



Designation: D 4373 – 9602

Standard Test Method for Rapid Determination of Calcium Carbonate Content of Soils¹

This standard is issued under the fixed designation D 4373; 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 This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.13 on Marine Geotechnics. Current edition approved Oct. 10, 1996; 2002. Published June 1997; November 2002. Originally published as D 4373 – 84. Last previous edition ~~D 4373 – 84 (1990)~~ ϵ D 4373 – 96.

1. Scope *

1.1 This test method covers the ~~quantitative~~ determination of the ~~calcium carbonate~~ (CaCO_3) content of soils and soft rock which can be readily broken down by mechanical effort. It is a gasometric method that utilizes a simple portable apparatus. ~~The test method is quickly performed. Results should be clearly stated as the calcite equivalent in percent because different carbonate species cover a wide range of percent calcite equivalent as shown below for soils containing calcium carbonate.~~

NOTE 1—The presence a number of carbonates:

Species	Cation	Calcite Equivalent, %
Magnesite	Mg	117.0
Dolomite	Ca, Mg	108.6
Calcite	Ca	100.0
Aragonite	Ca	100.0
Rhodocrosite	Mn	87.1
Siderite	Fe	86.4
Smithsonite	Zn	79.8
Witherite	Ba	50.7
Cerussite	Pb	37.5

For example, a 100 % dolomite $\text{CaMg}(\text{CO}_3)_2$ would be expected to yield 108.6 % calcite equivalent while 100 % siderite would yield only 86.4 % calcite equivalent. Calcite and reducing minerals such as sulfide and sulfate in the soil aragonite reactions will interfere with the determination of the amount of CaCO_3 present. Therefore, this test typically complete within about 10 min. This method is an approximate method that determines does not distinguish between the calcium carbonate species and such determination must be made using quantitative chemical analysis methods such as atomic absorption.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D 6026.

1.4 The method used to specify how data are collected, calculated, or recorded in this standard is not directly related to the accuracy to which the data can be applied in design or other uses, or both. How one applies the results obtained using this standard is beyond its scope.

1.5 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 precaution statements, see Section 6. 8.

2. Referenced Documents

2.1 ASTM Standards:

C 25 Methods for Chemical Analysis of Limestone, Quicklime and Hydrated Lime²

D 653 Terminology Relating to Soil, Rock, and Contained Fluids³

² Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing Annual Book of reagents not listed by the American Chemical Society, see *Analar ASTM Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, Vol 04.01.

³ Dunn, D. A., "Revised Techniques for Quantitative Calcium Carbonate Analysis Using the "Karbanat-Bombe," and Comparisons to Other Quantitative Carbonate Analysis Methods," *Journal*

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

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 Construction³

D 4753 Specification for Evaluating, Selecting and Specifying Balances and Scales for Use in Soil, Rock and Construction Materials Testing³

D 6026 Practice for Using Significant Digits in Calculating and Reporting Geotechnical Test Data⁴

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens⁵

3. Terminology

3.1 For definitions of terms used in this standard see Terminology D 653.

4. Summary of Test Method

2.1 The calcium

4.1 The carbonate content (calcite equivalent) of soil is determined by treating a 1-g dried soil specimen with hydrochloric acid (HCl) in an enclosed reactor vessel, reaction cylinder (reactor). Carbon dioxide (CO₂) gas is evolved during the reaction between the acid and carbonate fraction of the specimen. The resulting pressure generated in the closed reactor is proportional (see Fig. 1) to the carbonate content calcite equivalent of the specimen. This pressure is measured with a Bourdon tube suitable pressure gauge, or equivalent pressure-measuring device, that is pre-calibrated with reagent grade calcium carbonate.

35. Significance and Use

35.1 This test method is used to determine the presence and quantity of calcium carbonate in a soil specimen in terms of the calcite equivalent. The method is generally intended for use as an index of approximate carbonate content to assist with characterizing marine soils. Other test methods exist (such as Method C 25) to evaluate calcium carbonate equivalency for purposes of characterizing use of calcareous materials as soil modifiers or agricultural lining materials.

5.1.1 Calcium carbonates (CaCO₃) are known cementing agents, are water soluble at pH < 7, and are soft on the Mohs' scale compared to other soil minerals.

