Lab 2: Chemical Composition of Compounds

Part I. Identification Based on Percent Metal Compositions

Part II. Determination of Percent $\text{KClO}_3$ in a $\text{KCl/KClO}_3$ Mixture

Procedure Overview

- Inspect and clean the crucibles
- Assemble the setup: ring stand, iron ring, clay triangle, and Bunsen burner
- Heat the crucible and cover red hot, let cool, weigh the cooled crucible and cover, and record the mass
- Add a strip of Mg to the crucible, weigh the crucible containing the Mg, along with the crucible cover, and record the mass
- Place the covered crucible in the clay triangle, heat gently
- After the reaction is complete, let setup cool, weigh crucible containing the reaction product, along with the cover, and record the mass
- Using the remaining crucibles, repeat the above steps, beginning with heating the crucible red hot, for the other two samples: a copper(II) carbonate hydroxide compound and the $\text{KClO}_3$/KCl mixture

Figure 2-1. The experimental setup.
Materials for Chemical Composition of Compounds

To be checked out from the stockroom:
None

From your lab drawer:
3 crucibles with covers
Spatula

Provided in the lab:
Magnesium ribbon
Copper(II) carbonate hydroxide (solid)
Potassium chlorate (KClO₃)/Potassium chloride (KCl) mixture
Bunsen burner
Matches
Ring stand
Iron ring
Tongs
Clay triangle

Procedure

Part I. Identification Based on Percent Metal Composition

A. Magnesium

1. Inspect the crucible for cracks. If your crucible is cracked, exchange it for a new one at the stockroom. Clean the crucible and cover thoroughly. The crucible may be stained, so you will not be able to get it completely white. After cleaning, be sure to handle it ONLY with tongs.

2. Place a clay triangle on an iron ring clamped to a ring stand. Using tongs, transfer the crucible and its cover to the clay triangle and begin heating them with a Bunsen burner. (See Figure 2-1.) Adjust the burner to obtain a blue flame having an inner cone. The tip of the inner cone is the hottest part of the flame; use this part of the flame to heat the crucible red hot.

3. After heating the crucible, cool it in place on the ring stand for ~10–15 minutes. DO NOT TOUCH THE CRUCIBLE!

4. Weigh the crucible and cover to the nearest milligram and record the mass. Place a magnesium sample that is ~10 cm long into the crucible and weigh the crucible, cover, and sample together. Record this mass.

5. Cover the crucible with its cover and place it inside the clay triangle supported by a ring and ring stand. Heat gently to avoid losing sample by overflowing the crucible. After 10 minutes, using tongs to lift the lid slightly, check to make sure that most of the metal has reacted. If the metal is burning, replace the lid and continue heating gently until flames are no longer visible.

6. Remove the cover and heat the crucible with the hottest part of the flame for 5 minutes to complete combustion of the sample.
7. When the crucible and its contents have cooled (10–15 minutes), weigh them (with the cover) to the nearest milligram, and record this mass. Always use the same balance you used for the previous weighings.

8. Dispose of the magnesium product and any extra magnesium in the container designated for waste disposal. Clean the crucible and cover.

B. Copper in Copper(II) Carbonate Hydroxide

1. Repeat steps 1–3 from Part I.A. with the second crucible.

2. Weigh the crucible and cover to the nearest milligram (e.g., 0.001 g) and record the mass.

3. With the crucible and cover on the balance, press tare (balance to read 0.000 g). Weigh ~1 gram of the copper(II) carbonate hydroxide into the crucible. There is no need to record this mass.

4. Remove the crucible with sample from the balance. Press tare (balance to read 0.000 g). Weigh the crucible and cover with sample and record the mass.

5. Gently heat with the cover partially covering the crucible (see Figure 2-1) over a low flame for ~5 minutes. As the sample is heated, carbon dioxide gas and water vapor will be generated from the decomposition of copper(II) carbonate hydroxide. The incomplete covering of the crucible facilitates the escape of the gases.

6. After 5 minutes, increase the heating intensity and continue heating until the entire sample is converted to black CuO. After the last traces of green copper(II) carbonate hydroxide have disappeared, continue to heat the sample for at least two more minutes to ensure complete decomposition. Allow the crucible to cool to room temperature. DO NOT TOUCH THE CRUCIBLE!

7. Weigh the cooled crucible and cover with the CuO product. After recording the mass, discard the CuO, and any unreacted copper(II) carbonate hydroxide, in the waste jar provided. Clean the crucible and cover.

Part II. Determination of Percent KClO₃ in a KClO₃/KCl Mixture

1. Repeat steps 1–3 from Part I.A. with the third crucible.

2. Weigh the crucible and cover to the nearest milligram and record the mass.

3. With the crucible and cover on the balance, press tare (balance to read 0.000 g). Weigh out ~1 gram of the KClO₃/KCl mixture into the crucible. There is no need to record this mass.

4. Record the sample number of the KClO₃/KCl mixture. If you make a mistake, discard any unreacted KClO₃/KCl mixture in the waste jar provided.

5. Remove the crucible with the sample and cover from the balance. Press tare (the balance to read 0.000 g). Place the crucible with sample and cover on the balance and record the mass.

6. Gently heat with the cover partially covering the crucible (see Figure 2-1) over a low flame for ~5 minutes. As the sample is heated, the mixture will melt and oxygen (O₂) will be generated from the decomposition of KClO₃. As oxygen is generated, the molten mixture will bubble and some of the potassium chloride will spatter onto the crucible cover. Keep the crucible partially
covered during this step to prevent loss of potassium chloride. The incomplete covering facilitates the escape of oxygen gas.

7. After 5 minutes, increase the heating intensity. Continue heating for an additional 10 minutes. Allow the crucible to cool to room temperature.

8. Weigh the cooled crucible with the product and cover. After recording the mass, rinse the crucible with water while scrubbing to remove the potassium chloride. Rinse the potassium chloride down the drain. Be sure to also rinse and scrub the underside of the crucible cover.

9. Check that all reagent bottles are tightly capped when you are finished with them.

Waste Disposal
Place all metal oxide and any unreacted copper(II) carbonate hydroxide, magnesium ribbon, or potassium chlorate/potassium chloride mixture in the waste jar provided. Do not put water, paper, matches, or any other solids in the waste containers.

Before You Leave the Lab
1. Upon completing the work (includes all requested calculations), show your work to your TA.
2. Clean your lab bench and have your TA check your equipment drawer, lab bench, and lab notebook.
3. Obtain your TA’s signature in your lab notebook and turn in the carbon copies of your lab notebook pages associated with this experiment.

Information to Enter in your Notebook during the Lab
- Mass of empty crucible and cover
- Mass of crucible, cover, and Mg
- Mass of Mg
- Mass of crucible, cover, and product
- Mass of product
- Calculate the % Mg in your product
- Measured % Mg values reported by the rest of your quiz section
- Mass of empty crucible and cover
- Mass of crucible, cover, and sample
- Mass of sample
- Mass of crucible, cover, and product
- Mass of product
- Calculate the % Cu in your product
- Measured % Mg values reported by the rest of your quiz section
- Sample number of your unknown mixture
- Mass of empty crucible and cover
- Mass of crucible, cover, and KCl/KClO₃ mixture
• Mass of KCl/KClO₃ mixture
• Mass of crucible, cover, and product
• Mass lost upon heating
• Calculation of % KClO₃ in the mixture

• Any other notes or observations that will help you remember what happened during the experiment (helpful when you work on your post-lab report or need to explain your results)