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

CANDIDATE NAME

CENTRE NUMBER

CANDIDATE NUMBER

*2645126014*

**CHEMISTRY** **9701/32**

Paper 32 Advanced Practical Skills **May/June 2009**

**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 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 are advised to show all working in calculations.
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.

SP (SHW 00157 2/08) T67843/4


UNIVERSITY *of* CAMBRIDGE
International Examinations

**[Turn over**

For Examiner's Use

1 You are provided with the following.
**FB 1** is 0.023 mol dm$^{-3}$ potassium manganate(VII), $KMnO_4$.
**FB 2** is aqueous ethanedioic acid, $H_2C_2O_4$, made by dissolving the hydrated salt, $H_2C_2O_4.2H_2O$.
You are also provided with the following.
1.0 mol dm$^{-3}$ sulfuric acid, $H_2SO_4$
distilled water

You are required to determine the concentration, in g dm$^{-3}$, of hydrated ethanedioic acid, $H_2C_2O_4.2H_2O$, in **FB 2**.

**Dilution of FB 2**

**(a)** By using a burette, measure between 42.50 cm$^3$ and 43.00 cm$^3$ of **FB 2** into the 250 cm$^3$ graduated flask, labelled **FB 3**.
Record your burette readings and the volume of **FB 2** added to the flask in the space below.

Make up the contents of the flask to the 250 cm$^3$ mark with distilled water. Place the stopper in the flask and mix the contents thoroughly by slowly inverting the flask a number of times.

**Titration**

Fill a second burette with **FB 1**.

Pipette 25.0 cm$^3$ of **FB 3** into a conical flask. Use the measuring cylinder provided to add to the flask 25 cm$^3$ of 1.0 mol dm$^{-3}$ sulfuric acid and 40 cm$^3$ of distilled water.
Put the thermometer in the flask and heat the solution until the temperature is just over 65 °C.
Carefully remove the thermometer and place the hot flask under the burette. If the neck of the flask is too hot to hold safely, use a folded paper towel to hold the flask.
Run in 1 cm$^3$ of **FB 1**. Swirl the flask until the colour of the potassium manganate(VII) has disappeared then continue the titration as normal until a permanent pale pink colour is obtained. This is the end-point.

**If a brown colour appears during the titration**, reheat the flask to 65 °C. The brown colour should disappear and the titration can be completed as above.
**If the brown colour does not disappear on reheating**, discard the solution and start the titration again.

**Perform a rough (trial) titration and sufficient further titrations to obtain reliable results.**
Record your titration results in the space below. Make certain that your recorded results show the precision of your working.

| | |
|---|---|
| i | |
| ii | |
| iii | |
| iv | |
| v | |
| vi | |

[6]

*For Examiner's Use*

**(b)** From your titration results obtain a volume of **FB 1** to be used in your calculations. Show clearly how you obtained this volume.

[1]

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

**(c)** Calculate how many moles of $KMnO_4$ were run from the burette into the conical flask.

.................... mol of $KMnO_4$ were run from the burette into the conical flask.

Put the correct number of electrons into each of the following half-equations to balance the electrical charges.

$$MnO_4^- + 8H^+ + \text{........} e^- \longrightarrow Mn^{2+} + 4H_2O$$

$$C_2O_4^{2-} \longrightarrow 2CO_2 + \text{........} e^-$$

Calculate how many moles of ethanedioate ions, $C_2O_4^{2-}$, reacted with the $KMnO_4$ run from the burette.

.................... mol of ethanedioate ions reacted with the $KMnO_4$ run from the burette.

Calculate the concentration, in mol dm$^{-3}$, of $C_2O_4^{2-}$ in **FB 3**.

The concentration of $C_2O_4^{2-}$ in **FB 3** is .................... mol dm$^{-3}$.

Calculate the concentration, in mol dm$^{-3}$, of $C_2O_4^{2-}$ in **FB 2**.