5.2 This test method has limitations as follows:

5.2.1 If low carbonate contents (calcite equivalents) are measured, the user does not know whether the soil is low in carbonate content or contains cersusite, witherite, etc., which are carbonate species whose reactions with hydrochloric acid are either very slow or limited.

³ Annual Book of Sedimentary Petrology ASTM Standards, Vol-50, 1980, pp-632-637, 04.08.

⁴ Annual Book of ASTM Standards, Vol 04.09.

⁵ Annual Book of ASTM Standards, Vol 14.04.

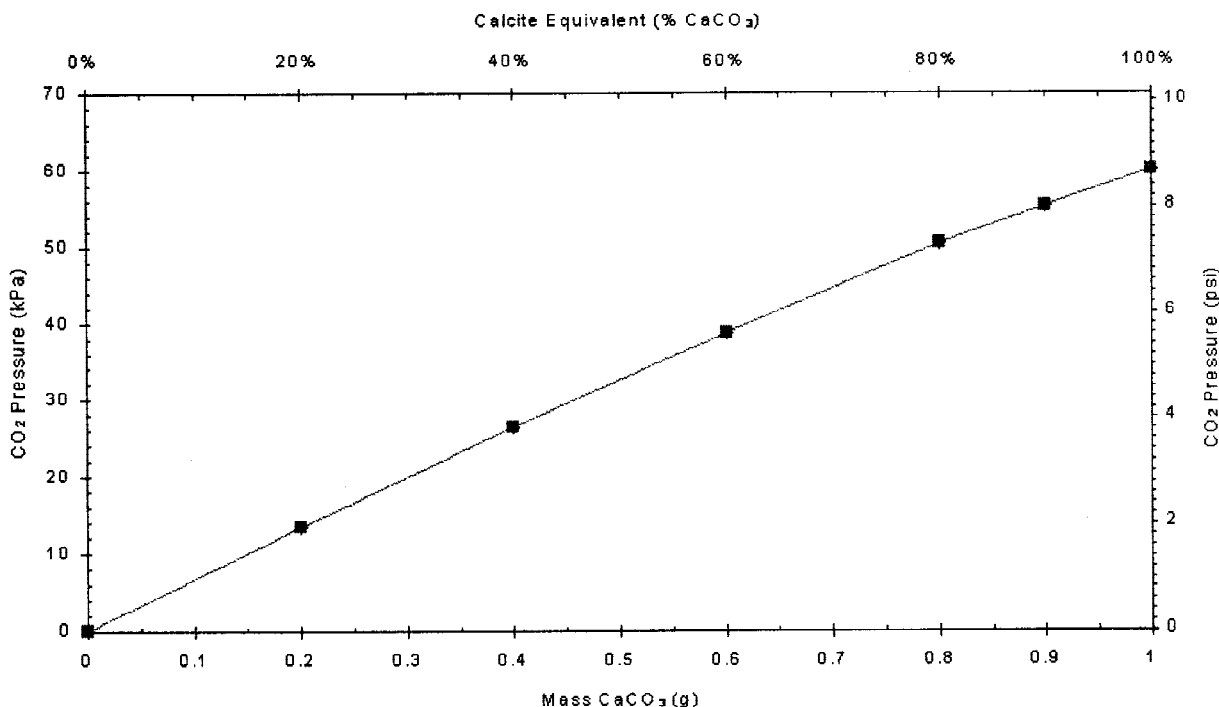


FIG. 1 Typical Calibration Curve for 0.374L Test Cell and 70 kPa (10 psi) Pressure Gauge

5.2.2 Testing times may be extensive (longer than 1 h) for some carbonate species (such as dolomite) if calcite equivalents within about 1 % are required.

5.2.3 The effects of specimen grain size, duration of testing, pH and specimen mass are discussed in the literature.⁶

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 D 3740 are generally considered capable of competent and objective testing/sampling/inspection, etc. Users of this standard are cautioned that compliance with Practice D 3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D 3740 provides a means of evaluating some of those factors.

