

EXPERIMENT

A Solubility Product Constant: Solubility product of an iodate salt

Goal

The goal of this laboratory experiment is to calculate the solubility product of a iodate salt.

Materials

- | | |
|--|--|
| <input type="checkbox"/> A 10mL pipet, a 150mL beaker, stirring rod | <input type="checkbox"/> solid KI _(s) |
| <input type="checkbox"/> Filter paper, funnel, stand, ring | <input type="checkbox"/> A 0.2M KIO _{3(aq)} solution |
| <input type="checkbox"/> A burette, stand and fisher clamp | <input type="checkbox"/> 2M HCl _(aq) and 2% starch solution |
| <input type="checkbox"/> A 1M Ca(NO ₃) _{2(aq)} solution | <input type="checkbox"/> A series of 125mL (or 250mL) erlenmeyers |

Background

When insoluble compounds dissolve in water an equilibrium between the solid and the dissolved ions establishes. Based on the stoichiometry of the compound we will obtain a different number of moles of ions in solution.

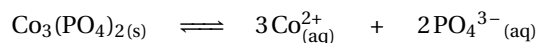
This experiment can be carried out in two different forms. The instructor can either give you an unknown saturated solution, or you can prepare it yourself. At the same time, the standardization of sodium thiosulfate can be carried out optionally, based on the instructor's indications.

Solubility equilibrium

Insoluble compounds dissolve in small amounts in water. Solubility equilibrium is the equilibrium that describes the dissolution of an insoluble compound to produce an aqueous solution of ions. For example, in the case of silver chloride, we have:



The solubility equilibrium above represents the dissolution of the insoluble salt—a solid—to produce ions, silver, and chloride in solution. In general terms, solubility equilibria start with a solid and generate ions in solution. To break down the insoluble compound into ions, we just need to take into account the stoichiometry of the compound. For example, for the case of Cobalt(II) phosphate, we have:



Solubility product in terms of molarities, K_{sp}

Silver chloride, AgCl is an insoluble compound in water which means this compound will not fully dissolve in water. Still, small quantities of the salt will indeed dissolve leading to a small amount of silver and chlorine ions. As this compound is insoluble the ion concentration will be very small. The dissociation equilibrium involved in the dissolution indicated below, is characterized by an equilibrium constant K_{sp} called *solubility product constant* or simply *solubility product*:



As pure solids are not included in any equilibrium constant, the formula above does not include $\text{AgCl}_{(s)}$. Hence, all solubility products simply result from the product of the molarities of the ions involved in the dissociation with the corresponding powers based on the stoichiometry of the salt. This is why the term *product* is included in its name. K_{sp} will have a different explicit expression depending on the formula of the insoluble compound—depending on its stoichiometry. For example, K_{sp} for calcium fluoride—a 1:2 compound—would be:



Solubility products are related to the solubility of chemicals. However, the relationship is not one-on-one. In other words, a larger K_{sp} does not necessarily imply a larger solubility. For example, K_{sp} for PbBr_2 is $6.6 \times 10^{-3}\text{M}$ whereas K_{sp} for MgCO_3 is $4.0 \times 10^{-5}\text{M}$. We have that K_{sp} for PbBr_2 is smaller than K_{sp} for MgCO_3 . However, the solubility of PbBr_2 is indeed larger than the solubility of MgCO_3 .

Relating solubility to the concentration of ions

We have that insoluble compounds dissolve to produce small amounts of ions in solutions. The concentration of these ions is directly related to solubility s using the stoichiometry of the compound. For example, for silver chloride—a salt with a 1:1 stoichiometry—we have that solubility has a 1 and 1 relationship with the concentration of ions:

$$[\text{Ag}^{+}] = s \text{ and } [\text{Cl}^{-}] = s$$

Similarly, for barium sulfate we have that solubility has a 1:1 relationship with the concentration of ions:

