

CHEMISTRY LAB MANUAL

2017-18

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						
	Add a further 2cm of depth of aqueous sodium hydroxide						

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								
Add a 2cm of depth of aqueous ammonia								
(b) To 1cm depth of solution add 1cm depth of aqueous sodium hydroxide and a piece of aluminium foil, heat the mixture								

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								
(d) To 1cm depth of solution in a test tube add 1cm depth of barium nitrate Then								

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		
FA2		
FA3		
FA4		
FA5		
FA6		
FA7		
FA8		

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.

I	
II	
III	
IV	
V	

One of the cations in **FA 6** is

(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	
add aqueous ammonia.	
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	
add dilute nitric acid.	

The anions in **FA 6** are and

[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.

<i>test</i>	<i>observations</i>
(i) To a 1 cm depth of FA 7 in a test-tube add aqueous sodium hydroxide and leave for a few minutes.	
(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	
(iii) pour a 1 cm depth of the mixture into a clean boiling tube and add a 3 cm depth of aqueous sodium hydroxide.	

I	
II	
III	
IV	
V	
VI	

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

.....

.....

(v) Explain your observations in **(iii)**.

.....

.....

[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.	
II			
III			
IV		To a 1 cm depth of FA 5 in a test-tube, add FA 1 , aqueous potassium manganate(VII), dropwise.	
		To a 1 cm depth of FA 6 in a test-tube, add FA 1 , aqueous potassium manganate(VII), dropwise.	

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

..... [4]

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

reagent(s)

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.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

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

FA 4 contains

FA 5 contains

FA 6 contains

(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)

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.

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

..... [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.

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**.

.....

[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.

<i>test</i>	<i>observations</i>
(i) Place a small spatula measure of FA 6 in a hard-glass test-tube and heat strongly.	
(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	
add aqueous sodium hydroxide.	
(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).	
(iv) To a 1 cm depth of the solution from (iii) in a test-tube, add aqueous sodium hydroxide.	
(v) To a 1 cm depth of the solution from (iii) in a test-tube, add aqueous ammonia.	
(vi) To a 1 cm depth of the solution from (iii) in a test-tube, add aqueous barium chloride or aqueous barium nitrate.	

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

cations

anions

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

.....

.....

.....

[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.

<i>test</i>	<i>observations</i>	
	FB 4	FB 5
(i) To a 1 cm depth of solution in a test-tube, add aqueous ammonia.		
(ii) To a 1 cm depth of solution in a test-tube, add a few drops of aqueous silver nitrate.		
(iii) To a 1 cm depth of solution in a test-tube add a few drops of aqueous barium nitrate.		

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

cation anion

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

cations anions

(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.

<i>test</i>	<i>observations</i>
To a 1 cm depth of FB 5 in a test-tube, add	

The cation in **FB 5** is

[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**?

.....

Carry out the following tests and complete the table below.

<i>test</i>	<i>observations</i>
(ii) Place a spatula measure of FB 6 in a hard-glass test-tube. Heat gently.	
(iii) Dissolve a small spatula measure of FB 6 in a 2 cm depth of distilled water in a test-tube. Use this solution for tests (iv) and (v).	
(iv) Pour about half the solution prepared in (iii) into a boiling tube and add aqueous sodium hydroxide, then	
gently warm the mixture.	
(v) To the remainder of the solution prepared in (iii), add a few drops of aqueous potassium iodide, then	
add a few drops of starch solution.	

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

FB 6 contains and

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

.....
[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.

<i>test</i>	<i>observations</i>	
	FA 5	FA 6
Add aqueous sodium hydroxide.		
Add aqueous ammonia.		
Add a 1 cm depth of dilute hydrochloric acid, then		
transfer the mixture into a boiling tube and warm gently.		
Add two or three drops of acidified aqueous potassium manganate(VII).		
Add a 1 cm depth of aqueous barium chloride or barium nitrate, then		
add dilute hydrochloric acid.		

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		
anion		

- (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**

.....

observations and conclusions

- (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**

.....

observations and conclusions

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.	
To a 1 cm depth of FA 5 in a test-tube, add aqueous ammonia.	
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,	
then add aqueous potassium manganate(VII) drop by drop.	
To a 1 cm depth of FA 5 in a test-tube, add a 1 cm depth of aqueous potassium iodide,	
followed by a few drops of starch indicator.	

[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 and

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

[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)

reasons(s)

.....

possible anion(s)

reasons(s)

..... [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.

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.

.....

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.....

.....

(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.

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.

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.....

[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.

[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** is

explanation

.....

.....

.....

The cation contained in solution **FB** is

explanation

.....

.....

.....

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

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.....

.....

[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 .	
Add 5 drops of aqueous silver nitrate to your solution of FA 7 .	

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.

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

.....
.....

- (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

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

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

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

.....
.....
.....
..... [9]

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

For
Examiner's
Use

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.

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

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.....
.....

