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Standard Test Method for Determining Cement Content of Fresh Soil-Cement (Heat of Neutralization Method)¹

This standard is issued under the fixed designation D5982; 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 procedure for determining the cement content of fresh soil-cement. This test method can be used for determining the cement content of specimens that contain 3 to 16 % cement. This test method is appropriate for soil-cement containing up to 55 % plus 4.75 mm (No. 4) sieve-size particles with a maximum particle size of 75 mm (3 in.). It should not be used for determining the Class F pozzolan content of these mixtures.
- 1.2 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.
- 1.2.1 The procedures used to specify how data are collected/recorded and calculated in this test method are regarded as the industry standard. In addition, they are representative of the significant digits that should generally 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; it is common practice to increase or reduce significant digits of reported data commensurate with such considerations. It is beyond the scope of this test method to consider significant digits used in analysis methods for engineering design.
- 1.3 <u>Units—The</u> values stated in SI units are to be regarded as the standard. The inch-pound units—values given in parentheses are for information only.mathematical conversions to inch-pound units, which are provided for information only and are not considered standard. Reporting of test results in units other than SI shall not be regarded as nonconformance with this standard.
- 1.3.1 The converted inch-pound units use the gravitational system of units. In this system, the pound (lbf) represents a unit of force (weight), while the unit for mass is slugs. The converted slug unit is not given, unless dynamic (F = ma) calculations are involved.
- 1.3.2 It is common practice in the engineering/construction profession to concurrently use pounds to represent both a unit of mass (lbm) and of force (lbf). This implicitly combines two separate systems of units; that is, the absolute system and the gravitational system. It is scientifically undesirable to combine the use of two separate sets of inch-pound units within a single standard. As stated, this standard includes the gravitational system of inch-pound units and does not use/present the slug unit for mass. However the use of balances or scales, recording pounds of mass (lbm) or recording in lbm/ft³ shall not be regarded as nonconformance with 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. Specific precautionary statements are given in Section 8.

2. Referenced Documents

2.1 ASTM Standards:²

C219 Terminology Relating to Hydraulic Cement

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

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

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.

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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.



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 in Geotechnical Data E644 Test Methods for Testing Industrial Resistance Thermometers

3. Terminology

- 3.1 Definitions:
- 3.1.1 Refer to Terminology D653 for terms relating to soil.
- 3.1.2 Refer to Terminology C219 for terms relating to hydraulic cement.
- 3.1 *Definitions*—For definitions of common technical terms used in this standard, refere to Terminology standards D653 and C219.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 *heat of neutralization*—the difference between the temperature of soil-cement/buffer solution after mixing and the average of buffer solution and soil-cement test specimen temperature before mixing.
 - 3.2.2 percent cement—percentage of cement by total mass of specimen.

4. Summary of Test Method

4.1 A representative sample of fresh soil-cement is obtained. The temperature of the soil-cement test specimen and a buffer solution are determined separately and recorded. The buffer solution is added to the soil-cement test specimen and vigorously mixed. After mixing, the temperature of the soil-cement/buffer solution mixture is determined and recorded. The heat of neutralization is calculated and, from a previously established calibration curve, the cement content of the test specimen is obtained.

5. Significance and Use

- 5.1 This procedure provides a means for reliably determining the cement content of soil-cement in approximately 15 to 20 min. The procedure can be used to determine the cement content of soil-cement to ± 1 percentage point by mass of dry sample of the actual cement content, that is generally adequate for most construction control applications.
- 5.2 The buffer solution reacts with the calcium hydroxide in the cement and may react with calcareous material in the soil to produce heat. Any reaction from calcareous soil is accommodated in the calibration curve but below 3 % cement content there is usually insufficient temperature reaction for reliable results. Because Class F pozzolan may not contain any calcium hydroxide for reaction, this test method will not determine percentage Class F pozzolan in the mix.

