

CANDIDATE  
NAME

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CENTRE  
NUMBER

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CANDIDATE  
NUMBER

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**CHEMISTRY**

**9701/33**

Paper 3 Advanced Practical Skills 1

**October/November 2016**

**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.

Qualitative Analysis Notes are printed on pages 10 and 11.  
A copy of the Periodic Table is printed on page 12.

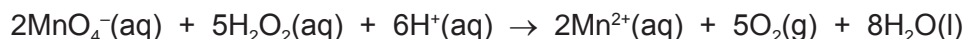
At the end of the examination, fasten all your work securely together.  
The number of marks is given in brackets [ ] at the end of each question or part question.

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

<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 Hydrogen peroxide,  $\text{H}_2\text{O}_2$ , can be oxidised to give oxygen,  $\text{O}_2$ . This reaction happens rapidly in the presence of acidified potassium manganate(VII),  $\text{KMnO}_4$ .



You will determine the concentration of a solution of hydrogen peroxide. You will first dilute the solution and then carry out a titration using acidified potassium manganate(VII),  $\text{KMnO}_4$ .

**FA 1** is aqueous hydrogen peroxide,  $\text{H}_2\text{O}_2$ .

**FA 2** is  $0.0200 \text{ mol dm}^{-3}$  potassium manganate(VII),  $\text{KMnO}_4$ .

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

### (a) Method

#### Dilution

- Pipette  $25.0 \text{ cm}^3$  of **FA 1** into the  $250 \text{ cm}^3$  volumetric flask.
- Add distilled water to make  $250 \text{ cm}^3$  of solution and shake the flask thoroughly.
- Label this diluted solution of hydrogen peroxide **FA 4**.

#### Titration

- Fill the burette with **FA 2**.
- Rinse the pipette thoroughly with distilled water and then with a little **FA 4**.
- Pipette  $25.0 \text{ cm}^3$  of **FA 4** into a conical flask.
- Use the measuring cylinder to add  $25 \text{ cm}^3$  of **FA 3** to the conical flask.
- Perform a rough titration and record 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 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 2** added in each accurate titration.

**Keep FA 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 2** to be used in your calculations. Show clearly how you obtained this value.

25.0 cm<sup>3</sup> of **FA 4** required ..... cm<sup>3</sup> of **FA 2**. [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 potassium manganate(VII) present in the volume of **FA 2** calculated in (b).

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

- (ii) Use your answer to (i) and the equation at the top of page 2 to calculate the number of moles of hydrogen peroxide present in 25.0 cm<sup>3</sup> of **FA 4**.

moles of  $\text{H}_2\text{O}_2$  = ..... mol

- (iii) Calculate the concentration, in mol dm<sup>-3</sup>, of  $\text{H}_2\text{O}_2$  in **FA 4**.

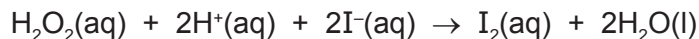
concentration of  $\text{H}_2\text{O}_2$  in **FA 4** = ..... mol dm<sup>-3</sup>

- (iv) Calculate the concentration, in mol dm<sup>-3</sup>, of  $\text{H}_2\text{O}_2$  in **FA 1**.

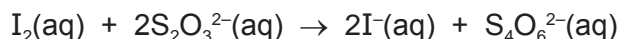
concentration of  $\text{H}_2\text{O}_2$  in **FA 1** = ..... mol dm<sup>-3</sup>  
[4]

[Total: 12]

- 2 In **Question 1**, hydrogen peroxide was oxidised to form oxygen. Hydrogen peroxide can also be reduced to form water. This occurs, for example, when hydrogen peroxide,  $\text{H}_2\text{O}_2$ , reacts with iodide ions,  $\text{I}^-(\text{aq})$ , to form iodine,  $\text{I}_2(\text{aq})$ .



The rate of this reaction can be measured by adding acidified hydrogen peroxide,  $\text{H}_2\text{O}_2$ , to a mixture of iodide ions,  $\text{I}^-$ , thiosulfate ions,  $\text{S}_2\text{O}_3^{2-}$ , and starch indicator. As the iodine is produced, it reacts immediately with the thiosulfate ions and is reduced back to iodide ions.