$$[\text{Ba}^{+2}] = s \text{ and } [\text{SO}_4^{-2}] = s$$

When the stoichiometry of the compound is not 1:1 we need to include the stoichiometry coefficients in the relationship between solubility and ion concentration. For example, for Ag_2SO_4 —a compound with 2:1 stoichiometry, we have that

$$[\text{Ag}^{+}] = 2 \cdot s \text{ and } [\text{SO}_4^{-2}] = s$$

This is because, for every mole of silver sulfate, we produce two moles of silver—and hence the factor two—and one mole of sulfate in solution. Similarly, for $\text{Nd}_2(\text{CO}_3)_3$ we have

$$[\text{Nd}^{+3}] = 2 \cdot s \text{ and } [\text{CO}_3^{-2}] = 3 \cdot s$$

Relating solubility to the concentration of ions

The solubility product is directly related to molar solubility s . We will demonstrate how to obtain this relationship using three examples. First, the solubility product of AgCl is:



As the concentration of each ion, Ag^{+} and Cl^{-} , is related to the molar solubility of the salt, we have

$$K_{sp} = [\text{Ag}^{+}] \cdot [\text{Cl}^{-}] = (s) \cdot (s) = s^2$$

Second, for silver sulfide, an insoluble compound with a more complex stoichiometry, we have:



Third, the solubility equilibrium and K_{sp} expression for $\text{Nd}_2(\text{CO}_3)_3$ is



As the ion concentrations are related to solubility, we have

$$K_{sp} = (2s)^2 \cdot (3s)^3 = 108s^5$$

K_{sp} in terms of molar solubility: general formula

For any insoluble salt A_xB_y , we have that K_{sp} is related to s by means of a general formula:

$$K_{sp} = a \cdot s^b \quad (1)$$

where:

$$a \text{ is } x^x \cdot y^y$$

$$b \text{ is } x + y$$

For example, for BaF_2 the constant a would be $1^1 \cdot 2^2$, that is four, whereas the constant b will be $1 + 2$ which equals three. As such, the expression of K_{sp} in terms of s would be: $K_{sp} = 4s^3$. This approach is useful when we need to compute the solubility product constant given the molar solubility.

Molar solubility in terms of K_{sp} : general formula

We previously explored the relationship between K_{sp} and molar solubility. Here we will explore the relationship between molar solubility and K_{sp} , simply solving for s in Equation ???. Again, for any insoluble salt A_xB_y , we have:

$$s = \left(\frac{K_{sp}}{a} \right)^{\frac{1}{b}} \quad (2)$$

where:

$$a \text{ is } x^x \cdot y^y$$

$$b \text{ is } x + y$$

For example, for BaF_2 the constant a would be $1^1 \cdot 2^2$, that is four, whereas the constant b will be $1 + 2$ which equals three. As such, the expression of s in terms of K_{sp} would be: $s = \left(\frac{K_{sp}}{4} \right)^{\frac{1}{3}}$. This approach is useful when the solubility product constant is given and we need to calculate the molar solubility. As the relation between molar solubility and K_{sp} is not a one-to-one relationship, K_{sp} is not directly related to solubility. For salts with a similar stoichiometry, it would be safe to compare solubilities in terms of K_{sp} . For example:

CuS	$K_{sp} = 8 \times 10^{-37}$	$s = 6 \times 10^{-19}$
PbS	$K_{sp} = 3 \times 10^{-28}$	$s = 1 \times 10^{-14}$
LiF	$K_{sp} = 2 \times 10^{-3}$	$s = 3 \times 10^{-2}$

and we have that the lower K_{sp} the lower solubility. When the salt stoichiometry differs

MgF ₂	$K_{sp} = 5 \times 10^{-11}$	$s = 2 \times 10^{-4}$
Li ₃ PO ₄	$K_{sp} = 2 \times 10^{-4}$	$s = 7 \times 10^{-2}$
Li ₂ CO ₃	$K_{sp} = 8 \times 10^{-4}$	$s = 6 \times 10^{-2}$

then an increase in K_{sp} does not necessarily follow an increase in solubility.

