READ THESE INSTRUCTIONS FIRST

Write your Centre number, candidate number and name on all the work you hand in.
Give details of the practical session and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen.
You may use a soft pencil for any diagrams, graphs or rough working.
Do not use staples, paper clips, highlighters, glue or correction fluid.
DO NOT WRITE IN ANY BARCODES.

Answer all questions.
Electronic calculators may be used.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 11 and 12.

At the end of the examination, fasten all your work securely together.
The number of marks is given in brackets [ ] at the end of each question or part question.
In this experiment you are to determine the relative formula mass of an iron(II) salt by titration with potassium manganate(VII).

FA 1 is the iron(II) salt.
FA 2 is 0.0200 mol dm\(^{-3}\) potassium manganate(VII), KMnO\(_4\).
FA 3 is dilute sulfuric acid, H\(_2\)SO\(_4\).

(a) Method

Preparing a solution of FA 1

- Weigh the 250 cm\(^3\) beaker and record the mass in the space below.
- Add all the FA 1 provided to the beaker. Weigh the beaker with FA 1 and record the mass.
- Calculate the mass of FA 1 used and record this in the space below.
- Use a measuring cylinder to add approximately 100 cm\(^3\) of FA 3 to the beaker. Stir until all the solid has dissolved.
- Transfer the solution into the 250 cm\(^3\) volumetric (graduated) flask labelled FA 4.
- Wash out the beaker thoroughly using distilled water and add the washings to the volumetric flask. Make the solution up to the mark using distilled water.
- Shake the flask thoroughly to mix the solution before using it for your titrations.
- This solution of the iron(II) salt is FA 4.

Titration

- Pipette 25.0 cm\(^3\) of FA 4 into a conical flask.
- Use a measuring cylinder to add 20 cm\(^3\) of FA 3 to the flask.
- Fill the burette with FA 2.
- Titrate FA 4 with FA 2 until the solution changes to a permanent pink colour.
- 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 FA 2 added in each accurate titration.

(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 FA 4 required ....................... cm³ of FA 2 [1]
(c) **Calculations**

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

(i) Calculate the number of moles of potassium manganate(VII) present in the volume of FA 2 calculated in (b).

\[ \text{moles of KMnO}_4 = \ldots \ldots \ldots \ldots \text{mol} \]

(ii) The half-equation for the reduction of a manganate(VII) ion is:

\[ \text{MnO}_4^- + 8\text{H}^+ + 5e^- \rightarrow \text{Mn}^{2+} + 4\text{H}_2\text{O} \]

Give the half-equation for the oxidation of an iron(II) ion to an iron(III) ion.

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

Therefore, 1 mole of manganate(VII) ions reacts with 5 moles of iron(II) ions.

(iii) Calculate the number of moles of iron(II) ions present in 25.0 cm$^3$ of solution FA 4.

\[ \text{moles of Fe}^{2+} \text{ in 25.0 cm}^3 \text{ of FA 4} = \ldots \ldots \ldots \ldots \text{mol} \]

(iv) Calculate the number of moles of iron(II) ions present in 250 cm$^3$ of solution FA 4.

\[ \text{moles of Fe}^{2+} \text{ in 250 cm}^3 \text{ of FA 4} = \ldots \ldots \ldots \ldots \text{mol} \]

(v) In 1 mole of the iron(II) salt, FA 1, there is 1 mole of iron(II) ions.

Use the mass of FA 1 you weighed out to calculate the relative formula mass of the iron(II) salt.

\[ \text{relative formula mass} = \ldots \ldots \ldots \ldots \]
(d) (i) A 25 cm³ pipette is accurate to ±0.06 cm³. Calculate the maximum percentage error when the pipette was used to measure solution FA 4.

\[
\text{percentage error in measuring FA 4} = \ldots \ldots \ldots \% \]

(ii) State the maximum error in the mass of the 250 cm³ beaker that you recorded in (a).

\[
\text{maximum error} = \ldots \ldots \ldots \text{g} \]

(iii) Calculate the maximum percentage error in the mass of FA 1 used in (a).

\[
\text{maximum percentage error} = \ldots \ldots \ldots \% \]

[2]

[Total: 15]
2 In this experiment you are to determine the formula of hydrated barium chloride, FA 5, by heating to remove the water of crystallisation. You will heat two separate samples. The anhydrous barium chloride does not decompose when heated.

FA 5 is hydrated barium chloride, BaCl₂·xH₂O

(a) Method

Record all weighings, in an appropriate form, in the space below.

- Record the mass of the empty crucible without its lid.
- Add between 2.0 and 2.4 g of FA 5 into the crucible. Record the mass of the crucible and its contents.
- Use a pipe-clay triangle to support the crucible and contents on a tripod.
- Heat the crucible and its contents gently for about one minute with the lid off. Then heat strongly for a further four minutes.
- Put the lid on the crucible and leave to cool for approximately 10 minutes.

While you are waiting for the crucible to cool, start work on Question 3.

