

CANDIDATE  
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**CHEMISTRY**

**9701/31**

Paper 3 Advanced Practical Skills 1

**May/June 2015**

**2 hours**

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

**MODIFIED LANGUAGE**

**READ THESE INSTRUCTIONS FIRST**

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 an HB pencil for any diagrams or graphs.  
Do not use staples, paper clips, glue or correction fluid.  
**DO NOT WRITE IN ANY BARCODES.**

Answer **all** questions.  
Electronic calculators may be used.  
You may lose marks if you do not show your working or if you do not use appropriate units.  
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 10 and 11.

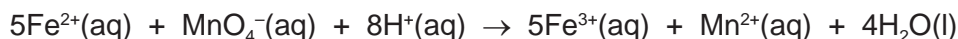
At the end of the examination, fasten all your work securely together.  
The number of marks is given in brackets [ ] at the end of each question or part question.

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

<b>For Examiner's Use</b>	
<b>1</b>	
<b>2</b>	
<b>3</b>	
<b>Total</b>	

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

- 1 In this question you will determine the concentration of iron(II) ions in **FA 2**. To do this you will do a titration using potassium manganate(VII) solution. The iron(II) ions,  $\text{Fe}^{2+}$ , are oxidised by the manganate(VII) ions,  $\text{MnO}_4^-$ .



When all the  $\text{Fe}^{2+}$  ions have been oxidised, the presence of unreacted  $\text{MnO}_4^-$  ions causes the solution to become a permanent pink colour.

**FA 1** contains  $0.0200 \text{ mol dm}^{-3}$  manganate(VII) ions,  $\text{MnO}_4^-$ .

**FA 2** is a solution containing iron(II) ions,  $\text{Fe}^{2+}$ .

**FA 3** is  $1.0 \text{ mol dm}^{-3}$  sulfuric acid,  $\text{H}_2\text{SO}_4$ .

**(a) Method**

- Fill the burette with **FA 1**.
- Use the pipette to transfer  $25.0 \text{ cm}^3$  of **FA 2** into the conical flask.
- Use the  $25 \text{ cm}^3$  measuring cylinder to add  $10 \text{ cm}^3$  of **FA 3** to the conical flask.
- Add **FA 1** from the burette into the conical flask until the solution becomes a permanent pink colour.
- Perform a **rough titration** and record your burette readings in the space below.

The rough titre is .....  $\text{cm}^3$ .

- Do as many accurate titrations as you think necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record in a suitable form below all of your burette readings and the volume of **FA 1** added in each accurate titration.

**Keep FA 2 to use in Question 3.**

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b) From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you obtained this value.

25.0 cm<sup>3</sup> of **FA 2** required ..... cm<sup>3</sup> of **FA 1**. [1]

**(c) Calculations**

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of manganate(VII) ions present in the volume of **FA 1** calculated in (b).

moles of MnO<sub>4</sub><sup>-</sup> = ..... mol

- (ii) Calculate the number of moles of iron(II) ions present in 25.0 cm<sup>3</sup> of **FA 2**.

moles of Fe<sup>2+</sup> = ..... mol

- (iii) Calculate the concentration, in mol dm<sup>-3</sup>, of iron(II) ions in **FA 2**.

concentration of Fe<sup>2+</sup> in **FA 2** = ..... mol dm<sup>-3</sup>

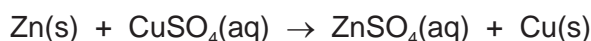
- (iv) **FA 2** was prepared by dissolving hydrated ammonium iron(II) sulfate, (NH<sub>4</sub>)<sub>2</sub>Fe(SO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O in distilled water. Calculate the mass of salt that would have to be dissolved in 1.00 dm<sup>3</sup> of water to prepare **FA 2**.  
(A<sub>r</sub>: H, 1.0; N, 14.0; O, 16.0; S, 32.1; Fe, 55.8)

I	
II	
III	
IV	

mass of (NH<sub>4</sub>)<sub>2</sub>Fe(SO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O = ..... g  
[4]

[Total: 12]

- 2 In this experiment you will measure the heat given out by the reaction of excess zinc with copper(II) sulfate solution and use this to estimate the concentration of the copper(II) sulfate.



**FA 4** is zinc powder.

**FA 5** is aqueous copper(II) sulfate,  $\text{CuSO}_4$ .

**(a) Method**

Read through the instructions carefully and prepare a table below for your results before starting any practical work.

