18A. A Solubility Product Constant

Introduction

The interaction of a slightly soluble ionic compound with its dissolved ions leads to another type of equilibrium (Ebbing/Gammon, Chapter 18). The equilibrium constant for this kind of equilibrium has a special name. It is called the *solubility product constant*.

Purpose

In this experiment, you will determine the solubility of $Ca(IO_3)_2$ (calcium iodate) and calculate its solubility product constant. You will also demonstrate that the solubility of $Ca(IO_3)_2$ is independent of the amount of water that is used to obtain a saturated solution of this substance.

Concept of the experiment

If the solubility of a slightly soluble ionic compound is measured, the solubility product constant for the substance can be calculated (Ebbing/Gammon, Example 18.3). This is the method that you will adopt for $Ca(IO_3)_2$ in this experiment. The equilibrium between solid $Ca(IO_3)_2$ and its ions in a saturated solution is

$$Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2IO_3^-(aq)$$

If some analytical technique is used to determine the concentration of either Ca^{2+} ions or IO_3^- ions in the saturated solution, the solubility of $Ca(IO_3)_2$ will be known and the solubility product constant (K_{sp}) can be calculated.

In this experiment, you will determine the concentration of IO_3^- ions through a titration with a standardized solution of $Na_2S_2O_3$ (sodium thiosulfate) in the presence of KI (potassium iodide), using starch as an indicator. A definition of standardization can be found in the experiment "An Acid–Base Titration Curve." Potassium iodide will react with the iodate ions to give I_2 as the sole product containing iodine. The reaction is

$$IO_3^-(aq) + 5I^-(aq) + 6H_3O^+(aq) \rightarrow 3I_2(aq) + 9H_2O(l)$$

The molecular iodine reacts with $S_2O_3^{2-}$ ions during the titration according to

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

where $S_4O_6^{2-}$ is the tetrathionate ion. When these two equations are combined, the net reaction occurring in the titration is obtained.

$$IO_3^-(aq) + 5I^-(aq) + 6H_3O^+(aq) \rightarrow 3I_2(aq) + 9H_2O(l)$$

$$3I_2(aq) + 6S_2O_3^{2-}(aq) \rightarrow 6I^-(aq) + 3S_4O_6^{2-}(aq)$$

$$IO_3^-(aq) + 6S_2O_3^{2-}(aq) + 6H_3O^+(aq) \rightarrow I^-(aq) + 3S_4O_6^{2-}(aq) + 9H_2O(l)$$

Starch is used as an indicator in this titration because it reacts with I_2 reversibly to form a dark blue color. I_2 is consumed in the titration, so the color fades as the titration progresses. You will know when a stoichiometric volume of the $Na_2S_2O_3$ solution has been added because at that point, one drop of that solution will cause the disappearance of the last trace of the blue color. A trial titration will allow you to find the approximate volume that is required before you do the first of two exact titrations.

Your laboratory instructor may ask you to standardize the $Na_2S_2O_3$ solution, using a known volume of a KIO_3 solution whose molarity is known accurately. If you are asked to do this, a calculation in the Prelaboratory Assignment will make the task easier. Alternatively, the standardized solution of $Na_2S_2O_3$ may be provided for you.

Procedure

Getting started

1. Obtain a 10-mL transfer pipet, a 50-mL buret, and 3 pieces of filter paper.
2. Ask your laboratory instructor whether you are to standardize the $Na_2S_2O_3$ solution.

