UNIVERSITY OF CAMBRIDGE INTERNATIONAL EXAMINATIONS
General Certificate of Education
Advanced Subsidiary Level and Advanced Level

| CANDIDATE NAME | |
|---|---|

| CENTRE NUMBER | | | | | | CANDIDATE NUMBER | | | | |
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**CHEMISTRY** **9701/32**

Advanced Practical Skills 2 **May/June 2012**

**2 hours**

Candidates answer on the Question Paper.

Additional Materials:     As listed in the Confidential Instructions

## 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.
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.

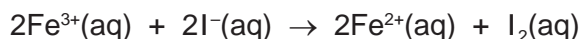Qualitative Analysis Notes are printed on pages 11 and 12.

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.

| Session |
|---|
| |
| Laboratory |
| |

| For Examiner's Use | |
|---|---|
| 1 | |
| 2 | |
| Total | |

This document consists of **12** printed pages.

UNIVERSITY of CAMBRIDGE
International Examinations

**[Turn over**

**1**  Iodide ions are oxidised by iron(III) ions in the presence of acid.

$$2Fe^{3+}(aq) + 2I^-(aq) \rightarrow 2Fe^{2+}(aq) + I_2(aq)$$

The rate of this reaction can be measured by adding thiosulfate ions, $S_2O_3^{2-}$, and some starch indicator to the mixture. As the iodine is produced, it reacts immediately with the thiosulfate ions and is reduced back to iodide ions.

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$
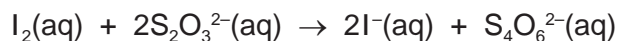
When all the thiosulfate ions have reacted, the iodine which continues to be produced then turns the starch indicator blue-black. The rate of reaction may be determined by timing how long it takes for the reaction mixture to turn blue-black.

You are to investigate how the rate of reaction is affected by changing the concentration of the reagents.

**FB 1** is $0.0500\,mol\,dm^{-3}$ aqueous acidified iron(III) chloride, $FeCl_3$.
**FB 2** is $0.0500\,mol\,dm^{-3}$ aqueous potassium iodide, $KI$.
**FB 3** is $0.00500\,mol\,dm^{-3}$ aqueous sodium thiosulfate, $Na_2S_2O_3$.
**FB 4** is starch indicator.

**Read through the instructions carefully and prepare a table for your results on page 3 before starting any practical work.**

**(a) Method**

**Experiment 1**
- Using a $25\,cm^3$ measuring cylinder add the following to a $100\,cm^3$ beaker:
  - $10\,cm^3$ of **FB 2**
  - $20\,cm^3$ of **FB 3**
  - $10\,cm^3$ of **FB 4**
- Fill the burette labelled **FB 1** with aqueous acidified iron(III) chloride, **FB 1**.
- Run $20.00\,cm^3$ of **FB 1** into a second $100\,cm^3$ beaker.
- Add the contents of the first beaker to the second beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second in the table that you have prepared on page 3.
- Wash out both beakers.

**Experiment 2**
- Using a $25\,cm^3$ measuring cylinder add the following to a $100\,cm^3$ beaker:
  - $10\,cm^3$ of **FB 2**
  - $20\,cm^3$ of **FB 3**
  - $10\,cm^3$ of **FB 4**
- Fill a second burette with distilled water.
- Run $10.00\,cm^3$ of **FB 1** into a second $100\,cm^3$ beaker.
- Run $10.00\,cm^3$ of distilled water into the beaker containing **FB 1**.
- Add the contents of the first beaker to the second beaker and start timing immediately.
- Stir the mixture once and place the beaker on a white tile.
- Stop timing as soon as the solution turns blue-black.
- Record this reaction time to the nearest second in the table that you have prepared on page 3.
- Wash out both beakers.

**Experiments 3 – 5**
- Carry out three further experiments to investigate how the reaction time cha̶̶̶̶̶
  with different volumes of iron(III) chloride.
  Remember that the combined volume of **FB 1** and distilled water must always be
  20.00 cm³.
  Do not use a volume of **FB 1** that is less than 6.00 cm³.

Record all your results in a single table. You should include the volume of iron(III)
chloride, the volume of distilled water and the reaction time.

| | |
|---|---|
| I | |
| II | |
| III | |
| IV | |
| V | |
| VI | |
| VII | |
| VIII | |
| IX | |

[9]

**(b)** In order to convert the times measured in the experiments into rates of reaction, it is
necessary first to work out the change in the iron(III) ion concentration that occurs from
the time when the mixtures are combined to the time when the solution turns blue-black.
You must show your working.

