

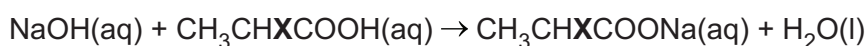
Quantitative analysis

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 You will investigate the enthalpy change of neutralisation, ΔH_{neut} , between aqueous sodium hydroxide of known concentration and a dilute organic acid. You will use your results to suggest the identity of the organic acid. The acid is a halogenocarboxylic acid containing one halogen atom, **X**, per molecule.



FB 1 is 1.90 mol dm^{-3} sodium hydroxide, NaOH.

FB 2 is a solution containing 312.5 g dm^{-3} of the organic acid $\text{CH}_3\text{CHXCOOH}$.

(a) Method

- Support the cup in the 250 cm^3 beaker.
- Pipette 25.0 cm^3 of **FB 1** into the cup.
- Place the thermometer into **FB 1**. Record the temperature of **FB 1** in Table 1.1. This is the temperature when the volume of **FB 2** is 0.00 cm^3 .
- Fill the burette with **FB 2**.
- Run 5.00 cm^3 of **FB 2** into the cup containing **FB 1**.
- Stir the mixture. Record the highest temperature observed.
- Run further 5.00 cm^3 portions of **FB 2** into the same cup.
- On each addition of **FB 2** stir the contents of the cup. Record the highest temperature after each addition.