- (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.

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.....
.....
..... [5]

[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.

<i>test</i>	<i>observations</i>
(i) To 1 cm depth of dilute hydrochloric acid in a test-tube add a small spatula measure of FB 5 .	
(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 .	
(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 .	

<i>test</i>	<i>observations</i>
(iv) Place a small spatula measure of FB 6 into a hard glass test-tube. Heat the contents gently.	
(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 .	
(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	
(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 .	

[6]

I	
II	
III	
IV	
V	
VI	

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

.....

- (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

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

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

.....

[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

and

- (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.

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

Fe^{2+} ions are contained in solution

Explain how your observations support your conclusion.

.....
.....

- (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

.....
.....

- (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

.....

[6]

[Total: 15]

I	
II	
III	
IV	
V	
VI	

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)				
(b)				
(c)				
(d)				
(e)				

[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		
FA 5		
FA 6		
FA 7		

[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.				
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.				
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.				

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

.....

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

.....

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

.....

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

.....

- (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.

.....

.....

.....

[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.

<i>test</i>	<i>observations</i>	
	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,		
add a small piece of aluminium foil.		
Place a spatula measure of solid in a hard-glass test-tube. Heat gently at first and then more strongly.		

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

anion or

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

.....

.....

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

observation for **FA 9** anion in **FA 9** is

observation for **FA 10** anion in **FA 10** is

[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 $\text{NO} \rightarrow$ (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

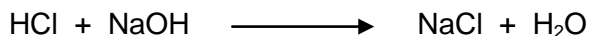
QUANTITATIVE ANALYSIS

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 .

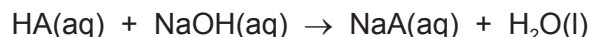
	Rough	1	2	3	4
Final burette reading / cm ³					
Initial burette reading / cm ³					
Volume of HCl added / cm ³					

Questions

- (a) How many moles of NaOH were present in 25.0 cm³ solution?
- (b) How many moles of HCl were present in the volume of acid you used to neutralise the NaOH solution?
- (c) What was the exact concentration of hydrochloric acid in mol dm⁻³?
- (d) Why was the conical flask placed on a piece of white tile?
- (e) Why were the pipette and burette washed with the solutions they were going to contain?
- (f) Why was the conical flask not washed with the alkali solution it was going to contain?
- (g) Explain why it does not matter if there is water already in the flask.
- (h) Explain why a conical flask was used and not a beaker.

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.

The rough titre is 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.

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.

25.0 cm³ of **FA 2** required 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.

moles of NaOH = 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**.

moles of HA = mol

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

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

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

M_r of HA =
[4]

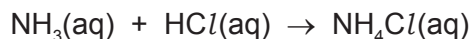
- (d) A student carrying out this method correctly with the same concentration of reactants obtained a titre of 28.30 cm³. Would this give a larger or smaller value of M_r than yours?
Explain your answer.

.....
.....
..... [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.

The rough titre is 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.

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.

25.0 cm^3 of **FB 1** required 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).

moles of HCl = mol

- (ii) Use your answer to (i) to determine the number of moles of ammonia present in 25.0 cm^3 of **FB 1**, pipetted into the conical flask.

moles of NH_3 = mol

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

concentration of NH_3 (diluted) in **FB 1** = 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'.

concentration of '.880 ammonia' = 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.)

percentage difference in concentration = % [1]

[Total: 12]

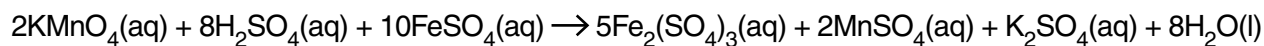
EXPERIMENT NO. 17

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.

The rough titre is 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.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

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

For
Examiner's
Use

25.0 cm³ of **FB 1** required 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.

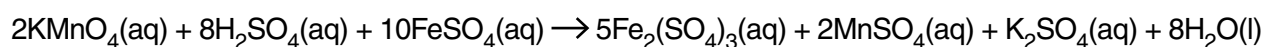
[A_r: O, 16.0; K, 39.1; Mn, 54.9]

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

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

..... mol of KMnO₄.

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



..... 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**.

The concentration of FeSO_4 in **FB 1** is 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]

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

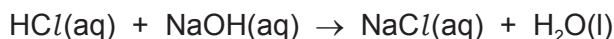
- (vi) **FB 1** is an aqueous solution containing 21.50 g dm^{-3} of FeSO_4 and $\text{Fe}_2(\text{SO}_4)_3$.
Calculate the percentage, by mass, of FeSO_4 in this mixture.

The mixture contains % 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.

The rough titre is 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.

I	
II	
III	
IV	
V	
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.

25.0 cm³ of **FA 1** required 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.

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

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

moles of NaOH = mol

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

moles of HCl = mol

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

concentration of HCl in **FA 2** = 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 cm³** 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 cm³** 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.

