



CANDIDATE NAME					
CENTRE NUMBER			CANDIDATE NUMBER		

CHEMISTRY 9701/33

**Advanced Practical Skills** 

October/November 2010

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Instructions to Supervisors

### **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 a soft pencil for any diagrams, graphs or rough working.

Do not use staples, paper clips, highlighters, glue or correction fluid.

DO NOT WRITE IN ANY BARCODES.

Answer all questions.

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 11 and 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						
Laborat	tory					

For Examiner's Use				
1				
2				
3				
Total				

This document consists of 11 printed pages and 1 blank page.



There are three questions on this paper. Question 2 should not be the last question attempted.

For Examiner's Use

1 You are to determine the concentration of hydrochloric acid, which supplies the H<sup>+</sup> ions in the following reaction.

$$IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow 3H_2O(I) + 3I_2(aq)$$

In the presence of an excess of  ${\rm IO_3}^-$  ions and an excess of  ${\rm I}^-$  ions, the amount of  ${\rm I_2}$  liberated is directly proportional to the amount of H<sup>+</sup> ions present and can be determined by titration with sodium thiosulfate,  ${\rm Na_2S_2O_3}$ .

You are provided with the following reactants.

**FA 1** hydrochloric acid **FA 2** containing 15.0 g dm $^{-3}$  sodium thiosulfate, Na $_2$ S $_2$ O $_3$ .5H $_2$ O aqueous potassium iodate(V), KIO $_3$  aqueous potassium iodide, KI

### (a) Method

- Fill a burette with FA 2.
- Pipette 25.0 cm<sup>3</sup> of **FA 1** into the conical flask.
- Use a 25 cm³ measuring cylinder to add to the flask 10 cm³ of aqueous potassium iodate(V) and 10 cm³ of aqueous potassium iodide. There is an excess of each of these reagents.
- Place the flask on a white tile.
- Titrate the liberated iodine with FA 2.
- During the titration the colour of the iodine in the solution will fade from red-brown to orange to yellow. The end-point occurs when the solution just goes colourless with the addition of a single drop of **FA 2**.
- You should perform a rough titration.
   In the space below record your burette readings for this rough titration.

The rough titre is	cm <sup>3</sup>
ino rough and lo	

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Record in a suitable form below all of your burette readings and the volume of FA 2
  added in each accurate titration.
- Make certain any recorded results show the precision of your practical work.

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[7]

(b)	From your Show clear				to	be	used	in	your	calculat	ion.

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25.0 cm<sup>3</sup> of **FA 1** require ...... cm<sup>3</sup> of **FA 2**. [1]

### **Calculations**

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

(c) (i) Calculate the concentration, in mol dm $^{-3}$ , of the sodium thiosulfate in FA 2. FA 2 contains 15.0 g dm $^{-3}$  Na $_2$ S $_2$ O $_3$ .5H $_2$ O. [ $A_r$ : H, 1.0; O, 16.0; Na, 23.0; S, 32.1]

The concentration of sodium thiosulfate in **FA 2** is ...... mol dm<sup>-3</sup>.

(ii) Calculate how many moles of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> are contained in the volume of **FA 2** recorded in **(b)**.

..... mol of  $\mathrm{Na_2S_2O_3}$ 

(iii) Calculate how many moles of iodine, I<sub>2</sub> reacted with the Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> in (ii).

$$2Na_2S_2O_3(aq) + I_2(aq) \rightarrow Na_2S_4O_6(aq) + 2NaI(aq)$$

..... mol of iodine reacted with the sodium thiosulfate.

(iv) Calculate how many moles of hydrochloric acid, HCl, reacted with an excess of potassium iodate(V) and an excess of potassium iodide to produce the amount of iodine calculated in (iii).

$$\mathrm{IO_3^-(aq)}$$
 +  $\mathrm{5I^-(aq)}$  +  $\mathrm{6H^+(aq)} \longrightarrow \mathrm{3H_2O(I)}$  +  $\mathrm{3I_2(aq)}$ 

..... mol of HC1 produced the amount of iodine calculated in (iii).

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	(V)	Calculate the concentration, in moldm 3, of HC1 in FA 1.	For Examiner's Use
(d)		The concentration of HC $l$ in <b>FA 1</b> is mol dm <sup>-3</sup> . [5]	
(u)		ach reading with a burette has a maximum error of ±0.05 cm <sup>3</sup> . rade B volumetric (bulb) pipettes are calibrated to ±0.06 cm <sup>3</sup> .	
	(i)	Calculate the maximum error in the volume run from the burette recorded in any titration.	
		The maximum error is cm <sup>3</sup> .	
	(ii)	Express the maximum error calculated in (i) as a percentage error for the volume calculated in (b).	
		The maximum error is %.	
	(iii)	Calculate the percentage error when 25.0 cm <sup>3</sup> of <b>FA 1</b> was pipetted into the conical flask.	
		The array was	
		The error was %. [2]	
		[Total: 15]	
			1

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2 FA 3 is powdered basic copper(II) carbonate, a hydrated mixture of copper(II) carbonate and copper(II) hydroxide.

