Adapted from the "Gravimetric Analysis" experiment in Chem 11 at Santa Monica College.

Purpose

To determine the identity of an unknown sulfate salt using a precipitation reaction and gravimetric analysis.

Learning Objectives

Students perform a precipitation reaction and quantitatively collect the precipitate.

Students demonstrate the use of a Bunsen burner with correct technique and in a safe manner.

Students implement correct technique to handle a crucible.

Students calculate the mass % of sulfate ion in an unknown salt.

Students identify the metal cation of their unknown salt using their experimental results and stoichiometry.

Students analyze error within experimentation.

Equipment

- 250-mL beaker
- Spatula
- Stirring rod
- 100-mL graduated cylinder
- 10-mL graduated cylinder
- Pasteur pipet and bulb
- Ring stand
- Wire gauze
- Bunsen burner and striker
- Crucible and lid
- Crucible tongs
- Ashless filter paper
- Clay triangle
- Funnel
- 250-mL Erlenmeyer flask

Chemicals

- Unknown metal sulfate
- 2 M HCl solution
- 0.1 M $BaCl_2$ solution

Introduction

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:

$$\text{Metal sulfate (aq)} + \text{Barium chloride (aq)} \longrightarrow \text{Barium sulfate (s)} + \text{Metal chloride (aq)}$$

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 $BaSO_4$ 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 $BaSO_4$ 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 $BaCl_2$ 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 $BaSO_4$. This may be achieved by lowering the pH with 2 M HCl and by increasing the temperature. The resulting decrease in the yield of the $BaSO_4$ is insignificant.

Lab Technique: Using a Bunsen Burner

A Bunsen burner is a piece of equipment used for the general purpose of heating substances. When connected to a gas line and lit with either a striker or a match, Bunsen burners generate a controllable, open flame that is hot enough for most experimental procedures. The flame produced consists of a visible outer cone and inner cone, in which the topmost tip of the inner cone is the hottest part of the flame, shown in Figure GA.1 and Figure GA.2. In case of emergency, completely shutting off the gas line is the best way to stop the flame. **Make sure the gas line is completely off at all times unless the Bunsen burner is in use.** [a]

[a]Description adapted from Laboratory Manual from Los Medanos College found at click here

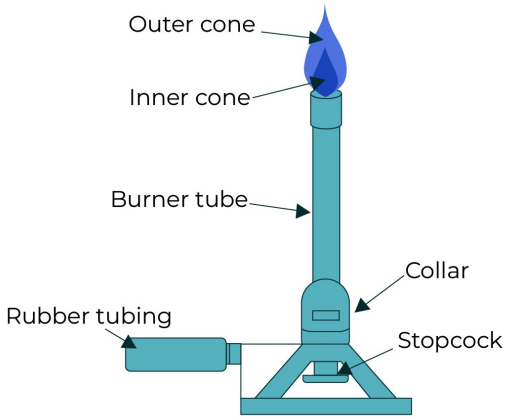

Figure GA.1: Diagram of the parts of a Bunsen burner.

