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CHEMISTRY

9701/31

Paper 3 Advanced Practical Skills 1

October/November 2016

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 In **Questions 1** and **2** you will determine the percentage purity of industrial grade calcium carbonate, CaCO_3 , by two different methods.

In the first method you will collect and measure the volume of gas given off in the reaction between a known mass of industrial grade calcium carbonate, in the form of small marble chips, and a known amount of dilute hydrochloric acid. The acid will be in excess. The impurities in the calcium carbonate will not react with the acid.



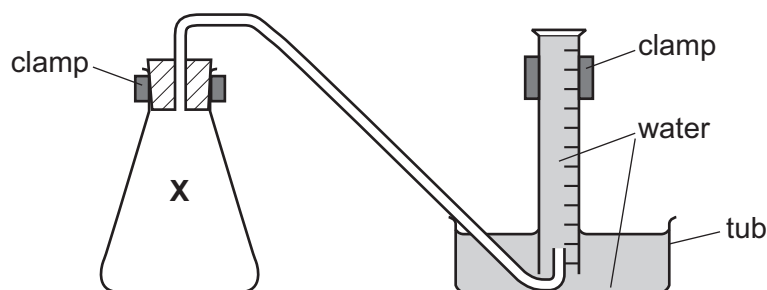
FA 1 is industrial grade calcium carbonate, CaCO_3 , in the form of small marble chips.

FA 2 is 2.00 mol dm^{-3} hydrochloric acid, HCl .

(a) Method

Read through the whole method before starting any practical work.

The diagram below may help you in setting up your apparatus.



- Fill the tub with water to a depth of about 5 cm.
- Fill the 250 cm^3 measuring cylinder **completely** with water. Hold a piece of paper towel firmly over the top, invert the measuring cylinder and place it in the water in the tub.
- Remove the paper towel and clamp the inverted measuring cylinder so the open end is in the water just above the base of the tub.
- Pipette 25.0 cm^3 of **FA 2** into the reaction flask labelled **X**.
- Check that the bung fits tightly in the neck of flask **X**, clamp flask **X** and place the end of the delivery tube into the inverted 250 cm^3 measuring cylinder.
- Weigh the container with **FA 1** and record the mass in the space on page 3.
- Remove the bung from the neck of the flask. Tip **FA 1** into the acid and replace the bung **immediately**. Remove the flask from the clamp and swirl it to mix the contents. Swirl the flask occasionally until no more gas is evolved. Replace the flask in the clamp.
- Reweigh the container and any residue of **FA 1** and record the mass in the space on page 3.
- Calculate and record in the space on page 3 the mass of **FA 1** used.
- When no more gas is given off, measure and record the final volume of gas in the measuring cylinder in the space on page 3.

Keep the contents of flask X for use in Question 2.

Results

[2]

(b) 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 carbon dioxide gas collected in the measuring cylinder. (Assume that 1 mole of gas occupies 24.0 dm³ under these conditions.)

moles of CO₂ = mol

- (ii) Use your answer to (i) and the Periodic Table on page 12 to calculate the mass of pure calcium carbonate in the sample of industrial grade calcium carbonate, **FA 1**.

mass of CaCO₃ = g

- (iii) Use your answer to (ii) and the mass of marble chips used in (a) to calculate a value for the percentage purity of the sample of industrial grade calcium carbonate, **FA 1**.

percentage purity of **FA 1** = %
[4]

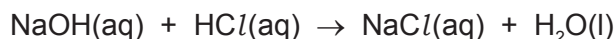
- (c) Not all the carbon dioxide given off in the reaction is collected in the measuring cylinder.

Suggest a change to the method which would lead to an increase in the volume of carbon dioxide collected.

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

[Total: 7]

- 2 You will determine the amount of hydrochloric acid remaining in flask **X** after the reaction with the marble chips in **Question 1**. You will do this by titration with sodium hydroxide of known concentration.



