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Calcimeter

Gauge-Read Model #152-95

Recording Model #152-96

Instruction Manual

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Ver. 2.2

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Table of Contents

Intro	2
Description	2
Components	3
Safety	4
Setup	5
<i>Recording Calcimeter</i>	<i>5</i>
<i>Gauge-Read Calcimeter</i>	<i>6</i>
Test Procedure	7
Creating a Calibration Curve	9
Chart Recorder	11
Maintenance	13

Intro

The OFITE Gauge-Read Calcimeter and the OFITE Recording Calcimeter are 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 Calcimeters can help determine the proper chemical treatment.

These instruments comply 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.

The OFITE Gauge-Read Calcimeter (152-95) features a pressure gauge on top of the reaction cell. With this model, the pressure within the reaction must be manually read from this gauge.

The OFITE Recording Calcimeter (152-96) includes a transducer and a chart recorder that automatically records the pressure readings without user intervention. This model was designed to make the test procedure easier by enabling you to run it virtually unattended.

Components

Gauge-Read Calcimeter (152-95):

- #142-54 O-ring
- #152-95-1 Gauge with Cover, 30 PSI, 4" Diameter
- #152-95-2 Bleed-Off Screw
- #152-95-3 Cell Cap
- #152-95-4 Reaction Cell
- #152-95-5 O-ring, $1\frac{3}{8}$ " \times $\frac{1}{16}$ "
- #152-95-6 Sample Cup

Recording Calcimeter (152-96):

#152-95 Calcimeter

- #142-54 O-ring
- #152-95-2 Bleed-Off Screw
- #152-95-3 Cell Cap
- #152-95-4 Reaction Cell
- #152-95-5 O-ring, $1\frac{3}{8}$ " \times $\frac{1}{16}$ "
- #152-95-6 Sample Cup
- #141-17 Clip for Graduated Cylinder
- #152-37 AC Power Cord, 3-Conductor
- #152-95 Calcimeter
- #152-96-2 Minigraph Recorder
- #152-96-3 Stainless Steel Case
- #152-96-4 Chart Paper for Minigraph
- #152-96-5 Pressure Transducer
- #152-96-6 Porcelain Mortar, 65 mL
- #152-96-7 Porcelain Pestle
- #152-96-8 #30 Gear Train
- #152-96-10 Power Supply
- #152-96-11 Female Cable Jack
- #152-96-12 Male Cable Plug
- #152-96-13 Insulated Banana Plug, Black
- #152-96-14 Insulated Banana Plug, Red
- #153-02 Brush; Graduate; $1\frac{1}{2}$ " \times $10\frac{3}{4}$ "
- #153-18 Glass Graduated Cylinder 10 mL \times $\frac{2}{10}$ mL
- #153-55 Silicone Stopcock Grease, 150 g Tube (Dow Corning)
- #166-03 Hand-held Balance, 0 - 320 g (0.1g)
- #275-03 *Hydrochloric Acid; 10%; 8 oz (UN #1789)
- #280-00 Wetting Agent; 1 oz
- #285-00-1 Calcium Carbonate; 100 g

Safety



Important

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.

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

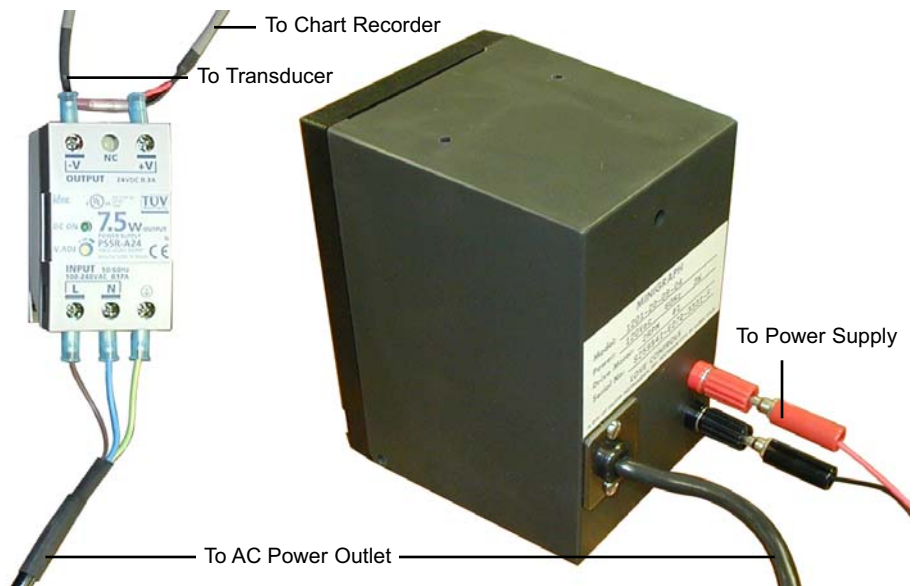
The OFITE Recording Calcimeter uses an electrically powered transducer and recorder to record the pressure in the reaction cell. Observe the following safety considerations at all times:

