Designation: D4373 − 14

Standard Test Method for Rapid Determination of Carbonate Content of Soils[1]

This standard is issued under the fixed designation D4373; 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 carbonate content of soils and soft rock which can be readily broken down by mechanical effort. It is a gasometric method that uses a simple portable apparatus. 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 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
Cerrusite	Pb	37.5

For example, a 100 % dolomite would be expected to yield 108.6 % calcite equivalent while 100 % siderite would yield only 86.4 % calcite equivalent. Calcite and aragonite reactions will typically complete within about 10 minutes. This method does not distinguish between the carbonate species and such determination must be made using quantitative chemical analysis methods such as atomic absorption.

1.2 *Units*—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 D6026.

1.3.1 The procedures used to specify how data are collected/recorded or calculated, in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should 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; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analytical methods for engineering design.

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.* For specific precaution statements, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D3042 Test Method for Insoluble Residue in Carbonate Aggregates

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

D6026 Practice for Using Significant Digits in Geotechnical Data

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Terminology

3.1 *Definitions*—For definitions of common technical terms used in this standard, refer to Terminology D653.

4. Summary of Test Method

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 reaction cylinder (reactor).

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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 D18.06 on Physical-Chemical Interactions of Soil and Rock.

Current edition approved May 1, 2014. Published June 2014. Originally approved in 1984. Last previous edition approved in 2007 as D4373 – 02 (2007). DOI: 10.1520/D4373-14.

[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.

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

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Carbon dioxide (CO_2) 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 calcite equivalent of the specimen. This pressure is measured with a suitable pressure gauge, or equivalent pressure-measuring device, that is pre-calibrated with reagent grade calcium carbonate.

5. Significance and Use

5.1 This test method is used to determine the presence and quantity of 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 C25 and Test Method D3042) 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 ($CaCO_3$) 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 cerrusite, witherite, and the like, which are carbonate species whose reactions with hydrochloric acid are either very slow or limited.

5.2.2 Testing times may be extensive (longer than 1 hour) 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.[3]

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 assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

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

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

6.1.2 *Pressure Gauge (Bourdon Tube-type or Electronic Pressure Transducer),* 70 kPa (10 psi), with an accuracy of 0.25 %, and a readability of 0.5 kPa (0.1 psi).

6.1.3 *Acid or Soil Container, or Both,* of clear plastic with a bail handle to hold 20 mL of acid. Optionally, in addition to, or in place of the acid container, use a soil container of clear plastic to hold the soil specimen. Acid added to dry soil often causes considerable splashing and use of soil container reduces splashing before reactor is sealed.