**(i)** Calculate how many moles of thiosulfate ions, $S_2O_3^{2-}$, are added in each experiment.

moles of $S_2O_3^{2-}$ = ................. mol

**(ii)** Calculate how many moles of iodine, $I_2$, reacted with the number of moles of
thiosulfate ions calculated in **(i)**.

moles of $I_2$ = ................. mol

**(iii)** Calculate how many moles of iron(III) ions, $Fe^{3+}$, reacted to form the number of
moles of iodine calculated in **(ii)**.

moles of $Fe^{3+}$ = ................. mol

**(iv)** Use your answer from **(iii)** to calculate the decrease in concentration of iron(III) ions
in the total reaction mixture (60 cm³) from the start of the experiment to the point
when the solution turned blue-black.

decrease in $Fe^{3+}$ concentration = ................. mol dm⁻³
[3]

For
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**(c)** The rate of the reaction can be represented by the following formula.

$$\text{'rate'} = \frac{\text{decrease in } Fe^{3+} \text{ concentration from } \textbf{(b)(iv)}}{\text{reaction time}} \times 10^6$$

Use your experimental results to complete the following table. Include the volume of **FB 1**, the reaction time and the 'rate' with their units.

If you were unable to answer **(b)(iv)**, you may assume that the decrease in $Fe^{3+}$ concentration is $2.25 \times 10^{-3} \, mol \, dm^{-3}$ (This is not the correct value).

| | | |
|---|---|---|
| | | |

[2]

**(d)** On the grid opposite, plot 'rate' against the volume of **FB 1**. Draw a line of best fit.
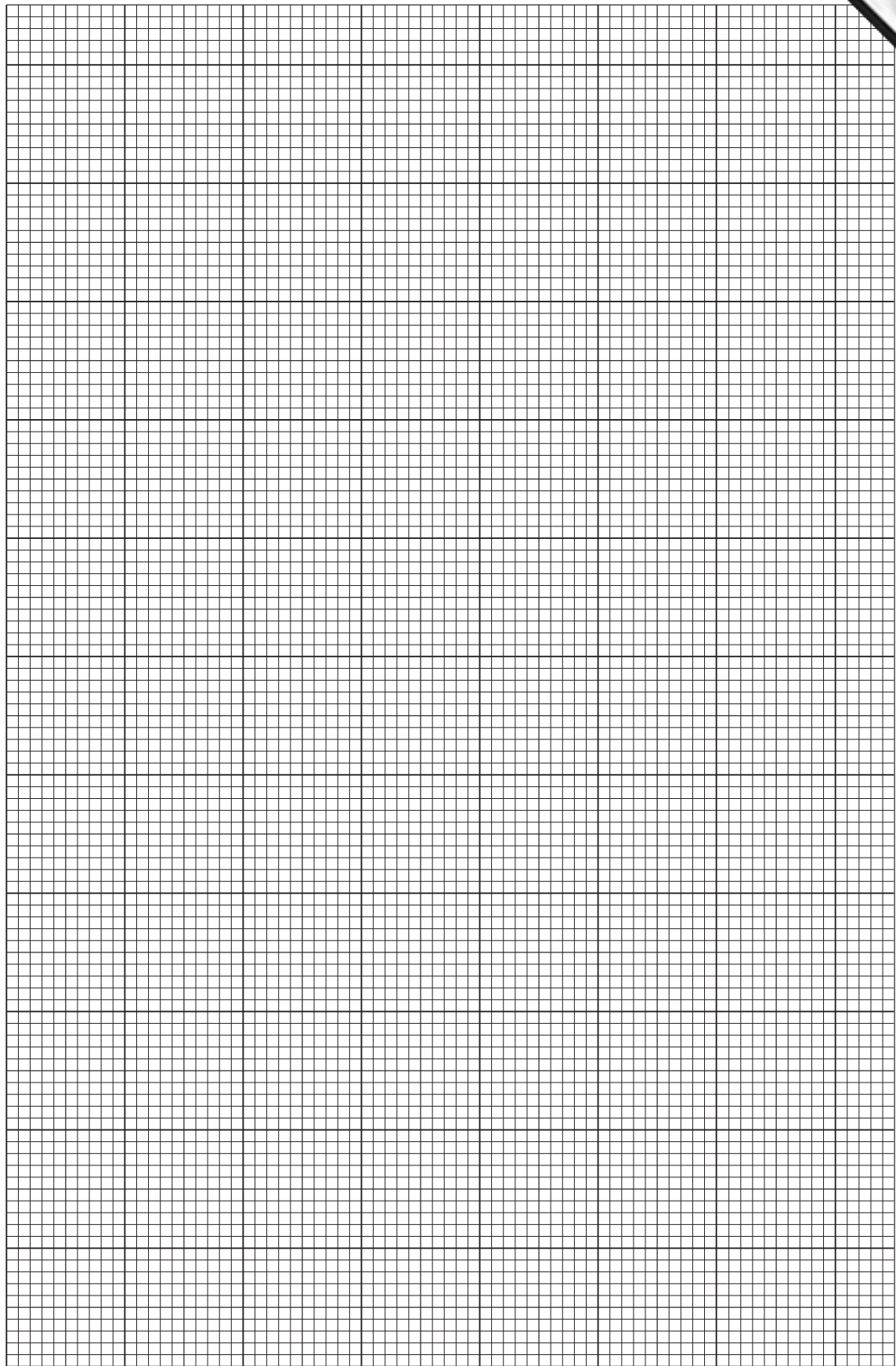
| I |  |
|---|---|
| II |  |
| III |  |
| IV |  |

[4]

**(e)** In your experiments, the volume of **FB 1** represents the concentration of iron(III) chloride. From your results, what conclusion can you draw about the relationship between the rate of reaction and the concentration of iron(III) chloride?