Procedure

Part A: Preparation of a saturated solution

- ☐ Step 1: – If your instructor assign you an unknow you can skip Part A.
- ☐ Step 2: – In a 150mL beaker mix 50mL of a 0.2M KIO₃ solution and 20mL of a 1M Ca(NO₃)₂ solution. Make sure you pick the reagents with the indicated concentration as in the lab there could be more diluted potassium iodate. Stir the resulting mix with a stirring rod. A precipitate will appear.
- ☐ Step 3: – Using gravity filtration (use a filter funnel and not a Büchner funnel), filter the precipitate. Make sure you rinse the beaker with water, to collect all precipitate leftovers.

- ☐ *Step 4:* – Wash the precipitate on the filter paper with three different volumes of water.
- ☐ *Step 5:* – Use a spatula to separate the precipitate into two halves. Dispose of each part in a clean labeled 100mL beaker. Put one of the beakers aside to be used in case of an accident. Use the other beaker in the rest of the experiment. If you do not use the first half of the precipitate, dispose of it at the end of the experiment following your instructor's indications.
- ☐ *Step 6:* – Add 40mL of distilled water to the beaker containing the precipitate. Use a stirring rod to stir the mixtures.
- ☐ *Step 7:* – This is the saturated solution. Let this solution rest for 30 minutes, occasionally stirring.
- ☐ *Step 8:* – It is important not to add additional water to this solution.

Part B: Standardization of thiosulfate: mixture preparation

- ☐ *Step 1:* – Locate the KIO_3 solution and write down its molarity in the results section.
- ☐ *Step 2:* – Prepare a diluted KIO_3 solution by pipetting 5mL of the KIO_3 solution into a 100mL volumetric flask and fill up with distilled water.
- ☐ *Step 3:* – Pipet 10mL of the diluted KIO_3 solution prepared in the previous step into a 125mL (or 250mL) Erlenmeyer flask. First, add 20mL of distilled water from a cylinder. Add 20 drops of 2M HCl and the equivalent of 1mL of solid KI, measured with a 10mL cylinder. Mind you can approximately measure the volume of a solid with a cylinder. Finally, add 40 drops of a 0.2% starch solution. Swirl the mixture to obtain a homogeneous mixture.
- ☐ *Step 4:* – Repeat the previous step two more times, preparing a total of three Erlenmeyers containing the iodate mixture.

Part B: Standardization of thiosulfate

- ☐ *Step 1:* – Ask your instructor if the thiosulfate solution is already standardized. If so, you can skip Part B.
- ☐ *Step 2:* – Rinse a buret with a small portion of $\text{Na}_2\text{S}_2\text{O}_3$ solution, discarding the solution used in the rinses.
- ☐ *Step 3:* – Fill a buret with $\text{Na}_2\text{S}_2\text{O}_3$ solution.
- ☐ *Step 4:* – Titrate one of the iodate samples adding increments of 1mL of sodium thiosulfate at a time. Swirl the mixture after each addition. You will have reached the equivalency point when the solution becomes colorless. Try placing the flask containing the analyte on a piece of paper. The white background will help see the color change when it occurs.
- ☐ *Step 5:* – Write down the volume of thiosulfate added to reach the equivalency point to the nearest 0.01mL. This will give an estimate of the volume needed to reach the endpoint.
- ☐ *Step 6:* – After having estimated the volume of titrate needed to reach the endpoint, we will now speed up the titration by rapidly titrating the second thiosulfate sample.
- ☐ *Step 7:* – Add thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) to a second iodate sample, stopping 3mL before the estimated endpoint.
- ☐ *Step 8:* – Add $\text{Na}_2\text{S}_2\text{O}_3$ drop by drop until you reach the endpoint and the solution becomes colorless. Record the buret reading to the nearest 0.01mL.
- ☐ *Step 9:* – Repeat the titration with the last iodate sample.

