

CANDIDATE
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CHEMISTRY

9701/32

Paper 3 Advanced Practical Skills 2

May/June 2014

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.

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 **11** printed pages and **1** blank page.



- 1 Carbon dioxide in the atmosphere dissolves in rain water making it acidic. When this water passes through limestone containing calcium carbonate, it forms solutions of calcium hydrogen carbonate.

In this experiment, you will determine the concentration of hydrogen carbonate ions in a solution by titration with hydrochloric acid.

FB 1 is $0.100 \text{ mol dm}^{-3}$ hydrochloric acid, HCl .

FB 2 contains an unknown concentration of hydrogen carbonate ions, HCO_3^- .
methyl orange indicator

(a) Method

- Fill the burette with **FB 1**.
- Pipette 25.0 cm^3 of **FB 2** into a conical flask.
- Add a few drops of methyl orange indicator to the conical flask.
- 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 certain 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 **FB 1** 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 cm³ of **FB 2** required cm³ of **FB 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 hydrochloric acid present in the volume of **FB 1** calculated in (b).

moles of HCl = mol

- (ii) Write the **ionic** equation for the reaction of **FB 1** with **FB 2**.

.....

- (iii) Calculate the concentration, in mol dm⁻³, of hydrogen carbonate ions in **FB 2**.

I	
II	

concentration of HCO₃⁻ in **FB 2** = mol dm⁻³
[2]

[Total: 10]

- 2 As well as hydrogen carbonate ions, water that has passed through rocks also contains a range of metal ions such as Ca^{2+} . The presence of Ca^{2+} ions leads to the water forming scum with certain types of soap. Soluble metal carbonates can be added to remove these ions. One such soluble carbonate is $\text{M}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$.

In this experiment you will heat a sample of the carbonate salt to remove the water of crystallisation and leave the anhydrous salt, M_2CO_3 . By working out how much water was lost you will be able to calculate the relative formula mass of $\text{M}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ and hence identify **M**.

FB 3 is the hydrated salt, $\text{M}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$.

(a) Method

- Weigh a clean, dry crucible and its lid.
- Transfer all the **FB 3** into the crucible and put the crucible lid on.
- Reweigh the crucible, the lid and the contents.
- Place the crucible on the pipe-clay triangle.
- Heat the crucible very gently for 5 minutes.
- Remove the lid and heat strongly for a further 5 minutes.
- Allow the crucible to cool.
You should start **Question 3** while cooling is taking place.
- When the crucible is cool enough to handle, reweigh the crucible, the lid and the contents.

Record, in an appropriate form in the space below, all your weighings and calculations including the mass of **FB 3** used and the mass of water lost.

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 mass of water lost on heating to calculate the number of moles of hydrated salt, $\text{M}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$, in the initial sample.
[A_r: H, 1.0; O, 16.0]

moles of $\text{M}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ = mol

- (ii) Calculate the relative formula mass of $M_2CO_3 \cdot 10H_2O$, **FB 3**.

relative formula mass of $M_2CO_3 \cdot 10H_2O$ =

- (iii) Identify **M** in the salt, **FB 3**.

[A_r: H, 1.0; Li, 6.9; C, 12.0; O, 16.0; Na, 23.0; K, 39.1; Rb, 85.5; Cs, 133]

I	
II	
III	
IV	
V	

M is
[5]

- (c) (i) State the maximum error in the mass of **FB 3** you recorded in (a).

maximum error in the mass of **FB 3** = g

- (ii) Calculate the maximum percentage error in the mass of water lost in (a).

percentage error =%
[1]

- (d) Suggest a change to the method that would improve the accuracy of this experiment. Explain your answer.

.....

 [2]

[Total: 12]

I	
II	
III	

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 name or correct formula of the element or compound must be given.

- (a) Water in rivers and streams contains a range of different cations and anions. A particular salt occurs in some samples of spring water. You will carry out tests to identify this salt.

FB 4 is a solution of the salt. It contains one cation, which is listed in the Qualitative Analysis Notes on page 10. It also contains one anion, which is either sulfate, SO_4^{2-} , or sulfite, SO_3^{2-} .

- (i) Choose reagents that will allow you to identify the cation in **FB 4**. Carry out suitable tests using these reagents and record your observations in the space below.

The cation in **FB 4** is

- (ii) Choose reagents to identify the anion in **FB 4**. Carry out suitable tests using these reagents and record your results in the space below.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

The anion in **FB 4** is

[8]

Before starting parts (b) and (c), half-fill a 250 cm³ beaker with water and heat with a Bunsen burner to approximately 60 °C. You will use this as a hot water bath.
Turn off the Bunsen burner.

- (b) **FB 5, FB 6** and **FB 7** are solutions each containing a single compound which could be ethanol, ethanal or propanone. To identify each compound you will react the samples with Tollens' reagent and with acidified potassium manganate(VII).

(i) **Preparation of Tollens' reagent**

- To approximately 2 cm depth of aqueous silver nitrate in a boiling tube, add approximately 0.5 cm depth of aqueous sodium hydroxide.
- Add aqueous ammonia a little at a time with continuous shaking until the brown precipitate **just** dissolves. Do not add an excess of ammonia.

test	observations		
	FB 5	FB 6	FB 7
To a 1 cm depth of each solution in a clean, dry test-tube add a few drops of the Tollens' reagent that you have prepared. Do not shake the tube. If no reaction is seen, warm the tube in the hot water bath.			
When you have made your observations, rinse out all the test-tubes and the boiling tube which were used with Tollens' reagent.			
To a 1 cm depth of each solution in a test-tube add a 1 cm depth of dilute sulfuric acid. Then add a few drops of aqueous potassium manganate(VII). If no reaction is seen, warm the tube in the hot water bath.	Do not carry out this test		

- (ii) Identify each compound.

FB contains ethanol.

FB contains ethanal.

FB contains propanone.

I	
II	
III	
IV	
V	

- (iii) Another reagent can be used with these samples to identify which compounds have a carbonyl group, C=O. This reagent has not been supplied so you cannot carry out this test. State the reagent and the expected observations of the three tests.

reagent:

expected observations

FB 5

FB 6

FB 7

[7]

- (c) **FB 8** is an aqueous solution of an organic compound. Carry out the following tests. You do not need to identify **FB 8**.

<i>test</i>	<i>observations</i>
To a 1 cm depth of FB 8 in a test-tube add a 1 cm depth of dilute sulfuric acid. Then add a few drops of aqueous potassium manganate(VII). If no reaction is seen, place the test-tube in the hot water bath and leave to stand.	
To a 1 cm depth of FB 8 in a test-tube carefully add a small spatula measure of sodium hydrogen carbonate.	

[3]

[Total: 18]

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
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, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; 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
sulfur dioxide, SO_2	turns acidified aqueous potassium manganate(VII) from purple to colourless

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