Objectives

- To experimentally analyze an unknown sulfate salt via a precipitation reaction, using the techniques associated with Gravimetric Analysis to collect and weigh the precipitate, and
- To calculate the percentage by mass of $\text{SO}_4^{-2}$ in the unknown sulfate salt via a stoichiometric analysis of the collected precipitate, and then use this percentage to identify the metal “M” present in the sulfate salt.

**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 goes all the way to completion, then the mass of the substance in the original sample can be determined.

In this experiment, the percentage by mass of sulfate in an unknown sulfate salt will be determined by gravimetric analysis. First, a pre-weighed sample of the unknown sulfate salt will be dissolved in water. Next, an excess of aqueous barium chloride is added to the aqueous solution of the unknown salt. This will result in the precipitation of all the sulfate ions as barium sulfate:

\[
\text{Metal sulfate (aq) + Barium chloride (aq)} \rightarrow \text{Barium sulfate (s) + Metal chloride (aq)}
\]

The barium sulfate precipitate is collected by filtration, dried and weighed. Since barium chloride is added in excess, and since the precipitation reaction goes to completion, we can assume that all of the sulfate is transferred from the original unknown sample to the precipitate. The mass of sulfate in the collected $\text{BaSO}_4$ precipitate can be calculated via its percent composition. This also yields the mass of sulfate in the original unknown since:

\[
\text{mass of sulfate in the precipitate} = \text{mass of sulfate in the unknown sample}
\]

Finally, using the mass of sulfate along with the initial mass of unknown used, the percentage by mass of sulfate in the original sample may now be calculated.

In order to obtain the best results, the collected $\text{BaSO}_4$ crystals should be as large as possible. This considerably aids the filtration process (larger crystals are less likely to be pass through the filter paper), and it also minimizes the amount of impurities adsorbed onto the crystals (smaller surface area). In general, larger 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 even further by slightly increasing the solubility of the $\text{BaSO}_4$. This may be achieved by lowering the pH with 6 M $\text{HCl}$ and by increasing the temperature. The resulting decrease in the yield of the $\text{BaSO}_4$ is insignificant.

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**Procedure**

**Chemicals**

Unknown solid sulfate salt, 6 M $\text{HCl}$ solution, and 0.1 M $\text{BaCl}_2$ solution
Equipment

250-mL beaker, analytical balance, stirring rod, 100-mL graduated cylinder, stand with ring clamp, wire screen, Bunsen burner, wash bottle with distilled water, crucible and lid, crucible tongs, ash-less filter paper, large funnel, 500-mL Erlenmeyer flask, clay triangle

Safety

Be very careful when handling 6 M \(\ce{HCl}) (aq). If this acid comes in contact with your skin or eyes you should immediately rinse the affected area with water for several minutes. Also remember that items heated in the Bunsen burner are very hot (especially the crucible), and to allow ample time for them to cool before touching.

Instructions

1. Weigh a clean, dry 250-mL beaker to the nearest 0.001 g using the analytical balance, and record this mass on your lab report. Next, add 0.30 – 0.35 grams of your unknown sample to the beaker. Record the combined mass of the beaker plus sample on your lab report.

2. Add 50 mL of distilled water, followed by 20 drops of 6 M \(\ce{HCl}) (aq), to the sample in the beaker. Stir the contents of the beaker until the sample has entirely dissolved. Leave the stirring rod in the beaker.

3. Obtain a stand with a ring clamp from the back of the lab. Place your wire screen on the ring and the beaker containing your dissolved sample on the wire screen. Use the Bunsen burner to heat the solution until it is nearly (but not quite) boiling. Turn the Bunsen burner off before the solution boils.

4. While heating the solution, measure out 25 mL of 0.1 M \(\ce{BaCl2}) (aq) using a 100-mL graduated cylinder. The graduated cylinder used should be clean (rinse with distilled water) but does not need to be dry.

5. Slowly add small portions of \(\ce{BaCl2}) (aq) to the beaker containing the hot solution. You should observe the formation of a white precipitate of \(\ce{BaSO4}) (s). Stir the contents of the beaker as you add the \(\ce{BaCl2}) solution. The addition of the \(\ce{BaCl2}) must be performed very slowly – this step should take you at least 3 minutes to complete! When finished, rinse any precipitate that remains on the stirring rod into the solution with a small amount of distilled water, and then allow the precipitate to settle in the beaker for about 20 minutes.

6. While the precipitate settles, prepare your crucible by heating it in the hottest part of the Bunsen burner flame for about 2 minutes (use the crucible tongs, cradling the crucible as demonstrated by the instructor – never pick up a crucible by pinching the walls). Repeat with the lid. Place the hot crucible and lid on the metal base of the stand to cool. Once they have cooled to room temperature weigh the crucible without the lid using the analytical balance, and record this mass on your lab report. You do not need to weigh the lid.

7. Obtain a piece of ash-less filter paper from your instructor and fold it into quarters. Open the folded paper into a cone and place it into your large funnel. Wet the filter paper with a small amount of distilled water so that it adheres to the funnel. Sit the funnel in the mouth of a 500-mL Erlenmeyer flask, which will be used to collect the filtrate.

