Gravimetric Analysis of a Metal Carbonate

Introduction

How do chemists determine the identity of a compound? A large variety of analytical techniques and procedures, ranging from instrumental methods such as spectroscopy and chromatography to more classical processes, such as qualitative and gravimetric analyses, have been created to accomplish that task. In this laboratory, the identity of a Group 1 metal carbonate is determined gravimetrically using a double-replacement precipitation reaction.

Concepts

- Double-replacement reaction
- Gravimetric analysis

Background

In this experiment, an unknown Group 1 metal carbonate, M_2CO_3, is analyzed to determine the identity of the Group 1 metal, M.

A known amount of the soluble unknown carbonate is dissolved in water to dissociate the compound into its ions (Equation 1).

$$M_2CO_3(s) \rightarrow 2M^+(aq) + CO_3^{2-}(aq) \qquad \textit{Equation 1}$$

When a solution of calcium chloride, $CaCl_2$, is added to this metal carbonate solution, a precipitate of calcium carbonate forms (Equation 2).

$$Ca^{2+}(aq) + CO_3^{2-}(aq) \rightarrow CaCO_3(s) \qquad \textit{Equation 2}$$

The overall reaction represents a double-replacement reaction with a precipitate formed (Equation 3).

$$CaCl_2(aq) + M_2CO_3(aq) \rightarrow CaCO_3(s) + 2MCl(aq) \qquad \textit{Equation 3}$$

The precipitated calcium carbonate is then filtered, dried, and weighed. The moles of calcium carbonate, $CaCO_3$, are equal to the moles of Group 1 metal carbonate, M_2CO_3, added to the original solution. Dividing the mass of the unknown carbonate by the moles of calcium carbonate yields the formula weight, and thus the identity, of the Group 1 metal carbonate.

Experiment Overview

The purpose of this lab is to determine the identity of a Group 1 metal carbonate compound by gravimetric analysis. The unknown is weighed and dissolved in water. A solution of calcium chloride is added to the metal carbonate solution to precipitate the carbonate ions as calcium carbonate. The precipitate is filtered, dried and weighed. From the data, the formula weight and identity of the unknown metal carbonate is determined.

Pre-Lab Questions *(Use a separate sheet of paper to answer the following questions.)*

An unknown metal carbonate was analyzed gravimetrically and yielded the following data.

Mass of crucible + M_2CO_3	12.627 g
Mass of crucible	10.655 g
Mass of M_2CO_3	1.972 g
Mass of filter paper	0.598 g
Mass of filter paper + $CaCO_3$	2.436 g
Mass of $CaCO_3$	1.838 g
Moles of $CaCO_3$	
Molar mass of M_2CO_3	
Identity of M_2CO_3	
Percent error	

1. From the mass of $CaCO_3$, calculate the moles of $CaCO_3$ precipitated.
2. Calculate the molar mass of the unknown.
3. Calculate the molar mass of the following Group 1 metal carbonates:
   a. Li_2CO_3
   b. Na_2CO_3
   c. K_2CO_3
4. What is the identity of M_2CO_3?
5. Calculate the percent error in the molar mass determination of M_2CO_3 by comparing the experimentally determined molar mass of M_2CO_3 to the known molar mass of the appropriate metal carbonate.

Materials

Chemicals

Calcium chloride solution, $CaCl_2$, 0.2 M, 125 mL

Unknown carbonate sample, M_2CO_3, 2 g

Water, distilled or deionized, 200 mL

Equipment

Analytical balance, 0.001-g or 0.0001-g precision

Beakers, 400-mL, 2

Bunsen burner

Crucible, 15-mL

Crucible tongs

Drying oven

Filter funnel

Filter paper, quantitative

Glass stirring rods, 2

Graduated cylinder, 250-mL

Heat-resistant pad

Ring stand and iron ring

Spatula

Triangle, clay, pipe stem

Wash bottle

Watch glass

Safety Precautions

The unknown solids are slightly toxic by ingestion and are skin irritants. Handle the crucible only with tongs. Do not touch the crucible with fingers or hands. There is a significant burn hazard associated with handling a crucible—remember that a hot crucible looks like a cold one. Wear chemical splash goggles and chemical-resistant gloves and apron. Wash hands thoroughly with soap and water before leaving the laboratory.