The rough titre is cm^3 .

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

I	
II	
III	
IV	
V	
VI	
VII	

[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.

25.0 cm^3 of **FB 1** required 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).

moles of HCl = 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).

moles of borax = mol

- (iii) Use your answer to (ii) to calculate the number of moles of borax in 1.00 dm^3 of **FB 1**.

moles of borax in 1.00 dm^3 **FB 1** = mol

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

M_r of borax =

- (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.

x =
[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.

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.

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.

[1]

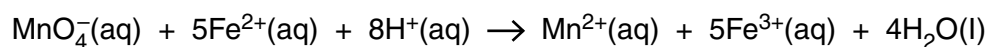
Calculations

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.

..... 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.



..... 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**.

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

Calculate the concentration, in mol dm^{-3} , of Fe^{2+} in **FB 1**.

Concentration of Fe^{2+} in **FB 1** = mol dm^{-3} .

Calculate, to **4 significant figures**, the concentration of $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$ in **FB 1** in g dm^{-3} .

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

i	
ii	
iii	
iv	
v	

FB 1 contains g dm^{-3} of $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$.
[5]

- (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.

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

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

smallest division on burette scale = cm^3

estimated error in reading a volume = \pm cm^3 [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 = \pm cm^3 [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.

.....
.....
.....[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.

The rough titre is cm^3 .

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make sure any recorded results show the precision of your practical work.
- Record in a suitable form below all of your burette readings and the volume of **FA 4** added in each accurate titration.

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.

Volume of **FA 4** required is cm^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 sodium thiosulfate in the volume of **FA 4** calculated in (b).

moles of $\text{Na}_2\text{S}_2\text{O}_3 = \dots\dots\dots \text{mol}$

- (ii) Use the equation below to calculate the number of moles of iodine that reacted with the sodium thiosulfate in the titration.



moles of $\text{I}_2 = \dots\dots\dots \text{mol}$

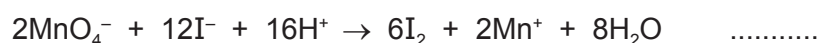
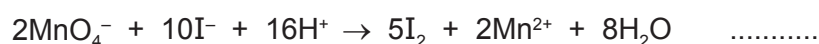
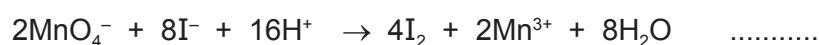
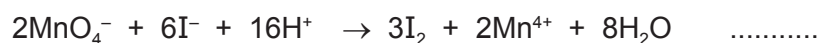
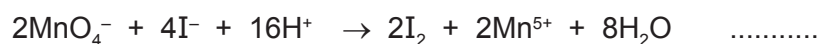
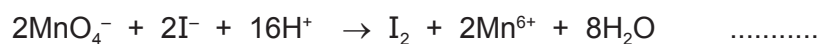
- (iii) Use information on page 2 to calculate the number of moles of potassium manganate(VII) in **FA 1** used in the titration.

moles of $\text{KMnO}_4 = \dots\dots\dots \text{mol}$

- (iv) From your answers to (ii) and (iii), calculate the number of moles of iodine produced by the reaction of **2.00** moles of potassium manganate(VII) with excess potassium iodide.

moles $\text{I}_2 = \dots\dots\dots \text{mol}$

- (v) Using your answer to (iv), put a tick next to the ionic equation that represents the reaction between **FA 1** and **FA 3**.



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

.....

.....

.....

[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.

percentage error = %

- (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.

.....

.....

.....

[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.

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.

The rough titre is 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.

*For
Examiner's
Use*

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 have obtained this value.

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

(c) Calculations

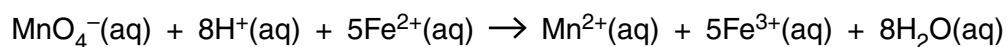
For
Examiner's
Use

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)**.

moles of MnO_4^- (aq) = mol

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



moles of Fe^{2+} (aq) in the conical flask = mol

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

moles of Fe^{2+} in the weighed sample = mol

I	
II	
III	
IV	
V	

- (iv) Calculate the percentage of iron in **FA 1**.
[A_r : Fe, 55.8]

the percentage of iron in **FA 1** = %
[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.

maximum individual error = g

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

maximum percentage error = % [2]

[Total: 16]

THERMAL DECOMPOSITION

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.

I	
II	
III	
IV	
V	

[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]

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

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

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

.....
.....
..... [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 1.80 g and 2.00 g 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.

I	
II	
III	
IV	
V	
VI	

[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**.

percentage loss in mass = %

- (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 = %

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

percentage purity = %
[3]

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

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

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

(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.

.....

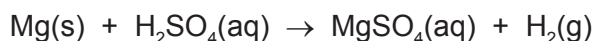
..... [1]

[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

[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)

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

.....

- (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.)

heat energy produced = J

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

enthalpy change = 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.)

heat energy produced = J

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

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

[5]

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

maximum error = °C.