- Support the plastic cup in the  $250\text{ cm}^3$  beaker.
- Use the  $50\text{ cm}^3$  measuring cylinder to transfer  $40\text{ cm}^3$  of **FA 5** into the plastic cup.
- Measure and record the initial temperature of the solution in the plastic cup.
- Start the stopwatch. Measure and record the temperature of the solution every 30 seconds up to and including the temperature at 2 minutes. Stir the solution frequently.
- At time  $t = 2\frac{1}{2}$  minutes, add **all** the powdered zinc to the solution in the plastic cup and stir the mixture.
- Record the temperature every 30 seconds from  $t = 3$  minutes up to and including  $t = 9$  minutes. Stir the solution constantly.

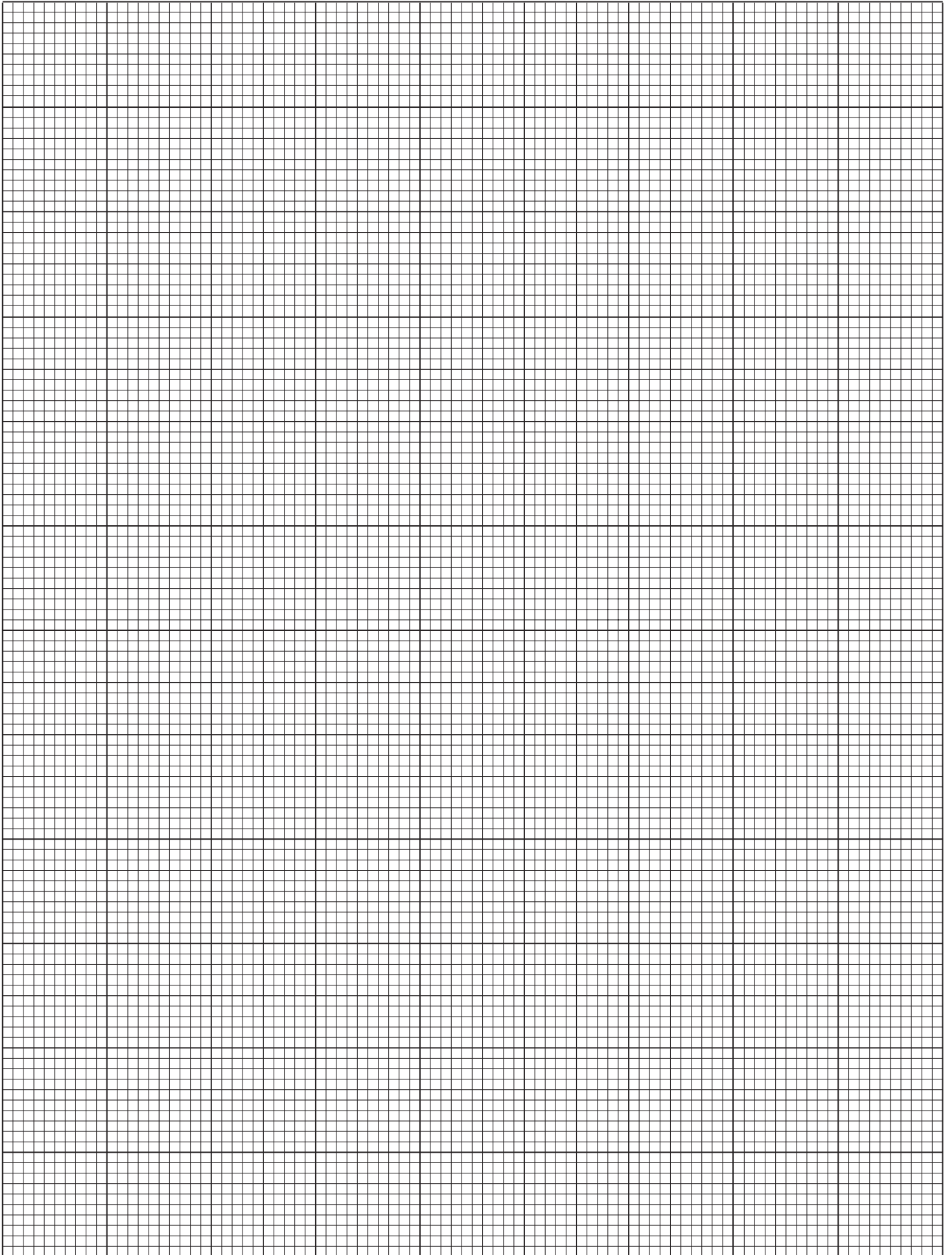
I	
II	
III	
IV	

[4]

- (b) (i)** On the grid opposite, plot the temperature ( $y$ -axis) against the time ( $x$ -axis). The scale for the temperature axis must allow you to plot a point with a temperature  $5^\circ\text{C}$  greater than the maximum temperature you recorded.
- (ii)** Draw the following best-fit **straight** lines on the graph.
- a line through the points between time  $t = 0$  minutes and time  $t = 2$  minutes
  - a line through the points between time  $t = 5$  minutes and time  $t = 9$  minutes
  - a vertical line at time  $t = 2\frac{1}{2}$  minutes
- (iii)** Extrapolate the first two straight lines so that they intersect the vertical line at time  $t = 2\frac{1}{2}$  minutes.  
Use these extrapolated lines to determine the theoretical temperature **change** at time  $t = 2\frac{1}{2}$  minutes.

