

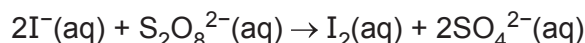
Quantitative analysis

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 Iodide ions in aqueous solution are oxidised to iodine by a variety of oxidising agents. One of these is the peroxodisulfate ion, $\text{S}_2\text{O}_8^{2-}$, which reacts as shown.



Sodium thiosulfate is added to the reaction mixture to react with iodine as it is produced. When all of the thiosulfate has reacted, further iodine produced reacts with starch indicator to give a dark colour.

You will carry out two experiments to investigate how the rate of this reaction is affected by changing the concentration of the peroxodisulfate ion.

FA 1 is $0.0200 \text{ mol dm}^{-3}$ potassium peroxodisulfate, $\text{K}_2\text{S}_2\text{O}_8$.

FA 2 is $0.00500 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

FA 3 is 1.00 mol dm^{-3} potassium iodide, KI .

FA 4 is starch indicator.

(a) Method

Experiment 1

- Label one of the 100 cm^3 beakers **A** and the other 100 cm^3 beaker **B**.
- Fill one burette with **FA 1**. Label this burette **FA 1**.
- Run 20.00 cm^3 of **FA 1** from the burette into beaker **A**.
- Fill the second burette with **FA 2**. Label this burette **FA 2**.
- Run 10.00 cm^3 of **FA 2** from the burette into beaker **B**.
- Use the measuring cylinder to add 20.0 cm^3 of **FA 3** to beaker **B**.
- Add 10 drops of **FA 4** to beaker **B**.
- Add the contents of beaker **A** to beaker **B** and start timing **immediately**.
- Stir the mixture once and place the beaker on the white tile.
- Stop timing as soon as the solution turns a dark colour.
- Record this time to the nearest second in the space for results.
- Wash out both beakers and dry them using paper towel.

Experiment 2

- Run 10.00 cm^3 of **FA 1** from the burette into beaker **A**.
- Run 10.00 cm^3 of **FA 2** from the burette into beaker **B**.
- Use the measuring cylinder to add 20.0 cm^3 of **FA 3** into beaker **B**.
- Use the same measuring cylinder to add 10.0 cm^3 of distilled water to beaker **B**.
- Add 10 drops of **FA 4** to beaker **B**.
- Add the contents of beaker **A** to beaker **B** and start timing **immediately**.
- Stir the mixture once and place the beaker on the white tile.
- Stop timing as soon as the solution turns a dark colour.
- Record this time to the nearest second.

Record all your data in a table. You should include the volume of **FA 1**, the volume of distilled water, the reaction time and the rate of reaction for both experiments.

Use the following formula to calculate the rate of reaction.

$$\text{rate} = \frac{1000}{\text{reaction time}}$$

Results

I	
II	
III	
IV	
V	
VI	

[6]

- (b) (i) Explain why the concentration of potassium peroxodisulfate used in each experiment is proportional to the volume of FA 1 used.

.....
 [1]

- (ii) A student thinks that the rate of reaction is proportional to the concentration of FA 1. Complete Table 1.1 to suggest volumes of reactants that could be used in a further experiment to confirm whether the student is correct. Do **not** carry out this experiment.

Table 1.1

volume / cm ³				volume
FA 1	FA 2	FA 3	distilled water	FA 4
				10 drops

[2]

- (c) A student correctly carried out the method in (a) but had been given a more concentrated solution of sodium thiosulfate. State how you would expect the student's times to differ from yours. Explain your answer.

.....
 [1]

- (d) The potassium iodide is in a large excess in Experiments 1 and 2. Suggest why a large excess of iodide ions is needed in these experiments.

.....
 [1]

[Total: 11]

- 2 You will carry out an experiment to determine the enthalpy change, ΔH , when one mole of ammonium chloride dissolves in water.

FA 5 is ammonium chloride, NH_4Cl .

(a) Method

- Weigh the container with **FA 5**. Record the mass in the space for results.
- Support the cup in the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 25.0 cm^3 of distilled water into the cup.
- Place the thermometer in the water and tilt the cup, if necessary, so that the bulb of the thermometer is fully covered. Record the temperature of the water at time $t = 0$.
- Start the stop-clock and leave it running for the whole experiment.
- Measure and record the temperature of the water in the cup every half minute for 2 minutes.
- At $t = 2\frac{1}{2}$ minutes, tip all the **FA 5** into the cup. Stir the contents of the cup.
- Measure and record the temperature of the contents of the cup at $t = 3$ minutes and then every half minute up to and including $t = 8$ minutes.
- Weigh the container with any residual **FA 5**. Record the mass.
- Calculate and record the mass of **FA 5** added.