Note 1—The quality of the result produced by 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/sampling/inspection/ and the like. UserUsers of this standard are cautioned that compliance with Practice D3740 does not in itself assureensure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

- 6.1 Balance or Scale—Chosen in accordance with Specification D4753.
- 6.1.1 A typical balance or scale used for obtaining the mass of a soil-cement test specimen must be readable to 0.01 kg or 0.01 lbm and have a minimum capacity of about 9.1-9 kg (20 lbm).
- 6.1.2 A typical balance or scale used for preparing the buffer solution (see 7.4) must be readable to 0.1 g and have a capacity of about 3000 g-g (6.6 lbm).
- 6.2 Digital Thermometer, 0 to 100°C range, readable to 0.1°C, conforming to the requirements of Test Methods E644. The digital thermometer must be equipped with a thermocouple probe as short as possible and no more than 130 mm (5 in.) in length.
- 6.3 Specimen Container, leakproof, widemouth plastic (Nalgene) container, 4-L (1-gal) capacity, with screw cap, minimum of three recommended. One cap should be pierced in the center to allow insertion of the thermometer probe.
 - 6.4 Timing Device, A a stopwatch or other suitable timing device readable to 1 s.
 - 6.5 Glass or Plastic Beaker, Approximately approximately 3000-mL (0.8-gal) capacity.
 - 6.6 Funnel, Widemouth widemouth funnel to fit mouth of specimen container (see 6.3).
 - 6.7 Hand Scoop, Flatbottom flatbottom and scoop with handle; approximate bowl dimensions, 75 by 150 mm (3-(3\frac{1}{2}) by 6 in.).
 - 6.8 Gloves, Protective protective gloves to be worn whenever handling buffer solution.
- 6.9 Specimen Container Holder, Angle angle irons, woodblocks, or other suitable material capable of holding the specimen container securely in an inverted position.
- 6.10 Buffer Container, Aa suitable container with pouring spout or spigot, preferably plastic, of sufficient capacity to hold a buffer solution supply for daily testing. Each test performed requires 1.5 L (0.4 gal) of buffer solution.



- 6.11 Pail, Plastic plastic pail, minimum 4-L (1-gal) capacity.
- 6.12 Spoon, Largelarge metal spoon for mixing the soil-cement calibration test specimens as described in Section 10.
- 6.13 *Mixing Container*, An-an 11- to 15-L (3- to 4-gal) container, preferably plastic, used for mixing the soil-cement calibration test specimens as described in Section 10.
- 6.14 Shovel or Large Scoop, Square square faced shovel or large scoop, used for obtaining fresh sample from belt, hopper, truck, or site.

7. Reagents and Materials

- 7.1 Distilled water is to be used for preparing the buffer solution as described in 7.4. Tapwater Potable water that is free of acids, alkalies, or oils and is suitable for drinking should be used for rinsing the thermocouple probe and lid.
- 7.2 *Sodium Acetate*—Anhydrous (crystalline) sodium acetate, technical grade or better, 225 g (0.5 lbm) required for each test specimen.
 - 7.3 Glacial Acetic Acid—Liquid glacial acetic acid, technical grade or better, 360 g (0.8 lbm) required for each test specimen.
 - 7.4 Preparation of Buffer Solution: Solution:
 - 7.4.1 Prepare 1.5 liters litres (0.4 gal) of buffer solution for each specimen.
 - 7.4.2 Dissolve 225 g (0.5 lbm) of anhydrous sodium acetate in 500 mL (17 oz) of distilled water, stirring constantly.

Note 2—A blender or malt mixer may be used to help dissolve the anhydrous sodium acetate.

- 7.4.3 Add 360 g of glacial acetic acid to the sodium acetate/water solution prepared in 7.4.2.
- 7.4.4 Add distilled water to bring the final volume to 1.5 L. <u>L (0.4 gal).</u> Mix thoroughly.

Note 3—Subsection 7.4 provides sufficient buffer for one test. If more than one test is to be performed, the proportions given should be adjusted to provide enough buffer solution to complete testing required for that day (see subsection 7.5).

7.5 Buffer solution is to be used within 24 h after it is mixed.

8. Safety Hazards

- 8.1 Chemicals used in this procedure contain acid that can be irritating to exposed skin. Perform this procedure only with adequate ventilation and with rinse water available. Wear rubber gloves, laboratory coat or apron, and safety glasses or goggles.
- 8.2 After mixing the soil-cement/buffer solution and before removing the lid, place a towel over the top of the specimen container. The pressure generated while mixing can cause fluid to spray as the lid is removed.

