Objective

- In this experiment you will investigate the dissolved iodate ion concentration in saturated solutions of calcium iodate hydrate, $Ca(IO_3)_2 \bullet xH_2O$ (solution 1), and calcium iodate hydrate, $Ca(IO_3)_2 \bullet xH_2O$ in KIO_3 (solution 2). You will determine the $[IO_3^-]$ and the $[Ca^{2+}]$ and use these values to determine the K_{sp} for $Ca(IO_3)_2 \bullet xH_2O$.

Skill

- Practice titrations with the application to solve for K_{sp}.

Introduction

In a saturated solution of $Ca(IO_3)_2 \bullet xH_2O$, the equilibrium between the dissolved ions and the solid is given by the equation:

$$Ca(IO_3)_2 \cdot xH_2O_{(s)} \leftrightarrows Ca^{2+}{}_{(aq)} + 2\ IO_3^-{}_{(aq)} + x\ H_2O_{(l)} \qquad \text{Equation 1}$$

The quantitative equilibrium expression describing the concentration of iodate and calcium ions at equilibrium is:

$$K_{sp} = [Ca^{2+}][IO_3^-]^2 \qquad \text{Equation 2}$$

Interestingly, this equilibrium between the solid and the dissolved ions produces a constant solubility product under a variety of conditions. If the solid is placed into 1 L of water, the solid dissolves (the reaction shown in equation 10-1 proceeds to the right) until the product of the dissolved ion concentrations becomes equivalent to the solubility product ($[Ca^{2+}]\ [IO_3^-]^2$).

If the solid is placed into a solution with aqueous potassium iodate as the solvent, not as much solid will dissolve since common ion (IO_3^-) already exists in the solution, thus the equilibrium is not driven as far to the right as in the previous example where pure water was used as the solvent. The product of ($[Ca^{2+}]\ [IO_3^-]^2$ *will still equal K_{sp}*, but since product (the iodate ions) is initially present, the amount of solid allowed to dissolve is curtailed. This reduction in solubility (of the solid) is known as the "Common Ion Effect".

In this experiment, you will investigate the dissolved iodate ion concentration in saturated solutions of calcium iodate by titrating solutions that are in equilibrium with the solid. The solutions will be titrated with a standard sodium thiosulfate solution. Once the iodate concentration is determined, this value allows us to calculate the concentration of calcium ions that also dissolved from the solid through the stoichiometry of the process.

You will use a standardized solution of sodium thiosulfate ($Na_2S_2O_3$) to titrate the iodine produced by reacting the iodate ions (in your saturated solution) with potassium iodide (KI). Iodine is produced from this reaction as follows:

$$IO_3^-{}_{(aq)} + 5\ I^-{}_{(aq)} + 6\ H_3O^+{}_{(aq)} \rightarrow 3\ I_{2\,(aq)} + 9\ H_2O_{(l)} \qquad \text{Equation 3}$$

The iodine (I_2) that is formed is titrated with thiosulfate:

$$I_{2\,(aq)} + 2\ S_2O_3^{2-}{}_{(aq)} \rightarrow 2\ I^-{}_{(aq)} + S_4O_6^{2-}{}_{(aq)} \qquad \text{Equation 4}$$

From equation 3, we can see that each iodate ion (IO_3^-) in solution will produce three molecules of iodine (I_2). From equation 4, we see the each molecule of I_2 reacts with two thiosulfate ions. From these two mole ratios, we can determine that each iodate ion in solution will react with six thiosulfate ions in the titration. You will need to use these two mole ratios in your stoichiometry calculation to determine the number of moles of iodate ion in the analyte solution.

Two solutions saturated with solid calcium iodate will be investigated. Solution 1 contains calcium iodate in pure water and Solution 2 contains calcium iodate in potassium iodate so that the effect of the common ion can be observed. However, in each case the calculated solubility product (K_{sp}) should maintain a constant value *if* the experiment has been carried out properly.

Acknowledgments

This experiment was adapted from "How do we determine the solubility and an equilibrium constant for a sparingly soluble salt? Purdue University's 1999 Chemistry 116 Lab Manual"

Equipment and Materials

Calcium iodate in H_2O (Solution 1)

Calcium iodate in KIO_3 (Solution 2)

0.240 M potassium iodide solution

0.0500 M sodium thiosulfate

1 M HCl

1% starch solution

Filter paper

Funnel

Beakers

8.00 mL volumetric pipette

250.0 mL Erlenmeyer flask

25.00 mL burette

Graduated cylinders

Experimental Procedure

For this experiment two teams will work together and share their data with each other when finished with the experiment tasks. It is possible a team may be one individual or a pair of students. One team will complete Part A with solution 1 and the other team will complete Part B with solution 2.

