Designation: D3155 – 11

Standard Test Method for
Lime Content of Uncured Soil-Lime Mixtures[1]

1. Scope*

1.1 This test method covers the determination of the lime content of soil-lime mixtures sampled from a project under construction or at the pug-mill, or both.

1.2 In soils with highly variable amounts of $CaCo_3$ (such as caliche), it may be difficult to obtain a representative sample.

1.3 The values stated in SI units are to be regarded as standard. The values stated in inch-pound units are provided for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C51 Terminology Relating to Lime and Limestone (as used by the Industry)
C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials
D653 Terminology Relating to Soil, Rock, and Contained Fluids
D1193 Specification for Reagent Water
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
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 *Definitions:*

3.1.1 Refer to Terminology C51 for terms relating to lime.

3.1.2 Refer to Terminology D653 for terms relating to soil.

4. Summary of Test Method

4.1 A representative specimen is obtained after thorough mixing of the soil and lime and sieving through a 4.75-mm (No. 4) sieve.

4.2 A 300-g specimen of the material passing the 4.75-mm (No. 4) sieve is shaken together with 600 mL of ammonium chloride solution.

4.3 A 10-mL aliquot of fluid from 4.2 is mixed with 100 mL of water, and sodium hydroxide is added until a pH of 13.0 to 13.5 is obtained.

4.4 Triethanolamine and indicator powder is added to the solution and EDTA is used to titrate to a clear blue endpoint.

4.5 The mL of EDTA used is compared to a calibration curve to determine percent lime by dry mass of the total specimen.

5. Significance and Use

5.1 This test method can be used to determine the lime content of uncured soil-lime mixtures.

5.1.1 Lime content in soil-lime mixtures is needed by agencies such as highway departments, to determine lime content in soil-lime mixtures for payments to contractors, to check compliance with specifications, or to check the efficacy of quality control measures.

5.1.2 Lime content is also needed by producers of soil-lime mixtures who have to determine lime content for production control purposes.

NOTE 1—The quality of the result produced by this standard 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/etc. Users of this standard are cautioned that compliance with Practice D3740 does not in itself ensure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

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

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[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 Jan. 1, 2011. Published February 2011. Originally approved in 1973. Last previous edition approved in 2006 as D3155-98(2006). DOI: 10.1520/D3155-11.

[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.

6. Apparatus

6.1 *Balance,* a class GPI balance having a capacity of 1000 g or more and meeting the requirements of Specification D4753 for a balance of 0.01-g readability.

6.2 *Glassware*—25-mL graduated cylinder, 1000-mL graduated cylinder, 2-L volumetric flask, 25-mL burets, 10-mL volumetric pipets, 250-mL Erlynmeyer flasks, medicine droppers.

6.3 *Plasticware*—1.9-L (2 qt) polyethylene containers with snap-on covers, 300-mm (12 in.) diameter plastic funnel, 19-L (5-gal) polyethylene bottles for ammonium chloride, 19-L (5-gal) polyethylene bottles for water.

6.4 *Buret Stand,* for 25-mL buret.

6.5 *Stirrer and Stirring Bar,* magnetic, or equivalent apparatus.

6.6 *Stirring Rods,* stainless steel, approximately 300 mm (12 in.) in length.

6.7 *pH Meter*—A pH meter equipped with reference electrode and low-sodium error glass pH-sensitive electrodes (or combination electrode) and a meter capable of displaying 0.01 units pH at 0.2 pH accuracy over a range from 0 to 14.

6.8 *Sieve,* 4.75-mm (No. 4), conforming to Specification E11 required for soils containing particles larger than this size.

7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references to water are understood to mean reagent water conforming to Specification D1193, Type II.

7.2 *Purity of Reagent Chemicals* —Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the committee on Analytical Reagents of the American Chemical Society[3], where such specifications are available. 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.

7.2.1 *Ammonium Chloride Solution, 100 g/L*—Transfer 1893 g of USP granular ammonium chloride (NH_4Cl) to a 19-L (5-gal) polyehtylene bottle. Dissolve it in 7.5 to 11.4 L (2 to 3 gal) of water. Dilute to 18.9 L (5 gal) with water, and mix well.