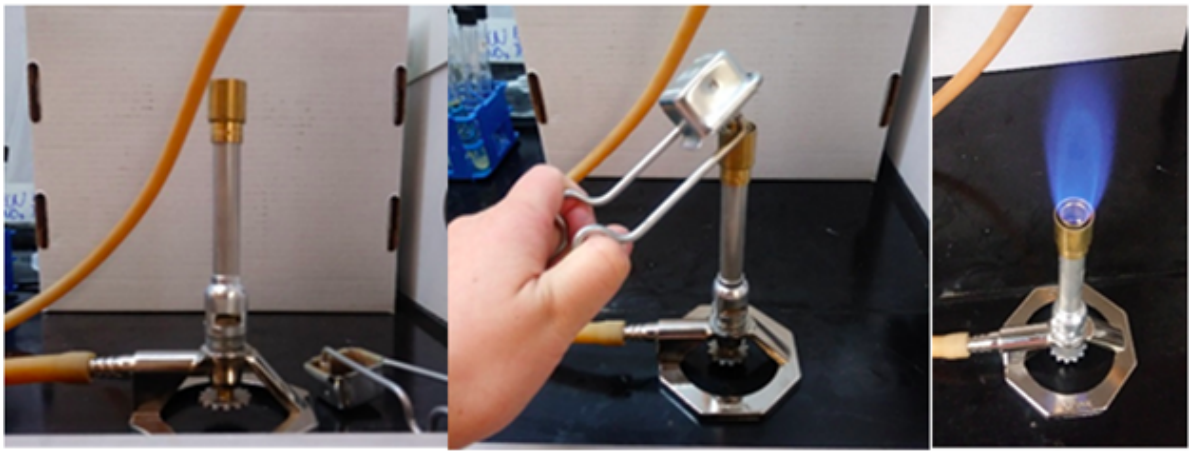

Figure GA.2: Pictures of the set up, lighting of, and a lit Bunsen burner.

To use a Bunsen burner, follow these steps:

1. Check the equipment for broken parts before use.
2. Inspect the rubber gas tubing for cracks by stretching it and looking for any major cracks throughout the tubing. If the tubing has cracks and leaks, discard it and get a new tubing.
3. Attach one end of the rubber tubing to the arm of the Bunsen burner and the other end to the gas line located on your lab bench.
4. Once the Bunsen burner and rubber tubing is secure, turn on the gas line. Quickly move onto step 5 to ensure flammable gas does not build up in the room.

5. If using a striker, squeeze the handle of the striker over the top of the Bunsen burner to generate a spark where gas is emitted. Do this until a flame appears. Adjust the size of the flame using the gas line knob or the stopcock as needed. If using matches, strike the head of the match against the striking strip on the back of the pack. Hold the flame up to the top of the Bunsen burner where gas is emitted until a flame appears. Adjust the size of the flame using the gas line knob or the stopcock as needed.

Precautions and tips for using a Bunsen burner:

- The gas lines located in the lab benches and in the fume hood have a gas pressure of 4 pounds per square inch (psi). For reference, keep in mind that the pressure needed to blow up a balloon is 15 psi (atmospheric pressure). The pressure of the gas lines is not a dangerous amount, but always be aware of its use. When the gas line is on, you will normally hear a slight hissing sound. Natural gas has no smell, but a trace amount of a sulfur compound is added so that it does have odor. You might begin to smell it from unburned gas. To avoid leaks, you should not have the Bunsen burner or the gas line on when not in use.
- Bunsen burners are generally safe to use as long as they are used properly and with common sense. For example, the Bunsen burner should be kept away from the edge of the table or anywhere it could easily be knocked over. Do not reach over the Bunsen burner, even if it is off. Be cautious when heating glass containers. If the container is too cold (from either a reaction, ice bath, or other freezing procedure), applying direct heat to the glass may cause it to crack or break.
- There are several features on the Bunsen burner meant for controlling the size of the flame, but generally, the most effective way is by controlling gas valve located on your lab bench or in the fume hood. The gas valve controls the amount of gas entering the Bunsen burner. Allowing more gas to enter the Bunsen burner creates a larger flame. Twisting the collar clockwise will reduce the amount of air entering the Bunsen burner, cutting off oxygen supply to the flame. The flame can also be adjusted by using the stopcock located at the bottom of the Bunsen burner, which adjusts the flow of gas entering the Bunsen burner.
- **Never leave an unattended flame.**
- Check the area around your Bunsen burner for flammable objects (paper towels, flammable solvents, etc.) prior to lighting the flame.
- Hot objects look just like cold objects. Wait sufficient time for objects to cool before handling them.
- Metals can conduct heat very well. Be aware that this may happen when heating objects with clamps.
- Hair and clothing should be tied down to avoid contact with any flame.
- Wear gloves when handling all chemicals in this experiment.