- When the crucible is cool, remove the lid, and weigh the crucible with the residue.
- Record the mass of anhydrous barium chloride remaining in the crucible after heating and the mass of water lost.

- To prepare for the second experiment, use a spatula to remove the residue from the crucible into the beaker labelled waste.
- Reweigh the empty crucible without its lid.
- Carry out the experiment again. This time use between 1.5 and 1.9 g of FA 5.
(b) **Calculation**

Show your working in each step.

(i) Calculate the **mean** number of moles of water removed from the hydrated salt in the experiments.

\[(A_r : H, 1.0; O, 16.0)\]

\[
\text{moles of } H_2O = \text{................. mol}
\]

(ii) Calculate the **mean** number of moles of anhydrous barium chloride produced in the experiments.

\[(A_r : Ba, 137; Cl, 35.5)\]

\[
\text{moles of } BaCl_2 = \text{............ mol}
\]

(iii) Calculate the value of \(x\) in the formula of hydrated barium chloride, BaCl\(_2\).xH\(_2\)O.

\[
x = \text{.................}
\]

\[\text{[3]}\]

(c) (i) Suggest how the experimental procedure could be modified to ensure that all of the water of crystallisation had been removed by heating hydrated FA 5.

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

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

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

(ii) Do you think that the results from your two experiments are consistent with each other? Justify your answer by carrying out appropriate calculations.

\[\text{[3]}\]

\[\text{[Total: 12]}\]
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. 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 is hydrated barium chloride.

FA 6 is the same iron(II) salt used in Question 1. It contains one other cation and one anion.

(i) Place a small spatula measure of FA 6 into a test-tube. Dissolve the solid in about a 5 cm depth of distilled water. Use the solution for the following tests.

<table>
<thead>
<tr>
<th>test</th>
<th>observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>To a 1 cm depth of aqueous FA 6 in a boiling tube, add aqueous sodium hydroxide until no further change occurs, then heat the mixture carefully.</td>
<td></td>
</tr>
<tr>
<td>Dissolve a few crystals of FA 5 in a 1 cm depth of distilled water in a test-tube. Add a 1 cm depth of FA 6, then add excess dilute hydrochloric acid to the mixture.</td>
<td></td>
</tr>
</tbody>
</table>
(ii) Identify the ions present in **FA 6**.

cations: Fe$^{2+}$ and ........................................... anion: ..............................................................

(iii) Give the ionic equation for the reaction of iron(II) ions with hydroxide ions.

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

(iv) Place a small spatula measure of **FA 6** into a **hard-glass** test-tube.
Heat gently, then strongly, until no further change is observed.
Record your observations in the space below.
(b) **FA 7, FA 8 and FA 9** are aqueous solutions. Each contains **one** cation and **one** anion. **FA 3** is dilute sulfuric acid, \( \text{H}_2\text{SO}_4 \).

Mix pairs of solutions so that you can complete the table below. For each test, use 1 cm depths of each solution in clean test-tubes. Record your observations in the table.

<table>
<thead>
<tr>
<th></th>
<th>FA 7</th>
<th>FA 8</th>
<th>FA 9</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>FA 3</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>FA 7</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>FA 8</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

From your observations, complete the following statements.

The anion in **FA 7** is ...................................................

The cation in **FA 8** is ..................................................

The anion in **FA 9** is ...................................................

[6]

