WORKSHEET NO. 1

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

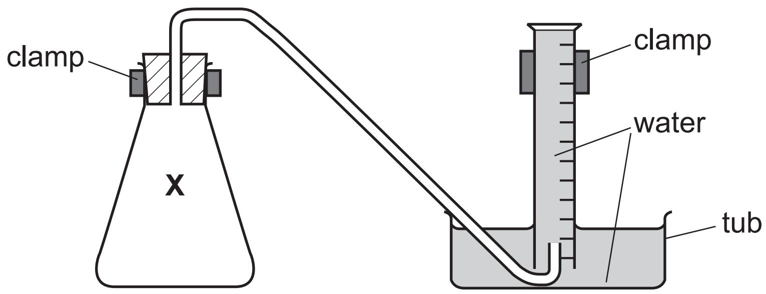
In the first method you will collect and measure the volume of gas given off in the reaction between a known mass of industrial grade calcium carbonate, in the form of small marble chips, and a known amount of dilute hydrochloric acid. The acid will be in excess. The impurities in the calcium carbonate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

$$CaCO_3(s) + 2HCl(aq) \rightarrow CaCl_2(aq) + H_2O(l) + CO_2(g)$$

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 2.
- 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 2.
- Calculate and record in the space on page 2 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 2.

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

Results

mass of tube + FA1 / g	21.82
mass of tube + residue / g	20.92
mass of FA1 used / g	0.90
Volume of gas collected / cm^3	198

[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 dm^3 under these conditions.)

1 mole —— 24.0 dm^3

x —— $\frac{198}{1000}$ dm^3

moles of CO_2 = 8.25×10^{-3} mol

(ii) Use your answer to **(i)** and the Periodic Table on page 11 to calculate the mass of pure calcium carbonate in the sample of industrial grade calcium carbonate, **FA 1**.

mole ratio

$CaCO_3$: CO_2

1 : 1

8.25×10^{-3} : x

8.25×10^{-3} mol

$n = \frac{m}{M_r}$

$8.25 \times 10^{-3} = \frac{m}{100.1}$

$m = 0.826$

mass of $CaCO_3$ = 0.826 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**.

$\frac{0.826}{0.90} \times 100$

percentage purity of **FA 1** = 91.8 %

[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.

Use a gas syringe to collect the gas as CO_2 is partially soluble in water.

[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.

$$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$$

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

FA 3 is 0.140 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.

Final burette reading /cm^3	25.40
Initial burette reading/cm^3	0.00
Volume of FA3 used /cm^3	25.40

The rough titre is 25.40 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.

	1	2	3	4
Final burette reading/cm^3	25.20	33.60	40.10	
Initial burette reading/cm^3	0.00	8.50	15.00	
Volume of FA3 used /cm^3	25.20	25.10	25.10	
Best titre		✓	✓	

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.

$$\frac{25.10 + 25.10}{2}$$

25.0 cm^3 of **FA 4** required 25.10 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)**.

$n = cV$

$= 0.140 \times \frac{25.10}{1000}$

moles of NaOH = 3.51×10^{-3} mol

(ii) Use your answer to **(i)** and the equation on page 3 to determine the number of moles of hydrochloric acid, HC*l*, present in the $25.0\,cm^3$ of **FA 4** pipetted in **(a)**.

mole ratio

HCl : NaOH

1 : 1

moles of HC*l* = 3.51×10^{-3} mol

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

$25\,cm^3$ — 3.51×10^{-3}

$250\,cm^3$ — x

moles of HC*l* remaining = 3.51×10^{-2} mol

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

$n = cV$

$= 2.00 \times \frac{25.0}{1000}$

moles of HC*l* pipetted into flask **X** = 0.0500 mol

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

$0.0500 - 3.51 \times 10^{-2}$

moles of HC*l* which reacted in flask **X** = 0.0149 mol

(vi) Use your answer to **(v)**, the equation in **Question 1** and the Periodic Table on page 11 to calculate the mass of pure calcium carbonate, $CaCO_3$, in the sample of industrial grade calcium carbonate, **FA 1**.

mole ratio
$CaCO_3 : HCl$
$1 : 2$
$x : 0.0149$
7.45×10^{-3} mol

$n = \frac{m}{M_r}$
$7.45 \times 10^{-3} = \frac{m}{100.1}$

mass of $CaCO_3$ = 0.746 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**.