A. Team A - Titrations of Saturated Calcium Iodate in Water (Solution 1)

Filter the Calcium Iodate Solution

1. Shake the mixture prior to pouring sample, to ensure the solution is saturated and homogeneous. Obtain 50 mL of the calcium iodate in water solution in a clean beaker. It is ok if this sample is hazy.
2. Prepare filter papers and funnels to gravity filter the solution.
3. Filter the solution and collect the filtrate in a clean dry labeled beaker. The filtrate must be clear. A hazy liquid indicates fine particles of solid present in the solution.
4. If the filtrate is hazy, filter the solution again. Your filtrate must be clear to proceed.

Prepare Analyte

1. Obtain about 35 mL of 1 M HCl in a clean beaker, labeled “1 M HCl.”
2. Obtain about 75 mL of 0.240 M KI solution in a 150 mL beaker, labeled “0.240 M KI”.
3. Obtain a clean dry 250 mL Erlenmeyer flask and add the following three materials:
    a. Use a graduated cylinder to add 25 mL of the KI solution.
    b. Use a 8.00 mL volumetric pipette to add 8.00 mL of the filtered $Ca(IO_3)_2$ solution.
    c. Use a graduated cylinder to add 10 mL of 1 M HCl from step 1.

The solutions should be orange/brown at this point.

Titrate

1. Obtain about 60 mL of 0.0500 M sodium thiosulfate standard solution, $Na_2S_2O_3$, in a clean beaker, labeled “0.0500 M $Na_2S_2O_3$.”

2. Record the actual concentration of the $Na_2S_2O_3$ standard solution on your data table.
3. Prepare a burette for performing titrations by rinsing and filling it with the 0.0500 M $Na_2S_2O_3$ standard solution.
4. Record your initial burette reading in Table 1 of the data tables.
5. Titrate carefully with $Na_2S_2O_3$ until the solution turns pale yellow (post-it note yellow). When the solution is yellow, do the following:
    a. Add 10 drops of 1% starch indicator (the solution should turn blue-black).
    b. Continue to *slowly titrate* until the solution just turns *colorless*.
6. Record your final burette reading in Table 1 of the data tables.

NOTE: The intermediate volume when the solution was post-it note yellow does not need to be recorded.

7. Repeat the preparation of the analyte and titration at least two more times. Your volume of titrant used should agree to within +/- 0.20 mL. See your instructor if your readings are outside this range.

B. Team B - Titrations of Saturated Calcium Iodate in KIO_3 (Solution 2)

Filter the Calcium iodate in potassium iodate solution

1. Shake the mixture prior to pouring sample, to ensure the solution is saturated and homogenous. Obtain 50 mL of the *calcium iodate in* KIO_3 solution in a clean beaker. It is ok if this sample is hazy.
2. Record the concentration of KIO_3 on your handout.
3. Prepare filter papers and funnels to gravity filter the solution.
4. Filter the solution and collect the filtrate in a clean dry labeled beaker. The filtrate must be clear. A hazy liquid indicates fine particles of solid present in the solution.
5. If the filtrate is hazy, filter the solution again. Your filtrate must be clear to proceed.

Prepare Analyte

1. Obtain about 35 mL of 1 M HCl into a clean beaker, labeled “1 M HCl.”
2. Obtain about 75 mL of the 0.240 M KI solution in a 150 mL beaker, labeled “0.240 M KI”.
3. Obtain a clean dry 250 mL Erlenmeyer flask and add the following three materials:
    a. Use a graduated cylinder to add 25 mL of the KI solution.
    b. Use a 8.00 mL volumetric pipette to add 8.00 mL of the filtered $Ca(IO_3)_2$ in KIO_3 solution.
    c. Use a graduated cylinder to add 10 mL of 1 M HCl from step 1.

    The solutions should be orange/brown at this point.

Titrate

1. Obtain about 60 mL of 0.0500 M sodium thiosulfate standard solution, $Na_2S_2O_3$, in a clean beaker, labeled "0.0500 M $Na_2S_2O_3$."
2. Record the actual concentration of the $Na_2S_2O_3$ standard solution on your data table.
3. Prepare a burette for performing titrations by rinsing and filling it with the 0.0500 M $Na_2S_2O_3$ standard solution.
4. Record your initial burette reading in Table 3 of the data tables.
5. Titrate carefully with $Na_2S_2O_3$ until the solution turns pale yellow (post-it note yellow). When the solution is yellow then do the following:
    a. Add 10 drops of 1% starch indicator (the solution should turn blue-black).
    b. Continue to *slowly titrate* until the solution just turns colorless.
6. Record your final burette reading in Table 3 of the data tables.

NOTE: The intermediate volume when the solution was post-it note yellow does not need to be recorded.

7. Repeat the preparation of the analyte and titration at least two more times. Your volume of titrant used for each trial should agree to within +/- 0.20 mL. See your instructor if your readings are outside this range.

Make sure you know who you are exchanging data with. You will need to upload images of both Table 1 and Table 3 in the report, no matter which part you completed in lab!

