

CANDIDATE
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CHEMISTRY

9701/36

Paper 3 Advanced Practical Skills 2

October/November 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	

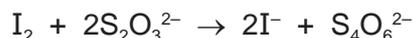
This document consists of **10** printed pages and **2** blank pages.

- 1 You are to determine the concentration of a solution of sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

To do this you will first produce a known amount of iodine by reacting iodate(V) ions, IO_3^- , with an excess of iodide ions, I^- . The equation for this reaction is below.



The amount of iodine produced in this reaction can be found by titrating with thiosulfate ions. The equation for this reaction is below.



FB 1 is aqueous sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FB 2 is aqueous potassium iodate(V) containing 3.60 g dm^{-3} KIO_3 .

FB 3 is sulfuric acid, H_2SO_4 .

FB 4 is aqueous potassium iodide, KI .

starch indicator

(a) Method

- Fill a burette with **FB 1**.
- Pipette 25.0 cm^3 of **FB 2** into the conical flask.
- Use the measuring cylinder to add 25 cm^3 of **FB 3** into the conical flask.
- Use the measuring cylinder to add 10 cm^3 of **FB 4** into the conical flask. Brown iodine solution is produced.
- Add **FB 1** from the burette until most of the iodine has been removed and the solution in the conical flask is yellow.
- Add 10 drops of starch indicator to the contents of the conical flask. The solution will turn blue-black.
- Continue adding **FB 1**, from the burette, until the blue-black colour just disappears.
- Carry out 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 **FB 1** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	

[6]

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

25.0 cm³ of **FB 2** required cm³ of **FB 1**. [1]

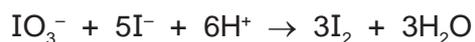
(c) Calculations

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

- (i) Calculate the number of moles of KIO₃ present in 25.0 cm³ of **FB 2**.
[A_r: O, 16.0; K, 39.1; I, 126.9]

moles of KIO₃ = mol

- (ii) The equations for the production of iodine and its titration with thiosulfate are shown below.



Use these equations to calculate the number of moles of thiosulfate present in the volume of **FB 1** you calculated in (b).

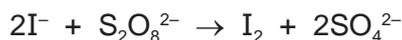
moles of S₂O₃²⁻ = mol

- (iii) Calculate the concentration, in mol dm⁻³, of sodium thiosulfate in **FB 1**.

concentration = mol dm⁻³
[5]

[Total: 12]

- 2 In Question 1 iodide ions were oxidised by iodate(V) ions. Iodide ions can also be oxidised by peroxodisulfate ions, $\text{S}_2\text{O}_8^{2-}$, and you are to investigate the rate of this reaction.



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



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

The rate of the reaction can be represented by rate = $\frac{1}{\text{reaction time}}$.

(a) Method

FB 4 is aqueous potassium iodide, KI.

FB 5 is $0.020 \text{ mol dm}^{-3}$ potassium peroxodisulfate, $\text{K}_2\text{S}_2\text{O}_8$.

FB 6 is $0.010 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

starch indicator

Read through the method before you start any practical work and prepare a suitable table for your results in the space at the top of page 5.

- Empty and wash thoroughly the burette that you used in Question 1.

Experiment 1

- Use the measuring cylinder to add 20 cm^3 of **FB 4** into a 100 cm^3 beaker.
- Use the 10 cm^3 pipette to add 10.0 cm^3 of **FB 6** to the beaker.
- Add 10 drops of starch indicator to the beaker.
- Fill a burette with **FB 5**.
- Run 20.00 cm^3 of **FB 5** into a second 100 cm^3 beaker.
- Add the contents of the first beaker to the second beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution goes blue-black.
- Record this reaction time to the nearest second.
- Wash out both beakers and shake dry.