The impurities in the calcium carbonate will not react with the alkali.

FA 3 is $0.140 \text{ mol dm}^{-3}$ sodium hydroxide, NaOH.
bromophenol blue indicator

(a) Method

- Transfer **all** the contents of flask **X** into the 250 cm^3 volumetric flask.
- Rinse flask **X** with distilled water and add the washings to the volumetric flask. Add distilled water up to the mark.
- Stopper the volumetric flask and mix the contents thoroughly. Label this solution **FA 4**.
- Rinse the pipette then use it to transfer 25.0 cm^3 of **FA 4** into a conical flask.
- Add about 10 drops of bromophenol blue indicator.
- Fill the burette with **FA 3**.
- 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.
- Record, in a suitable form below, all of your burette readings and the volume of **FA 3** added in each accurate titration.
- Make certain any recorded results show the precision of your practical work.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

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

25.0 cm^3 of **FA 4** required cm^3 of **FA 3**. [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 sodium hydroxide, NaOH, present in the volume of **FA 3** you calculated in **(b)**.

moles of NaOH = mol

- (ii)** Use your answer to **(i)** and the equation on page 4 to determine the number of moles of hydrochloric acid, HCl, present in the 25.0 cm³ of **FA 4** pipetted in **(a)**.

moles of HCl = mol

- (iii)** Use your answer to **(ii)** to calculate the number of moles of hydrochloric acid, HCl, remaining in flask **X** after the reaction in **1(a)**.

moles of HCl remaining = mol

- (iv)** Use the relevant information on page 2 to calculate the number of moles of hydrochloric acid, HCl, pipetted into flask **X** in **1(a)**.

moles of HCl pipetted into flask **X** = mol

- (v)** Use your answers to **(iii)** and **(iv)** to calculate the number of moles of hydrochloric acid, HCl, which reacted with the marble chips in flask **X**.

moles of HCl which reacted in flask **X** = mol

- (vi) Use your answer to (v), the equation in **Question 1** and the Periodic Table on page 12 to calculate the mass of pure calcium carbonate, CaCO_3 , in the sample of industrial grade calcium carbonate, **FA 1**.

mass of CaCO_3 = g

- (vii) Use your answer to (vi) and the mass of marble chips recorded in **1(a)** to calculate the percentage purity of **FA 1**.

percentage purity of **FA 1** = %
[5]

- (d) You have carried out two different methods to find the percentage purity of industrial grade calcium carbonate.

A source of error in **Question 1** is that some carbon dioxide escapes before the bung can be inserted.

How would this affect the percentage purity of **FA 1** calculated in the two questions? Explain your answers.

Question 1

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

Question 2

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

[3]

[Total: 16]

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.

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) **FA 5** and **FA 6** are solids each containing one cation and one anion.
Carry out the following tests and record your observations in the table below.

<i>test</i>	<i>observations</i>	
	FA 5	FA 6
(i) Place a spatula measure of solid in a hard-glass test-tube and heat gently at first, then		
heat strongly until no further change takes place.		
Leave the tube to cool completely then add a 2 cm depth of dilute sulfuric acid to the solid residue. Shake the contents of the tube then leave it to stand.		

test	observations	
	FA 5	FA 6
(ii) Place a spatula measure of solid in a boiling tube and add a 2 cm depth of dilute sulfuric acid.		
Keep the solutions formed in (ii) for tests (iii) and (iv).		
(iii) To a 1 cm depth of solution from (ii) in a test-tube, add aqueous sodium hydroxide.		
(iv) To a 1 cm depth of solution from (ii) in a test-tube, add aqueous ammonia.		

- (v) Identify as many ions as you can from your observations. Write 'unknown' where you have not been able to identify an ion.

FA 5: cation anion

FA 6: cation anion

- (vi) Write an equation, including state symbols, for the reaction between **FA 6** and dilute sulfuric acid.

.....
[12]

(b) **FA 7** is a solution containing one anion from those listed on page 11. The anion is either a halide or contains nitrogen.

