



Standard Test Method for Calcium Carbonate Content of Soils¹

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1. Scope

1.1 This test method covers the quantitative determination of the calcium carbonate (CaCO_3) content of soils. It is a gasometric method that utilizes a simple portable apparatus. The test method is quickly performed for soils containing calcium carbonate.

NOTE 1—The presence of dolomite $\text{CaMg}(\text{CO}_3)_2$ and reducing minerals such as sulfide and sulfate in the soil will interfere with the determination of the amount of CaCO_3 present. Therefore, this test method is an approximate method that determines the calcium carbonate equivalent.

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

1.3 *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.

2. Summary of Test Method

2.1 The calcium carbonate content of soil is determined by treating a 1-g dried soil specimen with hydrochloric acid (HCl) in an enclosed reactor vessel. Carbon dioxide 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 of the specimen. This pressure is measured with a bourdon tube pressure gage, or equivalent pressure-measuring device, that is precalibrated with reagent grade calcium carbonate.

3. Significance and Use

3.1 This test method is used to determine the presence and quantity of calcium carbonate in a soil specimen. Calcium carbonates are known cementing agents, are water soluble at $\text{pH} < 7$, and are soft on the Mohs' scale compared to other soil minerals.

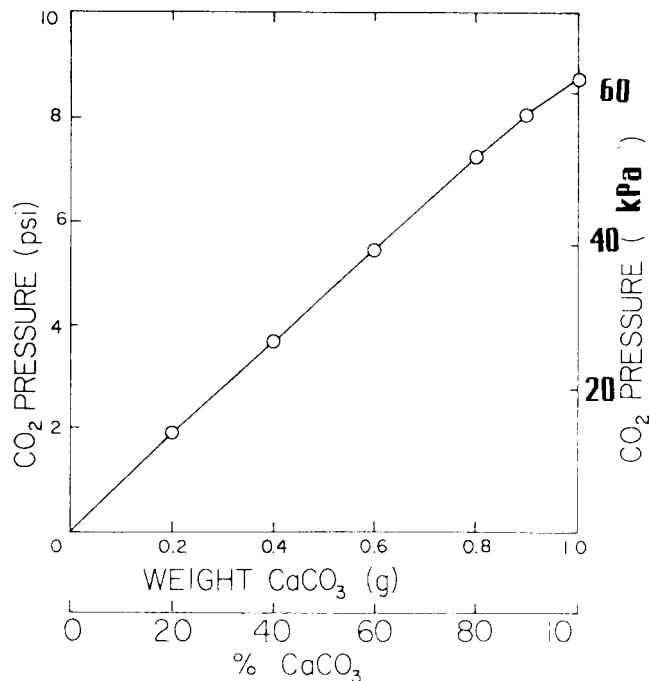


FIG. 1 Typical Calibration Curve for 0.374L Test Cell and 10 psi (69 kPa) Pressure Gage

4. Apparatus

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

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

4.1.2 *Pressure Gage*, 10 ± 0.1 psi ($70 \pm$ kPa).

4.1.3 *Container*, of clear plastic with a bail handle to hold 20 mL of acid.

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

4.2 *Balance*, with a sensitivity of 0.01 g.