6. Apparatus

46.1 *Rapid Carbonate Analyzer*—A schematic drawing of the rapid carbonate analyzer is shown in Fig. 2. The basic components of this apparatus include:

46.1.1 *Reaction Cylinder (Reactor)*, with threaded cap and O-ring seal to enclose the cylinder. A clear plastic cylinder allows viewing of effervescent reaction.

46.1.2 *Pressure Gauge (Bourdon Tube-type or Electronic Pressure Transducer)*, $\pm 70 \pm 0$ kPa (10 psi (7)), with an accuracy of 0 ± 0.25 %, and a readability of 0.5 kPa (0.1 psi).

46.1.3 *Acid Container*, of clear plastic with a bail handle to hold 20 mL of acid. Optionally, in lieu of the acid container, use a soil container of clear plastic to hold 1 g of soil, with approximate dimensions of 25 mm diameter by 6 mm high with a nominal 4 mm notch down the side of the soil container. Acid added to dry soil often causes considerable splashing and use of soil container reduces splashing before reactor is sealed.

6.1.4 *Pressure Relief Valve*, for safe release of CO₂ gas pressure.

46.2 *Balance*—The balance must meet the requirements of Specification D 4753 and this section. A Class GP1 balance with a sensitivity minimum capacity of 100 g and a readability of 0.01 g is required to measure the soil mass.

6.3 *Drying Oven*, thermostatically-controlled, preferably of the forced-draft type, meeting the requirements of Specification E 145 and capable of maintaining a uniform temperature of $110 \pm 5^\circ\text{C}$ throughout the drying chamber.

⁶ Demars, K.R., Chaney, R.C., Richter, J.A., "The Rapid Carbonate Analyzer," *Geotechnical Testing Journal*, ASTM, Vol. 6, No. 1, March 1981, pp. 30–34.

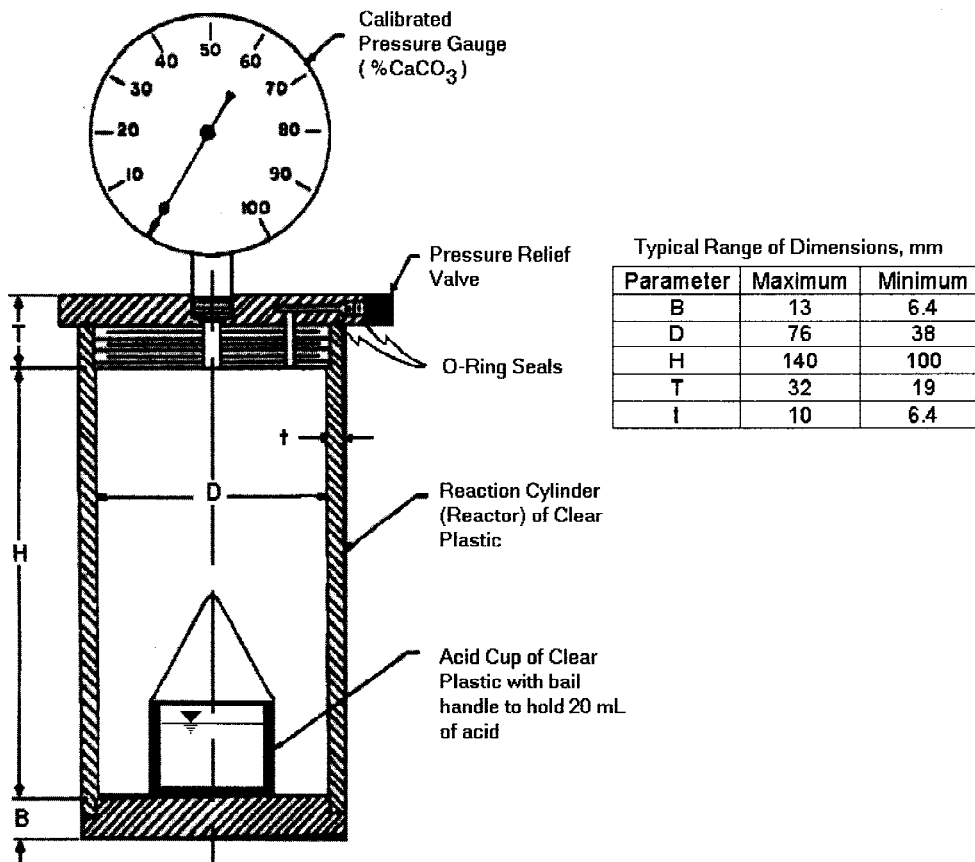


FIG. 2 Schematic Drawing of Rapid Carbonate Analyzer

7. Reagents

57.1 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.⁷ 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.

