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CHEMISTRY

9701/31

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 oxidation number of iodine in one of its compounds by titration.

FA 1 is a $0.0197 \text{ mol dm}^{-3}$ solution of the iodine-containing compound.

FA 2 is dilute sulfuric acid, H_2SO_4 .

FA 3 is aqueous potassium iodide, KI .

FA 4 is $0.105 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.
starch indicator

FA 1 reacts with excess acidified potassium iodide to produce iodine, I_2 . This iodine is then titrated with aqueous sodium thiosulfate using starch indicator.

(a) Method

- Fill the burette with **FA 4**.
- Pipette 25.0 cm^3 of **FA 1** into a conical flask.
- Using the measuring cylinder, add 10 cm^3 of **FA 2** to the same conical flask.
- Using the same measuring cylinder, add 20 cm^3 of **FA 3** to the mixture in the conical flask. The mixture will now be a red-brown colour, due to iodine produced.
- Carry out a rough titration by adding **FA 4** from the burette until the mixture becomes light brown.
- Then add 10 drops of starch indicator. The mixture will change to a dark blue colour.
- Continue titrating until the mixture becomes colourless. This is the end-point.

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 4** 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 4** to be used in your calculations. Show clearly how you obtained this value.

The iodine produced required cm^3 of **FA 4**. [1]

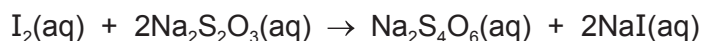
(c) Calculations

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

- (i) Calculate the number of moles of sodium thiosulfate in the volume of **FA 4** calculated in (b).

moles of $\text{Na}_2\text{S}_2\text{O}_3 = \dots\dots\dots \text{mol}$

- (ii) The equation for the reaction of iodine with sodium thiosulfate is shown.



Calculate the number of moles of iodine that reacted with the sodium thiosulfate calculated in (i).

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

- (iii) Use the information on page 2 to calculate the number of moles of iodine-containing compound in the 25 cm^3 of **FA 1** used in each titration.

moles of iodine-containing compound in 25 cm^3 **FA 1** = $\dots\dots\dots \text{mol}$

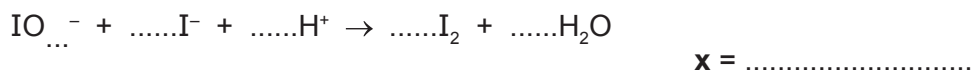
- (iv) Use your answers to (ii) and (iii) to calculate the number of moles of iodine produced when 1 mole of the iodine-containing compound in **FA 1** reacts with excess **FA 3**. Give your answer as an integer.

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

- (v) The anion in **FA 1** is IO_x^- where **x** is the number of oxygen atoms present in the formula.

Use your answer to (iv) to balance the ionic equation for the reaction between **FA 1** and **FA 3** under acidic conditions.

Hence deduce the value of **x** in the formula IO_x^- .



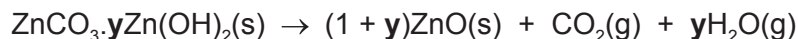
- (vi) Calculate the oxidation state of iodine in **FA 1**.
(If you were unable to calculate **x** in part (v), assume that **x** = 4.)

oxidation state of iodine = $\dots\dots\dots$
[6]

[Total: 14]

- 2 Zinc carbonate occurs in a basic form, which means that zinc hydroxide is also present. The chemical formula of basic zinc carbonate can be written as $\text{ZnCO}_3 \cdot y\text{Zn(OH)}_2$, where y may not be an integer. In this experiment you will heat basic zinc carbonate to decompose it and use your results to determine the value of y .

When basic zinc carbonate is heated, it decomposes as shown.



FA 5 is basic zinc carbonate, $\text{ZnCO}_3 \cdot y\text{Zn(OH)}_2$.

(a) Method

Read through the method before starting any practical work.

Prepare a table for all your results from Experiments 1 and 2 in the space on page 5.

Experiment 1

- Weigh a crucible with its lid and record the mass.
- Add 2.1–2.5 g of **FA 5** to the crucible. Weigh the crucible and lid with **FA 5** and record the mass.
- Place the crucible in the pipe-clay triangle on top of the tripod.
- Heat the crucible and contents gently for 1 minute with the lid on.
- Remove the lid. Heat the crucible and contents strongly, with the lid off, for approximately 4 minutes.
- Replace the lid and leave the crucible and residue to cool for at least 5 minutes, before re-weighing it with the lid on. Record the mass.
- **While the crucible is cooling, you may wish to begin work on Question 3.**
- Calculate, and record in your table, the mass of **FA 5** used and the mass of residue obtained.

