



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

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**CHEMISTRY**

**9701/31**

Paper 31 Advanced Practical Skills

**October/November 2008**

**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.

|                   |
|-------------------|
| <b>Session</b>    |
|                   |
| <b>Laboratory</b> |
|                   |

| <b>For Examiner's Use</b> |  |
|---------------------------|--|
| <b>1</b>                  |  |
| <b>2</b>                  |  |
| <b>3</b>                  |  |
| <b>Total</b>              |  |

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

- 1 **FA 1** contains the monoprotic (monobasic) acid  $\text{RCO}_2\text{H}$ . You are to determine the relative molecular mass,  $M_r$ , of the acid and deduce its molecular formula.

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You are provided with the following.

**FA 1**, the aqueous acid, containing  $38.68 \text{ g dm}^{-3} \text{ RCO}_2\text{H}$

**FA 2**, aqueous sodium hydroxide containing  $3.40 \text{ g dm}^{-3} \text{ NaOH}$

Phenolphthalein indicator.

**(a) Dilution of FA 1**

By using a burette measure between  $38.00 \text{ cm}^3$  and  $39.00 \text{ cm}^3$  of **FA 1** into the  $250 \text{ cm}^3$  graduated (volumetric) flask labelled **FA 3**.

Record your burette readings and the volume of **FA 1** added to the flask in the space below.

Make up the contents of the flask to the  $250 \text{ cm}^3$  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 **FA 3**, the diluted solution containing  $\text{RCO}_2\text{H}$ .

Pipette  $25.0 \text{ cm}^3$  of **FA 2** into the conical flask and add 2–3 drops of phenolphthalein indicator.

Titrate the sodium hydroxide in the flask with **FA 3** until the solution just turns colourless.

**Perform a rough (trial) titration and sufficient further titrations to obtain accurate 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]

- (b) From your titration results obtain a volume of **FA 3** to be used in your calculations. Show clearly how you obtained this volume.

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[1]

### Calculations

Show your working and appropriate significant figures in all of your calculations.

- (c) Calculate how many moles of NaOH have been pipetted into the conical flask.  
[ $A_r$ : H, 1.0; O, 16.0; Na, 23.0]

..... mol of NaOH were pipetted into the conical flask.

Use your titre volume in (b) and the answer above to calculate how many moles of  $\text{RCO}_2\text{H}$  are contained in  $250\text{ cm}^3$  of the diluted acid **FA 3**.

$250\text{ cm}^3$  of **FA 3** contains ..... mol of  $\text{RCO}_2\text{H}$ .

Use this answer to calculate the concentration, in  $\text{mol dm}^{-3}$ , of the undiluted acid in **FA 1**.

The concentration of  $\text{RCO}_2\text{H}$  in **FA 1** is .....  $\text{mol dm}^{-3}$ .

Use this answer to calculate, correct to **3 significant figures**, the relative molecular mass,  $M_r$ , of  $\text{RCO}_2\text{H}$ .

The relative molecular mass,  $M_r$ , of  $\text{RCO}_2\text{H}$  is .....

Use this answer to deduce the formula of the acid  $\text{RCO}_2\text{H}$ .

The formula of  $\text{RCO}_2\text{H}$  is .....

[5]

[Total: 12]

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

- 2 You are required to find the percentage by mass of water of crystallisation in **FA 4**, hydrated magnesium sulphate,  $\text{MgSO}_4 \cdot x\text{H}_2\text{O}$ .

The water contained in the crystals can be removed by heating the crystals.

### Method

- (a) Follow the instructions below to determine the mass of water driven off when heating magnesium sulphate crystals.

- Weigh the empty hard glass boiling-tube.
- Tip the contents of the tube labelled **FA 4** into the boiling-tube.
- Reweigh the boiling-tube and **FA 4**.
- Hold the boiling-tube in the holder provided and heat gently at first, then strongly for several minutes.
- Leave the boiling-tube to cool on a heat-proof mat.
- **Carry on with other parts of the paper, e.g. question 3, while the boiling-tube cools.**
- When cool weigh the boiling-tube and its contents.
- Continue the heating, cooling and weighing until you are satisfied that all of the water of crystallisation has been driven from the crystals.

