Designation: D 806 – 00

Standard Test Method for Cement Content of Hardened Soil-Cement Mixtures[1]

This standard is issued under the fixed designation D 806; 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 covers the determination by chemical analysis of cement content of hardened soil-cement mixtures.

1.2 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D 6026.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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.* For specific hazard precautions, see Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*

C 125 Terminology Relating to Concrete and Concrete Aggregates[2]

C 219 Terminology Relating to Hydraulic Cement[2]

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[3]

D 2901 Test Method for Cement Content of Freshly Mixed Soil-Cement[3]

D 3740 Practice for the Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and Rock Used in Engineering Design and Construction[3]

D 4753 Specification for Evaluating, Selecting, and Specifying Balances and Scales for Use in Soil, Rock, and Construction Materials Testing[3]

D 5982 Test Method for Determining Cement Content of Fresh Soil-Cement (Heat of Neutralization Method)[4]

D 6026 Practice for Using Significant Digits in Calculating and Reporting Geotechnical Test Data[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[2]

E 145 Specifications for Gravity-Convection and Forced-Ventilation Ovens[2]

E 832 Specification for Laboratory Filter Papers[2]

3. Terminology

3.1 *Definitions*:

3.1.1 Refer to Terminology D 653 for definitions of terms relating to soil.

3.1.2 Refer to Terminologies C 125 and C 219 for definitions of terms relating to cement.

4. Significance and Use

4.1 This test method determines cement content in mixtures of cement with soil or aggregate by chemical analysis. It was developed primarily for testing samples for which a significant degree of cement hydration or hardening has taken place. Test Methods D 2901 or D 5982 may be used for determining cement content of freshly mixed soil-cement mixtures.

4.2 This test method is based on determination by chemical analysis of the calcium oxide (CaO) content of the sample. The method may not be applicable to soil-cement materials containing soils or aggregates which yield significant amounts of dissolved calcium oxide (CaO) under the conditions of the test.

NOTE 1—The agency performing this test method can be evaluated in accordance with Practice D 3740. Not withstanding statements on precision and bias contained in this test method: the precision of this test method 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 D 3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D 3740 does not, in itself, ensure reliable testing. Reliable testing depends on many factors; Practice D 3740 provides a means of evaluating some of these factors.

5. Apparatus

5.1 *Analytical Balance*—An analytical balance conforming to the requirements of Class GP2 in Specification D 4753 and with Class S weights.

5.2 *Filter Paper*—Filter paper including Whatman No. 1, 11 and 15 cm in diameter; Whatman No. 41, 15 cm in diameter; and Whatman No. 2, 11 or 15 cm in diameter.

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[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.15 on Stabilization with Admixtures.

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[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] *Annual Book of ASTM Standards*, Vol 04.08.

[4] *Annual Book of ASTM Standards*, Vol 04.09.

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

5.3 *Fifty-Millilitre-Pipet*.

5.4 *Drying Oven*—Thermostatically controlled, preferably of the forced-air type, meeting the requirements of Specification E 145 and capable of maintaining a uniform temperature of 110 ± 5°C throughout the drying chamber.

5.5 *Miscellaneous Apparatus*—Supplementary equipment, such as electric ovens, hot plates, a small riffle, a No. 40- (425 µm-) sieve with bottom pan and cover, a cast iron mortar and pestle, and a ball mill if possible.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Potassium Permanganate, Standard Solution* (0.1 *N*)—Prepare and standardize a 0.1 *N* $KMnO_4$ solution.

NOTE 2—The use of a standard 0.1 *N* $KMnO_4$ solution is not necessary when the samples are titrated in accordance with 8.9 and the results are calculated in accordance with 9.2.

6.3 *Ammonium Nitrate Solution*—Dissolve 20 g of NH_4NO_3 in 1L of distilled water.

6.4 *Hydrochloric Acid (1 + 3)*—Add 200 mL of HCl (sp gr 1.19) to 600 mL of distilled water.

6.5 *Hydrochloric Acid (1 + 1)*—Add 25 mL of HCl (sp gr 1.19) to 25 mL of distilled water.

6.6 *Nitric Acid*—See Note 3.

