

CHEMISTRY LAB MANUAL

2016-17

Solved

[Signature]

QUALITATIVE ANALYSIS

EXPERIMENT NO. 1

FA1, FA2, FA3, FA4, FA5 and **FA6** are aqueous solutions each containing one of the ions Al^{3+} , Mg^{2+} , Cu^{2+} , Fe^{2+} , Fe^{3+} and Zn^{2+}

You will carry out the following tests on each of the solutions.

At each stage of the test you are to record any color changes seen, the formation of any precipitate and the solubility of such precipitates in an excess of the reagent added. Where gases are released they should be identified by a test, **describe in the appropriate place in your observations.**

Carry out the following tests. Record your observations in the spaces provided in the table.

TESTS	FA1	FA2	FA3	FA4	FA5	FA6
(a) To 1cm depth of solution in a test tube add 1cm depth of aqueous sodium hydroxide. Swirl the tube, then	White ppt	white ppt	blue ppt	green ppt turned brown on contact with air	red-brown ppt	white ppt
Add a further 2cm of depth of aqueous sodium hydroxide	soluble in excess	insoluble in excess	insoluble in excess	insoluble in excess	insoluble in excess	soluble in excess

TESTS	FA1	FA2	FA3	FA4	FA5	FA6
(b) To 1cm depth of solution in a test tube add 1cm depth of aqueous ammonia. Swirl the tube, then	white ppt	white ppt	blue ppt	green ppt turned brown on contact with air	red-brown ppt	white ppt
Add a further 2cm of depth of aqueous ammonia	insoluble in excess	insoluble in excess	soluble in excess gives a deep blue solution	insoluble in excess	insoluble in excess	soluble in excess

Results

solution	cation present
FA1	Al^{3+}
FA2	Mg^{2+}
FA3	Cu^{2+}
FA4	Fe^{2+}
FA5	Fe^{3+}
FA6	Zn^{2+}

EXPERIMENT NO. 2

FA1, FA2, FA3, FA4, FA5, FA6, FA7 and FA8 are aqueous solutions each containing one of the ions CO_3^{2-} , Cl^- , Br^- , I^- , NO_3^- , NO_2^- , SO_4^{2-} , SO_3^{2-} . You will carry out the following tests on each of the solutions.

At each stage of the test you are to record any color changes seen, the formation of any precipitate and the solubility of such precipitates in an excess of the reagent added. Where gases are released they should be identified by a test, **describe in the appropriate place in your observations.**
Carry out the following tests. Record your observations in the spaces provided in the table.

TESTS	FA1	FA2	FA3	FA4	FA5	FA6	FA7	FA8
(a) To 1cm depth of solution in a test tube add 1cm depth of silver nitrate then		White ppt	off-white ppt	pale yellow ppt				
Add a 2cm of depth of aqueous ammonia		Soluble in excess	partially soluble in excess	insoluble in excess				
(b) To 1cm depth of solution add 1cm depth of aqueous sodium hydroxide and a piece of aluminium foil, heat the mixture						a colorless gas produced which turned damp red litmus paper blue.		

TESTS	FA1	FA2	FA3	FA4	FA5	FA6	FA7	FA8
(c) To 1cm depth of solution in a test tube add 1cm depth of dilute hydrochloric acid	eff. of a colorless gas which gives white ppt with lime water					a pale brown gas produced		a colorless gas produced which turned acidified potassium manganate purple to colorless
(d) To 1cm depth of solution in a test tube add 1cm depth of barium nitrate Then Add a 2cm of depth of dilute hydrochloric acid							white ppt	white ppt
							insoluble in excess	soluble in excess

Use the Qualitative Analysis Notes to identify the anion present in each of the solutions.
 Complete the table below to identify each ion and to give supporting evidence from your observations.

solution	anion	Evidence
FA1	CO_3^{2-}	CO_2 gas produced by dilute HCl
FA2	Cl^-	white ppt with aq. AgNO_3 , soluble in excess of aq. NH_3
FA3	Br^-	off-white with aq. AgNO_3 , partially soluble in excess of aq. NH_3
FA4	I^-	pale yellow ppt with aq. AgNO_3 , insoluble in excess of aq. NH_3
FA5	NO_3^-	ammonia produced on heating with aq. NaOH and Al-foil
FA6	NO_2^-	pale brown gas produced by dilute HCl
FA7	SO_4^{2-}	white ppt with aq. Ba^{2+} , insoluble in excess of dilute HCl
FA8	SO_3^{2-}	white ppt with aq. Ba^{2+} , soluble in excess of dilute HCl

EXPERIMENT NO. 3

Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

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

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

(a) **FA 6** is a solution of two different salts. It contains two different cations, one of which is listed in the Qualitative Analysis Notes. It contains two anions, both of which are listed in the Qualitative Analysis Notes..

(i) Choose reagents that will allow you to identify one of the cations. Carry out suitable tests using these reagents and record your results in the space below.

tests	observations
To 1cm ³ of solution add 1cm ³ of aq. NaOH then in excess	white ppt soluble in excess
To 1cm ³ of solution add 1cm ³ of aq. NH ₃ then in excess	white ppt soluble in excess

I	
II	
III	
IV	
V	

One of the cations in **FA 6** is Zn^{2+}

(ii) Carry out the following tests to identify the two anions present in **FA 6**.

<i>test</i>	<i>observations</i>
To a 1 cm depth of FA 6 in a test-tube add a 1 cm depth of aqueous silver nitrate, then	off-white ppt
add aqueous ammonia.	partially soluble
To a 1 cm depth of FA 6 in a test-tube add a 1 cm depth of aqueous barium chloride (or aqueous barium nitrate), then	white ppt
add dilute nitric acid.	insoluble in excess

The anions in **FA 6** are Br^- and SO_4^{2-} .

[9]

VI	
VII	
VIII	
IX	

(b) FA 7 is an acidified solution of iron(II) sulfate, $\text{FeSO}_4(\text{aq})$.

Carry out the following tests and record your observations.

test	observations
(i) To a 1 cm depth of FA 7 in a test-tube add aqueous sodium hydroxide and leave for a few minutes.	green ppt turned brown on contact with air
(ii) To a 1 cm depth of FA 7 in a boiling tube add a 1 cm depth of dilute sulfuric acid followed by a 1 cm depth of '20 vol' hydrogen peroxide. Stir the mixture, then	no change / yellow solution
(iii) pour a 1 cm depth of the mixture into a clean boiling tube and add a 3 cm depth of aqueous sodium hydroxide.	red-brown ppt and eff. a gas produced which relights a glowing splint

I	
II	
III	
IV	
V	
VI	

(iv) What type of reaction takes place in (ii)?

Redox reaction

(v) Explain your observations in (iii).

Fe^{2+} oxidized to Fe^{3+} , so red-brown ppt with aq. NaOH.
 H_2O_2 decomposed, produced H_2O and O_2 .

[6]

[Total: 15]

EXPERIMENT NO. 4

Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

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

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

(a) **FA 4**, **FA 5** and **FA 6** are solutions, each containing one transition metal ion. One of the solutions also contains the ammonium ion. All the cations present are listed in the Qualitative Analysis Notes.

(i) Carry out the following tests on the three solutions.

	<i>test</i>	<i>observations</i>
I	To a 1 cm depth of FA 4 in a test-tube, add FA 1 , aqueous potassium manganate(VII), dropwise.	no change
II		
III		
IV	To a 1 cm depth of FA 5 in a test-tube, add FA 1 , aqueous potassium manganate(VII), dropwise.	purple color decolorizes, brown ppt formed
	To a 1 cm depth of FA 6 in a test-tube, add FA 1 , aqueous potassium manganate(VII), dropwise.	purple color of aq. KMnO_4 decolorizes

(ii) State which solution(s) contain ions which have been oxidised.

FA5 and FA6

[4]

(b) (i) Select a reagent or reagents to identify **all** the cations present in the three solutions.

reagent(s) aq. NaOH and aq. NH₃

Carry out experiments using your reagent(s) on each of **FA 4**, **FA 5** and **FA 6** and record your observations in a suitable form in the space below.

Tests	Observations		
	FA4	FA5	FA6
To 1cm depth of solution add 1cm ³ of aq. NaOH then in excess warm the mixture	red-brown ppt insoluble in excess no change	off-white ppt turned brown on contact with air insoluble in excess no change	green ppt turned brown on contact with air insoluble in excess a colorless gas produced which turned damp red litmus paper blue
To 1cm depth of a solution add 1cm ³ of aq. NH ₃ then in excess	red-brown ppt insoluble in excess	off-white ppt turned brown on contact with air insoluble in excess	green ppt turned brown on contact with air insoluble in excess

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

(ii) Use your observations to identify the cations present in the three solutions.

FA 4 contains Fe³⁺

FA 5 contains Mn²⁺

FA 6 contains Fe²⁺ and NH₄⁺

(c) Each of the solutions **FA 4**, **FA 5** and **FA 6** contains either a chloride or a sulfate ion.

(i) Choose a reagent or reagents to identify which solution(s) contain **chloride** ions.

reagent(s) *aq. AgNO₃*

Use your reagent(s) to carry out a test on each of **FA 4**, **FA 5** and **FA 6** and record your results in the space below.

test	Observations		
	FA4	FA5	FA6
<i>To 1cm depth of solution add 1cm depth of aq. AgNO₃</i>	<i>white ppt</i>	<i>no change</i>	<i>no change</i>

(ii) State which solution(s) contain a chloride ion.

FA4

[3]

[Total: 15]

EXPERIMENT NO. 5

Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

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

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

(a) In **Question 1** you used **FA 2**. This solution was prepared from hydrated ammonium iron(II) sulfate, $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$.

To a 1 cm depth of **FA 2** in a test-tube, add a small spatula measure of sodium carbonate. Record your observations.

test	Observation
To 1cm depth of FA2 in a test tube, add a small spatula measure of sodium carbonate	effervescence of a colorless gas produced which gave white ppt with Lime water.

Solutions containing Fe^{2+} ions can quickly be oxidised in air if they are prepared by dissolving the solid in distilled water.

Use your observations to suggest what other substance was added to solid $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ to prepare **FA 2**.

an acid / H^+

[2]

- (b) **FA 6** is a mixture of two salts, each of which contains a single cation and a single anion from those listed in the Qualitative Analysis Notes. Do the following tests and record your observations in the table below.

test	observations
(i) Place a small spatula measure of FA 6 in a hard-glass test-tube and heat strongly.	i) solid turned grey/black from green ii) Steamy fumes / water condensed. iii) Solid melts.
(ii) Place a small spatula measure of FA 6 in a test-tube and carefully add dilute sulfuric acid until the reaction is complete, then	effervescence of colorless gas, which gave white ppt with lime water. solid dissolved give a pale blue solution
add aqueous sodium hydroxide.	Blue ppt. insoluble in excess
(iii) To a 3 cm depth of distilled water in a boiling tube, add the remaining sample of FA 6 . Stir and then filter the mixture into a clean boiling tube. You will use this solution for tests (iv)–(vi).	X
(iv) To a 1 cm depth of the solution from (iii) in a test-tube, add aqueous sodium hydroxide.	white ppt. insoluble in excess
(v) To a 1 cm depth of the solution from (iii) in a test-tube, add aqueous ammonia.	white ppt. insoluble in excess
(vi) To a 1 cm depth of the solution from (iii) in a test-tube, add aqueous barium chloride or aqueous barium nitrate.	white ppt. formed

(vii) Suggest possible identities for the ions present in **FA 6**.

cations Cu^{2+} and Mg^{2+}

anions CO_3^{2-} and $\text{SO}_4^{2-} / \text{SO}_3^{2-}$

(viii) Describe a further test that would allow you to determine exactly which anions are present. Explain your choice. Do **not** do this test.

add any dilute strong acid other than H_2SO_4 in test
(vi) after $\text{BaCl}_2(\text{aq})$, ppt of BaSO_4 will be insoluble and
ppt of BaSO_3 will be soluble.

[11]

[Total: 13]

EXPERIMENT NO. 6

Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

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

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

- (a) **FB 4** and **FB 5** are solutions of salts each containing one cation and one anion from those listed in the Qualitative Analysis Notes. Carry out the following tests and record your observations in the table below.

test	observations	
	FB 4	FB 5
(i) To a 1 cm depth of solution in a test-tube, add aqueous ammonia.	off-white ppt. rapidly turned brown on contact with air. Insoluble in excess	white ppt. insoluble in excess
(ii) To a 1 cm depth of solution in a test-tube, add a few drops of aqueous silver nitrate.	white ppt.	no change
(iii) To a 1 cm depth of solution in a test-tube add a few drops of aqueous barium nitrate.	no change	white ppt.

(iv) Identify both ions in **FB 4**.

cation Mn^{2+} anion Cl^{-}

(v) Suggest the ions which may be present in **FB 5**.

cations Mg^{2+} / Al^{3+} anions SO_4^{2-} / SO_3^{2-}

(vi) Select a reagent which could be used in a further test on **FB 5** to identify the **cation** present. Carry out your test and record your observations.

test	observations
To a 1 cm depth of FB 5 in a test-tube, add $aq. NaOH$	white ppt. insoluble in excess

The cation in **FB 5** is Mg^{2+}

[7]

(b) **FB 6** is a pale purple salt containing two cations.

(i) What does this suggest about the identity of one of the cations in **FB 6**?

..... *Transition metal*

Carry out the following tests and complete the table below.

test	observations
(ii) Place a spatula measure of FB 6 in a hard-glass test-tube. Heat gently.	* Solid melted. * solid turned brown. * water condensed. * Steamy fumes
(iii) Dissolve a small spatula measure of FB 6 in a 2cm depth of distilled water in a test-tube. Use this solution for tests (iv) and (v).	solid dissolved a pale yellow solution formed
(iv) Pour about half the solution prepared in (iii) into a boiling tube and add aqueous sodium hydroxide, then	brown / red-brown ppt. formed insoluble in excess
gently warm the mixture.	a colorless gas produced which turned damp red Litmus paper blue.
(v) To the remainder of the solution prepared in (iii), add a few drops of aqueous potassium iodide, then	no change / solution turned yellow
add a few drops of starch solution.	solution turned purple / blue-black

(vi) Identify the cations present in **FB 6**.

FB 6 contains Fe^{3+} and NH_4^+

(vii) What type of reaction occurred when potassium iodide was added to **FB 6** in (v)?

..... redox reaction. oxidation of I^- and reduction of Fe^{3+}

[7]

[Total: 14]

EXPERIMENT NO. 7

Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

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

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

- (a) **FA 5** and **FA 6** are solutions each containing one cation and one anion. Use a 1 cm depth of **FA 5** or **FA 6** in a test-tube to carry out the following tests on the two solutions and record your observations.

test	observations	
	FA 5	FA 6
Add aqueous sodium hydroxide.	no change	white ppt. soluble in excess
Add aqueous ammonia.	no change	white ppt. insoluble in excess
Add a 1 cm depth of dilute hydrochloric acid, then	brown / pale-brown gas produced	no change
transfer the mixture into a boiling tube and warm gently.	brown / pale-brown gas produced	no change
Add two or three drops of acidified aqueous potassium manganate(VII).	Purple color of aq. KMnO_4 decolorises	Purple color of aq. KMnO_4 remain unchanged
Add a 1 cm depth of aqueous barium chloride or barium nitrate, then	no change	white ppt.
add dilute hydrochloric acid.	—	insoluble in HCl

Identify as many of the ions present in **FA 5** and **FA 6** as possible from your observations. If you are unable to identify any of the ions from your observations, write 'unknown' in the space.

	FA 5	FA 6
cation	unknown	Al^{3+}
anion	NO_2^-	SO_4^{2-}

- (b) **FA 7** is a solid with an anion containing the same element as one of the anions in either **FA 5** or **FA 6** but in a different oxidation state. Relevant anions are listed in the Qualitative Analysis Notes.

Place a spatula measure of **FA 7** in a boiling tube and add a 2 cm depth of distilled water. Shake the boiling tube to dissolve the solid and make a solution of **FA 7**.

- (i) Select reagents to test whether the anion in **FA 7** contains the same element as the anion in **FA 5**. Carry out your test(s) on the solution of **FA 7** and record your observations **and conclusions** in a suitable form in the space below.

reagents for testing **FA 7**

aq. NaOH + Al-Foil and heat

observations and conclusions

test	observation
To 1cm depth of solution in a test tube, add aq. NaOH and a piece of Al-Foil and heat the mixture	damp red litmus paper remains unchanged

Conclusion: NO_3^- is absent

- (ii) Select reagents to test whether the anion in **FA 7** contains the same element as the anion in **FA 6**. Carry out your test(s) on the solution of **FA 7** and record your observations **and conclusions** in a suitable form in the space below.

reagents for testing **FA 7**

aq. BaCl_2 followed by dilute HCl.

observations and conclusions

test	observations
To 1cm depth of solution in a test tube, add aq. BaCl_2	white ppt.
add dilute HCl in excess	ppt. soluble in HCl

[5]

[Total: 13]

EXPERIMENT NO. 8

Qualitative analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

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

Where reagents are selected for use in a test the full name or correct formula of the reagent must be given.

- (a) You are provided with solution **FA 5**. **FA 5** is an aqueous mixture of two salts and contains two cations and two anions. Carry out the following tests and complete the table below.

<i>test</i>	<i>observations</i>
To a 1 cm depth of FA 5 in a test-tube, add aqueous sodium hydroxide.	blue ppt. insoluble in excess
To a 1 cm depth of FA 5 in a test-tube, add aqueous ammonia.	blue ppt. soluble in excess produced a dark blue solution
To a 1 cm depth of FA 5 in a test-tube, add a 2 cm depth of dilute sulfuric acid, shake, and leave for about 1 minute,	brown gas produced
then add aqueous potassium manganate(VII) drop by drop.	purple color of aq. KMnO_4 decolorizes
To a 1 cm depth of FA 5 in a test-tube, add a 1 cm depth of aqueous potassium iodide,	brown ppt. a brown solution
followed by a few drops of starch indicator.	mixture turned blue-black

[5]

- (b) **FA 5** contains either or both a sulfate and/or a chloride. Select reagents and use them to carry out further tests on **FA 5** to positively identify which of these anions is present.

reagents *aq. AgNO₃* and *aq. BaCl₂ / Ba(NO₃)₂*

Record your tests and all your observations in a suitable form in the space below.

tests	Observations
(i) add <i>aq. AgNO₃</i>	<i>no change</i>
(ii) add <i>aq. BaCl₂ / Ba(NO₃)₂</i>	<i>white ppt</i>

[4]

- (c) Use your observations in (a) and (b) to suggest the identities of as many ions present in **FA 5** as possible. Give reasons for your deductions for one cation and one anion.

possible cation(s) *Cu²⁺*

reasons(s) *blue ppt. with both aq. NaOH and aq. NH₃*

possible anion(s) *NO₂⁻* and *SO₄²⁻*

reasons(s) *brown gas with dilute H₂SO₄*

..... *white ppt. with aq. BaCl₂* [4]

[Total: 13]

EXPERIMENT NO. 9

For
Examiner's
Use

You are provided with three solutions, **FB 6**, **FB 7** and **FB 8**, each containing one cation and one anion.

One or more of the solutions contains a halide ion. One or more of the solutions contains a sulphate or sulphite ion.

Identification of the anions in **FB 6**, **FB 7** and **FB 8**

(a) By reference to the Qualitative Analysis Notes you are to select and use

- (i) one reagent to precipitate any halide ion that is present,
- (ii) a second reagent to confirm the identity of any halide ion present.

Because the solutions are coloured you will need to remove traces of solution from the precipitates.

Record the tests performed, the practical procedures used and the observations made for each of the solutions.

Present this information as clearly as possible in a suitable format in the space below.

tests	Observations		
	FB6	FB7	FB8
To 1cm depth of solution in a test tube, add aq. AgNO_3	white ppt.	yellow ppt.	no change
----- add aq. NH_3 in excess	soluble in excess of aq. NH_3	insoluble in excess	—

i	
ii	
iii	
iv	
v	
vi	
vii	

Use your observations to identify any halide ions present in the solutions **FB 6**, **FB 7** and **FB 8** and state which ion is present in which solution.

..... *FB6 contains Cl^- ions.*

..... *FB7 contains I^- ions.*

..... *FB8 contains no halide ion*

.....

.....

(b) Select reagents and carry out tests

- (i) to show which of the solutions contains a sulphate ion or a sulphite ion, and
(ii) to establish which of these ions is present.

Record your tests and observations below.

tests	Observations		
	FB 6	FB 7	FB 8
To 1cm depth of solution in a test tube, add aq. BaCl_2 or $\text{Ba}(\text{NO}_3)_2$	no change	no change	white ppt.
add dil. HCl in excess	—	—	insoluble in excess of dil. HCl

State which of the ions, sulphate or sulphite, is present in which of the solutions **FB 6**, **FB 7** and **FB 8** and explain how you reached this conclusion from your tests above.

FB 8 contains SO_4^{2-} ions.
FB 8 gave white ppt. with aq. BaCl_2 , insoluble in dilute HCl.

[3]

Identification of the cations in FB 6, FB 7 and FB 8

(c) Using aqueous sodium hydroxide and aqueous ammonia it is possible to identify two of the cations present and to draw some conclusions about the nature of the remaining cation.

Carry out tests with these reagents, recording details of what you did and observed in a suitable format in the space below.

tests	Observations		
	FB 6	FB 7	FB 8
(i) To 1cm depth of solution in a test tube, add aq. NaOH little at a time until in excess	red-brown ppt. insoluble in excess	grey-green ppt. soluble in excess gave a dark green solution	blue ppt. turned brown insoluble in excess
(ii) To 1cm depth of solution in a test tube, add aq. NH_3 little at a time until in excess	red-brown ppt. insoluble in excess	grey-green ppt. insoluble in excess	blue-green ppt. insoluble in excess

[4]

(d) Explain how your observations in (c) identify **two** of the cations present and which of the solutions contain those cations.

