9. Determination of the solubility product of KIO₄ and an investigation of the common ion effect

Student Sheet

This experiment will allow you to calculate a value for K_{sp}, and also to see the common ion effect in operation.

Intended learning outcomes

At the end of this practical and its write up you should be able to:

- Make up standard solutions and prepare equilibrium mixtures
- Plot a graph
- Calculate concentrations and deduce K_{sp} values
- Calculate experimental errors and apparatus errors and use these to evaluate experimental accuracy
- Consider the implications of apparatus errors when undertaking a planning exercise.
- Understand the Common Ion Effect

Safety

Materials safety data sheets should be consulted so that the correct action can be taken in event of a spillage and/or accident.

	You must wear eye protection throughout this experiment
	Sodium nitrate is **oxidizing** and **harmful**
	Potassium nitrate is **oxidizing**
	Potassium iodate is **oxidising** and **irritant**
	Sulphuric acid is **corrosive**.

Background Theory

Potassium iodate(VII) is sparingly soluble in water (its saturated solution is about 0.02 mol dm⁻³).
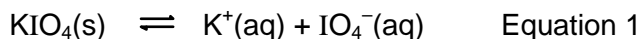
$$KIO_4(s) \rightleftharpoons K^+(aq) + IO_4^-(aq) \qquad \text{Equation 1}$$

Its solubility product is given by the expression:

$$K_{sp} = [K^+(aq)][IO_4^-(aq)] \qquad \text{Equation 2}$$

Its solubility (call it "s") is related to K_{sp} as follows:

$$s = [KIO_4(aq)] = [K^+(aq)] = [IO_4^-(aq)]$$

$$\therefore\ K_{sp} = s^2 \ \text{ or } \ s = \sqrt{K_{sp}}$$

The Common Ion Effect

If either K^+ ions or IO_4^- ions were to be added to the saturated solution of KIO_4 then, by Le Chatelier's principle, Equilibrium 1 would be displaced to the left. This would produce more solid KIO_4, and so the solubility of KIO_4 in this solution would be reduced. In other words, since the value of K_{sp} in Equation 1 is **constant**, if the $[K^+]$ were increased (for example, by the addition of another potassium salt), the $[IO_4^-]$ would *decrease* in order to keep K_{sp} the same. The ion which has been added, in this case the K^+ ion, is termed the **Common Ion**, as it is the same as one of the ions present in KIO_3. The decrease in solubility, which results when a **common ion** is added to a saturated solution of a sparingly soluble salt, is called the **Common Ion Effect**.

There is just one complication: The solubilities of ionic compounds in water are dependent on how many other ions (ions that may be totally unrelated to those in the salt) are in solution. To avoid complicated calculations, this experiment is designed to keep the **ionic strength** of the solution constant, by using the inert salt sodium nitrate in just the right amounts.

Basic principles

Solid potassium iodate(VII) is shaken with an aqueous mixture of $NaNO_3$ and KNO_3 (see table below) until equilibrium is reached. The excess solid is allowed to settle and the solution is filtered. A sample of the filtrate is taken, using a pipette, and its $[IO_4^-]$ is measured by titration.

Determining the concentration of IO_4^- ions in the filtrate

When acid and an excess of potassium iodide are added to the sample of filtrate, iodine is produced according to the reaction shown below:

$$IO_4^- \ + \ 8H^+ \ + \ 7I^- \ \rightarrow \ 4I_2 \ + \ 4H_2O$$

This solution of iodine is then titrated with a standard solution of sodium thiosulphate.

$$2S_2O_3^{2-} \ + \ I_2 \ \rightarrow \ S_4O_6^{2-} \ + \ 2I^-$$
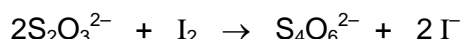
By considering both of the above equations, we can see that **1 mole** of IO_4^- is equivalent to **8 moles** of $S_2O_3^{2-}$. Thus, the $[IO_4^-]$ may be deduced.

As the addition of KNO_3 drives Equilibrium 1 to the left, the K^+ ions present when a new equilibrium is established will be those from the KNO_3 added, together with any K^+ ions present from Equation 1.

Method

This practical exercise is in two parts: part **A**, preparing the solutions prior to titration, and part **B**, performing the titrations. Part **A** may be done communally, but part **B** should be done individually or in pairs.

Obtain or prepare the following solutions:

Sodium nitrate: 500 cm^3 of 0.2 mol dm^{-3} (dissolve 8.50 g of the solid in distilled water and make up to 500 cm^3 in a volumetric flask)

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Potassium nitrate   250 cm^3 of 0.2 mol dm^{-3} (dissolve 5.05 g of the solid in distilled water and make up to 250 cm^3 in a volumetric flask)

Sodium thiosulphate 0.0500 mol dm^{-3} solution (this will be prepared for you)

Potassium iodide   1.0 mol dm^{-3} solution   (about 300 cm^3 may be needed in all)

Part A – making up the mixtures

- Take six 250 cm^3 bottles with stoppers. Weigh about 1 g of KIO$_4$ into each. Label them **A** to **F**.