57.2 Calcium Carbonate (CaCO₃).

57.3 Hydrochloric Acid (HCl), (in about 1 N solution)—Prepare 1 L of about 1 N solution by placing 80 mL of concentrated, reagent grade HCl in about 800 mL of commercial grade distilled water in a 1L volumetric flask and dilute to the mark with commercial grade distilled or demineralized water. Store in polyethylene bottle. Faster reaction times may achieved by increasing the concentration to higher normal solutions (up to about 3 N solution). Hydrochloric acid is also commercially available in a 1 N solution.

68. Safety Precautions

68.1 Use care in handling the hydrochloric acid so that no acid is spilled on either skin or clothing. If acid contacts the skin or eyes, immediately flush with large quantities of water. Process concentrated hydrochloric acid beneath a laboratory hood or in a well-ventilated area to reduce the inhalation of fumes.

68.2 The pressure relief valve of the carbonate analyzer should be opened following each test to dissipate gas pressure so that the cap can be safely removed.

79. Test Specimens

7.1 Select 20 or 30-g

9.1 Select 5 to 10-g specimens from a core or surface grab sample. Oven dry at $110 \pm 5^\circ\text{C}$ for a period of 12 to 24 h. Pulverize the entire sample with a mortar and pestle (or hammer) until all of the particles pass a No. 40 (0.425-mm) or finer sieve. Smaller particles react faster than larger particles when treated with acid.

810. Calibration

810.1 Calibration is accomplished by using reagent grade CaCO₃ to obtain the relationship between the weight mass of CaCO₃ and the pressure generated in the constant volume reactor. Each carbonate analyzer and pressure gage is individually calibrated. Prepare five sets of duplicate specimens with the following weights masses of CaCO₃:

810.1.1 Set 1—Two specimens at 0.2 ± 0.01 g, that is, analogous to 20 % calcium carbonate for a test specimen of 1 g.

810.1.2 Set 2—Two specimens at 0.4 ± 0.01 g.

810.1.3 Set 3—Two specimens at 0.6 ± 0.01 g.

810.1.4 Set 4—Two specimens at 0.8 ± 0.01 g.

810.1.5 Set 5—Two specimens at 1.0 ± 0.01 g.

8.2 Place a calibration

10.2 Place the specimen into the reactor and lower the filled 20-mL acid (using soil container if available). Add 20 ± 2 mL of acid solution into the reactor, being careful not to spill any acid. reactor (using acid container if available). Seal the reactor with top cap and close the pressure release thumbscrew.

8.3 Tilt relief valve. Tilt the reactor to initiate reaction between the acid and specimen. Mix the contents by swirling. If available, place the reactor on a swirling action and record mechanical shaker for the pressure reading when duration of the reaction test.

10.3 It is complete, usually 2 very rare that even calcite powder will completely react in less than 10 minutes. Therefore, it is essential to 3 min. Monitor monitor the pressure to make certain verify that the reaction cylinder is complete (pressure stabilized) and to confirm that the reactor is properly sealed.

8.4 Open sealed (pressure does not decrease). Record the pressure reading to the nearest 0.5 kPa (0.1 psi) or better.

10.4 Open the pressure release valve and remove the reactor top cap. Dispose of the contents and rinse the reactor with distilled or demineralized water.

8.5 Repeat 8.2 and 8.4

10.5 Repeat 10.2 through 10.4 for remaining calibration specimens.

10.6 Data reduction may use any one or all of the following:

10.6.1 Make a graph (Fig. 1) of CaCO₃ weight percent calcite equivalent (using mass of calcite added) versus reactor pressure. This calibration pressure in appropriate units for the gauge used.

10.6.2 Calibration data may be transferred to the face of the pressure gauge for a direct reading of percent CaCO₃ calcite equivalent as shown in Fig. 2.