The cation contained in solution **FB** 6 is Fe^{3+}

explanation

Red-brown ppt. with aq. NaOH and aq. NH_3
.....
.....

The cation contained in solution **FB** 7 is Cr^{3+}

explanation

Grey-green ppt. with aq. NaOH, soluble in excess and grey-green
ppt. insoluble with aq. NH_3 insoluble in excess.
.....
.....

What conclusion of a general nature about the third cation can you draw from your observations in (c)?

FB8 contains a transition metal ion. As it formed
colored ppt. with aq. NaOH and aq. NH_3 .
.....
.....
.....
.....

[2]

[Total: 16]

EXPERIMENT NO. 10

Qualitative Analysis

For
Examiner's
Use

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

When gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions should be attempted.

If any solution is warmed, a boiling tube MUST be used.

Rinse and re-use test-tubes and boiling tubes where possible.

Where reagents are selected for use in a test, the full name or correct formula of the reagents must be given.

- (a) **FA 7** contains one cation and one anion from those listed in the Qualitative Analysis Notes.

Put two spatula measures of **FA 7** into a test-tube.

Add about two-thirds of a test-tube of distilled water and dissolve the solid.

For each test that you carry out, use 1 cm depth of the solution of **FA 7**.

- (i) Carry out the following tests and complete the table below.

<i>test</i>	<i>observation(s)</i>
Add 5 drops of aqueous barium nitrate to your solution of FA 7 .	no change
Add 5 drops of aqueous silver nitrate to your solution of FA 7 .	no change

I	
II	
III	

- (ii) Put a **very small** spatula measure of solid **FA 7** into a hard glass test-tube. Hold the test-tube horizontally and heat it gently for a few seconds, then heat it strongly until no further change takes place. Leave the test-tube to cool to room temperature. *While cooling takes place, move on to (iv).* In the space below record the observations made at each stage in an appropriate form.

tests	observations
on gentle heating	solid melted
on strong heating	brown gas produced
on cooling	white solid formed

- (iii) State what deductions you can make about the identity of the anion in **FA 7** from the tests above.

..... Anion in FA7 might be NO_3^-

- (iv) Use the information in the Qualitative Analysis Notes to select a further test to confirm the identity of the anion in **FA 7**.

test Heat FA7 with aq. NaOH and Al-foil

Carry out **this test** and, in the space below, record the observation(s) made in an appropriate form. State your conclusion.

test	Observation
To 1cm depth of solution in a test tube, add aq. NaOH, Al-foil and heat	a colorless gas produced which turned damp red litmus paper blue.

FA7 contains NO_3^-

IV	
V	
VI	
VII	
VIII	
IX	

- (v) The cation in **FA 7** is aluminium ion, calcium ion or zinc ion. Select **one reagent** to identify the cation in **FA 7**.

reagent aqueous Ammonia

Use this reagent to carry out a test. Record the observation(s) made and identify the cation.

..... white ppt. soluble in excess

..... Zn^{2+} is present

(b) **FA 8** contains one cation from those listed on Qualitative Analysis Notes.

Put all of the **FA 8** into a test-tube.

Half fill the test-tube with distilled water and dissolve the solid.

- (i) To 1 cm depth of the solution of **FA 8** in a test-tube, add aqueous potassium iodide until the test-tube is half full. Allow the mixture to stand for two minutes.

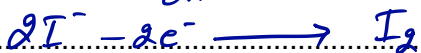
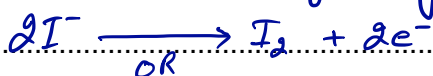
Use a dropping pipette to transfer about 1 cm³ of the mixture from the top of the test-tube to another test-tube. Add 5 drops of starch solution.

Record all of your observations.

tests	observations
To 1cm depth of solution in a test tube, add aq. KI	brown ppt. and brown solution formed
add starch solution	solution turned blue-black

- (ii) State what **type** of chemical behaviour has been shown by potassium iodide in this reaction. Give an ionic equation to justify your answer.

KI acts as reducing agent.



- (iii) To another 1 cm depth of solution of **FA 8** in a test-tube, add aqueous sodium hydroxide.

Record the observation(s) made.

Give the **ionic** equation for the reaction taking place.

blue ppt. formed, insoluble in excess of aq.

NaOH

Cu²⁺ is present.



[Total: 14]

I	
II	
III	
IV	
V	

EXPERIMENT NO. 11

For
Examiner's
Use

Qualitative analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

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

Where reagents are selected for use in a test the full name or correct formula of the reagent must be given.

- (a) Compounds **FB 5**, **FB 6** and **FB 7** contain the same non-metal but in three different oxidation states. You are provided with solid samples of **FB 5**, **FB 6** and **FB 7**. Carry out the tests described below and record your observations in the table.

test	observations
(i) To 1 cm depth of dilute hydrochloric acid in a test-tube add a small spatula measure of FB 5 .	Bubble of brown gas. Pale blue solution formed.
(ii) To 1 cm depth of dilute sulfuric acid in a boiling tube add the same depth of aqueous potassium iodide. Add a small spatula measure of FB 5 .	Colorless solution turned red-brown/ orange. Black/dark grey ppt./solid formed.
(iii) To 1 cm depth of dilute sulfuric acid in a test-tube add about ten drops of aqueous potassium manganate(VII). Add a small spatula measure of FB 5 .	Purple solution turned colorless. brown gas produced and solution turned pale blue.

test	observations
(iv) Place a small spatula measure of FB 6 into a hard glass test-tube. Heat the contents gently.	Solid sublimes. white fumes
(v) Place a small spatula measure of FB 6 into a boiling tube. Dissolve the solid in 1 cm depth of distilled water. Add 1 cm depth of aqueous sodium hydroxide. Warm the mixture with care .	a colorless gas produced which turned damp red Litmus paper blue
(vi) Place a small spatula measure of FB 7 into a hard glass test-tube. Heat the contents gently at first, then heat more strongly. Allow to stand for a few minutes	Solid melted on heating. a pale yellow solid formed on cooling.
(vii) Place a small spatula measure of FB 7 into a boiling tube. Dissolve the solid in about 1 cm depth of distilled water. Add 1 cm depth of aqueous sodium hydroxide. Warm the mixture with care .	No reaction. Damp red litmus paper remained unchanged

I	
II	
III	
IV	
V	
VI	

[6]

- (b) (i) From your observations in (a), identify the non-metal present in **FB 5**, **FB 6** and **FB 7**.

..... N

- (ii) Suggest the oxidation state of the non-metal in **FB 5** and **FB 6**.

The oxidation state of the non-metal in **FB 5** is +3

The oxidation state of the non-metal in **FB 6** is -3

- (iii) Suggest the type of reaction occurring in (a)(iii).

..... redox reaction

[3]

(c) Solid compounds containing Fe^{2+} and Ni^{2+} are usually green. One of these ions is present in **FB 8** and the other in **FB 9**. Both **FB8** and **FB9** are aqueous solutions.

(i) Use the Qualitative Analysis Notes to select two reagents that, **used in separate tests**, could identify the presence of the Fe^{2+} ion.

The reagents are *aq. NaOH*
and *aq. NH₃*

(ii) Use your chosen reagents to carry out tests on **both FB 8** and **FB 9**. Record your results in an appropriate form in the space below.

tests	Observations	
	FB8	FB9
To 1cm depth of solution in a test tube, add aq. NaOH little at a time until in excess	green ppt. insoluble in excess	green ppt. turned brown on contact with air. insoluble in excess
To 1cm depth of solution in a test tube, add aq. NH ₃ little at a time until in excess	solution turned blue.	green ppt. turned brown on contact with air. insoluble in excess

(iii) From the results of the tests in (ii), state which solution contains the iron(II) ions.

Fe^{2+} ions are contained in solution *FB9*

Explain how your observations support your conclusion.

..... *FB9 gave green ppt. with aq. NaOH and aq. NH₃.*

(iv) Aqueous EDTA is a reagent used to identify some transition metals. To 1 cm depth of the solution containing the nickel(II) ion, add 1 cm depth of aqueous EDTA.

observation

..... *Green solution turned blue.*

(v) State what you would expect to **see** if acidified potassium manganate(VII) was added to a sample of the solution containing the iron(II) ion.
Do not carry out this experiment.

expected observation *Purple color of aq. KMnO₄ decolorizes*

I	
II	
III	
IV	
V	
VI	

[6]

[Total: 15]

EXPERIMENT NO. 12

For
Examiner's
Use

You are provided with four aqueous solutions, **FA 4**, **FA 5**, **FA 6** and **FA 7**.

Each solution contains one of the following.

- an alcohol
- an aldehyde
- a carboxylic acid
- a ketone

You are to perform the tests below and from the results establish which type of organic compound is contained in each of **FA 4**, **FA 5**, **FA 6** and **FA 7**.

After each test discard the contents of the tubes into the 250 cm³ beaker, labelled organic waste. Rinse and re-use the tubes for the remaining tests.

Record your results in the table. Where no reaction has taken place, write 'no change' in the appropriate box in the table.

test (a)	Place 1 cm depth of each of the solutions FA 4 , FA 5 , FA 6 and FA 7 into separate test-tubes. To each tube add a small quantity of magnesium powder or turnings. Identify any gas given off and record the test you used to make the identification.
test (b)	Place 1 cm depth of each of the solutions FA 4 , FA 5 , FA 6 and FA 7 into separate test-tubes. To each tube add a small quantity of powdered sodium carbonate. Identify any gas given off and record the test you used to make the identification.
test (c)	Place 1 cm depth of each of the solutions FA 4 , FA 5 , FA 6 and FA 7 into separate test-tubes. To each tube add 1 cm depth of 2,4-dinitrophenylhydrazine reagent.
test (d)	Place 1 cm depth of each of the solutions FA 4 , FA 5 , FA 6 and FA 7 into separate test-tubes. Place 2 cm depth of aqueous silver nitrate in a boiling-tube and add to it 1 cm depth of aqueous sodium hydroxide. This will produce a precipitate of silver oxide. Use a dropping pipette to add dilute aqueous ammonia to this mixture until the precipitate of silver oxide just dissolves. Do not add an excess of aqueous ammonia. To each of the tubes containing FA 4 , FA 5 , FA 6 and FA 7 add 1 cm depth of the silver-containing solution you have just prepared.
test (e)	Place 1 cm depth of each of the solutions FA 4 , FA 5 , FA 6 and FA 7 into separate boiling-tubes. To each tube add a few drops of acidified potassium manganate(VII). Warm the tube gently.

test	FA 4	FA 5	FA 6	FA 7
(a)	no change	a colorless gas produced which gave ppt sound with lighted splint.	no change	no change
(b)	no change	a colorless gas produced which gave white ppt with lime water	no change	no change
(c)	Yellow ppt	no change	yellow ppt	no change
(d)	no change	no change	black ppt or silver mirror	no change
(e)	no change	no change	purple color of KMnO_4 disappear	purple color of KMnO_4 disappear

[8]

Identify the type of organic compound present in each of the solutions **FA 4**, **FA 5**, **FA 6** and **FA 7** and complete the table below.

	type of organic compound contained in the solution	confirmed by the observations in test(s)
FA 4	Ketone	c
FA 5	Carboxylic Acid	b
FA 6	Aldehyde	d
FA 7	Alcohol	e

[2]

[Total: 10]

EXPERIMENT NO. 13

Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

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

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

- (a) **FA 5, FA 6, FA 7** and **FA 8** are aqueous solutions of organic compounds. All of **FA 5, FA 6, FA 7** and **FA 8** contain carbon, hydrogen and oxygen only.

Half fill the 250 cm³ beaker with water and heat it to about 80 °C. Turn off the Bunsen burner. This will be used as a water bath.

To a 2 cm depth of aqueous silver nitrate in a boiling tube add 2 drops of aqueous sodium hydroxide and then add ammonia dropwise until the brown solid just disappears. This solution is Tollens' reagent and is needed in a test in (i).

- (i) Carry out the following tests on **FA 5**, **FA 6**, **FA 7** and **FA 8** and record your observations in the table.

test	observations			
	FA 5	FA 6	FA 7	FA 8
To a 1 cm depth in a test-tube, add a small spatula measure of sodium carbonate.	effervescence of a colorless gas which gave white ppt. with lime water	effervescence of a colorless gas which gave white ppt. with lime water	no change	no change
To a few drops in a test-tube, add a 1 cm depth of Tollens' reagent. Place the tube in the water bath and leave to stand. When you have completed this test rinse all tubes used.	black ppt. or silver mirror formed	no reaction	no reaction	black ppt. or silver mirror formed
To a 1 cm depth in a test-tube, add a few drops of acidified potassium manganate(VII). Place the tube in the water bath and leave to stand.	Purple solution of KMnO_4 turned colorless	no change	Purple solution of KMnO_4 turned colorless	Purple solution of KMnO_4 turned colorless

- (ii) Using your observations from the table, what functional group is present in both **FA 5** and **FA 6**?

Carboxylic acid ($-\text{CO}_2\text{H}$)

- (iii) Using your observations from the table, what functional group is present in both **FA 5** and **FA 8**?

aldehyde ($-\text{CHO}$)

- (iv) What **type** of reaction is occurring in the potassium manganate(VII) test?

redox reaction

- (v) Using your observations from the table, what functional group is present in **FA 7**?

1°/2° alcohol or alkene

- (vi) Suggest a test that would confirm the presence of the functional group in a pure sample of FA 7. Include the result you would expect the test to give.

Do not carry out this test.

* add Na metal \rightarrow H₂ gas will produced (for alcohol)
OR

* add Br₂ water \rightarrow solution will turn brown to colorless (for alkene)

[9]

- (b) FA 9 and FA 10 are solids that each contain one anion from those listed in the Qualitative Analysis Notes.

- (i) Carry out the following tests on FA 9 and FA 10 and record your observations in the table.

test	observations	
	FA 9	FA 10
To a spatula measure of solid in a boiling tube, add a 1 cm depth of aqueous sodium hydroxide. Warm, then,	solid dissolved, gave a white ppt.	solid dissolves formed a colorless solution
add a small piece of aluminium foil.	a colorless gas produced which turned damp red litmus paper blue.	bubbles of a colorless gas which turned damp red litmus paper blue
Place a spatula measure of solid in a hard-glass test-tube. Heat gently at first and then more strongly.	Solid melts. Brown gas produced. a pale yellow solid formed on cooling.	Solid melted to form a yellow liquid. Bubbles of a gas

- (ii) Using your observations from the table, which **two** anions could be present in FA 9 and FA 10?

anion NO₃⁻ or NO₂⁻

- (iii) Suggest a test that would allow you to decide which of the anions is present. State what observations you would expect.

..... Add dilute HCl, a brown gas will be produce from
..... NO₂⁻ ion.

- (iv) Carry out this test on FA 9 and FA 10 to decide which anion is present in each.

observation for FA 9 no change anion in FA 9 is NO₃⁻

observation for FA 10 no change anion in FA 10 is NO₃⁻

[7]

[Total: 16]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	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 heating	–
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	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	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

<i>ion</i>	<i>reaction with</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless NO \rightarrow (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids)

3 Tests for gases

<i>gas</i>	<i>test and test result</i>
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	"pops" with a lighted splint
oxygen, O_2	relights a glowing splint
sulfur dioxide, SO_2	turns acidified aqueous potassium manganate(VII) from purple to colourless

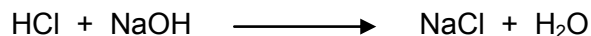
QUANTITATIVE ANALYSIS

TITRATION

EXPERIMENT NO. 14

INTRODUCTION TO TITRATION TECHNIQUES

An acid neutralizes a base to form a salt and water. Hydrochloric acid and sodium hydroxide are completely ionized in water. We say they are a strong acid or base because they are completely ionized in solution. The ions present in hydrochloric acid are $\text{H}^+(\text{aq})$ and $\text{Cl}^-(\text{aq})$ and in sodium hydroxide are $\text{Na}^+(\text{aq})$ and $\text{OH}^-(\text{aq})$.



You are going to use the technique of **titration** to produce a sodium chloride solution. Titration is a very accurate way of investigating the reaction of two solutions. It can be used to analyze the amount of a particular substance in a solution. This is known as **quantitative analysis**. In a titration, one solution is placed in a burette and the other is placed in a conical flask using a pipette. The solution in the burette is then run into the conical flask until there is a complete reaction. In this case you will completely neutralize a solution of sodium hydroxide with hydrochloric acid solution. You will use an indicator to tell you when there is complete neutralization. The indicator changes color at the exact point of neutralization. In this case you may use any acid-base indicator because you will be titrating a strong acid with a strong base.

Method

1. Wash the burette with distilled water (aka deionised water) and then rinse with a little of the hydrochloric acid.
2. Once the burette has been washed and rinsed out with the acid solution, fill it nearly to the top. Clamp the burette carefully and run a little acid through into the beaker until the tip becomes full. (Fill the burette with hydrochloric acid solution and ensure the tip is full.)
3. The pipette can be cleaned in a similar way to the burette, remembering to finish by washing it out with a little of the alkali solution. (A pipette safety filler is used to draw a measured volume of sodium hydroxide solution from the beaker into the pipette.)
4. Rinse the conical flask with some deionised water. In this case it does not matter if there is some water left in the flask after rinsing it.
5. Pipette exactly 25.0 cm^3 of the $0.100 \text{ mol dm}^{-3}$ sodium hydroxide solution into a clean conical flask. Now add two or three drops of acid-base indicator.
6. Now read the burette and record the reading in the middle row of a table like the one below. Be careful that your eye is level with the bottom of the meniscus or your reading will not be accurate.
7. Place the conical flask below the burette on a white tile. Run acid into the flask fairly quickly, shaking it all the time. As soon as the color of the indicator changes, close the tap and note the final burette reading. Record this result in your table above your initial reading. Subtract the initial reading from the final reading to give you the volume of acid added.
8. The first titration is a rough titration to give you an idea of the volume you need to add to exactly neutralise the acid. It is quite likely that you added a slight excess of acid as you were doing the titration quickly. Now repeat steps 2 to 7 but this time run in the acid quickly until you reach about 1 cm^3 less than the volume you added in the rough titration. Swirl the contents of the flask and add one drop of acid at a time from the burette until the indicator just changes colour. Record this volume. This should represent the exact volume you need to add to neutralise 25.0 cm^3 of $0.100 \text{ mol dm}^{-3}$ of sodium hydroxide.
9. To ensure that you have a reliable volume of alkali, you should repeat the whole titration again until you get two readings that agree within 0.10 cm^3 .

	Rough	1	2	3	4
Final burette reading / cm ³	25.80	25.40	36.30	33.80	
Initial burette reading / cm ³	0.00	0.00	11.00	8.50	
Volume of HCl added / cm ³	25.80	25.40	25.30	25.30	

$$\frac{25.30 + 25.30}{2} = 25.30 \text{ cm}^3$$

Questions

(a) How many moles of NaOH were present in 25.0 cm³ solution?

$$C = 0.100 \text{ mol dm}^{-3}$$

$$n = CV$$

$$V = 0.0250 \text{ dm}^3$$

$$= 0.100 \times 0.0250 =$$

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

(b) How many moles of HCl were present in the volume of acid you used to neutralise the NaOH solution?

mole ratio

HCl : NaOH

1 : 1

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

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

(c) What was the exact concentration of hydrochloric acid in mol dm⁻³?

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

$$C = \frac{n}{V} = \frac{2.50 \times 10^{-3}}{0.0253}$$

$$V = 0.0253 \text{ dm}^3$$

$$0.0988 \text{ mol dm}^{-3}$$

(d) Why was the conical flask placed on a piece of white tile?

To make the color change visible.

(e) Why were the pipette and burette washed with the solutions they were going to contain?

To prevent dilution of the chemicals they are going to contain.

(f) Why was the conical flask not washed with the alkali solution it was going to contain?

So to not add extra moles of alkali.

(g) Explain why it does not matter if there is water already in the flask.

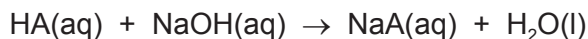
As the water will not change the number of moles of alkali.

(h) Explain why a conical flask was used and not a beaker.

To prevent from spillage.

EXPERIMENT NO. 15

HA is an organic acid. Solution **FA 1** was prepared by dissolving 13.1 g of solid HA in each dm³ of solution. You are to determine the relative molecular mass, M_r , of HA by titration with aqueous sodium hydroxide. The equation for the reaction between HA and sodium hydroxide is given below.



FA 1 is a solution containing 13.1 g dm⁻³ of organic acid, HA.

FA 2 is 0.100 mol dm⁻³ sodium hydroxide, NaOH.

bromothymol blue indicator

(a) Method

- Fill the burette with **FA 1**.
- Use the pipette to transfer 25.0 cm³ of **FA 2** into a conical flask.
- Add a few drops of bromothymol blue indicator. This indicator is blue in alkali and yellow in acid solutions.
- Perform a **rough titration** and record your burette readings in the space below.

Final burette reading/cm ³	25.60
Initial burette reading/cm ³	0.10
Volume of FA1 used/cm ³	25.50

The rough titre is25.50..... cm³.

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain 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 1** added in each accurate titration.

