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

CANDIDATE
NAME

CENTRE
NUMBER

CANDIDATE
NUMBER

**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, $Na_2CO_3.\textbf{x}H_2O$.

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

   **FB 1** is hydrated sodium carbonate, $Na_2CO_3.\textbf{x}H_2O$.
   **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.

$$Na_2CO_3.\textbf{x}H_2O \ + \ 2HCl \longrightarrow \ 2NaCl \ + \ CO_2 \ + \ (\textbf{x} + 1) \, H_2O$$
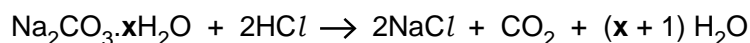
**(a) Method**

- 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.

**3**

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.

| | |
|---|---|
| I | |
| II | |
| III | |
| IV | |
| V | |
| VI | |
| VII | |

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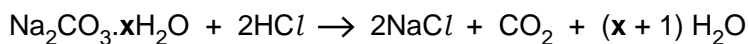
Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

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

.................. mol of $HCl$

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

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

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

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

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

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

| | |
|---|---|
| I | |
| II | |
| III | |
| IV | |
| V | |
| VI | |

relative formula mass = ....................

**5**

**(v)** Calculate the value of **x** in $Na_2CO_3.xH_2O$.
[$A_r$:  H, 1.0;  C, 12.0;  O, 16.0;  Na, 23.0]

**x** = .................. [6]

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

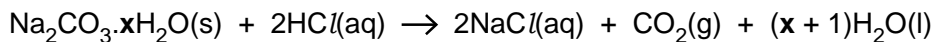What is the percentage error in the titre volume calculated in **(b)**?

percentage error =  ...................... % [1]

[Total: 15]

                                       **[Turn over**

**6**

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

The equation for this reaction is shown below.

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

**FB 3** is hydrated sodium carbonate, $Na_2CO_3.\textbf{x}H_2O$.
**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 | |
| II | |
| III | |
| IV | |
| V | |

[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 $1.0\,°C$.)

heat energy produced = .................................................... J

**(ii)** Calculate the number of moles of $Na_2CO_3.xH_2O$ 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 259 but note that this is **not** the correct value.

.................... mol of $Na_2CO_3.xH_2O$

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

$$Na_2CO_3.xH_2O(s) + 2HCl(aq) \longrightarrow 2NaCl(aq) + CO_2(g) + (x + 1)H_2O(l)$$
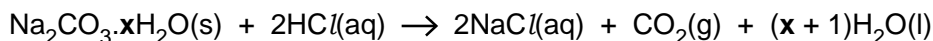
| | |
|---|---|
| I | |
| II | |
| III | |
| IV | |

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]

**8**

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 |  |

**9**

(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 |  |

**10**

**(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.<br><br>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.<br><br>...................................................<br><br>To this mixture add a small volume of hydrogen peroxide. | ......................................................... |

| | |
|---|---|
| I | |
| II | |
| III | |
| IV | |
| V | |
| VI | |
| VII | |

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]

**11**

**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.]

 **[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 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 |