I	
II	
III	

Results

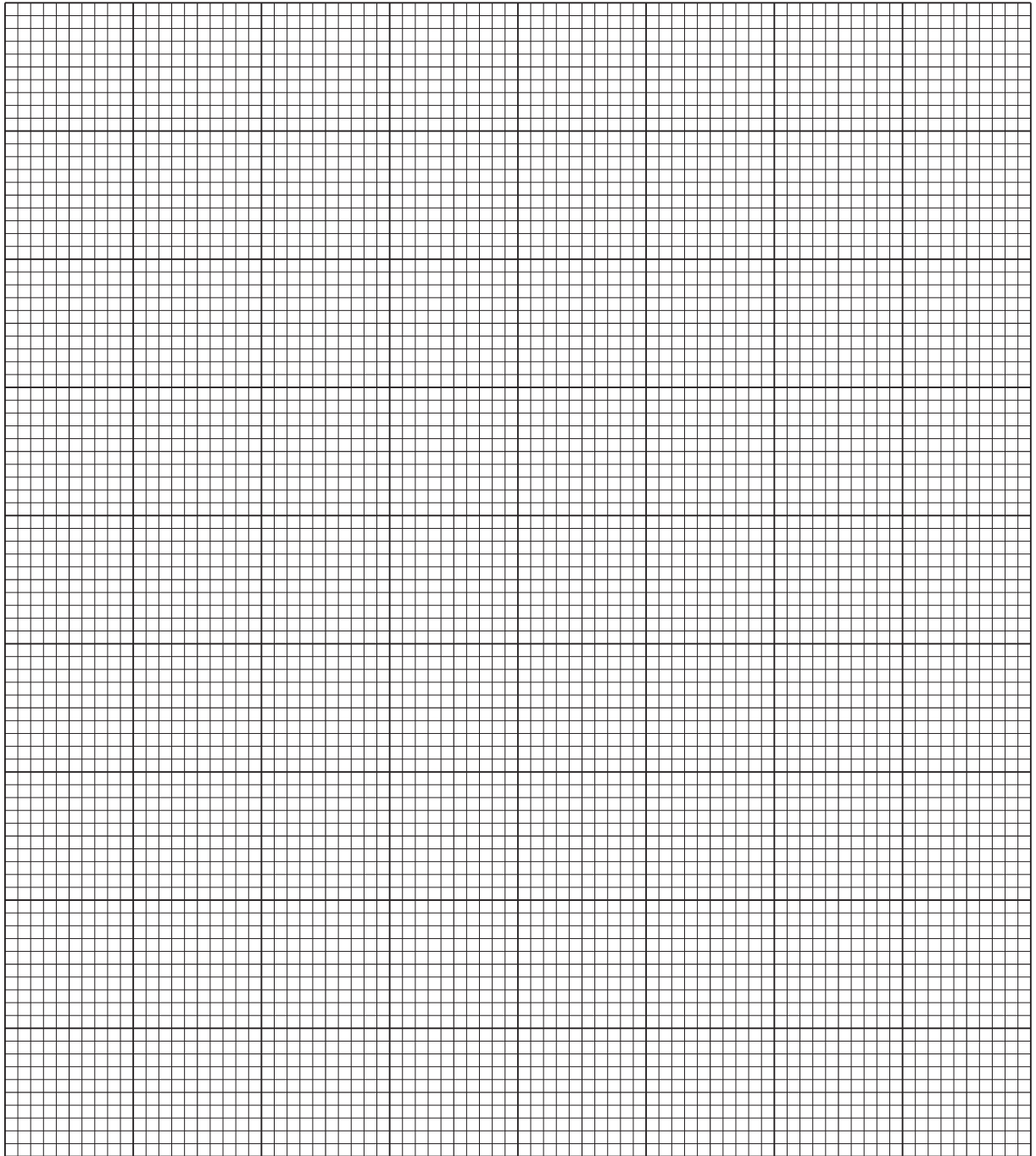
Table 1.1

total volume of FB 2 added / cm^3	0.00	5.00	10.00	15.00	20.00	25.00	30.00	35.00	40.00
temperature / $^{\circ}\text{C}$									

[3]

- (b) (i) Plot a graph of temperature (y-axis) against volume of **FB 2** added (x-axis) on the grid. Select a scale on the y-axis to include a temperature of 2°C above your maximum thermometer reading. Label any points you consider to be anomalous.

Draw two lines of best fit, the first for the increase in temperature and the second for after the maximum temperature has been reached. Extrapolate the two lines so they intersect. This intersection corresponds to the volume of **FB 2** required to form a neutral solution.



I	
II	
III	
IV	

[4]

(ii) Use your graph to determine the volume of **FB 2** required to neutralise 25.0 cm^3 of **FB 1**.

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

(c) Calculations

- (i) Calculate the energy change, in J, when the volume of **FB 2** recorded in **(b)(ii)** neutralises 25.0 cm³ of **FB 1**.

energy change = J [1]

- (ii) Calculate the amount, in mol, of sodium hydroxide, **FB 1**, pipetted into the cup.

amount of NaOH = mol [1]

- (iii) Calculate the enthalpy change of neutralisation, ΔH_{neut} , in kJ mol⁻¹, for 1.00 mol of sodium hydroxide reacting with **FB 2**.

$\Delta H_{\text{neut}} = \begin{matrix} \text{.....} \\ \text{sign} \end{matrix} \begin{matrix} \text{.....} \\ \text{value} \end{matrix} \text{ kJ mol}^{-1}$ [1]

- (iv) Use your answers to **(b)(ii)** and **(c)(ii)** and the information given on page 2 to calculate the relative formula mass, M_r , of the organic acid CH₃CHXCOOH. Show your working.

M_r of CH₃CHXCOOH = [1]

- (v) The acid is known to be one of the following: CH₃CHFCOOH, CH₃CHClCOOH, CH₃CHBrCOOH or CH₃CHICOOH. Use your answer to **(c)(iv)** to identify the acid used to make solution **FB 2**.

The acid in **FB 2** is [1]

- (vi) Calculate the percentage error in the relative formula mass, M_r , you calculated in **(c)(iv)**.

percentage error in M_r = % [1]

[Total: 14]

- 2 A gravimetric procedure can identify the metal in many metal carbonates. You will decompose a metal carbonate, MCO_3 , by heating to produce the metal oxide and carbon dioxide. You may assume this metal forms a stable metal oxide on heating. **M** is **not** a transition metal.