- (i) State the observation(s) you made while you were heating **FA 5**.

.....

- (ii) State the observation(s) you made once the residue had cooled down.

.....

Experiment 2

- Repeat the procedure used in **Experiment 1**, using 1.5–1.9 g of **FA 5** and using the other crucible and lid.
- Record the three balance readings made during the experiment.
- Calculate and record the mass of **FA 5** used and the mass of residue obtained.

Results

I	
II	
III	
IV	
V	
VI	

[6]

(b) Calculations

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

- (i) Calculate the relative formula mass, M_r , of zinc hydroxide, $\text{Zn}(\text{OH})_2$.

M_r of $\text{Zn}(\text{OH})_2$ =

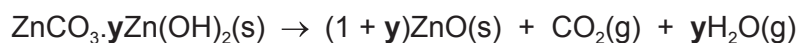
- (ii) Using your answer to (i), write down an expression, in terms of **y**, for the relative formula mass, M_r , of basic zinc carbonate, $\text{ZnCO}_3 \cdot y\text{Zn}(\text{OH})_2$.

M_r of $\text{ZnCO}_3 \cdot y\text{Zn}(\text{OH})_2$ =

- (iii) Using the mass of $\text{ZnCO}_3 \cdot y\text{Zn}(\text{OH})_2$ from **Experiment 1** and your answer to (ii), write down an expression, in terms of **y**, for the number of moles of $\text{ZnCO}_3 \cdot y\text{Zn}(\text{OH})_2$ that you heated in **Experiment 1**.

moles of $\text{ZnCO}_3 \cdot y\text{Zn}(\text{OH})_2$ = mol

- (iv) Using your answer to (iii) and the equation below, write an expression, in terms of **y**, for the number of moles of zinc oxide produced in **Experiment 1**.



moles of ZnO produced = mol

- (v) Use your results from **Experiment 1** to calculate the number of moles of zinc oxide, ZnO, obtained in the residue. You may assume complete decomposition has occurred.

moles of ZnO = mol

- (vi) Using your answers to (iv) and (v), calculate the value of **y** to one decimal place.

y =
[6]

- (c) (i) Apart from altering the balance or the masses of **FA 5** used, state **one** improvement you could make to the experimental procedure to improve its accuracy.

.....
.....
.....

- (ii) Which experiment should be more accurate, **Experiment 1** or **Experiment 2**? Explain your answer.

.....
.....
.....

[2]

[Total: 14]

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, **FA 7** and **FA 8** are solutions of salts.

Information about FA 6 , FA 7 and FA 8
• Each salt contains one cation and one anion. • One of the ions is sodium; the other five ions are listed in the Qualitative Analysis Notes. • Each salt contains a different nitrogen-containing ion. • FA 7 or FA 8 contains a halide ion.

(a) You will identify the **cations** present in **FA 6**, **FA 7** and **FA 8**.

To do this you will carry out **six** separate tests. You will use dilute sulfuric acid and aqueous sodium hydroxide separately with **FA 6**, **FA 7** and **FA 8**.

Use a 1 cm depth of each salt solution in a suitable tube for each test you carry out.

Record **all** of your observations in a table in the space below.

- (b) Name the reagents you would use to identify the halide ion present in either **FA 7** or **FA 8**.
Test **FA 7** and **FA 8** with these reagents and record your observations.

reagents used

unknown	observations	halide ion present ✓ / ✗
FA 7		
FA 8		

[2]

- (c) (i) Name the reagents you would use to confirm the presence of the nitrogen-containing **anions** in the two solutions that do **not** contain a halide ion. Test both solutions with these reagents and record your observations.

reagents used

unknown	observations
FA	
FA	

- (ii) Name the reagent you would use to positively identify one of the nitrogen-containing anions in the two solutions tested in (i). Test both solutions with this reagent. Record all your observations.

reagent used

unknown	observations
FA	
FA	

[4]

- (d) Use the information given in (a) and your observations in all tests to deduce the chemical formulae of the three salts.

FA 6 is **FA 7** is **FA 8** is

[2]

[Total: 12]

Qualitative Analysis Notes

1 Reactions of aqueous cations

<i>ion</i>	<i>reaction with</i>	
	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 —																								