Designation: D806 – 11

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

This standard is issued under the fixed designation D806; 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 D6026.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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:*[2]

C125 Terminology Relating to Concrete and Concrete Aggregates

C219 Terminology Relating to Hydraulic Cement

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D2901 Test Method for Cement Content of Freshly Mixed Soil-Cement

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

D5982 Test Method for Determining Cement Content of Fresh Soil-Cement (Heat of Neutralization Method)

D6026 Practice for Using Significant Digits in Geotechnical Data

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

E832 Specification for Laboratory Filter Papers

3. Terminology

3.1 *Definitions:*

3.1.1 Refer to Terminology D653 for definitions of terms relating to soil.

3.1.2 Refer to Terminologies C125 and C219 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 D2901 or D5982 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 D3740. 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 D3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D3740 does not, in itself, ensure reliable testing. Reliable testing depends on many factors; Practice D3740 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 D4753 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.

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

Current edition approved Nov. 1, 2011. Published January 2012. Originally approved in 1944. Last previous edition approved in 2006 as D806–00(2006). DOI: 10.1520/D0806-11.

[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**

5.3 *Fifty-Millilitre-Pipet.*

5.4 *Drying Oven*—Thermostatically controlled, preferably of the forced-air type, meeting the requirements of Specification E145 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.[3] 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 a 0.1 *N* $KMnO_4$ solution and standardize against primary standard grade oxalic acid, sodium oxalate or iron (II) ammonium sulfate hexahydrate.

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. However, the actual reagent concentration must be determined by titration against primary standard grade oxalic acid, sodium oxalate or iron (II) ammonium sulfate hexahydrate.

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 25 mL of water and then stir to obtain a thorough mixture. Then add 25 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) by dropwise addition of ammonium hydroxide, 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. Also, pH paper can be used to assure that enough ammonium hydroxide has been added.

8.5 Filter the hydroxides through an 11-cm Whatman No. 1 (or No. 41) filter paper, receiving the filtrate in the 600-mL

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

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 paper with 20 mL of hot HCl (1 + 3), directing the acid over the paper with a glass rod. Wash the paper several times with hot water, and then discard the paper. Dilute the solution to 75 mL.

NOTE 6—Instead of perforating the filter paper, the paper and precipitate may be transferred to the original beaker, the hydroxides dissolved with 20 mL of hot HCl (1 + 3) and diluted to 75 mL with water, and the procedure continued as described in 8.6. In this case, the reprecipitated hydroxides and pulp are subsequently removed simultaneously.

8.6 Make the solution slightly ammoniacal and boil 1 to 2 min. Allow the precipitate to settle, then decant through a Whatman No. 1 paper as before, receiving the filtrate in the 600-mL beaker previously set aside (8.5). Wash and police the beaker in which precipitation took place, finally washing the precipitate on the filter three or four times with NH_4NO_3 solution (20 g/L). Discard the hydroxide precipitate. Add 2 mL of NH_4OH (sp gr 0.90) to the filtrate, which will now have a volume of 250 to 350 mL. Heat the solution to boiling and add 10 mL of hot saturated ammonium oxalate solution. Keep the mixture near boiling until precipitate becomes granular; then set aside on a warm hot plate for 30 min or more. Before filtering off the calcium oxalate, verify completeness of precipitation, (Note 7) and make sure that a slight excess of NH_4OH is present. Filter the mixture through an 11-cm or 15-cm Whatman No. 2 filter paper, or if preferred a Whatman No. 42 paper, making sure that all the precipitate is being retained. Thoroughly clean with a rubber policeman the beaker in which precipitation took place, and transfer the contents to the filter with a stream of hot water. Wash the filter eight to ten times with hot water (not over 75 mL) (Note 8), using a stream from the wash bottle.

NOTE 7—The calcium oxalate precipitation is sometimes not complete and this results in low CaO values. The precipitation operation must be carried out with thoroughness and care.

NOTE 8—The filter may be washed four times each with NH_4OH (2 + 98) and hot water, in the order stated.

8.7 Carefully open the filter paper and wash the precipitate into the beaker in which the precipitation was effected. Dilute to 200 mL and add 10 mL of H_2SO_4 (1 + 1). Heat the solution just short of boiling, and titrate it with the standard $KMnO_4$ solution (Note 9) to a persistent pink color. Add the filter paper and macerate it. Continue the titration slowly until the pink color persists for 10 s.

NOTE 9—The temperature of the standard $KMnO_4$ solution should not vary from its standardization temperature so much as to cause a serious error in the determination of CaO. At ordinary room temperatures the volume of pure water changes to the extent of 0.01 to 0.04 % for each degree Celsius, depending on the temperature.

8.8 *Blank*—Make a blank determination, following the same procedure and using the same amounts of all reagents.

8.9 *Alternative Titration Procedure*—Titrate as described in 8.7, except that the $KMnO_4$ solution need not be a standard 0.1 *N* solution, but the same solution shall be used in titrating all the components. Omit the blank determination described in 8.8.

9. Calculation

9.1 Calculate the cement content of the soil-cement mixture as follows:

9.1.1 When the determination has been completed in accordance with 8.7 and 8.8:

9.1.1.1 Calculate the percentages of CaO in the soil, the cement, and the soil-cement mixture as follows:

$$\text{CaO, \%} = [((A - B)C \times 0.028)/D] \times 100 \quad (1)$$

where:

A = $KMnO_4$ solution required for titration of the sample, mL,
B = $KMnO_4$ solution required for titration of the blank, mL,
C = normality of the $KMnO_4$ solution,
D = sample represented by the aliquot titrated (Note 10), g, and
0.028 = CaO equivalent of 1 mL of 1.0 *N* $KMnO_4$ solution.

9.1.1.2 Calculate the percent cement by mass of soil as follows (Note 11):

$$\text{Cement, \%} = [(G - F)/(E - F)] \times 100 \quad (2)$$

where:

E = CaO in cement, %,
F = CaO in raw soil, %, and
G = CaO in soil-cement mixture,% .

NOTE 10—The aliquots titrated are equivalent to 1 g of soil or soil-cement and 0.2 g of cement.

NOTE 11—The value for percentage by mass of soil obtained in accordance with 9.1 or 9.2 is in terms of hydrated cement. Such values may be converted to an approximate equivalent of dry cement by multiplying them by the factor 1.04.

9.2 When the determination has been completed in accordance with 8.9, calculate the percent cement by mass of soil as follows (Note 11):

$$\text{Cement, \%} = [(J - I)/(5H - I)] \times 100 \quad (3)$$

where:

H = $KMnO_4$ solution required for titration of the sample of cement, mL,
I = $KMnO_4$ solution required for titration of the sample of raw soil, and mL,
J = $KMnO_4$ solution required for titration of the sample of soil-cement mixture, mL.

10. Precision and Bias

10.1 *Precision*—Test data on precision is not presented due to the nature of the soil materials being tested by this test method. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program.

10.1.1 Subcommittee D18.15 is seeking any data from the users of this test method that might be used to make a limited statement on precision.

10.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

11. Keywords

11.1 cement content; durability; soil-cement; soil-cement mixtures; soil stabilization

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this test method since the last issue, D806–00(2006), that may impact the use of this test method. (Approved November 1, 2011)

(*1*) Revised 6.2 to clarify the standardization of $KMnO_4$.
(*2*) Clarified Note 2.
(*3*) Revised 8.4 by adding method to make solution ammoniacal.
(*4*) Added additional alternative to Note 5 for assuring sufficient ammonium hydroxide addition.