- To each bottle add the appropriate quantities of aqueous sodium nitrate and aqueous potassium nitrate given in the table below. Use a 100 cm^3 measuring cylinder to measure out the NaNO$_3$ solutions. Use another 100 cm^3 measuring cylinder to measure out the KNO$_3$ solution for bottles **A** and **B**, but use a burette to measure out the KNO$_3$ solution for bottles **C** to **F**.

bottle	A	B	C	D	E	F
volume of NaNO$_3$ / cm^3	0	50	75	90	95	100
volume of KNO$_3$ / cm^3	100	50	25	10	5	0

- Place the stoppers tightly into the bottles and, keeping your finger over the stopper, shake each bottle for about 3–4 minutes to allow time for equilibrium to be reached. Allow the bottles to stand undisturbed until the excess solid mostly settles to the bottom. Try not to disturb the solid too much in the next step.

- Filter each solution through a dry, labelled, 250 cm^3 conical flask using a new dry filter paper each time. Place a 10 cm^3 pipette in or by each flask, and **do not mix them up during the subsequent titrations!**

Part B – titrating the solutions

- Fill a burette with the 0.0500 mol dm^{-3} sodium thiosulphate solution.
- Using a pipette, transfer 10.0 cm^3 of solution **A** into a 100 cm^3 conical flask.
- Using **separate** measuring cylinders, transfer to this flask about 5 cm^3 of 1.0 mol dm^{-3} KI and about 5 cm^3 of dilute sulphuric acid.
- Titrate the iodine produced in the solution with sodium thiosulphate from the burette, adding starch indicator near the endpoint (when the solution reaches a pale yellow colour).
- Repeat the above process with the solutions from all the other bottles, **using the bottle's own pipette each time, to avoid cross-contamination.**
- Your titre volumes should range from around 35 cm^3 down to about 2 cm^3, depending on the bottle you are titrating. So, be careful you do not overshoot the end-point!
- Enter your results in the table below.
- Complete your table by calculating the values for the remaining six columns.

  Note: The K$^+$ ions in each equilibrium solution come from two sources. Some are present in the KNO$_3$ solution added to each flask. The remainder come from the dissolved KIO$_4$. The total [K$^+$] is calculated by adding the [K$^+$] from the KNO$_3$ solution used, to the [K$^+$] from the dissolved KIO$_4$. The latter [K$^+$] will be the same as the [IO$_4^-$]

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The [K$^+$] from the KNO$_3$ = $\dfrac{\text{volume of KNO}_3 \text{ added} \times [\text{KNO}_3]}{\text{total volume}}$

$= \dfrac{\text{volume of KNO}_3 \text{ added} \times 0.200}{100}$

The [K$^+$] remaining from the saturated KIO$_4$ = [IO$_4^-$]

Total [K$^+$] = [IO$_4^-$] + $\dfrac{\text{volume of KNO}_3 \text{ added}}{500}$

the K_{sp} value of the solution in each flask is calculated using K_{sp} = [K$^+$][IO$_4^-$] mol dm^{-3}

Flask	initial burette reading / cm^3	final burette reading / cm^3	volume of thiosulphate used / cm^3	moles of thiosulphate used / mol	moles of IO$_4^-$ in 10 cm^3 sample / mol	[IO$_4^-$] / mol dm^{-3}	[K$^+$] / mol dm^{-3}	K_{sp} / mol^2 dm^{-6}
A								
B								
C								
D								
E								
F								

Analysis and evaluation of results

1 Plot a graph of [IO$_4^-$] (i.e. the solubility) against [K$^+$], drawing a line of best fit through your points. By considering the distribution of your plotted points around the line of best fit, comment on the quality of your results.

2 Calculate the mean of the six K_{sp} values in your table.

3 The data book value for the K_{sp} of KIO$_4$ (at 298 K) is 1.07×10^{-2} mol^2 dm^{-6}. Calculate the difference between your mean K_{sp} value and the 'book' value. Express this difference as a percentage of the 'book' value. This is your total experimental error.

4 Assuming that the maximum errors for the apparatus used in this experiment are as shown below, the total apparatus error is obtained by adding together the maximum percentage error in using each piece of apparatus.

You should use the titre to calculate the error in the burette and the volume of solution transferred in the pipette to calculate the error in the pipette.

Pipette = $\pm$ 0.06 cm^3

Burette = $\pm$ 0.10 cm^3 (from two readings)

Select the experiment in your table whose K_{sp} value is closest to the 'book' value. Use the data from **this** experiment to calculate the total apparatus error for **this** experiment.

5 By comparing the total experimental error with the total apparatus error, comment on the accuracy of **this** experiment.

6   Calculate the value of the total equipment error for Experiment **A** and for Experiment **F**.

- Explain why they are so different. Suggest why it would be unreasonable to expect the K_{sp} value obtained in experiment **A** to be as accurate as that obtained in experiment **F**.

- What might be learned from this when planning the quantities and apparatus to be used in a quantitative investigation?