When all the thiosulfate has reacted, the iodine then turns the starch indicator blue-black. The rate of reaction may be determined by timing how long it takes the reaction mixture to turn blue-black.

In this experiment you will measure two reaction times and use one of these to calculate the concentration of the thiosulfate ions in **FA 6**.

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

**FA 4** is the diluted hydrogen peroxide,  $\text{H}_2\text{O}_2$ , you prepared in **1(a)**.

**FA 5** is  $1.0 \text{ mol dm}^{-3}$  potassium iodide,  $\text{KI}$ .

**FA 6** is aqueous sodium thiosulfate,  $\text{Na}_2\text{S}_2\text{O}_3$ .

starch indicator

**Read through the instructions carefully before starting any practical work.**

**(a) Method**

- Use the measuring cylinder to transfer  $20 \text{ cm}^3$  of **FA 3** into the  $100 \text{ cm}^3$  beaker.
- Use the measuring cylinder to add  $10 \text{ cm}^3$  of **FA 4** into the  $100 \text{ cm}^3$  beaker.
- Rinse the measuring cylinder with distilled water and drain.
- Use the measuring cylinder to transfer  $20 \text{ cm}^3$  of **FA 5** into the  $250 \text{ cm}^3$  beaker.
- Use the measuring cylinder to transfer  $20 \text{ cm}^3$  of **FA 6** into the  $250 \text{ cm}^3$  beaker.
- Use the measuring cylinder to transfer  $10 \text{ cm}^3$  of starch indicator into the  $250 \text{ cm}^3$  beaker.
- Add the contents of the  $100 \text{ cm}^3$  beaker to the  $250 \text{ cm}^3$  beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second.
- Rinse both beakers and drain.

**Keep all FA solutions and starch indicator for use in (c).**

reaction time = ..... s [2]

**(b) Calculations**

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

- (i) The expression for the rate of reaction is shown.

$$\text{rate} = \frac{\text{concentration of iodine at the reaction time}}{\text{reaction time}}$$

The concentration of iodine at the reaction time is the concentration of iodine that would have been present at the time the blue-black colour appeared if no thiosulfate ions had been added.

In this experiment, you should assume the rate =  $2.61 \times 10^{-5} \text{ mol dm}^{-3} \text{ s}^{-1}$ .

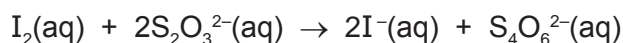
Use this value for the rate to calculate the concentration of iodine that would have been present at the reaction time if no thiosulfate ions had been added.

concentration of  $\text{I}_2$  = .....  $\text{mol dm}^{-3}$

- (ii) Use your answer to (i) and the total reaction volume, to calculate the number of moles of iodine that would have been present at the reaction time if no thiosulfate ions had been added.  
(If you were unable to calculate an answer to (i), you should use the value of  $1.32 \times 10^{-3} \text{ mol dm}^{-3}$ . This may not be the correct value.)

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

- (iii) Calculate the number of moles of thiosulfate ions that reacted with the moles of  $\text{I}_2$ , calculated in (ii).



moles of  $\text{S}_2\text{O}_3^{2-}$  = ..... mol

- (iv) Use your answer to (iii) to calculate the concentration of thiosulfate ions in **FA 6**.

concentration of  $\text{S}_2\text{O}_3^{2-}$  in **FA 6** = .....  $\text{mol dm}^{-3}$   
[4]

(c) Repeat the experiment in (a) using the following quantities of each reagent.

- add to the 100 cm<sup>3</sup> beaker  
20 cm<sup>3</sup> of **FA 3**  
10 cm<sup>3</sup> of **FA 4**
- add to the 250 cm<sup>3</sup> beaker  
20 cm<sup>3</sup> of **FA 5**  
10 cm<sup>3</sup> of **FA 6**  
10 cm<sup>3</sup> of starch indicator  
10 cm<sup>3</sup> of distilled water
- Add the contents of the 100 cm<sup>3</sup> beaker to the 250 cm<sup>3</sup> beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second.

reaction time = ..... s [1]

(d) (i) Explain the relationship between the value of the reaction time in (a) and the value of the reaction time in (c).