[Total: 13]
### Qualitative Analysis Notes

*Key: [ppt. = precipitate]*

#### 1 Reactions of aqueous cations

<table>
<thead>
<tr>
<th>ion</th>
<th>reaction with NaOH(aq)</th>
<th>reaction with NH₃(aq)</th>
</tr>
</thead>
<tbody>
<tr>
<td>aluminium, Al³⁺(aq)</td>
<td>white ppt.</td>
<td>white ppt.</td>
</tr>
<tr>
<td></td>
<td>soluble in excess</td>
<td>insoluble in excess</td>
</tr>
<tr>
<td>ammonium, NH₄⁺(aq)</td>
<td>no ppt.</td>
<td>—</td>
</tr>
<tr>
<td></td>
<td>ammonia produced on heating</td>
<td></td>
</tr>
<tr>
<td>barium, Ba²⁺(aq)</td>
<td>no ppt. (if reagents are pure)</td>
<td>no ppt.</td>
</tr>
<tr>
<td>calcium, Ca²⁺(aq)</td>
<td>white ppt. with high [Ca²⁺(aq)]</td>
<td>no ppt.</td>
</tr>
<tr>
<td>chromium(III), Cr³⁺(aq)</td>
<td>grey-green ppt. soluble in excess</td>
<td>grey-green ppt.</td>
</tr>
<tr>
<td></td>
<td>giving dark green solution</td>
<td>insoluble in excess</td>
</tr>
<tr>
<td>copper(II), Cu²⁺(aq)</td>
<td>pale blue ppt.</td>
<td>blue ppt. soluble in excess</td>
</tr>
<tr>
<td></td>
<td>insoluble in excess</td>
<td>giving dark blue solution</td>
</tr>
<tr>
<td>iron(II), Fe²⁺(aq)</td>
<td>green ppt. turning brown on contact with air</td>
<td>green ppt. turning brown on contact with air</td>
</tr>
<tr>
<td></td>
<td>insoluble in excess</td>
<td>insoluble in excess</td>
</tr>
<tr>
<td>iron(III), Fe³⁺(aq)</td>
<td>red-brown ppt.</td>
<td>red-brown ppt.</td>
</tr>
<tr>
<td></td>
<td>insoluble in excess</td>
<td>insoluble in excess</td>
</tr>
<tr>
<td>lead(II), Pb²⁺(aq)</td>
<td>white ppt.</td>
<td>white ppt.</td>
</tr>
<tr>
<td></td>
<td>soluble in excess</td>
<td>insoluble in excess</td>
</tr>
<tr>
<td>magnesium, Mg²⁺(aq)</td>
<td>white ppt.</td>
<td>white ppt.</td>
</tr>
<tr>
<td></td>
<td>insoluble in excess</td>
<td>insoluble in excess</td>
</tr>
<tr>
<td>manganese(II), Mn²⁺(aq)</td>
<td>off-white ppt. rapidly turning brown on contact with air</td>
<td>off-white ppt. rapidly turning brown on contact with air</td>
</tr>
<tr>
<td></td>
<td>insoluble in excess</td>
<td>insoluble in excess</td>
</tr>
<tr>
<td>zinc, Zn²⁺(aq)</td>
<td>white ppt.</td>
<td>white ppt.</td>
</tr>
<tr>
<td></td>
<td>soluble in excess</td>
<td>soluble in excess</td>
</tr>
</tbody>
</table>

[Lead(II) ions can be distinguished from aluminium ions by the insolubility of lead(II) chloride.]
2 Reactions of anions

<table>
<thead>
<tr>
<th>ion</th>
<th>reaction</th>
</tr>
</thead>
<tbody>
<tr>
<td>carbonate, CO₃²⁻</td>
<td>CO₂ liberated by dilute acids</td>
</tr>
<tr>
<td>chromate(VI), CrO₄²⁻(aq)</td>
<td>yellow solution turns orange with H⁺(aq); gives yellow ppt. with Ba²⁺(aq); gives bright yellow ppt. with Pb²⁺(aq)</td>
</tr>
<tr>
<td>chloride, Cl⁻(aq)</td>
<td>gives white ppt. with Ag⁺(aq) (soluble in NH₃(aq)); gives white ppt. with Pb²⁺(aq)</td>
</tr>
<tr>
<td>bromide, Br⁻(aq)</td>
<td>gives cream ppt. with Ag⁺(aq) (partially soluble in NH₃(aq)); gives white ppt. with Pb²⁺(aq)</td>
</tr>
<tr>
<td>iodide, I⁻(aq)</td>
<td>gives yellow ppt. with Ag⁺(aq) (insoluble in NH₃(aq)); gives yellow ppt. with Pb²⁺(aq)</td>
</tr>
<tr>
<td>nitrate, NO₃⁻(aq)</td>
<td>NH₃ liberated on heating with OH⁻(aq) and Al foil</td>
</tr>
<tr>
<td>nitrite, NO₂⁻(aq)</td>
<td>NH₃ liberated on heating with OH⁻(aq) and Al foil; NO liberated by dilute acids (colourless NO → (pale) brown NO₂ in air)</td>
</tr>
<tr>
<td>sulfate, SO₄²⁻(aq)</td>
<td>gives white ppt. with Ba²⁺(aq) or with Pb²⁺(aq) (insoluble in excess dilute strong acids)</td>
</tr>
<tr>
<td>sulfite, SO₃²⁻(aq)</td>
<td>SO₂ liberated with dilute acids; gives white ppt. with Ba²⁺(aq) (soluble in excess dilute strong acids)</td>
</tr>
</tbody>
</table>

3 Tests for gases

<table>
<thead>
<tr>
<th>gas</th>
<th>test and test result</th>
</tr>
</thead>
<tbody>
<tr>
<td>ammonia, NH₃</td>
<td>turns damp red litmus paper blue</td>
</tr>
<tr>
<td>carbon dioxide, CO₂</td>
<td>gives a white ppt. with limewater (ppt. dissolves with excess CO₂)</td>
</tr>
<tr>
<td>chlorine, Cl₂</td>
<td>bleaches damp litmus paper</td>
</tr>
<tr>
<td>hydrogen, H₂</td>
<td>“pops” with a lighted splint</td>
</tr>
<tr>
<td>oxygen, O₂</td>
<td>relights a glowing splint</td>
</tr>
<tr>
<td>sulfur dioxide, SO₂</td>
<td>turns acidified aqueous potassium dichromate(VI) from orange to green</td>
</tr>
</tbody>
</table>