	1	2	3
Final burette reading/cm ³	25.30	35.40	31.20
Initial burette reading/cm ³	0.10	10.00	6.10
Volume of FA1 used/cm ³	25.20	25.40	25.10

I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b) From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you obtained this value.

$$\frac{25.20 + 25.10}{2} = 25.15 \text{ cm}^3$$

25.0 cm³ of **FA 2** required25.15..... cm³ of **FA 1**. [1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of sodium hydroxide present in the volume of **FA 2** pipetted into the conical flask.

$$C = 0.100 \text{ mol dm}^{-3}$$
$$V = 0.0250 \text{ cm}^3$$
$$n = CV$$
$$= 0.100 \times 0.0250$$

moles of NaOH = 2.50×10^{-3} mol

- (ii) Use your answer to (c)(i) and the equation on page 2 to determine the number of moles of organic acid, HA, used to neutralise 25.0 cm³ of **FA 2**.

mole ratio

HA	:	NaOH
1	:	1
x	:	2.50×10^{-3}

moles of HA = 2.50×10^{-3} mol

- (iii) Use your answers to (b) and (c)(ii) to calculate the number of moles of HA in 1 dm³ of FA 1.

$$C = \frac{n}{V} = \frac{2.50 \times 10^{-3}}{0.0250}$$

= Concentration

moles of HA in 1 dm³ of **FA 1** = 0.100 mol

- (iv) Calculate the relative molecular mass, M_r , of the organic acid, HA.

$$n = \frac{m}{M_r}$$
$$M_r = \frac{g \cdot \text{dm}^{-3}}{\text{mol} \cdot \text{dm}^{-3}} = \frac{13.1}{0.100} = 131$$
$$\text{mol} \cdot \text{dm}^{-3} = \frac{g \cdot \text{dm}^{-3}}{M_r}$$

M_r of HA = 131 [4]

- (d) A student carrying out this method correctly with the same concentration of reactants obtained a titre of 28.30 cm³. Would this give a larger or smaller value of M_r than yours?

Explain your answer.

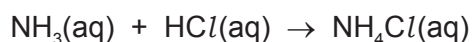
This would give larger value of M_r . As $C \propto \frac{1}{V}$ and $M_r \propto \frac{1}{C}$.

[1]

[Total: 13]

EXPERIMENT NO. 16

The concentration of aqueous ammonia used in qualitative analysis is 2 mol dm^{-3} but it is supplied in a much more concentrated form. This is referred to as '.880 ammonia'. You are to determine the concentration of '.880 ammonia' by titration of a solution of ammonia, **FB 1**, with hydrochloric acid of known concentration. The equation for the reaction is given below.



FB 1 is a dilute solution of ammonia, $\text{NH}_3(\text{aq})$. It was prepared by measuring out 5.91 cm^3 of the '.880 ammonia' and then adding distilled water until the solution had a volume of 1 dm^3 .

FB 2 is $0.100 \text{ mol dm}^{-3}$ hydrochloric acid, $\text{HCl}(\text{aq})$.

methyl orange indicator

(a) Method

- Fill the burette with **FB 2**.
- Use the pipette to transfer 25.0 cm^3 of **FB 1** into a conical flask.
- Add a few drops of methyl orange indicator.
- Perform a **rough titration** and record your burette readings in the space below.

Final burette reading/ cm^3	24.90
Initial burette reading/ cm^3	0.00
Volume of FB 2 added/ cm^3	24.90

The rough titre is24.90..... cm^3 .

- Carry out as many accurate titrations as you think necessary to achieve consistent results.
- Make certain 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 **FB 2** added in each accurate titration.

	1	2	3	4
Final burette reading/ cm^3	25.30	49.00	32.20	
Initial burette reading/ cm^3	0.70	24.60	7.80	
Volume of FB 2 used/ cm^3	24.60	24.40	24.40	

✓ ✓

I	
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[7]

- (b) From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you obtained this value.

$$\frac{24.40 + 24.40}{2} = 24.40$$

25.0 cm^3 of **FB 1** required24.40..... cm^3 of **FB 2**. [1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of hydrochloric acid present in the volume of **FB 2** calculated in (b).

$$C = 0.100 \text{ mol dm}^{-3}$$

$$V = 0.0244 \text{ dm}^3$$

$$n = CV$$

$$= 0.100 \times 0.0244$$

$$\text{moles of HCl} = 2.44 \times 10^{-3} \text{ mol}$$

- (ii) Use your answer to (i) to determine the number of moles of ammonia present in 25.0 cm³ of **FB 1**, pipetted into the conical flask.

$$\text{mole ratio } \text{NH}_3 : \text{HCl}$$

$$1 : 1$$
$$x : 2.44 \times 10^{-3}$$

$$\text{moles of NH}_3 = 2.44 \times 10^{-3} \text{ mol}$$

- (iii) Use your answer to (ii) to calculate the concentration, in mol dm⁻³, of the diluted ammonia, **FB 1**.

$$n = 2.44 \times 10^{-3} \text{ mol}$$

$$V = 0.0250 \text{ dm}^3$$

$$C = \frac{n}{V} = \frac{2.44 \times 10^{-3}}{0.0250} = 0.0976 \text{ mol dm}^{-3}$$

$$\text{concentration of NH}_3 \text{ (diluted) in FB 1} = 0.0976 \text{ mol dm}^{-3}$$

- (iv) Use your answer to (iii) and the information on page 2 to calculate the concentration, in mol dm⁻³, of '.880 ammonia'. * FB1 is dilute solution of '.880 ammonia'.

$$C_1 V_1 = C_2 V_2$$

$$C_1 \times \frac{5.91}{1000} = 0.0976 \times 1$$

$$C_1 = 16.5 \text{ mol dm}^{-3}$$

$$\text{concentration of '.880 ammonia'} = 16.5 \text{ mol dm}^{-3}$$

[3]

- (d) A student analysed a different sample of concentrated ammonia and determined the concentration to be 15.0 mol dm⁻³. Calculate the percentage difference in concentration of the '.880 ammonia' you have determined compared with that of the student.

(If you have been unable to complete the calculation, assume the concentration of '.880 ammonia' was 9.35 mol dm⁻³. This is not the correct value.)

$$\frac{16.5 - 15.0}{15.0} \times 100$$

$$\text{percentage difference in concentration} = 10.0 \% \text{ [1]}$$

[Total: 12]

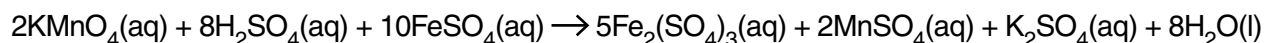
EXPERIMENT NO. 17

For
Examiner's
Use

FB 1 is an aqueous solution containing 21.50 g dm^{-3} of a mixture of iron(II) sulfate, FeSO_4 and iron(III) sulfate, $\text{Fe}_2(\text{SO}_4)_3$.

FB 2 is an aqueous solution containing 2.00 g dm^{-3} potassium manganate(VII), KMnO_4 .

In the presence of acid, the iron(II) sulfate is oxidised by potassium manganate(VII).



(a) Method

- Fill a burette with **FB 2**.
- Pipette 25.0 cm^3 of **FB 1** into the conical flask.
- Use a 25 cm^3 measuring cylinder to add 10 cm^3 of dilute sulfuric acid to the flask.
- Place the flask on a white tile.
- Carefully titrate with **FB 2** until the first permanent pink colour is obtained.

You should perform a **rough titration**.

In the space below record your burette readings for this rough titration.

Final burette reading/ cm^3	26.20
Initial burette reading/ cm^3	0.00
Volume of FB2 used/ cm^3	26.20

The rough titre is26.20..... cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Record in a suitable form below all of your burette readings and the volume of **FB 2** added in each accurate titration.
- Make certain any recorded results show the precision of your practical work.

	1	2	3	4	5
Final burette reading/ cm^3	26.40	41.00	31.40	36.10	
Initial burette reading/ cm^3	0.50	15.00	5.20	10.10	
Volume of FB2 used/ cm^3	25.90	26.00	26.20	26.00	



[7]

I	
II	
III	
IV	
V	
VI	
VII	

- (b) From your accurate titration results obtain a suitable value to be used in your calculation. Show clearly how you have obtained this value.

$$\frac{26.00 + 26.00}{2} = 26.00$$

25.0 cm³ of **FB 1** required 26.00 cm³ of **FB 2**. [1]

Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (c) (i) Calculate the concentration, in mol dm⁻³, of the potassium manganate(VII) in **FB 2**.

FB 2 contains 2.00 g dm⁻³ KMnO₄.
[A_r: O, 16.0; K, 39.1; Mn, 54.9]

$$n = \frac{m}{M_r}$$

$$\text{mol dm}^{-3} = \frac{\text{g dm}^{-3}}{M_r}$$

$$\text{mol dm}^{-3} = \frac{2.00}{158} = 0.0127 \text{ mol dm}^{-3}$$

The concentration of potassium manganate(VII) in **FB 2** is 0.0127 mol dm⁻³.

- (ii) Calculate how many moles of KMnO₄ were present in the volume calculated in (b).

$$C = 0.0127 \text{ mol dm}^{-3}$$

$$V = 0.0260 \text{ dm}^3$$

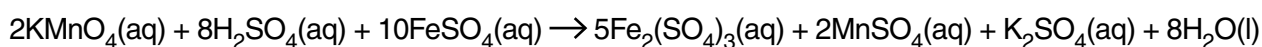
$$n = CV$$

$$= 0.0127 \times 0.0260$$

$$= 3.30 \times 10^{-4}$$

3.30 × 10⁻⁴ mol of KMnO₄.

- (iii) Calculate how many moles of iron(II) sulfate, FeSO₄, reacted with the potassium manganate(VII) in (ii).



$$\begin{array}{ccc} 2 & : & 10 \\ 1 & : & 5 \\ 3.30 \times 10^{-4} & : & x \end{array}$$

1.65 × 10⁻³ mol of FeSO₄ reacted with the potassium manganate(VII).

I	
II	
III	
IV	
V	

- (iv) Calculate the concentration, in mol dm^{-3} of FeSO_4 in **FB 1**.

$$n = 1.65 \times 10^{-3}$$

$$V = 0.0250 \text{ dm}^3$$

$$C = \frac{n}{V} = \frac{1.65 \times 10^{-3}}{0.0250} = 0.0660 \text{ mol dm}^{-3}$$

The concentration of FeSO_4 in **FB 1** is 0.0660 mol dm^{-3} .

- (v) Calculate the concentration, in g dm^{-3} , of FeSO_4 in **FB 1**.

[A_r : O, 16.0; S, 32.1; Fe, 55.8]

$$\text{mol dm}^{-3} = \frac{\text{g dm}^{-3}}{M_r}$$

$$\text{g dm}^{-3} = \text{mol dm}^{-3} \times M_r$$

$$= 0.0660 \times 151.9 = 10.0254$$

FB 1 contains 10.0 g dm^{-3} of FeSO_4 .

- (vi) **FB 1** is an aqueous solution containing 21.50 g dm^{-3} of FeSO_4 and $\text{Fe}_2(\text{SO}_4)_3$. Calculate the percentage, by mass, of FeSO_4 in this mixture.

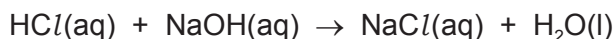
$$\frac{10.0}{21.50} \times 100 = 46.5\%$$

The mixture contains 46.5 % FeSO_4 .
[5]

[Total: 13]

EXPERIMENT NO. 18

You will determine the concentration of a solution of hydrochloric acid by diluting it and then titrating the diluted solution against an alkali.



FA 1 was made by dissolving 1.06 g of sodium hydroxide, NaOH, in distilled water to make 250 cm³ of solution.

FA 2 is hydrochloric acid, HCl.
methyl orange indicator

(a) Method

- Pipette 25.0 cm³ of **FA 2** into the 250 cm³ volumetric flask.
- Add distilled water to make 250 cm³ of solution and shake the flask thoroughly. Label this solution **FA 3**.
- Fill the burette with **FA 3**.
- Use the second pipette to transfer 25.0 cm³ of **FA 1** into a conical flask.
- Add about 5 drops of methyl orange.
- Perform a rough titration and record your burette readings in the space below. The end point is reached when the solution becomes a permanent pink colour.

Final burette reading/cm ³	26.20
Initial burette reading/cm ³	1.00
Volume of FA3 used/cm ³	25.20

The rough titre is 25.20 cm³.

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain 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 3** added in each accurate titration.

	1	2	3	4
Final burette reading/cm ³	33.40	49.60	25.00	49.70
Initial burette reading/cm ³	8.50	24.90	0.00	25.00
Volume of FA3 used/cm ³	24.90	24.70	25.00	24.70

I	
II	
III	
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VI	
VII	

✓ ✓

[7]

(b) From your accurate titration results, obtain a suitable value for the volume of **FA 3** to be used in your calculations. Show clearly how you obtained this value.

$$\frac{24.70 + 24.70}{2} =$$

25.0 cm³ of **FA 1** required 24.70 cm³ of **FA 3**. [1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

(i) Calculate the concentration, in mol dm^{-3} , of sodium hydroxide in **FA 1**.

Use the data in the Periodic Table on page 12.

$$\begin{aligned} M_r \text{ of NaOH} &= 40 \\ \text{mass} &= 1.06 \text{ g} / 250 \text{ cm}^3 \\ &= 4.24 \text{ g dm}^{-3} \end{aligned}$$
$$\text{mol dm}^{-3} = \frac{\text{g dm}^{-3}}{M_r} = \frac{4.24}{40} = 0.106$$

concentration of NaOH in **FA 1** = 0.106 mol dm^{-3}

(ii) Calculate the number of moles of sodium hydroxide present in 25.0 cm^3 of **FA 1**.

$$\begin{aligned} C &= 0.106 \text{ mol dm}^{-3} & n &= C V \\ V &= 0.0250 \text{ dm}^3 & &= 0.106 \times 0.0250 \\ & & &= 2.65 \times 10^{-3} \end{aligned}$$

moles of NaOH = 2.65×10^{-3} mol

(iii) Deduce the number of moles of hydrochloric acid present in the volume of **FA 3** you have calculated in (b).

$$\begin{aligned} \text{mole ratio} & \quad \text{HCl} : \text{NaOH} \\ & \quad 1 : 1 \\ x & : 2.65 \times 10^{-3} \\ & = 2.65 \times 10^{-3} \text{ mol} \end{aligned}$$

moles of HCl = 2.65×10^{-3} mol

(iv) Calculate the concentration, in mol dm^{-3} , of hydrochloric acid in **FA 2**.

$$\begin{aligned} n &= 2.65 \times 10^{-3} \\ V &= 0.0247 \text{ dm}^3 \\ C &= \frac{n}{V} = \frac{2.65 \times 10^{-3}}{0.0247} = 0.107 \text{ mol dm}^{-3} \end{aligned}$$

concentration of HCl in **FA 2** = 0.107 mol dm^{-3}

[5]

[Total: 13]

EXPERIMENT NO. 19

Borax is an alkali which has many uses. In this experiment you will determine x in the chemical formula of borax, $\text{Na}_2\text{B}_x\text{O}_7 \cdot 10\text{H}_2\text{O}$, by titration with hydrochloric acid.

FB 1 is a solution containing 15.5 g dm^{-3} of borax, $\text{Na}_2\text{B}_x\text{O}_7 \cdot 10\text{H}_2\text{O}$.

FB 2 is 2.00 mol dm^{-3} hydrochloric acid, HCl .

methyl orange indicator

(a) Method

Dilution of FB 2

- Pipette 10.0 cm^3 of **FB 2** into the 250 cm^3 volumetric flask.
- Make the solution up to 250 cm^3 using distilled water.
- Shake the solution in the volumetric flask thoroughly.
- This diluted solution of hydrochloric acid is **FB 3**. Label the volumetric flask **FB 3**.

Titration

- Fill the burette with **FB 3**.
- Pipette 25.0 cm^3 of **FB 1** into a conical flask.
- Add several drops of methyl orange.
- Perform a rough titration and record your burette readings in the space below.

Final burette reading/ cm^3	24.90
Initial burette reading/ cm^3	0.00
Volume of FB3 used/ cm^3	24.90

The rough titre is24.90..... 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 **FB 3** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	

	1	2	3	4
Final burette reading/ cm^3	49.40	24.60	49.30	
Initial burette reading/ cm^3	24.90	0.00	24.60	
Volume of FB3 used/ cm^3	24.50	24.60	24.70	

[7]

- (b) From your accurate titration results, obtain a suitable value for the volume of **FB 3** to be used in your calculations.

Show clearly how you obtained this value.

$$\frac{24.50 + 24.60}{2}$$

25.0 cm^3 of **FB 1** required24.55..... cm^3 of **FB 3**. [1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

(i) Calculate the number of moles of hydrochloric acid present in the volume of **FB 3** calculated in (b). *FB3 is a dilute solution of FB2.*

FB2 *FB3*
 $C_1 V_1 = C_2 V_2$
 $2.00 \times \frac{10.0}{1000} = C_2 \times \frac{250.0}{1000}$
 $C_2 = 0.0800 \text{ mol dm}^{-3}$
FB3

$C = 0.0800 \text{ mol dm}^{-3}$
 $V = 0.02455 \text{ dm}^3$

$n = C V$
 $= 0.0800 \times 0.02455$

moles of HCl = 1.96×10^{-3} mol

(ii) 1 mole of borax is neutralised by 2 moles of hydrochloric acid. Calculate the number of moles of borax that react with the hydrochloric acid in (i).

mole ratio borax : HCl
 1 : 2
 $x : 1.96 \times 10^{-3}$

moles of borax = 9.80×10^{-4} mol

(iii) Use your answer to (ii) to calculate the number of moles of borax in 1.00 dm³ of **FB 1**.

$n = 9.80 \times 10^{-4} \text{ mol}$
 $V = 0.0250 \text{ dm}^3$

$C = \frac{n}{V} = \frac{9.80 \times 10^{-4}}{0.0250} = 0.0392 \text{ mol dm}^{-3}$
 = Concentration

moles of borax in 1.00 dm³ **FB 1** = 0.0392 mol

(iv) Use your answer to (iii) and the information on page 2 to calculate the relative formula mass, M_r , of borax.

$\text{mol dm}^{-3} = 0.0392$
 $\text{g dm}^{-3} = 15.5$
 $M_r = ?$

$\text{mol dm}^{-3} = \frac{\text{g dm}^{-3}}{M_r}$
 $M_r = \frac{\text{g dm}^{-3}}{\text{mol dm}^{-3}} = \frac{15.5}{0.0392} = 395.41$

M_r of borax = 395

(v) Calculate x in the formula of borax, $\text{Na}_2\text{B}_x\text{O}_7 \cdot 10\text{H}_2\text{O}$. Use data from the Periodic Table.

$\text{Na} = 23 \times 2 = 46$
 $\text{O} = 16 \times 7 = 112$
 $\text{H}_2\text{O} = 18 \times 10 = 180$
338

$395 - 338 = 57$

$\frac{57}{10.8} = 5.28 \approx 5$

A_r of B = 10.8

$x = 5$
 [5]

[Total: 13]

EXPERIMENT NO. 20

For
Examiner's
Use

You are required to determine the concentration in g dm^{-3} of hydrated ammonium iron(II) sulphate, $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$, in the solution **FB 1**.

FB 1 contains hydrated ammonium iron(II) sulphate.

FB 2 is $0.0120 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

(a) Dilution of **FB 1**

By using a burette measure between 36.00 cm^3 and 37.00 cm^3 of **FB 1** into the 250 cm^3 graduated flask labelled **FB 3**.

Record your burette readings and the volume of **FB 1** added to the flask in the space below.

Final burette reading/ cm^3	36.50
Initial burette reading/ cm^3	0.00
Volume of FB 1 added/ cm^3	36.50

Make up the contents of the flask to the 250 cm^3 mark with distilled water. Place the stopper in the flask and mix the contents thoroughly by slowly inverting the flask a number of times.

Titration

Fill a second burette with **FB 2**.

Pipette 25.0 cm^3 of **FB 3** into a conical flask. Use a measuring cylinder to add approximately 10 cm^3 of 1.0 mol dm^{-3} sulphuric acid, H_2SO_4 , and titrate with **FB 2** until the first permanent pink colour remains in the solution.

Perform one rough (trial) titration and sufficient further titrations to obtain accurate results.

Record your titration results in the space below. Make certain that your recorded results show the precision of your working.

	Rough	1	2	3
Final burette reading/ cm^3	24.30	48.40	24.20	48.30
Initial burette reading/ cm^3	0.00	24.30	0.00	24.20
Volume of FB 2 used/ cm^3	24.30	24.10	24.20	24.10

✓

✓

i	
ii	
iii	
iv	
v	
vi	

[6]

- (b) From your titration results obtain a suitable volume of **FB 2** to be used in your calculations.
Show clearly how you obtained this volume.

$$\frac{24.10 + 24.10}{2} = 24.10 \text{ cm}^3$$

[1]

Calculations

Show your working and appropriate significant figures in all of your calculations.

- (c) Calculate how many moles of KMnO_4 were run from the burette during the titration.

$$C = 0.0120 \text{ mol dm}^{-3}$$

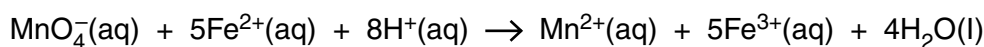
$$V = 0.0241 \text{ dm}^3$$

$$n = CV$$

$$= 0.0120 \times 0.0241$$

2.89×10^{-4} mol of KMnO_4 were run from the burette.

Calculate how many moles of Fe^{2+} ions reacted with the KMnO_4 run from the burette.



$$2.89 \times 10^{-4} : x$$

1.45×10^{-3} mol of Fe^{2+} reacted with the KMnO_4 run from the burette.

Calculate the concentration, in mol dm^{-3} , of Fe^{2+} in **FB 3**.

$$n = 1.45 \times 10^{-3}$$

$$V = 0.0250 \text{ dm}^3$$

$$C = \frac{n}{V} = \frac{1.45 \times 10^{-3}}{0.0250}$$

Concentration of Fe^{2+} in **FB 3** = 0.0580 mol dm^{-3} .

Calculate the concentration, in mol dm⁻³, of Fe²⁺ in **FB 1**.

$$C_1^{FB1} V_1 = C_2^{FB2} V_2$$

$$C_1 \times \frac{36.5}{1000} = 0.0580 \times \frac{250}{1000}$$

$$C_1 = 0.397 \text{ mol dm}^{-3}$$

Concentration of Fe²⁺ in **FB 1** = 0.397 mol dm⁻³.