Part C: Preparing three mixtures of the saturated solution

- ☐ *Step 1:* – The goal now is to prepare three samples of an acidified, saturated solution containing iodide. To do this we will first filter the saturated solution to eliminate the solid. After that, we will add acid and iodine to an exact volume of this solution.
- ☐ *Step 2:* – Filter the saturated solution obtained earlier in the experiment. Make sure the funnel and the paper are dry. Do not wash the precipitate with water. The filtrate is the solution to be titrated.
- ☐ *Step 3:* – Rinse a 10mL pipet with distilled water making sure the pipet is free of water at the end. Now rinse the same pipet two times with a small amount of the filtrate collected in the previous step (2mL), discarding the solution from the pipet.
- ☐ *Step 4:* – Pipet 10mL from the filtrate into a 125mL (or 250mL) Erlenmeyer flask. First, add 20mL of water distilled water from a cylinder. Add 20 drops of 2M HCl and the equivalent of 1mL of solid KI, measured with a 10mL cylinder. Mind you can approximately measure the volume of a solid with a cylinder. Finally, add 40 drops of a 0.2% starch solution. Swirl the mixture to obtain a homogeneous mixture.
- ☐ *Step 5:* – Repeat the previous step two more times to prepare a total of three mixtures.

Part D: Titration of the saturated solution mixtures: trial titration

- ☐ *Step 1:* – The final goal here is to calculate the molarity of the saturated solution, which corresponds to the solubility of the insoluble compound. Here we will estimate the equivalence point of the titration of sodium thiosulfate and sodium iodate.
- ☐ *Step 2:* – Rinse a buret with a small portion of $\text{Na}_2\text{S}_2\text{O}_3$ solution, discarding the solution used in the rinses.
- ☐ *Step 3:* – Fill a buret with $\text{Na}_2\text{S}_2\text{O}_3$ solution.
- ☐ *Step 4:* – Titrate one of the samples adding increments of 1mL of sodium thiosulfate at a time. Swirl the mixture after each addition. You will have reached the equivalency point when the solution becomes colorless. Try placing the flask containing the analyte on a piece of paper. The white background will help see the color change when it occurs.
- ☐ *Step 5:* – Write down the volume of thiosulfate added to reach the equivalency point to the nearest 0.01mL. This will give an estimate of the volume needed to reach the endpoint.

Part D: Titration of the saturated solution mixtures: exact titration

- ☐ *Step 1:* – After having estimated the volume of titrate needed to reach the endpoint, we will now speed up the titration by rapidly titrating the second thiosulfate sample.
- ☐ *Step 2:* – Add thiosulfate to a second sample of saturated solution, stopping 3mL before the estimated endpoint.
- ☐ *Step 3:* – Add $\text{Na}_2\text{S}_2\text{O}_3$ drop by drop until you reach the endpoint and the solution becomes colorless. Record the buret reading to the nearest 0.01mL.
- ☐ *Step 4:* – Repeat the procedure with the last saturated sample.

Calculations

0 This is the molarity of the potassium iodate solution, c_{KIO_3} .

1 This is the initial volume on the buret.

2 This is the final volume in the buret.

3 This is the volume of sodium thiosulfate used in the standardization, $v_{\text{Na}_2\text{S}_3\text{O}_3}$:

$$v_{\text{Na}_2\text{S}_3\text{O}_3} = 2 - 1$$

4 This is the molarity of thiosulfate, $c_{\text{Na}_2\text{S}_3\text{O}_3}$:

$$c_{\text{Na}_2\text{S}_3\text{O}_3} = 10 \times c_{\text{KIO}_3}^{\text{diluted}} / v_{\text{Na}_2\text{S}_3\text{O}_3} \times 6 = 10 \times 0.05 \times 0 / 3 \times 6$$

5 This is the average sodium thiosulfate concentration. If you do not standardize the reagent this value should be written in the bottle (0.025M).