9. Technical Hazards

- 9.1 The calibration curve is specific to the mix design and material used. If mix design percentages are changed or if the source of material is changed, a new calibration curve must be developed.
- 9.2 Soil-cement with gravel is highly susceptible to segregation during handling. To minimize segregation, use care in obtaining samples, selecting specimens, and during remixing and testing of the soil-cement specimen.
- 9.3 Soil-cement samples are to be tested within 60 min from the time water and cement first come into contact during mixing at the lab, mixing plant, or on site.
 - 9.4 Soil-cement samples must be protected from moisture loss prior to testing.
- 9.5 The temperature difference between the buffer solution and soil-cement specimen should not exceed 4°C. If the temperature difference is equal to or greater than 4°C, heat or cool the buffer solution to within 4°C of the soil-cement specimen.
- 9.6 Gelling or stiffening of the soil-cement/buffer solution mixture may occur when testing specimens having cement contents greater than 16 %. If this occurs, the ratio of mass of buffer solution to mass of soil-cement specimen (1.65 kg buffer solution to 1.50 kg soil-cement) (3.64 to 3.30 lbm) given in this test method cannot be used and a new mass ratio must be established.
- 9.7 The buffer solution used in the test method consists of glacial acetic acid, water, and sodium acetate. Glacial acetic acid is corrosive and ignitable and gives a distinct vinegar odor to the buffer mixture. Sodium acetate and calcium acetate (a reaction product) are not considered to be toxic or hazardous chemicals. The acidity of the buffer solution changes from pH 2 to pH 5.2 during testing and can be disposed of with the cement waste. If, after testing, a higher pH value is desired or if there is unreacted buffer solution, additional cement or lime may be added to the mixture prior to disposal.

10. Sampling

10.1 Obtain a fresh soil-cement sample using either the method described in 10.1.1 or 10.1.2. The method selected depends upon the reason for testing. Subsection 10.1.1 is to be used if the production process at the mixing plant is to be checked. Subsection 10.1.2 is to be used during placement of soil-cement at the time of construction.

- 10.1.1 Using a square faced shovel or large scoop, obtain a minimum 2-kg (5-lbm) sample from the mixing plant. The sample should be taken immediately after the soil-cement is deposited in the truck or onto the site. Segregation of the gravel fraction may occur during the dumping. When obtaining the sample, visually observe if it appears representative of the specified mixture proportions. If the sample does not appear representative, it should be discarded and a new sample obtained. Place the soil-cement sample in a large air-tight bucket or other suitable container (see 9.2 9.4).
- 10.1.2 Using a shovel or large scoop, obtain a minimum 2-kg (5-lbm) sample from the material obtained at the construction site for density or compressive strength testing, or both. When obtaining the sample, visually observe if it appears representative of the specified mixture proportions. If the sample does not appear representative, it should be discarded and a new sample obtained. Place the soil-cement sample in a large air-tight bucket or other suitable container (see 9.2 9.4).

11. Calibration and Standardization

- 11.1 Record all calibration data on an appropriate form or by electronic means. One example of a form that meets the data requirements is the form shown in Fig. ±X1.1.
- 11.2 Establish a calibration curve by determining the heat of neutralization of soil-cement test specimens prepared at known cement contents that bracket the value of percent cement to be used for construction.
- 11.3 Prepare nine 1.50 kg (3.30 lbm) soil-cement calibration specimens using the percentages of gravel, minus 4.75 mm (No. 4) material, and water as determined when the mixture was proportioned. The amount of cement added to the calibration specimens should be as follows: three calibration specimens should be two percentage points less cement than that specified for construction, three specimens two percentage points greater, and three specimens having the same percent cement as specified for construction. An example mixture proportion calculation for one 1.50 kg (3.30 lbm) specimen is shown on Fig. 2X1.2. Note that, in the example, water content values are required and were previously determined in accordance with Test Method D2216 in order to calculate the quantity of each size material needed.
- 11.3.1 In four separate containers place the mass, determined to the nearest 0.01 kg or 0.01 lbm, of gravel, minus 4.75 mm (No. 4) material, water and cement for one 1.50 kg (3.30 lbm) soil-cement calibration specimen.
- 11.3.2 Combine the gravel and minus 4.75 mm (No. 4) material with one-half the required amount of water in an 11- to 15-L (3- to 4-gal) container. Mix thoroughly using a large spoon or other suitable mixing device. Ensure that the gravel is evenly wetted and that no dry clods of minus 4.75 mm (No. 4) material are present in the calibration specimen.
- 11.3.3 Add the cement and the remainder of the water to the calibration specimen. Mix thoroughly to ensure an even distribution of cement throughout the specimen. Ensure that no cement clods are present and no cement is sticking to the sides of the container.
 - 11.3.4 Place the funnel into the mouth of the test specimen container.
- 11.3.5 Using the large spoon, place the soil-cement calibration specimen into the specimen container using care to prevent portions of the calibration specimen from sticking to the sides of the funnel. Ensure that all of the calibration specimen is removed from the mixing container and placed in the specimen container.
 - 11.3.6 Remove the funnel and secure the lid on the soil-cement calibration specimen container.
 - 11.4 Determine the heat of neutralization for the soil-cement calibration specimen in accordance with 13.5 13.20.
 - 11.5 Repeat subparagraphs 11.3.1 11.4 eight additional times to obtain nine heat of neutralization determinations.
- 11.6 Calculate the average of the "temperature difference" (heat rise) values obtained for the three trials performed at each of the three cement contents. The range (difference between highest and lowest) of the three individual temperatures at the same cement content should not exceed 3°C. If it does, repeat the test for that cement content. Record the value to the nearest 0.1°C as the "average temperature difference" as shown on Fig. ‡X1.1.
- 11.7 Prepare a plot of heat of neutralization (average temperature difference) versus cement content as shown on Fig. $4\underline{X1.1}$. Draw and calculate the best straight line through the three or more plotted points.
- 11.8 Determine the equation of the calibration line. It is recommended that a computer/calculator be used to perform a linear regression analysis to obtain the equation of the best-fit line through the data points obtained.

