OFITE®

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Calcimeter

Part No. 152-95

Instruction Manual

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Ver. 5

OFI Testing Equipment, Inc.

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Intro

The OFITE Calcimeter is used to determine the amount of Calcium Carbonate ($CaCO_3$) and Magnesium Carbonate (Dolomite) in a sample of alkaline earth carbonates such as oil well cores or drilled cuttings. Calcite build up in drilling fluids and in water treatment processes causes scaling problems. Data from the OFITE Calcimeter can help determine the proper chemical treatment.

This instrument complies with the ASTM D 4373 - 84 (Reapproved 1990) Standard Test Method for Calcium Carbonate Content in Soils. This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D -18.13 on Marine Geotechnics, published July 1984.

Description

In the OFITE Calcimeter, calcium carbonate and magnesium carbonate are reacted with 10 percent hydrochloric acid in a sealed reaction cell to form CO_2. As the CO_2 is released, the pressure build up is measured using either a pressure gauge or a pressure recorder. During the calibration process, a calibration curve is created by reacting HCl with pure, reagent-grade $CaCO_3$. By using a known weight of $CaCO_3$ reagent, you can determine the relationship between the amount of pressure released and the weight of $CaCO_3$ in the sample. Since all reaction cells are slightly different, this relationship will be different for each cell. Therefore a calibration curve is required to obtain accurate results.

The calcium carbonate content of soil (ASTM Procedure D 4373) is determined by treating a 1 g dried soil specimen with HCl in the reactor cell. The resulting pressure increase is then measured and compared to the calibration curve to determine the total weight of $CaCO_3$ in the test sample.

Components

#142-54	O-ring for Bleed-Off Screw
#152-95-2	Bleed-Off Screw
#152-95-3	Cell Cap
#152-95-4	Reaction Cell
#152-95-5	O-ring for Cap
#152-95-6	Sample Cup
#152-95-8	Gauge, Digital, 0 - 30 psi

Optional:

#152-96-6	Mortar, 65 mL, Porcelain
#152-96-7	Pestle, Porcelain
#153-02	Brush, Graduate, 1.5" × 10.75"
#153-18	Graduated Cylinder, 10 mL × .2 mL, Glass
#153-55	Stopcock Grease, Silicone
#166-03	Hand-held Balance, 0 - 320 g × .1 g
#275-03	Hydrochloric Acid, 10%, 8 oz UN 1789
#285-00-1	Calcium Carbonate, 100 g

Safety

The Calcimeter test relies on the reaction of $CaCO_3$ with hydrochloric acid. Hydrochloric acid is corrosive and may cause chemical burns. Use care in handling the acid. Avoid contact with skin, eyes, and clothing. In the event of exposure to skin or eyes, immediately flush with large quantities of water for at least 15 minutes. Do not inhale vapors. Process hydrochloric acid beneath a laboratory hood or in a well-ventilated area to reduce the risk of inhalation. Wear appropriate safety equipment at all times.

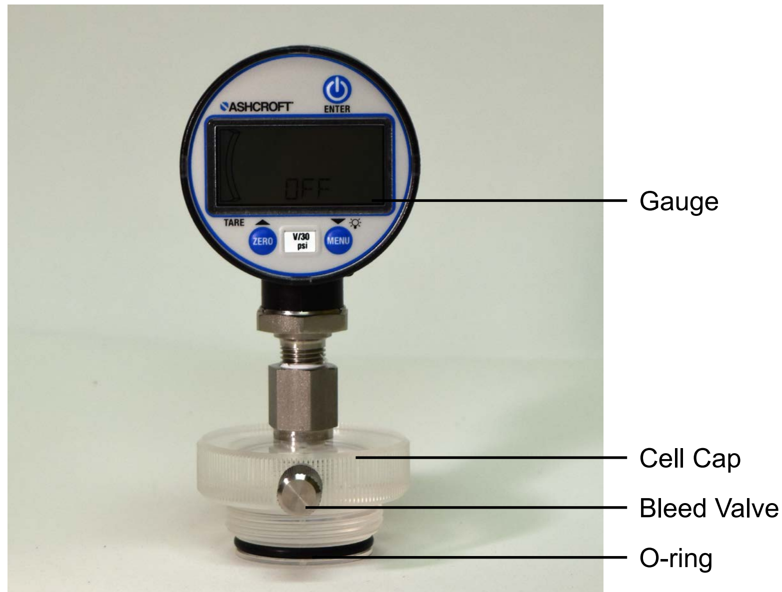
Do not take internally. In the event of accidental exposure, get medical attention immediately.