Calculate, to **4 significant figures**, the concentration of (NH₄)₂SO₄·FeSO₄·6H₂O in **FB 1** in g dm⁻³.

[A_r: Fe, 55.8; H, 1.0; N, 14.0; O, 16.0; S, 32.1]

$$M_r \text{ of } (\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O} = 399$$

$$\text{mol} \cdot \text{dm}^{-3} = \frac{\text{g} \cdot \text{dm}^{-3}}{M_r}$$

$$\text{g} \cdot \text{dm}^{-3} = \text{mol} \cdot \text{dm}^{-3} \times M_r$$

$$= 0.397 \times 399 = 155.6$$

FB 1 contains 155.6 g dm⁻³ of (NH₄)₂SO₄·FeSO₄·6H₂O. [5]

i	
ii	
iii	
iv	
v	

- (d) A student learns that the solution of the iron(II) salt has been prepared by dissolving the solid in distilled water that has absorbed air from the laboratory. Suggest a way in which the distilled water can be prepared and stored in the laboratory to ensure that it contains a minimum of dissolved air.

Heat the distilled water and store in a closed container. As the solubility of gas decreases as temperature increases.

..... [1]

- (e) Estimate the error in reading a volume from a burette.

smallest division on burette scale = 0.1 cm³

$$\text{error} = \frac{\text{Least Count}}{2}$$

estimated error in reading a volume = ± 0.05 cm³

[1]

- (f) A titre value is obtained by the difference between final and initial burette readings.

What is the **maximum** possible error in obtaining a titre reading?

estimated **maximum** error in the titre = ± 0.1 cm³

[1]

- (g) During one titration a student reads the burette twice.

Each reading has an error but the titre has no error. Explain how this can happen.

Error in same direction so cancel out.

He may recorded initial reading 0.05 cm³ greater than actual reading, and also recorded final burette reading 0.05 cm³ greater. [1]

[Total: 16]

EXPERIMENT NO. 21

In this experiment you will determine the ionic equation for the reaction of acidified potassium manganate(VII) with potassium iodide. Excess potassium iodide is used and the reaction produces iodine. The amount of iodine produced is measured by titration with sodium thiosulfate.

FA 1 is $0.0180 \text{ mol dm}^{-3}$ potassium manganate(VII), KMnO_4 .

FA 2 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4 .

FA 3 is $0.500 \text{ mol dm}^{-3}$ potassium iodide, KI .

FA 4 is $0.100 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.
starch indicator

(a) Method

- Pipette 25.0 cm^3 of **FA 1** into a conical flask.
- Use the measuring cylinder to add 25 cm^3 of **FA 2** to the conical flask.
- Use the measuring cylinder to add 20 cm^3 of **FA 3** to the conical flask.
- Fill the burette with **FA 4**.
- Carry out a rough titration. When the colour of the mixture becomes yellow/orange, add a few drops of starch indicator. Then titrate until the mixture goes colourless.
- Record all your burette readings in the space below.

Final burette reading/ cm^3	25.50
Initial burette reading/ cm^3	0.00
Volume of FA4 used/ cm^3	25.50

The rough titre is 25.50 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 4** added in each accurate titration.

	1	2	3
Final burette reading/ cm^3	25.20	35.60	40.70
Initial burette reading/ cm^3	0.00	10.30	15.50
Volume of FA4 used/ cm^3	25.20	25.30	25.20



I	
II	
III	
IV	
V	
VI	
VII	

[7]

- (b) From your accurate titration results, obtain a suitable value for the volume of **FA 4** to be used in your calculations.

Show clearly how you have obtained this value.

$$\frac{25.20 + 25.20}{2} :$$

Volume of **FA 4** required is 25.20 cm^3 . [1]

(vi) Prove that the iodide ion has been oxidised in the equation that you selected in (v).

- * An iodide ion loses an electron.....
- * Oxidation number of Iodine increases (-1 to 0).....
- * $2I^- \longrightarrow I_2 + 2e^-$

[5]

(d) (i) The error in calibration of the pipette you used is $\pm 0.06 \text{ cm}^3$.
Calculate the percentage error when measuring **FA 1**, using the pipette.

$$\begin{aligned} \% \text{ error} &= \frac{\text{error}}{\text{vol. measured}} \times 100 \\ &= \frac{0.06}{25.0} \times 100 = 0.240\% \end{aligned}$$

percentage error = 0.240 %

(ii) A student suggested that the experiment would be more accurate if a pipette was used to measure solution **FA 3**.

State and explain whether you agree with the student.

Not agree.....

As potassium iodide is already in excess.....

[2]

[Total: 15]

EXPERIMENT NO. 22

FA 1 is an iron salt in which all the iron is present as Fe^{2+} cations. You will work out the percentage of iron in this salt by titrating a solution of this salt with a standard solution aqueous potassium manganate(VII).

For
Examiner's
Use

FA 1 is an unknown iron(II) salt.

FA 2 is 1.00 mol dm^{-3} sulfuric acid.

FA 3 is $0.0100 \text{ mol dm}^{-3}$ potassium manganate(VII).

(a) Method

Weighing out the salt

- Weigh the tube containing **FA 1**.
- Tip the contents of the tube into a 250 cm^3 beaker.
- Re-weigh the empty tube.
- Record all your readings in a suitable form in the space below.

mass of tube + FA1 /g	35.05
mass of tube + residue/g	29.20
mass of FA1 /g	5.85

Preparing the solution

- To the salt in the beaker use a measuring cylinder to add approximately 150 cm^3 of **FA 2** and stir until the salt has dissolved.
- Pour the contents of the beaker carefully into the 250 cm^3 graduated (volumetric) flask using the small funnel.
- Rinse the contents of the beaker twice with a little distilled water and add these washings to the graduated flask.
- Fill the graduated flask to the line with distilled water. Shake carefully to ensure adequate mixing.

Titration

- Fill the burette with **FA 3**.
- Pipette 25.0 cm^3 of the solution of **FA 1** from the graduated flask into a conical flask.
- Titrate the solution of **FA 1** in the flask with **FA 3** until the first appearance of a permanent pink colour.

You should perform a **rough titration**.

In the space below record your burette readings for this rough titration.

Final burette reading/ cm^3	25.30
Initial burette reading/ cm^3	0.00
Volume of FA3 used/ cm^3	25.30

The rough titre is 25.30 cm^3 .

- Carry out as many accurate titrations as you think are necessary to obtain consistent results.
- Make certain any recorded results show the precision of your practical work.
- Record in an appropriate form below all of your burette readings and the volume of **FA 3** added in each accurate titration.

	1	2	3	4
Final burette reading/cm ³	24.70	49.50	24.80	
Initial burette reading/cm ³	0.00	24.70	0.00	
Volume of FA3 used/cm ³	24.70	24.80	24.80	

✓ ✓

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[7]

- (b) From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

$$\frac{24.80 + 24.80}{2}$$

25.0 cm³ of the solution of **FA 1** required 24.80 cm³ of **FA 3**.
[2]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

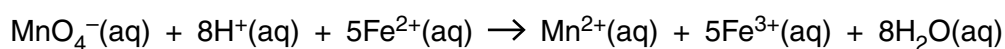
- (i) Calculate how many moles of MnO_4^- (aq) were present in the volume of **FA 3** calculated in (b).

$$C = 0.0100 \text{ mol dm}^{-3} \quad n = CV$$

$$V = 0.0248 \text{ dm}^3 \quad = 0.0100 \times 0.0248 = \boxed{2.48 \times 10^{-4} \text{ mol}}$$

$$\text{moles of } \text{MnO}_4^-(\text{aq}) = 2.48 \times 10^{-4} \text{ mol}$$

- (ii) Use the following equation to calculate how many moles of Fe^{2+} (aq) were present in the conical flask.



$$\begin{array}{ccc} 1 & : & 5 \\ 2.48 \times 10^{-4} & : & x \end{array} \quad \boxed{= 1.24 \times 10^{-3} \text{ mol}}$$

$$\text{moles of } \text{Fe}^{2+}(\text{aq}) \text{ in the conical flask} = 1.24 \times 10^{-3} \text{ mol}$$

- (iii) Calculate the number of moles of Fe^{2+} in your weighed sample of **FA 1**.

Weighed sample was dissolved and solution was made up to 250 cm^3

$$\begin{array}{ccc} 25.0 \text{ cm}^3 & \text{---} & 1.24 \times 10^{-3} \text{ mol} \\ 250 \text{ cm}^3 & \text{---} & x \end{array} \quad \boxed{= 0.0124 \text{ mol}}$$

$$\text{moles of } \text{Fe}^{2+} \text{ in the weighed sample} = 0.0124 \text{ mol}$$

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(iv) Calculate the percentage of iron in **FA 1**.

[A_r : Fe, 55.8]

$$m\% = \frac{\text{mass}}{A_r}$$

$$0.0124 = \frac{\text{mass}}{55.8}$$

$$\boxed{\text{mass of iron} = 0.692 \text{ g}}$$

$$\frac{0.692}{5.85} \times 100 = \boxed{11.8\%}$$

the percentage of iron in **FA 1** = 11.8 %
[5]

(d) There are a number of sources of potential error in this experiment. One of these involves the readings taken using the balance.

(i) State the maximum individual error in any single balance reading.

$$\text{error} = \frac{\text{Least Count}}{2} = \frac{0.01}{2}$$

maximum individual error = 0.005 g

(ii) Calculate the maximum percentage error in the mass of **FA 1** used in your experiment.

$$\begin{aligned} \% \text{ error} &= \frac{\text{error}}{\text{amount measured}} \times 100 \\ &= \frac{2(0.005)}{5.85} \times 100 \end{aligned} \quad \boxed{= 0.171\%}$$

maximum percentage error = 0.171 % [2]

[Total: 16]

**WATER
of
CRYSTALLIZATION**

EXPERIMENT NO. 23

FA 4 is an **impure** sample of hydrated magnesium sulfate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. When heated the water of crystallisation is driven off to leave anhydrous magnesium sulfate, MgSO_4 . The impurity does not give off water when heated. By determining how much water is present in the impure sample, the percentage purity can be calculated.

For
Examiner's
Use

(a) Method

- Weigh a clean dry crucible.
- Empty all of the **FA 4** into the crucible.
- Reweigh the crucible and its contents.
- Support the crucible in the pipe-clay triangle on top of a tripod.
- Heat the crucible gently for about 1 minute and then more strongly for a further 4 minutes.
- Allow the crucible to cool.
- When the crucible is cool enough to handle, reweigh the crucible and its contents.
- Repeat the cycle of heating and weighing as many times as you think necessary.

In the space below, record, in an appropriate form, all your weighings and include the mass of **FA 4** used and the mass of water that was lost.

mass of empty crucible /g	26.30
mass of crucible + FA4 /g	27.80
mass of FA4 before heating /g	1.50
mass of crucible + FA4 after heating /g	27.15
mass of crucible + FA4 after reheating /g	27.13
mass of FA4 after heating /g	0.83
mass of water lost /g	0.67

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[5]

(b) Calculations

Show your working and express your answers to **three** significant figures.

- (i) Using the mass of water that was lost on heating, calculate the mass of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ that was present in the initial sample of **FA 4**.
[A_r : H, 1.0; O, 16.0; Mg, 24.3; S, 32.1]

$$\begin{array}{r}
 \text{MgSO}_4 \cdot 7\text{H}_2\text{O} \quad \text{---} \quad 7\text{H}_2\text{O} \\
 246.4 \quad \text{---} \quad 126 \\
 x \quad \text{---} \quad 0.67
 \end{array}
 \quad \text{---} \quad \boxed{1.31 \text{ g}}$$

mass of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ = $\boxed{1.31}$ g [1]

- (ii) Calculate the percentage by mass of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in **FA 4**.

$$\frac{1.31}{1.50} \times 100 = \boxed{87.3\%}$$

percentage by mass of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in **FA 4** = $\boxed{87.3}$ % [1]

- (c) Suggest an improvement to the practical procedure that would give a more accurate value for the percentage by mass of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in **FA 4**.

Use larger mass of FA4 to reduce the percentage error of weighing machine. OR
use a lid to prevent spitting. [1]

[Total: 8]

EXPERIMENT NO. 24

FA 4 is an impure sample of hydrated calcium chloride, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$. On heating, hydrated calcium chloride loses its water of crystallisation.



You will determine the purity of **FA 4** by measuring the loss in mass that occurs when it is heated. The impurity present in **FA 4** is not decomposed on heating.

(a) Method

You should read the instructions carefully before starting any practical work and draw a table for your results in the space below.

- Weigh a crucible and record its mass.
- Add between 1.80g and 2.00g of **FA 4** into the crucible.
- Reweigh the crucible and its contents and record the mass.
- Place the crucible on the pipe-clay triangle and heat gently for 1 minute and then strongly for a further 2 minutes.
- Allow the crucible and its contents to cool. Reweigh the crucible and contents and record the mass.
- Heat the crucible strongly for a further 2 minutes. Allow it to cool. Reweigh the crucible and contents and record the mass.
- Repeat the heating, cooling and weighing until you are satisfied that all the water of crystallisation has been removed.
- Calculate and record the mass of **FA 4** used and the total mass of water lost.

mass of empty crucible /g	26.45
mass of crucible + FA4 /g	28.35
mass of FA4 before heating /g	1.90
mass of crucible + FA4 after heating /g	28.15
mass of crucible + FA4 after 1 st reheating /g	28.08
mass of crucible + FA4 after 2 nd reheating /g	28.07
mass of FA4 after heating /g	1.62
mass of water lost /g	0.28

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[6]

(b) Calculations

Show your working and appropriate significant figures in the final answer to **each** stage of your calculations.

(i) The percentage loss in mass on heating is defined as

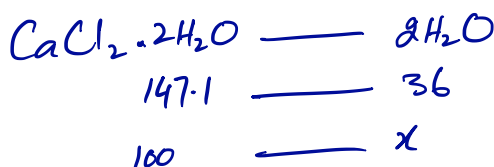
$$\frac{\text{the loss in mass on heating}}{\text{the original mass}} \times 100.$$

Calculate the percentage loss in mass of **FA 4**.

$$\frac{0.28}{1.90} \times 100$$

percentage loss in mass = 14.74 %

(ii) Calculate the percentage loss in mass when **pure** hydrated calcium chloride, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, is heated.



percentage loss in mass = 24.47 %

(iii) Use your results to (i) and (ii) to calculate the percentage purity of **FA 4**, impure $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$.

$$\frac{14.74}{24.47} \times 100$$

percentage purity = 60.2 %
[3]

(c) A student carried out this experiment using 2.60g of **FA 4**.

Suggest whether this experiment would give a more accurate result for the percentage purity of **FA 4**. Explain your answer.

Yes, as there will be less percentage error in weighing mass. [1]

OR

NO, because there is more water to be lost so more spitting and frothing so percentage error in mass lost will be greater.

(d) In your calculations you assumed that the impurity in **FA 4** does not decompose on heating.

State how the percentage purity that you calculated in (b)(iii) would change if the impurity were to decompose on heating.

Explain your answer.

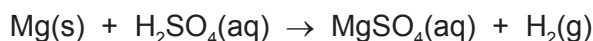
There would be the greater loss of mass therefore percentage
purity would increase. [1]

[Total: 11]

ENTHALPY

EXPERIMENT NO. 25

You will determine the enthalpy change, ΔH , for the reaction between magnesium and dilute sulfuric acid. The equation for the reaction is given below.



FA 3 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4 .

two different coiled lengths of magnesium ribbon, Mg.

(a) Method

Read through the method **before** starting any practical work and prepare a table for your results in the space below.

- Weigh the shorter piece of magnesium ribbon and record its mass.
- Support the plastic cup in the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 50 cm^3 of **FA 3** into the plastic cup.
- Place the thermometer in the **FA 3** in the plastic cup and record the initial temperature.
- Add the shorter piece of magnesium ribbon into the plastic cup. Ensure that all of the magnesium is in contact with the acid. (**Care**: acid spray may occur.)
- Stir the mixture and record the maximum temperature.
- Empty and rinse the plastic cup. Shake out any excess water.
- Repeat the experiment using the longer piece of magnesium ribbon and record all your data.

Results

	Shorter Piece	Longer Piece
mass of Magnesium ribbon/g	0.15	0.30
initial temperature / $^{\circ}\text{C}$	25.0	25.0
final temperature / $^{\circ}\text{C}$	38.0	50.0
change in temperature / $^{\circ}\text{C}$	13.0	25.0

[4]

(b) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Show by calculation that the sulfuric acid, **FA 3**, was used in excess in both experiments.
(A_r : Mg, 24.3)

moles of H_2SO_4

$$n = CV$$

$$= 1.00 \times \frac{50}{1000}$$

$$= 0.0500 \text{ moles}$$

moles of Magnesium

$$\text{Shorter piece: } \text{mole} = \frac{\text{mass}}{A_r} = \frac{0.15}{24.3} = 0.00617 \text{ moles}$$

$$\text{Longer piece: } \text{mole} = \frac{\text{mass}}{A_r} = \frac{0.30}{24.3} = 0.0124 \text{ moles}$$

Hence the no. of moles of H_2SO_4
are greater than Mg moles.

- (ii) State an observation which confirms that the sulfuric acid, **FA 3**, was in excess.

Magnesium ribbon completely dissolved / disappeared.

- (iii) Calculate the heat energy produced when the **shorter** piece of magnesium was added to **FA 3**.

(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm³ of solution by 1.0 °C.)

$$Q = mc\Delta T$$

$$= 50 \times 4.3 \times 13.0$$

heat energy produced = 2795 J

- (iv) Calculate the enthalpy change, in kJ mol⁻¹, for the reaction between the **shorter** piece of magnesium and the sulfuric acid.

$$0.00617 \text{ mole} \text{ --- } 2795 \text{ J}$$

$$1 \text{ mole} \text{ --- } x$$

$$= 452998 \text{ J/mol}$$

$$\frac{452998}{1000} = 452.998 \text{ kJ/mol}$$

enthalpy change = - 453.0 kJ mol⁻¹
(sign) (value)

- (v) Calculate the heat energy produced when the **longer** piece of magnesium was added to **FA 3**.

(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm³ of solution by 1.0 °C.)

$$Q = mc\Delta T$$

$$= 50 \times 4.3 \times 25.0$$

$$= 5375 \text{ J}$$

heat energy produced = 5375 J

- (vi) Calculate the enthalpy change, in kJ mol⁻¹, for the reaction between the **longer** piece of magnesium and the sulfuric acid.

$$0.0124 \text{ mole} \text{ --- } 5375 \text{ J}$$

$$1 \text{ mole} \text{ --- } x$$

$$433468 \text{ J/mol}$$

$$\frac{433467}{1000} = 433.467$$

enthalpy change = - 433.5 kJ mol⁻¹
(sign) (value)

[5]

- (c) (i) What is the maximum error in a reading of the thermometer used in this experiment?

$$\text{error} = \frac{\text{Least Count}}{2} = \frac{1}{2} = 0.5$$

maximum error = 0.5 °C.

- (ii) Which of your temperature changes has the higher percentage error?

ΔT for shorter piece

- (iii) Calculate this maximum percentage error.

$$\% \text{ error} = \frac{\text{error}}{\text{Amount}} \times 100 = \frac{2 \times 0.5}{13.0} \times 100$$

maximum percentage error in the temperature change = 7.69 %

[1]

- (d) Apart from errors due to heat loss and thermometer readings, suggest another significant source of error in this experiment. State what improvement could be made to the procedure to reduce this error.

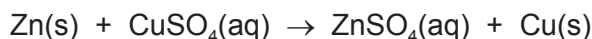
Error = Error in measuring out sulfuric acid with measuring cylinder.

Improvement = Use burette to transfer 50 cm^3 of H_2SO_4 . [2]

[Total: 12]

EXPERIMENT NO. 26

In this experiment you will measure the heat given out by the reaction of excess zinc with copper(II) sulfate solution and use this to estimate the concentration of the copper(II) sulfate.



FA 4 is zinc powder.

FA 5 is aqueous copper(II) sulfate, CuSO_4 .

(a) Method

Read through the instructions carefully and prepare a table below for your results before starting any practical work.

- Support the plastic cup in the 250 cm³ beaker.
- Use the 50 cm³ measuring cylinder to transfer 40 cm³ of **FA 5** into the plastic cup.
- Measure and record the initial temperature of the solution in the plastic cup.
- Start the stopwatch. Measure and record the temperature of the solution every 30 seconds up to and including the temperature at 2 minutes. Stir the solution frequently.
- At time $t = 2\frac{1}{2}$ minutes, add **all** the powdered zinc to the solution in the plastic cup and stir the mixture.
- Record the temperature every 30 seconds from $t = 3$ minutes up to and including $t = 9$ minutes. Stir the solution constantly.

time / minute	temperature / °C	time / minute	temperature / °C
0	24.0	5	44.0
$\frac{1}{2}$	24.0	$5\frac{1}{2}$	44.0
1	24.0	6	43.5
$1\frac{1}{2}$	24.0	$6\frac{1}{2}$	43.0
2	24.0	7	42.5
3	38.0	$7\frac{1}{2}$	42.0
$3\frac{1}{2}$	40.5	8	41.5
4	42.5	$8\frac{1}{2}$	41.0
$4\frac{1}{2}$	43.5	9	40.5

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[4]

(b) (i) On the grid opposite, plot the temperature (y-axis) against the time (x-axis). The scale for the temperature axis must allow you to plot a point with a temperature 5 °C greater than the maximum temperature you recorded.

(ii) Draw the following best-fit **straight** lines on the graph.

