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CHEMISTRY (US)

9185/35

Paper 3 Advanced Practical Skills 1

May/June 2015

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your Center 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 14 and 15.

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 **13** printed pages and **3** blank pages.

- 1 In this experiment you will determine the formula of iron(III) ammonium sulfate, $\text{FeNH}_4(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}$, where x is the number of molecules of water of crystallization.

A known mass of this iron(III) compound reacted with excess acidified potassium iodide to produce iodine. You will determine the amount of iodine produced by titrating the mixture with sodium thiosulfate.

FA 1 is $0.900 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FA 2 is a solution of iodine, I_2 , produced as outlined in the paragraph above.
starch indicator

(a) Method

Diluting FA 1

- Pipet 25.0 cm^3 of **FA 1** into the 250 cm^3 volumetric (graduated) flask.
- Make the solution up to the mark using distilled water.
- Shake the flask to mix the solution thoroughly before using it for your titrations.
- Label this diluted solution of sodium thiosulfate **FA 3**.
- Rinse the pipet with distilled water.

Keep FA 1 for use in Question 3.

Titration

- Fill the buret with **FA 3**.
- Use the pipet to transfer 25.0 cm^3 of **FA 2** into an Erlenmeyer flask.
- Add **FA 3** from the buret into the Erlenmeyer flask until the mixture becomes pale yellow.
- Then add 10 drops of starch indicator to give a blue-black color.
- Continue adding **FA 3** until this blue-black color disappears. This is the end-point of the titration.
- Perform a **rough titration** and record your buret readings in the space below.

The rough titer 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 table below, all of your buret readings and the volume of **FA 3** 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 calculation. Show clearly how you obtained this value.

25.0 cm³ of **FA 2** required cm³ of **FA 3**. [1]

(c) Calculations

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

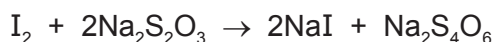
- (i) Using information on page 2, calculate the concentration, in mol dm⁻³, of sodium thiosulfate in **FA 3**.

concentration of Na₂S₂O₃ in **FA 3** = mol dm⁻³

- (ii) Calculate the number of moles of sodium thiosulfate present in the volume of **FA 3** calculated in (b).

moles of Na₂S₂O₃ = mol

- (iii) Use the equation below to calculate the number of moles of iodine that reacted with the sodium thiosulfate in (ii).



moles of I₂ = mol

- (iv) Calculate the concentration of I₂, in mol dm⁻³, in **FA 2**.

concentration of I₂ = mol dm⁻³

- (v) The iodine in **FA 2** was produced by the reaction of iron(III) ions with excess potassium iodide. Balance the equation for this reaction.



Use your answer to (iv) and this equation to calculate the number of moles of iron(III) ions that reacted to produce the iodine in 1.00 dm³ of **FA 2**.

moles of Fe³⁺ =

- (vi) The formula of the iron(III) compound is FeNH₄(SO₄)₂·xH₂O.
38.56 g of this compound was weighed out and added to excess aqueous acidified potassium iodide.
FA 2 was made by making the resulting solution of iodine up to 1.00 dm³ with distilled water.

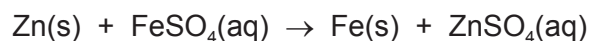
Use this information and your answer to (v) to calculate the number of moles of water of crystallization, **x**, in one mole of the iron(III) compound.

[A_r: H, 1.0; N, 14.0; O, 16.0; S, 32.1; Fe, 55.8]

x =
[6]

[Total: 14]

- 2 In this experiment you will determine the enthalpy change, ΔH , for the reaction of zinc and iron(II) sulfate.



In order to do this, you will determine the enthalpy changes for the reactions of zinc and iron with aqueous copper(II) sulfate. Excess of the two metals will be used during the determinations.

Then you will use Hess' Law to calculate the enthalpy change for the reaction above.

FA 4 is zinc, Zn.

FA 5 is iron, Fe.

FA 6 is $0.500 \text{ mol dm}^{-3}$ copper(II) sulfate, CuSO_4 .

- (a) Determination of the enthalpy change for the reaction of zinc, **FA 4**, with aqueous copper(II) sulfate, **FA 6**.

Method

- Support a plastic cup inside the 250 cm^3 beaker.
- Use the graduated cylinder to transfer 25 cm^3 of **FA 6** into the plastic cup.
- Measure and record the initial temperature of the solution in the space below.
- Add all the **FA 4** from the container to the **FA 6** in the plastic cup.
- Stir constantly until the maximum temperature is reached.
- Measure and record the maximum temperature. Tilt the cup if necessary to ensure the thermometer bulb is fully immersed.
- Calculate and record the temperature rise.

[3]

(b) Calculations

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

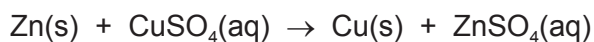
- (i) Calculate the energy produced during this reaction.
[Assume that **4.2 J** are needed to raise the temperature of 1.0 cm³ of solution by 1.0 °C.]

energy produced = J

- (ii) Calculate the number of moles of copper(II) sulfate in 25 cm³ of **FA 6**.

moles of CuSO₄ = mol

- (iii) Calculate the enthalpy change, in kJ mol⁻¹, for the reaction below.



enthalpy change = kJ mol⁻¹
sign value

[2]

- (c) Determination of the enthalpy change for the reaction of iron, **FA 5**, with aqueous copper(II) sulfate, **FA 6**.