Results



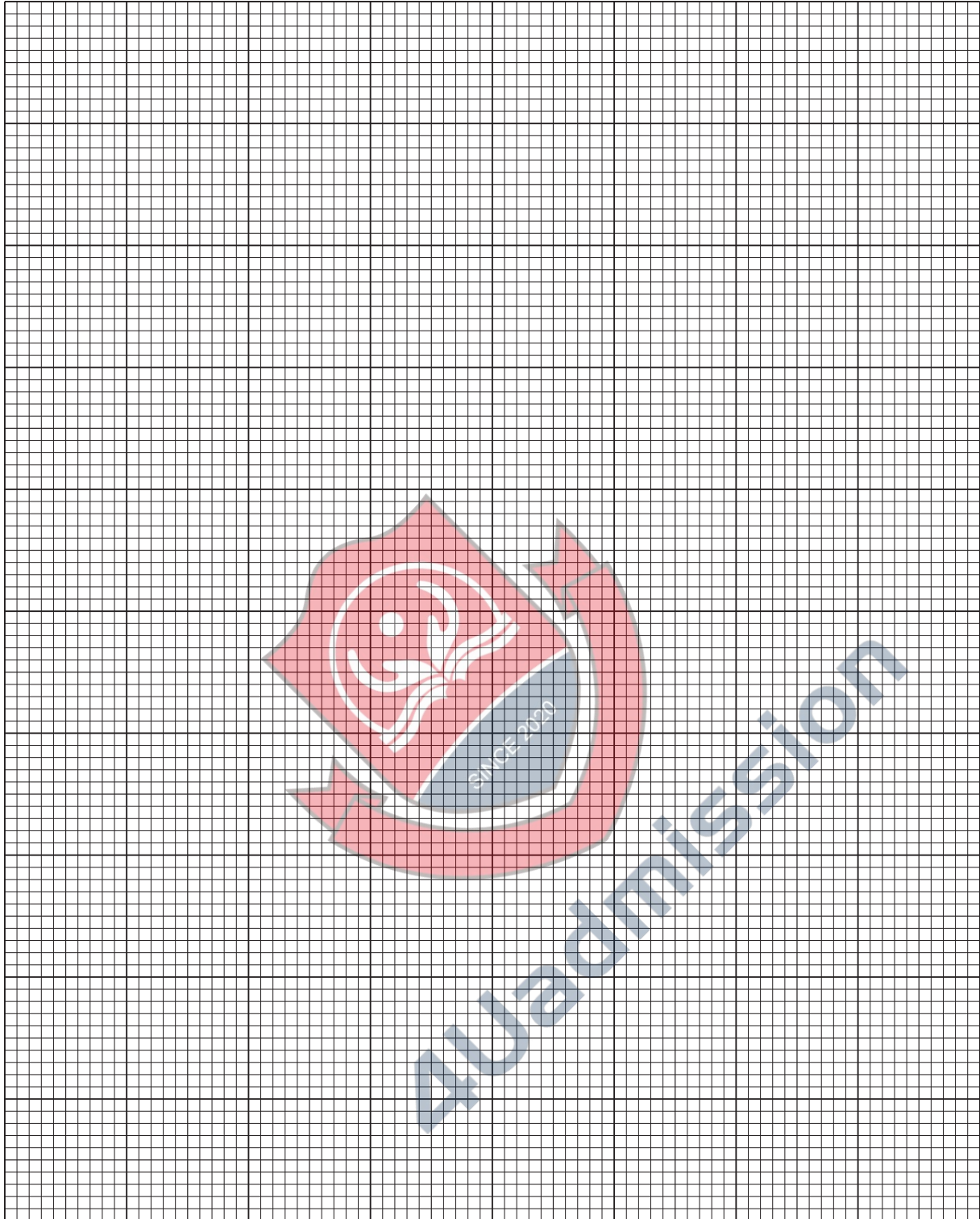
I	
II	
III	
IV	
V	

[5]

- (b)** Plot a graph of temperature (y-axis) against time (x-axis) on the grid. You should choose a scale that allows you to plot 2°C below the minimum temperature reached. Label any points you consider to be anomalous.

Draw **two** straight lines of best fit. One line is for the temperature before adding **FA 5** and the other line is for the warming of the solution once the minimum temperature has been reached.

Extrapolate both these lines to $t = 2\frac{1}{2}$ minutes.



