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## Gravimetric Analysis

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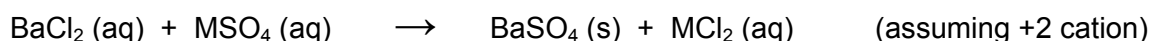
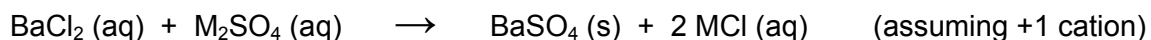
### Objectives

- To learn the techniques associated with gravimetric analysis.
- To use stoichiometry to calculate the percentage by mass of sulfate in an unknown sulfate salt.

### Background

Gravimetric analysis is a quantitative method for accurately determining the amount of a substance by selective precipitation of the substance from an aqueous solution. The precipitate is separated from the remaining aqueous solution by filtration and is then weighed. Assuming that the chemical formula for the precipitate is known and that the precipitation reaction is stoichiometric (goes to completion) the mass of the substance in the original sample can be determined.

In this experiment you will determine the percentage (by mass) of sulfate in an unknown sulfate salt by gravimetric analysis. First you will dissolve a measured mass of the unknown salt in water. Next you will add an excess of aqueous barium chloride to the aqueous solution of the unknown salt. This will result in the precipitation of the sulfate as barium sulfate.



The barium sulfate precipitate is collected by filtration, dried and weighed. The number of moles of sulfate can be determined from the mass of the barium sulfate. Since barium chloride is added in excess, and since the precipitation reaction is assumed to go to completion, the number of moles of sulfate recovered in the precipitate can be assumed to be equal to the number of moles of sulfate in the original sample allowing for the calculation of the percentage by mass of sulfate in the original sample.

In order to obtain the best results the  $\text{BaSO}_4$  crystals should be as large as possible. This facilitates filtration and washing of the crystals and the decreased surface area minimizes the amount of impurities adsorbed onto the crystals. Generally the largest crystals are obtained when the rate of precipitation is as low as possible. The rate of precipitation is minimized by slowly adding the  $\text{BaCl}_2$  solution to the aqueous mixture containing the unknown salt while continuously stirring the mixture. The rate of precipitation can be decreased still farther by slightly increasing the solubility of the  $\text{BaSO}_4$  (remember that a substance that is said to be insoluble is in fact very slightly soluble). The increase in solubility is achieved by lowering the pH with 6M HCl and by increasing the temperature. The resulting decrease in the yield of the  $\text{BaSO}_4$  is insignificant.

## Procedure

### Materials and Equipment

- Crucible and Cover
- Ashless filter paper (110 mm diameter)
- 0.1 M BaCl<sub>2</sub> solution
- 6 M HCl solution

### Safety

Be careful while handling 6 M HCl. If any HCl comes in contact with your skin or eyes you should immediately wash with water for several minutes. **You should be wearing your goggles at all times.** Make sure to allow ample time for the crucible to cool after it has been heated.

1. Measure and record the mass of a clean, dry 250 mL beaker using the analytical balance to the nearest 0.001 g. Add between 0.30 g and 0.35 g of your unknown sample to the 250 mL beaker and record the mass of the beaker plus sample to the nearest 0.001 g.
2. Add 50 mL of distilled water to the sample in the beaker. Next add 20 drops of 6 M HCl to the beaker. Stir the contents of the beaker until the sample has entirely dissolved. Leave the stirring rod in the beaker.
3. Measure 25 mL of 0.1 M BaCl<sub>2</sub> solution in a graduated cylinder. The graduated cylinder should be clean and rinsed with distilled water but does not need to be dry.
4. Heat the solution containing the sample in the 250 mL beaker until it is nearly (but not quite) boiling. Turn the burner off and **slowly** pour small portions of the BaCl<sub>2</sub> solution into the 250 mL beaker containing the sample. This step should take at least 3 minutes otherwise the BaCl<sub>2</sub> is being added to rapidly. Stir the contents of the beaker as you add the BaCl<sub>2</sub> solution. You should observe the formation of the white BaSO<sub>4</sub> precipitate. Rinse any precipitate that remains on the stirring rod into the solution with a small amount of distilled water and allow the precipitate to settle in the beaker for about 20 minutes.
5. While the precipitate settles prepare your crucible by heating it (with the cover on) in the hottest part of the Bunsen burner flame for about 2 minutes. After the crucible has cooled to room temperature record the mass of the crucible and the cover to the nearest 0.001 g using the analytical balance.
6. Obtain a piece of ashless filter paper and fold it into quarters. Open the folded paper into a cone, place it into a funnel and wet the filter paper with distilled water so that it adheres to the funnel. Place a 500 mL Erlenmeyer flask under the funnel to collect the filtrate.

7. After 20 minutes has passed slowly pour the mixture containing the  $\text{BaSO}_4$  precipitate down your stirring rod into the funnel. Be careful that the level of liquid in the funnel is never more than three-fourths of the way to the top of the filter paper. When the transfer is complete use your wash bottle (filled with distilled water) to rinse the residual precipitate from the beaker and the stirring rod into the funnel.
8. After all the liquid has drained from the funnel **very carefully** press the top edges of the filter paper together, and fold the filter paper into a compact package that will fit into the crucible. In order to avoid tearing the filter paper it is important that you do not use too much force. Place the folded filter paper into the crucible.
9. Set up your ring stand **in the fume hood** and support the crucible in a clay triangle that is attached to your ring stand. **Gently heat** the crucible **without** the cover to remove the water. After several minutes when you are sure the paper is dry, heat the crucible more vigorously so that the filter paper begins to char (turning from white, to brown, to black) but not so vigorously that the filter paper bursts into flame. If the filter paper bursts into flame you should smother it with your crucible cover and lessen the amount of heat. Continue to heat moderately until all of the filter paper has turned black.
10. Once all the filter paper has turned black heat the crucible **vigorously** with the **cover off** in the hottest part of the Bunsen burner flame so that the bottom of the crucible is **red hot**. The charred filter paper (carbon) will gradually combust and be converted into  $\text{CO}_2$  gas. When the filter paper is entirely combusted only the white  $\text{BaSO}_4$  should remain in the crucible. The crucible should be heated vigorously until there is no charred filter paper remaining.
11. Allow the crucible to cool. When the crucible has cooled to room temperature, record the mass of the crucible, the cover and its contents to the nearest 0.001g on the analytical balance.
12. Discard the  $\text{BaSO}_4$  in the proper waste container.