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

.....

- (iii) Calculate this maximum percentage error.

maximum percentage error in the temperature change = %
[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.

.....

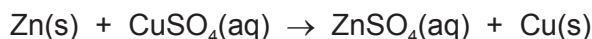
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..... [2]

[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 cm^3 beaker.
- Use the 50 cm^3 measuring cylinder to transfer 40 cm^3 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.

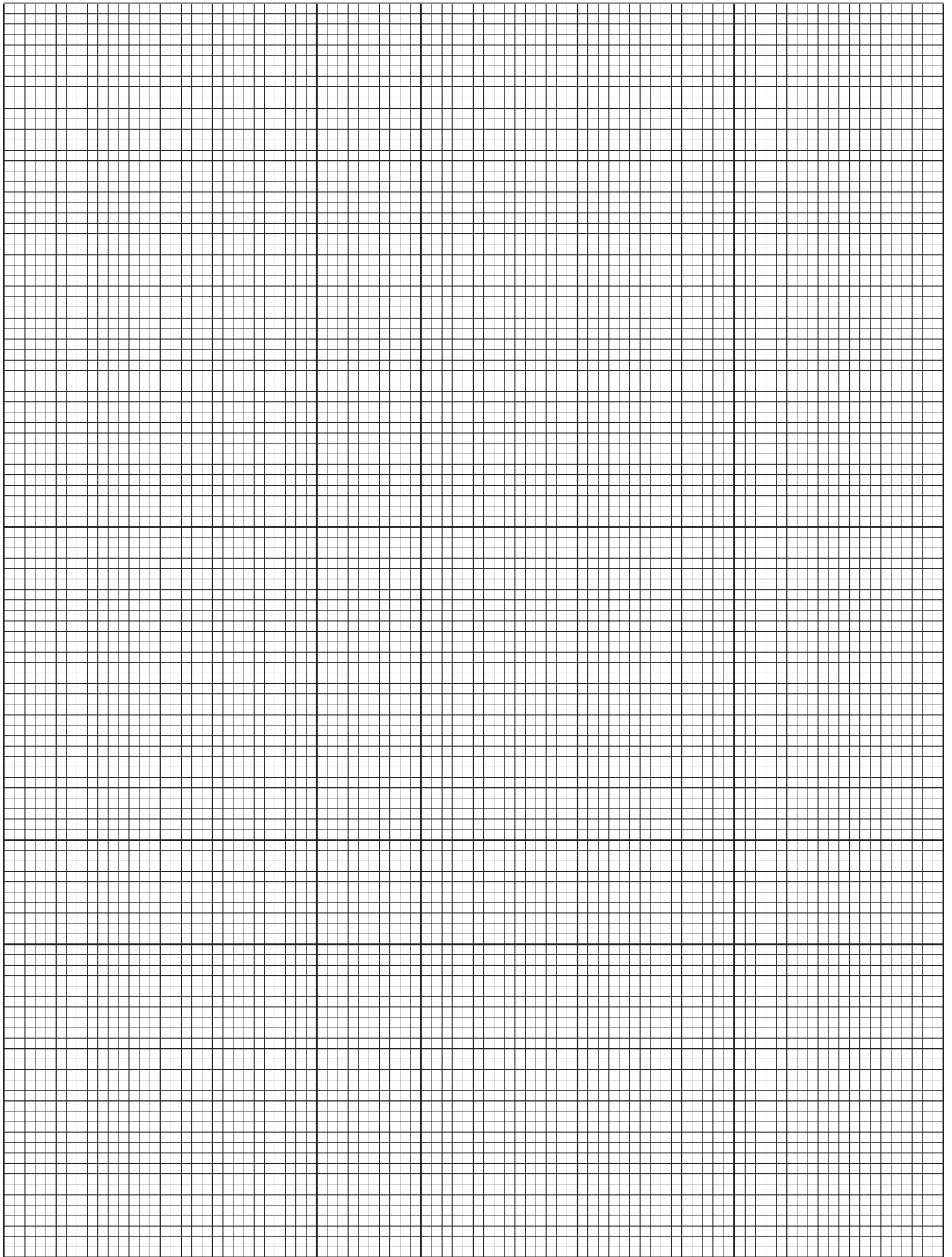
I	
II	
III	
IV	

[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.

change in temperature = $^\circ\text{C}$

[5]



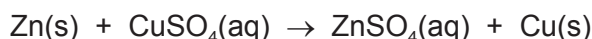
(c) Calculations

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.)

heat energy produced = 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.

moles of CuSO_4 = mol

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

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

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

maximum percentage error = %

- (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.

.....
.....
.....

- (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.

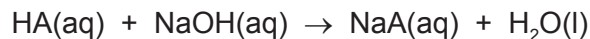
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[3]

[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.