The concentration of $C_2O_4^{2-}$ in **FB 2** is .................... mol dm$^{-3}$.

| | |
|---|---|
| i | |
| ii | |
| iii | |
| iv | |
| v | |

For Examiner's Use

Calculate the concentration, in $g\,dm^{-3}$, of $H_2C_2O_4.2H_2O$ in **FB 2**.
[$A_r$: H, 1.0; C, 12.0; O, 16.0]

**FB 2** contains .................... $g\,dm^{-3}$ $H_2C_2O_4.2H_2O$.
[5]

[Total: 12]

**2** You are provided with the following.
**FB 4**, anhydrous sodium carbonate, $Na_2CO_3$
**FB 5**, solid sodium hydrogencarbonate, $NaHCO_3$
$3.0\,mol\,dm^{-3}$ hydrochloric acid

You are to determine the enthalpy change of reaction, $\Delta H$, for the following reactions.

$$Na_2CO_3(s) + 2HCl(aq) \rightarrow 2NaCl(aq) + CO_2(g) + H_2O(l) \quad \Delta H_1$$

$$NaHCO_3(s) + HCl(aq) \rightarrow NaCl(aq) + CO_2(g) + H_2O(l) \quad \Delta H_2$$

**(a) Reaction of FB 4, $Na_2CO_3$, with an excess of $3.0\,mol\,dm^{-3}$ hydrochloric acid**

Read through the following instructions carefully before starting the experimental work.

- Support the plastic cup in the $250\,cm^3$ beaker provided.
- Use the measuring cylinder to transfer $50\,cm^3$ of $3.0\,mol\,dm^{-3}$ hydrochloric acid into the plastic cup.
- Weigh the tube containing **FB 4**, anhydrous sodium carbonate.
- Measure and record the steady temperature of the acid in the beaker.
- Add the contents of the tube to the acid in three separate lots, taking care that the mixture does not overflow.
- Stir and record the highest temperature obtained.
- Reweigh the tube containing residual **FB 4**.

Record in an appropriate form below all of your weighings and temperature measurements together with the mass, $\mathbf{m_1}$, of **FB 4** added and the temperature rise, $\Delta \mathbf{T_1}$.

[1]

For Examiner's Use

**(b)** Calculate the temperature rise per gram of **FB 4**, $Na_2CO_3$, used in the experiment.

$$\frac{\Delta \mathbf{T}_1}{\mathbf{m}_1} = \text{..................................... } °C\,g^{-1}$$ [2]

**(c)** Calculate the enthalpy change, $\Delta H_1$, for the following reaction.

$$Na_2CO_3(s) + 2HCl(aq) \longrightarrow 2NaCl(aq) + CO_2(g) + H_2O(l)$$

$$\Delta H_1 = -\left(22.79 \times \frac{\Delta \mathbf{T}_1}{\mathbf{m}_1}\right) \text{ kJ mol}^{-1}$$

$\Delta H_1 = -$ ...................................... $kJ\,mol^{-1}$

**(d)** **Reaction of FB 5, $NaHCO_3$, with an excess of 3.0 mol dm$^{-3}$ hydrochloric acid**

50 cm$^3$ of 3.0 mol dm$^{-3}$ hydrochloric acid contains 0.15 mol HC$l$.

Calculate the mass of $NaHCO_3$ that will react with 0.15 mol HC$l$.
[$A_r$: C, 12.0; H, 1.0; O, 16.0; Na, 23.0]

[1]

| |
|---|
| The reaction of $NaHCO_3$(s) and HC$l$(aq) is endothermic.<br>The expected **fall** in temperature when 1.0 g $NaHCO_3$(s) is added to 50 cm$^3$, an excess, of 3.0 mol dm$^{-3}$ HC$l$ is approximately 1.5 °C. |

**(e)** The error in reading a –10 °C to +110 °C thermometer is ±0.5 °C.

What is the maximum error when using two temperature measurements to calculate a temperature change?

The maximum error is ± ........................................ °C. [1]

**(f)** Determine the maximum percentage error in the calculated temperature change when 1.0 g of $NaHCO_3$ is added to 50 cm$^3$ of 3.0 mol dm$^{-3}$ hydrochloric acid.

The maximum error is ± .......................................... %. [1]

For Examiner's Use

**(g)** Use your answer to **(d)** and the expected temperature change of $-1.5\,°C\,g^{-1}$ to select a mass of **FB 5**, $NaHCO_3$, to use in an experiment with $50\,cm^3$ of $3.0\,mol\,dm^{-3}$ hydrochloric acid. The mass selected should give an appropriate, measurable, temperature fall.
**Note:** The hydrochloric acid should be in excess and the percentage error in temperature measurement should be kept to a minimum.

Mass of **FB 5** to be used = .................................................. g.

