

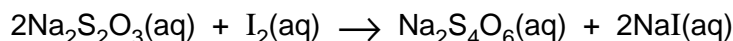


- 1 **FA 1** is an aqueous solution containing  $23.72 \text{ g dm}^{-3}$  of sodium thiosulphate,  $\text{Na}_2\text{S}_2\text{O}_3$ .  
**FA 2** is a solution of an oxidant, **X**, containing  $5.15 \text{ g dm}^{-3}$  of **X**.  
**FA 3** is a solution containing potassium iodide, KI.  
**FA 4** is  $1.00 \text{ mol dm}^{-3}$  sulphuric acid,  $\text{H}_2\text{SO}_4$ .

In the presence of acid, the oxidant **X** oxidises iodide ions to iodine.

1 mole of **X** produces 3 moles of iodine,  $\text{I}_2$

The iodine liberated can then be titrated with thiosulphate ions,  $\text{S}_2\text{O}_3^{2-}$ , to reduce the iodine back to iodide.



You are to determine the relative molecular mass of the oxidant **X**.

- (a) Pipette  $25.0 \text{ cm}^3$  of **FA 2** into a conical flask. Use the measuring cylinder provided to add an excess of iodide ions (approximately  $10 \text{ cm}^3$  of **FA 3**), and  $10 \text{ cm}^3$  of sulphuric acid, **FA 4**.

Titrate the iodine produced in the conical flask with **FA 1**. As the titration proceeds the colour of the iodine in solution will diminish. The end-point is reached when the colour disappears and the solution becomes colourless.

**There is no need to add starch indicator to find the end-point.**

Record your results in Table 1.1.

**Repeat the titration as many times as you think necessary to obtain accurate results.**

**Make certain that the recorded results show the precision of your practical work.**

**Table 1.1 Titration of Iodine with FA 1**

final burette reading / $\text{cm}^3$				
initial burette reading / $\text{cm}^3$				
volume of <b>FA 1</b> used / $\text{cm}^3$				

[2] + [6]

### Summary

$25.0 \text{ cm}^3$  of **FA 2** produced sufficient iodine to react with .....  $\text{cm}^3$  of **FA 1**.

Show which results you used to obtain this volume of **FA 1** by placing a tick (✓) under the readings in Table 1.1.

You are advised to show full working in all parts of the calculations.

- (b) Calculate how many moles of sodium thiosulphate,  $\text{Na}_2\text{S}_2\text{O}_3$ , were run from the burette during the titration.  
[ $A_r$ : Na, 23.0; S, 32.1; O, 16.0.]

[2]

- (c) Calculate how many moles of iodine,  $\text{I}_2$ , react with the sodium thiosulphate run from the burette.

[1]

- (d) Calculate how many moles of oxidant **X** were placed in the titration flask at the beginning of the titration.

[1]

- (e) Calculate the concentration, in  $\text{mol dm}^{-3}$ , of the oxidant **X** in **FA 2**.

[1]

- (f) Calculate the relative molecular mass,  $M_r$ , of the oxidant **X**.

[2]

[Total: 15]

- 2 **FA 5** contains **two cations** and **one anion** from the following list: ( $\text{Al}^{3+}$ ,  $\text{NH}_4^+$ ,  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Zn}^{2+}$ ;  $\text{CO}_3^{2-}$ ,  $\text{CrO}_4^{2-}$ ,  $\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$ ,  $\text{NO}_3^-$ ,  $\text{NO}_2^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{SO}_3^{2-}$ ).

In all tests, the reagent should be added gradually, with shaking after each addition.  
Record your observations in the spaces provided.

Your answers should include

- details of colour changes and precipitates formed,
- the names of gases evolved and details of the test used to identify each one.

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

**No additional or confirmatory tests for ions present should be attempted.**

**Candidates are reminded that definite deductions may be made from tests where there appears to be no reaction.**

Test	Observations [7]
<p><b>(a)</b> To 2 cm depth of <b>FA 5</b> in a test-tube, add 1 cm depth of aqueous silver nitrate.</p> <p>Leave the mixture to stand and continue with tests <b>(b)</b> to <b>(e)</b>.</p>	
<p><b>(b)</b> To 2 cm depth of <b>FA 5</b> in a boiling-tube, add 4 cm depth of aqueous sodium hydroxide. Stir thoroughly with the glass rod provided.</p> <p>Filter the mixture and retain the filtrate for tests <b>(c)</b>, <b>(d)</b> and <b>(e)</b>.</p>	
<p>Observe the residue in the filter paper after it has been exposed to the air for a few minutes.</p>	
<p><b>(c)</b> To 1 cm depth of the filtrate from <b>(b)</b> in a test-tube, add 2 cm depth of dilute nitric acid followed by aqueous silver nitrate.</p>	
<p><b>(d)</b> To 1 cm depth of the filtrate from <b>(b)</b> in a test-tube, add 2 cm depth of dilute hydrochloric acid followed by aqueous barium chloride.</p>	

