EXPERIMENT 1: Gravimetric Analysis of a Soluble Carbonate

General Introduction

The quantitative analysis of substances and mixtures is a major part of chemistry. It has provided us with our knowledge of the compositions of chemical substances, and is used to determine the formulas of new substances. Quantitative analysis is also widely used to determine the composition of mixtures, such as the concentration of a pollutant in a water sample, or the percentage of a metal in a ore sample.

The simplest way to measure the quantity of a substance is to weigh it. There are therefore many analytical techniques, collectively known as Gravimetric Analysis, based on weighing. For each analysis, a method must be devised so that the substance of interest can be quantitatively isolated (i.e. all of it) in a pure form, or as part of a compound of known composition. In this experiment, carbonate ions from a soluble metal carbonate will be quantitatively isolated as CaCO$_3$(s) by precipitation and filtration.

$$M_2$CO$_3$(aq) + CaCl$_2$(aq) → CaCO$_3$(s) + 2 MCl(aq)$$

The number of moles of carbonate ions in the original sample can be calculated from the mass of CaCO$_3$ obtained.

Laboratory Techniques

In this experiment you will learn or review the following laboratory techniques:

1. Use of a desiccator to prevent inaccuracy in weighings due to atmospheric water
2. Use of a sintered glass crucible for quantitative collection of a precipitate
3. Filtration under reduced pressure using a Buchner Funnel
4. To work safely with a flammable solvent

Objective

1. to determine the molar mass of the metal present in an unknown alkali metal carbonate

Procedure

NOTES:

The “Weighing by Difference” method should be used for all masses to be used in your calculations.

See appendix D.

The CaCO$_3$ precipitate must be collected quantitatively. Any loss of precipitate (e.g. left in the beaker or on the stirring rod) will result in an error in your analysis.

1. Obtain a clean dry sintered glass crucible from the oven. Place it in a desiccator and when it has cooled to room temperature (about 20 min.) make an identifying mark in pencil on the ground glass area on the side of the crucible. Then weigh it on the analytical balance.
2. Record the sample code of the alkali metal carbonate (M$_2$CO$_3$) assigned to you, and then weigh about 0.5 g of this compound into a weighing vial.
3. Weigh the vial with the metal carbonate in it on the analytical balance. Tip the contents of the vial into a 400 mL beaker. Re-weigh the vial on the same analytical balance.
4. Dissolve the metal carbonate in about 125 mL of deionized water. Do not lose any of this solution (e.g., on a glass rod used for stirring) or the final result will be inaccurate. If the carbonate does not dissolve completely, warm the solution, crush any crystals and stir to aid dissolving.
5. Warm the solution until it begins to steam but do not boil it. Slowly add and stir about 30 mL of 0.5 mol·L$^{-1}$ calcium chloride to the solution. This should provide an excess of calcium ions for the precipitation. Remove the beaker from
the ring stand and turn off the Bunsen. Periodically stir the mixture for about 5 minutes. This will cause the calcium carbonate precipitate to coagulate and will make the filtration easier.

6. Set up your filtration apparatus (see Figure 1) and turn on the aspirator.

7. Transfer the precipitate to the **pre-weighed** sintered glass crucible using your glass rod to direct the flow into the crucible. Careful work is essential at this stage.

8. Wash the precipitate in the crucible with 3 x 5 mL portions of water to remove all traces of soluble salts, breaking the suction between washings by pulling the hose off of the aspirator connector.

9. Finally, wash with two 5 mL portions of methanol to remove water. **CAUTION:** Methanol is a flammable organic solvent and must be kept well away from open flames. Its flash point is 10°C, below room temperature! Do not use methanol until directed to do so by a demonstrator. Leave the aspirator on until all liquid has been “pulled” through.

10. Place the crucible in the oven and dry it for 25 minutes at about 120°C - longer if there is a lot of traffic at the oven (i.e. if the oven door is being opened and closed often).

Allow the crucible and contents to cool to room temperature in a desiccator (about 20 min.), then weigh accurately.

![Figure 1](image)

11. Do the calculations and answer all questions for this lab in your Lab Report.

**Note:**

\[
\text{% discrepancy} = \frac{(\text{experimental value}) - (\text{accepted value})}{(\text{accepted value})} \times 100\%
\]
Sample Code ______________________

Table 1
Data for Analysis of Unknown Metal Carbonate

<table>
<thead>
<tr>
<th>Mass of alkali metal carbonate (sample A, B or C) + vial + cover /g</th>
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</thead>
<tbody>
<tr>
<td>Mass of vial + cover /g</td>
<td></td>
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<tr>
<td>Mass of alkali metal carbonate /g</td>
<td></td>
</tr>
<tr>
<td>Mass of sintered glass crucible + calcium carbonate /g</td>
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</tr>
<tr>
<td>Mass of sintered glass crucible /g</td>
<td></td>
</tr>
<tr>
<td>Mass of calcium carbonate /g</td>
<td></td>
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</tbody>
</table>

Calculations
1. Calculate the number of moles of calcium carbonate obtained.

2. Calculate the number of moles of alkali metal carbonate, $\text{M}_2\text{CO}_3$, used based on the following equation:

$$\text{M}_2\text{CO}_3(\text{aq}) + \text{CaCl}_2(\text{aq}) \rightarrow \text{CaCO}_3(\text{s}) + 2 \text{MCl}(\text{aq})$$
3. From the mass of alkali metal carbonate used and your answer to 2, calculate the molar mass of the alkali metal carbonate, \( M_2CO_3 \).

4. Calculate the molar mass of the alkali metal, \( M \), in the alkali metal carbonate.

5. Identify the alkali metal, \( M \).

6. Calculate the percentage discrepancy by comparing the value that you obtained with that given on your periodic table.
Questions

1. (a) What sources of error in this experiment might lead to a high value for the amount of CaCO$_3$(s) collected? Would the calculated molar mass of the alkali metal be high or low? **Explain clearly.**

(b) What sources of error in this experiment might lead to a low value for the amount of CaCO$_3$(s) collected? Would the calculated molar mass of the alkali metal be high or low?

2. If you were being extremely careful and had more time, how would you check to see if your CaCO$_3$(s) was completely dry after step 10 in the experiment?
Gravimetric Analysis of a Soluble Carbonate

An experiment designed to determine the atomic mass of the metal in an unknown soluble metal carbonate, $M_2CO_3$, involved precipitating all of the $CO_3^{2-}$ ion as calcium carbonate, $CaCO_3$, from a solution of the unknown metal carbonate. The following masses were obtained.

Data for Analysis of Unknown Metal Carbonate

| Mass of weighing vial + $M_2CO_3$/g | 6.7335 |
| Mass of weighing vial /g | 5.2332 |
| Mass of $M_2CO_3$/g | 1.5003 |
| Mass of sintered glass crucible + $CaCO_3$/g | 33.4200 |
| Mass of sintered glass crucible /g | 32.3350 |
| Mass of $CaCO_3$/g | 1.0850 |

Calculations

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2. Calculate the number of moles of alkali metal carbonate, $M_2CO_3$, used based on the following equation:

$$M_2CO_3(aq) + CaCl_2(aq) \rightarrow CaCO_3(s) + 2 MCl(aq)$$
Prelaboratory Exercise (cont.)

3. From the mass of alkali metal carbonate used and your answer to 2, calculate the molar mass of the alkali metal carbonate, $M_2CO_3$.

4. Calculate the molar mass of the alkali metal, M, in the alkali metal carbonate.

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