwatch,* readable to 0.1 minutes.

5.3 *Measuring Spoon,* capacity 0.2 to 0.3 mL.

5.4 *Drying oven,* capable of continuously heating at 110 ± 5 °C.

5.5 *Balance,* shall be capable of reproducing results within 0.0002 g with an accuracy of ±0.0002 g. Direct-reading balances shall have a sensitivity not exceeding 0.0001 g. Conventional two-pan balances shall have a maximum sensibility reciprocal of 0.0003 g. Any rapid weighing device that may be provided, such as a chain, damped motion, or heavy riders, shall not increase the basic inaccuracy by more than 0.0001 g at any reading and with any load within the rated capacity of the balance.

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[1] This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.69 on Miscellaneous Tests.

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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.

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

5.6 *Stirrer,* magnetic variable speed, with a TFE-fluorocarbon coated magnetic stirring rod or an overhead stirrer with a propeller.

6. Reagents and Materials

6.1 *Purity of Reagents*—All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[3]

6.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D1193, Type I. Other reagent water types (See Note 1) may be used, provided it is first ascertained that the water is of sufficiently high purity to permit its use without adversely affecting the precision and bias of the test method.

NOTE 1—Type II water was specified at the time of round robin testing of this test method.

6.3 *Barium Chloride*—Crystals of barium chloride ($BaCl_2 \cdot 2H_2O$) screened to 850 to 600 µm. To prepare in the laboratory, spread crystals over a large watch glass, desiccate for 24 h, screen to remove any crystals that are not 850 to 600 µm, and store in a clean, dry jar.

6.4 *Conditioning Reagent*—Place 30 mL of concentrated hydrochloric acid (HCl, sp gr 1.19), 300 mL reagent water, 100 mL 95 % ethanol or isopropanol and 75 g sodium chloride (NaCl) in a container. Add 50 mL glycerol and mix.

6.5 *Sulfate Solution, Standard* (1 mL = 0.100 mg SO_4)—Dissolve 0.1479 g of anhydrous sodium sulfate (Na_2SO_4) in water, and dilute with water to 1 L in a volumetric flask.

6.6 *pH paper,* readable to at least 0.5 pH units.

6.7 *Sodium Hydroxide, (0.1N)*—Dissolve 4 g of sodium hydroxide (NaOH) in water and dilute with water to 1L

6.8 *Hydrochloric Acid (0.1N)*—Dilute 8.6 mL of concentrated hydrochloric acid (HCl sp gr 1.19) to 1L with water

7. Permissible Variations

7.1 Both the referee method and qualified methods as described in Test Methods C114 are permissible as alternatives to the turbidimetric procedure.

8. Calibration and Standardization

8.1 Prepare standards by diluting 0.0, 2.0, 5.0, 10.0, 15.0, 20.0, 30.0, and 40.0 mL of standard sulfate solution with water to 100-mL volumes in volumetric flasks. These solutions will have sulfate ion concentrations of 0.0, 2.0, 5.0, 10.0, 15.0, 20.0, 30.0, and 40.0 mg/L, respectively. Follow the procedure given in Section 9 using appropriate amounts of the standard sulfate solution prepared in accordance with 6.5 and prepare a calibration curve showing sulfate ion content in mg/L plotted against the corresponding photometer readings.

8.2 A separate calibration curve must be prepared for each photometer and a new curve must be prepared if it is necessary to change the cell, lamp, or filter, or if any other alterations of instrument or reagents are made. Check the curve with each series of tests by running two or more solutions of known sulfate concentrations in the range of 5 to 40 mg SO_4/L.

NOTE 2—The slope of the curve may not be linear below 5 or above 40 mg SO_4/L for this method.

9. Procedure

9.1 Collect at least 100 g of representative soil for analysis. Dry the sample for 18 to 24 h at 110 °C. Crush to pass a 600 µm sieve. In a 400 mL beaker labeled Sample A, put a sample consisting of approximately 30 g. In a second 400 mL beaker labeled Sample B, place a sample of approximately 3 g. Record the mass of each sample to the nearest 0.001 g. Add 250 mL of deionized water to each beaker, and stir on a magnetic stirrer for at least 1.0 h but no more than 1.2 h. Filter the extraction through two dry medium texture filter papers under suction. Do not wash the residue. Repeat the filtration if solution is not clear. Check the pH of the filtrate using pH paper and, if needed, neutralize each filtrate to pH of 7 ± 1 using either 0.1N HCl or 0.1N NaOH.