Important

Refer to the Material Safety Data Sheet (MSDS) for more information on Hydrochloric Acid

Setup

Begin by carefully unpacking the equipment. Inspect each piece to ensure everything is clean and in good operating condition. Inspect the cell cap o-ring for cracks or wear.



Test Procedure

Before starting the test procedure, make sure the equipment is clean and in good operating condition. Verify that a calibration curve is available for the particular equipment to be used. If a calibration curve is not available, see the “Creating a Calibration Curve” section on page 8 to construct one.

1. Obtain a sample of core, drilled cuttings, or other solids that are to be analyzed. The sample should be dry and free of contaminants. Grind the sample to 100 mesh or finer, using a mortar and pestle and a 100-mesh sieve. If you do not know whether the sample has been dried, heat it in an oven at 220°F (105°C) for 12 to 24 hours.

2. Weigh approximately 1.0 - 1.4 g of the sample to the nearest .001 g.

3. Load the test sample in the reactor cell.

    a. Unscrew and remove the cell cap. Remove the acid cup from the reaction cell.

    b. Inspect the reaction cell and top. Make sure both are clean and dry.

    c. Make sure the reaction cell o-ring on the top and the o-ring on the bleed valve are in good condition. Use a light coating of vacuum grease on the o-ring seals. Make sure all pipes or tubing connections are tight and do not leak.

    d. Hold the reaction cell in a horizontal position and slide one piece of paper and its sample to the bottom.

    e. Return the cell to the vertical position and brush the paper with a small brush to remove traces of the sample, then remove the paper.

    f. Fill the acid cup with 20 mL 10% hydrochloric acid. Carefully place the cup into the cell. Be careful not to spill the HCl or get any on the bottom of the cup.

    g. Hand tighten the cell cap. Be careful not to splash any acid onto the sample.

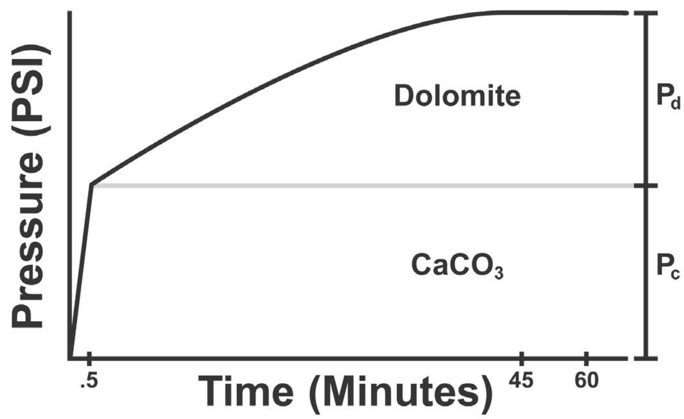
    h. Open the bleed valve until the pressure reading is zero. Then close the bleed valve tightly.

    i. Turn the cell back to the horizontal position to release acid from the cup.

4. Turn the reaction cell back to vertical and start timing the test. This will start the reaction between the HCl and the $CaCO_3$.

5. At 30 seconds, record the pressure as “$CaCO_3$ Pressure”. If the test sample contains any dolomite, there will be a pause, then a slow, second rise in pressure. Swirl the reaction cell and allow sufficient time for the reaction to finish. The reaction is complete when the pressure stops increasing. This should happen in 30 to 45 minutes. The final pressure value is the total $CaCO_3$ pressure plus the dolomite pressure. To calculate the dolomite pressure, subtract the $CaCO_3$ pressure (30 second reading) from the total pressure (30 - 45 minute reading).

6. Refer to the graph below to interpret the pressure readings.



7. Use the equations below to calculate the percentages of $CaCO_3$ and dolomite. For values of “Slope” refer to the “Creating a Calibration Curve” section below.

$$\%CaCO_3 = \frac{\text{(Pressure Reading, PSI) (100)}}{\text{(Sample Weight) (Average Slope)}}$$

$$\%\text{Dolomite} = \frac{\text{(Total Pressure - } CaCO_3 \text{ Pressure) (100) (.92)}}{\text{(Sample Weight) (Average Slope)}}$$

Creating a Calibration Curve

The volume of a calcimeter reaction cell determines the relationship between the pressure increase and the amount of CO_2 released. This relationship is constant for a given reaction cell. The calibration curve and calculated calibration factor are used to convert the amount of pressure released into a percentage of calcium carbonate. All points on the calibration curve represent 100% $CaCO_3$ (for that sample weight). Any number of samples can be used to construct the calibration curve. The following are recommended for accuracy.

