Applied Microbiology
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Standard Operating Procedure

AMBL-250-A

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Carbon Dioxide Determination of Carbonate Rock by Differential Loss on Ignition

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METHOD SUMMARY

This SOP describes a procedure for determining the carbon dioxide mass contained in a carbonate rock or other rocks that contain or are suspected of containing carbonates. The determination is based on the thermal decomposition of carbonate resulting in the loss of carbon dioxide mass that is measured gravimetrically. This method is adapted from *Methods of Chemical Analysis for Carbonate and Silicate Rocks*, Bulletin 152, Part 1 (State Geological Survey of Kansas, 1961).

ENVIRONMENTAL HEALTH AND SAFETY

Hazards Assessment: This method involves the use of a muffle furnace operated at extremely high temperatures (550°C and 1000°C). In appropriate handling of the furnace doors, heated exterior surfaces and samples being place in or removed from the furnace can lead to any one of the following types of burn classification.

First-degree (superficial) burn

A first-degree burn affects only the epidermis, or outer-most layer of skin. The burned skin will be red, painful, dry, and does not blister. Long-term damage to the tissue is uncommon but would typically result in an increase or decrease skin color.

Second-degree (partial thickness) burn

A second-degree burn affects the epidermis and a portion of the lower skin layer, the dermis. The burned area will be red, blistered, and may be swollen and painful.

Third-degree (full thickness) burn

A third-degree burn destroys both the epidermis and dermis, and may also damage the underlying bones, muscles, and tendons. Once bones, muscles, or tendons are also burned, this may also be referred to as a fourth-degree burn. The burned area appears white or charred, and the skin may peel. Since nerve endings are destroyed for this classification of burn, there will be no feeling in the burned area.

A burn of any degree, should be treated immediately. The information provided below is intended as general guidance only and is not to be taken as medical advice.

First-degree burn

A first-degree burn is comparable to a mild sunburn and first aid treatment may be applied by running cool water over the burn area. Do not apply ice. Apply an antibiotic cream and cover the burn area lightly with a sterilized gauze. Consult your medical provider for additional treatment information or to discuss use of over-the-counter pain medication.

Second-degree burn

A second-degree burn may be initially treated as done for a first-degree burn. Contact your healthcare provider as soon as possible and schedule appointment if recommended. Your doctor may prescribe a stronger antibiotic cream to kill bacteria. It has been suggested that elevating the burned area may reduce pain and swelling.

Third-degree burn

Seek medical attention immediately for a third-degree burn. Depending on the severity, a third-degree burn can be life-threatening and often requires skin grafts.

Safety Equipment and Engineering Controls: This method requires long tongs to be used when moving samples in and out of the furnace. Once the operation of the muffle furnace at 1000°C has concluded, allow the furnace to cool for a minimum of 1 hour before opening the door. Allow the furnace to cool for a minimum 30 minutes before opening the door when operating the muffle furnace at 550°C.

Personal Protective Equipment (PPE): This method requires the use of the following PPE.

Heat resistant gloves when moving sample in and out of the furnace.

Safety goggles or glasses

Laboratory coat

Analysis-derived Wastes and Disposal:

Waste Generated	Hazardous (Y / N)	Disposal
This procedure generates a rock residue.	N	May be disposed as a landfill waste.

METHOD DESCRIPTION

1.0 Introduction and Applicability

Thermal decomposition of carbonate to oxygen and carbon dioxide occurs at temperatures between 900 and 950°C. The following reaction shows this decomposition for calcium carbonate resulting in calcium oxide and carbon dioxide.

$$CaCO_3 \xrightarrow{+\Delta Heat} CaO + CO_2$$

The loss of carbon dioxide is determined gravimetrically by the difference between a sample's mass before and after being exposed to a temperature of 1000°C. This difference can be used to calculate the stoichiometric amount of carbonate that would have been originally present.

This method is particularly applicable to rocks containing carbonate, such as calcite, aragonite, and dolomite ($CaMg(CO_3)_2$), or soils derived from these weathered rocks. Rocks or soils suspected of containing carbonates may also be used with this method, but may result in unquantifiable results due to either low or negligible CO_2 mass loss during ignition.