6 This is the initial volume in the buret.

7 This is the final volume in the buret.

8 This is the volume of thiosulfate used, $v_{\text{Na}_2\text{S}_3\text{O}_3}$:

$$v_{\text{Na}_2\text{S}_3\text{O}_3} = 7 - 6$$

9 This is the concentration of iodate IO_3^- in the saturated solution, $c_{\text{IO}_3^-}$:

$$c_{\text{IO}_3^-} = v_{\text{Na}_2\text{S}_3\text{O}_3} \times c_{\text{Na}_2\text{S}_3\text{O}_3} / 60 = 8 \times 5 / 60$$

10 This is the solubility of the iodate salt, s .

$$s = c_{\text{IO}_3^-} / 2 = 9 / 2$$

11 This is the average solubility.

12 You obtain the solubility product by doing:

$$s = 4 \times 11^3$$

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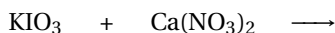
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Pre-lab Questions

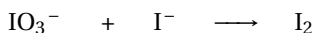
A Solubility Product Constant: Solubility product of an iodate salt

1. In the experiment, you will react potassium iodate with calcium nitrate to produce a precipitate. Indicate the name of the precipitate formed:



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2. In this experiment the iodate (IO_3^-) and iodide ions (I^-) react to produce molecular iodine (I_2) in acidic medium. Hence, in this redox reaction, a reactant is converted into products by simultaneous oxidation and reduction reactions. This type of redox reaction is referred to as disproportionation. Balance the corresponding disproportionation reaction.



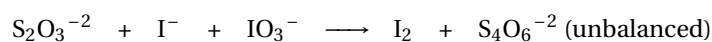
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3. In this experiment thiosulfate ($\text{S}_2\text{O}_3^{2-}$) is used in a redox titration. This compound reacts with molecular iodine (I_2), which has a reddish-brown color, to produce the colorless iodide (I^-) and tetrathionate. Hence, when all thiosulfate is finished along with the titration the reaction becomes colored. Balance the corresponding redox reaction in acidic medium.



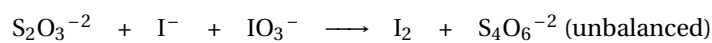
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4. Both reactions in the previous questions happen consecutively during the experiment. In the redox titration, first iodate reacts with iodine to produce iodine, which also reacts with thiosulfate to produce iodine. Combine both reactions to generate the overall reaction happening in the titration.



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5. You need 25mL of sodium thiosulfate 0.01M to reach the equivalency point in the titration of a 10mL sample of iodate. Using the balanced reaction from the previous question, carry stoichiometric calculations to calculate the molarity of iodate in the solution.



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**Results
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Part B: $\text{Na}_2\text{S}_2\text{O}_3$ standarization

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① Molarity of KIO_3 , $c_{\text{KIO}_3} =$

	1	2	3	4
① Initial Buret Volume (mL)				
② Final Buret Volume (mL)				
③ $\text{Na}_2\text{S}_2\text{O}_3$ Volume used (mL)				
④ $\text{Na}_2\text{S}_2\text{O}_3$ Concentration (M)				

⑤ Mean $\text{Na}_2\text{S}_2\text{O}_3$ Concentration (M) =

Part D: Titration of saturated solution

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⑤ Molarity of $\text{Na}_2\text{S}_2\text{O}_3$, $c_{\text{Na}_2\text{S}_2\text{O}_3} =$

		1	2	3	4
⑥	Initial Buret Volume (mL)				
⑦	Final Buret Volume (mL)				
⑧	$\text{Na}_2\text{S}_2\text{O}_3$ Volume used (mL)				
⑨	IO_3^- Concentration (M)				
⑩	Solubility (M)				

Unknown Number = _____

⑪ Mean solubility (M) =

⑫ Mean K_{sp} =

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Post-lab Questions

A Solubility Product Constant: Solubility product of an iodate salt

1. Write down the solubility equilibrium of calcium iodate.

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2. Write down the expression of the solubility constant of calcium iodate in terms of ion molarities.

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3. Write down the expression of the solubility constant of calcium iodate in terms of solubilities.

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4. Write down the expression of the solubility of calcium iodate in terms of the solubility product constant.

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