Clean-up

1. All solutions can be poured in the sink with running water.
2. Use hot soapy water to wash all glassware and appropriate equipment. Rinse well with hot tap water.
3. DI rinse all washed glassware and equipment then return to the place you obtained it. Let air dry.
4. Fill the DI water bottle that is at your lab bench.
5. Check the bin/drawer at your station and make sure all of the items were returned to it. If materials are missing, let your instructor know so that they can be replaced before the next class.
6. Clean the common area assigned to you by your instructor.
7. Return your personal items to your pack/bag.
8. Disinfect your work area(s) that cannot be washed in the sink with disinfectant spray. DO NOT wipe – Let disinfectant air dry.
9. Wash your hands before you leave the lab.

Calculations

A. Titrations of Saturated Calcium Iodate in Water (Solution 1)

This discussion will help determine the solubility, K_{sp}, of the $Ca(IO_3)_2 \cdot xH_2O$ in water.

Titration calculations use stoichiometry to determine the concentration of an unknown analyte from the concentration of a known titrant. You have much experience with this; however, this one is a little more complicated because several reactions are taking place. More than one mole ratio is needed to get from the concentration of the titrant to the concentration of the unknown analyte. The mole ratios you determined for the Preparation Activity should be recorded for use in the calculations.

Perform the calculations below in order to determine the experimental K_{sp} of the calcium iodate hydrate in water.

1. Calculate the number of moles of thiosulfate ion, $S_2O_3^{2-}$, that were used to reach equilibrium in the titration.
2. Calculate the number of moles of iodate ion, IO_3^-, from the calcium iodate hydrate which reacted with the thiosulfate ions, $S_2O_3^{2-}$, that came from the moles of sodium thiosulfate determined in the first calculation.
3. Calculate the concentration of iodate ions, $[IO_3^-]$, from the moles of iodate calculated above.
4. Calculate the concentration of calcium ions, $[Ca^{2+}]$, from the calcium iodate hydrate —see equation (1) to determine a mole ratio that will allow this concentration calculation.
5. Calculate the experimental K_{sp} for the calcium iodate hydrate in water (equation 2).

B. Titrations of Saturated Calcium Iodate in KIO_3 (Solution 2)

The following discussion will help determine the solubility, K_{sp}, of the calcium iodate in potassium iodate, $Ca(IO_3)_2 \cdot xH_2O$ in KIO_3.

1. Calculate the number of moles of thiosulfate ion, $S_2O_3^{2-}$, that were used to reach equilibrium in the titration.
2. Calculate the moles of iodate ions, IO_3^-, which reacted with the thiosulfate ions, $S_2O_3^{2-}$, from the titrant.

This is calculated the same way #2 was calculated for part A, although in this system the iodate ions came from both the calcium iodate hydrate and from the potassium iodate. This is the total number of moles of iodate ions in the system —this will be helpful in the 4th calculation below.

The calcium ion concentration cannot be directly determined from the total iodate ion concentration since some of the iodate ions came from the potassium iodate. This may sound complex, but it is not that difficult since the concentration of the potassium iodate in which the calcium iodate hydrate was dissolved is known.

3. Calculate the moles of iodate ion, IO_3^-, that came from the KIO_3.
4. Determine the moles of iodate ion, IO_3^-, which came from the calcium iodate hydrate, $Ca(IO_3)_2 \cdot xH_2O$. (Yes, it is a simple calculation.)
5. Calculate the concentration of the calcium ion, $[Ca^{2+}]$, —recall the amount that was pipetted into the flask for the titration (see equation 1).

It's important to understand that the K_{sp} will be calculated using the concentration of all of the iodate ions. When two numbers are multiplied together if one of them decreases what will have to happen to the other to give the same product? Yes, it will have to increase. Should the K_{sp} change? Think about what causes the K_{sp} to change, it will help you answer one of the questions.

6. Calculate the molar concentration of the iodate ion, $[IO_3^-]$.
7. Calculate the experimental K_{sp} for the calcium iodate hydrate in potassium iodate (see equation 2).

Calculations

A. Titrations of Saturated Calcium Iodate in Water (Solution 1)

1. Calculate the number of moles of thiosulfate ion, $S_2O_3^{2-}$, reacted.
2. Calculate the number of moles of iodate ion, IO_3^-, reacted
3. Calculate the molar concentration of iodate ions, $[IO_3^-]$.
4. Calculate the molar concentration of calcium ions, $[Ca^{2+}]$.
5. Calculate the experimental K_{sp} for the calcium iodate hydrate in water.

B. Titrations of Saturated Calcium Iodate in KIO_3 (Solution 2)

1. Calculate the number of moles of thiosulfate ion, $S_2O_3^{2-}$, reacted.
2. Calculate the total number of moles of iodate ions, IO_3^-, reacted.
3. Calculate the number of moles of iodate ions, IO_3^-, that came from KIO_3.
4. Calculate the number of moles of iodate ions, IO_3^-, that came from $Ca(IO_3)_2 \cdot xH_2O$.
5. Calculate the molar concentration of calcium ions, $[Ca^{2+}]$.
6. Calculate the total molar concentration of iodate ions, $[IO_3^-]$.
7. Calculate the experimental K_{sp} for the calcium iodate hydrate in potassium iodate.