- a line through the points between time $t = 0$ minutes and time $t = 2$ minutes
- a line through the points between time $t = 5$ minutes and time $t = 9$ minutes
- a vertical line at time $t = 2\frac{1}{2}$ minutes

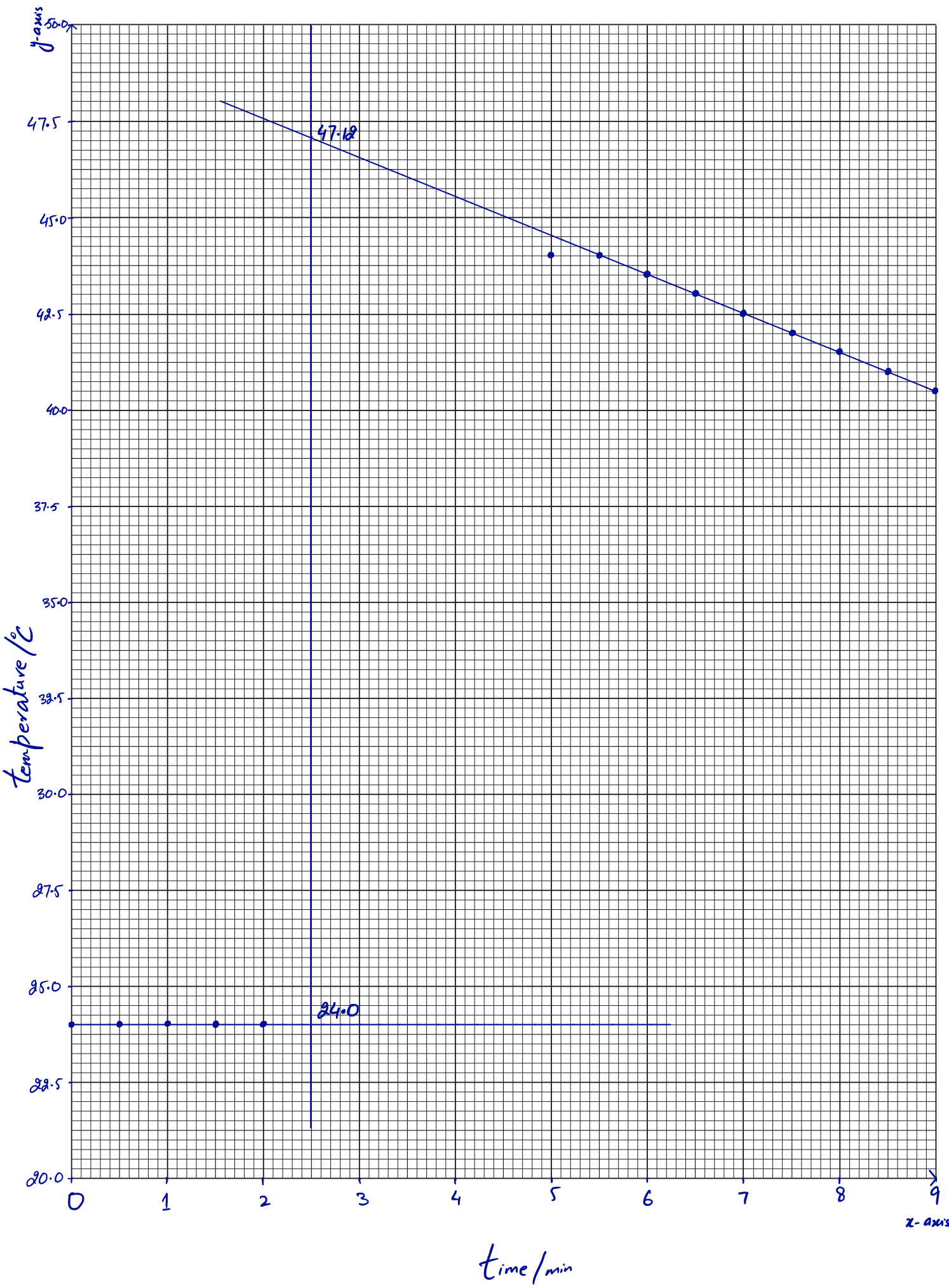
(iii) Extrapolate the first two straight lines so that they intersect the vertical line at time $t = 2\frac{1}{2}$ minutes.

Use these extrapolated lines to determine the theoretical temperature **change** at time $t = 2\frac{1}{2}$ minutes.

$$47.12 - 24.0 =$$

change in temperature = °C

[5]



(c) Calculations

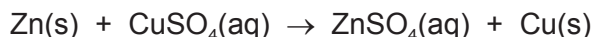
Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Use your answer to (b)(iii) to calculate the heat energy produced in the reaction. (Assume that 4.2 J are required to increase the temperature of 1 cm³ of solution by 1 °C.)

$$Q = mc\Delta T$$
$$= 40 \times 4.2 \times 23.12$$

heat energy produced = 3884 J

- (ii) The molar enthalpy change, ΔH , for the reaction shown below is -219 kJ mol^{-1} .



Use this value and your answer to (i) to calculate the number of moles of copper(II) sulfate in your reaction.

$$\frac{1 \text{ mol}}{2} \times \frac{219000}{3884}$$

moles of CuSO_4 = 0.0177 mol

- (iii) Use your answer to (ii), to calculate the concentration of copper(II) sulfate, in mol dm^{-3} , in FA 5.

$$C = \frac{n}{V} = \frac{0.0177}{0.0400}$$

concentration of CuSO_4 = 0.443 mol dm^{-3}
[3]

- (d) (i) Calculate the maximum percentage error in the highest temperature that you recorded in your results table.

$$\% \text{ error} = \frac{\text{error}}{\text{highest temp}} = \frac{0.5}{44.0}$$

maximum percentage error = 0.114 %

- (ii) A student suggested that the concentration of the copper(II) sulfate could be determined more accurately if a greater mass of zinc had been used. Explain whether you agree with this student.

Not agree. As zinc is already in excess.

- (iii) A second student suggested that the concentration of the copper(II) sulfate could be determined more accurately if a smaller volume of copper(II) sulfate was used. Explain whether you agree with this student.

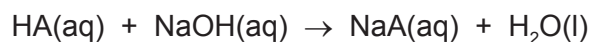
Not agree.
As smaller volume will have greater percentage error.

[3]

[Total: 15]

EXPERIMENT NO. 27

You are to determine the enthalpy change for the neutralisation reaction given below.



FA 3 is 1.80 mol dm^{-3} HA.

FA 4 is aqueous sodium hydroxide, NaOH.

(a) Method

Read through the instructions carefully and prepare a table below for your results before starting any practical work.

- Support the plastic cup in the 250 cm^3 beaker.
- Rinse and fill the burette with **FA 3**.
- Use the measuring cylinder to transfer 25 cm^3 of **FA 4** into the plastic cup.
- Place the thermometer in the plastic cup and record the temperature of the solution. Tilt the cup if necessary to ensure the thermometer bulb is fully immersed.
- Run 5.00 cm^3 of **FA 3** into the cup. Stir, and record the new temperature of the solution and the volume of **FA 3** added.
- Run a second 5.00 cm^3 of **FA 3** into the cup. Stir and record the new temperature and the total volume of **FA 3** added.
- Continue adding **FA 3** in 5.00 cm^3 portions. Stir and record each new temperature and total volume of **FA 3** until a total of 45.00 cm^3 has been added.

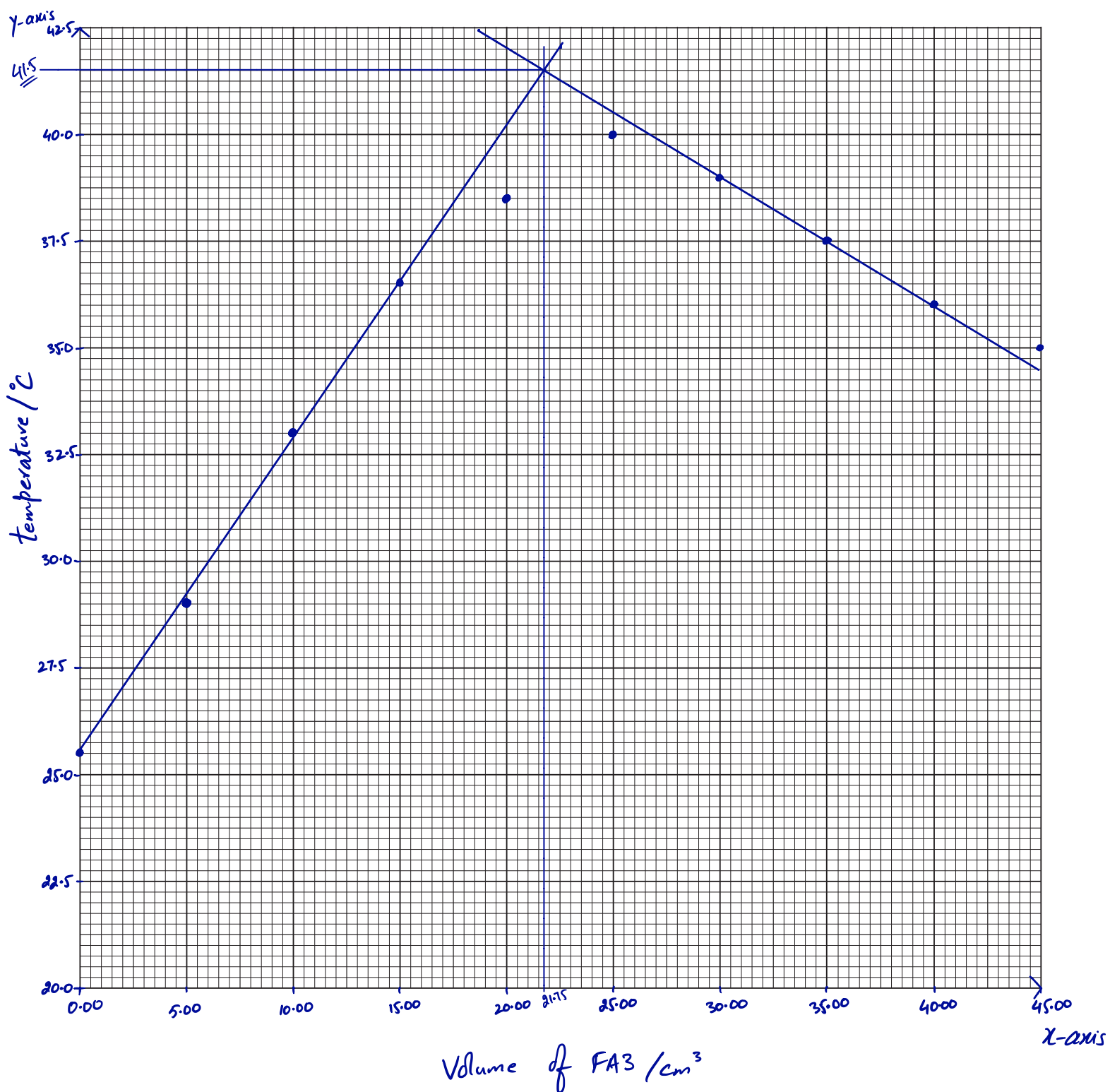
Results

Volume of FA3/cm ³	temperature/°C	Volume of FA3/cm ³	temperature/°C
0.00	25.5	25.00	40.0
5.00	29.0	30.00	39.0
10.00	33.0	35.00	37.5
15.00	36.5	40.00	36.0
20.00	38.5	45.00	35.0

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[4]

- (b) Plot a graph of temperature (y-axis) against total volume of **FA 3** added (x-axis) on the grid opposite. The temperature axis should allow you to include a point at least 2°C greater than the maximum temperature recorded.



Draw the best fit smooth curve or straight line through the two sets of points, one for the increase in temperature of the mixture and the other for the cooling of the solution once the reaction is complete. Extrapolate the two lines and determine the maximum **increase** in temperature and the corresponding volume of **FA 3** added for this increase in temperature.

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maximum temperature **increase** = ...16.0... °C

volume of **FA 3** = ...21.75... cm³
[4]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of HA present in the volume of **FA 3** recorded in (b).

$$n = CV$$
$$= 1.80 \times 0.02175$$

moles of HA = 0.0392 mol

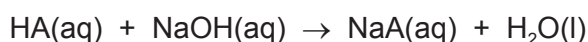
- (ii) Using your answers to (b), calculate the heat energy produced when **FA 3** neutralised 25 cm³ of sodium hydroxide.

(Assume that 4.2 J of heat energy changes the temperature of 1.0 cm³ of solution by 1.0 °C.)

$$m = 25 + 21.75$$
$$= 46.75$$
$$Q = mc\Delta T$$
$$= 46.75 \times 4.2 \times 16.0$$

heat energy produced = 3142 J

- (iii) Calculate the enthalpy change of neutralisation, in kJ mol⁻¹, for the reaction below.



$$\frac{0.0392 \text{ mol}}{1 \text{ mol}} \times \frac{3142 \text{ J}}{x}$$
$$= 80153 \text{ J/mol}$$

enthalpy change = 80.15 kJ mol⁻¹
(sign) (value)

[4]

- (d) The maximum error in a single thermometer reading is ±0.5 °C. Calculate the maximum percentage error in the increase in temperature recorded in (b).

$$\% \text{ error} = \frac{\text{error}}{\text{temperature}} = \frac{0.5 \times 2}{16.0} \times 100$$

maximum percentage error = 6.25 % [1]

- (e) When carrying out thermochemistry experiments in an A Level laboratory, the plastic cup is usually placed in a glass beaker. Give a reason for the use of the glass beaker.

Safety - to prevent plastic cup tipping over - greater stability OR
Reduce heat loss - air jacket. [1]

- (f) Apart from using a thermometer calibrated to a greater level of precision, suggest one improvement that could be made to the **method** carried out in (a).

Use burette or pipette to measure FA4 instead of measuring cylinder.
..... [1]

[Total: 15]

EXPERIMENT NO. 28

You are required to determine the molar enthalpy change of solution for ammonium chloride, **FA 1**.

*For
Examiner's
Use*

When an exothermic reaction takes place in a container such as a beaker, some of the evolved heat energy is absorbed by the beaker.

When an endothermic reaction takes place some of the required heat energy is supplied by the beaker.

The amount of heat energy evolved or supplied for a 1 °C change in temperature is known as the heat capacity of the beaker.

In preparation for your experiment to determine the molar enthalpy change of solution for **FA 1** you will first need to determine the approximate heat capacity of a 250 cm³ beaker.

Before starting any practical work read through the instructions in (a) and draw up a table to record your results.

(a) **Determining the approximate heat capacity of the 250 cm³ beaker**

When samples of hot and cold water are mixed in the 250 cm³ beaker, some heat is lost to the beaker in raising its temperature. To determine the approximate heat capacity of your 250 cm³ beaker, you will determine the maximum temperature rise when a sample of hot water is added to cold water in the beaker.

- Use a 50 cm³ measuring cylinder to transfer 50 cm³ of cold water into the 250 cm³ beaker.
- Use the 50 cm³ measuring cylinder to transfer 50 cm³ of cold water into a 100 cm³ beaker. Note the temperature of the water in this 100 cm³ beaker and heat it **carefully and gently** until the temperature of the water in it has increased by 45–50 °C then stop heating, *e.g. if the water is at 20.0 °C you should warm it to 65–70 °C.*
- Stir the cold water in the 250 cm³ beaker with the thermometer.
- Record the temperature of the cold water (this is the temperature at t = 0 min).
- Record the temperature each minute for 3 minutes.
- After you have taken the reading at t = 3 min, use the thermometer to stir the hot water in the 100 cm³ beaker.
- At t = 4 min, measure the temperature of the hot water and record this value in the box below.
- **Immediately** add the hot water from the 100 cm³ beaker to the cold water in the 250 cm³ beaker. Stir with the thermometer but do **not** record the temperature.
- Continue to stir the water throughout the experiment.
- Record the temperature at t = 5 min, and then every ½ **minute** until t = 8 min.
- Empty and rinse the 250 cm³ beaker. Dry it using a paper towel.
- Record all measurements of time and temperature obtained.

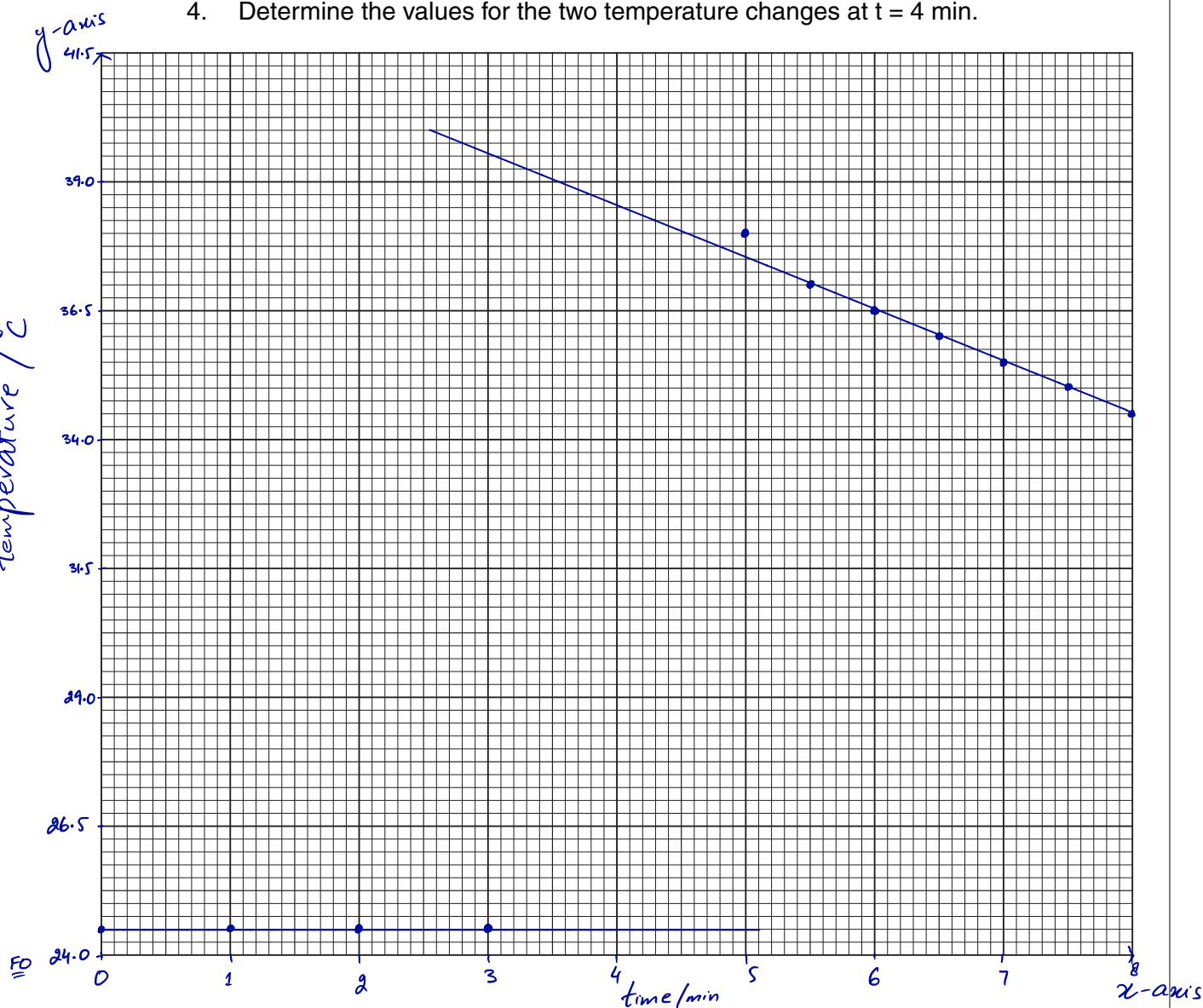
The temperature, T_1 , of the hot water at t = 4 min is °C.

Table of results

time / min	temperature / °C	time / min	temperature / °C
0	24.5	6	36.5
1	24.5	6½	36.0
2	24.5	7	35.5
3	24.5	7½	35.0
5	38.0	8	34.5
5½	37.0		

(b) Graph plotting

1. Plot a graph of the temperature of the water in the 250 cm³ beaker (y-axis) against time (x-axis) on the grid below.
Do **not** plot the temperature, T_1 , of the hot water at $t = 4$ min.
2. Draw two straight lines of best fit; one through the points up to $t = 3$ min; the second through the points from $t = 5$ min to $t = 8$ min. Extrapolate both lines to $t = 4$ min.
3. From the extrapolated lines read the minimum and the maximum temperatures at $t = 4$ min. Record these values in the spaces provided below.
4. Determine the values for the two temperature changes at $t = 4$ min.



Minimum temperature, T_2 , at $t = 4$ min is ...24.5... °C.

Maximum temperature, T_3 , at $t = 4$ min is ...38.5... °C.

Temperature rise for 50 cm³ of cold water in the 250 cm³ beaker, $(T_3 - T_2)$ is ...14.0... °C.

Temperature fall for 50 cm³ of hot water from the 100 cm³ beaker, $(T_1 - T_3)$ is ...17.5... °C.

[4]

(c) Calculations

Working should be shown in all calculations.

[4.2 J are absorbed or released when the temperature of 1.0 cm³ of **water** changes by 1.0 °C.]

- (i) Calculate the heat energy gained by the 50 cm³ of cold water in the 250 cm³ beaker.

$$Q = mc\Delta T$$

$$= 50 \times 4.2 \times 14.0$$

The heat energy gained by the cold water = ...2940 J.

- (ii) Calculate the heat energy lost by the 50 cm³ of hot water from the 100 cm³ beaker.

$$Q = mc\Delta T$$

$$= 50 \times 4.2 \times 17.5$$

The heat energy lost by the hot water = ...3675 J.

