Gypsum Analysis

1. **Scope**:

   This document provides 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. This method may be used to determine sulfate in other products such as potassium sulfate or magnesium sulfate.

2. **Principle**:

   The ground sample is dissolved in dilute HCl and the sulfate is precipitated as $BaSO_4$. The percent gypsum is calculated from the mass of $BaSO_4$. 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**:

   Equivalents are acceptable
   4.1. Aluminum moisture dishes with covers (VWR cat. # 25420-072)
   4.2. Drying Ovens – 45°C and 105°C
   4.3. 400mL 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)
   4.6. Porcelain Gooch Crucibles (Coors Cat. # 60148 or 60151)
   4.7. Vacuum set up
   4.8. Analytical balance
   4.9. Desiccator

5. **Reagents and Supplies**:

   5.1. Hydrochloric acid, conc.
   5.2. Barium chloride

6. **Free Water Determination**:

   6.1. Prepare samples as described in RA-SP-SMPL-PREP.

   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. **$BaSO_4$ Determination**:

7.1. Prepare 10% barium chloride solution by adding 100g $BaCl_2$ to a 1000mL volumetric flask. Add water to dissolve then fill to the mark. Filter through Whatman No. 42 paper.

7.2. Weigh 0.5 ±0.001g of the ground sample into a 400mL beaker.

7.3. Add 10mL HCl and then 50mL H_2O. Cover with a watch glass and gently boil for ~10 minutes.

7.4. 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.

7.5. Thoroughly clean beaker with a brush and rinse with DI water. Transfer filtrate back to cleaned beaker (volume should be approximately 250mL).

7.6. Cover with a watch glass and bring to a boil. Add 15mL of 10% $BaCl_2$ dropwise until solution is well-clouded ($BaSO_4$ forms as the precipitate). The remainder may be added more rapidly. Keep on a hot steam bath or keep warm at low temperature for at least one hour. Allow to cool for several hours.

7.7. 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.

7.8. Dry crucible and contents for 2 hours at ~105°C. Cool in a desiccator and weigh to obtain the mass of $BaSO_4$.

8. **Equations**:

$$\% \text{ Gypsum} = \frac{\text{mass of } BaSO_4 \times 0.7377}{\text{mass of sample}} \times 100$$

$$\% \text{ Sulfur} = \frac{\text{mass of } BaSO_4 \times 0.1374}{\text{mass of sample}} \times 100$$

$$\% \text{ Gypsum} = \frac{\% \text{ Sulfur}}{0.1863}$$

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

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:

$$0.7377 = \frac{\text{Molecular weight Gypsum}}{\text{Molecular weight } BaSO_4} = \frac{172.17}{233.38}$$

$$0.1374 = \frac{\text{Molecular weight Sulfur}}{\text{Molecular weight } BaSO_4} = \frac{32.07}{233.38}$$

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

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

Gypsum equivalent is calculated both using the mass of $BaSO_4$ and the percent calcium from mineral analysis and both values are reported.

Results and reruns are reported per client's request.

Approvals:

Revised By:

Stacy Aylesworth _____ 6/18/20

Stacy Aylesworth
Senior Environmental Scientist

Date

Approved By:

Maryam Khosravifard _____ 6/18/20

Maryam Khosravifard
Environmental Program Manager I

Date

Approved By:

Sarva Balachandra _____ 6/28/20

Sarva Balachandra
Quality Assurance Officer

Date

Revision Log:

Date	What was Revised? Why?
7/9/19	Added revision number and date. Revised approval page. Made minor grammatical changes
11/21/19	Added reference to sample prep SOP. Removed unnecessary steps
6/17/20	Changed reporting to using both sulfate and calcium results. Added more detail to preparing 10% barium chloride. Updated name of sample prep SOP.