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 In this experiment, you will carry out a titration to determine the concentration of a solution of sodium thiosulfate, $Na_2S_2O_3$.

You will first react iodate(V) ions, IO_3^-, with an excess of iodide ions, I^-, to produce a known amount of iodine, I_2. This iodine is then titrated with the solution of sodium thiosulfate of unknown concentration.

FB 1 is aqueous sodium thiosulfate, $Na_2S_2O_3$, of unknown concentration.
FB 2 is aqueous potassium iodate(V) containing $3.35\,g\,dm^{-3}$ KIO_3.
FB 3 is dilute sulfuric acid, H_2SO_4.
FB 4 is $0.500\,mol\,dm^{-3}$ potassium iodide, KI.
FB 5 is starch indicator.

(a) Method

- Fill the burette with **FB 1**.
- Pipette $25.0\,cm^3$ of **FB 2** into a conical flask.
- Use the measuring cylinder to add $10.0\,cm^3$ of **FB 3** to the conical flask.
- Use the same measuring cylinder to add $10.0\,cm^3$ of **FB 4** to the conical flask.
- Add **FB 1** from the burette until the solution turns yellow.
- Add 10–15 drops of **FB 5** to the solution in the conical flask until the mixture turns blue-black.
- Continue to add more **FB 1** from the burette until the mixture just becomes colourless.
- Perform a rough titration and record your burette readings.
- Discard the contents of the conical flask **immediately**. Rinse the conical flask with water.

initial reading/cm³	0.00
final reading / cm³	21.30
rough titre /cm³	21.30

rough titre = 21.30 cm³

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Discard the contents of the conical flask **immediately** after each titration. Rinse the conical flask with water.
- 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 **FB 1** added in each accurate titration.

Keep the remaining FB 1 for use in Question 3.

* 000080000003 * DFD

Results

	1	2
initial reading/cm^3	0.10	0.00
final reading/cm^3	21.10	21.10
titre/cm^3	21.00	21.10

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.

$$\frac{21.00 + 21.10}{2} =$$

25.0 cm^3 of **FB 2** required ....21.05.... cm^3 of **FB 1**. [1]

(c) Calculations

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

(ii) Calculate the amount, in mol, of IO_3^- ions in 25.0 cm^3 of **FB 2**.

$n = c\,(\text{in mol dm}^{-3}) \times V\,(\text{in dm}^{-3})$

$= \frac{3.35 \text{ g / dm}^3}{(23.0+126.9+48.00)\text{ g/mol}} \times (25.0 \times 10^{-3})$

amount of IO_3^- = ...3.91×10^{-4}.... mol [2]

(iii) The equations for the production of iodine and the titration of this iodine with thiosulfate ions are shown.

$$IO_3^- + 5I^- + 6H^+ \rightarrow 3I_2 + 3H_2O$$

$$3 \times (I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-})$$

Use these equations to calculate the amount, in mol, of thiosulfate ions in the volume of **FB 1** in **1(b)**.

$IO_3^- : S_2O_3^{2-}$

$1 : 2 \times 3$

$3.91 \times 10^{-4} : x$

$x = 2.348 \times 10^{-3}$

amount of $S_2O_3^{2-}$ = ...2.35×10^{-3}.... mol [1]

(iv) Calculate the concentration, in mol dm^{-3}, of sodium thiosulfate in **FB 1**.

$$C_{Na_2S_2O_3} = \frac{2.35 \times 10^{-3}}{21.05 \times 10^{-3}}$$

concentration of $Na_2S_2O_3$ = ...1.12×10^{-1}.... mol dm^{-3} [1]

[Total: 13]

2 You will carry out experiments using the hydrated salt $MX_2 \cdot 2H_2O$ and analyse the results to identify **M** and **X**.

M is the cation of a Group 2 metal.
X is the anion of an element in Group 17.

When $MX_2 \cdot 2H_2O$ is heated, the loss of mass is caused **only** by the water of crystallisation evaporating.

FB 6 is the hydrated salt, $MX_2 \cdot 2H_2O$.

(a) Method

(i) Thermal decomposition

- Weigh the empty crucible with its lid. Record the mass in the results section.
- Transfer all the **FB 6** from the container into the crucible.
- Weigh the crucible, lid and **FB 6**. Record the mass.
- Place the crucible, lid and contents on the pipeclay triangle.
- Heat the crucible gently, with the lid on, for approximately 1 minute.
- Heat the crucible strongly, with the lid off, for approximately 5 minutes.
- Replace the lid and leave the crucible to cool for at least 5 minutes.