Preparing saturated solutions of $Ca(IO_3)_2$

1. Prepare $Ca(IO_3)_2$ by adding 50 mL of 0.2 *M* KIO_3 to 20 mL of 1 *M* $Ca(NO_3)_2$ in a 150-mL beaker. Do not use the 0.0100 *M* solution of KIO_3 that will be available if you are to standardize your $Na_2S_2O_3$ solution.
2. Stir the mixture vigorously with a stirring rod. A white, crystalline precipitate of $Ca(IO_3)_2$ should form.
3. Let the mixture stand for a few minutes while you prepare for gravity filtration (see the Introduction to this manual).
4. Filter the precipitate. Rinse the remnants from the beaker onto the filter paper, using distilled water from a plastic wash bottle.
5. Wash the precipitate on the filter paper with three small portions of distilled water.
6. Place about 1/3 of the wet precipitate into a clean, labeled beaker, using a metal spatula. Place a similar portion of the precipitate into an identical beaker, using the same method. These beakers should have capacities of at least 100 mL. Save the remaining portion of the precipitate on the filter paper to use if an unforeseen accident occurs.
7. Use a graduated cylinder to add 40 mL of distilled water to the first beaker and 80 mL of distilled water to the second beaker.
8. Stir each beaker thoroughly, using separate stirring rods.
9. Allow each stirring rod to remain in its beaker and let each mixture stand for at least 30 min with occasional stirring.
10. Go on to the next parts of this experiment while you are waiting.

Cleaning and filling your buret

1. The Introduction to this manual provides instructions for using a buret. Clean your buret and fill it with the $Na_2S_2O_3$ solution, following those directions.

Standardizing the $Na_2S_2O_3$ solution (optional)

1. Pipet 10.0 mL of 0.0100 *M* KIO_3 into each of two clean 125-mL or 250-mL Erlenmeyer flasks. Do not use the 0.2 *M* solution of KIO_3.
2. Add about 20 mL of distilled water to each flask from a clean graduated cylinder.
3. Dissolve about 1 cm^3 of solid KI, as measured in a clean, dry 10-mL graduated cylinder, in each solution. Add 20 drops of 2 *M* HCl to each flask and swirl to obtain homogeneous solutions.

> **CAUTION: Handle hydrochloric acid with care. It can cause chemical burns in addition to ruining your clothing. If you spill any acid on you, wash the contaminated area thoroughly and report the incident to your laboratory instructor. You may require further treatment.**

4. Record the initial buret reading.
5. Place one of the flasks under the buret with the tip inside the mouth of the flask. Insert a piece of white paper under the flask.
6. Subtract 2 mL from the volume of the $Na_2S_2O_3$ solution that you calculated in the Prelaboratory Assignment. *Rapidly* add the resulting volume to the flask.
7. Rinse the walls of the flask with distilled water from a plastic wash bottle.
8. Add 40 drops of a 0.2% starch solution.
9. Continue the titration on a *drop-by-drop* basis. Swirl the flask rapidly after each drop. The titration will be finished when one drop causes the solution to become colorless.
10. Record the final buret reading.
11. Refill the buret, if necessary, and repeat Steps 4 through 10. If the volumes that were used in these titrations differ by more than 0.15 mL (about 3 drops), repeat the titration until two consecutive results have this precision.
12. Calculate and record the molarity from each of the two titrations. Obtain the mean molarity.

Analyzing the saturated solutions of $Ca(IO_3)_2$

1. The object of this part of the experiment is to determine the molarity of IO_3^- ions above each of the precipitates. Extraneous water must not be introduced during filtration and sampling, or the molarity of IO_3^- ions will no longer be that of a saturated solution.
2. Filter each mixture prepared in the first part of this experiment through a piece of *dry* filter paper and a *dry* filter funnel. Catch each filtrate in a clean, *dry* labeled beaker. Use a different piece of filter paper for each filtration.
3. *Do not wash* the precipitates on the filter paper.
4. Rinse the 10-mL pipet with distilled water and shake out as much water as you can. Rinse the pipet with two small portions (about 2 mL each) of the first filtrate. Discard these portions.
5. Pipet 10.0 mL of the first filtrate into a clean, labeled 125-mL or 250-mL Erlenmeyer flask.

6. Fill the buret with the $Na_2S_2O_3$ solution.
7. Add about 20 mL of distilled water to each flask from a clean graduated cylinder.
8. Dissolve about 1 cm^3 of solid KI, as measured in a clean, dry 10-mL graduated cylinder, in each solution. Add 20 drops of 2 *M* HCl to each flask and swirl to obtain homogeneous solutions.

CAUTION: Remember to handle the hydrochloric acid with care.

