

CANDIDATE  
NAME

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**CHEMISTRY**

**9701/31**

Paper 3 Advanced Practical Skills 1

**October/November 2015**

**2 hours**

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

**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.  
A copy of the Periodic Table is printed on page 12.

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 **12** printed pages and **1** insert.

- 1 In this experiment you will determine the ionic equation for the reaction of acidified potassium manganate(VII) with potassium iodide. Excess potassium iodide is used and the reaction produces iodine. The amount of iodine produced is measured by titration with sodium thiosulfate.

**FA 1** is  $0.0180 \text{ mol dm}^{-3}$  potassium manganate(VII),  $\text{KMnO}_4$ .

**FA 2** is  $1.00 \text{ mol dm}^{-3}$  sulfuric acid,  $\text{H}_2\text{SO}_4$ .

**FA 3** is  $0.500 \text{ mol dm}^{-3}$  potassium iodide, KI.

**FA 4** is  $0.100 \text{ mol dm}^{-3}$  sodium thiosulfate,  $\text{Na}_2\text{S}_2\text{O}_3$ .  
starch indicator

**(a) Method**

- Pipette  $25.0 \text{ cm}^3$  of **FA 1** into a conical flask.
- Use the measuring cylinder to add  $25 \text{ cm}^3$  of **FA 2** to the conical flask.
- Use the measuring cylinder to add  $20 \text{ cm}^3$  of **FA 3** to the conical flask.
- Fill the burette with **FA 4**.
- Carry out a rough titration. When the colour of the mixture becomes yellow/orange, add a few drops of starch indicator. Then titrate until the mixture goes colourless.
- Record all your burette readings in the space below.

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

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make sure 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 4** added in each accurate titration.

**Keep FA 1 and FA 2 for use in Question 3 and FA 4 for use in Question 2.**

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b)** From your accurate titration results, obtain a suitable value for the volume of **FA 4** to be used in your calculations.  
Show clearly how you have obtained this value.

Volume of **FA 4** required is .....  $\text{cm}^3$ . [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 sodium thiosulfate in the volume of **FA 4** calculated in (b).

moles of  $\text{Na}_2\text{S}_2\text{O}_3 = \dots\dots\dots$  mol

- (ii) Use the equation below to calculate the number of moles of iodine that reacted with the sodium thiosulfate in the titration.



moles of  $\text{I}_2 = \dots\dots\dots$  mol

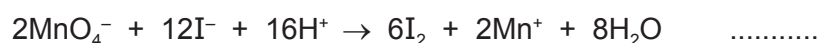
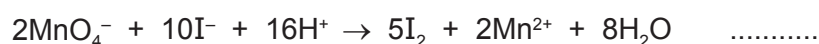
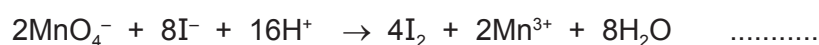
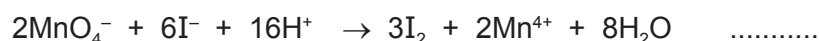
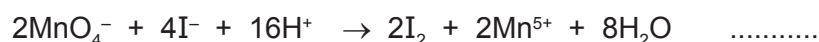
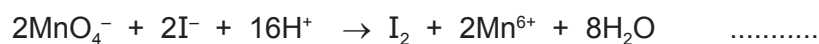
- (iii) Use information on page 2 to calculate the number of moles of potassium manganate(VII) in **FA 1** used in the titration.

moles of  $\text{KMnO}_4 = \dots\dots\dots$  mol

- (iv) From your answers to (ii) and (iii), calculate the number of moles of iodine produced by the reaction of **2.00** moles of potassium manganate(VII) with excess potassium iodide.

moles  $\text{I}_2 = \dots\dots\dots$  mol

- (v) Using your answer to (iv), put a tick next to the ionic equation that represents the reaction between **FA 1** and **FA 3**.



(vi) Prove that the iodide ion has been oxidised in the equation that you selected in (v).

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

[5]

(d) (i) The error in calibration of the pipette you used is  $\pm 0.06 \text{ cm}^3$ .  
Calculate the percentage error when measuring **FA 1**, using the pipette.

percentage error = ..... %

(ii) A student suggested that the experiment would be more accurate if a pipette was used to measure solution **FA 3**.  
State and explain whether you agree with the student.

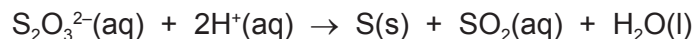
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[2]

[Total: 15]

- 2 In this experiment you will investigate how the rate of reaction between sodium thiosulfate and hydrochloric acid is affected by the concentration of the acid.

When aqueous thiosulfate ions react with hydrogen ions,  $\text{H}^+$ , in any acid, a pale yellow precipitate of sulfur is formed. The ionic equation for this reaction is given below.



The rate of the reaction can be determined by measuring the time taken to produce a fixed quantity of sulfur.

**FA 4** is  $0.10 \text{ mol dm}^{-3}$  sodium thiosulfate,  $\text{Na}_2\text{S}_2\text{O}_3$ .

**FA 5** is  $0.20 \text{ mol dm}^{-3}$  hydrochloric acid,  $\text{HCl}$ .

**(a) Method**

Record **all** your measurements, in an appropriate form, in the space below.

**Experiment 1**

- Use the larger measuring cylinder to transfer  $40 \text{ cm}^3$  of **FA 4** into the  $100 \text{ cm}^3$  beaker.
- Rinse the larger measuring cylinder thoroughly with water, then add  $30 \text{ cm}^3$  of **FA 5** to the beaker and start timing **immediately**.
- Stir the mixture once and place the beaker on top of the printed insert page provided.
- Look down through the solution in the beaker at the print on the insert.
- Stop timing as soon as the precipitate of sulfur makes the print on the insert invisible.
- Record the reaction time to the **nearest second**.
- Empty and rinse the  $100 \text{ cm}^3$  beaker.
- Dry the outside of the beaker ready for Experiment 2.

