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

CANDIDATE NAME

CENTRE NUMBER | | | | | | CANDIDATE NUMBER | | | | |

9572253701

CHEMISTRY **9701/32**

Advanced Practical Skills **May/June 2011**

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 **12** printed pages.

UNIVERSITY *of* CAMBRIDGE International Examinations

[Turn over

1 Many solid salts exist as hydrates. One example is washing soda – hydrated sodium carbonate, $Na_2CO_3.\mathbf{x}H_2O$.

You are to determine the value of **x** in $Na_2CO_3.\mathbf{x}H_2O$ by titration with hydrochloric acid.

FB 1 is hydrated sodium carbonate, $Na_2CO_3.\mathbf{x}H_2O$.
FB 2 is 0.200 mol dm^{-3} hydrochloric acid, HCl.
methyl orange indicator

The equation for the reaction between hydrated sodium carbonate and hydrochloric acid is shown below.

$$Na_2CO_3.\mathbf{x}H_2O + 2HCl \longrightarrow 2NaCl + CO_2 + (\mathbf{x} + 1)\,H_2O$$

(a) Method

- Weigh the tube containing **FB 1**, the hydrated sodium carbonate. Record the mass in the space below.
- Add all the **FB 1** into a 250 cm^3 glass beaker. Reweigh the tube containing any residual **FB 1**. Record the mass in the space below.
- Calculate and record the mass of **FB 1** used.

mass of **FB 1** used = ... g

- Use the 50 cm^3 measuring cylinder to add, in total, about 100 cm^3 of distilled water to the beaker.
- Stir with a glass rod until all the solid has dissolved.
- Pour the solution from the beaker into the 250 cm^3 graduated (volumetric) flask.
- Wash out the beaker thoroughly with distilled water and add the washings to the graduated flask.
- Make up the contents of the graduated flask to the 250 cm^3 mark with distilled water.
- Shake the flask to mix the solution of **FB 1**.
- Pipette 25.0 cm^3 of your solution of **FB 1** into a conical flask.
- Add to the flask a few drops of methyl orange indicator and place the flask on a white tile.
- Fill the burette with hydrochloric acid, **FB 2**.
- Titrate the solution of **FB 1** with the acid until the end-point is reached.

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.
- Make certain any recorded results show the precision of your practical work.
- Record, in an appropriate form below, all your burette readings and the volume of **FB 2** added in each accurate titration.

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[7]

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

25.0 cm^3 of **FB 1** required .... cm^3 of **FB 2**. [1]

(c) Calculations

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

(i) Calculate how many moles of HC*l* were present in the volume of **FB 2** calculated in **(b)**.

.... mol of HC*l*

(ii) Calculate how many moles of $Na_2CO_3.\mathbf{x}H_2O$ were present in 25.0 cm^3 of the solution of **FB 1**.

$$Na_2CO_3.\mathbf{x}H_2O + 2HCl \longrightarrow 2NaCl + CO_2 + (\mathbf{x} + 1)\ H_2O$$

.... mol of $Na_2CO_3.\mathbf{x}H_2O$

(iii) Calculate how many moles of $Na_2CO_3.\mathbf{x}H_2O$ were present in 250 cm^3 of the solution of **FB 1**.

.... mol of $Na_2CO_3.\mathbf{x}H_2O$

(iv) Use the mass of **FB 1** that you weighed out to calculate the relative formula mass of $Na_2CO_3.\mathbf{x}H_2O$.

relative formula mass = ...

I	
II	
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VI	

(v) Calculate the value of **x** in $Na_2CO_3.\mathbf{x}H_2O$.
[A_r: H, 1.0; C, 12.0; O, 16.0; Na, 23.0]

x = ... [6]

(d) The error in a single burette reading is $\pm 0.05\,cm^3$.

What is the percentage error in the titre volume calculated in **(b)**?

percentage error = ... % [1]

[Total: 15]

2 You are to determine the enthalpy change for the reaction of hydrated sodium car… $Na_2CO_3.\mathbf{x}H_2O$, with hydrochloric acid, $HCl(aq)$.

The equation for this reaction is shown below.

$$Na_2CO_3.\mathbf{x}H_2O(s) + 2HCl(aq) \longrightarrow 2NaCl(aq) + CO_2(g) + (\mathbf{x} + 1)H_2O(l)$$

FB 3 is hydrated sodium carbonate, $Na_2CO_3.\mathbf{x}H_2O$.
FB 4 is 4.00 mol dm^{-3} hydrochloric acid, HCl.

