UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
General Certificate of Education
Advanced Subsidiary Level and Advanced Level

CANDIDATE NAME									
CENTRE NUMBER						CANDIDATE NUMBER				

3933279290

CHEMISTRY **9701/31**

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
Laboratory

For Examiner's Use	
1	
2	
3	
Total	

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

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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^+ ions in the following reaction.

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

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

You are provided with the following reactants.

FA 1 hydrochloric acid
FA 2 containing 15.0 g dm^{-3} sodium thiosulfate, $Na_2S_2O_3.5H_2O$
aqueous potassium iodate(V), KIO_3
aqueous potassium iodide, KI

(a) Method

- Fill a burette with **FA 2**.
- Pipette 25.0 cm^3 of **FA 1** into the conical flask.
- Use a 25 cm^3 measuring cylinder to add to the flask 10 cm^3 of aqueous potassium iodate(V) **and** 10 cm^3 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^3

- 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.

I	
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IV	
V	
VI	
VII	

[7]

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(b) From your titration results obtain a suitable value to be used in your calculation. Show clearly how you have obtained this value.

For Examiner's Use

25.0 cm^3 of **FA 1** require ..... cm^3 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} $Na_2S_2O_3.5H_2O$.
[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^{-3}.

(ii) Calculate how many moles of $Na_2S_2O_3$ are contained in the volume of **FA 2** recorded in **(b)**.

..... mol of $Na_2S_2O_3$

(iii) Calculate how many moles of iodine, I_2 reacted with the $Na_2S_2O_3$ 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, HC*l*, reacted with an excess of potassium iodate(V) and an excess of potassium iodide to produce the amount of iodine calculated in **(iii)**.

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

..... mol of HC*l* produced the amount of iodine calculated in **(iii)**.

I	
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(v) Calculate the concentration, in mol dm^{-3}, of HCl in **FA 1**.

For Examiner's Use

The concentration of HCl in **FA 1** is ..... mol dm^{-3}.
[5]

(d)

Each reading with a burette has a maximum error of ±0.05 cm^3.
Grade B volumetric (bulb) pipettes are calibrated to ±0.06 cm^3.

(i) Calculate the maximum error in the volume run from the burette recorded in any titration.

The maximum error is ..... cm^3.

(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^3 of **FA 1** was pipetted into the conical flask.

The error was ..... %.
[2]

[Total: 15]

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2 **FA 3** is powdered basic copper(II) carbonate, a hydrated mixture of copper(II) carbonate and copper(II) hydroxide.
The approximate formula for the basic carbonate is $CuCO_3.Cu(OH)_2.H_2O$.

For Examiner's Use

When heated, basic copper(II) carbonate decomposes.

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

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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II	
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Calculations

For Examiner's Use

(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 $CuCO_3(s) \longrightarrow$ 1 mol $CO_2(g)$
1 mol $Cu(OH)_2(s) \longrightarrow$ 1 mol $H_2O(g)$

Assume that 1 mol of any sample of the solid basic carbonate contains 1 mol H_2O.

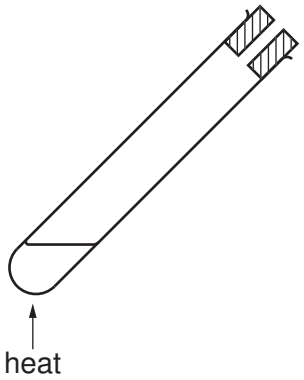
[M_r: CO_2, 44.0; H_2O, 18.0]

.....

.....

..... [1]

(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+}.

For Examiner's Use

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**.

I	
II	
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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.

You should rinse and reuse test-tubes where possible.

test		*observations*			
		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]

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(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.

For Examiner's Use

solution	*cation*	*supporting evidence*
FA 4		
FA 5		
FA 6		
FA 7		

[4]

I	
II	
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IV	

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 Pb^{2+} in one of the solutions **FA 4**, **FA 5**, **FA 6** and **FA 7**.

The reagent is ..... . [1]

(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.	

[5]

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

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

[1]

[Total: 15]

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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
lead(II), Pb^{2+}(aq)	white ppt. soluble in excess	white 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

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

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[Turn over

2 Reactions of anions

ion	*reaction*
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chromate(VI), CrO_4^{2-} (aq)	yellow solution turns orange with H^+(aq); gives yellow ppt. with Ba^{2+}(aq); gives bright yellow ppt. with Pb^{2+}(aq)
chloride, Cl^- (aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq)); gives white ppt. with Pb^{2+}(aq)
bromide, Br^- (aq)	gives cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq)); gives white ppt. with Pb^{2+}(aq)
iodide, I^- (aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(aq)); gives yellow ppt. with Pb^{2+}(aq)
nitrate, NO_3^- (aq)	NH_3 liberated on heating with OH^-(aq) and Al foil
nitrite, NO_2^- (aq)	NH_3 liberated on heating with OH^-(aq) and Al foil; NO liberated by dilute acids (colourless NO → (pale) brown NO_2 in air)
sulfate, SO_4^{2-} (aq)	gives white ppt. with Ba^{2+}(aq) or with Pb^{2+}(aq) (insoluble in excess dilute strong acid)
sulfite, SO_3^{2-} (aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in excess dilute strong acid)

3 Tests for gases

gas	*test and test result*
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 dichromate(VI) from orange to green

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