9.2 Adjust the temperature of the solution in the volumetric flasks to between 20 and 25 °C [68 to 77 °F].

9.3 Use an aliquot of 10 mL for specimen A1, 20 mL for specimen A2, 10 mL for specimen B1, and 20 mL for specimen B2. Dilute to a volume of 100 mL in a volumetric flask and follow the procedure below or Test Methods C114. Pour the 100 mL into a 250 mL beaker. Add 5.0 mL of conditioning reagent. Mix by stirring with a stir bar and magnetic stirrer. Add 0.3 g of the prepared $BaCl_2$ crystals using a measuring spoon and start the timer.

9.4 Stir exactly 1.0 min, at the same constant speed for all determinations.

9.5 Remove the beaker from the stirrer, pour solution into the cell, and put the cell into the photometer. Record the turbidity at 4.0 min after pouring the solution into the cell.

9.6 Determine the mg of SO_4 from the calibration curve.

10. Calculation or Interpretation of Results

10.1 Calculate the sulfate content (% by mass) for each specimen as follows:

$$P = \frac{2.5*M}{A*W} \tag{1}$$

where:

P = % SO_4 in dried soil
M = mg/L of SO_4 calculated from calibration curve
W = mass of soil placed into 400 mL beaker, in g
A = volume of aliquot specimen, in mL

10.2 Limits of values for results to be valid. (See Note 3)

Specimen	Lower Limit	Upper Limit
A1	0.04%	0.33%
A2	0.02%	0.17%
B1	0.41%	3.33%
B2	0.21%	1.67%

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[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

1. Determine which results fall within the above limits
2. Discard those that do not
3. Average all results that comply

NOTE 3—These values were derived from the limits on the sulfate test of 5 to 40 mg/L, and a solubility of sulfate of 1.6 g/L at pH 7 when calcium sulfate is present. The method is not applicable for soils outside this range. For those samples, see the Annex.

11. Report

11.1 Report the average obtained as % sulfate by mass of sample. If milligrams sulfate in kilograms of soil (mg SO_4/kg soil) is desired, multiply % sulfate by 10 000. Report results to nearest 0.01 % or 100 mg/kg.

12. Precision and Bias[4]

12.1 *Precision*—The standard deviation was found to vary with concentration, so therefore, a coefficient of variation was used for the precision statement. The single operator coefficient of variation was found to be 4.4 %. Therefore, results of two properly conducted tests by the same operator on the same material should not differ by more than 12.4 % of their average. The multilaboratory coefficient of variation was found to be 21.2 %. Therefore, results of two different laboratories on identical samples of a material should not differ from each other by more than 60 % of their average.

12.2 *Bias*—Since there is no accepted reference material suitable for determining bias, bias can not be determined.

13. Keywords

13.1 hydraulic cement; soil; Sulfate; turbidimeter

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C09-1029. Contact ASTM Customer Service at service@astm.org.

ANNEX

(Mandatory Information)

A1. REDUCING THE NUMBER OF SUB-SAMPLES AND SAMPLES OUT OF COMPLIANCE

A1.1 This procedure was written to accommodate laboratories that are not familiar with the soil under test. If prior knowledge is available, a lesser number of sub-samples, and/or dilutions may be used.

A1.2 If a sample falls above 3.33 % sulfate, then different sample masses and/or dilutions can be used. If this procedure is followed, then the mg SO_4 from the turbidimeter reading shall be maintained within 5 to 40 mg/L. If another procedure is used, then validation shall be provided showing that the results are within the linear range of the calibration.

SUMMARY OF CHANGES

Committee C09 has identified the location of selected changes to this test method since the last issue, C1580 – $09^{\varepsilon 1}$, that may impact the use of this test method. (Approved Oct. 1, 2015.)

(1) Added Section 3.

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