Procedure

1. Obtain a clean, dry 15-mL crucible.
2. Set up a Bunsen burner on a ring stand beneath a ring clamp holding a clay pipe stem triangle. Place the crucible in the clay triangle (see Figure 1). Do NOT light the Bunsen burner.
3. Light the Bunsen burner and brush the bottom of the crucible with the burner flame for about one minute. Turn off the Bunsen burner and allow the crucible to cool.
4. Using crucible tongs to handle the crucible, measure the mass of the clean, empty crucible to the nearest 0.001 g. Record the mass in the Data Table.
5. While the crucible is still on the balance, add approximately 2 g of the unknown carbonate to the crucible. Record the combined mass of the crucible and unknown carbonate in the Data Table.

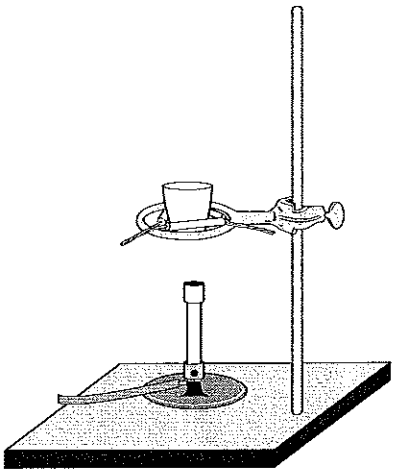

Figure 1.

6. Place the crucible on the clay triangle as shown in Figure 2. Light the Bunsen burner again and slowly heat the crucible by brushing the bottom of the crucible with the Bunsen burner flame for 2–3 minutes. Set the crucible to cool on a heat-resistant pad.
7. Weigh the crucible on an analytical balance. Record the mass in the Data Table.
8. Repeat steps 6 and 7 until the mass of the crucible and unknown carbonate no longer decreases. *Note:* The Group 1 metal carbonates are hydroscopic—they absorb water from the air. These heating steps are necessary to ensure the crucible is dry and the carbonate samples are anhydrous when massed.

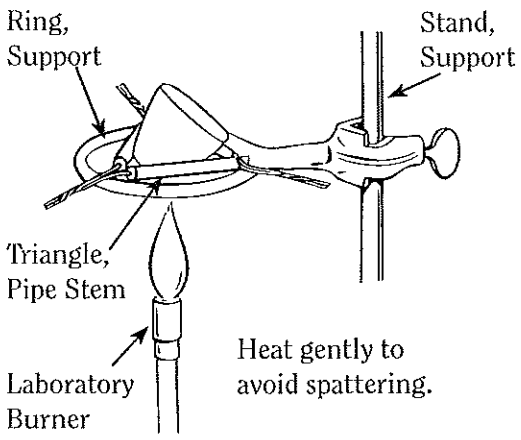

Figure 2.

9. Add the crucible contents to a 400-mL beaker.
10. Add about 200 mL of distilled or deionized water to the beaker and stir to dissolve the unknown carbonate.
11. Add about 125 mL of the 0.2 M $CaCl_2$ solution to the 400-mL beaker and stir.
12. Let the precipitate settle (5 minutes).
13. Obtain a piece of quantitative filter paper. Weigh the filter paper on the analytical balance. Record the mass of the filter paper in the Data Table.

14. Fold the filter paper into a cone. First fold the filter paper in half and crease. Next, fold the filter paper almost in half again, leaving about a 5° angle between the folded edges (see Figure 3).
15. Tear off the corner of the top edge, open the filter paper into a cone shape, and place the torn corner in the bottom of the cone.

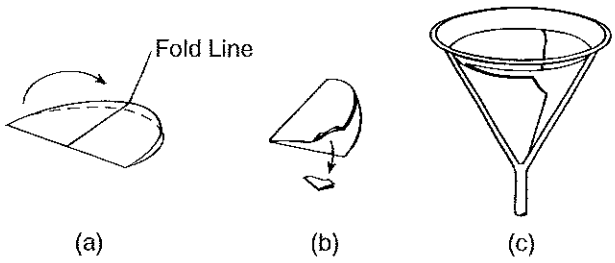

Figure 3.