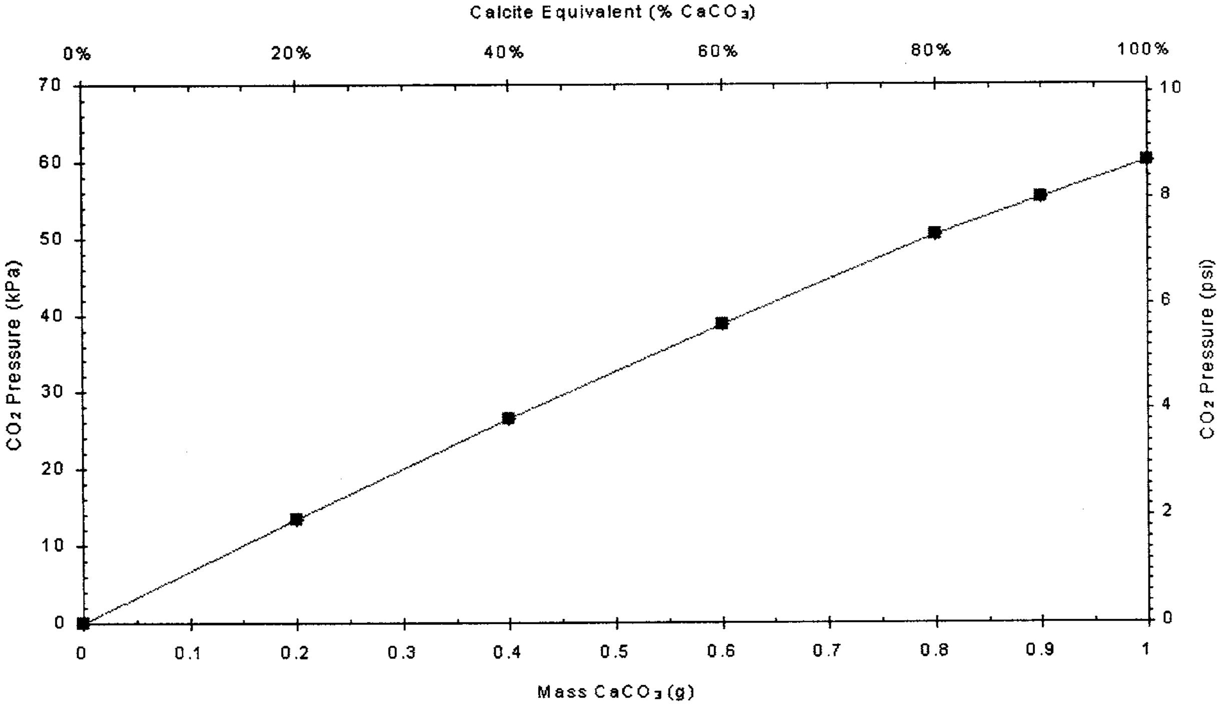

FIG. 1 Typical Calibration Curve for 0.374L Test Cell and 70 kPa (10 psi) Pressure Gauge

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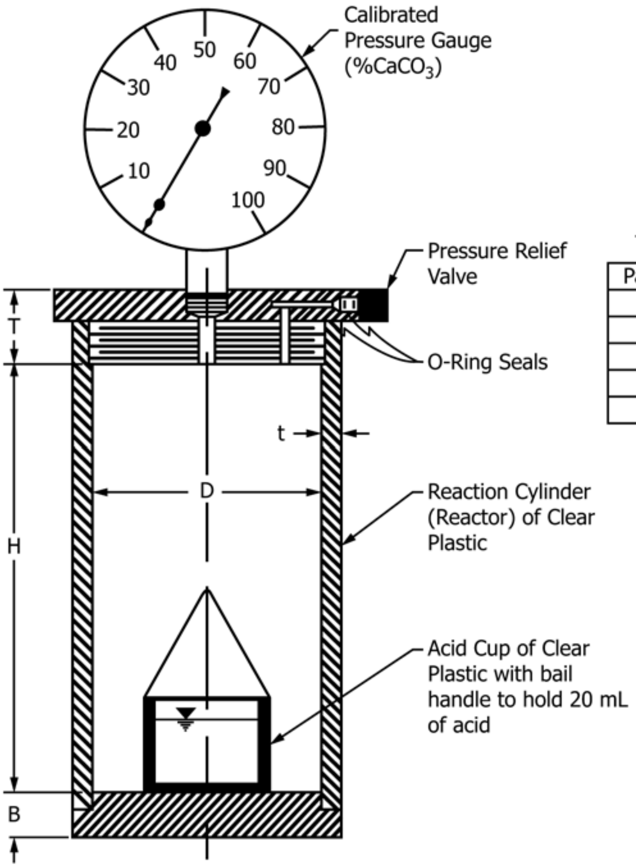
[3] Demars, K.R., Chaney, R.C., Richter, J.A., "The Rapid Carbonate Analyzer," *Geotechnical Testing Journal*, ASTM, Vol. 6, No. 1, March 1981, pp. 30–34.



Typical Range of Dimensions, mm

Parameter	Maximum	Minimum
B	13	6.4
D	76	38
H	140	100
T	32	19
t	10	6.4

FIG. 2 Schematic Drawing of Rapid Carbonate Analyzer

6.1.4 *Pressure Relief Valve,* for safe release of CO_2 gas pressure.

6.2 *Balance*—The balance must meet the requirements of Specification D4753 and this section. A Class GP1 balance with a 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 E145 and capable of maintaining a uniform temperature of 110 ± 5°C throughout the drying chamber.

6.4 *Sieve*—No. 40 (425 µm) or finer sieve that conforms to the requirements of Specification E11.

6.5 *Mortar and Rubber-Covered Pestle (Optional)*—Apparatus suitable for breaking up aggregations of air-dried soil particles without breaking individual particles.

6.6 *Mechanical Agitation Device (Optional)*—A platform, wrist action or similar type shaker having a gyratory, orbital, reciprocating, or similar motion to assist in the reaction process by continuously agitating the soaking soil.

6.7 *Graduated Cylinder, Flask, Pipet, or the Like*——A glass or equivalent inert device with enough capacity to hold 20 ± 2 mL of hydrochloric acid and readable to the nearest 1 mL.

