3. GRAVIMETRIC ANALYSIS OF A PRECIPITATE

Initial Question

Chemists can find the identity of unknown compounds using techniques such as qualitative analysis, chromatography, spectroscopy, and gravimetric analysis. Gravimetric analysis, which uses a balance to determine the mass of a substance, is one of the oldest and most accurate quantitative methods for determining the amount of an analyte in a sample.

Can you determine the amount and identity of an unknown component of a substance?

Materials and Equipment

Model 1

- Beakers (4), glass, 100-mL
- Beral pipets (4)
- Unknown A (alkali metal carbonate), 5.0 g
- 0.10 M Sodium nitrate, ($NaNO_3$), 5 drops
- 0.10 M Potassium chloride, (KCl), 5 drops
- 0.10 M Ammonium nitrate, (NH_4NO_3), 5 drops
- 0.10 M Calcium chloride, ($CaCl_2$), 5 drops
- Stirring rod
- Marking pen (to label beakers)
- Distilled water, 200 mL

Model 2 and Applying Your Knowledge

- Beaker, glass, 100-mL
- Filtration funnel
- Erlenmeyer flask, glass, 250-mL
- Filter paper, Whatman® Ashless, #42
- Watch glass, 100-mm
- Analytical balance, 0.001 g precision, 1 per class
- Stirring rod
- Pencil
- Wash bottle with distilled water
- Drying oven, 1 per class

Model 2

- Unknown A (same unknown as Model 1), 1.00 g
- 0.25 M Calcium chloride ($CaCl_2$), 50 mL[2]
- Distilled water, 100 mL

Applying Your Knowledge

- Unknown B, 2.00 g
- 0.50 M Potassium nitrate (KNO_3), 20 mL
- 0.50 M Lithium chloride (LiCl), 20 mL
- 0.50 M Calcium nitrate ($Ca(NO_3)_2$), 20 mL
- 0.50 M Sodium chloride (NaCl), 20 mL
- Distilled water, as needed

Safety

Add these important safety precautions to your normal laboratory procedures:

- Wear your goggles.

Getting Your Brain in Gear

1. In this lab you will be given an unknown alkali metal carbonate. Your lab group will have to determine the identity of that compound through gravimetric analysis. The unknown could be: lithium carbonate, sodium carbonate, potassium carbonate, or cesium carbonate.

   a. Does each of these compounds have the same ratio of metal atoms to carbonate ions? Explain your answer.

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   b. What information do you need to find the percent mass of an ion, like carbonate, in a compound?

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   c. Using the periodic table, find the percentage of carbonate by mass in each of the carbonates.

   Lithium carbonate (Li_2CO_3): _____

   Sodium carbonate (Na_2CO_3): _____

   Potassium carbonate (K_2CO_3): _____

   Cesium carbonate (Cs_2CO_3): _____

   d. Why is the percentage of carbonate different for each of the compounds listed above while the ratio of carbonate to metal ion is the same?

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   e. When identifying an unknown, why is it helpful to know the chemical percent composition?

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MODEL 1

Building Model 1 – Precipitating an Unknown

1. Obtain four 100-mL beakers and label them "A," "B," "C," "D."

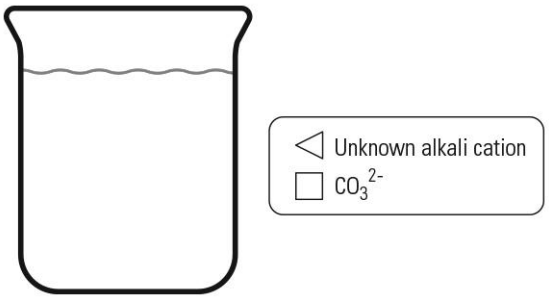
2. Add approximately 50 mL of distilled water to each beaker.

3. Into each of the 100-mL beakers add a *pea size* amount of Unknown A, which is an alkali metal carbonate.

4. Use a stirring rod to mix and dissolve the unknown carbonate.

5. In the beaker below draw the unknown carbonate solution as a particulate-level representation. Use the particulate key as a guide.

   NOTE: Use "M" when referring to the unknown alkali metal.



