

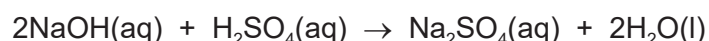
Quantitative analysis

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 determine the concentration of sulfuric acid by reaction with a known concentration of sodium hydroxide using a thermometric method. The equation for the reaction is shown.



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

FA 2 is dilute sulfuric acid, H_2SO_4 .

(a) Method

- Place the cup in the 250 cm^3 beaker.
- Use the 25 cm^3 measuring cylinder to transfer 25.0 cm^3 of **FA 1** into the cup.
- Place the thermometer into the solution in the cup and record its temperature in the table of results.
- Fill a burette with **FA 2**.
- Run 5.00 cm^3 of **FA 2** into the solution in the cup.
- Stir the mixture and record the highest temperature reached.
- Repeat adding 5.00 cm^3 volumes of **FA 2** into the solution in the cup until 45.00 cm^3 has been added. Record the highest temperature reached after each addition.

Results

volume of FA 2 added / cm^3	0.00	5.00	10.00	15.00	20.00
temperature of solution / $^{\circ}\text{C}$					

volume of FA 2 added / cm^3	25.00	30.00	35.00	40.00	45.00
temperature of solution / $^{\circ}\text{C}$					

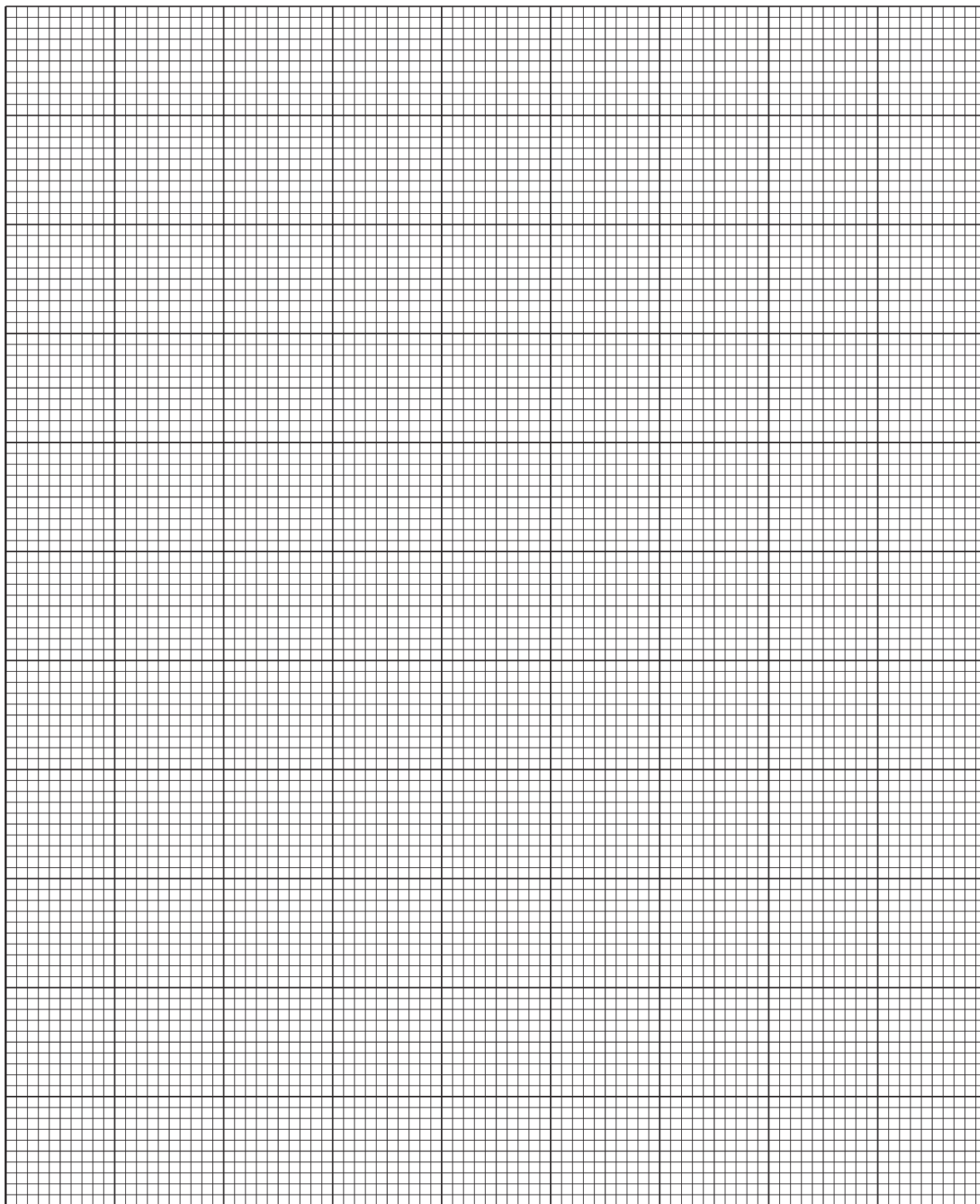
[3]