1. Prepare five sets of duplicate specimens with the following masses of $CaCO_3$:

   0.2 ± 0.01 g
   0.4 ± 0.01 g
   0.6 ± 0.01 g
   0.8 ± 0.01 g
   1.0 ± 0.01 g

2. Load a calibration sample.

   Perform the procedure outlined in the "Test Procedure", step 3.

3. Tip the cell and allow acid to run out of the cup onto the sample. Swirl the cell gently and continuously until a constant pressure is obtained. This will take at least 10 minutes. Keep the reactants in the lower part of the cell to avoid getting acid into the pressure gauge or pressure transducer. As soon as the reaction has started, observe the rapidly rising pressure. Record the peak pressure to the nearest 0.1 PSI (0.5 kPa) as the $CaCO_3$ pressure for the sample weight used.

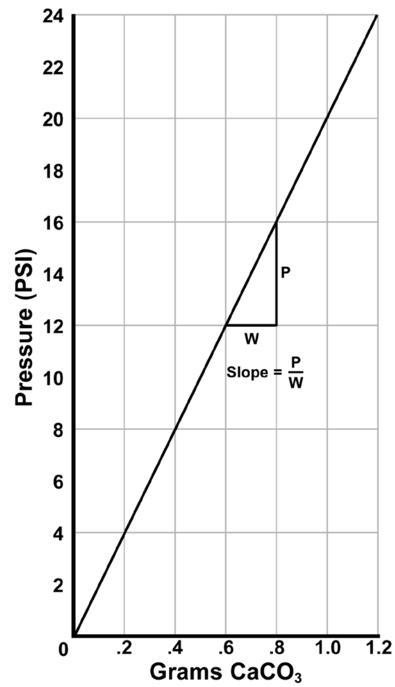
   ☑ Tip

   If a mechanical shaker is available, it may be used to agitate the cell rather than swirling the reactants. Agitate the sample for 10 minutes.

4. Repeat steps 2 and 3 for each of the remaining samples you prepared in step 1.

5. For each sample create a plot on linear graph paper. On the x-axis plot grams of $CaCO_3$ and on the y-axis plot the final pressure in PSI. Draw a straight line through the average of the points. The graph on page 9 shows a sample calibration curve.

6. Because the relationship between pressure and sample size is linear, you may assume the curve to be a straight line with a constant slope. As the graph on page 9 illustrates, the slope of the curve is 2 PSI / .1 g $CaCO_3$, or 20 PSI / 1.0 g $CaCO_3$, resulting in a slope of 20. Therefore, the calibration factor is .05 × 100 = 5. This number is the slope or average slope for the equipment. It is a function of the volume of the reaction cell.



Sample Calibration Curve

As shown by the equations below, the slope can be written as a “Cell Factor” to multiply the pressure reading to directly obtain percentages of Calcium Carbonate and Dolomite.

As described above Slope = 20 PSI for a 1g sample therefore

$$\%\ CaCO_3 = \frac{(\text{Pressure})\ (100)}{(1\ \text{g})\ (20)} = 5 \times \text{Pressure}$$

$$\%\ \text{Dolomite} = \frac{(\text{Total Pressure - } CaCO_3 \text{ Pressure})(.92)(100)}{(1\ \text{g} \times 20)}$$

or

$$\%\ \text{Dolomite} = 4.6 \times (\text{Total Pressure - } CaCO_3 \text{ Pressure})$$

Maintenance

After each test, thoroughly clean the cell and acid cup with water and a mild soap. Use only alcohol-free cleaners. Alcohol can damage the plexiglass cell.

Testing For Leaks

Leaks in the pressure system are the most common case of inaccurate readings.

1. Periodically inspect the reaction cell and replace the o-rings if they are dry, cracked, or worn.

2. Clean the pipe-thread connection between the reaction cell top and the pressure gauge or transducer with a brush and soap suds. If repairs are necessary, disassemble the pipe-threaded connection between the reaction cell and the gauge or transducer. Use teflon tape to re-seal the connection.