6. To determine the identity of the unknown, the mass of the unknown and the mass of carbonate in the unknown needs to be obtained. Brainstorm ways to separate the alkali metal ions from the carbonate ions.

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7. Into Beaker A add 5 drops of 0.1 M $NaNO_3$.

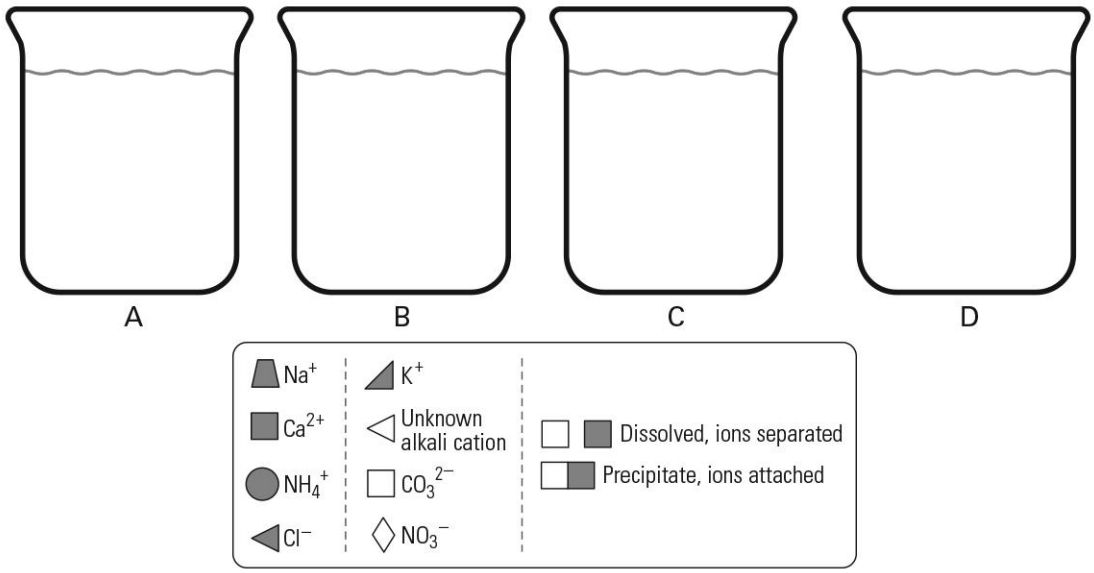
8. Into Beaker B add 5 drops of 0.1 M NH_4NO_3.

9. Into Beaker C add 5 drops of 0.1 M $CaCl_2$.

10. Into Beaker D add 5 drops of 0.1 M KCl.

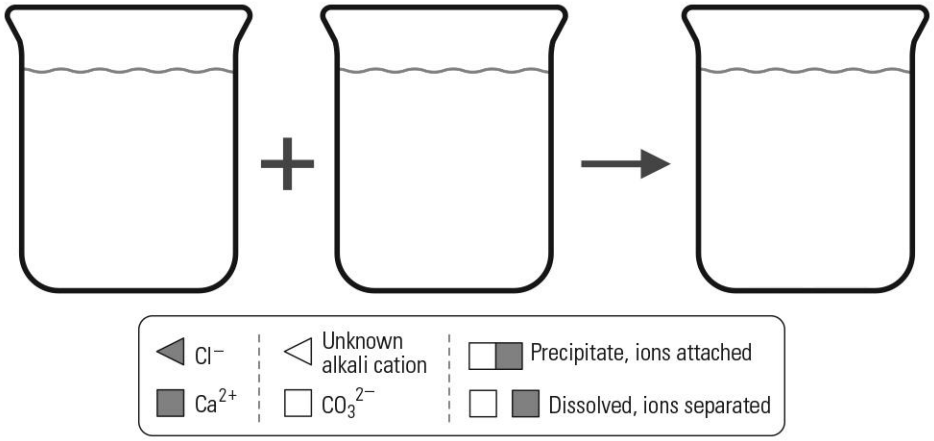
11. In the beakers in Model 1 draw particulate-level pictures of the resulting solutions in Beakers A-D. Use the particulate key as a guide.