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The approximate formula for the basic carbonate is CuCO<sub>3</sub>.Cu(OH)<sub>2</sub>.H<sub>2</sub>O.

When heated, basic copper(II) carbonate decomposes.

$$CuCO_3.Cu(OH)_2.H_2O(s) \rightarrow 2CuO(s) + CO_2(g) + 2H_2O(g)$$

You are to determine the change in mass as the solid is heated and decomposed.

- (a) Method Read through the instructions before starting any practical work.
  - Record all weighings in an appropriate form in the space below.
  - Weigh and record the mass of an empty boiling-tube.
  - Tip the contents of the tube labelled FA 3 into the weighed boiling-tube. Reweigh and record the total mass of the boiling-tube and FA 3.
  - Heat FA 3 in the boiling-tube very gently until the vigorous decomposition of the copper carbonate has stopped; then heat more strongly for 1 to 2 minutes. Take care not to lose any solid from the tube during the initial heating.
  - Warm the upper parts of the boiling-tube to evaporate any water that may have condensed while heating the carbonate.
  - Place the hot tube on a heat-proof mat and leave to cool.
  - You are advised to continue with part (d) of this question or to start another question while the tube cools.
  - When cool, reweigh the boiling-tube and the residual copper(II) oxide.
  - Reheat, cool and reweigh the tube until you are satisfied decomposition is complete.

### **Results**

In an appropriate form, in the space below, record all of your balance readings, the mass of basic copper(II) carbonate and the mass of residual copper oxide.

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[6]

#### **Calculations**

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(b) Calculate the loss in mass during the experiment as a percentage of the mass of solid heated.

[1]

(c) The theoretical loss in mass is 33.5%.

The proportions of  $CuCO_3$  and  $Cu(OH)_2$  in the basic carbonate can vary from the 1:1 ratio given in the formula.

Make use of the following information to account for the difference between the value you have calculated in **(b)** and the theoretical percentage loss in mass.

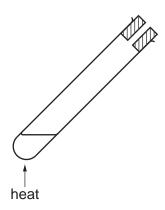
1 mol CuCO<sub>3</sub>(s)  $\rightarrow$  1 mol CO<sub>2</sub>(g) 1 mol Cu(OH)<sub>2</sub>(s)  $\rightarrow$  1 mol H<sub>2</sub>O(g)

Assume that 1 mol of any sample of the solid basic carbonate contains 1 mol H<sub>2</sub>O.

 $[M_r: CO_2, 44.0; H_2O, 18.0]$ 

[11]

(d) Add to the diagram below additional standard laboratory apparatus that would enable you to collect and measure the volume of carbon dioxide evolved in the experiment. Ensure that your apparatus does not also collect and measure any of the water vapour evolved.



[2]

[Total: 10]

**3 FA 4**, **FA 5**, **FA 6** and **FA 7** are aqueous solutions each containing one of the ions  $Al^{3+}$ ,  $Mg^{2+}$ ,  $Pb^{2+}$ ,  $Zn^{2+}$ .

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You will carry out the following tests on each of the solutions.

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.

(a) Carry out the following tests. Record your observations in the spaces provided in the table.

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## You should rinse and reuse test-tubes where possible.

	40.04		observations							
	test	FA 4	FA 5	FA 6	FA 7					
(i)	To 1 cm depth of solution in a test-tube add 1 cm depth of aqueous sodium hydroxide. Swirl the tube, then									
	add a further 2 cm depth of aqueous sodium hydroxide.									
(ii)	To 1 cm depth of solution in a test-tube add 1 cm depth of aqueous ammonia. Swirl the tube, then									
	add a further 2 cm depth of aqueous ammonia.									
(iii)	To 1 cm depth of solution in a test-tube add 1 cm depth of aqueous potassium iodide.									

[4]

(b) Use the Qualitative Analysis Notes on page 11 to identify the cation present in each of the solutions. Complete the table below to identify each ion and to give supporting evidence from your observations. solution cation supporting evidence **FA 4 FA** 5 FA 6 **FA** 7 [4]

For Examiner's Use

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## Do not carry out the following test.

(c)	Use the Qualitative Analysis Notes on pages 11 and 12 to select a further reagent that
	could be used to confirm the presence of Pb2+ in one of the solutions FA 4, FA 5, FA 6
	and <b>FA 7</b> .