Method

- Support the second plastic cup inside the beaker.
- Use the graduated cylinder to transfer 25 cm³ of **FA 6** into the plastic cup.
- Measure and record the initial temperature of the solution in the space below.
- Add all the **FA 5** from the container to the **FA 6** in the plastic cup.
- Stir constantly until the maximum temperature is reached.
- Measure and record the maximum temperature. Tilt the cup if necessary to ensure the thermometer bulb is fully immersed.
- Calculate and record the temperature rise.

Keep solution **FA 6** for use in Question 3.

[2]

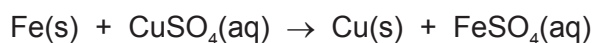
(d) Calculations

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

- (i) Calculate the energy produced during this reaction.
[Assume that **4.2 J** are needed to raise the temperature of 1.0 cm³ of solution by 1.0 °C.]

energy produced = J

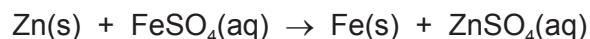
- (ii) Calculate the enthalpy change, in kJ mol⁻¹, for the reaction below.



enthalpy change = kJ mol⁻¹
sign value

[2]

- (e) Use your values for the enthalpy changes calculated in (b)(iii) and (d)(ii) to calculate the enthalpy change for the reaction below.



Show clearly how you obtained your answer by drawing a Hess' Law energy cycle.

(If you were unable to calculate the enthalpy changes, assume that the value in (b)(iii) is -210 kJ mol^{-1} and the value in (d)(ii) is -144 kJ mol^{-1} . Note: these are not the correct values.)

enthalpy change = kJ mol^{-1}
sign value

[2]

- (f) (i) Calculate the maximum percentage error in the temperature **rise** in (c).

percentage error = %

- (ii) Apart from using a more accurately calibrated thermometer, suggest **one** improvement that could be made to this experiment that would increase the accuracy.

.....

[2]

[Total: 13]

3 Qualitative Analysis

At each stage of any test you are to record details of the following.

- color 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 large test-tube (for boiling) MUST be used.

Rinse and reuse test-tubes where possible.

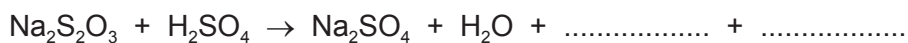
Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

- (a) **FA 1**, used in **Question 1**, is an aqueous solution of sodium thiosulfate.
FA 6, used in **Question 2**, is an aqueous solution of copper(II) sulfate.
FA 7 is solid sodium thiosulfate.

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

<i>test</i>	<i>observations</i>
(i) Using a spatula, place 2 or 3 crystals of FA 7 into a hard-glass test-tube. Heat gently for several seconds, then	
heat strongly.	
(ii) To a 1 cm depth of dilute sulfuric acid in a large test-tube (for boiling), add a few crystals of FA 7 . Observe until no further change occurs, then	
warm the mixture, gently and carefully .	
(iii) To a 1 cm depth of aqueous potassium iodide in a test-tube, add a few drops of FA 6 , aqueous copper(II) sulfate, then	
add FA 1 , aqueous sodium thiosulfate, to the mixture until no further change occurs.	

- (iv) Using your observations in (ii), complete the equation below by giving the formulae of the other two products.



[5]

(b) **FA 8** and **FA 9** are aqueous solutions.

Each contains **one** cation and **one** anion from those listed on pages 14 and 15.

Carry out the tests and record all your observations in the table.

For each test, use a 1 cm depth of **FA 8** or **FA 9** in a test-tube.

<i>test</i>	<i>observations</i>	
	FA 8	FA 9
(i) Add aqueous sodium hydroxide.		
(ii) Add a few drops of aqueous silver nitrate followed by aqueous ammonia.		
(iii) Add aqueous ammonia.		
(iv) Add an equal depth of dilute sulfuric acid.		
(v) Add an equal depth of FA 9 .		X

(vi) Using your observations, identify **three** of the ions present in **FA 8** and **FA 9**. Write 'unknown' next to the ion that you cannot identify directly from your observations.

ions in **FA 8**: cation anion

ions in **FA 9**: cation anion

(vii) The 'unknown' ion in (vi) can be identified by elimination, using the lists of ions on pages 14 and 15.

Choose **one** positive test that would confirm the identity of this ion. Name the reagent(s) you would use and state what you would observe if the test was positive.

Do not carry out this test.

'unknown' ion tested for

reagent(s)

observation(s)

[8]

[Total: 13]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	reaction with	
	NaOH(aq)	NH ₃ (aq)
aluminum, 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)	gray-green ppt. soluble in excess giving dark green solution	gray-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 (colorless $\text{NO} \rightarrow$ (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 colorless

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