FB 3 is the metal carbonate MCO_3 .

(a) Method

- Weigh the crucible with its lid. Record the mass.
- Add between 0.90 g and 1.10 g of **FB 3** to the crucible.
- Weigh the crucible, lid and **FB 3**. Record the mass.
- Place the crucible on the pipe-clay triangle.
- Gently heat the crucible and contents for approximately 1 minute with the lid on.
- Remove the lid. Then heat the crucible and contents strongly for approximately 5 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 3.

- When the crucible is cool, weigh the crucible with its lid and contents. Record the mass.
- Calculate and record the mass of **FB 3** added to the crucible, the mass of residue obtained and the mass loss.

Keep the residue for use in 2(d).

Results

I	
II	
III	
IV	
V	

[5]

(b) Calculations

- (i) Calculate the amount, in mol, of carbon dioxide lost on heating **FB 3**.

amount of CO_2 = mol [1]

- (ii) Use your answer to **(b)(i)** and your data from **(a)** to calculate the relative formula mass, M_r , of MCO_3 .

M_r of MCO_3 = [1]

- (iii) Use your answer to **(b)(ii)** to suggest the identity of metal **M**.
Show your working.

M is [1]

- (c) A student carrying out the same experiment as in **(a)** spills a small quantity of solid just before carrying out the final weighing.
State what effect this would have on the value of the M_r that is calculated for MCO_3 .
Explain your answer.

.....

 [1]

- (d) Normally, in this experiment, you would reheat and reweigh the crucible and contents until the mass is constant to ensure all the metal carbonate has decomposed.
Suggest a **chemical** test to determine whether all the metal carbonate has decomposed.

Record your test, observation and conclusion.

test

observation

conclusion

[2]

[Total: 11]

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 Half fill the 250cm³ beaker with water and place it on a gauze on the tripod. Heat the water until boiling then switch off your Bunsen burner. This will be your hot water bath.

(a) **FB 4**, **FB 5** and **FB 6** are acids with the same concentration. Only one is an organic acid.

- (i) Carry out the following tests and record your observations in Table 3.1.
Use a 1 cm depth of **FB 4**, **FB 5** or **FB 6** in a test-tube for each test.

Table 3.1

<i>test</i>	<i>observations</i>		
	FB 4	FB 5	FB 6
Test 1 Add 1 or 2 drops of acidified aqueous potassium manganate(VII), then			
place the test-tube in the hot water bath.			
Test 2 Add a few copper turnings and place the test-tube in the hot water bath for a few minutes.			
Test 3 Add a 1 cm strip of magnesium ribbon, then			
leave the test-tube for 3 minutes and then shake the test-tube gently.			
At the end of the experiments rinse the test-tubes thoroughly.			

[7]

- (ii) Each of **FB 4**, **FB 5** and **FB 6** is one of the following acids: methanoic acid, nitric acid or sulfuric acid.

Use your observations to suggest the identity of each acid. Explain your answers.

FB 4 is

explanation

.....

FB 5 is

explanation

.....

FB 6 is

explanation

.....

[3]

- (b) The halogenocarboxylic acid in **FB 2** was hydrolysed by heating with excess aqueous sodium hydroxide. The resulting solution is **FB 7**.

- (i) Carry out a test to check the identity of the halogen atom present in **FB 2**.
Use a 1 cm depth of **FB 7** in a test-tube for your test.
State your reagents and record your observations at each stage of your test.

From this test **only**, give the identity of the halogen present in **FB 2**.

The halogen is

[3]

- (ii) Give the equation for the hydrolysis reaction of the halogenocarboxylic acid, **FB 2**, with excess hot aqueous sodium hydroxide.
(If you were unable to identify the halogen in (b)(i), then use the formula $\text{CH}_3\text{CHXCOOH}$.)

..... [2]

[Total: 15]

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													13	14	15	16	17	18			
														1 H hydrogen 1.0								2 He helium 4.0

lanthanoids

actinoids

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