- (b) (i) Plot a graph of temperature (y-axis) against volume of acid added (x-axis) on the grid provided. Select a scale on the y-axis to include a temperature 4.0°C above the highest temperature you recorded.
Label any points you consider to be anomalous.

Draw two lines of best fit, one for the rise in temperature and one for after the maximum temperature has been reached.

Extrapolate the two lines so they intersect.

[4]



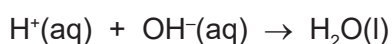
- (ii) Use your graph to determine the volume of sulfuric acid, **FA 2**, required to neutralise 25.0 cm³ of sodium hydroxide, **FA 1**.

volume of H₂SO₄ = cm³ [1]

- (iii) Calculate the concentration of sulfuric acid in **FA 2**.

concentration of $\text{H}_2\text{SO}_4 = \dots\dots\dots \text{mol dm}^{-3}$ [1]

- (c) A student carrying out the same procedure used the results from their graph to determine the enthalpy of neutralisation for the reaction.



- (i) State how the student used their graph to determine the value of ΔT for use in the equation $q = mc\Delta T$.

.....
 [1]

- (ii) The student correctly calculated the value of ΔH for the reaction as $\Delta H = -55.2 \text{ kJ mol}^{-1}$. The theoretical value for $\Delta H_{\text{neut}}^\ominus$ given in the student's textbook is $-57.6 \text{ kJ mol}^{-1}$.

Calculate the percentage error in the student's result compared with the theoretical value.

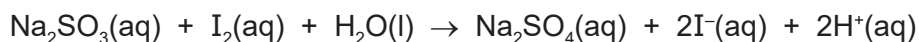
percentage error = % [1]

- (iii) Suggest why the student's result was less negative than the theoretical value. Explain your answer.

.....
 [1]

[Total: 12]

- 2 Solid sodium sulfite is often provided as the hydrated salt, $\text{Na}_2\text{SO}_3 \cdot x\text{H}_2\text{O}$, where x is an integer. You will determine x by using a solution of this sodium sulfite and reacting it with an excess of aqueous iodine.



The amount of iodine remaining will be determined by titration using a known concentration of sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.



FA 3 is a solution containing 31.50 g dm^{-3} of hydrated sodium sulfite, $\text{Na}_2\text{SO}_3 \cdot x\text{H}_2\text{O}$.

FA 4 is $0.100 \text{ mol dm}^{-3}$ iodine, I_2 .

FA 5 is $0.100 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FA 6 is starch indicator.

(a) Method

- Pipette **10.0** cm^3 of **FA 3** into a conical flask.
- Pipette **25.0** cm^3 of **FA 4** into the same flask.
- Swirl the flask to mix the contents.
- Fill the second burette with **FA 5**.
- Add **FA 5** to the flask until the mixture is yellow.
- Add approximately 10 drops of **FA 6**.
- Complete the rough titration by adding **FA 5** until the mixture is colourless.
- 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.
- Make sure any recorded results show the precision of your practical work.
- Record, in a suitable form below, all of your burette readings and the volume of **FA 5** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	

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

10.0 cm³ of **FA 3** plus 25.0 cm³ of **FA 4** required cm³ of **FA 5**. [1]

(c) Calculations

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

- (ii) Use your answer to (b) to calculate the amount, in mol, of sodium thiosulfate, **FA 5**, required to react with the excess iodine which remained in the conical flask.

amount of Na₂S₂O₃ = mol

Hence calculate the amount, in mol, of iodine, **FA 4**, remaining in the conical flask.

amount of I₂ remaining = mol [1]

- (iii) Calculate the amount, in mol, of iodine, **FA 4**, added to the conical flask.

amount of I₂ added = mol

Hence calculate the amount, in mol, of iodine that reacted with the 10.0 cm³ of sodium sulfite, **FA 3**.

amount of I₂ that reacted with Na₂SO₃ = mol [1]

- (iv) Use your final answer to (c)(iii) and the information on page 5 to calculate the amount, in mol, of sodium sulfite present in 1.00 dm³ of **FA 3**.

amount of Na₂SO₃ in 1.00 dm³ = mol [1]



(v) Use your answer to (c)(iv) to calculate the value of x in $\text{Na}_2\text{SO}_3 \cdot x\text{H}_2\text{O}$.