5. Reagents

5.1 *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,

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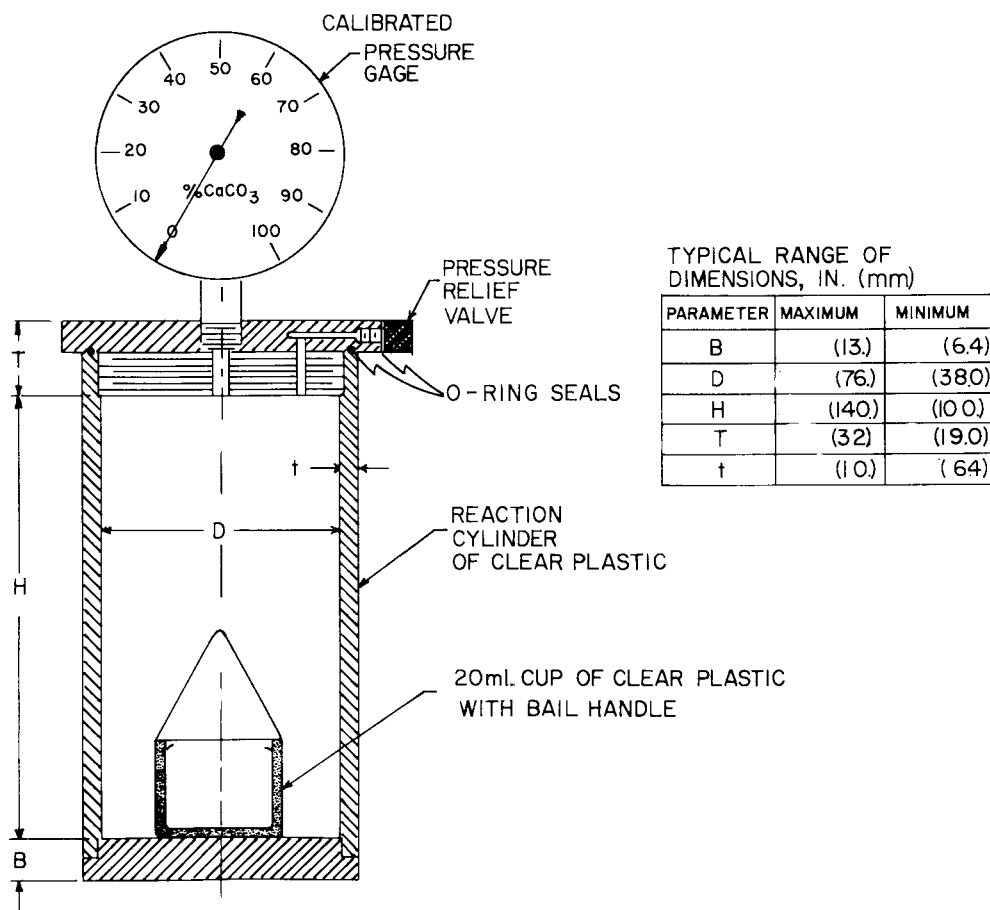


FIG. 2 Schematic Drawing of Rapid Carbonate Analyzer

where such specifications are available.² 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.

5.2 Calcium Carbonate (CaCO₃).

5.3 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 a 1L volumetric flask and dilute to the mark with distilled or demineralized water. Store in polyethylene bottle.

6. Safety Precautions

6.1 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.

6.2 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.

² 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.

7. Test Specimens

7.1 Select 20 or 30-g specimens from a core or surface grab sample. Oven dry at 105°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. 10 (2-mm) sieve. Smaller particles react faster than larger particles when treated with acid.

8. Calibration

8.1 Calibration is accomplished by using reagent grade CaCO₃ to obtain the relationship between the weight 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 of CaCO₃:

- 8.1.1 Set 1—Two specimens at 0.2 g, that is, analogous to 20 % calcium carbonate for a test specimen of 1 g.
- 8.1.2 Set 2—Two specimens at 0.4 g.
- 8.1.3 Set 3—Two specimens at 0.6 g.
- 8.1.4 Set 4—Two specimens at 0.8 g.
- 8.1.5 Set 5—Two specimens at 1.0 g.

8.2 Place a calibration specimen in the reactor and lower the filled 20-mL acid container into the reactor, being careful not to spill any acid. Seal reactor with top cap and close pressure release thumbscrew.

8.3 Tilt reactor to initiate reaction between acid and specimen. Mix contents by a swirling action and record the pressure reading when the reaction is complete, usually 2 to 3 min.

Monitor pressure to make certain that the reaction cylinder is properly sealed.

8.4 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 for remaining specimens and prepare a graph (Fig. 1) of CaCO_3 weight versus reactor pressure. This calibration data may be transferred to the face of the pressure gage for a direct reading of percent CaCO_3 , as shown in Fig. 2.

9. Procedure

9.1 Weigh a 1 ± 0.01 -g specimen from the pulverized soil. Follow the procedures described in 8.2-8.4 to obtain a direct measurement of carbonate content. If there is no pressure response, but a minor effervescent reaction is observed, repeat the experiment with 2 g of dry solids in the reactor and divide this carbonate reading by 2 to obtain the true carbonate content.

NOTE 2—If dolomite is present the reaction will continue for 30 to 40 min or more. The dolomite content may be estimated by taking the difference in readings at 30 s and 35 min.

10. Report

10.1 Report all results to the nearest 1 %.

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

11. Precision and Bias

11.1 *Precision*—No interlaboratory testing program has as yet been conducted using this test method to determine multilaboratory precision. A round robin to determine this information will probably not take place because of the expense and economics of industry.

11.2 *Bias*—The bias of the procedure in 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 information on performance statistics of this test is provided in the literature.³

12. Keywords

12.1 calcareous soils; carbonate content; marine soils

³ 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.

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