



## Standard Test Method for Cement Content of Freshly Mixed Soil-Cement<sup>1</sup>

This standard is issued under the fixed designation D 2901; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope \*

1.1 This test method covers determination of the cement content of samples of freshly-mixed soil-cement.

1.2 *This standard does not purport to address all of the safety problems, 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.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 806 Test Method for Cement Content of Soil-Cement Mixtures<sup>2</sup>

D 1293 Test Methods for pH of Water<sup>3</sup>

D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction<sup>2</sup>

D 4753 Specification for Evaluating, Selecting, and Specifying Balances and Scales for Use in Testing Soil, Rock, and Related Construction Materials<sup>2</sup>

E 11 Specification for Wire-Cloth Sieves for Testing Purposes<sup>4</sup>

### 3. Summary of Test Method

3.1 A weighed sample of the fresh material passing a 4.75 mm sieve is extracted with an ammonium chloride solution. After allowing the sample to settle, an aliquot of the supernatant is diluted with water. The pH is adjusted to 13 and triethanolamine is added to complex interferants. The sample is titrated with EDTA solution to the blue endpoint of hydroxynaphthol blue indicator. The cement content is read from a calibration curve prepared from titrations of several known cement content mixes made with the same water, soil, and cement as the sample.

### 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 as a relatively quick test for use at the construction site to determine compliance with specifications. The method is for materials tested soon after the completion of mixing; Test Method D 806 may be used for testing samples for which a significant degree of cement hydration or hardening has taken place.

NOTE 1—Notwithstanding the 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 which 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 assure reliable testing. Reliable testing depends on many factors; Practice D 3740 provides a means of evaluating some of those factors.

### 5. Apparatus

5.1 *Balance*, meeting Specification D 4753 (Class GP2) and having a capacity of 1000 g or more and a sensitivity of 0.1 g.

5.2 *Glassware*—A 25-mL and a 1000-mL graduated cylinder, 25-mL burets, 10-mL syringes or volumetric pipets, 250-mL beakers, medicine droppers.

5.3 *Plasticware*, 2-L (2-qt) polyethylene containers with snap-on covers, 300 mm (12-in.) diameter plastic funnel, 20-L polyethylene bottles for ammonium chloride and distilled or demineralized water.

5.4 *Buret Stand* for 25-mL buret.

5.5 *Magnetic Stirrer and Stirring Bar*.

5.6 *Stirring Rods*, stainless steel stirring rods approximately 300 mm (12-in.) long.

5.7 *pH Meter, or Indicator Paper* (pH range from 10 to 14)—A portable, combination-electrode pH meter is recommended to obtain accurate results as the pH adjustment is made. Indicator paper can be used as an alternate, but less accurate, means of measuring the pH adjustment.

5.8 *Sieve*—A 4.75 mm (No. 4) sieve conforming to the requirements of Specification E 11.

### 6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

<sup>1</sup> 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.

Current edition approved Jan. 10, 1999. Published April 1999. Original edition published as D 2901 – 70. Last previous edition D 2901 – 93.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.08.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>5</sup> 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 *Ammonium Chloride Solution* (10 %)—Transfer 2000 g of granular ammonium chloride (NH<sub>4</sub>Cl) to a 20-L plastic bottle. Make up to 20 L with warm, distilled or demineralized water and mix well.

6.3 *EDTA Solution* (0.1 M), is available as a standardized, pre-prepared solution from many chemical supply houses.<sup>6</sup> Or, this solution may be prepared by the user as follows: Dissolve 74.45 g of disodium (ethylenedinitrilo) tetraacetate dihydrate (Na<sub>2</sub>C<sub>10</sub>H<sub>14</sub>N<sub>2</sub>O<sub>8</sub>·2H<sub>2</sub>O) powder in approximately 1 L of warm, distilled or demineralized water in a beaker. Cool to room temperature, transfer quantitatively to a 2-L volumetric flask and dilute to the mark with distilled or demineralized water. Store in a polyethylene bottle.

6.4 *Hydroxynaphthol Blue Indicator Powder*.<sup>7</sup>

6.5 *Sodium Hydroxide Solution* (50 %), is available as a low-in-carbonate, pre-prepared reagent.<sup>8</sup> Dilute 1+1 with distilled or demineralized water for use.

6.5.1 Or, this solution may be prepared by the user as follows: Very slowly and cautiously add 500 g of sodium hydroxide (NaOH) pellets to 600 mL of distilled or demineralized water and allow to cool to room temperature. Dilute to 1 L with distilled or demineralized water. Store in a plastic bottle. Dilute 1+1 with distilled or demineralized water for use.