1. Make sure the power switch is off before connecting the power cable to an electrical outlet.
2. Make sure you are using a three-wire grounding type power cable and outlet.
3. Always unplug the power cable before opening the recorder to replace chart paper or any other maintenance or repair work.

Setup

Recording Calcimeter

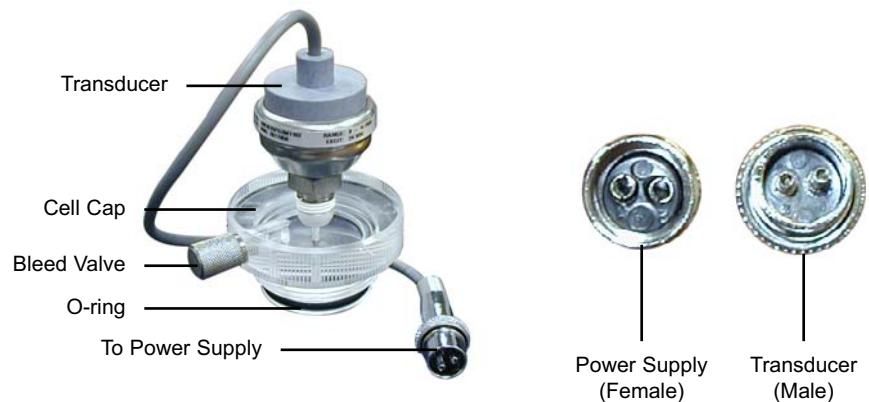
1. Begin by carefully unpacking the equipment. Inspect each piece to ensure everything is clean and in good operating condition. Inspect the cell cap o-ring for cracks or wear.
2. Verify that the chart recorder has enough paper and that it is loaded properly. For more information, see the "Chart Recorder" section on page 2.
3. Plug the power supply into the back of the chart recorder. Make sure the red cord goes into the red plug and the black cord goes into the black plug.



Power Supply

Chart Recorder

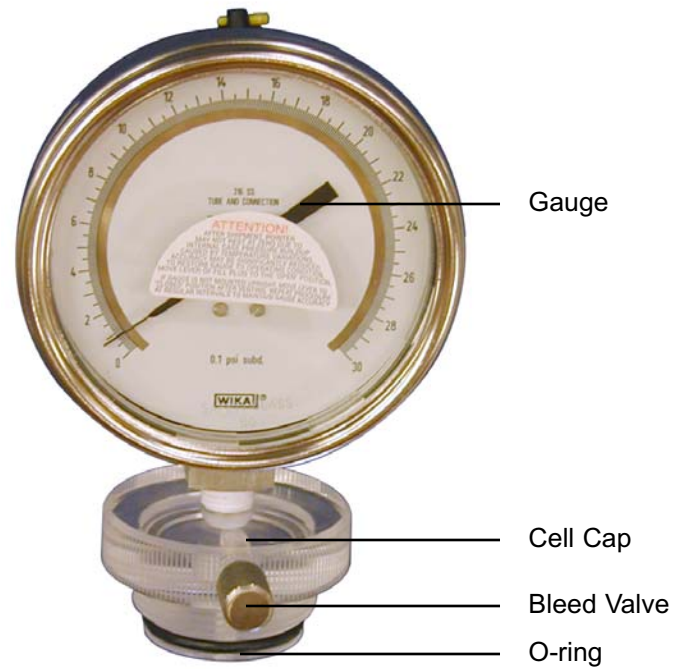
4. Connect the transducer to the power supply. The cable connectors are keyed to prevent an incorrect connection. Be sure to tighten the cable lock to keep the connection secure.
5. Plug the chart recorder and power supply into an appropriate AC power outlet.