Predicted temperature fall = ................................................ °C.
[1]

**(h)** **Read through the instructions before starting any practical work.**

- Empty, rinse, and shake dry the plastic cup used in **(a)**.
- Support the plastic cup in the $250\,cm^3$ beaker provided.
- Use the measuring cylinder to transfer $50\,cm^3$ of $3.0\,mol\,dm^{-3}$ hydrochloric acid into the plastic cup.
- Weigh the empty tube labelled $NaHCO_3$.
- Add the mass of **FB 5** you have selected in **(g)** to the tube and reweigh.
- Measure and record the steady temperature of the acid in the beaker.
- Add the contents of the tube to the acid in three separate lots, taking care that the mixture does not overflow.
- Stir and record the lowest temperature obtained.
- Reweigh the tube containing residual **FB 5**.

Record in an appropriate form below all of your weighings and temperature measurements together with the mass, $\mathbf{m_2}$, of **FB 5** added and the temperature fall, $\Delta\mathbf{T_2}$.

[2]

**(i)** Calculate the temperature fall per gram of **FB 5**, $NaHCO_3$, used in the experiment.

$\dfrac{\Delta T_2}{m_2}$ = ......................................... $°C\,g^{-1}$
[3]

For Examiner's Use

**(j)** Calculate the enthalpy change, $\Delta H_2$, for the following reaction.

$$NaHCO_3(s) + HCl(aq) \rightarrow NaCl(aq) + CO_2(g) + H_2O(l)$$

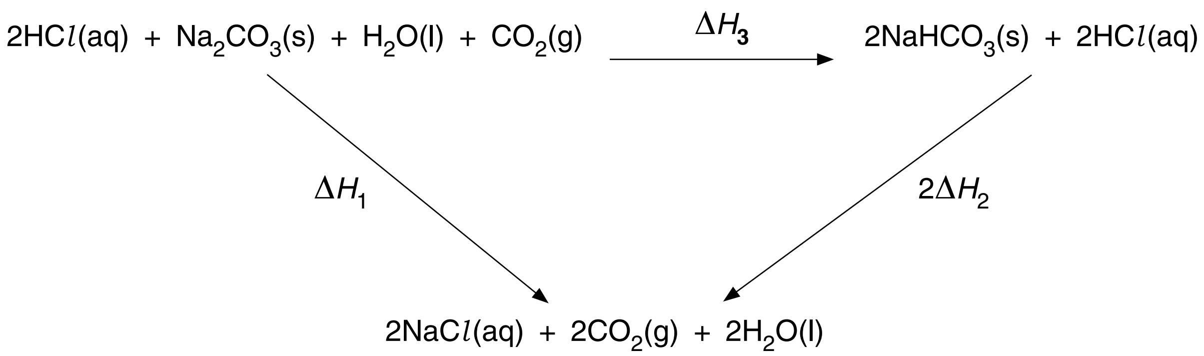
$$\Delta H_2 = +\left(18.06 \times \frac{\Delta \mathbf{T}_2}{\mathbf{m}_2}\right)\ \text{kJ mol}^{-1}$$

$\Delta H_2$ = + ...................................... kJ mol$^{-1}$

**(k)** It is not possible to measure experimentally the enthalpy change, $\Delta H_3$, for the following reaction as it does not take place in the laboratory.

$$Na_2CO_3(s) + CO_2(g) + H_2O(l) \rightarrow 2NaHCO_3(s)$$

It is possible, however, to calculate a "theoretical" value of $\Delta H_3$ for this reaction from the results of the experiments you have carried out and a Hess cycle.




Derive an equation to link $\Delta H_1$, $\Delta H_2$, and $\Delta H_3$.

Use your equation and the results from **(c)** and **(j)** to calculate a value for $\Delta H_3$.

$\Delta H_3$ = ...................................... kJ mol$^{-1}$

[2]

**(l)** Suggest a modification to the experimental method in order to reduce the transfer of heat energy to or from the contents of the plastic cup during the experiment.

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

.................................................................................................................................... [1]

[Total: 15]

For Examiner's Use

**3** **FB 6** and **FB 7** each contain one of the following sodium halides, NaC*l*, NaBr, NaI.

**(a)** Place half of the solid **FB 6** provided in a test-tube. Half fill the test-tube with distilled water and shake to dissolve the solid. Label the tube **FB 6**.
Do the same with **FB 7**, labelling the tube **FB 7**.
Keep the remaining solid for **(c)**.

**(b)** You are to select appropriate reagents from those provided and to perform tests to identify which halide ion is present in **FB 6** and which in **FB 7**.
**Retain some of the FB 7 solution for test (d).**

In an appropriate form below record the tests performed and the results of those tests.

| | |
|---|---|
| i | |
| ii | |
| iii | |
| iv | |

From the recorded observations the following halides are identified.