6.6 *Triethanolamine Solution* (20 %)—Dilute 20 mL of reagent grade triethanolamine (HOCH<sub>2</sub>CH<sub>2</sub>)<sub>3</sub>N to 100 mL with distilled or demineralized water.

6.7 *pH 7 and pH 12.5 Buffer Solutions*, used to calibrate pH meter.

## 7. Sampling

7.1 Take representative samples of the soil-cement mixture at the completion of mixing. Test the samples immediately or place in covered plastic containers and test within 30 min of the completion of mixing. Depending on whether the sample contains material retained on a 4.75-mm sieve, follow either 7.1.1 or 7.1.2 below.

7.1.1 For soil-cement mixtures with 100 % passing a 4.75-mm sieve, weigh a 300.0-g portion and titrate as described in Section 9.

7.1.2 For soil-cement mixtures with material retained on a 4.75-mm sieve, weigh a 700.0-g sample. Screen the sample thoroughly on a 4.75-mm sieve until all the material retained is free of smaller adhering particles. Weigh and record as  $M_{fcw}$  the

total material passing the sieve. Mix the material passing the sieve and weigh a 300.0-g portion and titrate as described in Section 9.

NOTE 2—If a correction is to be made for variations in water content, determine the water content,  $w'$ , of a separate portion of the material passing a 4.75-mm sieve. Computations for the correction are given in Note 7.

## 8. Preparation of Calibration Curve

8.1 From the materials to be used for construction, prepare three sets of duplicate samples at the design water content containing the following amounts of cement: Set 1, two samples at 75 % of the design cement content, Set 2, two samples at 100 % of the design cement content, and Set 3, two samples at 125 % of the design cement content.

NOTE 3—If necessary, additional calibration points may be established to cover a wider range of cement contents.

For each sample calculate quantities of soil, cement, and water as follows:

$$M_s = S[(1 + w/100)(1 + C/100)] \quad (1)$$

$$M_r = (R/100) \times M_s$$

$$M_f = M_s - M_r$$

$$M_c = (C/100) \times M_s$$

$$V_w = (w/100)(M_s + M_c)$$

where:

$w$  = design water content, percent by dry mass of soil and cement,  
 $C$  = cement content, percent by dry mass of soil,  
 $R$  = percent material retained on a 4.75-mm (No. 4) sieve,  
 $S$  = sample size, 300.0 g when 100 % of the soil passes a 4.75-mm sieve, 700.0 g when part of the soil is retained on a 4.75-mm sieve.

$M_s$  = total oven-dry mass of soil, g

$M_r$  = mass of material retained on a 4.75-mm sieve, g,

$M_f$  = mass of material passing 4.75-mm sieve, g,

$M_c$  = mass of cement, g, and

$V_w$  = volume of water, mL.

For each sample mix the soil and cement thoroughly to a uniform color. Add the water and mix thoroughly.

NOTE 4—The moisture content of air-dry soil will have a slight effect on the accuracy of the calibration results. This may be corrected by using quantities of soil and water calculated as follows:

$$M_f' = (1 + w_s/100) \times M_f \quad (2)$$

$$V_w' = V_w - (M_f' - M_f) \quad (3)$$

where:

$w_s$  = moisture content of air-dry soil passing 4.75-mm sieve, %.

8.2 Depending on whether the sample contains material retained on the 4.75-mm sieve, follow either 8.2.1 or 8.2.2 as follows:

8.2.1 For soils with 100 % passing a 4.75-mm sieve, titrate each 300.0-g sample as described in Section 9. After titrating the six samples, construct a graph showing millilitres of EDTA solution *versus* percent cement by weight using average figures from Sets 1, 2, and 3.

8.2.2 For soils with material retained on a 4.75-mm sieve, thoroughly screen each 700.0-g sample on the sieve until all the material retained is free of smaller, adhering particles. Mix the material passing the sieve and weigh a 300.0-g portion and titrate as described in Section 9. After titrating the six samples,

<sup>5</sup> *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.

<sup>6</sup> Available from Fisher Scientific, Pittsburgh, PA.

<sup>7</sup> Available from Mallinckrodt Chemical Works, St. Louis, MO, or Fisher Scientific, Pittsburgh, PA.