12. Conditioning

12.1 Perform the calibration as described in Section 11 and the procedure as described in Section 13 in an area that is isolated from drafts and heat sources i.e., sources, that is, relatively uniform temperature. It is recommended that the calibration and the testing be performed at the same location.

13. Procedure

- 13.1 All test data are to be recorded on an appropriate form or by electronic means. Fig. $3\underline{X1.3}$ is an illustration of a form meeting the data requirements.
 - 13.2 Place a 4-L (1-gal) specimen container on the scale. Place the funnel into the mouth of the specimen container.
- 13.3 Using a small scoop, place 1.50 kg (3.30 lbm) of the soil-cement sample obtained in Section 9 into the specimen container using care to prevent portions of the specimen from sticking to the sides of the funnel.



- 13.4 Remove the funnel and secure the lid on the soil-cement specimen container.
- 13.5 Carefully obtain 1.65 kg (3.64 lbm) of the buffer solution as prepared in 7.4 (see 8.1).
- 13.6 Submerge the thermometer probe into the buffer solution.
- 13.7 Determine the temperature of the buffer solution after 1 min and record the value to the nearest 0.1°C as the "buffer temperature".temperature."
- 13.8 Thoroughly rinse the thermometer probe and lid using fresh water. Carefully and thoroughly dry the thermometer probe and lid.
 - 13.9 Remove the lid from the soil-cement specimen container.
 - 13.10 Secure the lid that has been pierced by the thermometer probe on the soil-cement specimen container.
- 13.11 Invert the soil-cement specimen container onto a stand. Shake the specimen container to ensure that the entire specimen is at the bottom and that the thermocouple probe is completely covered by the specimen.
- 13.12 Determine the temperature of the soil-cement after 2 min, and record the value to the nearest 0.1°C as the "soil-cement temperature" as shown on Fig. 3X1.3. Remove the specimen container from the stand, and remove the thermometer probe and lid.
- 13.12.1 Verify that the temperature of the buffer and the soil-cement are within 4°C (see 9.5). If not, heat or cool the buffer solution as needed before continuing.
 - 13.13 Add the buffer solution to the soil-cement.
 - 13.14 Secure a lid onto the soil-cement specimen container.
- 13.15 With one hand on the specimen container top and the other on the test specimen container bottom, vigorously shake and rotate the container for 4 min to mix the soil-cement/buffer solution. During the mixing process, continually invert and then upright the specimen container through 180° while also continuously shaking the specimen container. This ensures an even and thorough mixing of the soil-cement/buffer solution.
- 13.16 Remove the lid (see Note 4), and secure the lid pierced by thermometer probe onto the specimen container. Invert the specimen container onto a stand, and determine the temperature of the soil-cement/buffer solution mixture after 1 min.minute.
- Note 4—It is recommended that a towel be placed over the top of the lid before removal; the pressure generated while mixing could cause fluid to spray as the lid is removed.
 - 13.17 Record the value obtained in 13.16 to the nearest 0.1°C as the "mixture temperature" as shown on Fig. 3X1.3.
 - 13.18 Remove the specimen container from the stand and carefully remove the thermometer probe and lid.
- 13.19 Calculate the average of the buffer temperature and the soil-cement temperature and record the value to the nearest 0.1°C as the "average temperature" as shown on Fig. 3X1.3.
- 13.20 Calculate the difference between the mixture temperature and the average temperature and record the value to the nearest 0.1°C as the "temperature difference".difference."
- 13.21 Using either the calibration line equation determined in 11.8 or the calibration curve determined in 11.7 (shown on Fig. ±X1.1), determine the cement content of the specimen.
 - 13.22 Record the value to the nearest 0.1 % as the "calculated cement content (%)" as shown on Fig. 3X1.3.
 - 13.23 Pour the soil-cement/buffer solution mixture into an appropriate waste container for fresh concrete and cap securely.
- Note 5—Because of the acetic acid, the soil-cement/buffer solution mixture will have a vinegar smell. The pH of the soil-cement/buffer solution should now be approximately 6.5 versus 2.5 of the glacial acetic acid-sodium acetate buffer solution alone. It can be safely disposed of with regular waste concrete or soil-cement.
 - 13.24 Thoroughly wash the test specimen container, thermometer probe and lid, scoop, and funnel with water.

