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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.

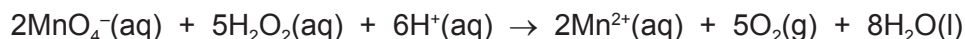
At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [] at the end of each question or part question.

Session
Laboratory

For Examiner's Use	
1	
2	
3	
Total	

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

- 1 Hydrogen peroxide, H_2O_2 , can be oxidised to give oxygen, O_2 . This reaction happens rapidly in the presence of acidified potassium manganate(VII), 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), KMnO_4 .

FA 1 is aqueous hydrogen peroxide, H_2O_2 .

FA 2 is $0.0200 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

FA 3 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4 .

(a) Method

Dilution

- Pipette 25.0 cm^3 of **FA 1** into the 250 cm^3 volumetric flask.
- Add distilled water to make 250 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 cm^3 of **FA 4** into a conical flask.
- Use the measuring cylinder to add 25 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 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 cm³ of **FA 4** required cm³ 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 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 cm³ of **FA 4**.

moles of H_2O_2 = mol

- (iii) Calculate the concentration, in mol dm⁻³, of H_2O_2 in **FA 4**.

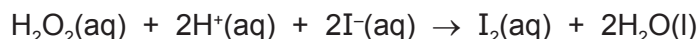
concentration of H_2O_2 in **FA 4** = mol dm⁻³

- (iv) Calculate the concentration, in mol dm⁻³, of H_2O_2 in **FA 1**.

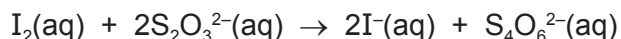
concentration of H_2O_2 in **FA 1** = mol dm⁻³
[4]

[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, H_2O_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, H_2O_2 , to a mixture of iodide ions, 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 mol dm^{-3} sulfuric acid, H_2SO_4 .

FA 4 is the diluted hydrogen peroxide, H_2O_2 , you prepared in **1(a)**.

FA 5 is 1.0 mol dm^{-3} potassium iodide, 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 cm^3 of **FA 3** into the 100 cm^3 beaker.
- Use the measuring cylinder to add 10 cm^3 of **FA 4** into the 100 cm^3 beaker.
- Rinse the measuring cylinder with distilled water and drain.
- Use the measuring cylinder to transfer 20 cm^3 of **FA 5** into the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 20 cm^3 of **FA 6** into the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 10 cm^3 of starch indicator into the 250 cm^3 beaker.
- Add the contents of the 100 cm^3 beaker to the 250 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.

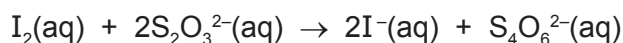
$$\text{concentration of } \text{I}_2 = \dots\dots\dots \text{ mol dm}^{-3}$$

- (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.)

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

- (iii) Calculate the number of moles of thiosulfate ions that reacted with the moles of I_2 , calculated in (ii).



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

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

$$\text{concentration of } \text{S}_2\text{O}_3^{2-} \text{ in FA 6} = \dots\dots\dots \text{ mol dm}^{-3}$$

[4]

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

- add to the 100 cm³ beaker
20 cm³ of **FA 3**
10 cm³ of **FA 4**
- add to the 250 cm³ beaker
20 cm³ of **FA 5**
10 cm³ of **FA 6**
10 cm³ of starch indicator
10 cm³ of distilled water
- Add the contents of the 100 cm³ beaker to the 250 cm³ 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	FA 7	FA 8	FA 9	FA 10
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 FA 10 in a boiling tube add a 0.5 cm depth of 20 'vol' hydrogen peroxide, then add one 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 FA 11 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]

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Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

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

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	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, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown 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, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	"pops" with a lighted splint
oxygen, O_2	relights a glowing splint

The Periodic Table of Elements

Group																			
1	2	Key												13	14	15	16	17	18
		atomic number atomic symbol name relative atomic mass																	
3	4																		
Li lithium 6.9	Be beryllium 9.0																		
11	12																		
Na sodium 23.0	Mg magnesium 24.3																		
19	20																		
K potassium 39.1	Ca calcium 40.1																		
37	38																		
Rb rubidium 85.5	Sr strontium 87.6																		
55	56																		
Cs caesium 132.9	Ba barium 137.3																		
87	88																		
Fr francium —	Ra radium —																		
														</					