Studies of a Precipitation Reaction

Prelab Assignment
Read the entire lab. Write an objective and any hazards associated with this lab in your laboratory notebook. Answer the following 6 questions in your laboratory notebook before coming to lab.

1. (a) What do you expect to see (literally) if a compound is solubilized?

   (b) Write the equations for the solubilization or disassociation of (1) calcium nitrate and (2) potassium iodate in water. Iodate is a polyatomic ion with the formula $\text{IO}_3^-$.

2. What do you expect to see (literally) if a compound precipitates?

3. Do you expect a reversible reaction to go to completion? In other words do you expect all of the reactants to be converted completely to products in a reversible reaction and why or why not?

4. List at least three reasons why making detailed observations is important in solving chemistry problems?
5. Read the second full paragraph of the Introduction section of this lab write up. Carefully consider all of the differences between the example of a well and a poorly stated hypothesis. Then answer the following questions.
(a) Why is “A chemical reaction will occur when calcium nitrate is mixed with potassium iodate” not a well stated hypothesis?

(b) Propose a more specific, clearly stated, and testable hypothesis that could be used for part (a) of this question.

6. List at least two considerations in designing a good experiment.
Introduction

Over time the rates (or speed) of the forward and reverse reactions of reversible reactions become equal. Products are being formed and broken down at the same rate. After this point in time the concentrations of the reactants and products no longer change with time or become constant. This does not mean the reaction has stopped or that the concentrations of the reactants and products are equal to each other. This state is called equilibrium. The ratio of the concentration of the products raised to the power of their stoichiometric coefficient(s) to the concentration of the reactants raised to the power of their stoichiometric coefficient(s) at equilibrium is called the equilibrium constant. Irreversible reactions cannot reach equilibrium because there is no reverse reaction.

The Process of Science

In science it is important to make careful observations and record all observations in detail. Observations may be qualitative or quantitative. Quantitative observations involve numerical values and generally result from measurements. Qualitative observations are recording what you can physically sense. It is seldom possible to know what phenomena or details are important in advance. It is better to record irrelevant details than to miss details. Irrelevant details may be ignored. Missed observations cannot necessarily be repeated and may cause an incorrect hypothesis to be formed or cause the phenomena of interest not to be understood at all. Review the observations you have made during an experiment before you leave the laboratory to ensure (1) you have considered all comparative information and (2) your observations have not been contaminated by expectations or belief. Part of the point of making observations is to determine what is unusual. Therefore, it is important to record what you see, smell, otherwise sense or measure that is not expected as well as what is expected. When you place a strong acid on an egg white you see the color change from clear to opaque white. This is an observation. If you record that the egg cooked, you have contaminated your observation with a belief that the white color was caused by the egg being cooked. Allowing expectations or beliefs to impact your observations may lead to incorrect hypotheses and/or an inability to understand the phenomena under study.

A hypothesis is based on previous observations or previous knowledge and therefore a hypothesis cannot be formed without a set of reliable and detailed observations and/or a thorough literature review. A hypothesis is a possible explanation for the observations that have been made or a prediction of what will happen. In forming a hypothesis trends in observations or data and relationships between observations or data are examined and causes and effects are explored. A hypothesis is a statement and is not a question. A good scientific hypothesis must be testable by an experiment. A hypothesis must be stated so that it can be proven true or false by an experiment. In other words the difference between a true and false outcome must be clearly stated in the hypothesis. A hypothesis should be detailed. It should be stated so that it can be answered with either yes (true) or no (false). An example of a vague poorly stated hypothesis is: a precipitate will form when Ca(NO$_3$)$_2$ and NaOH solutions are mixed. An example of a clearly stated testable hypothesis is: the solid compound Ca(OH)$_2$ will precipitate within 30 minutes after 1 M solutions of Ca(NO$_3$)$_2$ and NaOH are mixed.

In most experiments some property or factor is altered or varied and the response of another property or factor is observed and recorded. The factor that is changed is called the
dependent variable, and the response is called the independent variable. In a well designed experiment only one factor is varied at a time. If more than one factor is allowed to vary at the same time it is not possible to draw verifiable conclusions concerning the cause of the response observed. In chemistry it is important that all of the variables impacting the system are identified. Experiments should be designed so that only one factor is varied at a time. The number of steps in an experiment should be minimized in order to minimize error.

Procedure

Problem Statement: What is the nature of the reaction between calcium nitrate, \( \text{Ca(NO}_3\text{)}_2 \), and potassium iodate, \( \text{KIO}_3 \)?

