INSTRUCTIONS:
OFI CALCIMETER
PART No. 152-95

The OFITE calcimeter is used for the determination of alkaline earth carbonates such as calcium carbonate and dolomite.

EQUIPMENT:

1. Plexiglass CO2 cell body and cap
2. Acid cup for plexiglass cell
3. Pressure gauge, 30 psi

OPTIONAL EQUIPMENT AND REAGENTS:

1. Shaker, mild agitation
2. Balance, portable or analytical, 0.1 mg
3. Morter and pestle
4. Pipette, 10 ml – PART NO. 153-40 with safety bulb PART NO. 153-41
5. Glazed paper or equivalent
6. Lubriseal PART NO. 153-55
7. Graph paper
8. Brush, small
9. Hydrochloric acid 10%
10. Calcium carbonate, PART NO. 285-00
11. Carrying case, ss, PART NO. 144-35

PROCEDURE:

A. Determination of a calibration curve

1. Weigh 0.200g, 0.400g, 0.600g, etc. up to 1.40g CaCO3 on glazed paper.
2. Transfer weighed sample to bottom of clean and dry CO2 cell.

3. Pour 10 ml of 10% hydrochloric acid solution into acid cup and lower into cell. Do not spill or get any of the acid solution on bottom of cup.
4. Inspect cell o rings and apply a light coating of lubriseal.
5. Tighten cell cap being careful not to splash acid solution onto sample.
6. Check pressure gauge to make sure it is at 0 psi before mixing reactants.
7. Tilt cell and allow acid solution to run out of cup onto weighed sample. Swirl continuously by hand until a constant pressure is obtained. Keep reactants in lower part of cell to avoid getting acid into pressure gauge.
8. Plot grams of CaCO3 versus pressure on regular graph paper.
9. Compute the average slope of the calibration curve by obtaining form the curve two high pressure points (P2 – P1) and corresponding weights (W2 – W1). Compute the slope form the formula below. Repeat the slope calculation for two low pressure points and corresponding weights. Finally, compute an average slope for the curve using the two slopes just computed.

   CALCULATIONS:

   $$\text{SLOPE} = \frac{P2 - P1}{W2 - W1}$$ P = Pressure (PSI), W = Weight (grams)

   $$\text{AVERAGE SLOPE: } \frac{\text{Slope1} + \text{Slope 2}}{2}$$
10. Cell should be calibrated monthly.

B. SAMPLE TREATMENT

1. Grind sample to at least 100 mesh with mortar & pestle.
2. Weigh approximately (1.0 to 1.4 grams) sample to nearest 0.01 gram or better depending on type of balance available.
3. Follow steps 1 through 8 as in part A.
4. As soon as the cell is tilted to start the reaction, observe and record the rapidly rising pressure at its peak. Record this as CaCO3 pressure. If dolomite is present after a slight pause, a second rise in pressure will occur. The dolomite reaction is slow, so continue to swirl cell and allow sufficient time for completion, i.e. when pressure remains constant for 15 – 20 minutes. This final value of pressure is the total CaCO3 plus dolomite pressure. Use the following formula for determining percentages.

   $$\%\ \text{CaCO3 as recorded} = \frac{(\text{Pressure reading, PSI})\ (100)}{(\text{Sample weight})\ (\text{Avg. slope})}$$

   $$\%\ \text{Dolomite as recorded} = \frac{(\text{Total pressure-pressure CaCO3})\ (100)\ (0.92)}{(\text{Sample weight})\ (\text{Avg. slope})}$$

C. REMARKS

1. Because of potential leaks in the pressure system, periodically check and replace if necessary, CO2 cell, or O-rings.
2. A serious leak will show up during a test as a drop in pressure after the normal pressure build up from the CO2 reaction.
3. To check for leaks, react (CaCO3 with HCL) to a pressure of 10 – 15 psi and let the cell set for one hour, a drop in pressure build up indicates a leak is present.
4. The results of test run on samples should agree with the initial calibration curve to within +- 5%. If no leaks are present and they do not, perform the following checks:

    a. Check for sticking gauge.
    b. Make sure balance is clean, shield balance from air currents and vibration as much as possible.
    c. Be sure CO2 cell is clean and dry.
    d. Check for impurities in reagents. Moisture in CaCO3 standart will introduce an error.

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