**FB 6** contains ....................................................

**FB 7** contains ....................................................

[4]

**(c)** Carry out the following tests. **[Care: unpleasant fumes may be produced]**

| *test* | *observations* | |
|---|---|---|
| | **FB 6** | **FB 7** |
| Place the remaining solid in a clean, dry test-tube and add 5 drops of concentrated sulfuric acid (**care: the concentrated acid is very corrosive**),<br><br>then as soon as you have made your observation, | | |
| half fill the test-tube with distilled water to dissolve the remaining solid and any fumes produced. | | |
| Transfer 1 cm depth of the resulting solution to a test-tube and add a few drops of starch solution. | | |

[2]

**(d)** Carry out the following tests.

| *test* | *observations* |
|---|---|
| Place 1 cm depth of the solution of **FB 7** prepared in **(a)** in a test-tube.<br><br>Add 1 cm depth of aqueous bromine,<br>**[Care: unpleasant fumes]**<br>then, | |
| add a few drops of starch solution. | |

[1]

**(e)** Use your observations and knowledge of halogen chemistry to explain the reactions in **(c)** and identify the chemical behaviour of the concentrated sulfuric acid in the reaction.

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

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

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

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

Use your observations and knowledge of halogen chemistry to explain what happens when the solutions are mixed in **(d)**.

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

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

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

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

[3]

For Examiner's Use

(f) **FB 8** and **FB 9** each contain one cation from those listed on page 11.

Carry out the following tests to identify the cation present in each solution.

| *test* | *observations* | |
|---|---|---|
| | **FB 8** | **FB 9** |
| To 1 cm depth of solution in a test-tube, add aqueous sodium hydroxide a little at a time<br><br>then, | | |
| add an excess of the reagent to give no more than 4 cm depth of solution in the test-tube. | | |
| To 1 cm depth of solution in a test-tube, add aqueous ammonia a little at a time<br><br>then, | | |
| add an excess of the reagent to give no more than 4 cm depth of solution in the test-tube. | | |

The cation present in **FB 8** is ..................................................

The cation present in **FB 9** is ..................................................
[3]

[Total: 13]

## 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.<br>soluble in excess | white ppt.<br>insoluble in excess |
| ammonium, $NH_4^+$(aq) | no ppt.<br>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.<br>insoluble in excess |
| copper(II), $Cu^{2+}$(aq) | pale blue ppt.<br>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<br>insoluble in excess | green ppt. turning brown on contact with air<br>insoluble in excess |
| iron(III), $Fe^{3+}$(aq) | red-brown ppt.<br>insoluble in excess | red-brown ppt.<br>insoluble in excess |
| lead(II), $Pb^{2+}$(aq) | white ppt.<br>soluble in excess | white ppt.<br>insoluble in excess |
| magnesium, $Mg^{2+}$(aq) | white ppt.<br>insoluble in excess | white ppt.<br>insoluble in excess |
| manganese(II), $Mn^{2+}$(aq) | off-white ppt. rapidly turning brown on contact with air<br>insoluble in excess | off-white ppt. rapidly turning brown on contact with air<br>insoluble in excess |
| zinc, $Zn^{2+}$(aq) | white ppt.<br>soluble in excess | white ppt.<br>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 $H^+(aq)$;<br>gives yellow ppt. with $Ba^{2+}(aq)$;<br>gives bright yellow ppt. with $Pb^{2+}(aq)$ |
| chloride, $Cl^-$ (aq) | gives white ppt. with $Ag^+(aq)$ (soluble in $NH_3(aq)$);<br>gives white ppt. with $Pb^{2+}(aq)$ |
| bromide, $Br^-$ (aq) | gives cream ppt. with $Ag^+(aq)$ (partially soluble in $NH_3(aq)$);<br>gives white ppt. with $Pb^{2+}(aq)$ |
| iodide, $I^-$ (aq) | gives yellow ppt. with $Ag^+(aq)$ (insoluble in $NH_3(aq)$);<br>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,<br>NO liberated by dilute acids<br>(colourless NO $\longrightarrow$ (pale) brown $NO_2$ in air) |
| sulfate, $SO_4^{2-}(aq)$ | gives white ppt. with $Ba^{2+}(aq)$ (insoluble in excess dilute strong acid)<br>gives white ppt. with $Pb^{2+}(aq)$ |
| sulfite, $SO_3^{2-}(aq)$ | $SO_2$ liberated with dilute acids;<br>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<br>(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 aqueous acidified potassium dichromate(VI) (aq) from orange to green |