I. Data Collection: Interaction of calcium nitrate and potassium iodate
A. Wash and label two 100 or 150 mL beakers. Obtain, about 40 mL of 0.2 M calcium nitrate, \( \text{Ca(NO}_3\text{)}_2 \), and 55 mL of 0.1 M potassium iodate, \( \text{KIO}_3 \), stock solutions in the clean labeled beakers – one solution in each beaker. Record the molarity of each stock solution in your laboratory notebook.

![beakers of Ca(NO₃)₂ and KIO₃ stock solutions]

Wash and label two 250 mL beakers. Prepare the following two solutions – one in each beaker – using a graduated cylinder and **using the calcium nitrate and potassium iodate solutions you obtained previously and put into your 100 or 150 mL beakers**.

Solution 1: \( 25 \text{ mL Ca(NO}_3\text{)}_2 + 25 \text{ mL KIO}_3 - 50 \text{ mL total} \)
Solution 2: \( 15 \text{ mL distilled H}_2\text{O} + 10 \text{ mL Ca(NO}_3\text{)}_2 + 25 \text{ mL KIO}_3 - 50 \text{ mL total} \)

![beakers used as reaction vessels]
Mix each solution with a separate stirring rod, rubbing the tip of the rod against the bottom or sides of the beakers. This is a process called **scratching**. Scratching produces an unpleasant sound something like fingernails being scraped across a chalk board. Scratching causes imperfections on the surface of the beaker and provides a surface for precipitation of a solid to occur thus speeding up the precipitation process. Scratch the beaker with the glass rod frequently until a solid has begun to form. Then let the two solutions stand for 20-30 minutes. Proceed to part B while you are waiting. Record all of your observations in your laboratory notebook.

B. Calcium nitrate dissociates in water to form the calcium cation and nitrate anion as illustrated in the following reaction

\[ \text{Ca(NO}_3\text{)_2(aq)} \rightarrow \text{Ca}^{2+}(\text{aq}) + 2\text{NO}_3^{-}(\text{aq}). \]

Potassium iodate dissociates in water to form the potassium cation and iodate anion as illustrated in the following reaction

\[ \text{KIO}_3(\text{aq)} \rightarrow \text{K}^{+}(\text{aq}) + \text{IO}_3^{-}(\text{aq}). \]

Assume the reaction of calcium nitrate and potassium iodate is a precipitation reaction. Record in your laboratory notebook what possible salts or ionic compounds could form from the reaction of calcium nitrate and potassium iodate. Review your solubility guidelines and record the whether or not each of these salts is soluble in water. Write balanced chemical equations for all possible reactions from the mixing of calcium nitrate and potassium iodate in your laboratory notebook. Remember that reactions may be either reversible or irreversible. Based on this information form a hypothesis predicting the precipitate(s) formed by the mixing of calcium nitrate and potassium iodate as well as what ions remain in solution. Record (1) the problem your hypothesis addresses or tries to answer and (2) your hypothesis in your laboratory notebook. Discuss your problem statement and hypothesis with your lab instructor, refine your hypothesis and record the refined hypothesis approved by your lab instructor in your lab notebook.

II. Data Collection: Weight of the precipitates

A **filtrate** is the solution that has passed through a filter.

A. Label and weigh two pieces of filter paper and record their masses in your laboratory notebook. Fold the weighed filter paper as illustrated by your instructor. Insert the folded filter paper into a filter funnel. Place the filter funnel in a clean Erlenmeyer flask. Seat (or seal) the filter to the funnel surface by filtering approximately 5 mL of distilled water through the filter. Gently swirl your solution to suspend the solids. Quickly and carefully pour the solution into the filter funnel. Make sure you pour the solution from beaker 1 into filter 1. Use a stirring rod and rubber policemen to transfer (push) as much of the precipitate as possible into the filter funnel. Use a wash bottle of distilled water to wash any remaining precipitate out of your beaker and into the filter funnel.
Wash the precipitate and the surface of your filter with approximately 2 mL of distilled water from a wash bottle and allow the water to completely drain from the filter. Repeat this washing step at least 4 more times. Gently remove the filter paper from the funnel, place it on a watch glass and allow it to air dry. Save your filtrates for further analyses.

B. Based on the molar concentrations of your stock solution and the volumes of solution used, calculate the number of moles of calcium nitrate and potassium iodate added to each solution. Using these numbers of moles and the balanced chemical equations you deduced previously in part I B of this lab, calculate the expected mass of the precipitate (also called the theoretical yield) for solution 1 and solution 2. What is the limiting reagent in each case (in solutions 1 and 2)? Record your calculations in your laboratory notebook.

III. Data Collection: Tests of the supernatant/filtrate identities

A supernatant is the solution standing above a settled precipitate in the bottom of a container.

