

CANDIDATE
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CHEMISTRY

9701/31

Paper 3 Advanced Practical Skills 1

May/June 2014

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.

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 , is unstable and decomposes to give water and oxygen. In addition to the usual units of concentration, mol dm^{-3} , the concentration of a solution of hydrogen peroxide can also be given in 'volume strength' or 'vol'. For example, in **Question 3** you will use a solution of '20 vol' H_2O_2 . This term means that when 1 dm^3 of this solution is completely decomposed it generates 20 dm^3 of oxygen at room temperature and pressure.

The aim of the following titration is to determine the volume strength of a solution of hydrogen peroxide. To do this you will titrate an acidified solution of hydrogen peroxide with potassium manganate(VII) solution.

FA 1 is $0.0200 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

FA 2 is aqueous hydrogen peroxide, H_2O_2 .

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

(a) Method

- Fill the burette with **FA 1**.
- Pipette 25.0 cm^3 of **FA 2** into the conical flask.
- Use the measuring cylinder to add 25 cm^3 of **FA 3** to the conical flask.
- Run **FA 1** from the burette into the conical flask until the pink colour remains.
- 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 1** added in each accurate titration.

I	
II	
III	
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V	
VI	

[6]

- (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 cm³ of **FA 2** required cm³ 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 potassium manganate(VII) present in the volume of **FA 1** calculated in (b).

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

- (ii) Use the following equation and your answer to (i) to calculate the number of moles of hydrogen peroxide used in each titration.



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

- (iii) Calculate the concentration, in mol dm⁻³, of H_2O_2 in **FA 2**.

concentration of $\text{H}_2\text{O}_2 = \dots\dots\dots$ mol dm⁻³

- (iv) Write an equation for the decomposition of hydrogen peroxide to produce oxygen and water.

.....

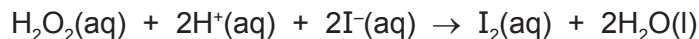
- (v) Calculate the concentration in 'volume strength' of H_2O_2 in **FA 2**. You must assume that the molar volume of oxygen is 24.0 dm³ mol⁻¹ at room temperature and pressure.

I	
II	
III	
IV	
V	

concentration of **FA 2** = 'vol'
[5]

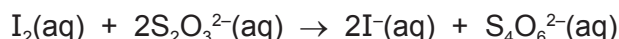
[Total: 12]

- 2 An acidified solution of hydrogen peroxide is able to oxidise iodide ions, $\text{I}^{-}(\text{aq})$, to iodine, $\text{I}_2(\text{aq})$.



In this experiment, you will investigate how the rate of this reaction depends on the concentration of the hydrogen peroxide and on the concentration of the iodide ions.

The rate of this reaction can be measured by adding thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$, and starch indicator to the mixture. 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.

FA 2 is aqueous hydrogen peroxide, H_2O_2 .

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

FA 4 is 0.50 mol dm^{-3} potassium iodide, KI.

FA 5 is $0.025 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

starch indicator

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

(a) Method

Experiment 1

- Use the measuring cylinder to transfer the following volumes into the same 100 cm^3 beaker.
 - 10 cm^3 of **FA 3**
 - 20 cm^3 of **FA 4**
 - 10 cm^3 of **FA 5**
 - 10 cm^3 of distilled water
- Add 10 drops of starch indicator to the beaker.
- Rinse the measuring cylinder with water and shake dry.
- Use the measuring cylinder to measure 10 cm^3 of **FA 2**.
- Add the contents of the measuring cylinder to the 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.
- Wash out the beaker and the measuring cylinder with water and shake dry.

Experiment 2

- Use the measuring cylinder to transfer the following volumes into the same 100 cm^3 beaker.
 - 10 cm^3 of **FA 3**
 - 20 cm^3 of **FA 4**
 - 10 cm^3 of **FA 5**
- Add 10 drops of starch indicator to the beaker.
- Rinse the measuring cylinder with water and shake dry.
- Use the measuring cylinder to measure 20 cm^3 of **FA 2**.

- Add the contents of the measuring cylinder to the 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.
- Wash out the beaker and the measuring cylinder with water and shake dry.