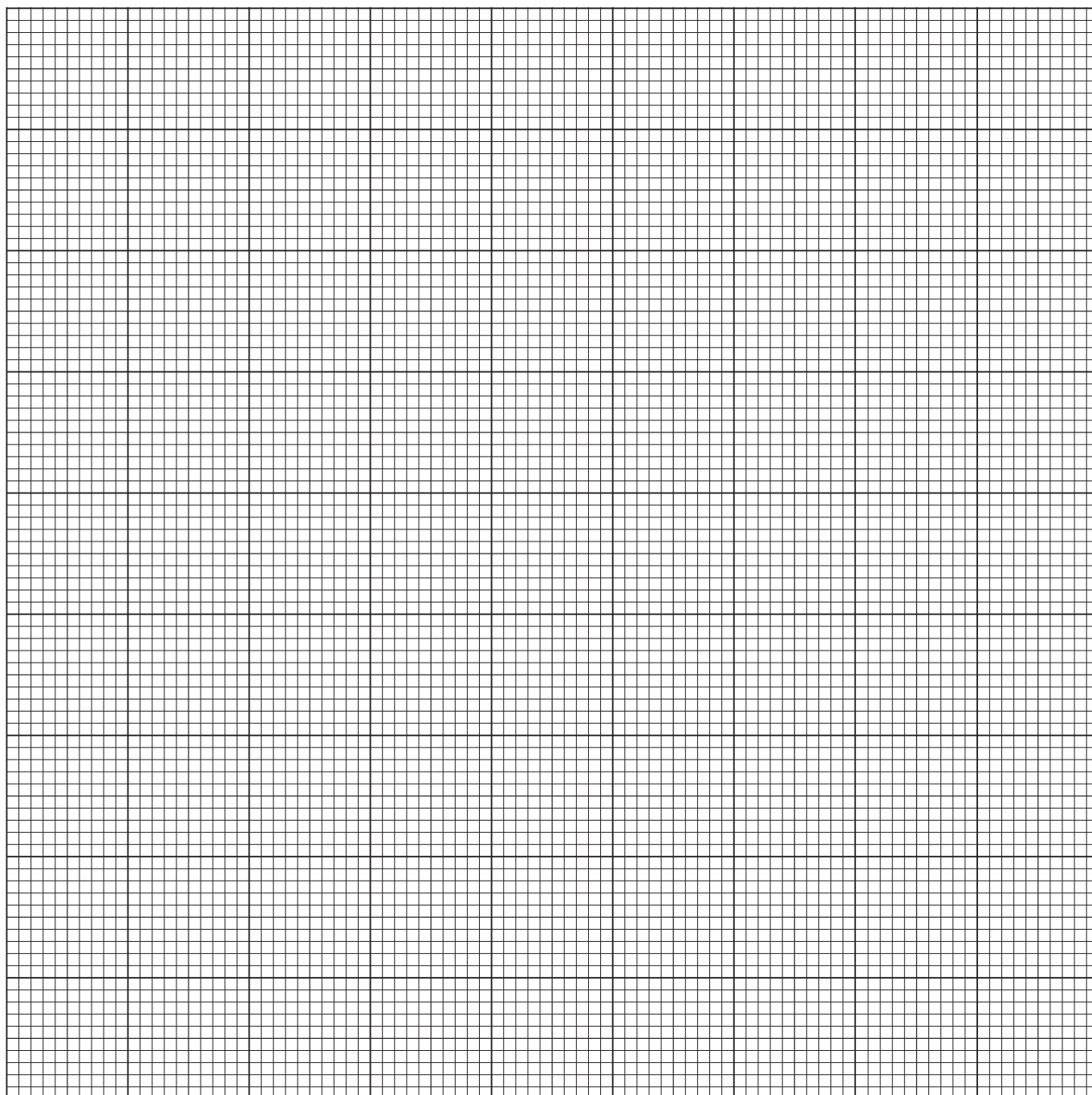
- Support the plastic cup in the 250 cm^3 beaker.
- Rinse and fill the burette with **FA 3**.
- Use the measuring cylinder to transfer 25 cm^3 of **FA 4** into the plastic cup.
- Place the thermometer in the plastic cup and record the temperature of the solution. Tilt the cup if necessary to ensure the 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

I	
II	
III	
IV	

[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.

I	
II	
III	
IV	

maximum temperature **increase** = °C

volume of **FA 3** = cm³
[4]

(c) Calculations

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

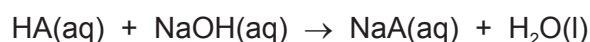
- (i) Calculate the number of moles of HA present in the volume of **FA 3** recorded in (b).

moles of HA = 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.)

heat energy produced = J

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



enthalpy change = 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).

maximum percentage error = % [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.

.....
..... [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).

.....
..... [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 250cm^3 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.