Measure and transfer about 5 mL of 0.3 M Na₃PO₄ solution into a small test tube. Measure about 5 mL of 1 M HCl into a separate small test tube.

Test tubes of Na₃PO₄ and HCl stock solutions

A. Positive tests (tests of your Ca(NO₃)₂ and KIO₃ stock solutions)

Place about 1 mL of the stock 0.2 M Ca(NO₃)₂ solution from your 100 or 150 mL beaker in a test tube. Place about 1 mL of 0.1 M KIO₃ stock solution from your 100 or 150 mL beaker in a separate test tube. Note that 20 drops is equal to about 1 mL.
100 or 150 mL beakers of Ca(NO\textsubscript{3})\textsubscript{2} and KIO\textsubscript{3} stock solutions

Calcium cation, Ca\textsuperscript{2+}, test: Add about 10 drops (0.5 mL) of 0.3 M Na\textsubscript{3}PO\textsubscript{4} solution from your small test tube to the test tube containing 1 mL of stock 0.2 M calcium nitrate solution. Record your observations in your laboratory notebook. The reaction of calcium cation with phosphate anions is illustrated below:

\[
3\text{Ca}^{2+}(aq) + 2\text{PO}_4^{3-}(aq) \rightarrow \text{Ca}_3(\text{PO}_4)_2(s).
\]

Iodate, IO\textsubscript{3}⁻, test: Dissolve about 0.5 cm\textsuperscript{3} (about 1/4 inch on a spatula) of KI in the 1 mL of stock 0.1 M potassium iodate solution in your test tube; then add about 1 mL of 1 M HCl to the test tube. Record your observations in your laboratory notebook. The reaction of the iodate anion with the iodide anion and hydrogen cation is illustrated below:

\[
\text{IO}_3^-(aq) + 5\text{I}^-(aq) + 6\text{H}^+(aq) \rightarrow 3\text{I}_2(aq) + 3\text{H}_2\text{O}.
\]

red liquid
B. Filtrate tests

Repeat the calcium cation test using 1 mL of solution 1 filtrate rather than 1 mL of 0.1 M Ca(NO$_3$)$_2$ stock solution. Repeat the test again using 1 mL of solution 2 filtrate. Compare your results for the calcium cation test using solution 1 and solution 2 filtrates to each other and to the results using the 0.2 M Ca(NO$_3$)$_2$ stock solution. Record your observations in your laboratory notebook.
Repeat the iodate test using 1 mL of solution 1 filtrate rather than 1 mL of 0.1 M KIO₃ stock solution. Repeat the test again using 1 mL of solution 2 filtrate. Compare your results for the iodate test using solution 1 and solution 2 filtrates to each other and to the results using the 0.1 M KIO₃ stock solution. Record your observations in your laboratory notebook.
IV. Data Collection: Testing your hypothesis

Do your observations from section III support or refute your hypothesis? In other words do you expect any calcium cation and iodate anion to remain in your supernatant and filtrate after 30 minutes and why or why not? Record your observations and conclusions in your laboratory notebook.
Problem Statement:

Initial hypothesis statement:

Refined hypothesis statement approved by your lab instructor:

Molarity of stock Ca(NO$_3$)$_2$ solution (listed on the bottle)  M (moles/L)

Molarity of stock KIO$_3$ solution (listed on the bottle)  M

<table>
<thead>
<tr>
<th>mL Ca(NO$_3$)$_2$ solution:</th>
<th>Solution 1</th>
<th>Solution 2</th>
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<tbody>
<tr>
<td>mL KIO$_3$ solution:</td>
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<tr>
<td>mass of filter paper:</td>
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<td>mass of filter &amp; precipitate:</td>
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<td>mass of precipitate:</td>
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<tr>
<td>moles of Ca(NO$_3$)$_2$:</td>
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<td>moles KIO$_3$:</td>
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<td>expected mass of precipitate, Ca(IO$_3$)$_2$:  (or theoretical yield)</td>
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<td>% yield:</td>
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<td>(mass of precipitate/theoretical yield) x 100</td>
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Questions:

1. Calculate the percent yield for solutions 1 and 2. How do your actual precipitate masses compare to the masses you predicted by calculation, in other words were your percent yields high or low? If your percent yields were low make a list of at least 4 possible explanations for the low yield.

2. Are your test results for the presence of Ca$^{2+}$ and IO$_3^-$ in the supernatants consistent with the limiting reagent and excess reagent calculations in part II B of this laboratory exercise? Why do you think your results do or do not agree with your predictions?

3. Did the results from the experiment you designed to test your hypothesis support or refute your hypothesis? How might you have improved your hypothesis and your test methods?