7.2.2 *EDTA Solution (0.1 M)*—Dissolve 74.45 g of reagent grade disodium (ethylenedinitrilo) tetraacetate dihydrate ($Na_2C_{10}H_{14}N_2\ O_8 \cdot 2H_2O$) powder in about 1 L (0.26 gal) of warm water in a beaker. Cool to room temperature, transfer to a 2-L volumetric flask, and dilute to 2 L with water. Store in a polyethylene bottle.

7.2.3 *Sodium Hydroxide Solution, 250 g/L*—With constant stirring, cautiously add 250 g of reagent grade sodium hydroxide (NaOH) pellets to 300 mL of water, stir until dissolved, and allow to cool to room temperature. Dilute to 1 L with water. Store in a polyethylene bottle.

7.2.4 *Triethanolamine Solution, 200 g/L*—Mix 100 mL of reagent grade triethanolamine ($(HOCH_2CH_2)_3N$) in 200 to 300 mL of water in a beaker, dilute to 500 mL, mix well, and store in a polyethylene bottle.

7.2.5 *Hydroxynaphthol Blue Indicator Powder*(CAS No. 63451354)[4]—An indicator for the determination of Calcium by titration with EDTA.

8. Preparation of Calibration Curve

8.1 From the soil and lime materials to be used for construction, prepare three sets of duplicate specimens at the design water content in order that they contain the following amount of lime:

8.1.1 *Set 1*—Two samples at 75 % of the design lime content,

8.1.2 *Set 2*—Two samples at 100 % of the design lime content,

8.1.3 *Set 3*—Two samples at 125 % of the design lime content.

8.2 For each specimen, compute quantities of soil, lime and water as follows:

$$M_s = S/(1+W/100) \times (1+L/100) \tag{1}$$

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

$$M_f = M_s - M_r$$

$$M_l = (L/100) \times M_s$$

$$V_w = (W/100)\,(M_s - M_l)$$

M_s = total dry mass of soil, g,
M_r = mass of material retained on 4.75-mm (No. 4) sieve, g,
M_f = mass of material passing 4.75-mm (No. 4) sieve, g,
M_l = mass of lime, g,
V_w = volume of water, mL,
W = design water content, dry mass % of soil,
L = lime content, dry mass % of soil,
R = material retained on a 4.75-mm (No. 4) sieve, %, and
S = specimen size: 300.0 g when 100 % of the soil passes a 4.75-mm (No. 4) sieve; 700.0 g when soil is retained on a 4.75-mm (No. 4) sieve.

8.3 Mix the dry soil and lime thoroughly to a uniform color. Add the water and mix thoroughly until uniformly moist.

8.4 For soils with 100 % passing a 4.75-mm (No. 4) sieve, titrate each 300-g specimen as described in Section 10. After titrating the six specimens, construct a graph showing mL of EDTA solution versus mass percent lime using average figures from Sets 1, 2, and 3.

8.5 For soils with material retained on a 4.75-mm (No. 4) sieve, weigh a 700-g specimen. Screen the specimen thoroughly on a 4.75-mm (No. 4) sieve until all the material retained is free of smaller adhering particles. Mix the material passing the sieve, weigh a 300 g specimen, and titrate as described in Section 10. After titrating six specimens, construct

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[3] *Reagent Chemicals, American Chemicals Society Specifications,* American Chemical Society, Washington, DC. For suggestions of the testing of reagents not listed by American Chemical *Society Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K. and the *United States Pharmacopoeia*.

[4] Available from Mallinckrodt Balur, Inc., Phillipsburg, NJ, as Product Code Number 5630.

a graph showing mL of EDTA solution, using average figures from Sets 1, 2, and 3, versus grams of lime, $M_{l\ 300}$, in a 300-g specimen computed by:

$$M_{l\ 300} = [300/(700 - M_r)] \times M_l \qquad (2)$$

9. Test Specimen

9.1 During construction, take representative samples of the soil-lime mixture at the completion of mixing and test the samples immediately. If the samples cannot be tested immediately, place them in covered plastic containers and test them within 8 h after complete mixing. In this case, the calibration curve (see Section 8) should be established using soil-lime mixtures mellowed for a time period commensurate to within 1 h of testing of field samples.

9.1.1 For soils with 100 % passing a 4.75-mm (No. 4) sieve, weigh a 300-g specimen and titrate as described in Section 10.

9.1.2 For soils with material retained on a 4.75-mm (No. 4) sieve, weigh a 700-g specimen. Screen the specimen thoroughly on a 4.75-mm (No. 4) sieve until all the material retained is free of smaller adhering particles. Determine the mass of the total material passing the sieve and record as M_{flw}. Mix the material passing the sieve and weigh a 300-g specimen and titrate as described in Section 10.