2.0 Apparatus

a. Convection oven operated at 103-105°C for drying sample to a constant weight.

b. Muffle furnace, operated at 550 ± 50°C for igniting and removing organic carbon and operated at 1000 ± 15°C for thermally decomposing inorganic carbon in carbonates to carbon dioxide gas.

c. Evaporating Dish, for sample drying and ignition, made of porcelain (optionally use platinum).

d. Analytical balance capable of weighing to the nearest 0.0001 g or less.

e. Desiccator, glass

f. Mechanical ball mill (optional)

g. Mortar and pestle

h. Sieves, 2.0 mm (10 mesh, 0.0787 in) and 0.250 mm (60 mesh, 0.0098 in.)

3.0 Reagents

a. Carbonate standard. Calcium carbonate (ACS grade, ≥95%).

4.0 Procedure

4.1 Sample Preparation

Using cleaned equipment, mechanically or manually crush and grind the sample to a coarse size that can pass through a 2.0 mm (No. 10) sieve. Remove the larger material from the sieve and continue crushing the material until it passes.

Thoroughly mix this coarse material, and if necessary, quarter and split it to reduce the sample to approximately 100 g. Continue to grind and reduce the material size until it passes a 0.250 mm (No. 60) sieve.

Remix the sample thoroughly for several minutes because grinding and sieving tends to segregate the soft and hard portions of the sample. Remixing is done to assure sample homogeneity. This is particularly important for minimizing variability when the material is subsampled to obtain separate portions for analysis.

4.2 Determine Dry Mass of Sample

A. Pretreat evaporating dish prior to its first use and determine the tare mass.
   1. Clean and dry the dish and place it into the muffle furnace.
   2. Bring the furnace temperature up to 1000°C and leave at this temperature for 1 hour.
   3. Turn off the muffle furnace, allowing it to cool for at least an hour or preferably until the temperature is less than 500°C.
   4. Remove the dish, cool in a glass desiccator to room temperature and weigh to the nearest 1 mg. This is the tared mass.

   Note: It is important that the evaporating dish and sample have reached room temperature before weighing. It is not uncommon to experience a gradual loss of mass displayed over a period of 5 to 20 minutes when cooling has not achieved a stable temperature. When this condition is observed to be occurring, allow the balance time to reach a minimum mass before recording. This is typically characterized by the mass reaching a minimum value and then increasing.

B. Dry the sample to constant weight and determine dry mass and moisture content of sample. Prepare from 3 to 5 separate portions of the sample.

1. Place approximately two to five grams of sample into each clean, dry and tared dish. Weigh and record the combined mass of each dish and sample to the nearest 1 mg.
2. Calculated the initial sample mass in each dish by subtracting the tared dish mass from the combined mass.
3. Place the dish into the 103-105°C oven for no less than 1 hour. Remove the dish, place it in a glass desiccator and cool to room temperature.
3. Weigh and record the dried combined sample and dish mass.
4. Calculate the dried sample mass by subtracting the tared dish mass from the dried combined mass difference.
5. Repeat this drying and weighing cycle until the change in dried sample mass between the last weighing and the most recent previous weighing is no greater than 1% of the previous dried sample mass. For example: If the previous dried sample mass is 2,851 mg, then the last dried sample mass can differ by no more than 28.5 mg. Report this final mass as the dry sample mass.
6. Alternatively, dry the sample in the 103-105°C oven overnight, weight and record the dried combined mass, calculate the dried sample mass and record this as the final dry sample mass.

4.3 Determine Moisture Content of Sample

A. The moisture content of the sample is the proportion of the initial sample mass that is water and is expressed as a percentage. Calculate the sample's moisture content as follows.

$$W\ (\%) = \frac{M_T - M_{103}}{M_T} \times 100$$

where: W (%) = sample moisture content
M_T = total initial sample mass, mg
M_{103} = dry sample mass, mg

B. Report moisture content of the sample to the nearest 1%.

4.3 Determine Organic Content of Sample

A. Organic content of the sample material is represented by the proportion of the dry sample mass that is lost at a temperature of 550°C and is expressed as a percentage.