Test	Observations
<p>(e) Place 1 cm depth of the filtrate from (b) in a boiling-tube and warm the tube gently.  <b>Take care as a solution containing sodium hydroxide may bump on heating and eject hot corrosive sodium hydroxide.</b></p>	
<p>(f) Observe the mixture left to stand in test (a).</p>	
<p>Use a teat pipette to remove the solution from the precipitate formed, then add 2 cm depth of distilled water to wash the precipitate. Allow the precipitate to settle and again use a teat pipette to remove the solution.</p> <p>Dissolve the solid in 2 cm depth of dilute aqueous nitric acid. You may need to cautiously warm the mixture. Use this solution in the test below.</p>	
<p>Add dilute hydrochloric acid to the solid dissolved in nitric acid.</p>	

Use the information in the Qualitative Analysis Tables on pages 6 and 7 to identify the ions present in **FA 5**.

The **cations** present in **FA 5** are ..... and .....

The **anion** present in **FA 5** is ..... [1]

Which observations support your choice of these ions?

.....  
 ..... [1]

What is the identity of the solid formed and dissolved in test (f)? Give a reason.

.....  
 ..... [1]

[Total: 10]

## QUALITATIVE ANALYSIS NOTES

[Key: ppt. = precipitate]

### 1 Reactions of aqueous cations

ion	reaction with	
	NaOH(aq)	NH <sub>3</sub> (aq)
aluminium, Al <sup>3+</sup> (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH <sub>4</sub> <sup>+</sup> (aq)	ammonia produced on heating	
barium, Ba <sup>2+</sup> (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca <sup>2+</sup> (aq)	white ppt. with high [Ca <sup>2+</sup> (aq)]	no ppt.
chromium(III), Cr <sup>3+</sup> (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu <sup>2+</sup> (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe <sup>2+</sup> (aq)	green ppt. insoluble in excess	green ppt. insoluble in excess
iron(III), Fe <sup>3+</sup> (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
lead(II), Pb <sup>2+</sup> (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
magnesium, Mg <sup>2+</sup> (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn <sup>2+</sup> (aq)	off-white ppt. insoluble in excess	off-white ppt. insoluble in excess
zinc, Zn <sup>2+</sup> (aq)	white ppt. soluble in excess	white ppt. soluble in excess

[Lead(II) ions can be distinguished from aluminium ions by the insolubility of lead(II) chloride.]

## 2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, $\text{CO}_3^{2-}(\text{aq})$	$\text{CO}_2$ liberated by dilute acids
chromate(VI), $\text{CrO}_4^{2-}(\text{aq})$	yellow solution turns orange with $\text{H}^+(\text{aq})$ ; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$ ; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$ ); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$ ); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$ ); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
nitrate, $\text{NO}_3^-(\text{aq})$	$\text{NH}_3$ liberated on heating with $\text{OH}^-(\text{aq})$ and $\text{Al}$ foil
nitrite, $\text{NO}_2^-(\text{aq})$	$\text{NH}_3$ liberated on heating with $\text{OH}^-(\text{aq})$ and $\text{Al}$ foil, $\text{NO}$ liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown $\text{NO}_2$ in air)
sulphate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acid)
sulphite, $\text{SO}_3^{2-}(\text{aq})$	$\text{SO}_2$ liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acid)

## 3 Tests for gases

<i>gas</i>	<i>test and test result</i>
ammonia, $\text{NH}_3$	turns damp red litmus paper blue
carbon dioxide, $\text{CO}_2$	gives a white ppt. with limewater (ppt. dissolves with excess $\text{CO}_2$ )
chlorine, $\text{Cl}_2$	bleaches damp litmus paper
hydrogen, $\text{H}_2$	pops with a lighted splint
oxygen, $\text{O}_2$	relights a glowing splint
sulphur dioxide, $\text{SO}_2$	turns potassium dichromate(VI) (aq) from orange to green

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