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## 9701/31

**May/June 2007**

**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 ON 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 10 and 11.

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>

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

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

- 1 You are required to find the percentage purity of a sample of sodium carbonate,  $\text{Na}_2\text{CO}_3$ .

**FA 1** contains  $4.50 \text{ g dm}^{-3}$  of the impure sodium carbonate.

**FA 2** is  $0.50 \text{ mol dm}^{-3}$  hydrochloric acid,  $\text{HCl}$ .

**(a) Dilution of FA 2**

By using a burette, measure between  $33.00 \text{ cm}^3$  and  $34.00 \text{ cm}^3$  of **FA 2** into the  $250 \text{ cm}^3$  graduated flask labelled **FA 3**.

Record your burette readings and the volume of **FA 2** 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 of hydrochloric acid.

Pipette  $25.0 \text{ cm}^3$  of **FA 1** into a conical flask. Add a few drops of methyl orange indicator and titrate with **FA 3**.

**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 suitable volume of **FA 3** to be used in your calculations.

Show clearly how you obtained this volume.

[1]

**Calculations**

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

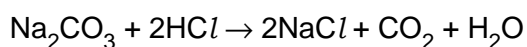
- (c) Calculate how many moles of  $\text{HCl}$  are contained in the **FA 2** run into the graduated flask.

.....mol of  $\text{HCl}$  were run into the graduated flask.

Calculate how many moles of  $\text{HCl}$  are contained in the volume of **FA 3** which reacted with  $25.0\text{ cm}^3$  of **FA 1**.

.....mol of  $\text{HCl}$  reacted with  $25.0\text{ cm}^3$  of **FA 1**.

Use this answer to calculate how many moles of sodium carbonate,  $\text{Na}_2\text{CO}_3$ , are present in  $1.00\text{ dm}^3$  of **FA 1**.



.....mol of  $\text{Na}_2\text{CO}_3$  are present in  $1.00\text{ dm}^3$  of **FA 1**.

Calculate the mass of sodium carbonate,  $\text{Na}_2\text{CO}_3$ , in  $1.00\text{ dm}^3$  of **FA 1**.  
[ $A_r$ : C, 12.0; O, 16.0; Na, 23.0]

**FA 1** is ..... $\text{g dm}^{-3}$  of  $\text{Na}_2\text{CO}_3$ .

Calculate, to **3 significant figures**, the percentage purity of the sodium carbonate,  $\text{Na}_2\text{CO}_3$ , dissolved in **FA 1**.

The percentage purity of the sodium carbonate dissolved in **FA 1** is .....%.

[5]

i	
ii	
iii	
iv	
v	

- (d) Look at the scale on the 25 cm<sup>3</sup> measuring cylinder provided. Record the smallest scale division on the measuring cylinder and estimate the error in reading the scale.

smallest division = ..... cm<sup>3</sup>

estimated error = ± ..... cm<sup>3</sup>

If 25 cm<sup>3</sup> of **FA 1** is measured with a measuring cylinder, calculate the estimated percentage error.

The estimated error is ..... %.

Your pipette is calibrated with an error of ±0.06 cm<sup>3</sup>.

Calculate the percentage error when measuring 25.0 cm<sup>3</sup> of solution with this pipette.

The error is ..... %.

[2]

- (e) Use the measuring cylinder to place  $25\text{ cm}^3$  of **FA 1** into a conical flask.  
Add methyl orange indicator as before and titrate with **FA 3**.  
Repeat the titration **once** using the measuring cylinder. Record your results below.

Would you expect to be able to obtain consistent titres using a measuring cylinder?  
Explain your answer.

.....

.....

.....

.....

.....[1]

- (f) A student suspects that the presence of dissolved carbon dioxide affects the end-point of the titration.  
Suggest a simple modification to the experimental technique to eliminate the dissolved carbon dioxide as the titration is performed.

.....

.....

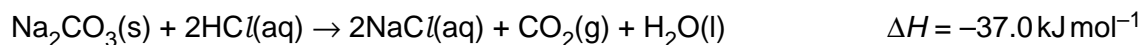
.....

.....[1]

[Total: 16]

- 2 Read through the question before starting any practical work.