Experiment 2

- Use the measuring cylinder to add 20 cm^3 of **FB 4** into a 100 cm^3 beaker.
- Use the 10 cm^3 pipette to add 10.0 cm^3 of **FB 6** to the beaker.
- Add 10 drops of starch indicator to the beaker.
- Fill the second burette with distilled water.
- Run 10.00 cm^3 of **FB 5** into the second 100 cm^3 beaker.
- Run 10.00 cm^3 of distilled water into this second beaker containing **FB 5**.
- Add the contents of the first beaker to the second beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution goes blue-black.
- Record this reaction time to the nearest second.
- Wash out both beakers and shake dry.

Record your results in the space below. You should show the volume of **FB 5**, the volume of water and the reaction time for each experiment.

[3]

- (b)** Carry out **three** further experiments to investigate how the reaction time changes with different volumes of peroxodisulfate. Remember that the combined volume of peroxodisulfate solution, **FB 5**, and distilled water must always be 20.00 cm³. Do not use a volume of **FB 5** that is less than 4.00 cm³.

Record the volume of **FB 5**, the volume of water and the reaction time for each experiment.

I	
II	
III	
IV	

[4]

- (c)** Use your results from **(a)** and **(b)** to complete the table below. You should show the volume of **FB 5**, the reaction time and the volume of **FB 5** × reaction time.

[2]

- (d) A student who had carried out these experiments concluded that the rate of reaction was directly proportional to the volume of **FB 5**.

Using your values from (c), explain whether your results agree with this conclusion.

.....
.....
.....
..... [1]

- (e) Another student thought that the experiment could be made more accurate by giving longer reaction times. To do this he repeated **Experiment 1** with the same volumes but using $0.100 \text{ mol dm}^{-3}$ sodium thiosulfate instead of **FB 6**. He found that the reaction never turned blue-black. Explain why.

.....
.....
..... [1]

- (f) Describe how you could modify this experiment to investigate the effect of the concentration of iodide ions, **FB 4**, on the rate of the reaction.

.....
.....
.....
..... [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.

Half fill the 250 cm³ beaker with water and heat it until the water is approximately 60 °C. Leave to stand. This water will be used as a water bath. Turn off the Bunsen burner when the water is hot.

(a) FB 7 and FB 8 are aqueous solutions. One of the elements present in the different ions in **FB 7** and **FB 8** is the same.

Carry out the following tests on **FB 7** and **FB 8** and complete the table.

<i>test</i>	<i>observations</i>
(i) To a 1 cm depth of FB 7 in a test-tube add a 1 cm depth of aqueous silver nitrate.	
(ii) To a 1 cm depth of FB 7 in a test-tube add aqueous sodium hydroxide.	
(iii) To a 1 cm depth of FB 7 in a test-tube add a 1 cm depth of aqueous sodium hydroxide and then a 1 cm depth of hydrogen peroxide. Leave to stand.	
(iv) Before starting this test, the Bunsen burner must be turned off. To a 2 cm depth of dilute sulfuric acid add a few drops of FB 8 then add a 1 cm depth of ethanol. Leave to stand in the water bath.	

[5]

(b) What element is present in both **FB 7** and **FB 8**?

..... [1]

(c) Solid **FB 9** and aqueous solution **FB 10** both contain the same cation.

(i) Transfer approximately half of the **FB 9** into a hard-glass test-tube and heat.

observations

.....

(ii) Transfer the remaining **FB 9** into a boiling tube and carefully add dilute sulfuric acid.

observations

.....

(iii) To a 1 cm depth of **FB 10** in a test-tube, add a piece of magnesium ribbon. Leave to stand.

observations

.....

(iv) To a 1 cm depth of **FB 10** in a test-tube, add an equal depth of concentrated hydrochloric acid (**care: this is very corrosive**).

observations

.....

(v) Suggest which cation is present in both **FB 9** and **FB 10**.

.....

(vi) What change in the oxidation state of magnesium is occurring during reaction (c)(iii)?

Oxidation state change from to

[8]

(d) In which part of the Periodic Table are the elements identified as being present in **FB 7**, **FB 8**, **FB 9** and **FB 10**?

..... [1]

[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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