**Experiment 2**

- Rinse the larger measuring cylinder, then use it to transfer  $40 \text{ cm}^3$  of **FA 4** into the  $100 \text{ cm}^3$  beaker.
- Use the smaller measuring cylinder to add  $10 \text{ cm}^3$  of distilled water to the beaker.
- Use the same measuring cylinder to add  $20 \text{ cm}^3$  of **FA 5** to the mixture in the beaker and start timing **immediately**.
- Stir the mixture once and place the beaker on top of the printed insert page provided.
- Stop timing as soon as the print on the insert becomes invisible.
- Record the reaction time to the **nearest second**.
- Empty and rinse the  $100 \text{ cm}^3$  beaker.
- Dry the outside of the beaker ready for Experiment 3.

**Experiment 3**

- Carry out the reaction using a mixture of  $40 \text{ cm}^3$  of **FA 4**,  $20 \text{ cm}^3$  of distilled water and  $10 \text{ cm}^3$  of **FA 5**.
- Measure and record the reaction time to the **nearest second**.

I	
II	
III	
IV	

[4]

- (b) (i) The 'rate of reaction' can be represented by the formula below.

$$\text{'rate of reaction'} = \frac{1000}{\text{reaction time}}$$

Use this formula to calculate the 'rate of reaction' for Experiments 1 and 3.  
Give the unit.

'rate of reaction' for Experiment 1 ..... unit .....

'rate of reaction' for Experiment 3 ..... unit .....

- (ii) Calculate the initial concentrations of hydrochloric acid in the reaction mixtures in Experiments 1 and 3.

initial concentration of HCl in Experiment 1 = ..... mol dm<sup>-3</sup>

initial concentration of HCl in Experiment 3 = ..... mol dm<sup>-3</sup>

- (iii) How is the 'rate of reaction' affected by the concentration of hydrochloric acid in the mixture?

.....  
.....

- (iv) Predict how the reaction time measured in Experiment 1 would have been affected if the experiment had been carried out using 0.20 mol dm<sup>-3</sup> sulfuric acid instead of 0.20 mol dm<sup>-3</sup> hydrochloric acid.

Explain your answer.

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

- (v) Predict how the reaction time measured in Experiment 3 would have been affected if the experiment had been carried out in a 250 cm<sup>3</sup> beaker instead of a 100 cm<sup>3</sup> beaker.

Explain your answer.

.....  
.....

[5]

[Total: 9]

### 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.

**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) **FA 6** is a sodium compound containing one anion listed on page 11.

Dissolve the **FA 6** provided in about 15 cm<sup>3</sup> of distilled water in a boiling tube. Carry out the following tests and record your observations in the table below.

<i>test</i>	<i>observations</i>
<p>(i) To a 1cm depth of the solution of <b>FA 6</b> in a test-tube, add a few drops of aqueous barium chloride or aqueous barium nitrate, then</p> <hr style="border-top: 1px dashed black;"/> <p>add dilute hydrochloric acid.</p>	
<p>(ii) To a 1cm depth of the solution of <b>FA 6</b> in a test-tube, add an equal volume of aqueous hydrogen peroxide, then</p> <hr style="border-top: 1px dashed black;"/> <p>add a few drops of aqueous barium chloride or aqueous barium nitrate, then</p> <hr style="border-top: 1px dashed black;"/> <p>add dilute hydrochloric acid.</p>	

<i>test</i>	<i>observations</i>
<p>(iii) To a 1 cm depth of the solution of <b>FA 6</b> in a boiling tube, add an equal volume of <b>FA 2</b>, sulfuric acid, then</p> <p>heat the mixture <b>gently and cautiously</b>.</p>	
<p>(iv) To a 1 cm depth of the solution of <b>FA 6</b> in a test-tube, add an equal volume of aqueous sodium hydroxide, then</p> <p>add a few drops of <b>FA 1</b>, aqueous potassium manganate(VII), then</p> <p>add <b>FA 2</b>, sulfuric acid.</p>	

(v) Identify the anion in **FA 6**, and state **one** piece of evidence for your identification.

anion .....

evidence .....

.....

.....

(vi) Give the chemical equation for the reaction between **FA 6** and hydrogen peroxide,  $\text{H}_2\text{O}_2$ , in test (ii). State symbols are **not** required.

.....

[7]



- (b) **FA 7**, **FA 8**, **FA 9** and **FA 10** each contain one cation from the list on page 10. You will attempt to identify the cations by testing with aqueous sodium hydroxide and aqueous ammonia. In each case, use a 1 cm depth of the solution in a test-tube.

(i) Complete the table below.

test	observations			
	FA 7	FA 8	FA 9	FA 10
add sodium hydroxide				
add aqueous ammonia				

- (ii) Use your observations to identify, as far as possible, the cation present in each solution. If alternative identities are possible, state this clearly.

FA 7 cation .....

FA 8 cation .....

FA 9 cation .....

FA 10 cation .....

- (iii) Give the ionic equation for the reaction of **one** of your cations with a few drops of sodium hydroxide. State symbols are **not** required.

.....

- (iv) The precipitates obtained when alkalis are added to solutions of certain cations are sometimes difficult to see. Suggest how, using no additional apparatus, the experiment could be repeated in a way that would make these precipitates more visible.

.....

.....

[9]

[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)	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