I	
II	
III	
IV	

[4]

(c) (i) Use your graph to determine the temperature change, ΔT , at $t = 2\frac{1}{2}$ minutes.

ΔT at $t = 2\frac{1}{2}$ minutes = °C [1]

- (iii)** Calculate the amount, in mol, of ammonium chloride used.

amount of NH_4Cl = mol [1]

- $$\Delta H = \text{.....} \text{ kJ mol}^{-1} \quad [1]$$

- Assume that the maximum uncertainty in a single thermometer reading is $\pm 0.5^\circ\text{C}$.

Show your working.

maximum percentage error = % [1]

[Total: 14]

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 a 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 (a) **FA 6** is a salt containing a Group 1 ion and an anion that consists of a transition metal and a non-metal element.

- (i) Transfer **FA 6** into a hard-glass test-tube. Heat the tube gently at first and then strongly. Record all your observations and identify the gas produced.
The residue is **FA 7**. You will use **FA 7** in (a)(ii).

observations

.....

gas produced

[3]

Allow FA 7 to cool before starting (a)(ii).

While FA 7 is cooling you may wish to continue with (b)(i).

- (ii) Put a 2 cm depth of acidified aqueous potassium manganate(VII) in a test-tube. Add the same depth of aqueous sodium hydroxide. Then add **FA 7** and stir using the glass rod for about 30 seconds. Filter the mixture and collect the filtrate.
Record your observations.

.....

Put a 1 cm depth of the filtrate in a test-tube. Add sulfuric acid until in excess.
Record your observations.

.....

[2]

- (b) (i) **FA 8** and **FA 9** are both aqueous solutions of salts. **FA 8** contains one cation and one anion. **FA 9** contains two cations and one anion. One of the cations and both anions are listed in the Qualitative analysis notes.

Carry out the following tests and record your observations in Table 3.1.
For each test use a 1 cm depth of **FA 8** or **FA 9** in a test-tube.

Table 3.1

<i>test</i>	<i>observations</i>	
	FA 8	FA 9
Test 1 Add sulfuric acid.		
Test 2 Add aqueous sodium hydroxide, then transfer the mixture into a boiling tube and warm.		
Test 3 Add a few drops of aqueous barium chloride or aqueous barium nitrate, then add nitric acid.		
Test 4 Add FA 8 with shaking until in excess.		

[7]

- (ii) Deduce the identity of the **three** ions listed in the Qualitative analysis notes that are present in **FA 8** and **FA 9**. Suggest the identity of **one** other cation. Give the formula of each ion. If you cannot identify an ion write 'unknown'.

FA 8 contains and

FA 9 contains and and

[2]

- (iii) Write an ionic equation for **one** reaction that occurred in Test 2 in Table 3.1. Include state symbols.

..... [1]

[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	Key												13	14	15	16	17	18		
		<div>1 H hydrogen 1.0</div>																			
		<div>atomic number atomic symbol name relative atomic mass</div>																			
3	4													5	6	7	8	9	10	11	12
Li lithium 6.9	Be beryllium 9.0													B boron 10.8	C carbon 12.0	N nitrogen 14.0	O oxygen 16.0	F fluorine 19.0	Ne neon 20.2		
11	12													13	14	15	16	17	18		
Na sodium 23.0	Mg magnesium 24.3													Al aluminium 27.0	Si silicon 28.1	P phosphorus 31.0	S sulfur 32.1	Cl chlorine 35.5	Ar argon 39.9		
19	20													31	32	33	34	35	36		
K potassium 39.1	Ca calcium 40.1													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													49	50	51	52	53	54		
Rb rubidium 85.5	Sr strontium 87.6													In indium 114.8	Sn tin 118.7	Sb antimony 121.6	Te tellurium 127.6	I iodine 126.9	Xe xenon 131.3		
55	56													81	82	83	84	85	86		
Cs caesium 132.9	Ba barium 137.3													Tl thallium 204.4	Pb lead 207.2	Bi bismuth 209.0	Po polonium —	At astatine —	Rn radon —		
87	88													113	114	115	116	117	118		
Fr francium —	Ra radium —													Nh nihonium —	Fl flerovium —	Mc moscovium —	Lv livermorium —	Ts tennessine —	Og oganesson —		

lanthanoids	57	La	lanthanum	138.9	58	Ce	cerium	140.1	59	Pr	praseodymium	140.9	60	Nd	neodymium	144.2	61	Pm	promethium	—	62	Sm	samarium	150.4	63	Eu	euroium	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
	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	—