Experiment 3

- Use the measuring cylinder to transfer the following volumes into the same 100 cm³ beaker.
 - 10 cm³ of **FA 3**
 - 10 cm³ of **FA 4**
 - 10 cm³ of **FA 5**
 - 10 cm³ of distilled water
- Add 10 drops of starch indicator to the beaker.
- Rinse the measuring cylinder with water and shake dry.
- Use the measuring cylinder to measure 20 cm³ of **FA 2**.
- Add the contents of the measuring cylinder to the 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.

Record all your results in a single table. You should include the volume of hydrogen peroxide, the volume of potassium iodide, the volume of distilled water and the reaction time. You should also include the 'rate of reaction' which is given by the following expression.

$$\text{rate of reaction} = \frac{1}{\text{reaction time}}$$

I	
II	
III	
IV	
V	
VI	

[6]

(b) From your results, what can you conclude about how the 'rate of reaction' is affected by,

(i) the concentration of hydrogen peroxide,

.....

(ii) the concentration of potassium iodide?

.....

[1]

- (c) It is not possible to draw a reliable conclusion about the effect of changing the concentration of a reagent on the 'rate of reaction' from only two experiments.

Suggest **three** experiments that could be carried out in addition to **Experiments 2** and **3** to investigate the effect of changing the concentration of **FA 4**, potassium iodide. In each case give the volumes of each solution/liquid that would be used.

Do not carry out these experiments.

[2]

- (d) A student was unable to complete all three experiments on the same day but had to return to the investigation the following morning. The student noticed that the results were significantly different.

Give **two** possible reasons for these differences and explain how the student could have overcome these problems.

.....
.....
..... [2]

- (e) There are many sources of error in these experiments. In the following, assume that the only error involves measuring the reaction time.

- (i) If the error in each recorded time is ± 1 s, calculate the percentage error in the time recorded in **Experiment 1**.

percentage error = %

- (ii) Another student decided to repeat **Experiment 1** but used $0.050 \text{ mol dm}^{-3}$ sodium thiosulfate in place of **FA 5**.

How would this change the percentage error in the recorded time? Explain your answer.

.....
.....
..... [2]

[Total: 13]

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) **FA 6** is a solution of two different salts. It contains two different cations, one of which is listed in the Qualitative Analysis Notes on page 10. It contains two anions, both of which are listed in the Qualitative Analysis Notes on page 11.

- (i) Choose reagents that will allow you to identify one of the cations. Carry out suitable tests using these reagents and record your results in the space below.

I	
II	
III	
IV	
V	

One of the cations in **FA 6** is

(ii) Carry out the following tests to identify the two anions present in **FA 6**.

<i>test</i>	<i>observations</i>
To a 1 cm depth of FA 6 in a test-tube add a 1 cm depth of aqueous silver nitrate, then	
add aqueous ammonia.	
To a 1 cm depth of FA 6 in a test-tube add a 1 cm depth of aqueous barium chloride (or aqueous barium nitrate), then	
add dilute nitric acid.	

The anions in **FA 6** are and

[9]

VI	
VII	
VIII	
IX	

(b) **FA 7** is an acidified solution of iron(II) sulfate, $\text{FeSO}_4(\text{aq})$.

Carry out the following tests and record your observations.

<i>test</i>	<i>observations</i>
(i) To a 1 cm depth of FA 7 in a test-tube add aqueous sodium hydroxide and leave for a few minutes.	
(ii) To a 1 cm depth of FA 7 in a boiling tube add a 1 cm depth of dilute sulfuric acid followed by a 1 cm depth of '20 vol' hydrogen peroxide. Stir the mixture, then	
(iii) pour a 1 cm depth of the mixture into a clean boiling tube and add a 3 cm depth of aqueous sodium hydroxide.	

I	
II	
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V	
VI	

(iv) What type of reaction takes place in (ii)?

.....

.....

(v) Explain your observations in (iii).

.....

.....

[6]

[Total: 15]

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 giving dark green solution	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, $\text{SO}_3^{2-}(\text{aq})$	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, 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
sulfur dioxide, SO_2	turns acidified aqueous potassium manganate(VII) from purple to colourless

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