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 answer to **each** step of your calculations.

1 Magnesium reacts with different acids, including organic acids, to form a salt and hydrogen. You will measure the volume of hydrogen formed when a known mass of magnesium reacts with excess carboxylic acid.

FA 1 is magnesium, Mg.
FA 2 is a carboxylic acid, RCH_2COOH. R does **not** contain an acidic hydrogen.

(a) **Method**

- Weigh the container with **FA 1**. Record the mass in the space for results.
- Fill the tub with water to a depth of approximately 5 cm.
- Fill the 250 cm^3 measuring cylinder completely with water. Holding 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.
- Use the 50 cm^3 measuring cylinder to transfer 50.0 cm^3 of **FA 2** into the flask labelled **X**. Check that the bung fits tightly into the neck of flask **X**, clamp flask **X** and place the end of the delivery tube into the inverted 250 cm^3 measuring cylinder.
- Remove the bung from the neck of the flask. Add **all** the **FA 1** to the acid in the flask and replace the bung **immediately**. Remove the flask from the clamp and swirl it to mix the contents.
- Replace the flask in the clamp and leave until the fizzing stops. Swirl the flask occasionally.
- Weigh the container with any residual **FA 1**. Record the mass.
- Calculate and record the mass of **FA 1** that reacts with the acid.
- When no more gas is collected, record the final volume of gas produced to the nearest cm^3.

You may wish to start Question 2 or Question 3 while the gas is being collected.

Keep FA 2 for use in Question 3.

Results

Mass of container with FA1/g	36.58
Mass of container with residual FA1/g	36.43
Mass of FA1 reacted/g	0.15

I	
II	
III	

[3]

(b) **Calculations**

(i) Calculate the amount, in mol, of magnesium added to flask **X**.

$n = \frac{0.159}{24.3\ g \cdot mol^{-1}} = 6.17 \times 10^{-3}$ mol

amount of Mg = 6.17 × 10⁻³ mol [1]

(ii) Write an ionic equation for the reaction between magnesium and the carboxylic acid. Include state symbols.

$Mg(s) + 2RCH_2COOH(aq) \rightarrow H_2(g) + Mg^{2+}(aq) + 2RCH_2COO^-(aq)$ [1]

(iii) Calculate the volume, in cm^3, of hydrogen expected to form in the reaction.

$n_{H_2} = 6.17 \times 10^{-3}$ mol

$V_{H_2} = 6.17 \times 10^{-3}\ mol \times 24.00 \times 10^3\ cm^3/mol$

$= 148\ cm^3$

volume of H_2 = 148 cm^3 [1]

(c) A student carries out the method in **1(a)**. The volume of hydrogen collected is smaller than expected.

Suggest **two** reasons why the student collected a smaller volume of hydrogen than expected.

reason 1 The produced hydrogen escapes.

reason 2 The Mg is not pure, with MgO on the surface. MgO reacts with acid to form water.

[2]

(d) State the uncertainty in a single balance reading for the balance you used in **1(a)**.

uncertainty = ± 0.01 g

Hence, calculate the maximum percentage error in the mass of magnesium, **FA 1**, used.

得3 ← $\frac{0.01 \times 2}{0.15} \times 100\% = 13.3\%$

2次

maximum percentage error = 13.3 % [1]

[Total: 9]

2 Carboxylic acids may have other atoms or groups substituted for hydrogen in the chain. R'CHZCOOH is the monosubstituted acid of $R'CH_2COOH$, where **Z** is a halogen. R' may be H or C_nH_{2n+1}.

You will carry out a titration using sodium hydroxide of known concentration to find the relative molecular mass, M_r, of the monosubstituted carboxylic acid, R'CHZCOOH.

$$R'CHZCOOH(aq) + NaOH(aq) \rightarrow R'CHZCOONa(aq) + H_2O(l)$$

The end-point is the appearance of the first permanent blue colour. Assume that the sodium hydroxide is too dilute for any significant hydrolysis of the halogen.

FA 3 is 0.0950 mol dm^{-3} sodium hydroxide, NaOH.
FA 4 is 15.28 g dm^{-3} monosubstituted carboxylic acid, R'CHZCOOH.
FA 5 is thymolphthalein indicator.

滴定剂为 0.100 mol·dm^{-3}

HCl

(a) **Method**

- Fill the burette with **FA 3**.
- Pipette 25.0 cm^3 of **FA 4** into a conical flask.
- Add a few drops of **FA 5** to the same conical flask.
- Perform a rough titration and record your burette readings.

Initial reading /cm³	0.15
Final reading /cm³	26.95
Titre	26.80

rough titre = 26.80 cm³

- 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 all your burette readings and the volume of **FA 3** added in each accurate titration.

Accurate titration	①	②
Initial reading /cm³	0.10	0.25
Final reading /cm³	26.40	26.55
Titre /cm³	26.30	26.30

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.

$\frac{26.30 + 26.30}{2} = 26.30$

25.0 cm^3 of **FA 4** required 26.30 cm^3 of **FA 3**. [1]

(c) **Calculations**

(i) Give your answers to **2(c)(ii)**, **2(c)(iii)** and **2(c)(iv)** to an appropriate number of significant figures. [1]

(ii) Calculate the amount, in mol, of sodium hydroxide in the volume of **FA 3** in **2(b)**.