- (iii) The difference between the values calculated in (i) and (ii) is an approximate value for the total heat energy absorbed by the 250 cm³ beaker during the experiment. The heat capacity of the beaker is the amount of heat energy absorbed for a 1 °C change in temperature.

approximate heat capacity of the 250 cm³ beaker = $\frac{(\text{heat energy lost}) - (\text{heat energy gained})}{(T_3 - T_2)}$ J °C⁻¹

Use your answers to (i) and (ii) and the temperature rise from (b) to calculate the approximate heat capacity of the 250 cm³ beaker.

$$\frac{3675 - 2940}{14.0}$$

The approximate heat capacity of the 250 cm³ beaker = ...52.2 J °C⁻¹.

[1]

(d) Determining the enthalpy change of solution for ammonium chloride

Follow the instructions below to find the temperature change when a known mass of solid ammonium chloride dissolves in water.

You are provided with two samples of ammonium chloride. You should use the sample labelled **NH₄Cl** in **experiment 1** and the sample labelled **FA 1** in **experiment 2**.

Experiment 1

- Enter all results in the table below.
- Weigh the stoppered tube containing ammonium chloride, which is labelled **NH₄Cl**.
- Use the 50 cm³ measuring cylinder to transfer 100 cm³ of cold water into the rinsed and dried 250 cm³ beaker used in **(a)**.
- Stir the water in the beaker with the thermometer and record the temperature.
- Add the solid from the weighed tube to the water.
- Stir the mixture constantly with the thermometer.
- Record the minimum temperature obtained in the solution.
- Reweigh the tube labelled **NH₄Cl**, its stopper and any residual ammonium chloride.
- Empty and rinse the beaker and dry it using a paper towel.

Experiment 2

- Enter all results in the table below.
- Weigh a clean, dry, boiling-tube.
- Weigh between 9.8 g and 10.2 g of **FA 1**, ammonium chloride, into the boiling-tube.
- Repeat the procedure in **experiment 1** and record the minimum temperature obtained when this mass of **FA 1** dissolves in 100 cm³ of water.
- Reweigh the boiling-tube and any residual ammonium chloride.

Results

	experiment 1	experiment 2
mass of tube + ammonium chloride / g	44.90	39.80
mass of empty tube / g		29.70
mass of tube + residual ammonium chloride / g	29.70	29.75
mass of ammonium chloride / g	15.20	10.05
initial temperature of water / °C	25.5	26.0
minimum temperature obtained / °C	16.0	20.0
temperature fall, ΔT / °C	9.5	6.0

[6]

(e) Calculations

Working should be shown in all calculations.

- (i) Use the temperature fall from (d), **experiment 1**, to calculate the change in heat energy of the solution.
[4.3 J are absorbed or released when the temperature of 1.0 cm³ of **solution** changes by 1.0 °C.]

$$Q = mc\Delta T$$

$$= 100 \times 4.3 \times 9.5$$

The change in heat energy of the solution =4085 J.

- (ii) To calculate the total change in heat energy as ammonium chloride dissolves in water, the change in heat energy of the 250 cm³ beaker has to be added to the change in heat energy of the solution.
Explain why these two changes in heat energy have to be added together.

During experiment beaker also absorb or lose heat energy, so we'll add this to calculate total heat energy.

- (iii) Use your answer in (i) above and the approximate heat capacity of the 250 cm³ beaker calculated in (c)(iii) to calculate the combined change in heat energy of the beaker and solution.

$$4085 + (52.2 \times 9.5)$$

The combined change in heat energy of the beaker and solution =4581 J.

- (iv) Calculate how many moles of **FA 1**, NH₄Cl, were used in (d), **experiment 1**.
[A_r: Cl, 35.5; H, 1.0; N, 14.0]

$$\text{mole} = \frac{\text{mass}}{M_r} = \frac{15.20}{53.5}$$

0.284 mol of **FA 1** were used in **experiment 1**.

i	
ii	
iii	
iv	

- (v) Calculate the enthalpy change when 1 mol of **FA 1** dissolves in an excess of water. This is the molar enthalpy change of solution, $\Delta H_{\text{solution}}(\text{NH}_4\text{Cl})$. Make certain that your answer is given in kJ mol^{-1} and has the appropriate sign.

$$\begin{array}{r} 0.284 \text{ mol} \quad \text{---} \quad 4581 \text{ J} \\ 1 \text{ mol} \quad \quad \quad \times \\ \hline = 16130 \text{ J/mol} \end{array}$$

$$\Delta H_{\text{solution}}(\text{NH}_4\text{Cl}) = \text{.....} \quad \text{+} \quad \text{.....} \quad \text{16.13} \quad \text{kJ mol}^{-1}.$$

sign calculated value

- (vi) Explain the significance of the sign you have given in (v) and how it is related to your experimental results.

..... The reaction is endothermic

OR

..... Temperature falls during reaction

[8]

v	
vi	
vii	
viii	

(f) Evaluation

A data book value for the molar enthalpy change of solution, $\Delta H_{\text{solution}}(\text{NH}_4\text{Cl})$, is $+15.2 \text{ kJ mol}^{-1}$.

The value you have obtained may be significantly different from this value.

Calculate the difference between your value of $\Delta H_{\text{solution}}(\text{NH}_4\text{Cl})$ and the data book value. Record this difference below. Express this difference as a percentage of the data book value.

$$16.13 - 15.2$$

$$\text{difference} = \dots\dots\dots 0.930 \text{ kJ mol}^{-1}$$

$$\frac{0.930}{15.2} \times 100$$

$$\text{percentage difference} = \dots\dots\dots 6.12 \dots\dots \%$$

[1]

(g) Sources of error

Describe **one** major source of error in this experiment. Suggest an improvement which would significantly increase the accuracy of the experiment. Explain why your suggestion would produce a more accurate value.

description of major source of error

Heat gained from the surrounding.

suggested improvement

Use plastic beaker or lagging for insulation.

explanation of why suggestion would increase experimental accuracy

Less heat gained from surrounding will lead to accurate results.

[2]

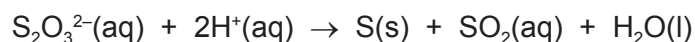
[Total: 25]

RATE OF REACTION

EXPERIMENT NO. 29

In this experiment you will investigate how the rate of reaction between sodium thiosulfate and hydrochloric acid is affected by the concentration of the acid.

When aqueous thiosulfate ions react with hydrogen ions, H^+ , in any acid, a pale yellow precipitate of sulfur is formed. The ionic equation for this reaction is given below.



The rate of the reaction can be determined by measuring the time taken to produce a fixed quantity of sulfur.

FA 4 is 0.10 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.

FA 5 is 0.20 mol dm^{-3} hydrochloric acid, HCl .

(a) Method

Record **all** your measurements, in an appropriate form, in the space below.

Experiment 1

- Use the larger measuring cylinder to transfer 40 cm^3 of **FA 4** into the 100 cm^3 beaker.
- Rinse the larger measuring cylinder thoroughly with water, then add 30 cm^3 of **FA 5** to the beaker and start timing **immediately**.
- Stir the mixture once and place the beaker on top of the printed insert page provided.
- Look down through the solution in the beaker at the print on the insert.
- Stop timing as soon as the precipitate of sulfur makes the print on the insert invisible.
- Record the reaction time to the **nearest second**.
- Empty and rinse the 100 cm^3 beaker.
- Dry the outside of the beaker ready for Experiment 2.

Experiment 2

- Rinse the larger measuring cylinder, then use it to transfer 40 cm^3 of **FA 4** into the 100 cm^3 beaker.
- Use the smaller measuring cylinder to add 10 cm^3 of distilled water to the beaker.
- Use the same measuring cylinder to add 20 cm^3 of **FA 5** to the mixture in the beaker and start timing **immediately**.
- Stir the mixture once and place the beaker on top of the printed insert page provided.
- Stop timing as soon as the print on the insert becomes invisible.
- Record the reaction time to the **nearest second**.
- Empty and rinse the 100 cm^3 beaker.
- Dry the outside of the beaker ready for Experiment 3.

Experiment 3

- Carry out the reaction using a mixture of 40 cm^3 of **FA 4**, 20 cm^3 of distilled water and 10 cm^3 of **FA 5**.
- Measure and record the reaction time to the **nearest second**.

Experiment No.	Volume of FA5/ cm^3	Volume of water/ cm^3	Time /s
01	30	0	78
02	20	10	96
03	10	20	144

I	
II	
III	
IV	

[4]

- (b) (i) The 'rate of reaction' can be represented by the formula below.

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

Use this formula to calculate the 'rate of reaction' for Experiments 1 and 3.
Give the unit.

'rate of reaction' for Experiment 1 12.8 unit s⁻¹.....

'rate of reaction' for Experiment 3 6.94 unit s⁻¹.....

- (ii) Calculate the initial concentrations of hydrochloric acid in the reaction mixtures in Experiments 1 and 3.

Experiment # 01

$$n = CV$$

$$= 0.20 \times 0.030$$

$$= 6.00 \times 10^{-3} \text{ mol}$$

$$C = \frac{n}{V} = \frac{6.00 \times 10^{-3}}{0.070} = 0.0857 \text{ mol dm}^{-3}$$

Experiment # 02

$$n = CV$$

$$= 0.200 \times 0.010$$

$$= 2.00 \times 10^{-3} \text{ mol}$$

$$C = \frac{n}{V} = \frac{2.00 \times 10^{-3}}{0.070} = 0.0286 \text{ mol dm}^{-3}$$

initial concentration of HCl in Experiment 1 = 0.0857 mol dm⁻³

initial concentration of HCl in Experiment 3 = 0.0286 mol dm⁻³

- (iii) How is the 'rate of reaction' affected by the concentration of hydrochloric acid in the mixture?

Rate of reaction increases with increase of concentration.

- (iv) Predict how the reaction time measured in Experiment 1 would have been affected if the experiment had been carried out using 0.20 mol dm⁻³ sulfuric acid instead of 0.20 mol dm⁻³ hydrochloric acid.

Explain your answer.

Time will be shorter for sulfuric acid, as sulfuric acid has a greater concentration of H⁺ ions.

- (v) Predict how the reaction time measured in Experiment 3 would have been affected if the experiment had been carried out in a 250 cm³ beaker instead of a 100 cm³ beaker.

Explain your answer.

Reaction time will be greater due to less depth of solution in 250 cm³ beaker.

[5]

[Total: 9]

EXPERIMENT NO. 30

Metal carbonates react with dilute acids to produce carbon dioxide. You will identify the metal, **M**, in a metal carbonate, M_2CO_3 , by measuring the volume of carbon dioxide produced during the reaction of M_2CO_3 with excess hydrochloric acid.



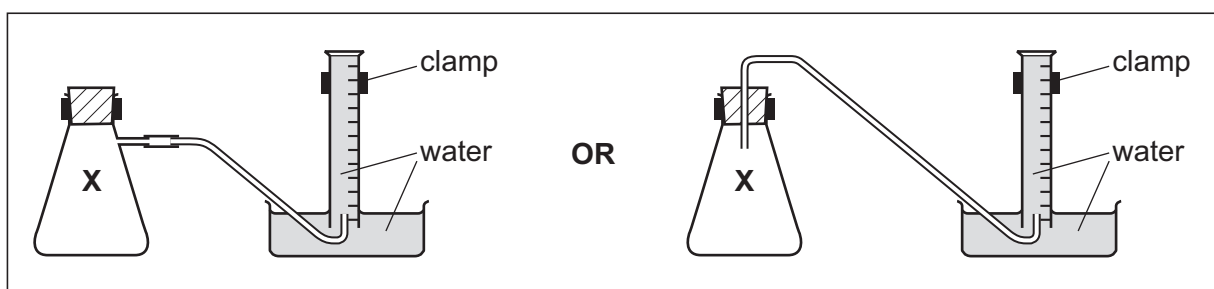
FA 2 is hydrochloric acid, HCl , as used in **Question 1**.

FA 4 is M_2CO_3 .

(a) Method

Read **all** instructions before starting your practical work.

The diagrams below may help you in setting up your apparatus.



- Fill the tub with water to a depth of about 5 cm.
- Fill the 250 cm³ measuring cylinder **completely** with water. Hold 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 cm³ measuring cylinder to place 50 cm³ of **FA 2** into the reaction flask, labelled **X**.
- Check that the bung fits tightly in the neck of flask **X**, clamp flask **X**, and place the end of the delivery tube into the inverted 250 cm³ measuring cylinder.
- Weigh the container with **FA 4** and record the mass in the space below.
- Remove the bung from the neck of the flask. Tip all the **FA 4** into the acid in the flask and replace the bung **immediately**. Remove the flask from the clamp and swirl it to mix the contents.
- Swirl the flask occasionally until no more gas is evolved. Replace the flask in the clamp.
- Reweigh the container and record the mass, and the mass of **FA 4** used, in the space below.
- When no more gas is collected, measure and record the final volume of gas in the measuring cylinder in the space below.

mass of tube + FA4 /g	21.70
mass of tube + residue /g	20.80
mass of FA4 used /g	0.90
initial volume of measuring cylinder /cm ³	26
final volume of measuring cylinder /cm ³	27
volume of CO ₂ collected /cm ³	201

[2]

(b) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Use the volume of gas you collected to calculate the number of moles of gas produced. [Assume that 1 mole of gas occupies 24.0 dm³ under these conditions.]

$$\frac{201 \text{ cm}^3}{1000} = 0.201 \text{ dm}^3$$

$$\begin{array}{l} 24 \text{ dm}^3 \text{ --- } 1 \text{ mol} \\ 0.201 \text{ dm}^3 \text{ --- } x \end{array}$$

$$\text{moles of gas} = \dots\dots\dots 8.38 \times 10^{-3} \text{ mol}$$

- (ii) Use your answer to (i) to deduce the number of moles of M₂CO₃ used in the reaction.

mole ratio

$$\begin{array}{ccc} \text{M}_2\text{CO}_3 & : & \text{CO}_2 \\ 1 & : & 1 \\ 8.38 \times 10^{-3} & : & 8.38 \times 10^{-3} \end{array} \quad \text{moles of M}_2\text{CO}_3 = \dots\dots\dots 8.38 \times 10^{-3} \text{ mol}$$

- (iii) Use your answer to (ii) and the mass of FA 4 used to calculate the relative formula mass, M_r, of M₂CO₃.

$$\text{mol} = \frac{\text{mass}}{M_r}$$
$$8.38 \times 10^{-3} = \frac{0.90}{M_r} = 107.4$$
$$M_r \text{ of M}_2\text{CO}_3 = \dots\dots\dots 107.4$$

- (iv) Use your answer to (iii) and the Periodic Table on page 12 to identify metal M. Explain your answer.

$$\begin{array}{l} M_r \text{ of M}_2\text{CO}_3 = 107.4 \qquad \qquad \qquad M_r \text{ of CO}_3 = 60 \\ M_r \text{ of M}_2 = 107.4 - 60 = 47.4 \\ A_r \text{ of M} = \frac{47.4}{2} = \boxed{23.7} \end{array}$$

M is Sodium - Na

As it is the nearest.

(c) (i) A 250 cm³ measuring cylinder can be read to ±1 cm³.

Calculate the maximum percentage error in your reading of the volume of gas.

$$\frac{1 \times 2}{201} \times 100$$

maximum percentage error = ...0.995... %

(ii) It is likely that the volume of carbon dioxide that you collected was less than the theoretical volume.

Give **two** reasons why this volume is likely to be less than the theoretical volume.

In each case, suggest and explain a modification to the practical procedure that could help to reduce the difference in volume.

reason ... gas dissolves in water/solution ...

modification ... Use a gas syringe instead of measuring cylinder for gas collection ...

reason ... gas escapes before stopper inserted ...

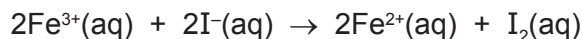
modification ... Use more (excess) volume of a lower concentration of acid ...

[5]

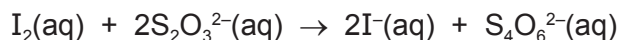
[Total: 11]

EXPERIMENT NO. 31

You will investigate the rate of reaction between iron(III) ions, Fe^{3+} , and iodide ions, I^- .



The iodine, I_2 , produced can be reacted immediately with thiosulfate ions, $\text{S}_2\text{O}_3^{2-}$.



When all the thiosulfate has been used, the iodine produced will turn starch indicator blue-black. The rate of the reaction can therefore be measured by finding the time for the blue-black colour to appear.

FB 1 is aqueous iron(III) chloride, FeCl_3 .

FB 2 is aqueous potassium iodide, KI.

FB 3 is $0.0060 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

starch indicator

You are advised to read the instructions before starting any practical work.

(a) Method

Experiment 1

- Fill a burette with **FB 1**.
- Run 20.00 cm^3 of **FB 1** into a 100 cm^3 beaker.
- Use the measuring cylinder to place the following in a second 100 cm^3 beaker.
 - 10 cm^3 of **FB 2**
 - 20 cm^3 of **FB 3**
 - 10 cm^3 of starch indicator
- Add the contents of the second beaker to the first beaker and start timing.
- Stir the mixture once and place the beaker on the white tile.
- The mixture turns brown and then yellow before turning a blue-black colour. Stop timing when this **blue-black colour** appears.
- Record in your table the volume of **FB 1** used, the volume of distilled water used and the time to the **nearest second** for the blue-black colour to appear.
- Wash both beakers.

For each of **Experiments 2-6** you should complete your results table to show the volume of **FB 1** used, the volume of distilled water used and the time taken to the **nearest second** for the blue-black colour to appear.

Experiment 2

- Fill the other burette with distilled water.
- Run 10.00 cm^3 of **FB 1** into a 100 cm^3 beaker.
- Run 10.00 cm^3 of distilled water into the same beaker.
- Use the measuring cylinder to place the following in a second 100 cm^3 beaker.
 - 10 cm^3 of **FB 2**
 - 20 cm^3 of **FB 3**
 - 10 cm^3 of starch indicator
- Add the contents of the second beaker to the first beaker and start timing.
- Stir the mixture once and place the beaker on the white tile.
- Stop timing when a blue-black colour appears.
- Wash both beakers.

Experiments 3-6

Carry out **four** further experiments to investigate the effect of changing the concentration of $\text{Fe}^{3+}(\text{aq})$ by altering the volume of aqueous FeCl_3 , **FB 1**, used.

You should not use a volume of **FB 1** that is less than 6.00 cm^3 and the total volume of the reaction mixture must always be 60 cm^3 .

Exp #	Volume of FB1/ cm^3	Volume of water/ cm^3	Reaction Time/s
01	20.00	0.00	23
02	10.00	10.00	36
03	6.00	14.00	45
04	8.00	12.00	40
05	13.00	7.00	32
06	16.00	4.00	27

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

(b) Calculations

The rate of reaction can be found by calculating the change in concentration of Fe³⁺(aq) that occurred when enough iodine was produced to change the colour of the indicator to blue-black.

Use your data and the equations on page 2 to carry out the following calculations.

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of thiosulfate ions, S₂O₃²⁻ used in each experiment in (a).

$$n = CV$$

$$= 0.0060 \times \frac{20}{1000}$$

moles S₂O₃²⁻ = ... 1.20 × 10⁻⁴ mol

- (ii) Calculate the number of moles of iodine, I₂, that react with the number of moles of S₂O₃²⁻ in (i).

mole ratio S₂O₃²⁻ : I₂

$$2 : 1$$

$$1.20 \times 10^{-4} : x$$

moles I₂ = ... 6.00 × 10⁻⁵ mol

- (iii) Calculate the number of moles of iron(III) ions, Fe³⁺, that were used to produce the number of moles of iodine in (ii).

mole ratio Fe³⁺ : I₂

$$2 : 1$$

$$x : 6.00 \times 10^{-5}$$

moles Fe³⁺ = ... 1.20 × 10⁻⁴ mol

- (iv) When the moles of Fe³⁺ that you calculated in (iii) reacted, a change in the concentration of moles of Fe³⁺ occurred. Calculate this change in concentration.

$$C = \frac{n}{V} = \frac{1.20 \times 10^{-4}}{60/1000} = 2.00 \times 10^{-3}$$

change in concentration of Fe³⁺(aq) = ... 2.00 × 10⁻³ mol dm⁻³

- (v) The following formula can be used as a measure of the 'rate of reaction'.

