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AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Test Method for Lime Content of Uncured Soil-Lime Mixtures[1]

This standard is issued under the fixed designation D 3155; 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 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 metric 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 51 Definitions of Terms Relating to Lime and Limestone[2]

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[2]

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

D 1193 Specification for Reagent Water[4]

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

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

3. Terminology

3.1 *Definitions:*

3.1.1 Refer to Terminology C 51 for terms relating to lime.

3.1.2 Refer to Terminology D 653 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 minimum 4.75-mm (No. 4) material 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 mixes is needed by agencies such as highway departments, that have to determine lime content in soil-lime 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—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 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 several factors; Practice D 3740 provides means of evaluating some of those factors.

6. Apparatus

6.1 *Balance*, a class GPI balance having a capacity of 1000 g or more and meeting the requirements of Specification D 4753.

6.2 *Glassware*—25-mL graduated cylinder, 1000-mL graduated cylinder, 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.

[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 04.01.

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

[4] *Annual Book of ASTM Standards*, Vol 02.05..

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

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 Indicator*—Supply of pH indicator paper or an approved pH meter with a pH range from 10 to 14.

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

7. Reagents

7.1 *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[5], 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.

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

7.1.2 *EDTA Solution (0.1 M)*—Dissolve 74.45 g of a reagent grade disodium (ethylenedinitrilo) tetraacetate dihydrate ($Na_2C_{10}H_{14}N_2O_8 \cdot 2H_2O$) powder in about 1 L of warm water in a beaker. Cool to room temperature, transfer quantitatively to a 2-L volumetric flask, and fill to the mark with water. Store in a polyethylene bottle.

7.1.3 *Sodium Hydroxide Solution, 500g/L*—With constant stirring, cautiously add 500 g of reagent grade sodium hydroxide (NaOH) pellets to 600 mL of water, stir until dissolved, and allow to cool to room temperature. Dilute to 1 L with water. Store in a plastic bottle. Dilute with an equal volume of water for use.

7.1.4 *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 plastic bottle.

7.1.5 *Hydroxynaphthol Blue Indicator Powder* (CAS No. 63451354)[6]—If preferred, Cal Red may be used as an indicator.[7]

7.2 *Purity of Water*—Unless otherwise indicated, references to water are understood to mean reagent water conforming to Specification D 1193, Type II.

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:

$$M_s = S/(1 + W/100) \times (1 + L/100) \quad (1)$$

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

$$M_f = M_s - M_r$$

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

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

where:

- 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 part of the 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, thoroughly screen each 700-g specimen on the 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 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:

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

9. Test Specimen

9.1 During construction, take representative samples of the soil-lime mixture at the completion of mixing. Test the samples immediately or place them in covered plastic containers and test them within 8 h after completing mixing.

NOTE 2—If the construction samples are obtained more than 8 h after the initial addition of the lime, then the calibration curve (see Section 8) should be obtained from a soil-lime mixture mellowed for a time period commensurate with field conditions.

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

[5] *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*.

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

[7] Available from Pfaltz and Bauer, Waterbury, CT, as Product Code H12970.

retained is free of smaller adhering particles. Weigh and record as M_{flw} the total material passing the sieve. Mix the material passing the sieve and weigh a 300-g specimen and titrate as described in Section 10.

NOTE 3—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 6.

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 add 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 indicator paper or approved pH meter. (Use a stirring rod to transfer drops of solution to the indicator paper). Add (4) drops of triethanolamine solution and then add about 0.2 g of the 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 4—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 5—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 6—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 3), %, 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 interlaboratory 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.[8]

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

[8] These numbers represent, respectively, the (1s) and (d2s) limits as described in Practice C 670.

SUMMARY OF CHANGES

Committee D-18 has identified the location of selected changes to this standard since the last issue (D 3155-96) that may impact the use of this standard.

(*1*) New Section 2, Reference Documents, has been added.
(*2*) Section 3, Significance and Use, has been revised.
(*3*) Section 4.1 added class of balance to be used.
(*4*) Section 5.1, Purity of Reagent Chemicals was added.
(*5*) Section 7.2, Purity of Water, was added.
(*6*) Note 1 was added.
(*7*) Section 10, Precision and Bias, has been rewritten.
(*8*) Section 13, Keywords, has been added.
(*9*) A Summary of Changes Section has been added.
(*10*) The term "moisture content" was changed to "water content" throughout.
(*11*) The term "weight" was changed to "mass" throughout.
(*12*) The term "sample" was changed to "specimen" where indicated by ASTM format guide.
(*13*) The symbol *M* for moisture content was changed to *W* for water content throughout.
(*14*) The symbol *W* for weight was changed to *M* for mass throughout.
(*15*) Metric units were given precedence over English units throughout.
(*16*) Section 1.2 has been added.
(*17*) Section numbers and note numbers have been changed as needed.
(*18*) Parentheses were added to Eq 1 for clarification.
(*19*) In the definition of V_w in Eq 1, the "equals" sign between M_s and M_l was changed to a "minus" sign.
(*20*) Availability of indicator powders has been confirmed and updated.