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

(ii) A student states that the error in the **total volume** of the reaction mixture in (a) is the same as the error in the **total volume** of the reaction mixture in (c).

State whether or not you agree with the student and explain your answer.

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

[4]

[Total: 11]

### 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 7, FA 8, FA 9** and **FA 10** are solutions that each contain a metal cation from those listed in the Qualitative Analysis Notes on page 10.

Carry out tests using reagents that will allow you to identify which metal ions are present.

Draw a single table to record your observations.

Complete the table below with your conclusions.

solution	<b>FA 7</b>	<b>FA 8</b>	<b>FA 9</b>	<b>FA 10</b>
metal ion present				

[11]

(b) Carry out the following test and record your observations.

<i>test</i>	<i>observations</i>
To a 1 cm depth of <b>FA 10</b> in a boiling tube add a 0.5 cm depth of 20 'vol' hydrogen peroxide, then add <b>one</b> drop of aqueous sodium hydroxide.	

[2]

(c) **FA 11** is a solid that contains one anion from those listed in the Qualitative Analysis Notes on page 11.

Place all of **FA 11** into a boiling tube and add a 3 cm depth of distilled water. Stir until all the solid has dissolved.

(i) Carry out the following test and record your observations.

<i>test</i>	<i>observations</i>
To a 1 cm depth of the solution of <b>FA 11</b> in a test-tube add dilute sulfuric acid.	

(ii) Use your observations from (i) to identify the anion in **FA 11**.

The anion is .....

(iii) Use the remaining solution of **FA 11** in the boiling tube to carry out a further test to support your identification of the anion.  
Record details of this test.

[4]

[Total: 17]





## 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	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, $\text{SO}_3^{2-}(\text{aq})$	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