⁷ *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

~~9. 1.~~

~~10.6.3 Use a least squares regression fit of the calibration data to arrive at an equation for calculating the percent calcite equivalent from the pressure data.~~

11. Procedure

~~9.1 Weigh~~

~~11.1 Select a 1 ± 0.01 -g specimen from the pulverized soil. Follow the procedures described in ~~8.2-8.4~~ 10.2-10.4 to obtain a direct measurement of ~~carbonate content~~. the calcite equivalent. If there is no pressure response, but a minor effervescent reaction is observed, repeat the experiment with 2, 5, or 10 g of dry solids in the reactor and divide this carbonate reading by 2, 5, or 10, respectively to obtain the true calcite equivalent.~~

~~11.2 After a 10 min reaction time, read the pressure value to obtain the carbonate content.~~

~~NOTE 2—If dolomite is present content. If one wishes to evaluate whether other carbonate species are present, take additional readings as shown in 11.3~~

~~11.3 Continue reading pressure until the reaction is complete using the following criteria: (a) The change in calcite equivalent is less than 0.3 % over a ten-min time period for ~~30~~ testing time up to ~~40 min or more~~; 120 min; and (b) The ~~dolomite content may be estimated by taking the difference change~~ in readings at 30 s and 35 calcite equivalent is less than 0.3 % over a 30-min time period for testing time greater than 120 min.~~

102. Report

~~102.1 Report all results to the nearest 1 %.~~

~~102.2 List sample source, project name and location (if applicable).~~

113. Precision and Bias

~~113.1 Precision—No interlaboratory testing program has as yet been conducted using—Due to the nature of the soil and rock materials tested by this test method it is either not feasible or too costly at this time to determine multilaboratory precision. A round robin produce multiple specimens that have uniform physical properties. Any variation observed in the data is just as likely to determine this information will probably not take place because be due to specimen variation as to operator or laboratory testing variation. Subcommittee D18.13 welcomes proposals that would allow for development of the expense and economics of industry.~~

~~11.2— a valid precision statement.~~

~~13.2 Bias—The bias of the procedure in—There is no accepted reference value for this test method has as not yet been conducted. A round robin to determine this information will probably not take place because of the expense and economics of industry.~~

~~11.3—Some method, therefore, bias cannot be determined.~~

~~13.3 Some information on performance statistics of this test is provided in the literature.~~

~~12. 8~~

14. Keywords

~~124.1 calcareous soils; calcite equivalent; carbonate content; Karbanat Bombe; marine soils~~

⁸ Dunn, D. A., "Revised Techniques for Quantitative Calcium Carbonate Analysis Using the "Karbanat-Bombe," and Comparisons to Other Quantitative Carbonate Analysis Methods," *Journal of Sedimentary Petrology*, Vol 50, 1980, pp. 632-637.

SUMMARY OF CHANGES

In accordance with ASTM Committee D18 policy, this section identifies the location of changes to this standard since the last edition (1996) that may impact the use of this standard.

- (1) Significant revisions to the standard were incorporated throughout most of the standard, including a name change (removal of word “calcium”).
- (2) Scope—Rewritten to explain that measurement of the carbonate content is actually the calcite equivalent and that the method also applies to soft rock. Added statements on use of Practice D 6026.
- (3) Addition of Referenced Documents and Terminology sections.
- (4) Summary of Test Method—References to calcium carbonate revised to calcite equivalents and revisions for consistency in nomenclature for the reactor vessel.
- (5) Significance and Use—Added discussion on limitations and reference to D 3740.
- (6) Apparatus—Added oven specification, revised pressure gage and balance specifications, added optional soil container inside reactor.
- (7) Reagents—Eliminated use of demineralized water.
- (8) Test Specimens—Reduced amount of material to be prepared for the test. Specified finer sieve size for maximum particle size to speed up reaction times.
- (9) Calibration—Added tolerances for masses of reagent and acid to be used.
- (10) Procedure—Added options to increase specimen mass to obtain higher resolution for low carbonate content materials.
- (11) Precision and Bias—Revised to conform with D18 policy.
- (12) Keywords—Added calcite equivalent.
- (13) Summary of Changes—Added.

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