Setup

Gauge-Read
Calcimeter

1. 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.

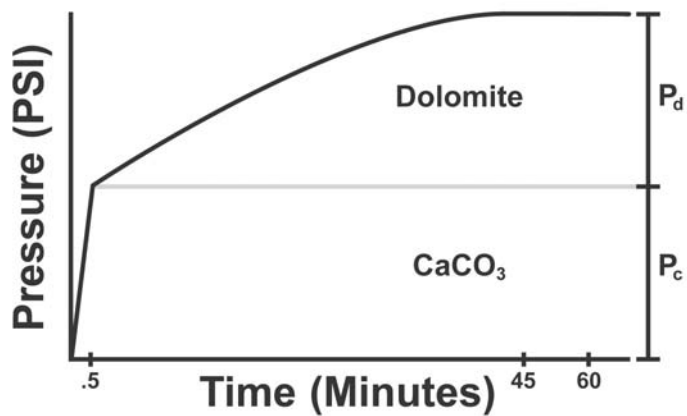


Test Procedure

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. Brush the paper with a small brush to remove traces of the sample, then remove the paper.
 - e. 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.
 - f. Hand tighten the cell cap. Be careful not to splash any acid onto the sample.
 - g. Open the bleed valve until the pressure reading is zero. Then close the bleed valve tightly.
4. Turn the reaction cell back to vertical and start timing the test. This will start the reaction between the HCl and the CaCO₃.

- At 30 seconds, record the pressure as “CaCO₃ 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 CaCO₃ pressure plus the dolomite pressure. To calculate the dolomite pressure, subtract the CaCO₃ pressure (30 second reading) from the total pressure (30 - 45 minute reading).
- Refer to the graph below to interpret the pressure readings.



- Use the equations below to calculate the percentages of CaCO₃ and dolomite. For values of “Slope” refer to the “Creating a Calibration Curve” section below.

$$\% \text{CaCO}_3 = \frac{(\text{Pressure Reading, PSI}) (100)}{(\text{Sample Weight}) (\text{Average Slope})}$$

$$\% \text{Dolomite} = \frac{(\text{Total Pressure} - \text{CaCO}_3 \text{ Pressure}) (100) (.92)}{(\text{Sample Weight}) (\text{Average Slope})}$$

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.

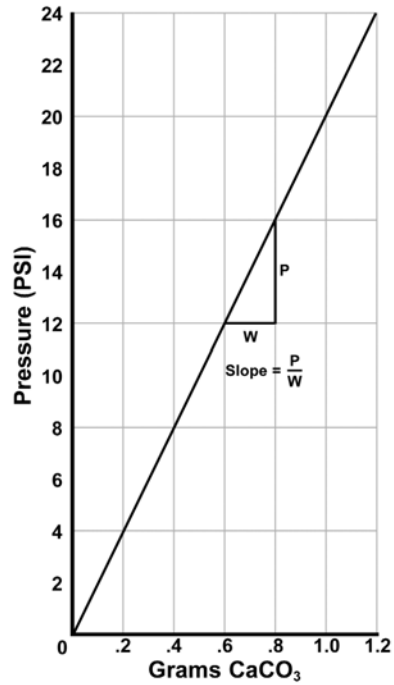
3. Tip the cell and allow acid to run out of the cup onto the sample. Swirl the cell gently and continuously until a constant pressure is obtained. This will take at least 10 minutes. Keep the reactants in the lower part of the cell to avoid getting acid into the pressure gauge or pressure transducer. 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 $.05 \times 100 = 5$. 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 by to directly obtain percentages of Calcium Carbonate and Dolomite.

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

$$\% \text{ CaCO}_3 = \frac{(\text{Pressure}) (100)}{(1 \text{ g}) (20)} = 5 \times \text{Pressure}$$

$$\% \text{ Dolomite} = \frac{(\text{Total Pressure} - \text{CaCO}_3 \text{ Pressure})(.92)(100)}{(1 \text{ g} \times 20)}$$

or

$$\% \text{ Dolomite} = 4.6 \times (\text{Total Pressure} - \text{CaCO}_3 \text{ Pressure})$$

Chart Recorder

The OFITE Recording Calcimeter differs from the pressure gauge model only in that a pressure transducer and small strip chart recorder are provided to replace the pressure gauge. It is highly recommended that you carefully read the instruction manual for the chart recorder before attempting to use the equipment.