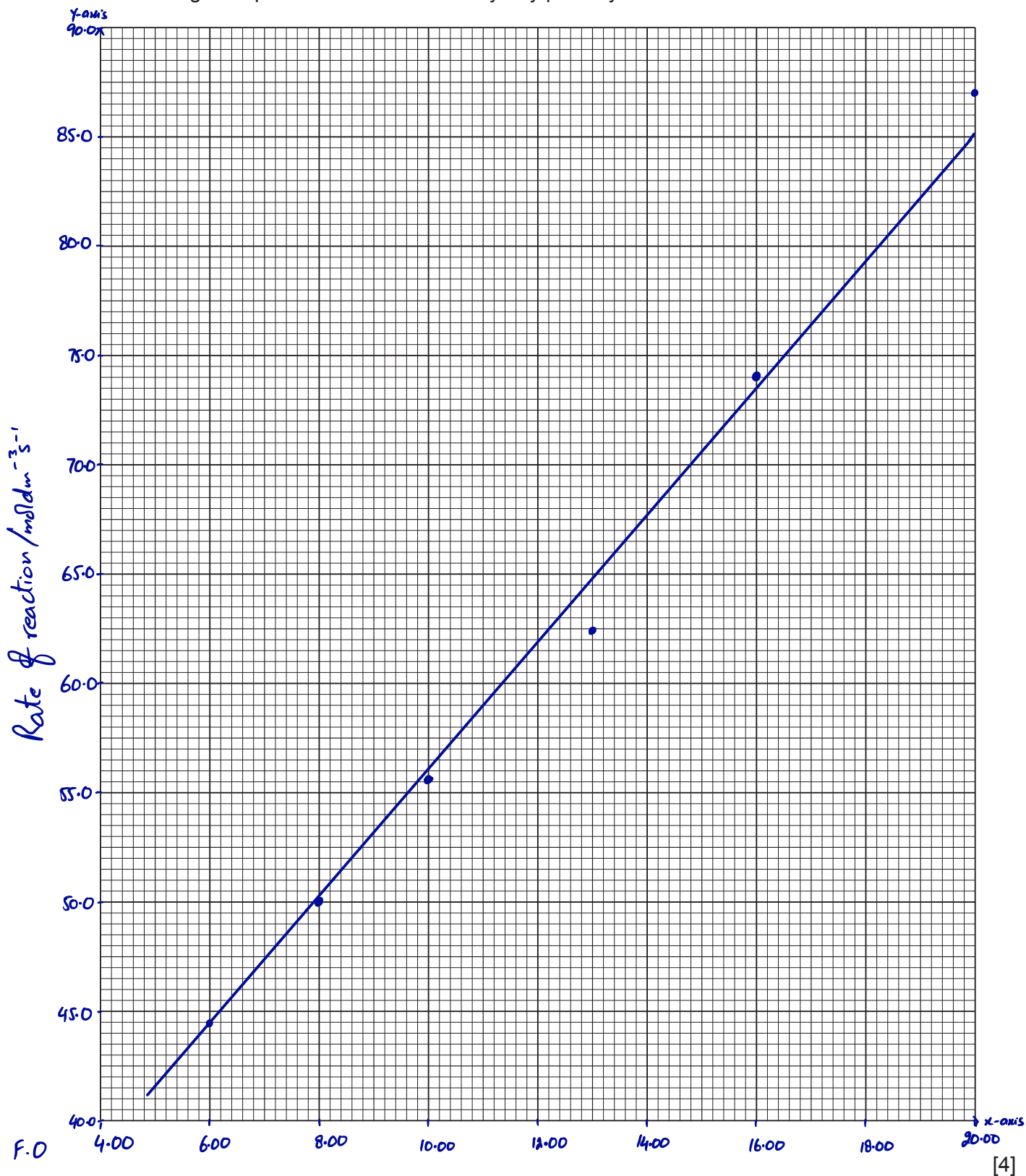
$$\text{'rate of reaction'} = \frac{\text{change in concentration of Fe}^{3+}(\text{aq})}{\text{reaction time}} \times 10^6$$

Complete the table to show the volume of **FB 1**, the reaction time and the rate in **Experiments 1-6**. You should include units.

If you were unable to calculate a value for the change in concentration of Fe³⁺(aq) in (iv), you should assume it is 2.50 × 10⁻³ mol dm⁻³. (Note: this is not the correct value.)

Experiment	Volume of FB1 / cm ³	Reaction Time / s	Rate of reaction / mol.dm ⁻³ .s ⁻¹
1	20.00	23	87.0
2	10.00	36	55.6
3	6.00	45	44.4
4	8.00	40	50.0
5	13.00	32	62.5
6	16.00	27	74.1

(c) On the grid, plot the rate (y-axis) against the volume of **FB 1** (x-axis). Draw a line of best fit through the points. You should identify any points you consider anomalous.



Volume of FB 1 / cm³

- (d) Using your graph, what conclusion can you reach about the effect of changing the concentration of FeCl_3 on the rate of the reaction between $\text{Fe}^{3+}(\text{aq})$ and $\text{I}^{-}(\text{aq})$?

Rate of reaction increases as concentration of Fe^{3+} increases.
Straight line shows rate is proportional to concentration
or volume. [2]

- (e) A student wanted to investigate how changing the concentration of I^{-} would affect the rate of reaction. Explain how this investigation could be carried out.

Alter volume of FB_2/KI whilst keeping other volume of FB_1 constant, add water to keep total volume constant.
[2]

- (f) It was found, by carrying out experiments similar to those used in (a), that increasing the concentration of I^{-} increased the rate of the reaction.

The student suggested modifications to the method as used in (a). In each case, state what the effect would be on the **reaction time** in Experiment 1 and explain how these changes would affect the **possible errors** in the measurements.

Suggested modification 1

The reaction was carried out using the same volumes of all reagents but with the concentrations of **FB 1** and **FB 2** being double their original values.

Reaction time will be less.
Less accurate since greater percentage error in time.

Suggested modification 2

The reaction was carried out using half the volume of all reagents.

Reaction time will stay same.
Less accurate since greater percentage error in volume.

[4]

- (g) (i) Which of the experiments you carried out in (a) had the greatest percentage error in the reaction time?

Experiment #01, as it has shortest reaction time.

- (ii) Calculate this percentage error. Assume that the error in measuring the reaction time is ± 0.5 s.

$$\frac{0.5}{23} \times 100$$

percentage error = 2.17 %
[2]

[Total: 27]

RANDOM PAPERS

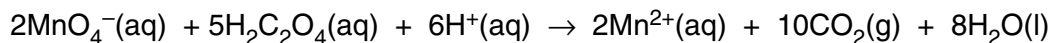
EXPERIMENT NO. 32

FA 1 is a solution containing 5.00 g dm^{-3} of hydrated ethanedioic acid, $\text{H}_2\text{C}_2\text{O}_4 \cdot x\text{H}_2\text{O}$.

FA 2 is a solution containing 2.37 g dm^{-3} of potassium manganate(VII), KMnO_4 .

You are also provided with 1.00 mol dm^{-3} sulphuric acid, H_2SO_4 .

In the presence of acid, potassium manganate(VII) oxidises ethanedioic acid;



You are to determine the value of x in $\text{H}_2\text{C}_2\text{O}_4 \cdot x\text{H}_2\text{O}$.

(a) Fill the burette with **FA 2**.

Pipette 25.0 cm^3 of **FA 1** into a conical flask. Use the measuring cylinder provided to add to the flask 25 cm^3 of 1.00 mol dm^{-3} sulphuric acid and 40 cm^3 of distilled water.

Heat the solution in the flask until the temperature is just over 65°C . The exact temperature is not important.

Be careful when handling hot solutions.

Remove the thermometer and carefully place the hot flask under the burette. If the neck of the flask is too hot to hold safely, use a folded paper towel to hold the flask. Run in about 1 cm^3 of **FA 2**. Swirl the flask until the colour of the manganate(VII) ions has disappeared then continue the titration as normal until a permanent pale pink colour is obtained. This is the end point. Record the burette readings in Table 1.1.

If a brown colour appears during the titration, reheat the flask to 65°C . The brown colour should disappear and the titration can then be completed.

If the brown colour does **not** disappear on reheating, discard the solution and restart the titration.

Repeat the titration as many times as you think necessary to obtain accurate results.

Make certain that the recorded results show the precision of your practical work.

Table 1.1 Titration of FA 1 with FA 2

final burette reading / cm^3	25.40	35.50	38.50	
initial burette reading / cm^3	0.00	10.30	13.40	
volume of FA 2 used / cm^3	25.40	25.20	25.10	
		✓	✓	

Summary

$$\frac{25.20 + 25.10}{2}$$

25.0 cm^3 of **FA 1** reacted with ... 25.15 ... cm^3 of **FA 2**.

Show which results you used to obtain this volume of **FA 2** by placing a tick (✓) under the readings in Table 1.1.

[7]

You are advised to show full working in all parts of the calculations.

- (b) Calculate how many moles of potassium manganate(VII), KMnO_4 , were run from the burette during the titration.

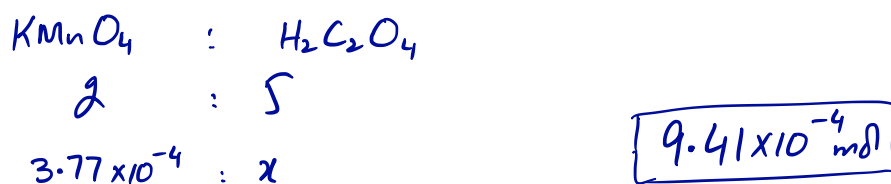
[A_r : K, 39.1; Mn, 54.9; O, 16.0.]

$$n = \frac{m}{M_r} = \frac{2.37}{158} = 0.0150 \text{ mol dm}^{-3}$$

$$n = cV = 0.0150 \times \frac{25.10}{1000} = 3.77 \times 10^{-4} \text{ mol}$$

[2]

- (c) Calculate how many moles of ethanedioic acid, $\text{H}_2\text{C}_2\text{O}_4$, reacted with the potassium manganate(VII) run from the burette.



[1]

- (d) Calculate the mass of $\text{H}_2\text{C}_2\text{O}_4$ in each dm^3 of FA 1

[A_r : H, 1.0; C, 12.0; O, 16.0.]

$$c = \frac{n}{V} = \frac{9.41 \times 10^{-4}}{25.0/1000} = 0.0377 \text{ mol dm}^{-3}$$

$$0.0377 \text{ mol dm}^{-3} = \frac{g \cdot \text{dm}^{-3}}{M_r}$$

$$0.0377 = \frac{g \cdot \text{dm}^{-3}}{90}$$

$$3.39 \text{ g dm}^{-3}$$

[3]

- (e) Calculate the mass of water in the 5.00 g of $\text{H}_2\text{C}_2\text{O}_4 \cdot x\text{H}_2\text{O}$.

$$5.00 - 3.39 = 1.61 \text{ g}$$

[1]

- (f) Calculate the value of x , in $\text{H}_2\text{C}_2\text{O}_4 \cdot x\text{H}_2\text{O}$.

$$\text{H}_2\text{C}_2\text{O}_4 = \frac{3.39}{90} = 0.0377$$

$$\text{H}_2\text{O} = \frac{1.61}{18} = 0.0894$$

$$\begin{array}{ccc} \text{H}_2\text{C}_2\text{O}_4 & & \text{H}_2\text{O} \\ 0.0377 & \text{---} & 0.0894 \\ 1 & \text{---} & x \\ \hline 2.37 & \approx & 2 \end{array}$$

[1]

[Total: 15]

EXPERIMENT NO. 33

In this experiment you will determine the concentration of a solution of sulfuric acid by titration.

FA 1 is sulfuric acid, H_2SO_4 .

FA 2 is aqueous sodium hydroxide, containing 4.20 g NaOH dissolved in 1.00 dm^3 of water.
methyl orange indicator

(a) Method

Dilution of FA 1

- Use a measuring cylinder to measure 10.0 cm^3 of **FA 1** into the 250 cm^3 volumetric flask.
- Make the solution up to the mark using distilled water.
- Shake the flask thoroughly.
- This diluted solution of sulfuric acid is **FA 3**. Label the flask **FA 3**.

Titration

- Fill the burette with **FA 2**.
- Pipette 25.0 cm^3 of **FA 3** into a conical flask.
- Add a few drops of methyl orange indicator.
- Perform a rough titration and record your burette readings in the space below. The end point is reached when the solution turns a permanent pale yellow colour.

Final burette reading/ cm^3	25.60
Initial burette reading/ cm^3	0.00
Volume of FA2 used/ cm^3	25.60

The rough titre is 25.60 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 2** added in each accurate titration.

Final burette reading/ cm^3	39.30	33.70	40.50	
Initial burette reading/ cm^3	14.00	8.50	15.20	
Volume of FA2 used/ cm^3	25.30	25.20	25.30	
	✓		✓	

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VII	

[7]

- (b) From your accurate titration results, obtain a suitable value for the volume of **FA 2** to be used in your calculations.
Show clearly how you obtained this value.

$$\frac{25.30 + 25.30}{2}$$

25.0 cm³ of **FA 3** required 25.30 cm³ of **FA 2**. [1]

(c) **Calculations**

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of sodium hydroxide present in the volume of **FA 2** calculated in (b).

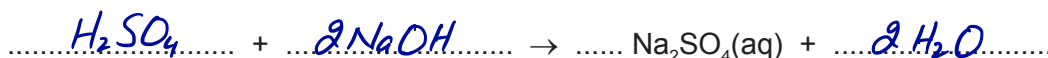
Mr of NaOH is 40.

$$n = \frac{m}{M_r} = \frac{4.20}{40} = \boxed{0.105 \text{ mol dm}^{-3}}$$

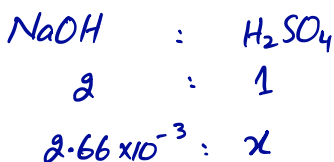
$$n = cV = 0.105 \times \frac{25.30}{1000}$$

moles of NaOH = 2.66 x 10⁻³ mol

- (ii) Complete the equation for the reaction of sulfuric acid with sodium hydroxide. State symbols are required.



- (iii) Use your answers to (i) and (ii) to calculate the number of moles of sulfuric acid used in each titration.



moles of H₂SO₄ = 1.33 x 10⁻³ mol

- (iv) Calculate the concentration, in mol dm⁻³, of sulfuric acid in **FA 3**.

$$c = \frac{n}{V} = \frac{1.33 \times 10^{-3}}{25.0/1000}$$

concentration of H₂SO₄ in **FA 3** = 0.0532 mol dm⁻³

- (v) Calculate the concentration, in mol dm⁻³, of sulfuric acid in **FA 1**.

$$\begin{array}{l} c_1 V_1 = c_2 V_2 \\ c_1 \times \frac{10.0}{1000} = 0.0532 \times \frac{25.0}{1000} \end{array}$$

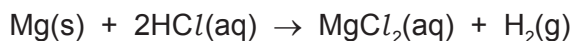
concentration of H₂SO₄ in **FA 1** = 1.33 mol dm⁻³

[5]

[Total: 13]

EXPERIMENT NO. 34

You will determine the enthalpy change, ΔH , of the reaction between magnesium and hydrochloric acid. To do this you will measure the change in temperature when a piece of magnesium ribbon reacts with an excess of hydrochloric acid.



FA 1 is hydrochloric acid, HCl.

FA 2 is magnesium ribbon, Mg.

(a) Method

- Weigh the **FA 2** and record the mass in the space below.
- Support the plastic cup in the 250 cm³ beaker.
- Coil the **FA 2** so that it will fit into the bottom of the plastic cup then remove it.
- Use the measuring cylinder to transfer 25 cm³ of **FA 1** into the plastic cup.
- Place the thermometer in the acid and tilt the cup if necessary so that the bulb of the thermometer is fully covered. Record the temperature at time = 0 in the table of results.
- Start timing and do not stop the clock until the whole experiment has been completed at time = 8 minutes.
- Record the temperature of the acid every half minute for 2 minutes.
- At time = 2 $\frac{1}{2}$ minutes carefully drop the coil of **FA 2** into the acid and stir the mixture.
- Record the temperature of the mixture at time = 3 minutes and complete the table by recording the temperature every half minute. Stir the mixture between thermometer readings.

Results

mass of **FA 2** = 0.21 g

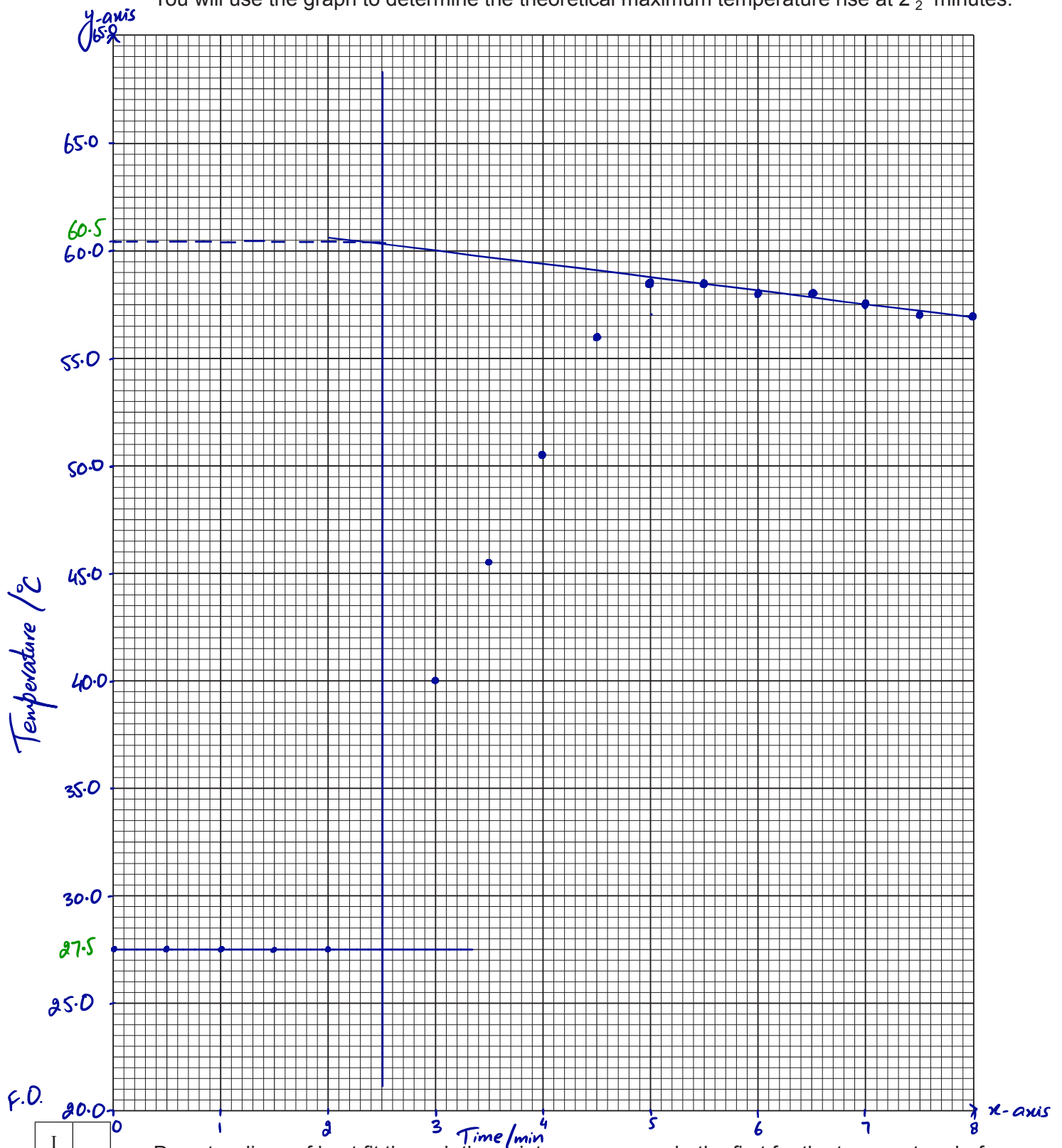
time / minutes	0	$\frac{1}{2}$	1	$1\frac{1}{2}$	2	$2\frac{1}{2}$	3	$3\frac{1}{2}$	4
temperature / °C	<u>27.5</u>	<u>27.5</u>	<u>27.5</u>	<u>27.5</u>	<u>27.5</u>		<u>40.0</u>	<u>45.5</u>	<u>50.5</u>

time / minutes	$4\frac{1}{2}$	5	$5\frac{1}{2}$	6	$6\frac{1}{2}$	7	$7\frac{1}{2}$	8
temperature / °C	<u>56.0</u>	<u>58.5</u>	<u>58.5</u>	<u>58.0</u>	<u>58.0</u>	<u>57.5</u>	<u>57.0</u>	<u>57.0</u>

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[4]

- (b) Plot a graph of temperature on the y-axis against time on the x-axis on the grid below. The scale for the temperature axis should extend 10 °C greater than the maximum temperature you recorded. You will use the graph to determine the theoretical maximum temperature rise at 2½ minutes.



I	
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Draw two lines of best fit through the points on your graph, the first for the temperature before adding **FA 2** and the second for the cooling of the mixture once the reaction is complete. Extrapolate the two lines to 2½ minutes, draw a vertical line between the two and determine the theoretical rise in temperature.

$$60.5 - 27.5$$

theoretical rise in temperature at 2½ minutes = 33.0 °C [4]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Use your answer to (b) to calculate the heat energy, in joules, given out when **FA 2** is added to **FA 1**.

(Assume 4.2 J of heat energy raises the temperature of 1.0 cm³ of the mixture by 1.0 °C.)

$$= mc \Delta T$$
$$= 25 \times 4.2 \times 33.0$$

heat energy evolved = 3465 J

- (ii) Use your answer to (i) and Ar of Mg (24.3) to calculate the enthalpy change, in kJ mol⁻¹, when 1 mole of magnesium, **FA 2**, reacts with hydrochloric acid, **FA 1**.

$$\text{Mg} = \frac{0.21}{24.3}$$
$$= 8.64 \times 10^{-3} \text{ mol}$$

$$\left. \begin{array}{l} 8.64 \times 10^{-3} \text{ mol} \quad \text{---} \quad 3465 \\ 1 \text{ mol} \quad \quad \quad \text{---} \quad x \end{array} \right\} = \frac{401642 \text{ J mol}^{-1}}{1000}$$

enthalpy change = - 401 kJ mol⁻¹
(sign) (value)

[3]

- (d) A student carried out the same procedure using the same concentration of sulfuric acid, H₂SO₄, instead of hydrochloric acid. Before starting the experiment the student predicted that the enthalpy change would be twice that with hydrochloric acid.

Was the student correct? Explain your answer.

Incorrect, as acid is already in excess, so there will be no change in ΔT. [1]

- (e) The enthalpy change determined in (c)(ii) is only an approximation of the actual value.

Suggest and explain one improvement you would make to the method in (a) to increase the accuracy of the experiment.

Use a lid or lagging to prevent heat loss. OR
Use a burette or pipette to measure FA1. [1]

[Total: 13]

EXPERIMENT NO. 35

For
Examiner's
Use

- 1 The identity of a metal, M, can be found by titrating a solution of its carbonate with hydrochloric acid of known concentration.

FA 1 is $0.950 \text{ mol dm}^{-3}$ hydrochloric acid, HCl.

FA 3 is a solution of the metal carbonate, M_2CO_3 , of concentration 6.90 gdm^{-3}

(a) Method

Dilution of the acid

- Pipette 25.0 cm^3 of **FA 1** into the 250 cm^3 volumetric (graduated) flask labelled **FA 4**.
- Add distilled water to make the total volume 250 cm^3 .
- Stopper the flask and mix the contents thoroughly.