## The Periodic Table of Elements

		Group															
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">1 H hydrogen 1.0</div> <div style="border: 1px solid black; padding: 2px;">2 He helium 4.0</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">3 Li lithium 6.9</div> <div style="border: 1px solid black; padding: 2px;">4 Be beryllium 9.0</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">5 B boron 10.8</div> <div style="border: 1px solid black; padding: 2px;">6 C carbon 12.0</div> <div style="border: 1px solid black; padding: 2px;">7 N nitrogen 14.0</div> <div style="border: 1px solid black; padding: 2px;">8 O oxygen 16.0</div> <div style="border: 1px solid black; padding: 2px;">9 F fluorine 19.0</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">11 Na sodium 23.0</div> <div style="border: 1px solid black; padding: 2px;">12 Mg magnesium 24.3</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">13 Al aluminium 27.0</div> <div style="border: 1px solid black; padding: 2px;">14 Si silicon 28.1</div> <div style="border: 1px solid black; padding: 2px;">15 P phosphorus 31.0</div> <div style="border: 1px solid black; padding: 2px;">16 S sulfur 32.1</div> <div style="border: 1px solid black; padding: 2px;">17 Cl chlorine 35.5</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">19 K potassium 39.1</div> <div style="border: 1px solid black; padding: 2px;">20 Ca calcium 40.1</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">21 Sc scandium 45.0</div> <div style="border: 1px solid black; padding: 2px;">22 Ti titanium 47.9</div> <div style="border: 1px solid black; padding: 2px;">23 V vanadium 50.9</div> <div style="border: 1px solid black; padding: 2px;">24 Cr chromium 52.0</div> <div style="border: 1px solid black; padding: 2px;">25 Mn manganese 54.9</div> <div style="border: 1px solid black; padding: 2px;">26 Fe iron 55.8</div> <div style="border: 1px solid black; padding: 2px;">27 Co cobalt 58.9</div> <div style="border: 1px solid black; padding: 2px;">28 Ni nickel 58.7</div> <div style="border: 1px solid black; padding: 2px;">29 Cu copper 63.5</div> <div style="border: 1px solid black; padding: 2px;">30 Zn zinc 65.4</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">37 Rb rubidium 85.5</div> <div style="border: 1px solid black; padding: 2px;">38 Sr strontium 87.6</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">39 Y yttrium 88.9</div> <div style="border: 1px solid black; padding: 2px;">40 Zr zirconium 91.2</div> <div style="border: 1px solid black; padding: 2px;">41 Nb niobium 92.9</div> <div style="border: 1px solid black; padding: 2px;">42 Mo molybdenum 95.9</div> <div style="border: 1px solid black; padding: 2px;">43 Tc technetium —</div> <div style="border: 1px solid black; padding: 2px;">44 Ru ruthenium 101.1</div> <div style="border: 1px solid black; padding: 2px;">45 Rh rhodium 106.4</div> <div style="border: 1px solid black; padding: 2px;">46 Pd palladium 106.4</div> <div style="border: 1px solid black; padding: 2px;">47 Ag silver 107.9</div> <div style="border: 1px solid black; padding: 2px;">48 Cd cadmium 112.4</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">55 Cs caesium 132.9</div> <div style="border: 1px solid black; padding: 2px;">56 Ba barium 137.3</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">57–71 lanthanoids</div> <div style="border: 1px solid black; padding: 2px;">72 Hf hafnium 178.5</div> <div style="border: 1px solid black; padding: 2px;">73 Ta tantalum 180.9</div> <div style="border: 1px solid black; padding: 2px;">74 W tungsten 183.8</div> <div style="border: 1px solid black; padding: 2px;">75 Re rhenium 186.2</div> <div style="border: 1px solid black; padding: 2px;">76 Os osmium 190.2</div> <div style="border: 1px solid black; padding: 2px;">77 Ir iridium 192.2</div> <div style="border: 1px solid black; padding: 2px;">78 Pt platinum 195.1</div> <div style="border: 1px solid black; padding: 2px;">79 Au gold 197.0</div> <div style="border: 1px solid black; padding: 2px;">80 Hg mercury 200.6</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">87 Fr francium —</div> <div style="border: 1px solid black; padding: 2px;">88 Ra radium —</div> </div>															
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">89–103 actinoids</div> <div style="border: 1px solid black; padding: 2px;">104 Rf rutherfordium —</div> <div style="border: 1px solid black; padding: 2px;">105 Db dubnium —</div> <div style="border: 1px solid black; padding: 2px;">106 Sg seaborgium —</div> <div style="border: 1px solid black; padding: 2px;">107 Bh bohrium —</div> <div style="border: 1px solid black; padding: 2px;">108 Hs hassium —</div> <div style="border: 1px solid black; padding: 2px;">109 Mt meitnerium —</div> <div style="border: 1px solid black; padding: 2px;">110 Ds darmstadtium —</div> <div style="border: 1px solid black; padding: 2px;">111 Rg roentgenium —</div> <div style="border: 1px solid black; padding: 2px;">112 Cn copernicium —</div> </div>															

lanthanoids

actinoids

57 La lanthanum 138.9	58 Ce cerium 140.1	59 Pr praseodymium 140.9	60 Nd neodymium 144.4	61 Pm promethium —	62 Sm samarium 150.4	63 Eu europium 152.0	64 Gd gadolinium 157.3	65 Tb terbium 158.9	66 Dy dysprosium 162.5	67 Ho holmium 164.9	68 Er erbium 167.3	69 Tm thulium 168.9	70 Yb ytterbium 173.1	71 Lu lutetium 175.0
89 Ac actinium —	90 Th thorium 232.0	91 Pa protactinium 231.0	92 U uranium 238.0	93 Np neptunium —	94 Pu plutonium —	95 Am americium —	96 Cm curium —	97 Bk berkelium —	98 Cf californium —	99 Es einsteinium —	100 Fm fermium —	101 Md mendelevium —	102 No nobelium —	103 Lr lawrencium —