FLORIDA DEPARTMENT OF TRANSPORTATION
State Materials Office
5007 NE 39th Avenue
Gainesville, Florida 32609

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Florida Test Method for Carbonates and Organic Matter in Base Materials

Designation: FM 5-514

1. SCOPE

This method covers the chemical analysis of cemented coquina and bank run shell carbonates of calcium and magnesium and organic matter such as wood trash or other vegetation.

Note 1: The values stated in SI units are to be regarded as the standard. The values given in parenthesis are for information only.

2. REFERENCED DOCUMENTS

2.1 AASHTO Standard:

M 231 Specification for Weighing Devices in the Testing of Materials

2.2 ASTM Standard:

E 50 Recommended Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

2.3 Other Documents:

FM 1-R 076 Florida Test Method for Reducing Field Samples of Aggregate to Testing Size

FM 5-532 Florida Test Method for the Field Evaluation of Sealants for Shoulder Joints

Hydrochloric Acid Safety Data Sheets (SDS)

Ammonium Hydroxide Safety Data Sheets (SDS)

3. OUTLINE OF METHOD

3.1 Impurities are determined as acid insoluble matter and as the ammonium hydroxide precipitate; carbonates are then calculated by difference. Organic matter is determined as the loss on ashing of acid insoluble matter. For determination of carbonates only refer to ***Section 8***.

4. APPARATUS

4.1 Bunsen burners with tripods or suitable hot plates.

4.2 Analytical balance sensitive to 0.1 mg complying with AASHTO M 231, Class A.

4.3 300 mL glass beakers or other convenient size.

4.4 50.00 mL capacity dispensing burette.

4.5 15 mL nominal capacity porcelain crucibles.

4.6 Desiccator with Drierite or any other efficient desiccant.

4.7 Drying gravity oven, capable of maintaining a temperature of 105-115°C. (221-239°F)

4.8 Glass Filtering funnels, 60° nominal, 75 mm nominal top diameter.

4.9 25 mL nominal capacity Gooch crucibles.

4.10 Jaw crusher and grinder capable of reducing dried samples to 90% passing the 425-μm (No. 40) sieve and with 100% passing the 2.00-mm (No. 10) sieve. Manual crushing of samples may be substituted for mechanical jaw crushers provided the integrity of the sample is maintained.

4.11 Muffle furnace capable of maintaining temperatures of 600±50°C (1030-1200°F) and 900±50°C (1550-1750°F).

4.12 Vacuum filter flask, 1 L capacity.

4.13 Stirring glass rods and rubber policeman.

4.14 Wash bottles, polyethylene squeeze type, filled with Deionized (DI) or distilled water.

5. REAGENTS AND SUPPLIES

5.1 Hydrochloric acid (1 + 5) solution: Add 1 volume of reagent grade concentrated hydrochloric acid to 5 volumes distilled or deionized water and mix.

5.2 Ammonium hydroxide (1 + 5) solution: Add 1 volume of reagent grade ammonium hydroxide to 5 volumes distilled or deionized water and mix.

5.3 Glass microfiber filter discs such as Whatman No. 934-AH, 21 mm diameter.

5.4 Quantitative filter paper, Whatman No. 41, 125 mm diameter.

5.5 Phenolphthalein Indicator: Dissolve 1 g indicator grade phenolphthalein in 100 mL reagent grade methanol or denatured alcohol. As an alternative commercially available 1% (w/v) alcoholic phenolphthalein solution may be used.

6. SAMPLE PREPARATION

6.1 If the material is damp when received from the field, it shall be dried until it becomes friable under the trowel. It may be air dried or by use of a drying apparatus provided the temperature does not exceed 60°C (140°F).

Note 2: Minimum gross sample size for Cemented Coquina, Shell Rock and Bank Run Shell materials shall be 23 kg (50 lb.).

6.2 For materials to be used as base or stabilizers, the entire sample shall be passed through a crusher set at a maximum opening of 19 mm (3/4 in), with an under tolerance of 3 mm (1/8 in). Alternately, the entire sample may be crushed before the drying operation in ***Section 6.1***.

6.3 Reduce samples using FM 1-R 076 for oven drying to test size of at least 1.4 kg, (3 lb.) for lime rock, and at least 2.3 kg [5 lb.] for Cemented Coquina, Shell Rock and Bank Run Shell).

6.4 Oven dry the split from ***Section 6.3*** at 110±5°C (230±9°F) for 12 h minimum (overnight is convenient).

6.5 Crush the split sample again, if needed, to a size that will pass the pulverizer throat.

6.6 Split the crushed material until a sample weighing at least 0.1 kg (1/4 lb.) or approximately 0.25 L (1/2 pt.) is obtained. For Cemented Coquina, Shell Rock and Bank Run Shell at least 0.5 kg (1 lb.) or approximately 1 L (1 qt) is required.

6.7 Pass the entire final sample through a pulverizer so that 90% passes 425-μm (No. 40) and 100% passes 2.00-mm (No. 10) sieve, and place in a moisture free container to assist analysis. This may require two passes through the pulverizer. For Cemented Coquina, Shell Rock and Bank Run Shell, split the pulverized material until a sample weighing at least 0.1 kg (1/4 lb.) or approximately 0.25 L (1/2 pt.) is obtained.