6.7 *Ammonium Oxalate Solution* (5 %)—50 g of ammonium oxalate. (**Warning**—In addition to other precautions, this is done by adding the acid, slowly while stirring, to the water to avoid a sudden temperature rise that could cause boiling and spattering of the acid solution.)

6.8 *Ammonium Hydroxide, NH_4OH* (sp gr 0.90).

6.9 *Sulfuric Acid (1 + 1)*—Add 500 mL H_2SO_4 (sp gr 1.84) to 500 mL of distilled water.

7. Samples

7.1 Samples of the following shall be selected for the test:

7.1.1 *Raw Soil*, representative of the soil phase of the soil-cement mixture.

7.1.2 *Cement*, representative of the cement phase of the soil-cement mixture, and

7.1.3 *Soil-cement Mixture* to be analyzed.

7.2 The gross laboratory sample of each component shall be approximately 200 g. This may be obtained by reducing the sample in bulk and, if necessary, in particle size through the use of drying, riffling and grinding processes.

8. Procedure

8.1 Dry 25 g of each of the samples in an oven to constant mass at 110 ± 5°C (230 ± 9°F) to remove free water. Reduce the samples to pass a No. 40- (425 µm-) sieve.

8.2 Using an analytical balance, prepare the following amounts for each of the samples: raw soil, 5 g; soil-cement mixture, 5 g; and cement, 1 g. Place each of the weighed samples in a 250-mL beaker. Add 50 mL of HCl (1 + 1) (Note 3) to each sample, cover, and boil *gently* for 5 min on the hot plate.

NOTE 3—In the case of the cement sample, it is usually preferable first to add 40 mL of water and then stir to obtain a thorough mixture. Then add 10 mL of HCl (sp gr 1.19) and boil *gently* just long enough to obtain decomposition of the cement. Vigorous or extended boiling of soil or cement samples is seldom necessary, and often results in much slower filtration.

8.3 Add 25 mL of hot water to the beakers, stir, allow to settle momentarily, and then decant the contents through a Whatman No. 1 filter paper (Note 4), preferably 15 cm in diameter. The filtrate should be received in a 250-mL volumetric flask. When the liquid has passed through the filter paper, wash the residue once by decantation, using hot water; then transfer it to the filter, using a stream of hot water. The beaker should be rapidly policed, the loosened material being transferred to the filter paper. The material on the filter should then be washed an additional four times, each washing consisting of 10 to 15 mL of hot water directed in a stream from the wash bottle. Very small amounts of residue will occasionally pass through the filter. These ordinarily may be disregarded.

NOTE 4—In the case of the soil and soil-cement samples, the bulk of the residue sometimes slows filtration appreciably. No difficulty is usually encountered from cement samples, and, as a rule, soil samples may be filtered and washed in less than 30 min. Some soil-cement mixtures require more time, but, if this period exceeds 1 h, subsequent filtration in similar cases may be more rapid if a No. 41 paper is substituted for the No. 1 paper. Slow filtration in such cases is generally caused by excessive boiling, resulting in gelation of the silica, which materially retards filtration.

8.4 When washing has been completed, discard the filter, and dilute the filtrate in the volumetric flask to 250 mL with cold water. The temperature of the solution should be near the calibration point of the flask. Agitate the flask to mix the contents thoroughly, then remove a 50-mL aliquot and transfer to the original 250-mL beaker (8.2), using a 50-mL pipet. Dilute to 100 mL. Make the solution slightly ammoniacal (Note 5), boil 1 to 2 min, and allow the hydroxides to settle.

NOTE 5—If the samples contain ferrous iron it is desirable to add a few drops of HNO_3 before precipitation of the hydroxides.

8.5 Filter the hydroxides through an 11-cm Whatman No. 1 (or No. 41) filter paper, receiving the filtrate in the 600-mL beaker. Wash the original 250-mL beaker into the filter once with a stream of hot NH_4NO_3 solution (20 g/L), and follow by washing the hydroxide precipitate once or twice with hot NH_4NO_3 solution (20 g/L). Set the filtrate aside, and place the original beaker under the funnel. Perforate the paper with a rod (Note 6), and wash the hydroxides down into the original beaker, using a stream of hot NH_4NO_3 solution (20 g/L) to remove most of the precipitate from the filter paper. Treat the

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[5] "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 "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."