change in temperature = .....  $^\circ\text{C}$

[5]



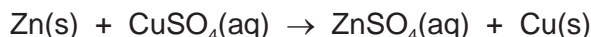
**(c) Calculations**

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Use your answer to **(b)(iii)** to calculate the heat energy produced in the reaction.  
(Assume that **4.2 J** are required to increase the temperature of 1 cm<sup>3</sup> of solution by 1 °C.)

heat energy produced = ..... J

- (ii) The molar enthalpy change,  $\Delta H$ , for the reaction shown below is  $-219 \text{ kJ mol}^{-1}$ .



Use this value and your answer to **(i)** to calculate the number of moles of copper(II) sulfate in your reaction.

moles of  $\text{CuSO}_4$  = ..... mol

- (iii) Use your answer to **(ii)**, to calculate the concentration of copper(II) sulfate, in  $\text{mol dm}^{-3}$ , in **FA 5**.

concentration of  $\text{CuSO}_4$  = .....  $\text{mol dm}^{-3}$   
[3]

- (d) (i) Calculate the maximum percentage error in the highest temperature that you recorded in your results table.

maximum percentage error = ..... %

- (ii) A student suggested that the concentration of the copper(II) sulfate could be determined more accurately if a greater mass of zinc had been used.  
Explain whether you agree with this student.

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

- (iii) A second student suggested that the concentration of the copper(II) sulfate could be determined more accurately if a smaller volume of copper(II) sulfate was used.  
Explain whether you agree with this student.

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

[3]

[Total: 15]

### 3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates 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 observations.**

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.**

**If any solution is warmed, a boiling tube MUST be used.**

Rinse and reuse test-tubes and boiling tubes where possible.

**Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.**

(a) In **Question 1** you used **FA 2**. This solution was prepared from hydrated ammonium iron(II) sulfate,  $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ .

To a 1 cm depth of **FA 2** in a test-tube, add a small spatula measure of sodium carbonate.

Record your observations.

Solutions containing  $\text{Fe}^{2+}$  ions can quickly be oxidised in air if they are prepared by dissolving the solid in distilled water.

Use your observations to suggest what other substance was added to solid

$(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  to prepare **FA 2**.

.....

[2]

- (b) **FA 6** is a mixture of two salts, each of which contains a single cation and a single anion from those listed in the Qualitative Analysis Notes on pages 10 and 11. Do the following tests and record your observations in the table below.

<i>test</i>	<i>observations</i>
(i) Place a small spatula measure of <b>FA 6</b> in a hard-glass test-tube and heat strongly.	
(ii) Place a small spatula measure of <b>FA 6</b> in a test-tube and carefully add dilute sulfuric acid until the reaction is complete, then	
add aqueous sodium hydroxide.	
(iii) To a 3 cm depth of distilled water in a boiling tube, add the remaining sample of <b>FA 6</b> . Stir and then filter the mixture into a clean boiling tube. You will use this solution for tests (iv)–(vi).	X
(iv) To a 1 cm depth of the solution from (iii) in a test-tube, add aqueous sodium hydroxide.	
(v) To a 1 cm depth of the solution from (iii) in a test-tube, add aqueous ammonia.	
(vi) To a 1 cm depth of the solution from (iii) in a test-tube, add aqueous barium chloride or aqueous barium nitrate.	



(vii) Suggest possible identities for the ions present in **FA 6**.

cations .....

anions .....

(viii) Describe a further test that would allow you to determine exactly which anions are present. Explain your choice. Do **not** do this test.

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

[11]

[Total: 13]

## 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)	no ppt. 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. turning brown on contact with air insoluble in excess	green ppt. turning brown on contact with air insoluble in excess
iron(III), Fe <sup>3+</sup> (aq)	red-brown ppt. insoluble in excess	red-brown 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. rapidly turning brown on contact with air insoluble in excess	off-white ppt. rapidly turning brown on contact with air insoluble in excess
zinc, Zn <sup>2+</sup> (aq)	white ppt. soluble in excess	white ppt. soluble in excess

## 2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, $\text{CO}_3^{2-}$	$\text{CO}_2$ liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$ )
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$ )
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$ )
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)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	$\text{SO}_2$ liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong 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
sulfur dioxide, $\text{SO}_2$	turns acidified aqueous potassium manganate(VII) from purple to colourless

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