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 (No. 4) sieve. Computations for the correction are given in Note 5.

10. Titration

10.1 Place each 300-g specimen in a 1.9-L (2-qt) polyethylene container and add 600 mL of NH_4Cl solution. Place the cover on the container and shake the container for 2 min (±2 s). Allow the mixture to settle for 4 min (±2 s). Pipet a 10-mL aliquot of the supernatant solution into a 250-mL Erlenmeyer flask and dilute with 100 mL of water. While thoroughly mixing on a magnetic stirrer, add drops of NaOH solution until a pH between 13.0 and 13.5 is obtained, as measured by the approved pH meter. Add 4 drops of triethanolamine solution and then add approximately 0.2 g of the hydroxynaphthol blue indicator powder. While the solution is being stirred on the magnetic stirrer, titrate with EDTA solution to a pure blue end point, and record the quantity in milliliters.

NOTE 3—A sharper endpoint may sometimes be obtained by adding approximately half of the anticipated quantity of EDTA solution before the addition of the NaOH solution.

NOTE 4—All equipment must be kept scrupulously clean by thorough rinsing with water. All reagents must be stored in polyethylene containers.

11. Calculation

11.1 If 100 % of the soil passes a 4.75-mm (No. 4) sieve, read the lime content by dry mass directly from the calibration curve corresponding to the titration results in mL of EDTA for the test specimen.

11.2 If the soil contains material retained on a 4.75-mm (No. 4) sieve, read grams of lime from the calibration curve corresponding to the titration results in mL of EDTA for the test specimen. Compute *A* and *B* as follows:

$$A = (M_{flw}/300) \times M_{l\ 300} \qquad (3)$$

$$B = 700/[1 + (W/100)]$$

where:

A = lime in 700-g specimen, g,
B = soil and lime in 700-g specimen, g,
M_{flw} = soil passing the 4.75-mm (No. 4) sieve as described in 9.1.2, g,
$M_{l\ 300}$ = lime read from calibration curve, g, and,
W = design water content, %.

Then compute *L*, percent lime by dry mass of total specimen:

$$L = [A/(B - A)] \times 100 \qquad (4)$$

NOTE 5—Variations of water content will have slight effect on the accuracy of test. Correction for water content variation may be computed as follows:

$$L' = \frac{1 + (W'/100)}{1 + [V_w/(M_f + M_t)]} \times L \qquad (5)$$

where:

L' = lime corrected for water content variation, %,
L = lime determined from test specimen, %,
W' = water content of test sample as determined in Section 9 (Note 2), %, and,
V_w, M_f, and M_t = are quantities computed in Section 8 for Calibration Set 2.

12. Precision and Bias

12.1 From an inter-laboratory study based on 60 test results obtained from 3 soils, 5 laboratories, and tests at 4 different lime contents, the following statements can be made:

12.1.1 *Single Operator Precision*—Because only one test was performed on a given soil type at a given percent lime content within a single laboratory, no single operator precision statement can be made.

12.1.2 *Multilaboratory Precision*—The multilaboratory standard deviation has been found to be 0.23 % of lime content and 0.34 mL of EDTA.[5]

12.1.3 *Bias*—When experimental results are compared with known values from accurately compounded specimens, the bias of the test method is found within 95% confidence to lie between –0.02 and 0.73 % of the true lime content.

13. Keywords

13.1 EDTA; lime; soil; soil-lime

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[5] These numbers represent, respectively, the (1s) and (d2s) limits as described in Practice C670.

SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this test method since the last issue, D3155–98(2006), that may impact the use of this test method. (Approved January 1, 2011)

(*1*) Added reference to Specification E11 in 6.8 and Referenced Documents.
(*2*) Revised Note 1 to add the most recent version of the minimum requirements for agencies caveat for D18 compliance.
(*3*) Added the guidance to improve and update the procedure in 6.7.
(*4*) Added the guidance to clarify the chemical's function in the test procedure in 7.2.5.
(*5*) Added the guidance to clarify the procedure for removing small adherent particles from gravel-sized particles in 8.5.
(*6*) Added the guidance to clarify the importance of creating a calibration curve commensurate to sample mellowing and sample testing times in 9.1. Incorporated Old Note 2 into this section and renumbered subsequent notes.

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