(i) You are to select suitable reagents to determine the identity of this anion. Record these in a suitable form below.

(ii) Use these reagents to carry out tests to identify the anion in **FA 7**.

Record your observations and conclusions in the space below.

[5]

[Total: 17]

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	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

The Periodic Table of Elements

		Group																																	
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18																		
		<div style="display: flex; justify-content: space-between; align-items: center;"> <div style="border: 1px solid black; padding: 2px;">1 H hydrogen 1.0</div> <div style="border: 1px solid black; padding: 2px;"> Key atomic number atomic symbol name relative atomic mass </div> </div>																																	
3 Li lithium 6.9	4 Be beryllium 9.0	11 Na sodium 23.0	12 Mg magnesium 24.3	19 K potassium 39.1	20 Ca calcium 40.1	21 Sc scandium 45.0	22 Ti titanium 47.9	23 V vanadium 50.9	24 Cr chromium 52.0	25 Mn manganese 54.9	26 Fe iron 55.8	27 Co cobalt 58.9	28 Ni nickel 58.7	29 Cu copper 63.5	30 Zn zinc 65.4	31 Ga gallium 69.7	32 Ge germanium 72.6	33 As arsenic 74.9	34 Se selenium 79.0	35 Br bromine 79.9	36 Kr krypton 83.8														
37 Rb rubidium 85.5	38 Sr strontium 87.6	39 Y yttrium 88.9	40 Zr zirconium 91.2	41 Nb niobium 92.9	42 Mo molybdenum 95.9	43 Tc technetium —	44 Ru ruthenium 101.1	45 Rh rhodium 102.9	46 Pd palladium 106.4	47 Ag silver 107.9	48 Cd cadmium 112.4	49 In indium 114.8	50 Sn tin 118.7	51 Sb antimony 121.8	52 Te tellurium 127.6	53 I iodine 126.9	54 Xe xenon 131.3	55 Cs caesium 132.9	56 Ba barium 137.3	57–71 lanthanoids	72 Hf hafnium 178.5	73 Ta tantalum 180.9	74 W tungsten 183.8	75 Re rhenium 186.2	76 Os osmium 190.2	77 Ir iridium 192.2	78 Pt platinum 195.1	79 Au gold 197.0	80 Hg mercury 200.6	81 Tl thallium 204.4	82 Pb lead 207.2	83 Bi bismuth 209.0	84 Po polonium —	85 At astatine —	86 Rn radon —
87 Fr francium —	88 Ra radium —	89–103 actinoids	104 Rf rutherfordium —	105 Db dubnium —	106 Sg seaborgium —	107 Bh bohrium —	108 Hs hassium —	109 Mt meitnerium —	110 Ds darmstadtium —	111 Rg roentgenium —	112 Cn copernicium —	113 Nh nihonium —	114 Fl flerovium —	115 Mc moscovium —	116 Lv livermorium —	117 Ts tennessine —	118 Og oganeson —																		

lanthanoids

actinoids

57 La lanthanum 138.9	58 Ce cerium 140.1	59 Pr praseodymium 140.9	60 Nd neodymium 144.4	61 Pm promethium —	62 Sm samarium 150.4	63 Eu europium 152.0	64 Gd gadolinium 157.3	65 Tb terbium 158.9	66 Dy dysprosium 162.5	67 Ho holmium 164.9	68 Er erbium 167.3	69 Tm thulium 168.9	70 Yb ytterbium 173.1	71 Lu lutetium 175.0
89 Ac actinium —	90 Th thorium 232.0	91 Pa protactinium 231.0	92 U uranium 238.0	93 Np neptunium —	94 Pu plutonium —	95 Am americium —	96 Cm curium —	97 Bk berkelium —	98 Cf californium —	99 Es einsteinium —	100 Fm fermium —	101 Md mendelevium —	102 No nobelium —	103 Lr lawrencium —