16. Place the cone into the filter funnel. Position the paper tight against the funnel walls and moisten the paper with about 5 mL of deionized water from a wash bottle. *Note:* After adding the water, use index fingers to seat the filter paper tightly against the sides of the funnel so that little, if any, air gaps are visible in the stem as the water filters through.
17. Set up the ring stand and iron ring and place the funnel in the ring. Let the funnel drain into a second 400-mL beaker (see Figure 4).
18. Using a stirring rod, decant the liquid from the 400-mL beaker into the funnel. Be sure to keep the liquid level below the top of the filter paper cone (Figure 5).
19. When all but approximately 10 mL of the liquid has been transferred, swirl the beaker to suspend the precipitated $CaCO_3$. Transfer this to the funnel, again making sure not to fill the cone above the top of the filter paper.
20. Rinse the flask with small amounts of distilled or deionized water from the wash bottle and then transfer the washings to the filter.
21. When all the solid has been transferred to the filter paper, rinse the solid with three small portions of distilled or deionized water. Allow the funnel to drain completely.
22. Obtain a watch glass. Using a microspatula, take the filter paper out of the funnel and place it in the center of the watch glass. Be careful not to tear the paper or to lose any part of the solid.
23. Using the microspatula, carefully open the filter paper into a circle on the watch glass. Place the watch glass and filter paper in a drying oven set at 110–120 °C.
24. Allow the filter paper to dry for 10–15 minutes. Remove the watch glass from the oven using crucible tongs. Use the spatula to break up the $CaCO_3$ into small particles.

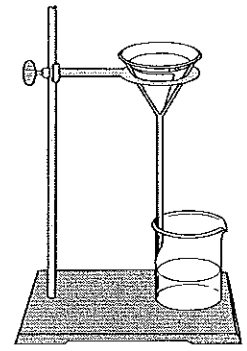

Figure 4.

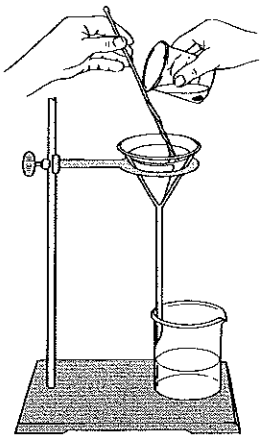

Figure 5.

25. Return the watch glass to the drying oven for an additional 5 minutes.
26. Remove the watch glass from the oven and set it aside to cool.
27. When cool, weigh the filter paper and the solid $CaCO_3$ on an analytical balance. Record the mass in the Data Table.
28. Repeat steps 25–27, until the mass readings do not change by more than 0.005 g.

Disposal and Cleanup

Your teacher will provide disposal and cleanup instructions.

Data Table

Mass of crucible	g
Mass of crucible + M_2CO_3	g
Mass of crucible + M_2CO_3 (dried) (1st weighing)	g
Mass of crucible + M_2CO_3 (dried) (2nd weighing)	g
Mass of M_2CO_3	g
Mass of filter paper	g
Mass of filter paper + $CaCO_3$	g
Mass of filter paper + $CaCO_3$ (2nd weighing)	g
Mass of $CaCO_3$	g
Moles of $CaCO_3$	mol
Molar mass of M_2CO_3	g/mol
Identity of M_2CO_3	
Percent error	

Post-Lab Calculations and Analysis *(Show all work.)*

1. Calculate the moles of precipitated calcium carbonate, $CaCO_3$. Enter this value in the Data Table.

2. Calculate the molar mass of the unknown carbonate. Enter this value in the Data Table.

3. From the calculated molar mass, identify the unknown. Calculate the percent error in the molar mass value. Enter both values in the Data Table.

4. Review the procedure and list possible sources of errors that would cause the molar mass of the unknown to be (a) too high or (b) too low.