$\frac{0.746}{0.90} \times 100$

percentage purity of **FA 1** = 82.9 %

[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

Percentage purity lower as loss of gas means fewer moles / less mass of $CaCO_3$

Question 2

No change / same percentage as same amount of acid reacts OR amount of acid left is same.

[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.

test	*observations*	
	FA 5	**FA 6**
(i) Place a spatula measure of solid in a hard-glass test-tube and heat gently at first, then	Solid melts	green solid turns black
heat strongly until no further change takes place.	Brown gas	a colorless gas which formed white ppt with Lime water.
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.	Solid dissolves form a colorless solution	Solid dissolves form a blue solution

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.	Solid dissolved, a colorless solution formed.	effervescence of gas and blue solution formed
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.	no change	blue ppt. insoluble in excess
(iv) To a 1 cm depth of solution from **(ii)** in a test-tube, add aqueous ammonia.	no change	blue ppt. soluble in excess to give a dark blue solution

(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 ....Unknown.... anion ....NO_3^-....

FA 6: cation ....Cu^{2+}.... anion ....CO_3^{2-}....

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

....$CuCO_{3(s)} + H_2SO_{4(aq)} \longrightarrow CuSO_{4(aq)} + H_2O_{(l)} + CO_{2(g)}$....

[12]

(b) **FA 7** is a solution containing one anion from those listed on page 10. 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.

Halides	add aq. $AgNO_3$ followed by aq. NH_3
NO_3^-	add aq. NaOH, Al-foil and heat
NO_2^-	add dilute HCl

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

Record your observations and conclusions in the space below.

tests	observations
a) To 1cm depth of solution add 1cm depth of aq. $AgNO_3$	Cream ppt. formed
add aq. NH_3 in excess	ppt. partially soluble in aq. NH_3
b) To 1cm depth of solution add 1cm depth of aq. NaOH, Al-foil and warm the mixture	Damp red Litmus paper remains red
c) To 1cm depth of solution add 1cm depth of dilute HCl	no change

Conclusion ⟶ FA7 contains Br^-

[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

ion	*reaction*
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, Cl^-(aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq))
bromide, Br^-(aq)	gives cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq))
iodide, I^-(aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(aq))
nitrate, NO_3^-(aq)	NH_3 liberated on heating with OH^-(aq) and Al foil
nitrite, NO_2^-(aq)	NH_3 liberated on heating with OH^-(aq) and Al foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO_2 in air)
sulfate, SO_4^{2-}(aq)	gives white ppt. with Ba^{2+}(aq) (insoluble in excess dilute strong acids)
sulfite, SO_3^{2-}(aq)	gives white ppt. with Ba^{2+}(aq) (soluble in excess dilute strong acids)

3 Tests for gases

gas	*test and test result*
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

Key

atomic number
atomic symbol
name
relative atomic mass

Group 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
							1 H hydrogen 1.0										2 He helium 4.0
3 Li lithium 6.9	4 Be beryllium 9.0											5 B boron 10.8	6 C carbon 12.0	7 N nitrogen 14.0	8 O oxygen 16.0	9 F fluorine 19.0	10 Ne neon 20.2
11 Na sodium 23.0	12 Mg magnesium 24.3											13 Al aluminium 27.0	14 Si silicon 28.1	15 P phosphorus 31.0	16 S sulfur 32.1	17 Cl chlorine 35.5	18 Ar argon 39.9
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 –		114 Fl flerovium –		116 Lv livermorium –		

lanthanoids	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
actinoids	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 –