$x = \dots\dots\dots$ [2]

(d) A student suggests that sodium carbonate should be added to each mixture of sodium sulfite and iodine in the conical flask before titrating with sodium thiosulfate.

State whether you agree with the student. Explain your answer.

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

[Total: 15]

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 FA 7 and FA 8 are solutions containing a total of three cations and two anions. Two of the cations and both of the anions are listed in the Qualitative analysis notes.

(a) (i) Carry out the following tests and record your observations. Use a fresh 1 cm depth of solution in a test-tube for each test.

test	observations	
	FA 7	FA 8
Test 1 Add a 1 cm depth of dilute nitric or hydrochloric acid and allow to stand for 2 minutes, then		
add a few drops of aqueous barium nitrate or aqueous barium chloride.		
Test 2 Add a few drops of acidified aqueous potassium manganate(VII).		
Test 3 Add a few drops of aqueous iron(III) chloride and allow to stand for 1 minute.		

- (ii) From your test results, give the formulae of the anions present in **FA 7** and **FA 8**. If the tests do not allow you to positively identify an anion, write 'unknown'.

anion in **FA 7** =

anion in **FA 8** =

[2]

- (b) (i) Select reagents for tests to identify as many of the cations as possible in **FA 7** and **FA 8**. Carry out your tests and record your reagents, conditions and observations.

[4]

- (ii) From your test results, give the formulae of as many cations as possible in **FA 7** and **FA 8**. If the tests do not allow you to positively identify a cation, write 'unknown'.

FA 7 contains

FA 8 contains

[2]

- (iii) Write an ionic equation for **one** reaction you observed in (b)(i). Include state symbols.

..... [1]

[Total: 13]

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 $\text{J g}^{-1} \text{ K}^{-1}$)

Group																
1	2	Group														
		1 H hydrogen 1.0														
		Key														
		atomic number atomic symbol name relative atomic mass														
3	4															
Li lithium 6.9	Be beryllium 9.0															
11	12															
Na sodium 23.0	Mg magnesium 24.3															
19	20															
K potassium 39.1	Ca calcium 40.1															
37	38															
Rb rubidium 85.5	Sr strontium 87.6															
55	56															
Cs caesium 132.9	Ba barium 137.3															
87	88															
Fr francium —	Ra radium —															
21	22															
Sc scandium 45.0	Ti titanium 47.9															
39	40															
Y yttrium 88.9	Zr zirconium 91.2															
71	72															
La lanthanoids —	Hf hafnium 178.5															
89–103	Rf rutherfordium —															
actinoids —	Db dubnium —															
105	106															
Sg seaborgium —	Bh bohrium —															
107	108															
Re rhenium 186.2	Hs hassium —															
75	76															
Os osmium 190.2	Ru ruthenium 101.1															
77	78															
Ir iridium 192.2	Rh rhodium 102.9															
79	80															
Au gold 197.0	Cd cadmium 112.4															
81	82															
Tl thallium 204.4	Pb lead 207.2															
83	84															
Bi bismuth 209.0	Po polonium —															
85	86															
I iodine 126.9	At astatine —															
51	52															
Sb antimony 121.8	Te tellurium 127.6															
53	54															
Br bromine 79.9	Kr krypton 83.8															
35	36															
Se selenium 79.0	Zn zinc 65.4															
33	34															
As arsenic 74.9	Ga gallium 69.7															
12	13															
10	11															
8	9															
Fe iron 55.8	Co cobalt 58.9															
26	27															
Ni nickel 58.7	Cu copper 63.5															
28	29															
10	11															
8	9															
12	13															
5	6															
3	4															
Al aluminium 27.0	Si silicon 28.1															
13	14															
P phosphorus 31.0	S sulfur 32.1															
15	16															
7	8															
N nitrogen 14.0	O oxygen 16.0															
9	10															
F fluorine 19.0	Ne neon 20.2															
2																
He helium 4.0																

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	euporium	152.0	64	Gd	gadolinum	157.3	65	Tb	terbium	158.9	66	Dy	dysprosium	162.5	67	Ho	holmium	164.9	68	Er	erbium	168.9	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	—