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## 9701/31

**May/June 2008**

**2 hours**

Additional Materials: As listed in the Instructions to Supervisors

Write your Centre number, candidate number and name on all the work you hand in.  
Give details of the practical session and laboratory where appropriate, in the boxes provided.  
Write in dark blue or black pen.  
You may use a soft pencil for any diagrams, graphs or rough working.  
Do **not** use staples, paper clips, highlighters, glue or correction fluid.  
**DO NOT WRITE IN ANY BARCODES.**

Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 11 and 12.

At the end of the examination, fasten all your work securely together.  
The number of marks is given in brackets [ ] at the end of each question or part question.

<b>Session</b>
<b>Laboratory</b>

For Examiner's Use	
1	
2	
Total	

This document consists of **11** printed pages and **1** blank page.

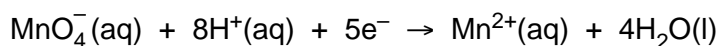
# 1 Read through question 1 before starting any practical work.

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You are provided with the following reagents.

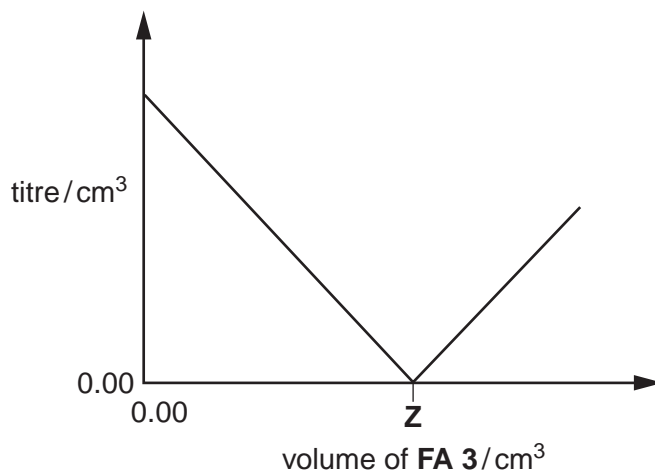
- **FA 1** containing  $15.68 \text{ g dm}^{-3}$  of hydrated ammonium iron(II) sulphate  $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$
- **FA 2**,  $0.015 \text{ mol dm}^{-3}$  potassium manganate(VII),  $\text{KMnO}_4$
- **FA 3** containing  $0.025 \text{ mol dm}^{-3}$  of a reagent **X**
- $1.0 \text{ mol dm}^{-3}$  sulphuric acid,  $\text{H}_2\text{SO}_4$

Iron(II) ions,  $\text{Fe}^{2+}$ , are oxidised by acidified manganate(VII) ions.



Reagent **X** oxidises  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  and is also oxidised by acidified  $\text{MnO}_4^-$ .

If varying volumes of **FA 3**, containing reagent **X**, are added to  $25.0 \text{ cm}^3$  of **FA 1** in the presence of  $\text{H}_2\text{SO}_4$  and the mixtures are titrated against **FA 2**, a graph of the results can be drawn as shown.



You are to determine experimentally

- **Z**, the exact volume of **FA 3** which reacts with  $25.0 \text{ cm}^3$  of **FA 1**,
- the mole ratio for the reaction of **FA 1** with reagent **X**.

## (a) Method

- Fill a burette with **FA 2**.
- Pipette  $25.0 \text{ cm}^3$  of **FA 1** into a conical flask.
- Use a measuring cylinder to add approximately  $10 \text{ cm}^3$  of  $1.0 \text{ mol dm}^{-3}$   $\text{H}_2\text{SO}_4$  to the solution in the flask.
- Titrate the **FA 1** in the flask with **FA 2** until the first permanent pink colour remains in the solution.

The end-point should be found after the addition of approximately  $13 \text{ cm}^3$  of **FA 2**.

**One** titration, performed accurately, will be sufficient.

You are reminded that just before the end-point the pink colour from a single drop of **FA 2** spreads through the whole of the solution before disappearing.

Record your titration results in the space below.

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[1]

**(b) Method**

- Fill the second burette with **FA 3**.
- Empty and rinse the conical flask used in part **(a)**.
- Pipette 25.0 cm<sup>3</sup> of **FA 1** into the conical flask and add 10 cm<sup>3</sup> of H<sub>2</sub>SO<sub>4</sub> using a measuring cylinder.
- Run 12.00 cm<sup>3</sup> of **FA 3** from the second burette into the flask.
- Titrate against **FA 2** until the first permanent pink colour remains in the solution.

The end-point should be found after the addition of approximately 5 cm<sup>3</sup> of **FA 2**.

**One** titration, performed accurately, will be sufficient.

You are reminded that just before the end-point the pink colour from a single drop of **FA 2** spreads through the whole of the solution before disappearing.