14. Calculations

- 14.1 Calculations required to obtain a calibration line equation as described in Section 11 are shown on Fig. ±X1.1.
- 14.2 Calculations required to determine the cement content (%) of the soil-cement test specimens are shown on Fig. 3X1.3.
- 14.3 An example mixture-proportioning calculation for a 1.50 kg (3.30 lbm) soil-cement calibration test specimen is shown on Fig. 2X1.2.

15. ReportReport: 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 1.2.
- 15.2 Record as a minimum the following general information (data):
- 15.2.1 Location where sample were taken



- 15.2.2 Sample information including project identification, date and time of sampling, and sampling personal.
- 15.3 The report is to consist of the information given on the following examples of completed and checked Record as a minimum the sampling and testing data given in the following example forms:
 - 15.3.1 "Cement Content—Heat of Neutralization Calibration Data," Fig. <u>4X1.1</u>.
 - 15.3.2 "Cement Content—Heat of Neutralization Determination," (Calibration Test Specimens Preparation)" Fig. 3X1.2.
 - 15.3.3 "Cement Content—Heat of Neutralization Determination," Fig. X1.3.
 - 15.4 Note any unusual conditions or other data considered necessary to properly interpret test results.
- 15.3 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026 and described in Section 1.

16. Precision and Bias

- 16.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results are written for specimens containing from 3 to 15 % cement and up to 50 % plus No. 4 (4.75-mm) sieve size material. The results are detailed in Fig. 3X1.3 and are summarized as follows:
- 16.1.1 Repeatability for Calibration Specimens—The single-operator standard deviation of a single test result (a test result defined in this procedure as the average of three separate measurements) was found to be 0.5°C. Therefore, results of two properly conducted tests by the same operator (each consisting of the average of three calibration specimens of the same cement content) should not differ by more than 1.5°C.
- 16.1.2 Repeatability for Test <u>Specimens—Specimens—The</u> single operator standard deviation of a single test result (a test result is defined in this procedure as the average of two separate measurements) was found to be 0.14 of a percentage point of cement content. Therefore, results of two properly conducted tests by the same operator (each consisting of the average of two cement content determinations) should not exceed 0.40 percentage points of cement content. The range (difference between the highest and the lowest) of the two individual cement content determinations used in calculating a test result should not exceed 0.55 percentage point of cement content.
- 16.1.3 Reproducibility for Calibration Specimens—The multilaboratory standard deviation of a single test result (a test result is defined in this procedure as the average of three separate measurements) was found to be 0.8°C. Therefore, results of two properly conducted tests in different laboratories on the same soil-cement mix should not differ by more than 2.3°C.
- 16.1.4 Reproducibility for Test Specimens—Specimens—The multilaboratory standard deviation of a single test result (a test result is defined in this procedure as the average of two separate measurements) was found to be 0.26 of a percentage point of cement content. Therefore, results of two properly conducted tests in different laboratories on the same soil-cement mix should not differ by more than 0.74 percentage point of cement content.
- 16.2 Bias—When experimental results are compared with known values from accurately compounded samples, the results of the test method are found with 95 % confidence to lie between ± 0.55 % of actual cement content and exhibit no bias.

17. Keywords

17.1 cement content; heat of neutralization; soil cement; soil stabilization

APPENDIX

X1. SAMPLING AND TESTING DATA FORMS

(Nonmandatory Information)

X1.1 See Figs. X1.1-X1.3.