During the cooling period, 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 the mass of **FB 6** added to the crucible, the mass of the residue obtained and the mass loss. Record these masses.

Results

mass of crucible + lid / g	17.80
mass of crucible + lid + FB 6 / g	19.80
mass of crucible + lid + residue / g	19.50
mass of FB 6 / g	2.00
mass of residue / g	1.70
mass of loss / g	0.30

I	
II	
III	
IV	

[4]

(ii) Anion test

- Use a small amount of your residue from **2(a)(i)** to carry out a test to identify anion **X**. Describe how you carry out your test.
- Record your observations and conclusion.

add $AgNO_3$ (aq)

white ppt, dissolves in dilute NH_3 (aq)

anion **X** .... Cl^- ....

[3]

(b) **Calculations**

(i) Calculate the amount, in mol, of water lost during the thermal decomposition.

$$\frac{0.30}{18.0} = 0.0167$$

amount of H_2O lost = 0.0167 mol [1]

(ii) Use your answers to **2(a)(i)** and **2(b)(i)** to calculate the relative formula mass, M_r, of MX_2.

$$\frac{1.70}{0.0167 \times 0.5} = 203.6$$

M_r of MX_2 = 203.6 [1]

(iii) Use your answers to **2(a)(ii)** and **2(b)(ii)** to calculate the atomic mass, A_r, of **M**. Identify cation **M**.

$$203.6 - 2(35.5) = 132.6$$

A_r of **M** = 132.6

cation **M** Ba^{2+}

[1]

(c) (i) Calculate the percentage error in the value for the M_r of MX_2 that you calculated in **2(b)(ii)** compared to the actual value for the M_r of MX_2.
Show your working.

$$\frac{|208.3 - 203.6|}{208.3} \times 100$$

percentage error = 2.3 % [1]

(ii) Suggest an improvement to the method given in **2(a)(i)** to reduce the percentage error in the value of M_r calculated.

heat to constant mass

[1]

[Total: 12]

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) (i) **FB 7** is solid potassium manganate(VII).

Transfer a small spatula measure of **FB 7** into a hard-glass test-tube. Heat the test-tube gently at first, and then more strongly.

Record your observations. Identify the gas produced.

black solid forms

gas produced

relights a glowing splint

gas produced O_2

[2]

(ii) The reaction that occurs in **3(a)(i)** is a redox reaction.
Explain why this reaction is a redox reaction by referring to one or more of the elements in **FB 7**.

oxidation number of oxygen went from -2 (MnO_4^-) to zero (O_2) [1]

(b) **FB 8** is a solution containing one cation and two anions.
FB 9 is a solution containing one cation and one anion.
All the anions 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 **FB 8** or **FB 9** in a test-tube for each test.

Table 3.1

test	observations	
	FB 8	**FB 9**
Test 1 Add acidified aqueous potassium manganate(VII) dropwise.	turns colorless upon adding the purple drops	no change
Test 2 Add a 1 cm strip of magnesium ribbon.	no change/ very small effervecence	fizzing "pops" with a lighted splint
Test 3 Add aqueous barium chloride or aqueous barium nitrate.	white ppt (soluble in HCl(aq))	white ppt (insoluble in HCl(aq))
Test 4 Add a 1 cm depth of **FB 1**, aqueous sodium thiosulfate. Leave to stand for 1 minute. **Immediately after the test, empty and thoroughly rinse the test-tube.**	no change	pale yellow ppt choking smell gas turns acidified $KMnO_4$ colorless

[5]

(ii) Carry out tests to identify the cation in **FB 8**.
Record your tests and observations in a suitable form.

You **must** use a boiling tube if any liquid is heated.

Test	observation
add NaOH (aq)	no change
then warm	gas produced. turns damp red litmus paper to blue

[3]

(iii) Use your observations in **3(b)(i)** and **3(b)(ii)** to identify the ions present in **FB 8**. If you **cannot** positively identify an ion or ions, write 'unknown'.

cation in **FB 8** NH_4^+

anions in **FB 8** SO_3^{2-} and unknown

[2]

(iv) Deduce the formula of **FB 9**.

FB 9 H_2SO_4 [1]

(v) Write an ionic equation for **one** reaction in **3(b)(i)** involving **FB 9**.
Include state symbols.

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

[Total: 15]

$Ba^{2+}_{(aq)} + SO_4^{2-}{}_{(aq)} \longrightarrow BaSO_{4(s)}$

$S_2O^{2-}_{3(aq)} + 2H^+_{(aq)} \longrightarrow S_{(s)} + SO_{2(g)} + H_2O_{(l)}$