7   The range of titration results in your experiments **A** – **F**, will be wide. In terms of the Common Ion Effect, explain why this is so.

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9. Determination of the solubility product of KIO_4 and an investigation of the common ion effect

### Teachers' Notes

Intended lesson outcomes

These are detailed on the pupil sheet.

Safety

The main points are included on the pupil sheet but it is the teacher's responsibility to ensure that a full risk assessment is carried out prior to the practical session.

Background Theory

Potassium iodate(VII) is sparingly soluble in water.
(Its saturated solution is about 0.02 mol dm^{-3}.)

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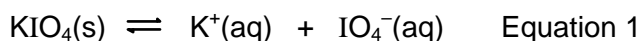
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$$\therefore K_{sp} = s^2 \text{ or } s = \sqrt{K_{sp}}$$

You may need to emphasise that the above relationship, $K_{sp} = s^2$, only holds true when no additional common ions are present. In the presence of additional common ions, Equation 1 is driven to the left. This results in the concentration of the common ion will be greater than was present before the addition (because more has been added), while the concentration of the other ion will be less than it was originally as the equilibrium has moved to the left, and more KIO_4 has precipitated. Thus, $[KIO_4(aq)] \neq [K^+(aq)]$, so $K_{sp} = s^2$ fails. Equation 2 must now be used.

There is no real need to spend time discussing the significance of the ionic strength of a solution. Such a discussion will add little to the understanding of the class and may well cause confusion.

It is critical that the solutions are made up with precision and are allowed to come to equilibrium before use. The actual time required will depend on temperature and so the solutions should be left as long as possible before use.

Each sample should be filtered prior to use. Care must be exercised to prevent cross-contamination; so clean apparatus and a new filter paper should be used each time. The analysis of each filtrate is done by treating a 10.0 cm^3 portion with excess aqueous potassium iodide and dilute sulphuric acid, and titrating the iodine produced against standard sodium thiosulphate solution. It might prove useful to review the chemistry involved in these processes before the experiment starts.

The analysis and evaluation of results section gives pupils the opportunity to practice their graphical skills but also requires them to evaluate their results critically. They should identify any anomalous points and interpret the scatter of their points relative to the best fit line to evaluate the reliability of their results (point 1).

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The comparison of their mean K_{sp} value with the data book value (points 2 & 3) will give a measure of experimental accuracy. However, as each of the six experiments will have a different titre value, the total apparatus error will be different in each case. To overcome this problem, pupils are instructed to select the experiment that generates the K_{sp} value closest to the data book K_{sp} value (points 4 & 5). Here, as there is an actual titre value, the apparatus error can be calculated and the comparison between apparatus error and experimental error can be made.

Before the class tackles the errors analysis, it would be worth spending a little time reviewing this process. Assuming that the experimental error is greater than the total apparatus error, students should conclude that the additional error is caused by problems in the procedure used, or in the level of skill shown of the operator. You may wish to extend the exercise at this point by asking your students to identify the source(s) of these additional errors and to suggest steps that might be taken to eliminate or reduce them.

The aim of points 5 & 6 is to draw attention to the critical importance of the size of the titre value in determining the apparatus error. In experiment A, the titre will be small, as the high $[KNO_3]$ will drive Equilibrium1 strongly to the left (leaving a low $[IO_4^-]$). The maximum burette error will, therefore, be high. The opposite argument applies in experiment E.

A discussion of this point with the class should result in sensible volumes being suggested when planning quantitative investigations.

Point 7 tests students' understanding of the Common Ion Effect.

Technical Information

Requirements per student/group

Apparatus

- six 250 cm^3 bottles with stoppers
- access to a balance weighing to 0.01 g
- access to a burette containing aqueous potassium nitrate
- a burette to hold the sodium thiosulphate solution
- six 10 cm^3 pipettes – one only needed if it is washed between experiments
- filter papers and filter funnels
- one 500 cm^3 volumetric flask – not needed if $NaNO_3$(aq) made up centrally
- one 250 cm^3 volumetric flask – not needed if KNO_3(aq) made up centrally
- one 100 cm^3 conical flask
- two 10 cm^3 measuring cylinders
- two 100 cm^3 measuring cylinders

Chemicals

- about 10 g of solid sodium nitrate (or 500 cm^3 of 0.200 mol dm^{-3} solution)
- about 6 g of solid potassium nitrate (or 250 cm^3 of 0.200 mol dm^{-3} solution)
- about 10 g of solid potassium iodate(VII) [periodate]
- about 200 cm^3 of 0.0500 mol dm^{-3} sodium thiosulphate solution
- about 100 cm^3 of 1.0 mol dm^{-3} potassium iodide solution
- about 100 cm^3 of 1.0 mol dm^{-3} sulphuric acid

Safety

The main points are included on the Student Sheet but it is the teacher's responsibility to ensure that a full risk assessment is carried out prior to the practical session. MSDS sheets should be consulted so that the correct action can be taken in event of a spillage and/or accident.

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