

CANDIDATE
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CHEMISTRY

9701/33

Paper 3 Advanced Practical Skills 1

May/June 2017

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 an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

Answer **all** questions.
Electronic calculators may be used.
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 10 and 11.
A copy of the Periodic Table is printed on page 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.

- 1 Sodium hydrogencarbonate, NaHCO_3 , is used as baking soda in cooking. Baking soda may also contain small amounts of other chemicals. In this experiment, you will determine the percentage purity by mass of an impure sample of NaHCO_3 by titration with sulfuric acid.

FA 1 is $0.0500 \text{ mol dm}^{-3}$ sulfuric acid, H_2SO_4 .

FA 2 is impure NaHCO_3 .
methyl orange

(a) Method

Preparing a solution of FA 2

- Weigh the stoppered container of **FA 2**. Record the mass in the space below.
- Tip all the **FA 2** into the beaker.
- Reweigh the container with its stopper. Record the mass.
- Calculate and record the mass of **FA 2** used.
- Add approximately 100 cm^3 of distilled water to the **FA 2** in the beaker.
- Stir the mixture with a glass rod until all the **FA 2** has dissolved.
- Transfer this solution into the 250 cm^3 volumetric flask.
- Wash the beaker with distilled water and transfer the washings to the volumetric flask.
- Rinse the glass rod with distilled water and transfer the washings to the volumetric flask.
- Make up the solution in the volumetric flask to the mark using distilled water.
- Shake the flask thoroughly.
- This solution of impure NaHCO_3 is **FA 3**. Label the flask **FA 3**.

Results

Titration

- Fill the burette with **FA 1**.
- Pipette 25.0 cm^3 of **FA 3** into a conical flask.
- Add several drops of methyl orange.
- Perform a rough titration and record your burette readings in the space below.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make sure any recorded results show the precision of your practical work.
- Record in a suitable form below all of your burette readings and the volume of **FA 1** added in each accurate titration.

Keep **FA 1** for use in Question 2.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

[8]

- (b) From your accurate titration results, obtain a suitable value for the volume of **FA 1** to be used in your calculations.
Show clearly how you obtained this value.

25.0 cm³ of **FA 3** required cm³ of **FA 1**. [1]

(c) **Calculations**

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

- (i) Calculate the number of moles of sulfuric acid present in the volume of **FA 1** calculated in (b).

moles of H₂SO₄ = mol

- (ii) Balance the equation for the reaction of sulfuric acid and sodium hydrogencarbonate. State symbols are not required.



- (iii) Using your answers to (i) and (ii), calculate the number of moles of sodium hydrogencarbonate used in each titration.

moles of NaHCO₃ = mol

- (iv) Using your answer to (iii), calculate the mass of sodium hydrogencarbonate present in the mass of **FA 2** used to prepare **FA 3**.

mass of NaHCO_3 = g

- (v) Calculate the percentage purity by mass of the impure sodium hydrogencarbonate sample, **FA 2**.

percentage purity by mass of impure NaHCO_3 , **FA 2** = %

- (vi) What did you assume about the impurities in **FA 2** when you calculated the percentage purity?

.....

- (vii) A volumetric flask was labelled $250.0 \pm 0.10 \text{ cm}^3$.

Calculate the maximum percentage error when using this volumetric flask.

maximum percentage error = %
 [7]

[Total: 16]

- 2 When baking soda is heated, carbon dioxide is produced. In this experiment you will investigate the reaction taking place when the sodium hydrogencarbonate in baking soda is thermally decomposed.

FA 4 is baking soda (impure NaHCO_3). Its composition is the same as that of **FA 2**.

(a) Method

Record all your readings in the space below.

- Weigh the crucible with its lid.
- Transfer all the **FA 4** from the container into the crucible.
- Weigh the crucible, lid and **FA 4**.
- Calculate and record the mass of **FA 4** used.
- Place the crucible and contents on a pipe-clay triangle.
- Heat gently, with the lid on, for approximately one minute.
- Heat strongly, with the lid off, for a further three minutes.
- Replace the lid and leave the crucible to cool for at least five minutes.

While the crucible is cooling you may wish to begin work on Question 3.