6.8 Clean the crusher, pulverizer, and splitters between samples to avoid contamination of the next sample.

7. METHOD

7.1 Give the sample a final mixing by stirring with a spatula.

7.2 Weigh approximately 1 g of sample in a 300 mL glass beaker. Record the weight.

7.3 Slowly, add 20 mL of hydrochloric acid (1 + 5) to beaker.

7.4 Heat the solution using a Bunsen burner flame or hot plate until it momentarily boils. Remove the beaker from heat source and allow to stand until the last of the gas has evolved from the sample.

7.5 Insert a microfiber filter disk in tared Gooch crucible and weigh. To filter, transfer all solid material in the beaker to the Gooch crucible by a combination of scrubbing with glass stirring rod with policeman and washing with water. Wash down walls and bottom of Gooch crucible twice with water. Save filtrate (see ***Section 7.8***).

Note 3: A tared Gooch crucible is prepared by heating in a muffle furnace at 950 ±50°C (1550-1750°F) for 30 min. and cooled in a desiccator.

7.6 Dry Gooch crucible for 2 h at 105-115°C (221-239°F), cool in desiccator to room temperature and weigh. Increase in mass represents insoluble silica, clay, and organic matter (Residue A). The mass of Residue A is the mass of the Gooch crucible

with residue (g) – mass of tared Gooch crucible with filter (g).

7.7 Ignite Gooch crucible at 600±50°C (1030-1200°F) for 30 min, cool to room temperature in desiccator, and weigh. Loss in mass represents organic matter (Loss B). The mass of Loss B is the mass of the Gooch crucible with residue (g) – mass of tared Gooch crucible with filter (g).

7.8 Transfer filtrate from ***Section 7.5*** back to original beaker, add 3 drops of phenolphthalein and add ammonium hydroxide (1 + 5) to neutralize to a faint pink color. Heat the solution and boil momentarily, then allow to cool until precipitate has settled enough for rapid filtration. Place the Whatman 41 filter paper in glass funnel and filter the insoluble residue. Use the wash bottle with DI or distilled water to rinse the filter and funnel. Wash paper five times, allowing paper to drain between washings. Scrub the walls of the beaker using a glass stirring rod with rubber policeman. Use water to transfer all insoluble residue from beaker and rinse rubber policeman. Filter all rinses.

7.9 Carefully, remove the filter from funnel and fold to contain insoluble residue. Place inside a tared porcelain crucible. Ignite crucible with filter at 900±50°C (1550-1750°F) for 30-45 minutes. Cool crucible and contents in desiccator and weigh. The mass of the residue (Residue C) is the mass of the crucible with residue (g) – mass of tared crucible (g).

Note 4: A tared crucible is prepared by heating in a muffle furnace at 950 ±50°C (1550-1750°F) for 30 min. and weighed after cooling in a desiccator.

7.10 Calculations:

% Carbonates of calcium and magnesium = (1.000 - Residues A and C) x 100

% Organic Matter = Loss B (step 7.7) x 100.

8. SHORT METHOD- FOR CARBONATES ONLY

8.1 Give the sample a final mixing by stirring with a spatula. Weigh approximately 1 g of sample in a 300 mL beaker. Record the weight.

8.2 Add 20 mL of hydrochloric acid (1 + 5) solution.

8.3 Heat to boiling using a hot plate or Bunsen burner with tripod.

8.4 Remove from heat. Allow to stand until the last of the gas has evolved from the sample.

8.5 Add 3 drops of phenolphthalein solution.

8.6 Add ammonium hydroxide (1 + 5) solution to neutralize to a faint pink color and boil momentarily.

8.7 Place the Whatman 41 filter paper in glass funnel and filter the insoluble residue. Use the wash bottle with DI or distilled water to rinse the filter and funnel. Wash paper five times, allowing paper to drain between washings. Scrub the walls of the beaker using a glass stirring rod with rubber policeman. Use water to transfer all insoluble residue from beaker and rinse rubber policeman. Filter all rinses.

8.8 Carefully, remove the filter from funnel and fold to contain insoluble residue. Place inside a tared porcelain crucible (See ***Note 4***.). Ignite crucible with filter at 900±50°C (1550-1750°F) for 30-45 minutes.

8.9 Cool in a desiccator and weigh contents of crucible with the insoluble residue (R). The mass of insoluble residue (R) is the mass of crucible with the residue(g) - mass of tared crucible (g).

8.10 The carbonate content is calculated as follows:

$$C = \frac{(W - R)}{W} \times 100$$

Where:
C = % of carbonates of calcium and magnesium
W = mass of sample, g
R = mass of insoluble residue, g

9. REPORTING

9.1 Report % of Carbonates and % Organic Matter to the nearest whole number.

10. PRECISION

10.1 Cooperative tests have shown that these differences between labs are reasonable:

Carbonate Range	
90% and up	± 0.4%
50% - 89%	± 0.9%

11. SAFETY

11.1 The concentrated acid and alkali reagents should be handled under an efficient fume hood. Follow all Safety Measurements on SDS. The diluted solutions may be used outside the hood, in a well-ventilated area, without known harm. Avoid contact of all reagents with the skin and eyes. In the case of accidental splashes in the eyes, flush thoroughly with water and contact a physician.

Additionally, no employee is to use any of the hazardous reagents until thoroughly trained in the procedure and shown the proper use of testing equipment.

ASTM E 50 provides additional general safety considerations on how to handle chemical reagents and use of proper protective equipment.