Record your titration results in the space below.

[1]

- (c)** The volume of **FA 3** added to the flask in **(b)** reacts with some but not all of the **FA 1** present. Calculate the difference between the titres obtained in parts **(a)** and **(b)**. Use this difference and the volume of **FA 3** added to the flask in **(b)** to calculate the volume of **FA 3** that you would expect to react with all of the Fe<sup>2+</sup> ions in 25.0 cm<sup>3</sup> of **FA 1**.

volume of **FA 3** = ..... cm<sup>3</sup>  
[1]

- (d) The value you have obtained in (c) is an **approximate** value of **Z**. You are to perform four more titrations, **each** with a **different** volume of **FA 3** added to 25.0 cm<sup>3</sup> of **FA 1**, in order to plot a graph of the form shown on page 2 and to obtain an exact value for **Z**.

**One** titration, performed accurately, will be sufficient for each volume of **FA 3** added.

The volume of **FA 3** you have obtained in (c) will help you to choose suitable volumes of **FA 3** to be added for each titration.

(If you were unable to calculate the volume of **FA 3** in (c) assume that the value lies in the range 19.0 cm<sup>3</sup> to 21.0 cm<sup>3</sup>.)

Remember

- you should not use more than 40.0 cm<sup>3</sup> of **FA 3** for any single titration,
- you already have data for titrations with no **FA 3** added and with 12.00 cm<sup>3</sup> added.

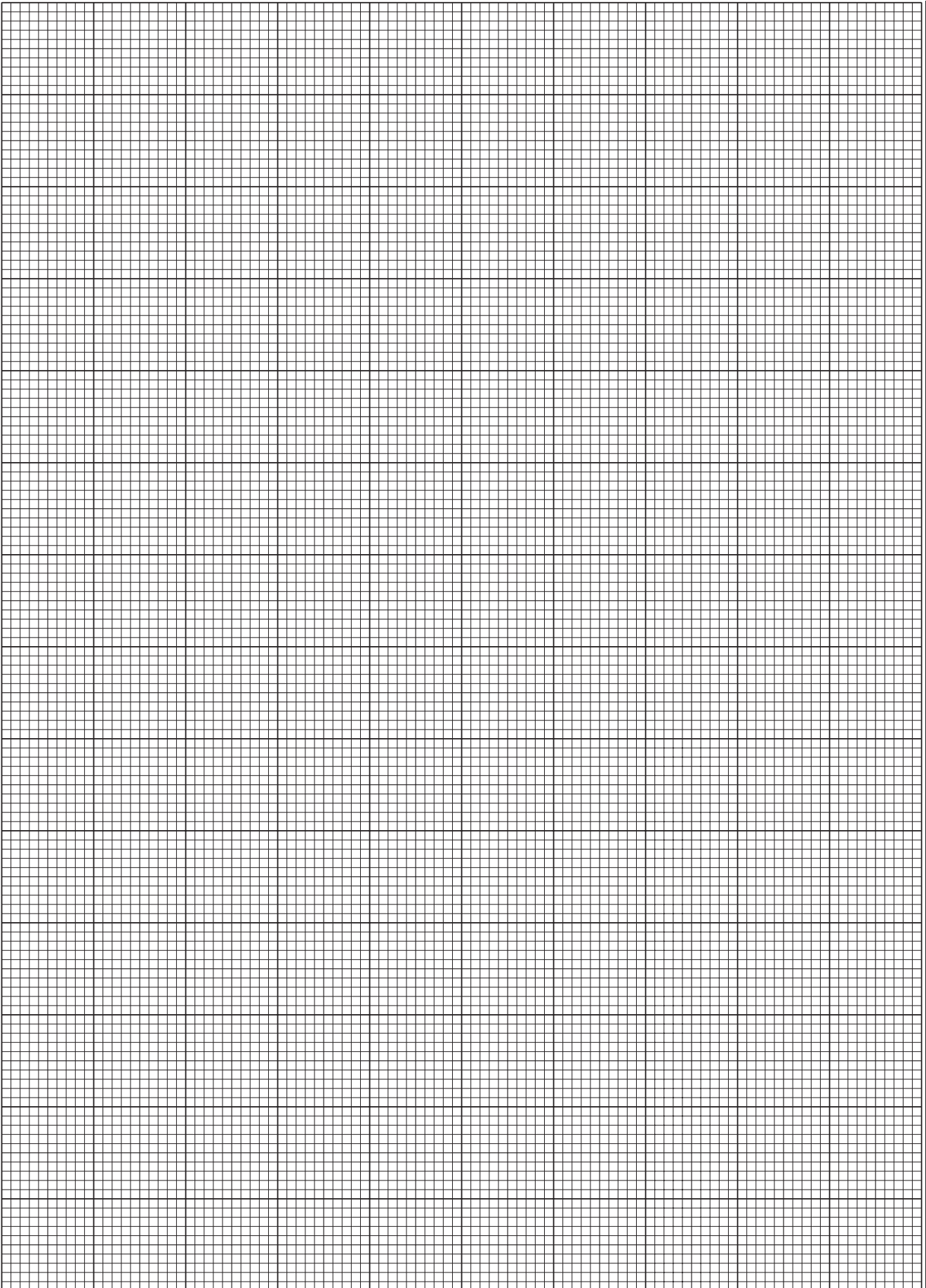
Prepare a table in the space below and use it to record the titration results for each volume of **FA 3** added. Include in your table the titre values from parts (a) and (b).