- When it is cool, weigh the crucible with its lid and contents.
- Heat strongly, with the lid off, for a further two minutes.
- Replace the lid and leave the crucible to cool for at least five minutes.
- When it is cool, weigh the crucible with its lid and contents.
- Calculate and record the mass of residue obtained.
- This residue is **FA 5**. Keep this for use in **2(d)**.

Results

I	
II	
III	
IV	

[4]

(b) Calculations

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

- (i) Use the percentage purity by mass of **FA 2** you calculated in **1(c)(v)**, to calculate the mass of sodium hydrogencarbonate in the sample of **FA 4** that you weighed out.

(If you were unable to carry out the calculation in **1(c)(v)**, assume that the percentage purity by mass of **FA 2** is 95.8%.)

mass of NaHCO₃ in **FA 4** weighed out = g

- (ii) Calculate the mass of impurity present in your sample of **FA 4**.

mass of impurity = g

- (iii) The impurity in **FA 4** does not decompose when it is heated. This means that the residue, **FA 5**, contains the mass of impurity calculated in (ii) together with the solid decomposition product of sodium hydrogencarbonate.

Calculate the mass of the solid decomposition product.

mass of solid decomposition product = g

- (iv) Use your answers to (i) and (iii) to calculate the mass of solid decomposition product that would be obtained if 84.0 g of **pure** sodium hydrogencarbonate were heated.

mass of solid decomposition product = g

- (v) A student carried out the experiment by heating to constant mass and calculated that heating 84.0 g of pure NaHCO₃ would produce 52.3 g of the solid decomposition product. The student then suggested the following equation for the thermal decomposition of sodium hydrogencarbonate.



Use data from the Periodic Table on page 12 to explain why the student's suggestion cannot be correct.

.....

[4]

(c) (i) Why was the lid put on while the crucible and its contents cooled?

.....

(ii) The experiment could be made more accurate by heating to constant mass or using a more accurate balance. Suggest a further improvement to make the experiment more accurate.

.....

[2]

(d) (i) Pour a 1 cm depth of sulfuric acid, **FA 1**, into a test-tube.
 Add some **FA 5** from the crucible to the acid in the test-tube.
 Record all your observations.

.....

(ii) Use your observation(s) in (i) to identify an anion present in **FA 5**.
 Explain your answer.

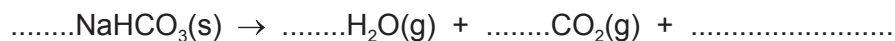
identity

explanation

.....

(iii) Steam is one of **three** products obtained when sodium hydrogencarbonate is thermally decomposed.

Use your answer in (ii) to complete and balance the equation for the thermal decomposition of sodium hydrogencarbonate. Include state symbols.



(iv) State whether the balanced equation in (iii) agrees with the student's results given in **2(b)(v)**.
 Show working in order to explain your answer.

.....

[4]

[Total: 14]

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 reagents are selected for use in a test, the **name** or **correct formula** of the element or compound must be given.

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.

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.

(a) (i) **FA 6** and **FA 7** are aqueous solutions.

Each solution contains one cation and one anion from those listed in the Qualitative Analysis Notes.

Use 1 cm depths of **FA 6** or **FA 7** in test-tubes for the following tests.

Complete the table by recording your observations.

<i>test</i>	<i>observations</i>	
	FA 6	FA 7
Add a few drops of aqueous barium chloride or aqueous barium nitrate, then add dilute nitric acid.		
Add a few drops of aqueous silver nitrate.		
Add a small spatula measure of sodium carbonate. Shake the mixture.		

(ii) From your observations, deduce which solution, **FA 6** or **FA 7**, has the lower pH. Give your evidence.

solution with lower pH

evidence

.....

[4]

(b) Choose **two** reagents that would allow you to identify the cations in **FA 6** and **FA 7**.

reagents and

Use these reagents to test solutions **FA 6** and **FA 7**.
Record all your observations in the space below.

[4]

(c) Deduce the chemical **formulae** of **FA 6** and **FA 7**.

FA 6

FA 7

[2]

[Total: 10]

Qualitative Analysis Notes

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)	faint white ppt. is nearly always observed unless 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	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
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

2 Reactions of anions

<i>ion</i>	<i>reaction</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids)

3 Tests for gases

<i>gas</i>	<i>test and test result</i>
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