6.8 *Miscellaneous Items*—Items such as a spatula, scoop, or hammer may be useful.

7. Reagents

7.1 *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.[4]

7.2 *Calcium Carbonate* ($CaCO_3$). Reagent grade (98-100 %) for calibration of reaction cylinder.

7.3 *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 distilled water (pH = 7) in a 1-L volumetric flask. Dilute to the mark with commercial grade distilled 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.

7.4 *Water*—Distilled or demineralized water is the only permissible test fluid.

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[4] *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 *Annual 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.

8. Hazards

8.1 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.

8.2 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.

9. Test Specimens

9.1 Select 5 to 10-g specimens from a core or surface grab sample. Oven dry at 110 ± 5°C for a period of 12 to 24 hours. 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.

10. Calibration

10.1 Calibration is accomplished by using reagent grade $CaCO_3$ to obtain the relationship between the mass of $CaCO_3$ 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 masses of $CaCO_3$:

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

10.1.2 *Set 2*—Two specimens at 0.4 ± 0.01 g.

10.1.3 *Set 3*—Two specimens at 0.6 ± 0.01 g.

10.1.4 *Set 4*—Two specimens at 0.8 ± 0.01 g.

10.1.5 *Set 5*—Two specimens at 1.0 ± 0.01 g.

10.2 Place the specimen into the reactor (using soil container if available). Add 20 ± 2 mL of acid solution into the reactor (using acid container if available). Seal the reactor with top cap and close the pressure relief valve. Tilt the reactor to initiate reaction between the acid and specimen. Mix the contents by swirling or gentle shaking. If available, place the reactor on a mechanical agitation device for the duration of the test.

10.3 It is very rare that even calcite powder will completely react in less than 10 minutes. Therefore, it is essential to monitor the pressure to verify that the reaction is complete (pressure stabilized) and to confirm that the reactor is properly 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 water.

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 of percent calcite equivalent (using mass of calcite added) versus pressure in appropriate units for the gauge used.

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

11.1 Select a 1 ± 0.01-g specimen from the pulverized soil. Follow the procedures described in 10.2 – 10.4 to obtain a direct measurement of the calcite equivalent. If there is no pressure response, but a minor effervescent reaction is observed, repeat the experiment with 2 ± 0.01 g, 5 ± 0.01 g, or 10 ± 0.01 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-minute reaction time, read the pressure value to obtain the carbonate 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 10-minute time period for testing time up to 120 minutes; or (*b*) The change in calcite equivalent is less than 0.3 % over a 30-minute time period for testing time greater than 120 minutes.

11.4 Take the pressure reading obtained above and determine the corresponding percent calcite equivalent from the data reduction method chosen in 10.6 to obtain the Carbonate Content (percent calcite equivalent).

12. Report: Test Data Sheet(s)/Form(s)

12.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as described below, is covered in 1.3.

12.2 Record as a minimum the following general information (data):

12.2.1 Record the Carbonate Content (percent calcite equivalent) to the nearest 1 %.

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

12.2.3 Record the mass of the test specimen to the nearest 0.01 grams.

12.2.4 Record the technician/operator name or initials and date of test.

13. Precision and Bias

13.1 *Precision*—Test data on precision is not presented due to the nature of the soil and rock materials tested by this method. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program. Also, it is either not feasible or too costly to produce multiple specimens that have uniform physical properties. Any variation observed in the data is just as likely to be due to specimen variation as to operator or laboratory testing variation.

13.1.1 Subcommittee D18.13 is seeking any data from the users of this test method that might be used to make a limited statement on precision.

13.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

13.3 Some information on performance statistics of this test is provided in the literature.[5]

14. Keywords

14.1 calcareous soils; calcite equivalent; carbonate content; Karbanat Bombe; marine soils

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (2002 (Reapproved 2007)) that may impact the use of this standard.

(1) Minor editorial changes in 1.1, 1.3.1, 10.2, and Section 11.
(2) Added reference to Test Method D3042 and Specification E11 in Section 2.
(3) Removed dimension requirements of soil cup, add sieve, and various miscellaneous test apparatus items in Section 6.
(4) Added clarifications on reagent grades for $CaCO_3$ and distilled water in Section 7.
(5) Renamed Section 8 to Hazards.
(6) Clarification on determination of carbonate content in Section 11.
(7) Revision of Section 12 for clarity of Report requirements.
(8) Revised 13.1 on Precision.

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[5] 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.