$n = c \cdot V = 0.0950\ mol \cdot dm^{-3} \cdot 26.30 \times 10^{-3}\ dm^{3}$

$= 2.50 \times 10^{-3}\ mol$

amount of NaOH = 2.50×10^{-3} mol [1]

(iii) Calculate the concentration, in mol dm^{-3}, of the monosubstituted carboxylic acid in **FA 4**.

$n_{acid} = 2.50 \times 10^{-3}\ mol$.

$c = \frac{n}{V} = \frac{2.50 \times 10^{-3}\ mol}{2.50 \times 10^{-2}\ dm^{3}} = 0.100\ mol \cdot dm^{-3}$

concentration of R'CHZCOOH = 0.100 mol dm^{-3} [1]

(iv) Calculate the relative molecular mass, M_r, of the monosubstituted carboxylic acid in **FA 4**.
Show your working.

$M = \frac{15.289}{0.100\ mol} = 152.89\ g \cdot mol^{-1} \simeq 153\ g \cdot mol^{-1}$

M_r of R'CHZCOOH = 153 [1]

(d) (i) A fresh sample of **FA 4** reacts with excess hot aqueous sodium hydroxide. The resulting solution is neutralised with nitric acid. This solution is **FA 6**.
Carry out a test on **FA 6** to determine the identity of **Z**.

reagents Aqueous $AgNO_3$, Aqueous NH_3

observations Cream ppt. forms, partially soluble in aqueous NH_3

identity of **Z** Br

[2]

(ii) Suggest why nitric acid is added to make **FA 6** before carrying out your test in **2(d)(i)**.

To neutralise excess sodium hydroxide.

$2Ag^{+} + 2OH^{-} \rightarrow Ag_2O + H_2O$ Ag_2O is brown ppt. [1]

(e) Use your answers to **2(c)(iv)** and **2(d)(i)** to calculate the relative formula mass of group R'. Hence, identify group R' in **FA 4**.

$153 - 32 - 13 - 80 - 13$

$= 15$

relative formula mass of R' = 15

R' in **FA 4** CH_3

[2]

(f) Two students correctly carry out the method in **2(a)** using the same concentrations of **FA 3** and **FA 4** in separate experiments. The difference between the students' mean accurate titres is 0.35 cm^3.

Suggest why the students identify the same R' group.
(Do **not** attempt a calculation.)

The difference is insignificant compared with the total volume of FA4 required.

..... [1]

[Total: 18]

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. If any solid is heated, a hard-glass test-tube must be used.

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

No additional tests should be attempted.

3 Half-fill the 250 cm^3 beaker with water and place it on a tripod and gauze. Heat the water until boiling then switch off your Bunsen burner. This is your hot water-bath.

(a) Some organic acids contain an unsaturated hydrocarbon group.
Carry out an experiment using the reagents supplied to determine whether the R group in FA 2 is unsaturated.
Record your test, observations and conclusion.

test Add acidified $KMnO_4$ (aq) to FA2, then put the test tube into hot water-b

observations No change.

conclusion R group in FA2 is ~~is~~ saturated.

[2]

实际为 sulfuric acid.

(b) FA 7 and FA 8 are aqueous solutions of ionic compounds.
FA 7 contains one cation and two anions.
FA 8 contains one cation and one anion.
Three of the five ions are listed in the Qualitative analysis notes.

(i) Carry out the following tests and record your observations in Table 3.1.

Use a 1 cm depth of FA 7 or FA 8 in a test-tube for each test.

Table 3.1

Cr^{3+}, SO_4^{2-}, NO_3^- → observations ← OH^-

test	observations	
	FA 7	FA 8
Test 1 Add aqueous sodium hydroxide, then	Grey-green ppt., soluble in excess, giving a dark green solution	No change
pour the mixture into a boiling tube and heat gently, then	No change.	
add a piece of aluminium foil.	Fizzing. The produced gas turns damp red litmus paper blue.	
Test 2 Add aqueous ammonia.	Grey-green ppt., insoluble in excess.	No change
Test 3 Add aqueous copper(II) sulfate.	No change	Pale blue ppt. → $Cu(OH)_2$
Test 4 Add aqueous barium chloride or aqueous barium nitrate, then	White ppt.	faint white ppt. → $Ba(OH)_2$
add nitric acid.	No change	White ppt. dissolves, giving a colourless solution.
Test 5 Add aqueous silver nitrate.	No change.	Brown ppt. → Ag_2O
Test 6 Add FA 8 until in excess.	Grey-green ppt., soluble in excess, giving a dark green solution	

[7]

Cr^{3+} ← (Test 1)
NO_3^-/NO_2^- ← (add a piece of aluminium foil.)
Cr^{3+} ← (Test 2)
SO_4^{2-} ← (Test 4)
Cr^{3+} ← (Test 6)

(ii) Identify the cations in **FA 7** and **FA 8**. If you are unable to positively identify an ion or ions, write 'unknown'.

cation in **FA 7** Cr^{3+} cation in **FA 8** unknown [1]

(iii) Suggest the possible identities of the anions in **FA 7**. Give reasons for your answer.

SO_4^{2-}. It reacts with Ba^{2+} to form white ppt., which is insoluble in nitric acid.

/ NO_3^- or NO_2^-. Because it reacts with Al foil to form NH_3 under alkaline condition. [2]

(iv) Identify the anion in **FA 8**.

anion in **FA 8** OH^- [1]

[Total: 13]