9. Record the initial buret reading.
10. Place one of the flasks under the buret with the tip inside the mouth of the flask. Insert a piece of white paper under the flask.
11. Add 40 drops of a 0.2% starch solution.
12. Begin a trial titration by adding increments of about 1 mL of the $Na_2S_2O_3$ solution. Swirl the solution after each addition.
13. The trial titration is complete when the addition of about 1 mL causes the solution to become colorless. Record the buret reading.
14. Repeat Steps 5 through 10 with a second sample of the first filtrate. This is an exact titration.
15. Subtract 1 mL from the volume found in the trial titration. Rapidly add the resulting volume to the flask from the buret.
16. Rinse the walls of the flask with distilled water.
17. Add 40 drops of the starch solution.
18. Continue the titration on a *drop-by-drop* basis, swirling after each drop, until one drop causes the complete disappearance of the color.
19. Record the buret reading.
20. Rinse the pipet with two small portions (about 2 mL each) of the second filtrate.
21. Using a 10.0-mL sample from the second filtrate, repeat Steps 5 through 10 and Steps 15 through 19. This is an exact titration.
22. Calculate the molarity of IO_3^- ions in each saturated solution.

A Solubility Product Constant

Date: ..... Student name: .....

Course: ..... Team members: .....

Section: .....

Instructor: .....

Prelaboratory assignment

1. Provide definitions for the following terms:

   a. Solubility

   b. Saturated solution

   c. Solubility product constant

   d. Standardization

2. a. Give the chemical equation that describes the equilibrium between calcium iodate and its ions in a saturated solution.

   b. Give the chemical equation for the net reaction that occurs during the titration.

3. a. How will you know when a stoichiometric amount of the $Na_2S_2O_3$ solution has been added in the titration?

   b. What is the purpose of the trial titration?

4. You may be required to standardize a solution of $Na_2S_2O_3$ in this experiment. The approximate molarity of this solution will be 0.025 *M*. You will take 10.0 mL of a 0.0100 *M* KIO_3 solution, dissolve excess amounts of KI in it, and, using the $Na_2S_2O_3$ solution, titrate to the disappearance of the starch–I_2 color. Using the approximate molarity of the $Na_2S_2O_3$ solution, calculate the approximate volume of the solution that will be required in the titration.

5. What safety precaution must be observed during this experiment?

A Solubility Product Constant

Date: ..... Student name: .....

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Section: .....

Instructor: .....

Results

1. *Standardizing the $Na_2S_2O_3$ solution (optional)*

Sample	**1**	**2**	**3**
KIO_3 solution taken (mL)			
Moles of KIO_3			
Final buret reading (mL)			
Initial buret reading (mL)			
Volume of $Na_2S_2O_3$ solution (mL)			
Molarity of $Na_2S_2O_3$ solution (mol/L)			
Mean molarity (mol/L)			

Calculations:

2. *Analyzing the saturated solutions*

   a. Trial titration

      Final buret reading (mL): .....

      Initial buret reading (mL): .....

      Volume of $Na_2S_2O_3$ solution (mL): .....

   b. Exact titrations

Filtrate	**1**	**2**
Final buret reading (mL)	.....	.....
Initial buret reading (mL)	.....	.....
Volume of $Na_2S_2O_3$ solution (mL)	.....	.....
Moles of $Na_2S_2O_3$ used	.....	.....
Moles of IO_3^- present initially	.....	.....
Molarity of IO_3^- (mol/L)	.....	.....

Calculations:

Student name: ..... Course/Section: ..... Date: .....

Questions

1. a. Calculate the solubility of $Ca(IO_3)_2$ from your results with the first and second filtrates.

   b. Why should these solubilities be identical? Explain.

   c. Calculate K_{sp} for $Ca(IO_3)_2$, using the mean solubility.

2. Discuss the effects of errors introduced from the following sources:

   a. The precipitate of $Ca(IO_3)_2$, when obtained originally, is not washed with distilled water.

b. The concentration of KIO_3 used for standardizing the solution of $Na_2S_2O_3$ is somewhat greater than 0.0100 *M*.

c. Extraneous water is introduced from wet filter paper or wet funnels or by washing the precipitate during the final filtration of $Ca(IO_3)_2$.

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