The percentage purity of the sodium carbonate can also be determined by measuring the temperature change when a weighed sample of the solid carbonate reacts with an excess of hydrochloric acid and the following information is used.



You are provided with the following.

**FA 4**, impure solid sodium carbonate

**FA 5**,  $2.0 \text{ mol dm}^{-3}$  hydrochloric acid

### Measurement of temperature change

- (a) Follow the instructions below to determine the percentage purity of the sodium carbonate.

You will carry out the experiment twice.

- Weigh the empty weighing bottle.
- Reweigh the bottle with between 4.00 g and 4.50 g of **FA 4**.
- Support the plastic cup in the  $250 \text{ cm}^3$  beaker and add to it, from a measuring cylinder,  $50 \text{ cm}^3$  of **FA 5**.
- Measure and record the steady temperature of the **FA 5** in the plastic cup.
- Add the **FA 4** from the weighing bottle to the plastic cup, a little at a time to prevent acid spray. Stir and record the highest temperature reached.
- Reweigh the empty weighing bottle.

In an appropriate format in the space below, record

- all measurements of mass and temperature,
- the mass of **FA 4** used, **m**,
- the temperature rise,  $\Delta T$ .

Empty and rinse the plastic cup.

Repeat the experiment.

### Results

**Calculations**

- (b) For **each** experiment calculate  $\frac{\Delta T}{m}$ , the temperature rise per gram of **FA 4** used.

first experiment  $\frac{\Delta T}{m} = \dots\dots\dots ^\circ\text{C g}^{-1}$

second experiment  $\frac{\Delta T}{m} = \dots\dots\dots ^\circ\text{C g}^{-1}$

Calculate the mean value of  $\frac{\Delta T}{m}$ .

The mean value of  $\frac{\Delta T}{m} = \dots\dots\dots ^\circ\text{C g}^{-1}$   
[2]

- (c) Is one repeat of the experiment sufficient or should it be repeated again? Explain your answer.

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

- (d) Calculate the percentage purity of the sodium carbonate using the following expression.  
Note, this value is likely to be different from the one you obtained in question 1.

$$\text{purity} = \frac{\Delta T}{m} \times \frac{1}{37.0} \times 2279\%$$

purity = .....% [1]

[Total: 8]

- 3 **FA 6** and **FA 7** are solids **each** containing one cation and one anion from those listed on page 10 and page 11.

You will dissolve each solid in dilute nitric acid,  $\text{HNO}_3$ , and use the solutions formed in reactions with aqueous sodium hydroxide,  $\text{NaOH}$ , and aqueous ammonia,  $\text{NH}_3$ .

At each stage you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of any precipitate 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) Preparation of the solutions**

Dissolve each solid separately in dilute  $\text{HNO}_3$  in a  $100\text{ cm}^3$  beaker. Use the **minimum** quantity of acid, added a little at a time and warm to dissolve the solid if necessary. Then add distilled water to each solution to give a total volume of about  $60\text{ cm}^3$ .

Record your observations in the space below.

[4]

**(b) Reactions of the solutions formed in (a) with  $\text{NaOH(aq)}$  and  $\text{NH}_3(\text{aq})$**

Use separate portions of each of the solutions formed in **(a)** in reactions with aqueous  $\text{NaOH}$  and with aqueous  $\text{NH}_3$ , added until in excess.

For each test use 1 cm depth of solution in a boiling-tube.

Record details of the tests performed and the observations made.



[6]

(c) Both cations and one anion can now be definitely identified.

cation in **FA 6**.....

evidence .....

cation in **FA 7** .....

evidence .....

The anion in **FA** ..... is .....

evidence .....

[3]

(d) Tests to identify the remaining anion. Do not carry out these tests.

From the Quantitative Analysis Notes for anions select **two** reagents which could be used, in **one** test, to indicate the presence of chloride ions in one of the solutions.

Describe how you would use these reagents and the expected observations if the chloride ion were present.

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

Select another reagent that would also indicate the presence of a chloride ion in the solution. Describe the expected observation if the chloride ion were present.

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

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

## 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 (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 potassium dichromate(VI) (aq) from orange to green

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