

CANDIDATE
NAME

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CENTRE
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CANDIDATE
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CHEMISTRY

9701/33

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.

Qualitative Analysis Notes are printed on pages 10 and 11.
A 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.

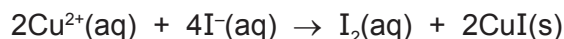
Session	
Laboratory	

For Examiner's Use	
1	
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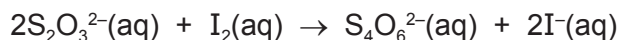
This document consists of **12** printed pages.

- 1 The formula of hydrated copper(II) sulfate is $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$ where x is the number of moles of water of crystallisation in one mole of salt. You will determine the value of x by titration.

When aqueous copper(II) ions react with aqueous iodide ions, I^- , iodine is produced.



The amount of iodine, I_2 , produced can be found by titrating it with aqueous thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$.



FA 1 is aqueous $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$ containing 26.2 g dm^{-3} .

FA 2 is $0.100 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FA 3 is aqueous potassium iodide, KI.

starch indicator

(a) Method

- Pipette 25.0 cm^3 of **FA 1** into a conical flask.
- Use the measuring cylinder to add 15 cm^3 of **FA 3**, an excess of KI, to the conical flask. The solution will turn brown because iodine is formed.
- Fill the burette with **FA 2**.
- Add **FA 2** from the burette until the colour of the mixture changes to pale brown.
- Add 10 drops of starch indicator. The mixture will turn blue-black.
- Continue adding **FA 2** from the burette until the dark colour suddenly disappears to leave an off-white solid. This is the end point of the titration.
- 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 your burette readings and the volume of **FA 2** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (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 **FA 1** required cm³ of **FA 2**. [1]

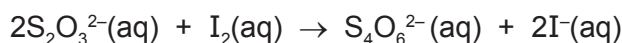
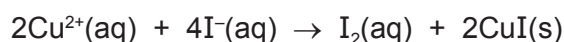
(c) Calculations

Show your working and appropriate significant figures in **each** step of your calculations.

- (i) Calculate the number of moles of thiosulfate ions present in the volume of **FA 2** you have calculated in (b).

moles of S₂O₃²⁻ = mol

- (ii) Use your answer to (i), and the equations for the reactions involved, to deduce the number of moles of Cu²⁺ present in 25.0 cm³ of **FA 1**.



moles of Cu²⁺ = mol

- (iii) Use your answer to (ii) and the mass of CuSO₄.xH₂O present in the solution, to calculate the relative molecular mass, M_r, of CuSO₄.xH₂O.

M_r of CuSO₄.xH₂O =

- (iv) Determine the value of **x**.
(Use data from the Periodic Table on page 12.)

x =
[6]

[Total: 14]

- 2 **FA 4** is an impure sample of hydrated calcium chloride, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$. On heating, hydrated calcium chloride loses its water of crystallisation.



You will determine the purity of **FA 4** by measuring the loss in mass that occurs when it is heated. The impurity present in **FA 4** is not decomposed on heating.

(a) Method

You should read the instructions carefully before starting any practical work and draw a table for your results in the space below.

- Weigh a crucible and record its mass.
- Add between 1.80g and 2.00g of **FA 4** into the crucible.
- Reweigh the crucible and its contents and record the mass.
- Place the crucible on the pipe-clay triangle and heat gently for 1 minute and then strongly for a further 2 minutes.
- Allow the crucible and its contents to cool. Reweigh the crucible and contents and record the mass.
- Heat the crucible strongly for a further 2 minutes. Allow it to cool. Reweigh the crucible and contents and record the mass.
- Repeat the heating, cooling and weighing until you are satisfied that all the water of crystallisation has been removed.
- Calculate and record the mass of **FA 4** used and the total mass of water lost.

While you are waiting for the crucible to cool, you may wish to start work on Question 3.

I	
II	
III	
IV	
V	
VI	

[6]

(b) Calculations

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

- (i) The percentage loss in mass on heating is defined as

$$\frac{\text{the loss in mass on heating}}{\text{the original mass}} \times 100.$$

Calculate the percentage loss in mass of **FA 4**.

percentage loss in mass = %

- (ii) Calculate the percentage loss in mass when **pure** hydrated calcium chloride, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, is heated.

percentage loss in mass = %

- (iii) Use your results to (i) and (ii) to calculate the percentage purity of **FA 4**, impure $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$.

percentage purity = %
[3]

- (c) A student carried out this experiment using 2.60 g of **FA 4**.

Suggest whether this experiment would give a more accurate result for the percentage purity of **FA 4**. Explain your answer.

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

(d) In your calculations you assumed that the impurity in **FA 4** does not decompose on heating.

State how the percentage purity that you calculated in (b)(iii) would change if the impurity were to decompose on heating.

Explain your answer.

.....

..... [1]

[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) (i) **FA 5, FA 6** and **FA 7** are aqueous solutions each containing one anion and one cation.

Carry out the experiments described below and record your observations for each solution in the table.

	FA 5	FA 6	FA 7
To a 1 cm depth in a test-tube, add a 1 cm depth of aqueous sodium carbonate.			
To a 1 cm depth in a test-tube, add a 1 cm depth of aqueous copper(II) sulfate.			
To a 1 cm depth in a test-tube, add a 1 cm depth of aqueous barium chloride or aqueous barium nitrate.			

(ii) What ion is present in **both FA 6** and **FA 7**?

.....

(iii) The anion in **FA 5** is one of carbonate, chloride, nitrate or sulfate.

Which anion is present in **FA 5**?

.....

(iv) Write the ionic equation, including state symbols, for the reaction between **FA 5** and aqueous copper(II) sulfate.

.....

[7]

(b) **FA 8** contains two anions and two cations from the lists on pages 10 and 11.

- To a 5 cm depth of distilled water in a boiling tube, add all the **FA 8**.
- Shake the boiling tube thoroughly for one minute to make sure that no more of the solid will dissolve.
- Filter the mixture into a clean boiling tube.
- Place the filter funnel in a conical flask and wash the residue with a little distilled water.
- **Keep both filtrate and residue for tests (i) and (ii) below.**

(i) **Tests on the filtrate (the solution in the boiling tube)**

Carry out the following tests and record your observations in the table below.

<i>test</i>	<i>observations</i>
To a 1 cm depth of the filtrate in a test-tube, add aqueous sodium hydroxide, then	
add aqueous hydrogen peroxide.	

(ii) **Tests on the residue**

Carry out the following tests and record your observations in the table below.

<i>test</i>	<i>observations</i>
Place the funnel containing the residue into a clean boiling tube. Pour approximately 5 cm ³ of dilute nitric acid onto the residue. Collect a 1 cm depth of solution in the boiling tube. Remove the funnel and return it to the conical flask.	
To this solution in the boiling tube, add aqueous sodium hydroxide.	

(iii) Identify **two** cations present in **FA 8**.

cations present and

(iv) Identify **one** anion present in **FA 8**.

anion present

(v) Suggest what type of reaction is happening when hydrogen peroxide is added in test (b)(i).

..... [8]

[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

