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CHEMISTRY

9701/33

Paper 3 Advanced Practical Skills 1

October/November 2017

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.

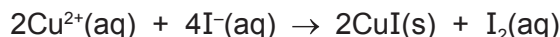
At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [] at the end of each question or part question.

Session
Laboratory

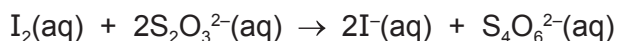
For Examiner's Use	
1	
2	
3	
Total	

This document consists of **12** printed pages.

- 1 In this experiment you will determine the value of **x** in the formula for hydrated copper(II) sulfate, $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$. You will first react a solution of Cu^{2+} ions with excess iodide ions, I^- . This reaction produces iodine.



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



FA 1 is $0.150 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FA 2 is dilute sulfuric acid.

FA 3 is 1.00 mol dm^{-3} potassium iodide, KI .

FA 4 is a solution made by dissolving 32.5 g of $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$ in 1.00 dm^3 of solution.
starch indicator

(a) Method

- Fill the burette with **FA 1**.
- Pipette 25.0 cm^3 of **FA 4** into a conical flask.
- Use the measuring cylinder to add 10 cm^3 of **FA 2** to the same conical flask.
- Use the measuring cylinder to add 10 cm^3 of **FA 3** to the same conical flask. The mixture will become brown because of the formation of I_2 , and will become cloudy because of the formation of the white precipitate of CuI .
- Add **FA 1** from the burette until the mixture becomes a light brown colour.
- Add 10 to 20 drops of starch indicator until the mixture becomes blue-black.
- Continue to titrate with **FA 1** until the blue-black colour disappears leaving a mixture with an off-white solid. This is the end-point.
- You should test that the end-point has been reached by adding 2 more drops of starch indicator. If the titration has reached the end-point the added starch indicator will cause no change in colour.
- 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 sure 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	
IV	
V	
VI	
VII	

[7]

- (b) From your accurate titration results, obtain a suitable value for the volume of **FA 1** to be used in your calculations.
Show clearly how you obtained this value.

25.0 cm³ of **FA 4** 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 thiosulfate ions in the volume of **FA 1** calculated in (b).

moles of S₂O₃²⁻ = mol

- (ii) Using the equations on page 2, calculate the number of moles of copper(II) ions in 25.0 cm³ of **FA 4**.

moles of Cu²⁺ = mol

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

concentration of Cu²⁺ in **FA 4** = mol dm⁻³

- (iv) Calculate the value of **x** in CuSO₄·**x**H₂O.

x =
[5]

- (d) (i) Calculate the maximum percentage error in one of your accurate titres.

maximum percentage error = %

- (ii) A student suggests that the experiment could be made more accurate if the volume of **FA 3** was measured using a burette.

Give a reason why the student might make this suggestion.

.....

.....

.....

Explain why this change would **not** improve the accuracy of the experiment.

.....

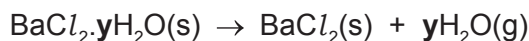
.....

[3]

[Total: 16]

Question 2 starts on the next page.

- 2 In this experiment you will determine the value of **y** in the formula for hydrated barium chloride, $\text{BaCl}_2 \cdot y\text{H}_2\text{O}$. You will do this by measuring the mass loss when a sample of hydrated barium chloride is heated.



FA 5 is hydrated barium chloride, $\text{BaCl}_2 \cdot y\text{H}_2\text{O}$.

(a) Method

Before starting any practical work, read through all the instructions and prepare a table for your results in the space provided.

- Weigh the crucible with a lid and record the mass.
- Add all the **FA 5** to the crucible.
- Reweigh the crucible with the lid and **FA 5**. Record the mass.
- Place the crucible in the pipe-clay triangle on top of a tripod.
- Heat the crucible **gently** with the lid on for about 1 minute.
- Remove the lid and then heat more strongly for a further 4 minutes.
- Replace the lid and allow the crucible to cool.
- **While the crucible is cooling you may wish to begin work on Question 3.**
- Once the crucible has cooled, reweigh the crucible with the lid and contents. Record the mass.
- Calculate and record the mass of **FA 5** used, the mass of the residue and the mass of water lost.

I	
II	
III	
IV	
V	

[5]

(b) 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 barium chloride in the residue. You may assume all the water has been removed.

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

- (ii) Calculate the number of moles of water lost.

moles of H_2O lost = $\dots\dots\dots$ mol

- (iii) Calculate the value of **y** in $\text{BaCl}_2 \cdot y\text{H}_2\text{O}$.

y = $\dots\dots\dots$
[3]

- (c) (i) For this experiment to give an accurate value for **y**, anhydrous barium chloride must be thermally stable.

Explain fully what would happen to the value of **y** if BaCl_2 were to decompose slightly during heating.

$\dots\dots\dots$
 $\dots\dots\dots$
 $\dots\dots\dots$

- (ii) Starting with the same mass of hydrated barium chloride, suggest how you could modify the experiment to determine more accurately the mass of water lost.

$\dots\dots\dots$
 $\dots\dots\dots$
[3]

[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 reagents are selected for use in a test, the **name** or **correct formula** of the element or compound must be given.

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.

FA 6 and **FA 7** are both salts which contain cations and anions from those listed in the Qualitative Analysis Notes. Each salt contains a single cation and a single anion.

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

test	observations	
	FA 6	FA 7
(i) Place a small spatula measure of the solid in a hard-glass test-tube and heat gently at first, then		
more strongly.		

(ii) From your observations, what is present in both salts?

.....

[4]

(b) Prepare solutions of **FA 6** and **FA 7** by placing the rest of each solid into separate 100 cm³ beakers. Add approximately 30 cm³ of distilled water to each beaker and stir until fully dissolved. Use these solutions for tests in (b).

- (i) Carry out tests to determine the cation present in each solution.
Record your tests and results in the space below.

- (ii) Carry out all the following tests and record your observations.

test	observations	
	solution of FA 6	solution of FA 7
To a 1 cm depth of solution in a test-tube add a 1 cm depth of barium chloride or barium nitrate, then		
add an excess of hydrochloric acid or nitric acid.		
To a 1 cm depth of solution in a test-tube add a 1 cm depth of silver nitrate.		

- (iii) Identify the ions present in each salt.

FA 6 contains and

FA 7 contains and

[9]

[Total: 13]

Qualitative Analysis Notes

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)	faint white ppt. is nearly always observed unless 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												1 H hydrogen 1.0											
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 —																								