

Quantitative Analysis

Read through the whole method before starting any practical work. Where appropriate, prepare a table for your results in the space provided.

Show the precision of the apparatus you used in the data you record.

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

- 1 Some metal carbonates occur in a basic form which means that the metal hydroxide is also present. The formula of one form of basic zinc carbonate is $\text{ZnCO}_3 \cdot 2\text{Zn(OH)}_2 \cdot x\text{H}_2\text{O}$, where **x** is an integer.

In this experiment you will carry out a thermal decomposition to find the relative formula mass, M_r , and the value of **x** for a sample of basic zinc carbonate.

FA 1 is basic zinc carbonate, $\text{ZnCO}_3 \cdot 2\text{Zn(OH)}_2 \cdot x\text{H}_2\text{O}$.

(a) Method

- Weigh the empty crucible with its lid. Record the mass.
- Transfer all the **FA 1** from the container into the crucible.
- Weigh the crucible, lid and **FA 1**. Record the mass.
- Place the crucible and contents on a pipe-clay triangle.
- Heat the crucible gently, with the lid on, for approximately 1 minute.
- Heat strongly, with the lid off, for a further 4 minutes.
- Replace the lid and leave the crucible to cool for at least 5 minutes.

While the crucible is cooling you may wish to begin work on Question 2 or 3.

- When the crucible is cool, weigh the crucible with its lid and contents. Record the mass.
- Place the crucible and contents on the pipe-clay triangle. Remove the lid.
- Heat the crucible strongly for a further 2 minutes.
- Replace the lid and leave the crucible to cool for at least 5 minutes.
- When the crucible is cool, reweigh the crucible with its lid and contents. Record the mass.
- Calculate and record the mass of **FA 1** used.
- Calculate and record the mass of residue obtained.

Results

I	
II	
III	
IV	



(b) Calculations

The equation for the thermal decomposition is shown.



- (i) Calculate the amount, in mol, of zinc oxide, ZnO, formed after heating.

amount of ZnO = mol

Hence, calculate the amount, in mol, of basic zinc carbonate in your sample of **FA 1**.

amount of $\text{ZnCO}_3 \cdot 2\text{Zn(OH)}_2 \cdot x\text{H}_2\text{O}$ = mol
[1]

- (ii) Use your answer to **(b)(i)** and your results in **(a)** to calculate the relative formula mass, M_r , of basic zinc carbonate, **FA 1**.

M_r of $\text{ZnCO}_3 \cdot 2\text{Zn(OH)}_2 \cdot x\text{H}_2\text{O}$ = [1]

- (iii) Use the Periodic Table to calculate the relative formula mass, M_r , of $\text{ZnCO}_3 \cdot 2\text{Zn(OH)}_2$.

M_r of $\text{ZnCO}_3 \cdot 2\text{Zn(OH)}_2$ = [1]

- (iv) Use your answers to **(b)(ii)** and **(b)(iii)** to determine the value of **x** in $\text{ZnCO}_3 \cdot 2\text{Zn(OH)}_2 \cdot x\text{H}_2\text{O}$.
Show your working.

x = [2]

- 2 In this experiment you will determine the relative formula mass, M_r , of basic zinc carbonate, $\text{ZnCO}_3 \cdot 2\text{Zn}(\text{OH})_2 \cdot x\text{H}_2\text{O}$, by an alternative method.

A known mass of basic zinc carbonate is reacted with a known volume and concentration of hydrochloric acid, HCl . The acid added is in excess. You will titrate portions of the resulting solution with sodium hydroxide, NaOH , of known concentration.

FA 2 has been prepared as follows:

3.52 g of basic zinc carbonate, $\text{ZnCO}_3 \cdot 2\text{Zn}(\text{OH})_2 \cdot x\text{H}_2\text{O}$, is reacted with 100 cm^3 of 2.00 mol dm^{-3} hydrochloric acid, HCl . The resulting solution is diluted to 1.00 dm^3 with distilled water.

FA 3 is 0.150 mol dm^{-3} sodium hydroxide, NaOH .

FA 4 is bromophenol blue indicator.

(a) Method

- Fill the burette with **FA 3**.
- Pipette 25.0 cm^3 of **FA 2** into a conical flask.
- Add several drops of **FA 4** 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 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 your burette readings and the volume of **FA 3** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b) From your accurate titration results, calculate a suitable mean value to use in your calculations. Show clearly how you obtain the mean value.

25.0 cm^3 of **FA 2** required cm^3 of **FA 3**. [1]

(c) Calculations

- (i) Give your answers to **(c)(ii)**, **(c)(iii)**, **(c)(iv)** and **(c)(vi)** to an appropriate number of significant figures. [1]

- (ii) Calculate the amount, in mol, of sodium hydroxide present in the volume of **FA 3** in **(b)**.

amount of NaOH = mol [1]

- (iii) Use your answer to **(c)(ii)** to calculate the amount, in mol, of hydrochloric acid present in 1.00 dm³ of **FA 2**.

amount of HCl in 1.00 dm³ of **FA 2** = mol [1]

- (iv) Use your answer to **(c)(iii)** and the information about the preparation of **FA 2** to calculate the amount, in mol, of hydrochloric acid that reacted with 3.52 g of basic zinc carbonate.

amount of HCl that reacted = mol [1]

- (v) Using the equation given in question **1(b)** as a guide, complete the equation for the reaction of hydrochloric acid with basic zinc carbonate. State symbols are **not** required.