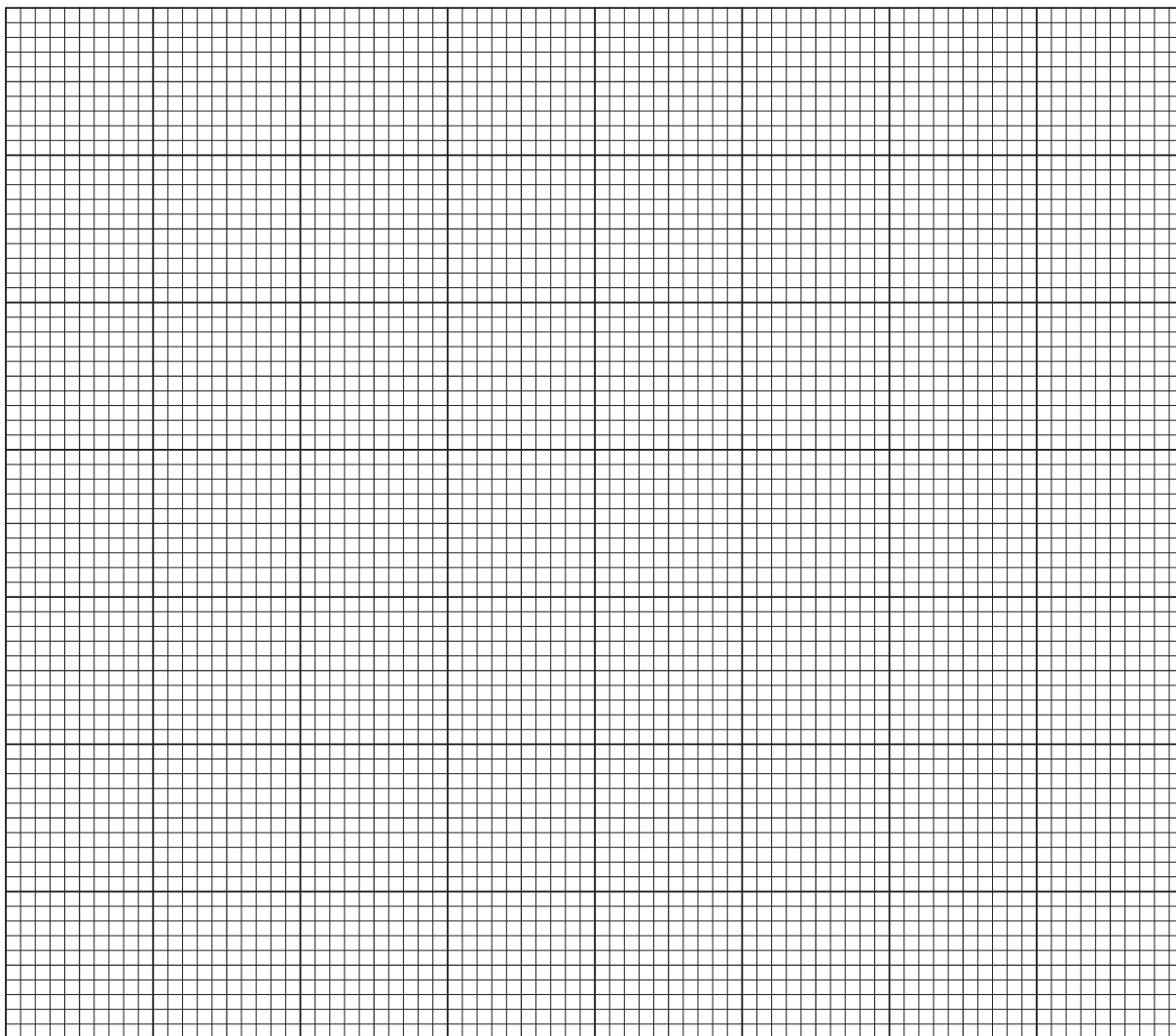
- Use a 50 cm³ measuring cylinder to transfer 50 cm³ of cold water into the 250 cm³ beaker.
- Use the 50 cm³ measuring cylinder to transfer 50 cm³ of cold water into a 100 cm³ beaker. Note the temperature of the water in this 100 cm³ beaker and heat it **carefully and gently** until the temperature of 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 $\frac{1}{2}$ **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

(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 °C.

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

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

Temperature fall for 50 cm³ of hot water from the 100 cm³ beaker, $(T_1 - T_3)$ is °C.
[4]

(c) Calculations

Working should be shown in all calculations.

[**4.2 J** are absorbed or released when the temperature of 1.0 cm^3 of **water** changes by 1.0°C .]

- (i) Calculate the heat energy gained by the 50 cm^3 of cold water in the 250 cm^3 beaker.

The heat energy gained by the cold water = J.

- (ii) Calculate the heat energy lost by the 50 cm^3 of hot water from the 100 cm^3 beaker.