Titration

- Fill the burette with diluted hydrochloric acid, **FA 4**.
- Use a clean pipette to transfer 25.0 cm^3 of **FA 3** into a conical flask.
- Titrate **FA 3** with **FA 4** using the indicator provided.
- Perform a rough titration and record your burette readings in the space below.

Final burette reading/ cm^3	24.90
Initial burette reading/ cm^3	0.00
Volume of FA4 used/ cm^3	24.90

The rough titre is24.90..... cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain 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 4** added in each accurate titration.

Final burette reading/ cm^3	24.50	49.10	25.00	
Initial burette reading/ cm^3	0.00	24.50	0.50	
Volume of FA4 used/ cm^3	24.50	24.60	24.50	
	✓		✓	

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[7]

- (b) From your accurate titration results, obtain a suitable value to be used in your calculations. Show clearly how you obtained this value.

$$\frac{24.50 + 24.50}{2}$$

25.0 cm³ of **FA 3** required 24.50 cm³ of **FA 4**
[1]

(c) Calculation

The equation for the reaction between hydrochloric acid and the metal carbonate is given below.



- (i) Calculate the number of moles of hydrochloric acid present in the volume in (b).

$$C_1 V_1 = C_2 V_2$$

$$0.0950 \times \frac{25.0}{1000} = C_2 \times \frac{25.0}{1000}$$

$$C_2 = 0.0950 \text{ mol dm}^{-3}$$

$$n = CV$$

$$= 0.0950 \times \frac{24.50}{1000}$$

moles of HCl = 2.33 × 10⁻³ mol

- (ii) Hence, calculate the number of moles of M₂CO₃ present in 25.0 cm³ of **FA 3**.

mole ratio M₂CO₃ : HCl

$$1 : 2$$

$$x : 2.33 \times 10^{-3}$$

moles of M₂CO₃ = 1.16 × 10⁻³ mol

- (iii) Calculate the concentration of M₂CO₃ in **FA 3** in mol dm⁻³.

$$C = \frac{n}{V} = \frac{1.16 \times 10^{-3}}{25.0/1000}$$

concentration of M₂CO₃ = 0.0464 mol dm⁻³

- (iv) Use your answer to (iii), and the fact that **FA 3** contains 6.90 g dm⁻³, to determine the relative atomic mass, A_r, of M.

$$\text{mol dm}^{-3} = \frac{\text{g dm}^{-3}}{M_r}$$

$$0.0464 = \frac{6.90}{M_r}$$

$$M_r = 148.7$$

$$148.7 - 60 = \frac{88.7}{2} = 44.4$$

A_r of M = 44.4

- (v) Use your answer to (iv) and the Periodic Table to suggest the identity of M.



M is K [5]

EXPERIMENT NO. 36

- 1 In this experiment you will determine the identity of the Group 2 metal, **X**, in the carbonate, **XCO₃**. To do this you will react a known mass of **XCO₃** with **excess** hydrochloric acid, **HCl**, and measure the mass of carbon dioxide that is given off.

FA 1 is **XCO₃**.

FA 2 is hydrochloric acid, **HCl**.

(a) Method

- Weigh the stoppered tube containing **FA 1** and record its mass.
- Use the measuring cylinder to transfer 25 cm³ of **FA 2** into the 250 cm³ beaker.
- Weigh the beaker containing the acid and record the mass.
- Carefully add all the sample of **FA 1** to the acid in the beaker.
- Stir the mixture until there is no further reaction.
- Reweigh the beaker and its contents and record the mass.
- Reweigh the stoppered tube containing any residual **FA 1** and record its mass.
- Calculate the mass of **FA 1** added to the acid and record this value.
- Calculate the mass of carbon dioxide given off and record this value.

mass of tube + FA1 /g	23.30
mass of tube + residue /g	21.30
mass of FA1 used /g	2.00
mass of beaker + acid /g	126.30
mass of beaker + acid + FA1 before reaction/g	128.30
mass of beaker + acid + FA1 after reaction/g	127.40
mass of CO ₂ given off /g	0.90

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[7]

(b) Calculations

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

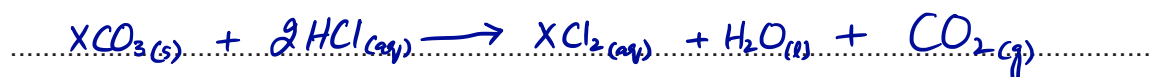
- (i) Calculate the number of moles of carbon dioxide given off when **XCO₃** reacted with the acid.

Use the data in the Periodic Table.

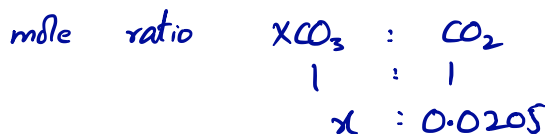
$$n = \frac{m}{M_r} = \frac{0.90}{44} = 0.0205 \text{ mol}$$

moles of CO₂ = 0.0205 mol

- (ii) Write the equation for the reaction of **FA 1**, **XCO₃**, with hydrochloric acid, **HCl**. Include state symbols.



- (iii) Use your answers to (i) and (ii) to calculate the number of moles of XCO_3 that were added to the acid.



moles of $\text{XCO}_3 = 0.0205$ mol

- (iv) Use your answer to (iii) to calculate the relative atomic mass, A_r , of X . Identify X .

$$n = \frac{m}{M_r}$$

$$0.0205 = \frac{2.00}{M_r}$$

$$M_r \text{ of } \text{XCO}_3 = 97.6$$

$97.6 - 60 = 37.6$ $\text{CO}_3 = 60$

I	
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A_r of $\text{X} = 37.6$

X is Ca

[5]

- (c) One of the sources of error in this experiment is that it is very difficult to reduce acid spraying out of the beaker when the metal carbonate is added to the acid.

- (i) Explain what effect this acid spray would have on the value you calculated for the relative atomic mass, A_r , of X .

A_r would be shorter, as acid spray will lead to greater mass of CO_2 so greater number of moles of CO_2 .

- (ii) Why is a small amount of acid spray not likely to cause an error in the identification of X ?

Small loss in mass so not much difference in A_r so does not cause confusion in identity of X .

- (iii) How could you minimise acid spraying out of the beaker?

- * Use conical flask / taller beaker OR
- * Add solid little at a time / slowly OR
- * Use less solid or less concentrated acid.

[3]

[Total: 15]

EXPERIMENT NO. 37

- 1 In this experiment you will determine the relative atomic mass of magnesium by thermal decomposition of hydrated magnesium sulfate.



FB 4 is hydrated magnesium sulfate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$.

(a) Method

Record all your weighings in the space below.

- Weigh the crucible with its lid.
- Transfer all **FB 4** into the crucible.
- Weigh the crucible, lid and **FB 4**.
- Place the crucible on the pipe-clay triangle.
- Heat the crucible gently with the lid **on**, for about one minute.
- Then heat the crucible strongly, without the lid, for a further four minutes.
- Leave the crucible and its contents to cool with the lid on, for several minutes.
- When the crucible has cooled, weigh it, with the lid and contents.
- Calculate and record the mass of anhydrous magnesium sulfate produced and the mass of water lost.

mass of crucible/g	35.07
mass of crucible + FB4 /g	37.44
mass of crucible + FB4 after heating/g	36.23
mass of anhydrous MgSO_4 /g	1.16
mass of H_2O lost /g	1.21

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III	

[3]

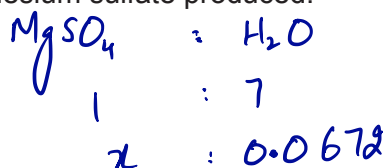
(b) Calculations

- (i) Calculate the number of moles of water lost during heating.

$$\frac{1.21}{18} =$$

moles of H_2O = 0.0672 mol

- (ii) Use the **equation above** and **your answer to (i)** to calculate the number of moles of anhydrous magnesium sulfate produced.



moles of MgSO_4 = 9.60×10^{-3} mol

- (iii) Use your weighings and your answer to (ii) to calculate the relative formula mass, M_r , of anhydrous magnesium sulfate.

$$n = \frac{m}{M_r}$$
$$9.60 \times 10^{-3} = \frac{1.16}{M_r}$$

$$M_r = 120.8$$

$$M_r \text{ of } \text{MgSO}_4 = 120.8$$

- (iv) From your answer to (iii), calculate the relative atomic mass, A_r , of magnesium.

$$\text{SO}_4 = 32.1 + 64$$
$$= 96.1$$

$$120.8 - 96.1 = 24.7$$

$$A_r \text{ of Mg} = 24.7$$

[4]

- (c) (i) How could the experiment be improved to ensure that the magnesium sulfate had been completely dehydrated?

Reheat until we get constant mass of crucible and
FB4.

- (ii) Why is the lid put on the crucible during cooling?

To prevent addition of moisture in anhydrous
magnesium sulfate.

[2]

[Total: 9]

EXPERIMENT NO. 38

You are to determine the enthalpy change of solution, in kJ mol^{-1} , of ammonium chloride.

FB 3 is approximately 9 g of solid ammonium chloride, NH_4Cl .
distilled water

(a) Method

Read through the instructions carefully and prepare a table below for your results before starting any practical work.

- Weigh the plastic cup and record the balance reading.
- Add between 2.9 and 3.1 g of ammonium chloride, **FB 3**, and record the new balance reading.
- Place the plastic cup in the 250 cm^3 beaker.
- Pour 25 cm^3 of distilled water into the measuring cylinder.
- Place the thermometer in the water and record the initial temperature in the table of results.
- Pour the 25 cm^3 of distilled water into the plastic cup.
- Stir the contents of the cup and record the lowest temperature of the solution. Tilt the cup if necessary to ensure the thermometer bulb is fully immersed.
- Repeat the procedure using the other plastic cup. Use between 4.9 and 5.1 g of **FB 3** for this experiment.
- Record the mass of **FB 3** used and the change in temperature for **each** experiment.

Results

	Experiment # 1	Experiment # 2
mass of plastic cup/g	2.14	2.14
mass of plastic cup + FB3/g	5.14	7.14
mass of FB3 used/g	3.00	5.00
initial temperature / $^{\circ}\text{C}$	28.0	28.5
final temperature / $^{\circ}\text{C}$	20.0	15.5
change in temperature / $^{\circ}\text{C}$	8.0	13.0

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[5]

- (b) (i) Calculate the mean mass of **FB 3** used. Give your answer to **two** decimal places.

$$\frac{3.00 + 5.00}{2}$$

mean mass of **FB 3** = 4.00 g

- (ii) Calculate the mean temperature change. Give your answer to **one** decimal place.

$$\frac{8.0 + 13.0}{2}$$

mean temperature change = 10.5 °C
[2]

(c) **Calculations**

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

- (i) Calculate the number of moles of NH_4Cl present in the mass of **FB 3** calculated in (b)(i).
[A_r: H, 1.0; N, 14.0; Cl, 35.5]

$$n = \frac{m}{M_r} = \frac{4.00}{53.5} = 0.0748$$

moles of NH_4Cl = 0.0748 mol

- (ii) Use your answers to (b)(ii) and (c)(i) to calculate the enthalpy change of solution, in kJ mol^{-1} , of NH_4Cl .
(Assume that **4.2 J** of heat energy changes the temperature of 1.0 cm^3 of solution by 1.0°C .)

$$Q = mc\Delta T = 25.0 \times 4.2 \times 10.5 = 1103 \text{ J or } 1.10 \text{ kJ}$$

$$\left. \begin{array}{l} 0.0748 \text{ mol} \text{ --- } 1.10 \text{ kJ} \\ 1 \text{ mol} \text{ --- } x \\ \hline T = 14.7 \text{ kJ mol}^{-1} \end{array} \right\}$$

enthalpy change = + 14.7 kJ mol^{-1}
(sign) (value)

[4]

- (d) (i) Which of the two experiments in (a), the first or the second, had the greater percentage error in the value calculated for the temperature change?

Temperature change of experiment 1

- (ii) Given that the error in a single thermometer reading is $\pm 0.5^\circ\text{C}$, calculate the percentage error in the temperature change for the experiment you gave in (d)(i).

$$\frac{0.5 \times 2}{8.0} \times 100$$

$$\% \text{ error} = \frac{\text{error}}{\text{value}} \times 100$$

percentage error = 12.5 %
[1]

- (e) (i) A student suggested that it would have been better to calculate the enthalpy change for each experiment separately. What would be the advantage of this suggestion? Explain your answer fully.

To check whether further experiments should be carried out. OR
To find out whether mass added is proportional to ΔT . OR
To compare the two answers.

- (ii) Another student suggested that the procedure would be improved by covering the plastic cup with a lid. Explain whether this would improve the procedure.

No improvement as temperature drops so no heat loss as reaction is endothermic. OR Improvement as prevents heat gain from surroundings. [2]

[Total: 14]

EXPERIMENT NO. 39

Qualitative Analysis

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.

No additional tests for ions present should be attempted.

If any solution is warmed, a boiling tube MUST be used.

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

Where reagents are selected for use in a test, the name or correct formula of the element or compound must be given.

- (a) A sample of limestone was reacted with dilute nitric acid to give solution **FB 6**. This sample of limestone contained calcium carbonate, CaCO_3 , and one other salt. This additional salt contains a single cation and a single anion. By carrying out the following tests you will be able to suggest identities of the additional ions.

test	observations
(i) To a 1 cm depth of FB 6 in a test-tube add aqueous ammonia.	white ppt. insoluble in excess
(ii) To a 1 cm depth of FB 6 in a test-tube add a 1 cm depth of aqueous silver nitrate.	no change
(iii) To a 1 cm depth of FB 6 in a test-tube add a 1 cm depth of aqueous barium chloride or barium nitrate.	white ppt.

(iv) Suggest all possible identities for the ions present in **FB 6**, apart from Ca^{2+} and NO_3^- .

Mg^{2+} or Al^{3+} and SO_4^{2-} or SO_3^{2-}

(v) Select a reagent to use in a further test on **FB 6** to confirm that one of the cations you have listed in (iv) is **not** present in **FB 6**. Carry out your test and complete the table.

test	observations	conclusion
To a 1 cm depth of FB 6 in a test-tube add add aq. NaOH	white ppt. insoluble in excess	Mg^{2+} is present Al^{3+} is absent

I	
II	
III	
IV	
V	
VI	

[6]

(b) You are provided with a solid, **FB 7**. By carrying out the following tests you will be able to identify three of the ions present in **FB 7**.

test	observations
(i) Place a spatula measure of FB 7 in a hard-glass test-tube. Heat gently at first, then	no change
heat more strongly until no further change is seen, then	solid turns yellow
allow the tube to cool.	solid turns yellow to white

I	
II	
III	
IV	
V	
VI	
VII	

test	observations
<p>(ii) Place a spatula measure of FB 7 in a test-tube.</p> <p>Add about a 5 cm depth of dilute nitric acid.</p> <p>You will use the solution formed for tests (iii) to (v).</p>	<p>effervescence of colorless gas which formed white ppt. with lime water.</p> <p>solid dissolves form a yellow solution</p>
<p>(iii) To a 1 cm depth of the solution in a test-tube add a few drops of aqueous potassium manganate(VII), then</p>	<p>solution turns orange brown</p>
<p>add a few drops of starch solution.</p>	<p>solution turns blue-black</p>
<p>(iv) To a 1 cm depth of the solution in a test-tube add a few drops of aqueous silver nitrate.</p>	<p>yellow ppt. formed</p>
<p>(v) To a 1 cm depth of the solution in a test-tube add aqueous ammonia.</p>	<p>white ppt.</p> <p>soluble in excess</p>

Use the Qualitative Analysis Notes to identify **three** of the ions present.

CO_3^{2-} , I^- and Zn^{2+}

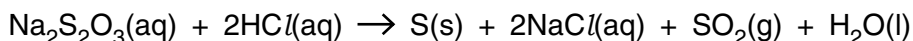
[7]

[Total: 13]

EXPERIMENT NO. 40

- 1 You are to investigate how the rate of formation of sulfur varies with the concentration of sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$, in the reaction below.

For
Examiner's
Use



Care should be taken to avoid inhalation of $\text{SO}_2(\text{g})$ that is given off during this reaction.

You are provided with the following.

- FA 1**, $0.15 \text{ mol dm}^{-3} \text{ Na}_2\text{S}_2\text{O}_3$ a measuring cylinder to measure 50 cm^3
FA 4, $2.0 \text{ mol dm}^{-3} \text{ HCl}$ a measuring cylinder or marked tube to measure 5 cm^3
 a printed insert a stop clock or clock with seconds hand

(a) Method – Read through the instructions before starting any practical work.

- Using the larger measuring cylinder transfer 50 cm^3 of **FA 1** into a 250 cm^3 beaker.
- Measure 5 cm^3 of **FA 4** in the smaller measuring cylinder.
- Tip the **FA 4** into the **FA 1** in the beaker and **immediately** start timing.
- Swirl the beaker to mix the solution and place it on top of the printed insert.
- View the printed insert from above so that it is seen through the solution.
- Note the time when the printing on the insert just disappears.

- Empty and rinse the beaker. Shake out as much of the rinse water as possible and dry the outside of the beaker.
- Repeat the experiment using 25 cm^3 of **FA 1** and 25 cm^3 of distilled water. Add 5 cm^3 of **FA 4** to start the reaction.

- Select suitable volumes of **FA 1** and distilled water for **one** further experiment to investigate the effect of sodium thiosulfate concentration on the rate of reaction. *Remember to use 5 cm^3 of **FA 4** and to keep the total volume of **FA 1** and distilled water constant.*

In an appropriate form record the following below:

- all measurements of volume and time (to the nearest second) for each experiment,
- calculated values of $1/t_{\text{time}}$ which are a measure of the rate of reaction.

Results

Experiment no.	Volume of FA1/ cm^3	Volume of $\text{H}_2\text{O}/\text{cm}^3$	Time /s	$1/t_{\text{time}} / \text{s}^{-1}$
01	50	0	25	0.0400
02	25	25	48	0.0208
03	10	40	132	0.00758

i	
ii	
iii	
iv	
v	
vi	
vii	
viii	
ix	

- (b) The total volume in each experiment is constant. Using volumes from the first two experiments, show by simple calculation that the volume of **FA 1** used is a measure of its concentration in the reaction mixture.

$$\frac{50}{55} = \frac{25}{55} \quad \text{Concentration of FA1 decreased by 50\%}$$

[1]

- (c) What is the relationship between the rate of reaction and the time taken?

Rate of reaction is inversely proportional to the reaction time.

[1]

- (d) For **each** experiment calculate the numerical value of (volume of **FA 1** × time).

experiment	(volume of FA 1 × time)/(cm ³ s)
1	1250
2	1200
3	1320

Use your results in (a) and these calculated values to deduce the relationship between the concentration of Na₂S₂O₃ and the rate of formation of sulfur.

Rate of formation of sulfur is independent of concentration of sodium thiosulfate.

[2]

- (e) Outline briefly how you would modify the experimental method to investigate the effect of temperature change on the reaction rate.

Use constant volume of all solutions and change temperature by using thermostatically controlled water bath.

[1]

[Total: 14]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	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 heating	–
barium, Ba ²⁺ (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca ²⁺ (aq)	white ppt. with high [Ca ²⁺ (aq)]	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	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

<i>ion</i>	<i>reaction with</i>
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$)
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$)
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$)
nitrate, $\text{NO}_3^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	NH_3 liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; NO liberated by dilute acids (colourless NO \rightarrow (pale) brown NO_2 in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	SO_2 liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids)

3 Tests for gases

<i>gas</i>	<i>test and test result</i>
ammonia, NH_3	turns damp red litmus paper blue
carbon dioxide, CO_2	gives a white ppt. with limewater (ppt. dissolves with excess CO_2)
chlorine, Cl_2	bleaches damp litmus paper
hydrogen, H_2	"pops" with a lighted splint
oxygen, O_2	relights a glowing splint
sulfur dioxide, SO_2	turns acidified aqueous potassium manganate(VII) from purple to colourless

The Periodic Table of Elements

Group																											
1	2	Key										13	14	15	16	17	18										
		1 H hydrogen 1.0																									
		atomic number atomic symbol name relative atomic mass																									
3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20										
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	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	K potassium 39.1	Ca calcium 40.1										
11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28										
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	K potassium 39.1	Ca calcium 40.1	Sc scandium 45.0	Ti titanium 47.9	V vanadium 50.9	Cr chromium 52.0	Mn manganese 54.9	Fe iron 55.8	Co cobalt 58.9	Ni nickel 58.7										
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54										
Rb rubidium 85.5	Sr strontium 87.6	Y yttrium 88.9	Zr zirconium 91.2	Nb niobium 92.9	Mo molybdenum 95.9	Tc technetium —	Ru ruthenium 101.1	Rh rhodium 102.9	Pd palladium 106.4	Ag silver 107.9	Cd cadmium 112.4	In indium 114.8	Sn tin 118.7	Sb antimony 121.8	Te tellurium 127.6	I iodine 126.9	Xe xenon 131.3										
55	56	57-71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86										
Cs caesium 132.9	Ba barium 137.3	lanthanoids	Hf hafnium 178.5	Ta tantalum 180.9	W tungsten 183.8	Re rhenium 186.2	Os osmium 190.2	Ir iridium 192.2	Pt platinum 195.1	Au gold 197.0	Hg mercury 200.6	Tl thallium 204.4	Pb lead 207.2	Bi bismuth 209.0	Po polonium —	At astatine —	Rn radon —										
87	88	89-103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118										
Fr francium —	Ra radium —	actinoids	Rf rutherfordium —	Db dubnium —	Sg seaborgium —	Bh bohrium —	Hs hassium —	Mt meitnerium —	Ds darmstadtium —	Rg roentgenium —	Cn copernicium —	Nh nihonium —	Fl flerovium —	Lv livermorium —	Ts tennessine —	Og oganesson —	—										

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