- (vi) Use your answer to **(c)(iv)** and the equation in **(c)(v)** to calculate the amount, in mol, of basic zinc carbonate in 3.52 g.

amount of $\text{ZnCO}_3 \cdot 2\text{Zn}(\text{OH})_2 \cdot x\text{H}_2\text{O}$ = mol

Hence, calculate the relative formula mass, M_r , of basic zinc carbonate.

M_r of $\text{ZnCO}_3 \cdot 2\text{Zn}(\text{OH})_2 \cdot x\text{H}_2\text{O}$ = [2]



- (d) A student correctly followed all the instructions in (a) to (c) of this question. The formula mass the student calculated was **smaller** than the correct value. The student suggests that this may be because the concentration of sodium hydroxide, **FA 3**, is greater than $0.150 \text{ mol dm}^{-3}$.

Explain why the student is correct.

.....

.....

.....

..... [2]

[Total: 17]

BLANK PAGE

Qualitative Analysis

For each test you should record all your observations in the spaces provided.

Examples of observations include:

- colour changes seen
- the formation of any precipitate and its solubility (where appropriate) in an excess of the reagent added
- the formation of any gas and its identification (where appropriate) by a suitable test.

You should record clearly at what stage in a test an observation is made.

Where no change is observed you should write 'no change'.

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

If any solution is warmed, a boiling tube must be used.

Rinse and reuse test-tubes and boiling tubes where possible.

No additional tests should be attempted.

- 3 (a) **FA 5** contains one cation and one anion, both of which are listed in the Qualitative analysis notes. **FA 5** is insoluble in water.

Transfer approximately half of the sample of **FA 5** into a hard-glass test-tube. Heat the test-tube gently at first, then more strongly, until no further change occurs.
Record **all** your observations.

.....

.....

.....

..... [2]

- (b) **FA 6** is an aqueous solution containing one cation and one anion. The anion is listed in the Qualitative analysis notes. **FA 6** does **not** contain sulfur.

- (i) To a 2 cm depth of **FA 6** in a test-tube, add a piece of magnesium ribbon.
Record **all** your observations.

.....

.....

..... [2]

- (ii) Write an ionic equation for the reaction between **FA 6** and magnesium. Include state symbols.

..... [1]

- (c) (i) Add a small spatula measure of **FA 5** to a 2 cm depth of **FA 6** in a boiling tube. Record **all** your observations.

The product of this test is **FA 7**. Label the boiling tube **FA 7**.

.....

 [1]

- (ii) To a 1 cm depth of **FA 7** in a test-tube, add aqueous ammonia. Record **all** your observations.

.....
 [1]

- (d) (i) The same anion is present in both **FA 6** and **FA 7**. The identity of this anion can be confirmed by testing either **FA 6** or **FA 7** with a pair of reagents.

Select two pairs of reagents which may be used to identify the anion present in both **FA 6** and **FA 7**.

first pair of reagents:

..... and

second pair of reagents:

..... and

[2]

- (ii) Use **FA 6** to carry out each test using the two pairs of reagents you have selected. Record **all** your observations in a suitable form below.

[2]

- (e) Use your observations in (a) to (d) to identify the ions present in **FA 5** and **FA 6**. If you are unable to identify an ion write 'unknown'.

	cation	anion
FA 5		
FA 6		

Qualitative analysis notes

1 Reactions of cations

cation	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 warming	—
barium, Ba ²⁺ (aq)	faint white ppt. is observed unless [Ba ²⁺ (aq)] is very low	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. unless [Ca ²⁺ (aq)] is very low	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	pale 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

anion	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/off-white ppt. with Ag ⁺ (aq) (partially soluble in NH ₃ (aq))
iodide, I ⁻ (aq)	gives pale 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; decolourises acidified aqueous KMnO ₄
sulfate, SO ₄ ²⁻ (aq)	gives white ppt. with Ba ²⁺ (aq) (insoluble in excess dilute strong acids); gives white ppt. with high [Ca ²⁺ (aq)]
sulfite, SO ₃ ²⁻ (aq)	gives white ppt. with Ba ²⁺ (aq) (soluble in excess dilute strong acids); decolourises acidified aqueous KMnO ₄
thiosulfate, S ₂ O ₃ ²⁻ (aq)	gives off-white/pale yellow ppt. slowly with H ⁺

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
hydrogen, H_2	'pops' with a lighted splint
oxygen, O_2	relights a glowing splint

4 Tests for elements

element	test and test result
iodine, I_2	gives blue-black colour on addition of starch solution

Important values, constants and standards

molar gas constant	$R = 8.31 \text{ J K}^{-1} \text{ mol}^{-1}$
Faraday constant	$F = 9.65 \times 10^4 \text{ C mol}^{-1}$
Avogadro constant	$L = 6.022 \times 10^{23} \text{ mol}^{-1}$
electronic charge	$e = -1.60 \times 10^{-19} \text{ C}$
molar volume of gas	$V_m = 22.4 \text{ dm}^3 \text{ mol}^{-1}$ at s.t.p. (101 kPa and 273 K) $V_m = 24.0 \text{ dm}^3 \text{ mol}^{-1}$ at room conditions
ionic product of water	$K_w = 1.00 \times 10^{-14} \text{ mol}^2 \text{ dm}^{-6}$ (at 298 K (25 °C))
specific heat capacity of water	$c = 4.18 \text{ kJ kg}^{-1} \text{ K}^{-1}$ (4.18 $\text{J g}^{-1} \text{ K}^{-1}$)

The Periodic Table of Elements

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

lanthanoids

actinoids

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