<sup>8</sup> Available from Fisher Scientific, Pittsburgh, PA.

construct a graph showing millilitres of EDTA solution, using average figures from Sets 1, 2, and 3, *versus* grams of cement,  $M_{c300}$ , in a 300-g sample computed as follows:

$$M_{c300} = [300/(700 - M_r)] \times M_c \quad (4)$$

8.3 Calibration curves are different for each soil type and may be nonlinear.

8.4 Rerun calibration curve each time a new batch of chemicals is introduced.

8.5 Inaccuracies result when the calcium content of test samples differs from that of the calibration samples. Calibration tests should be rerun whenever the samples vary significantly in calcium content, as measured by titration tests on the raw soils (Section 9). Inaccuracies may also result if the source of water used in the calibration samples differs from that of the test sampler.

## 9. Procedure for Titration

9.1 Place each 300.0-g sample into a 2-L (2-qt) polyethylene container and add 500 mL of  $\text{NH}_4\text{Cl}$  solution. Place a cover on the container and shake the mixture for 2 min  $\pm$  2 s. Allow the mixture to settle for 4 min  $\pm$  2 s to obtain a clear supernatant. Syringe or pipet a 10-mL aliquot of the supernatant solution into a 250-mL beaker containing a magnetic stirring bar, add 100 mL of distilled or demineralized water. While thoroughly mixing on a magnetic stirrer, add drops of NaOH solution with a medicine dropper until a pH between 13.0 and 13.5 is obtained as measured by a pH meter or the indicator paper. When a pH meter is used, refer to Test Methods D 1293 for calibration of the meter. When indicator paper is used, use a stirring rod to transfer drops of solution to the indicator paper. After adjusting the pH, wait about 20 to 30 s to insure that the pH does not drift downward from the specified range. Add four drops of triethanolamine solution and add about 0.2 g of indicator powder. Continue stirring the solution on the magnetic stirrer. Titrate with EDTA solution to a pure blue end point. Record the millilitres of EDTA required to obtain the end point.

NOTE 5—A sharper end point may be obtained by adding approximately 90 % of the anticipated quantity of EDTA solution before the addition of NaOH solution.

NOTE 6—All equipment must be kept scrupulously clean by thorough rinsing with distilled or demineralized water. All reagents must be stored in polyethylene containers.

## 10. Calculation

10.1 Depending on whether the sample contains material retained on a 4.75-mm sieve, follow either 10.1.1 or 10.1.2 below.

10.1.1 If 100 % of the soil-cement mixture passes a 4.75-mm sieve, read the cement content by dry weight of soil (exclusive of cement) directly from the calibration curve corresponding to the titration results in millilitres of EDTA for the test sample.

10.1.2 If the soil contains material retained on a 4.75-mm sieve, read grams of cement from the calibration curve corresponding to the titration results in millilitres of EDTA for the test sample. Calculate  $A$  and  $B$  as follows:

$$A = (M_{fcw}/300) \times M_{c300} \quad (5)$$

$$B = 700/[1 + (w/100)] \quad (6)$$

where:

$M_{fcw}$  = grams of moist soil and cement passing the 4.75-mm sieve described in 7.1.2,

$M_{c300}$  = grams of cement read from calibration curve,

$w$  = design water content, %,

$A$  = grams of cement in 700-g sample, and

$B$  = grams of soil and cement in 700-g sample

Then compute  $C$ , percent cement by dry mass of total sample (exclusive of cement) as follows:

$$C = [A/(B - A)] \times 100 \quad (7)$$

NOTE 7—Variations of water content will have a slight effect on the accuracy of test. Correction for water variation,  $C'$ , may be computed as follows:

$$C' = \frac{1 + (w'/100)}{1 + [V_w/(M_f + M_c)]} \times C \quad (8)$$

where

$C'$  = percent cement corrected for water variation,

$C$  = percent cement determined from test sample,

$w'$  = percent water of test sample as determined in Note 2, and

$V_w$ ,  $M_f$  and  $M_c$  are quantities computed in Section 8 for calibration Set 2.

## 11. Precision and Bias

11.1 In the process of development of D 2901-70, interlaboratory testing programs were conducted in 1967 and 1968. Results of these tests showed an average error in cement content determination of 3.6 % [(error)/(cement content)  $\times$  100] from which a coefficient of variation of 4.5 % is estimated. On duplicate specimens used in the calibration process, the average deviation in millilitres of EDTA was 2.4 % [(deviation in millilitres)/(average millilitres)  $\times$  100].

## 12. Keywords

12.1 cement content; soil-cement

**SUMMARY OF CHANGES**

In accordance with D-18 policy, this section identifies the location of the changes to this standard since the last edition (D 2901–93) that may impact the use of this standard.

- (1) Added Practice D 3740 to Section 2, Referenced Documents.
- (2) Added Note 1 for the Practice D 3740 statement.
- (3) Renumbered subsequent notes.
- (4) Summary of Changes section was added.

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