Make sure that in this experiment you use the hydrochloric acid labelled FA 4.

(a) Method

- Support the plastic cup in a 250 cm^3 beaker.
- Use a measuring cylinder to transfer 25 cm^3 of **FB 4** into the plastic cup.
- Tilt the beaker so that the bulb of the thermometer is covered by the solution.
- Measure and record the temperature of the solution.
- Measure and record the mass of the tube containing **FB 3**.
- **Carefully** tip all the hydrated sodium carbonate from the weighed tube into the plastic cup.
- **There will be effervescence. Add the solid in small portions with constant stirring using the thermometer.**
- Record the lowest temperature obtained.
- Reweigh the tube containing any residual **FB 3**.

In the space below, record, in an appropriate form,

- both balance readings,
- both temperature measurements,
- the mass of **FB 3** used in the experiment,
- the fall in temperature.

I	
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[5]

(b) Calculation

Show your working and express your answers to **three** significant figures.

(i) Calculate the heat energy change involved in the reaction.
(You may assume that 4.3 J are required to change the temperature of 1.0 cm^3 of any solution by 1.0 °C.)

(ii) Calculate the number of moles of $Na_2CO_3.\mathbf{x}H_2O$ you used in **2(a)**.
You will need to use the relative formula mass you calculated in **1(c)(v)**.
If you were unable to calculate the relative formula mass in **1(c),** assume it is 25
but note that this is **not** the correct value.

.... mol of $Na_2CO_3.\mathbf{x}H_2O$

(iii) Calculate the enthalpy change, in $kJ\,mol^{-1}$, for the following reaction.

$$Na_2CO_3.\mathbf{x}H_2O(s) + 2HCl(aq) \rightarrow 2NaCl(aq) + CO_2(g) + (\mathbf{x} + 1)H_2O(l)$$

I	
II	
III	
IV	

enthalpy change = ... $kJ\,mol^{-1}$

sign *value*

[4]

(c) In experiments carried out to determine enthalpy changes, heat transfer between the surroundings and the reactants is a significant source of error. This problem can be limited by improved insulation.

Apart from modifications made to minimise heat transfer, suggest **one** possible improvement you could make to the apparatus or procedure to make the determination of the enthalpy change more accurate.

....

....

.... [1]

[Total: 10]

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 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.

If any solution is warmed, a boiling tube MUST be used.

Rinse and reuse test-tubes and boiling tubes where possible.

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

(a) (i) You are provided with three solutions **FB 5**, **FB 6** and **FB 7**, each of which contains a single cation. One of these cations is aluminium, one is magnesium and the other is lead.

Use the information in the Qualitative Analysis Notes on page 11 to select reagents that would enable you to determine the cation in **FB 5**, **FB 6** and **FB 7**.

reagents ....

Carry out suitable tests and record the results of your experiments in an appropriate form in the space below.

[4]

I	
II	
III	
IV	

(ii) Complete the table below.

	FB 5	**FB 6**	**FB 7**
cation			

What is the **minimum** evidence from your observations, that enables you to identify these cations?

The minimum evidence for the cation in **FB 5** is ....

....

The minimum evidence for the cation in **FB 6** is ....

....

The minimum evidence for the cation in **FB 7** is ....

....

[4]

I	
II	
III	
IV	

(b) You are provided with solid **FB 8**.

Carry out the tests and complete the following table.

test	*observations*
(i) To a spatula measure of **FB 8**, in a test-tube, add about a 1 cm depth of distilled water to make a solution. To this solution add 4 pieces of magnesium ribbon.	
(ii) To a small spatula measure of **FB 8**, in a boiling tube, add 3 cm depth of aqueous sodium hydroxide. Warm gently and carefully.	
(iii) To a spatula measure of **FB 8**, in a test-tube, add about a 1 cm depth of distilled water to make a solution. To this solution add an equal volume of aqueous sodium hydroxide. .... To this mixture add a small volume of hydrogen peroxide.	 ....

I	
II	
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Identify the metal ion present in **FB 8**.

metal ion = ...

State the change in oxidation number (state) of this metal ion that is occurring in test **(i)**.

oxidation number (state) changes from .... to ....

State the change in oxidation number (state) of this metal ion that is occurring in test **(iii)**.

oxidation number (state) changes from .... to ....

[7]

[Total: 15]

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.]

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 acids)
sulfite, SO_3^{2-} (aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in excess dilute strong acids)

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