Model 1 – Precipitating an Unknown



Analyzing Model 1 – Precipitating an Unknown

12. Draw a particulate-level representation of the reaction that occurred in Beaker C. Start with three molecules of M_2CO_3, where "M" refers to the unknown alkali metal, and three molecules of $CaCl_2$. Use the particulate key as a guide. Please observe the Law of Conservation of Mass in your drawings.



13. Use your drawings of the reaction in Beaker C to describe the pathway the carbonate molecules followed throughout this reaction.

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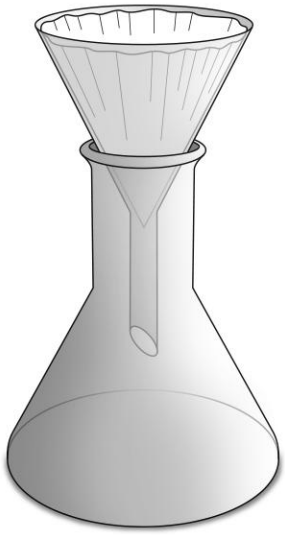
MODEL 2

Building Model 2 – Finding the Mass of Carbonate in an Unknown

1. Place a clean 100-mL beaker on an analytical balance and tare the mass of the beaker.
2. Add approximately 1.00 g of your unknown carbonate into the beaker. Record the exact mass (to three decimal places) in the Model 2 Data Table.
3. Add 20 mL of distilled water and stir with a stirring rod until dissolved.
4. Add 30 ml of 0.25 M $CaCl_2$ to the solution containing the unknown carbonate and stir to dissolve.
5. Assemble a filtration setup as in the diagram to the right using the Erlenmeyer flask and funnel.
6. Write your initials on a piece of filter paper with a pencil. Record the mass (to three decimal places) of the dry filter paper in the Model 2 Data Table. Then fold the filter paper and place it into the funnel.
7. Slowly pour the contents of the beaker with your precipitate into the funnel. Do not overfill the filter paper.
8. Rinse the beaker and stirring rod with deionized water to ensure that all the precipitate is in the funnel.
9. Continue until all of the filtrate has moved through the filter paper into the flask.



10. What type of substance is small enough to pass through the filter paper?

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11. What type of substance is large enough to get caught in the filter?

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12. How would the amount of your yield be affected if some of the carbonate was not precipitated?

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13. Where would the non-precipitated ions be after the filtration is complete?

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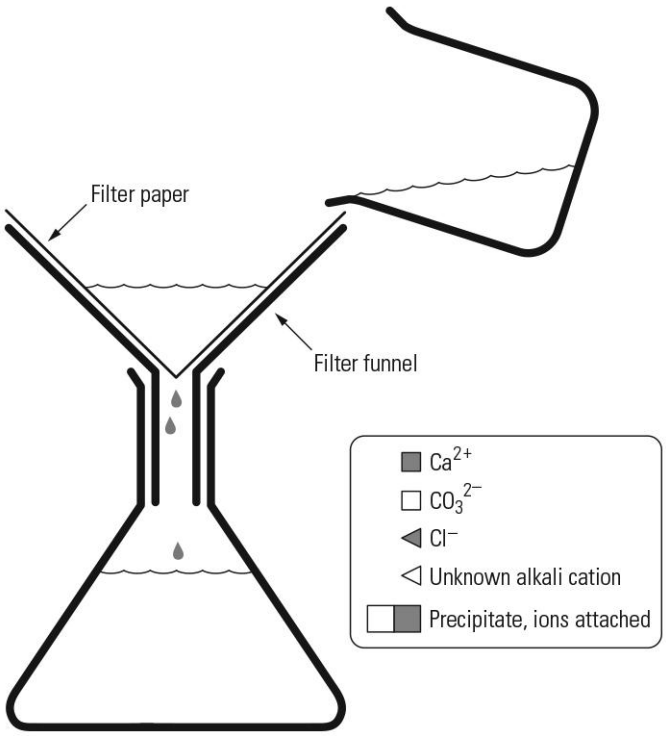
14. In the filtration setup below, draw particulate-level pictures of the products being filtered. Use the particulate key as a guide.



15. How can you determine if any carbonate ions went through the filter?

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16. What action should you take if carbonate ions passed through the filter?