The reagon to minimum in [1]	The reagent is	. ['	1]	
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(d) FA 8 contains aqueous copper(II) ions.

Carry out the following tests and make careful observations of all that happens in each experiment.

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	test	observations
(i)	To 2 cm depth of FA 8 in a boiling-tube add 1 spatula measure of zinc metal powder.  Leave to stand for 1 minute, then add 2 cm depth of distilled water and	
	leave to stand for a further 2 minutes.	
(ii)	To 1 cm depth of FA 8 in a test-tube add 1 cm depth of concentrated hydrochloric acid. (Care: corrosive) Retain the mixture for use in (iii).	
(iii)	Using a dropping pipette transfer 1 cm depth of the solution in (ii) into another test-tube.	
	Add 1 cm depth of water and shake the tube.	

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[5]

(e) From your observations in (d)(i) complete the equation below:

$$Cu^{2+}(aq) + Zn(s) \rightarrow$$

[1]

[Total: 15]

# **Qualitative Analysis Notes**

Key: [ ppt. = precipitate ]

## 1 Reactions of aqueous cations

	reaction	on with
ion	NaOH(aq)	NH <sub>3</sub> (aq)
aluminium, Al <sup>3+</sup> (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH <sub>4</sub> +(aq)	no ppt. ammonia produced on heating	
barium, Ba <sup>2+</sup> (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca <sup>2+</sup> (aq)	white ppt. with high [Ca <sup>2+</sup> (aq)]	no ppt.
chromium(III), Cr <sup>3+</sup> (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu <sup>2+</sup> (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe <sup>2+</sup> (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 <sup>3+</sup> (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
lead(II), Pb <sup>2+</sup> (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
magnesium, Mg <sup>2+</sup> (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn <sup>2+</sup> (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 <sup>2+</sup> (aq)	white ppt. soluble in excess	white ppt. soluble in excess

[Lead(II) ions can be distinguished from aluminium ions by the insolubility of lead(II) chloride.]

## 2 Reactions of anions

ion	reaction
carbonate,	CO <sub>2</sub> liberated by dilute acids
CO <sub>3</sub> <sup>2-</sup>	
chromate(VI), CrO <sub>4</sub> <sup>2-</sup> (aq)	yellow solution turns orange with H <sup>+</sup> (aq); gives yellow ppt. with Ba <sup>2+</sup> (aq); gives bright yellow ppt. with Pb <sup>2+</sup> (aq)
chloride,	gives white ppt. with Ag <sup>+</sup> (aq) (soluble in NH <sub>3</sub> (aq));
C <i>l</i> ⁻(aq)	gives white ppt. with Pb <sup>2+</sup> (aq)
bromide,	gives cream ppt. with Ag <sup>+</sup> (aq) (partially soluble in NH <sub>3</sub> (aq));
Br <sup>-</sup> (aq)	gives white ppt. with Pb <sup>2+</sup> (aq)
iodide,	gives yellow ppt. with Ag <sup>+</sup> (aq) (insoluble in NH <sub>3</sub> (aq));
I⁻(aq)	gives yellow ppt. with Pb <sup>2+</sup> (aq)
nitrate,	$\mathrm{NH_3}$ liberated on heating with $\mathrm{OH^-}(\mathrm{aq})$ and $\mathrm{A}\mathit{l}$ foil
NO <sub>3</sub> <sup>-</sup> (aq)	
nitrite,	$\mathrm{NH_3}$ liberated on heating with $\mathrm{OH^-}(\mathrm{aq})$ and $\mathrm{A}\mathit{l}$ foil;
NO <sub>2</sub> <sup>-</sup> (aq)	NO liberated by dilute acids (colourless NO → (pale) brown NO <sub>2</sub> in air)
sulfate,	gives white ppt. with Ba <sup>2+</sup> (aq) or with Pb <sup>2+</sup> (aq) (insoluble in excess dilute
SO <sub>4</sub> <sup>2-</sup> (aq)	strong acid)
sulfite,	SO <sub>2</sub> liberated with dilute acids;
SO <sub>3</sub> <sup>2-</sup> (aq)	gives white ppt. with Ba <sup>2+</sup> (aq) (soluble in excess dilute strong acid)

## 3 Tests for gases

gas	test and test result
ammonia, NH <sub>3</sub>	turns damp red litmus paper blue
carbon dioxide, CO <sub>2</sub>	gives a white ppt. with limewater (ppt. dissolves with excess CO <sub>2</sub> )
chlorine, Cl <sub>2</sub>	bleaches damp litmus paper
hydrogen, H <sub>2</sub>	"pops" with a lighted splint
oxygen, O <sub>2</sub>	relights a glowing splint
sulfur dioxide, SO <sub>2</sub>	turns acidified aqueous potassium dichromate(VI) from orange to green

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