Procedure:

Safety Precautions

- Follow all PPE guidelines.
- Absolutely NO substances found in a chemistry lab should be ingested.
- Be very careful when handling 2 M 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. Let your AI know in the case of any exposure to acid on your skin.
- If any 2 M HCl (aq) is spilled (even a drop), let your AI know so it can be appropriately neutralized and cleaned up.
- Items heated in the Bunsen burner are very hot (especially the crucible), and to allow ample time for them to cool before touching.
- Any items that are too hot to touch should not be placed on the lab bench. Either leave them where they were heated or on the base of the ring stand.
- Only hold the crucible cradled in the crucible tongs, never pinching the side of the container.
- Collected precipitate should be disposed of in the solid waste container. Filtrate should be disposed of in the aqueous waste container.
- Never leave a flame unattended.
- Turn gas off while Bunsen burner is not in use.

Each lab group (2–3 students as directed by your AI) will work independently. You may NOT share data with other lab groups.

Precipitation Reaction with Unknown Metal Sulfate and Barium Chloride

1. **Data Table:** Create a data table on the notebook pages at the end of this procedure that includes the following: Unknown Number, Mass of 250-mL beaker (g), Mass of 250-mL beaker and unknown (g), Mass of Unknown (g). Fill in this data table as you complete this experiment

2. Obtain your sample of unknown metal sulfate and write down the unknown number in your lab notebook. Failing to provide your unknown number in your post lab report will result in a 5-point penalty.

3. Weigh a clean, dry 250-mL beaker and record this mass in your lab notebook.

4. Add 0.30 g of your unknown sample to the beaker. Record the combined mass of the beaker plus sample.

5. Measure 3 mL of 2 M HCl (aq) in a 10-mL graduated cylinder, transferring the acid with a Pasteur pipet.

6. Add 50 mL of distilled water, followed by 3 mL of 2 M 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.**

7. Place a wire gauze on the ring portion of a ring clamp and place the beaker containing your dissolved sample on the wire gauze as shown in Figure GA.3. 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. Safety: The beaker should remain on the wire gauze until completely cooled.

8. While heating the solution, one partner should monitor the Bunsen burner and the other should measure out 25 mL of 0.1 M $BaCl_2$ using a graduated cylinder. The graduated cylinder used should be clean (rinse with distilled water) but does not need to be dry.

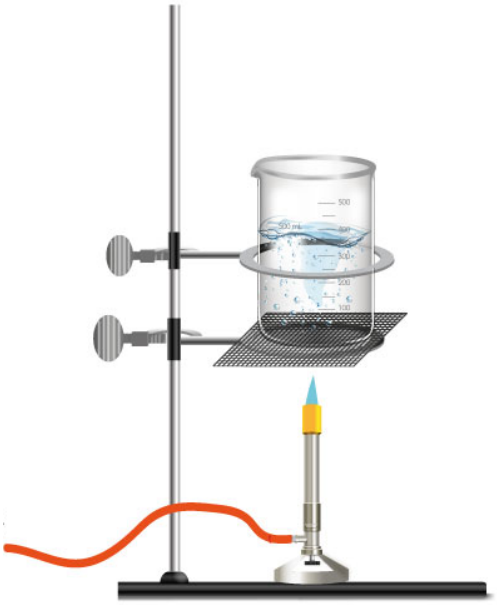

Figure GA.3: Appropriate setup to heat a beaker with a Bunsen burner.

9. Slowly add the 25 mL of 0.1 M $BaCl_2$ in small portions to the beaker containing the hot solution. You should observe the formation of a white precipitate of $BaSO_4$. Stir the contents of the beaker as you add the $BaCl_2$ solution. The addition of the $BaCl_2$ 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.

Collecting and Drying Precipitate

1. Data Table: Create a data table on the notebook pages at the end of this procedure that includes the following: Mass of crucible (g), Mass of crucible and dried $BaSO_4$ precipitate (g), and Mass % of $BaSO_4$ precipitate (g). Fill in this data table as you complete this experiment.

2. 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 and shown in Figure GA.4–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 and record this mass. You do not need to weigh the lid.