In an appropriate form record below

- all measurements of mass,
- the final mass of the residue and the mass of water driven off.

### Results

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

[7]

**Calculations**

- (b) Calculate the percentage by mass of water of crystallisation in the crystals.

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Hydrated magnesium sulphate crystals contain ..... % of water by mass.  
[1]

- (c) Use the relative molecular mass information provided to complete the table below.  
[ $M_r$ :  $\text{MgSO}_4$ , 120.4;  $\text{H}_2\text{O}$ , 18.0]

| possible value of $x$ in $\text{MgSO}_4 \cdot x\text{H}_2\text{O}$ | % water by mass |
|--|-----------------|
| 1  | 13.0            |
| 2  | 23.0            |
| 3  |                 |
| 4  | 37.4            |
| 5  | 42.8            |
| 6  | 47.3            |
| 7  | 51.1            |
| 8  | 54.5            |
| 9  |                 |
| 10   | 59.9            |

Use your answer in (b) and the information in the table to find the experimental value of  $x$  in the formula  $\text{MgSO}_4 \cdot x\text{H}_2\text{O}$ .

..... is the value of  $x$  in  $\text{MgSO}_4 \cdot x\text{H}_2\text{O}$ .  
[2]

- (d) A student is instructed to repeat the whole experiment to ensure **reliability** of results. Explain how repeating the experiment would lead to greater **reliability** in the experimental results.

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

- (e) On a balance weighing to 1 decimal place assume the maximum error is  $\pm 0.1$  g. What mass would have to be weighed out on this balance to result in an error of 0.04%?

The mass is ..... g.  
[1]

[Total: 12]

- 3 **FA 5** is a solid metal oxide. **FA 6** and **FA 7** are aqueous solutions.

You will carry out specified tests to deduce

- the chemical properties of **FA 5** and **FA 7**,
- the identities of the anion and cation present in **FA 6**.

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) Pour 1 cm depth of **FA 7** into a boiling-tube, stand the tube in a test-tube rack and, using a spatula, add a **very small** amount of the solid **FA 5**.  
A vigorous reaction will be observed.

The gas evolved is one of carbon dioxide, hydrogen or oxygen.

By considering the density of each gas compared to that of air, you are to decide which of these three gases you should test for first. Explain your answer.

[ $A_r$ : C, 12.0; H, 1.0; O, 16.0]

[1 mol of any gas occupies approximately  $24 \text{ dm}^3$  at room temperature and pressure.]

[ $24 \text{ dm}^3$  of air at room temperature and pressure has a mass of approximately 25.6 g.]

.....

.....

.....

Add a further 1 cm depth of **FA 7** to the boiling-tube. Test and identify the gas given off. Record in a suitable form the test or tests performed and the observation made for any test carried out.

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Deduce the identity of the gas evolved.

The gas evolved is .....  
[4]

- (b)** Place 1 cm depth of 10% potassium iodide solution, KI, in a test-tube and add a **very small** amount of **FA 5**. Observe, then add a few drops of starch solution.

Record the tests used and all of your observations in an appropriate form below.

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

[2]

**(c) Conclusions about the chemical properties of FA 5**

In test **(a)** **FA 5** remained unchanged at the end of the reaction.

**FA 5** was acting as ..... in this reaction.

In test **(b)** **FA 5** was acting as .....

Where in the Periodic Table might you find the metallic element contained in **FA 5**?

.....  
[2]

- (d)** To 1 cm depth of **FA 6** in a boiling-tube add an equal volume of **FA 7**. Mix thoroughly by gently shaking the tube.  
Using aqueous sodium hydroxide and aqueous ammonia you are to identify the cation present in **FA 6**, and the cation present in the mixture of **FA 6** and **FA 7**.

Record all of your observations in the table below.

| <i>test</i>   | <i>observations</i> |  |
|---|---------------------|--|
|   | <b>FA 6</b>         | mixture of <b>FA 6</b> and <b>FA 7</b> |
| To 1 cm depth of solution in a test-tube add, drop by drop, 1 cm depth of aqueous sodium hydroxide.<br><br>Stir the mixture, then add a further 1 cm depth of aqueous sodium hydroxide. |                     |  |
| To 1 cm depth of solution in a test-tube add, drop by drop, 1 cm depth of aqueous ammonia.<br><br>Stir the mixture, then add a further 1 cm depth of aqueous ammonia.                   |                     |  |

**Conclusions**

The cation present in **FA 6** is .....