The heat energy lost by the hot water = 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 cm^3 beaker during the experiment. The heat capacity of the beaker is the amount of heat energy absorbed for a 1°C change in temperature.

$$\text{approximate heat capacity of the } 250\text{ cm}^3 \text{ beaker} = \frac{(\text{heat energy lost}) - (\text{heat energy gained})}{(T_3 - T_2)} \text{ J}^\circ\text{C}^{-1}$$

Use your answers to (i) and (ii) and the temperature rise from (b) to calculate the approximate heat capacity of the 250 cm^3 beaker.

The approximate heat capacity of the 250 cm^3 beaker = $\text{J}^\circ\text{C}^{-1}$.

[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		
mass of empty tube / g		
mass of tube + residual ammonium chloride / g		
mass of ammonium chloride / g		
initial temperature of water / °C		
minimum temperature obtained / °C		
temperature fall, ΔT / °C		

[6]

(e) Calculations

For
Examiner's
Use

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.]

The change in heat energy of the solution = 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.

.....
.....
.....

- (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.

The combined change in heat energy of the beaker and solution = 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]

i	
ii	
iii	
iv	

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

- (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.

For
Examiner's
Use

$$\Delta H_{\text{solution}}(\text{NH}_4\text{Cl}) = \begin{matrix} \text{.....} & \text{.....} & \text{kJ mol}^{-1}. \\ \text{sign} & \text{calculated value} \end{matrix}$$

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

.....

.....

.....

[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.

difference = kJ mol^{-1}

percentage difference = %
[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

.....
.....

suggested improvement

.....
.....

explanation of why suggestion would increase experimental accuracy

.....
.....

[2]

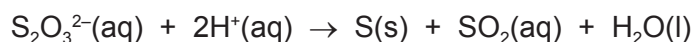
[Total: 25]

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, $\text{Na}_2\text{S}_2\text{O}_3$.

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**.

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 unit

'rate of reaction' for Experiment 3 unit

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

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

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

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

.....
.....

- (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.

.....
.....
.....

- (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.

.....
.....

[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.



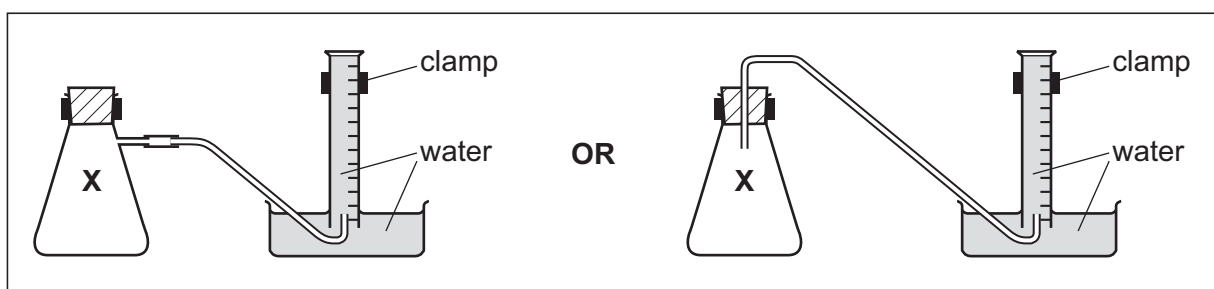
FA 2 is hydrochloric acid, HCl , as used in **Question 1**.

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.

(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.]

moles of gas = mol

- (ii) Use your answer to (i) to deduce the number of moles of **M**₂CO₃ used in the reaction.

moles of **M**₂CO₃ = mol

- (iii) Use your answer to (ii) and the mass of **FA 4** used to calculate the relative formula mass, *M*_r, of **M**₂CO₃.

*M*_r of **M**₂CO₃ =

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

M is

.....

.....

[4]

- (c) (i) A 250 cm^3 measuring cylinder can be read to $\pm 1\text{ cm}^3$.

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

maximum percentage error = %

- (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

.....

modification

.....

.....

reason

.....

modification

.....

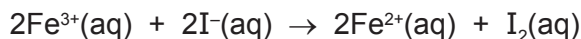
.....

[5]

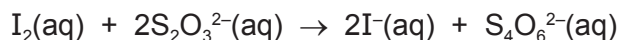
[Total: 11]

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 .

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

(b) Calculations

The rate of reaction can be found by calculating the change in concentration of $\text{Fe}^{3+}(\text{aq})$ 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, $\text{S}_2\text{O}_3^{2-}$ used in each experiment in (a).

moles $\text{S}_2\text{O}_3^{2-}$ = mol

- (ii) Calculate the number of moles of iodine, I_2 , that react with the number of moles of $\text{S}_2\text{O}_3^{2-}$ in (i).

moles I_2 = mol

- (iii) Calculate the number of moles of iron(III) ions, Fe^{3+} , that were used to produce the number of moles of iodine in (ii).

moles Fe^{3+} = mol

- (iv) When the moles of Fe^{3+} that you calculated in (iii) reacted, a change in the concentration of moles of Fe^{3+} occurred. Calculate this change in concentration.

