



Designation: D4373 – 21

Standard Test Method for Rapid Determination of Carbonate Content of Soils¹

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 standard. The values given in parentheses 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 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

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.

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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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

3. Terminology

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

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

4. Summary of Test Method

4.1 A representative sample is oven dried and then pulverized to pass a 0.425 mm (No. 40) or finer sieve. A 1 g specimen is obtained from the representative sample and placed inside the reaction cylinder (reactor). Hydrochloric acid is added to the reactor using a container and the reactor sealed. The reactor is tilted and then shaken to allow the acid to react with the specimen. After the pressure reading has stabilized, the pressure reading is taken. The resulting pressure generated in the closed reactor is proportional to the calcite equivalent of the specimen.

5. Significance and Use

5.1 This test method is used to determine the presence and quantity of carbonate in a soil or soft rock 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, but can be used for other soils and soft rock. 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 $\text{pH} < 7$, 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. 1. The analyzer must be calibrated prior to initial use, after repair, and once every 12 months (Section 10). The basic components of this apparatus include:

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.

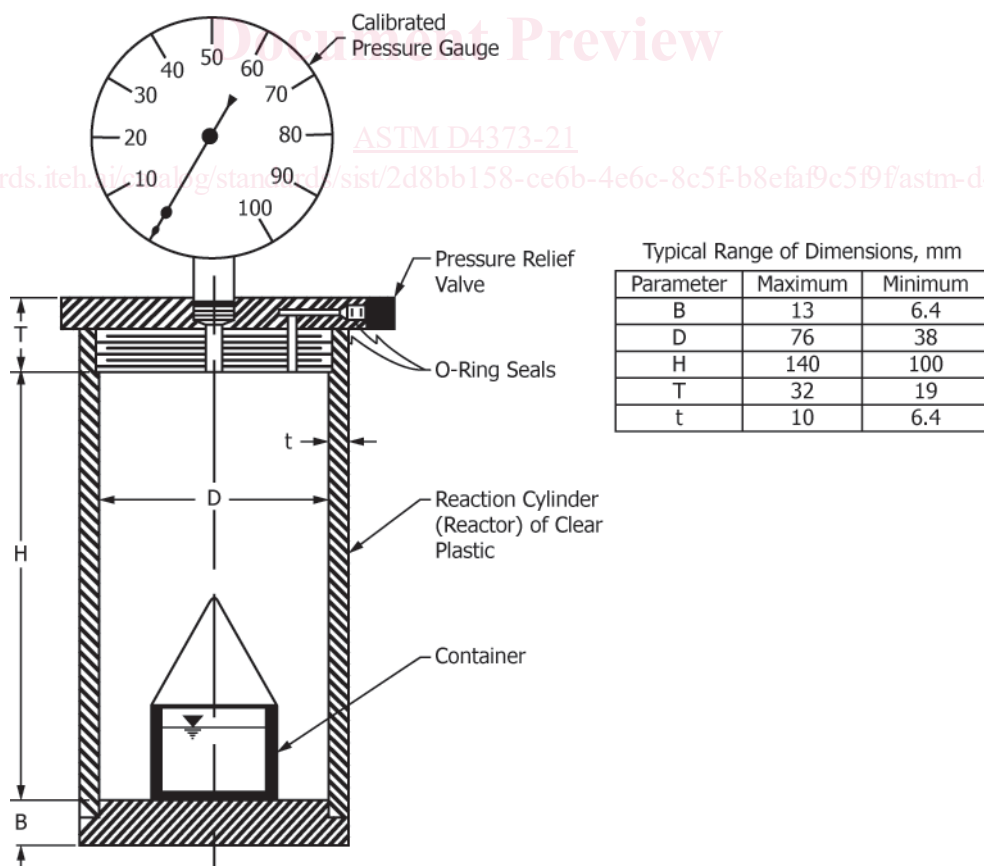


FIG. 1 Schematic Drawing of Rapid Carbonate Analyzer

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

6.1.2 *Pressure Gauge*—A Bourdon Tube-type or Electronic Pressure Transducer with a capacity of 70 kPa (10 psi), an accuracy of 1.5 %, and a readability of 0.5 kPa (0.1 psi).

6.1.3 *Container*—A clear plastic container 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/soft rock specimen. 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*—Used for safe release of CO₂ gas pressure.

6.2 *Balance*—The balance shall conform to the requirements of Guide **D4753**. A Class GP1 balance with a minimum capacity of 100 g and a readability of 0.01 g is needed to measure the soil/soft rock mass.

6.3 *Drying Oven*—Vented, thermostatically controlled oven capable of maintaining a uniform temperature of 110 ± 5°C (230 ± 9°F) throughout the drying chamber (preferably of the forced-draft type).

6.4 *Sieve*—425 μm (No. 40) 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 (CaCO₃)*. 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 be 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.

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. Sampling and Test Specimens

9.1 Select a 5 to 10 g representative sample from a core or surface grab sample. Oven dry at 110 ± 5°C (230 ± 9°F) 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 0.425-mm (No. 40) or finer sieve. Smaller particles react faster than larger particles when treated with acid. Proceed to Section **11** for testing.

10. Calibration

10.1 Calibration is accomplished by using reagent grade CaCO₃ to obtain the relationship between the 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 reagent grade calcium carbonate specimens with the following masses of CaCO₃:

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 important 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.

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.