The cation present in the mixture of **FA 6** and **FA 7** is .....

When **FA 6** and **FA 7** reacted together, **FA 7** was acting as .....

[5]

|            |  |
|------------|--|
| <b>i</b>   |  |
| <b>ii</b>  |  |
| <b>iii</b> |  |
| <b>iv</b>  |  |
| <b>v</b>   |  |

- (e) Solution **FA 6** was prepared using a bottle of solid whose label was partly missing. The solid is believed to contain the sulphate ion,  $\text{SO}_4^{2-}$ , but may contain the sulphite ion,  $\text{SO}_3^{2-}$ .

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Use*

By selecting appropriate reagents from those listed on page 12 of the qualitative analysis notes show that sulphate ions,  $\text{SO}_4^{2-}$ , are present.

Record your tests, observations and conclusions in an appropriate form below.

[3]

[Total: 16]



### Qualitative Analysis Notes

Key: [ppt. = precipitate]

#### 1 Reactions of aqueous cations

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

| <i>ion</i>                                | <i>reaction</i>  |
|---|--|
| carbonate,<br>$\text{CO}_3^{2-}$          | $\text{CO}_2$ liberated by dilute acids  |
| chromate(VI),<br>$\text{CrO}_4^{2-}$ (aq) | yellow solution turns orange with $\text{H}^+$ (aq);<br>gives yellow ppt. with $\text{Ba}^{2+}$ (aq);<br>gives bright yellow ppt. with $\text{Pb}^{2+}$ (aq)                               |
| chloride,<br>$\text{Cl}^-$ (aq)           | gives white ppt. with $\text{Ag}^+$ (aq) (soluble in $\text{NH}_3$ (aq));<br>gives white ppt. with $\text{Pb}^{2+}$ (aq)   |
| bromide,<br>$\text{Br}^-$ (aq)            | gives pale cream ppt. with $\text{Ag}^+$ (aq) (partially soluble in $\text{NH}_3$ (aq));<br>gives white ppt. with $\text{Pb}^{2+}$ (aq)  |
| iodide,<br>$\text{I}^-$ (aq)              | gives yellow ppt. with $\text{Ag}^+$ (aq) (insoluble in $\text{NH}_3$ (aq));<br>gives yellow ppt. with $\text{Pb}^{2+}$ (aq)   |
| nitrate,<br>$\text{NO}_3^-$ (aq)          | $\text{NH}_3$ liberated on heating with $\text{OH}^-$ (aq) and Al foil   |
| nitrite,<br>$\text{NO}_2^-$ (aq)          | $\text{NH}_3$ liberated on heating with $\text{OH}^-$ (aq) and Al foil;<br>$\text{NO}$ liberated by dilute acids<br>(colourless $\text{NO} \rightarrow$ (pale) brown $\text{NO}_2$ in air) |
| sulphate,<br>$\text{SO}_4^{2-}$ (aq)      | gives white ppt. with $\text{Ba}^{2+}$ (aq) or with $\text{Pb}^{2+}$ (aq) (insoluble in excess dilute strong acids)  |
| sulphite,<br>$\text{SO}_3^{2-}$ (aq)      | $\text{SO}_2$ liberated with dilute acids;<br>gives white ppt. with $\text{Ba}^{2+}$ (aq) (soluble in excess dilute strong acids)  |

## 3 Tests for gases

| <i>gas</i>                     | <i>test and test result</i>  |
|--------------------------------|--|
| ammonia, $\text{NH}_3$         | turns damp red litmus paper blue   |
| carbon dioxide, $\text{CO}_2$  | gives a white ppt. with limewater<br>(ppt. dissolves with excess $\text{CO}_2$ ) |
| chlorine, $\text{Cl}_2$        | bleaches damp litmus paper   |
| hydrogen, $\text{H}_2$         | 'pops' with a lighted splint   |
| oxygen, $\text{O}_2$           | relights a glowing splint  |
| sulphur dioxide, $\text{SO}_2$ | turns aqueous potassium dichromate(VI) (aq) from orange to green                 |

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