Figure GA.4: How to hold a crucible in crucible tongs.

> **Lab Technique of the Day: Assembly and use of a Bunsen burner.** Each lab partner must show your teaching team member the technique used for putting the pieces of a Bunsen burner together, light the burner, adjust the flame, and turn the burner off.

3. Obtain a piece of ash-less filter paper fold it into quarters. Open the folded paper into a cone as shown in Figure GA.5 and place it into your large funnel. Wet the filter paper with a few drops of distilled water so that it adheres to the funnel. Sit the funnel in the mouth of a 250-mL Erlenmeyer flask, which will be used to collect the filtrate.

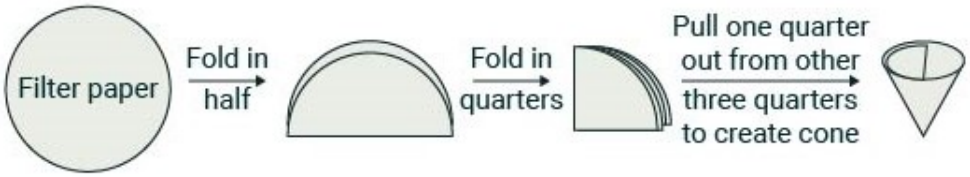

Figure GA.5: How to fold filter paper to fit into a funnel.

4. After 20 minutes has passed for your precipitate to settle, slowly pour the mixture containing the $BaSO_4$ 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 the distilled water wash bottle to rinse the residual precipitate from the beaker and the stirring rod into the funnel. Use the smallest amount of water possible while also adequately washing the beaker.

5. After all the liquid has drained from the funnel, while 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.

6. 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 as shown in Figure GA.6. 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.

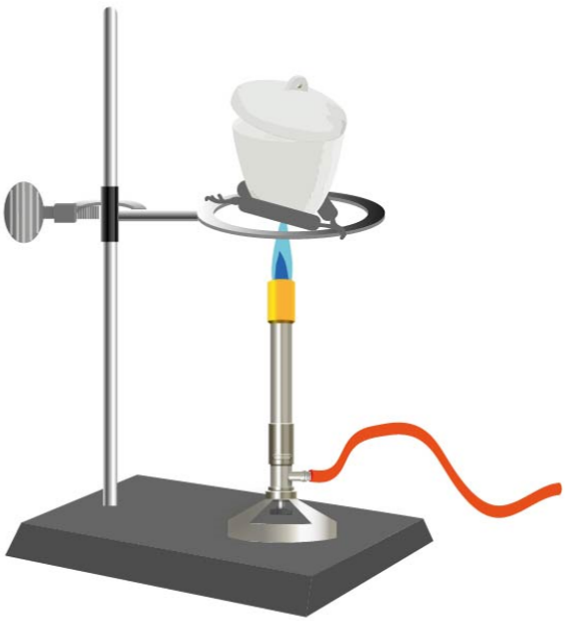

Figure GA.6: Appropriate set up for heating a crucible with a Bunsen burner.

7. 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 CO_2 gas. When the filter paper is entirely combusted only the white $BaSO_4$ should remain in the crucible. Continue to heat the crucible vigorously until no charred filter paper remains.

8. 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.

9. 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 7, then record this mass on your lab report. If the mass has decreased by more that 0.005 grams, then either the $BaSO_4$ 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.

Cleanup

- Barium sulfate should be discarded into the solid waste container.
- Filtrate solution should be discarded into the aqueous waste container.
- Ensure all glassware has been cleaned and returned to appropriate location before leaving.
- The crucible, wire gauze, and clay triangle are not disposable; please make sure it is cleaned and returned to the fume hood with the equipment.
- Wipe down your workspace (both bench and fume hood).
- Enter your data into the Data Entry portion of Labflow (both lab partners).
- Check out with your AI.
    - Show your clean workspace.
    - Show your completed lab notebook with all of your data.
    - Double check that your data is entered correctly into Labflow and have your AI scan your QR code.
- Log out of computers.