8. After 20 minutes has passed, slowly pour the mixture containing the \(\ce{BaSO4}) 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.

9. After all the liquid has drained from the funnel, wearing gloves very carefully press the top edges of the filter paper together, and gently fold the filter paper into a compact package that will fit into the crucible. It is important that you do not use too much force in order to avoid tearing the filter paper. Place the folded filter paper into the crucible.
10. Take your stand, ring clamp and Bunsen burner over to a fume hood. Place your clay triangle on the ring and the crucible in the clay triangle for support. *Gently heat the crucible without the lid to remove the water.* Once the paper appears to be dry (after several minutes), *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 cover it with the crucible lid to put out the flame, then reduce the amount of heat and remove the lid. Continue to heat moderately with the lid off until all of the filter paper has turned black.

11. Once all the filter paper has turned black, *vigorously heat the crucible without the lid* in the hottest part of the Bunsen burner flame so that the bottom of the crucible is red hot. The center of the flame should now be directly on the crucible. The charred filter paper (carbon) will gradually combust and be converted into \(\ce{CO2}\) gas. When the filter paper is entirely combusted only the white \(\ce{BaSO4}\) should remain in the crucible. Continue to heat the crucible vigorously until no charred filter paper remains. This should take 5 - 10 minutes.

12. Allow the crucible to cool to room temperature (this takes at least 5 minutes). Weigh the crucible *without the lid* and its contents on the analytical balance. Record this mass.

13. Place the crucible and its contents back in the clay triangle and heat vigorously with the lid off for an additional 5 minutes. Then allow it to cool again and reweigh the crucible and its contents *without the lid*. If the mass is within 0.005 grams of that in step 12, then record this mass on your lab report. If the mass has decreased by more than 0.005 grams, then either the \(\ce{BaSO4}\) is still wet or not all of the filter paper has combusted and you should repeat this step until you achieve a consistent mass, making sure the crucible is in the hottest part of the flame.

14. Discard the \(\ce{BaSO4}\) in the proper waste container, then clean up as directed by your instructor.
Pre-laboratory Assignment: Gravimetric Analysis

Suppose that 0.323 g of an unknown sulfate salt is dissolved in 50 mL of water. The solution is acidified with 6 M \( \ce{HCl} \), heated, and an excess of aqueous \( \ce{BaCl_2} \) is slowly added to the mixture resulting in the formation of a white precipitate.

1. Assuming that 0.433 g of precipitate is recovered calculate the percent by mass of \( \ce{SO_4^{2-}} \) in the unknown salt.

2. If it is assumed that the salt is an alkali sulfate determine the identity of the alkali cation.
Lab Report: Gravimetric Analysis of an Unknown Sulfate

Experimental Data

Unknown Sulfate ID Code:

- Mass of empty 250-mL beaker
- Mass of 250-mL beaker and unknown sulfate
- Mass of unknown sulfate
- Mass of empty crucible (without lid)
- Mass of crucible (without lid) and barium sulfate
- Mass of barium sulfate

Calculations and Conclusions

1. Calculate the mass of sulfate in the unknown sample. Clearly show each step of your calculation.

2. Calculate the percentage by mass of sulfate in the unknown sample. Show your work.

3. Calculate the percentage by mass of the metal in the unknown sample. Show your work.

4. The cation in your unknown metal sulfate is one of the following:
   - $\text{Al}^{3+}$
   - $\text{Na}^+$
   - $\text{Ni}^{2+}$
   - $\text{K}^+$
   - $\text{NH}^{4+}$
   - $\text{Cd}^{2+}$

Use this information along with your experimental results to determine which cation it is.

- Unknown Sulfate ID Code:
- Identity of Cation:

Show all of your work with clear, logical steps below. Clearly explain how your calculations here along with your
experimental results for #2 and/or #3 allowed you to identify the cation in the metal sulfate.

Questions

1. Suppose an unknown metal sulfate is found to be 72.07% \(\text{SO}_4^{2-}\). Assuming the charge on the metal cation is +3, determine the identity of the cation.

2. The unknown metal sulfates are hygroscopic and will absorb water from air. The unknowns must thus be kept in desiccators to remove any absorbed water. How would your results be affected if your unknown sample was not desiccated? Would this error cause your calculation of the mass percent of sulfate in the unknown to be too high or too low? Explain.

3. In this experiment you used an excess of the \(\text{BaCl}_2\) solution. How would your results be affected if you did not use an excess of the \(\text{BaCl}_2\) solution? Would this error cause your calculation of the mass percent of sulfate in the unknown to be too high or too low? Explain.

4. In the last step of the procedure, you vigorously heated the \(\text{BaSO}_4\) precipitate wrapped in filter paper in a crucible. How would your results be affected if tiny pieces of the filter paper still remained mixed in with the \(\text{BaSO}_4\) after heating? Would this error cause your calculation of the mass percent of sulfate in the unknown to be too high or too low? Explain.