The unit is a Love Controls 1200 series Minigraph Recorder. It uses an “inkless writing system”, which uses a metal stylus to tap a dotted line on pressure sensitive chart paper. It has been adapted for use with the Calcimeter by adding a pressure transducer and power supply. The unit will record the pressure changes within the reactor cell as they occur. The percentage of calcium carbonate and dolomite can be calculated by applying the calibration factor to the chart reading.

The transducer in the chart recorder has a range from 0 to 15 PSI. However, the chart readings represent milliamperes, not pressure. In order to convert the reading to PSI, figure the percentage of full scale that is being displayed. Then multiply that percentage by 15. This will give you the PSI reading.

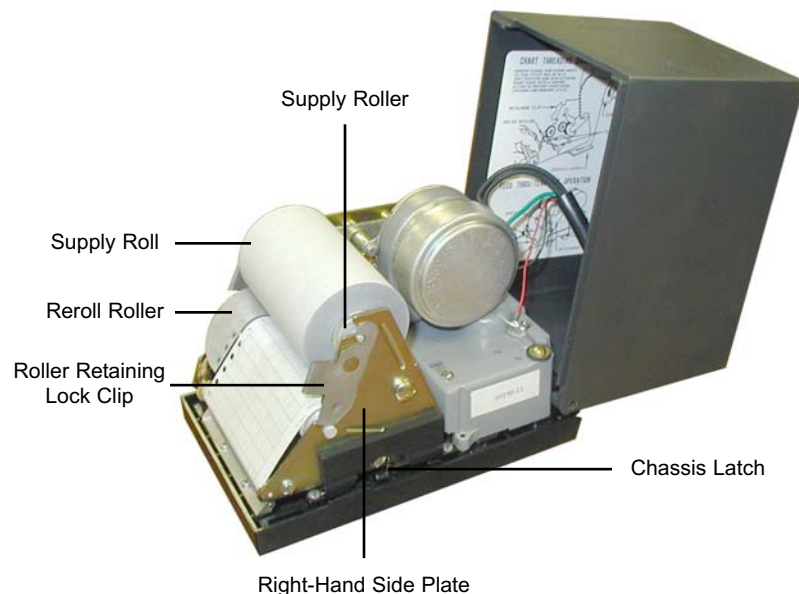
The chart paper included with the recorder shows 10 large divisions. If the chart paper shows a value on the third division (30% of full scale), the pressure reading is 4.5 PSI ($15 \text{ PSI} \times .3 = 4.5$).

There are two methods for installing chart paper. The Reroll method routes the chart onto a roller for storage and review at a later time. The Feed Through method routes the chart upwards through the top of the door so that it may be viewed easily during recording. OFITE recommends using the Feed Through method.

1. Remove all signal and power inputs to the instrument before changing the chart paper.
2. Press down on the door release latch and carefully swing the door downward.



3. Flip the chassis latch outward.
4. Roll the o-rings from the center of the feed through the roller to fit into the groove on each side of the roller.
5. Flip up the roller retaining lock clips.
6. Remove the supply roller from the instrument. Slide the supply roller into the perforated end of a roll of chart paper.
7. Hold the loose end of the chart paper in your left hand and the supply roll in your right hand so that the blank side of the chart paper is facing upward between your left hand and the roller. Slip the chart paper under the right hand side plate. Carefully maneuver the chart paper so that the perforations fit into the sprocket and drive rollers.
8. Press the supply roll tension arm toward the left side plate. Set the supply roller pins into the notches of the side plates and snap the roller retaining lock clips down into the lock position.
9. Pull the chart paper out from the supply roll until it extends out beyond the top of the door.
10. Press down on the right-hand side plate. Flip the chassis latch up (press the latch against the side plate until it snaps into place). Close the door. Reconnect the signal and power cables. Rotate the thumbwheel on the outside of the door to advance the chart paper as desired.



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 plexi-glass 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 Chlorox 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.

Recorder Not Responding To Pressure Changes In Reaction Cell.

1. Make sure the power cable is plugged into the electrical outlet and that power is available at the outlet.
2. Verify the power switch on the recorder is ON.
3. Check the plug and receptacle going into the recorder to make sure they are making a good connection.
4. Check the plug and receptacle connecting the transducer to the recorder. Make sure this plug and receptacle are making a good connection.