   1. Place the combined dry sample and dish into the muffle furnace at a temperature of 550°C for 25 minutes.
   2. Turn off the furnace and allow it to cool for at least 30 minutes.
   3. Remove the sample dish, place it into a glass desiccator and continue cooling to room temperature.
   4. Weigh and record the remaining inorganic sample mass to the nearest mg.

B. Calculate the organic content of the dry sample mass as follows.

$$OC\ (\%) = \frac{M_{103} - M_{550}}{M_{103}} \times 100$$

where:
$OC\ (\%)$ = sample organic content
M_{103} = dry sample mass, mg
M_{550} = remaining inorganic sample mass after ignition at 550°C, mg

C. Report organic content of the dry sample to the nearest 1%.

4.4 Determine Carbonate Content of Sample

A. Carbonate content is determined as the loss of carbon dioxide that occurs when carbonate is decomposed at 1000°C and is reported as a percentage.

   1. Return the combined sample and dish mass into the 550°C muffle furnace and set the furnace to 1000°C.
   2. Once the temperature reaches 1000°C, leave the sample in the furnace for at least one hour.
   3. Turn off the furnace and allow it to cool for at least an hour or preferably until the temperature is less than 500°C.
   4. Remove and place the sample in a glass desiccator and to continue to cool to room temperature.
   5. Weigh and record the combined sample and dish mass to the nearest mg.

B. Calculate the carbon dioxide mass loss and content of dry sample mass as follows.

$$CO_2\ (mg) = M_{550} - M_{1000}$$

where: CO_2 (mg) = sample carbon dioxide mass loss
M_{550} = remaining inorganic sample mass after ignition at 550°C, mg
M_{1000} = remaining inorganic sample mass after ignition at 1000°C, mg

and

$$CO_2\ (\%) = \frac{M_{550} - M_{1000}}{M_{103}} \times 100$$

where: CO_2 (%) = sample carbon dioxide content
M_{103} = dry sample mass, mg
M_{550} = remaining inorganic sample mass after ignition at 550°C, mg
M_{1000} = remaining inorganic sample mass after ignition at 1000°C, mg

C. Report carbon dioxide mass to the nearest mg and carbon dioxide content to the nearest 1%.

D. Calculate calcium carbonate mass and content, and carbonate mass and content of dry sample mass as follows.

$$CaCO_3\ (mg) = CO_2\ (mg) \times \frac{\left(MW_{CaCO_3}\right)}{\left(MW_{CO_2}\right)}$$

and

$$CaCO_3\ (\%) = \frac{CaCO_3\ (mg)}{M_{103}\ (mg)} \times 100$$

and

$$CO_3^{-2}\ (mg) = CO_2\ (mg) \times \frac{\left(MW_{CO_3^{-2}}\right)}{\left(MW_{CO_2}\right)}$$

and

$$CO_3^{-2}\ (\%) = \frac{CO_3^{-2}\ (mg)}{M_{103}\ (mg)} \times 100$$

where: $CaCO_3$ (mg) = sample calcium carbonate mass
MW_{CaCO3} = molecular weight of calcium carbonate
MW_{CO2} = molecular weight of carbon dioxide
$CaCO_3$ (%) = sample calcium carbonate content
$CO^{-2}{}_3$ (mg) = sample carbonate mass
$MW_{CO_3^{-2}}$ = molecular weight of carbon dioxide
$CO^{-2}{}_3$ (%) = sample carbonate content

E. Report calcium carbonate and carbonate mass to nearest mg of dry sample mass, and calcium carbonate and carbonate content to nearest 1%.

5.0 Quality Control

Periodically prepare and weigh three samples of approximately 5g of calcium carbonate (ACS grade) and run these through the entire procedure.

A. Accuracy. A single laboratory study determined the three-sample bias of carbon dioxide loss from calcium carbonate reagent (99.731% purity) to be -3.4% without correcting for reagent impurities and 2.6% after correcting for impurities.

B. Precision. The same single laboratory study determined the three-sample standard deviation of 0.0389 grams.

6.0 References

1. State Geological Survey of Kansas (1961) Walter E. Hill, Jr., Wanda N. Waugh, O. Karmie Galle, and Russell T. Runnels. *Methods of Chemical Analysis for Carbonate and Silicate Rocks*, Bulletin 152, Part 1, University of Kansas Publications, Lawrence, Kansas.