

Gypsum Analysis

1. **Scope:**

To provide a standardized procedure for the analysis of natural gypsum or gypsite. This method is not suitable for the analysis of by-product gypsums that contain free sulfuric acid and/or other sulfates.

2. **Principle:**

The ground sample is dissolved in dilute HCl and the sulfate precipitated as BaSO₄. The percent gypsum is calculated from the weight of BaSO₄. All analyses are reported based on the material as received and not on a dry weight basis.

3. **Safety:**

Read the SDS for all materials before use.

4. **Apparatus and Equipment:**

- 4.1. Aluminum moisture dishes (preferably with covers), 75 mm dia x 18 mm h, (VWR cat. # 25420-072 or equivalent)
- 4.2. Drying Ovens – 45°C and 105°C
- 4.3. 400 mL beakers
- 4.4. Hot plate or steam bath
- 4.5. Whatman binder free glass microfiber filter, Type (GF/F), Particle retention 0.7 µm (Whatman Cat. #1825-024 or equivalent)
- 4.6. Coors Porcelain Gooch Crucibles, 25 -40 mL capacity, with 21 – 24 mm base (Coors Cat. # 60148 and 60151 respectively, or equivalent)
- 4.7. Vacuum set up
- 4.8. Analytical balance
- 4.9. Desiccator

5. **Reagents and Supplies:**

- 5.1. Hydrochloric acid, conc.
- 5.2. 10% barium chloride (100 g BaCl₂ in 900 mL H₂O, filtered through Whatman No. 42 paper)

6. **Free Water Determination:**

- 6.1. Prepare samples as described in FEEDFERT-SP-3.
- 6.2. Mix sample thoroughly and weigh 20.00g into an aluminum dish. Dry at 45°C for a minimum of 8 hours or overnight. Keep covered and store in a desiccator to cool.
- 6.3. Re-weigh the cooled sample and calculate the weight loss as the moisture (or “free water”) in the material.

7. **BaSO₄ Determination:**

- 7.1. Weigh 0.5±0.001g of the ground sample into a 400 mL beaker.
- 7.2. Add 10 mL HCl and then 50 mL H₂O. Cover with a watch glass and gently boil for ≈10 minutes.
- 7.3. Filter boiling solution with vacuum through a glass microfiber filter in a Gooch crucible. Rinse beaker with hot water and pour the rinse onto the filter. Repeat as needed to be certain that all material is washed onto the filter.
- 7.4. Thoroughly clean beaker with a brush and rinse with DI water. Transfer filtrate back to cleaned beaker (volume should be approximately 250 mL).
- 7.5. Cover with a watch glass and bring to a boil. Add 15 mL of 10% BaCl₂. Add dropwise until solution is well-clouded. The remainder may be added more rapidly. BaSO₄ forms as the precipitate. Keep on a hot steam bath or low temperature for at least one hour. Allow to cool for several hours.
- 7.6. Dry an additional clean Gooch crucible with a glass microfiber filter at ~105°C for at least one hour. Cool in a desiccator and record the weight. Filter cooled solution with vacuum through the crucible. Wash filter at least 5 times with room temperature DI water.
- 7.7. Dry crucible and contents for 2 hours at ~105°C. Cool in a desiccator and weigh to obtain the mass of BaSO₄.

8. **Equations:**

$$\% \text{ Gypsum} = \frac{\text{mass of BaSO}_4 \times 0.7377}{\text{mass of sample}} \times 100$$

$$\% \text{ Sulfur} = \frac{\text{mass of BaSO}_4 \times 0.1374}{\text{mass of sample}} \times 100$$

$$\% \text{ Gypsum} = \frac{\% \text{ Sulfur}}{0.1862}$$

$$\% \text{ Calcium} = \% \text{ Gypsum} \times 0.2328$$

Correction for loss of moisture during grinding:

$$\% \text{ Gypsum, as received} = \frac{100 - \% \text{ moisture (unground)}}{100 - \% \text{ moisture (ground)}} \times \% \text{ Gypsum (ground)}$$

Note:

$$0.7377 = \frac{\text{Molecular weight Gypsum}}{\text{Molecular weight BaSO}_4} = \frac{172.17}{233.38}$$

$$0.1374 = \frac{\text{Molecular weight Sulfur}}{\text{Molecular weight BaSO}_4} = \frac{32.07}{233.38}$$

$$0.1863 = \frac{\text{Molecular weight Sulfur}}{\text{Molecular weight Gypsum}} = \frac{32.07}{172.17}$$

$$0.2328 = \frac{\text{Molecular weight Calcium}}{\text{Molecular weight Gypsum}} = \frac{40.08}{172.17}$$

If there is any question about the presence of sulfates other than gypsum, both total sulfate and total calcium should be determined, and the percentage of gypsum calculated from the lower component.

Results and reruns are reported per client's request.

Approvals:

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