3. Check for plugging in the connection between the reaction cell and the gauge or transducer. Also check the gauge entrance or the transducer barrel and diaphragm for build-up of calcium deposit over long periods of time. A warm bleach wash should clean these parts.

4. To check for leaks, pressurize the instrument as described in “Creating a Calibration Curve” above using a .6 g sample. Let the unit stand for at least one hour. The pressure within the reaction cell should remain stable unless leaks are present.

Calibration Data Does Not Give a Straight-Line

If there are no leaks in the system, but results are not giving a straight-line calibration curve or data is otherwise questionable, check the following:

1. Make sure the scale or balance is clean. Shield the balance from air currents and vibration as much as possible when weighing samples or $CaCO_3$ for calibration.

2. Check the reaction cell for contaminants. Be sure the cell is clean and dry.

3. Check for impurities in reagents. Moisture in $CaCO_3$ will result in low-pressure readings.

Warranty and Return Policy

Warranty:

OFI Testing Equipment, Inc. (OFITE) warrants that the products shall be free from liens and defects in title, and shall conform in all respects to the terms of the sales order and the specifications applicable to the products. All products shall be furnished subject to OFITE's standard manufacturing variations and practices. Unless the warranty period is otherwise extended in writing, the following warranty shall apply: if, at any time prior to twelve (12) months from the date of invoice, the products, or any part thereof, do not conform to these warranties or to the specifications applicable thereto, and OFITE is so notified in writing upon discovery, OFITE shall promptly repair or replace the defective products. Notwithstanding the foregoing, OFITE's warranty obligations shall not extend to any use by the buyer of the products in conditions more severe than OFITE's recommendations, nor to any defects which were visually observable by the buyer but which are not promptly brought to OFITE's attention.

In the event that the buyer has purchased installation and commissioning services on applicable products, the above warranty shall extend for an additional period of twelve (12) months from the date of the original warranty expiration for such products.

In the event that OFITE is requested to provide customized research and development for the buyer, OFITE shall use its best efforts but makes no guarantees to the buyer that any products will be provided.

OFITE makes no other warranties or guarantees to the buyer, either express or implied, and the warranties provided in this clause shall be exclusive of any other warranties including ANY IMPLIED OR STATUTORY WARRANTIES OF FITNESS FOR PURPOSE, MERCHANTABILITY, AND OTHER STATUTORY REMEDIES WHICH ARE WAIVED.

This limited warranty does not cover any losses or damages that occur as a result of:

- Improper installation or maintenance of the products
- Misuse
- Neglect
- Adjustment by non-authorized sources
- Improper environment
- Excessive or inadequate heating or air conditioning or electrical power failures, surges, or other irregularities
- Equipment, products, or material not manufactured by OFITE
- Firmware or hardware that have been modified or altered by a third party
- Consumable parts (bearings, accessories, etc.)

Returns and Repairs:

Items being returned must be carefully packaged to prevent damage in shipment and insured against possible damage or loss. OFITE will not be responsible for equipment damaged due to insufficient packaging.

Any non-defective items returned to OFITE within ninety (90) days of invoice are subject to a 15% restocking fee. Items returned must be received by OFITE in original condition for it to be accepted. Reagents and special order items will not be accepted for return or refund.

OFITE employs experienced personnel to service and repair equipment manufactured by us, as well as other companies. To help expedite the repair process, please include a repair form with all equipment sent to OFITE for repair. Be sure to include your name, company name, phone number, email address, detailed description of work to be done, purchase order number, and a shipping address for returning the equipment. All repairs performed as "repair as needed" are subject to the ninety (90) day limited warranty. All "Certified Repairs" are subject to the twelve (12) month limited warranty.

Returns and potential warranty repairs require a Return Material Authorization (RMA) number. An RMA form is available from your sales or service representative.

Please ship all equipment (with the RMA number for returns or warranty repairs) to the following address:

OFI Testing Equipment, Inc.
Attn: Repair Department
11302 Steeplecrest Dr.
Houston, TX 77065
USA

OFITE also offers competitive service contracts for repairing and/or maintaining your lab equipment, including equipment from other manufacturers. For more information about our technical support and repair services, please contact techservice@ofite.com.