change in concentration of $\text{Fe}^{3+}(\text{aq})$ = mol dm^{-3}

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

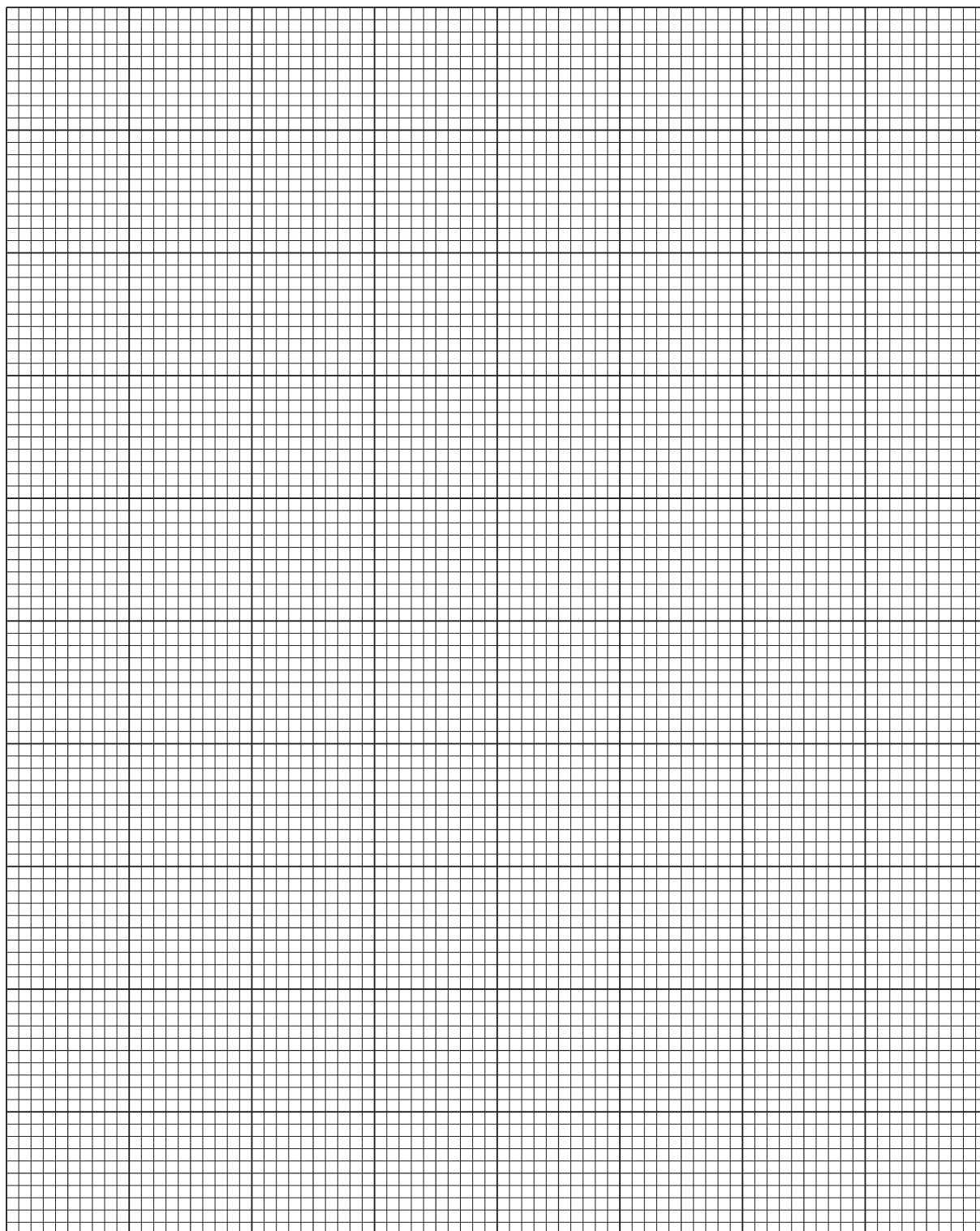
$$\text{'rate of reaction'} = \frac{\text{change in concentration of } \text{Fe}^{3+}(\text{aq})}{\text{reaction time}} \times 10^6$$

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 $\text{Fe}^{3+}(\text{aq})$ in (iv), you should assume it is $2.50 \times 10^{-3} \text{ mol dm}^{-3}$. (Note: this is not the correct value.)

Experiment			
1			
2			
3			
4			
5			
6			

- (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.



- (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 $\text{I}^{-}(\text{aq})$?

.....
.....
..... [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.

.....
.....
.....
..... [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.

.....
.....
.....

Suggested modification 2

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

.....
.....
.....

[4]

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

.....

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

percentage error = %
[2]

[Total: 27]

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 $\text{NO} \rightarrow$ (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

Group										
1	2				13	14	15	16	17	18

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

actinoids