[6]

- (e) Use the grid on the opposite page to plot a graph of titre against volume of **FA 3** added.

Draw **two** straight lines through the plotted points to find **Z**, the volume of **FA 3** that just reacts with the Fe<sup>2+</sup> ions in 25.0 cm<sup>3</sup> of **FA 1**.

**Z**, the volume of **FA 3** read from the graph is ..... cm<sup>3</sup>.



- (f) Circle on the graph **one** point where a repeat titration might be appropriate and justify your decision. If you do not think that any titration needs to be repeated, explain why you have come to that conclusion.

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.....

.....

.....

[1]

### Calculations

Show your working and appropriate significant figures in **all** of your calculations. [2]

- (g) Calculate how many moles of  $\text{Fe}^{2+}$  ions were pipetted into the flask.  
[ $A_r$ : Fe, 55.8; H, 1.0; N, 14.0; O, 16.0; S, 32.1]

..... mol of  $\text{Fe}^{2+}$  were pipetted into the flask. [2]

- (h) Calculate how many moles of **X** are present in **Z**, the volume of **FA 3** read from your graph.

..... mol of **X** were present in .....  $\text{cm}^3$  of **FA 3**. [1]

- (i) Calculate, to 3 significant figures, the number of moles of  $\text{Fe}^{2+}$  ions that react with 1 mol of **X**.

[2]

[Total: 24]

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- 2 The three solutions **FA 4**, **FA 5**, and **FA 6** each contain **one** of the following.
- aluminium sulphate,  $Al_2(SO_4)_3$
  - ammonium iodide,  $NH_4I$
  - zinc nitrate,  $Zn(NO_3)_2$

- (a) Use the information on page 12 to select two suitable reagents to use to discover which solution contains iodide ions.

Record, in the space below, the reagents used and the observations made.

From these tests, solution **FA** ..... contains iodide ions. [5]

You are to perform the tests given in the table opposite on each of **FA 4**, **FA 5** and **FA 6** to identify, where possible, the cation and anion present in each solution.

Record details of colour changes seen, the formation of any precipitate and the solubility of any such precipitate in an excess of the reagent added.

**Where gases are released they should be identified by a test**, described in the appropriate place in your table.

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

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

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



test	observations with <b>FA 4</b>	observations with <b>FA 5</b>	observations with <b>FA 6</b>
<b>(b)</b> To 1 cm depth of solution in a test-tube, add aqueous sodium hydroxide drop-by-drop until it is in excess.			
<b>(c)</b> To 1 cm depth of solution in a test-tube, add aqueous ammonia drop-by-drop until it is in excess.			
<b>(d)</b> To 1 cm depth of solution in a test-tube, add aqueous barium chloride,  then			
add dilute hydrochloric acid.			
<b>(e)</b> To 1 cm depth of solution in a boiling-tube add 2 cm depth of water and 1 cm depth of aqueous lead(II) nitrate,  then			
if a precipitate has formed, cautiously warm until the solution boils,  then			
cool the tube by standing it in a beaker of cold water.			

[6]

- (f) For each of the solutions **FA 4**, **FA 5**, and **FA 6**, summarise the evidence from the tests performed to identify the cations and anions present. State clearly where a cation or an anion has **not** been specifically identified.

**FA 4** contains .....

supporting evidence

.....  
 .....  
 .....

**FA 5** contains .....

supporting evidence

.....  
 .....  
 .....

**FA 6** contains .....

supporting evidence

.....  
 .....  
 .....

[4]

- (g) When testing a solution containing both  $\text{NH}_4\text{I}$  and  $\text{Zn}(\text{NO}_3)_2$ , suggest why a student should identify the  $\text{NH}_4^+$  ion before attempting to identify the  $\text{NO}_3^-$  ion. The Qualitative Analysis Notes on pages 11 and 12 should help you to answer this.

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

[Total: 16]

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

## 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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