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17. After you make sure all the carbonate has been removed from the filtrate, carefully place the wet filter paper with the $CaCO_3$ onto a watch glass and then into a drying oven overnight. This will remove any remaining water to ensure accurate mass measurements.

The Next Day

18. Remove the filter paper from the drying oven and measure its mass using an analytical balance. Record the mass (to three decimal places) in the Model 2 Data Table and determine the mass of the calcium carbonate precipitate.

Model 2 – Finding the Mass of Carbonate in an Unknown

Table 1: Model 2 Data Table – Finding the mass of carbonate in an unknown

Parameter	Mass (g)
Alkali metal carbonate (Unknown A)	
Dry filter paper	
Dry filter paper plus $CaCO_3$	
$CaCO_3$	

Analyzing Model 2 – Finding the Mass of Carbonate in an Unknown

19. Use the periodic table to determine the percentage of carbonate in calcium carbonate.

20. Knowing the percentage of carbonate in $CaCO_3$ and the mass of calcium carbonate, find the mass of carbonate in the unknown alkali metal carbonate (Unknown A).

21. Find the percentage of carbonate in the alkali metal carbonate using the mass of the carbonate from Unknown A in Model 2 and the total mass of the alkali metal carbonate.

22. The possible unknown compounds in this lab include lithium carbonate, sodium carbonate, potassium carbonate, and cesium carbonate. Using the percentages calculated in the Getting Your Brain in Gear section of the lab, identify the unknown and explain why you are confident with the methods of data collection that lead you to this conclusion.

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Connecting to Theory

When water quality is tested, a precipitate is intentionally formed. From the color of the precipitate, the identity of the dissolved ions can be determined. Chemists consult the Solubility Rules when they want to force ions to precipitate. The Solubility Rules are designed so that the user can cross reference cations with anions and determine if they will precipitate. Once the precipitate is formed, it can be dried and measured using gravimetric techniques.

Table 2: Abridged Solubility Rules

Ion	Solubility	Exceptions
NO_3^- (Nitrate)	Soluble	None
ClO_4^- (Chlorate)	Soluble	None
Halogens except F	Soluble	Ag^+, Hg_2^{2+}, Pb^{2+}
F^- (Fluoride)	Soluble	Ca^{2+}, Ba^{2+}, Sr^{2+}, Hg_2^{2+}, Pb^{2+}, Ag^+
SO_4^{2-} (Sulfate)	Soluble	Ca^{2+}, Ba^{2+}, Sr^{2+}, Hg^{2+}, Pb^{2+}, Ag^+
CO_3^{2-} (Carbonate)	Insoluble	Alkali Metals and NH_4^+
PO_4^{3-} (Phosphate)	Insoluble	Alkali Metals and NH_4^+
OH^- (Hydroxide)	Insoluble	Alkali Metals and Ca^{2+}, Ba^{2+}, Sr^{2+}
Alkali metals	Soluble	None
NH_4^+ (Ammonium)	Soluble	None

Applying Your Knowledge – Identifying an Unknown

An unknown alkali metal carbonate was discovered in a pharmacy. The pharmacist would like to identify it. If the unknown is lithium carbonate, it can be used as a drug to treat bipolar disorders. All other alkali carbonates: potassium carbonate, cesium carbonate, and sodium carbonate, will not be useful in treating this type of disorder.

Your lab group can use the following chemicals to identify the unknown:

- 2.0 g of unknown alkali metal carbonate
- 20.0 mL of the following solutions 0.5 M KNO_3, 0.5 M LiCl, 0.5 M $Ca(NO_3)_2$, and 0.5 M NaCl.

All other standard laboratory equipment is available. Consult your instructor with special requests. You do not need to use all of the materials provided.

1. Design a procedure to find the identity of the unknown.

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2. Create a data table to organize your data.

3. Before you start the lab, have your instructor approve your lab procedure for safety, but not on the accuracy of your proposed procedure.

4. Show the calculations needed to identify the unknown.

5. Did the pharmacist have lithium carbonate or a different compound? How did you come to this conclusion?

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6. Discuss the limitations of the laboratory techniques you used and how you can improve the accuracy of your results. You may use diagrams to supplement your explanations.

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