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**(f)** One source of error in this experiment arises from measuring the volumes of solutions.

  **(i)** Calculate the maximum percentage error in the volume of **FB 1** used in **Experiment 1**.

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

  **(ii)** Other than errors involving measurements of volumes, suggest an additional source of error in these experiments.

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  **(iii)** In this experiment, thiosulfate ions reduce iodine while iron(III) ions oxidise iodide ions. What other reaction might take place that would affect your confidence in the conclusions you made in **(e)**?

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

...............................................................................................................................
[4]

| | |
|---|---|
| I | |
| II | |
| III | |
| IV | |

For Examiner's Use

*For
miner's*

**(g) (i)** Carry out one additional experiment using the following volumes of each r
Use the same method as in **(a)**, mix **FB 2**, **FB 3** and **FB 4** together and star
reaction by adding this mixture to **FB 1** and the distilled water.

- 10.00 cm$^3$ of **FB 1**
- 20.00 cm$^3$ of distilled water
- 10 cm$^3$ of **FB 2**
- 10 cm$^3$ of **FB 3**
- 10 cm$^3$ of **FB 4**

Record the time for the reaction to go blue-black.

**(ii)** Explain the relationship between this time and the one you recorded in **Experiment 2**.

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[2]

[Total: 26]

## 2   Qualitative Analysis

At each stage in 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 reagents must be given.**

**(a)** **FB 5**, **FB 6**, **FB 7** and **FB 8** are aqueous solutions each of which contains a single cation and a single anion. Some of the ions present are listed below.

$$Pb^{2+} \qquad Ba^{2+} \qquad H^+ \qquad CrO_4^{2-} \qquad SO_4^{2-} \qquad Cl^-$$

By observing the reactions that occur when pairs of the solutions are mixed together, you will be able to identify which solution contains which of these ions.

Use 1 cm depth of each solution in a test-tube and record your observations in the following table.

|  | FB 6 | FB 7 | FB 8 |
|---|---|---|---|
| **FB 5** |  |  |  |
| **FB 6** | ▓ |  |  |
| **FB 7** | ▓ | ▓ |  |

|  |  |
|---|---|
| I |  |
| II |  |
| III |  |
| IV |  |
| V |  |

[5]

**(b)** From your observations, deduce which solution contains each of the following ions.

| ion | $Pb^{2+}$ | $Ba^{2+}$ | $H^+$ | $CrO_4^{2-}$ | $SO_4^{2-}$ | $Cl^-$ |
|---|---|---|---|---|---|---|
| solution |  |  |  |  |  |  |

|  |  |
|---|---|
| I |  |
| II |  |
| III |  |

[3]

**(c)** The anion in **FB 9** is either the nitrite ion, $NO_2^-$, or the nitrate ion, $NO_3^-$.

For
miner's

   **(i)** Describe a test you could carry out that would give positive results for both of these ions.

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

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

  **(ii)** Describe a test you could carry out that would distinguish between these two ions.

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

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

 **(iii)** Carry out both of these tests and record your results in an appropriate form in the space below.

| | |
|---|---|
| I | |
| II | |
| III | |
| IV | |
| V | |
| VI | |

  **(iv)** Which anion is present in **FB 9**?

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

[6]

[Total: 14]

## Qualitative Analysis Notes

*Key: [ppt. = precipitate]*

## 1    Reactions of aqueous cations

| ion | reaction with | |
| --- | --- | --- |
| | NaOH(aq) | NH$_3$(aq) |
| aluminium, Al$^{3+}$(aq) | white ppt. soluble in excess | white ppt. insoluble in excess |
| ammonium, NH$_4^+$(aq) | no ppt. ammonia produced on heating | – |
| barium, Ba$^{2+}$(aq) | no ppt. (if reagents are pure) | no ppt. |
| calcium, Ca$^{2+}$(aq) | white ppt. with high [Ca$^{2+}$(aq)] | no ppt. |
| chromium(III), Cr$^{3+}$(aq) | grey-green ppt. soluble in excess giving dark green solution | grey-green ppt. insoluble in excess |
| copper(II), Cu$^{2+}$(aq) | pale blue ppt. insoluble in excess | blue ppt. soluble in excess giving dark blue solution |
| iron(II), Fe$^{2+}$(aq) | green ppt. turning brown on contact with air insoluble in excess | green ppt. turning brown on contact with air insoluble in excess |
| iron(III), Fe$^{3+}$(aq) | red-brown ppt. insoluble in excess | red-brown ppt. insoluble in excess |
| lead(II), Pb$^{2+}$(aq) | white ppt. soluble in excess | white 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 |

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

## 2 Reactions of anions

| ion | reaction |
|---|---|
| carbonate, $CO_3^{2-}$ | $CO_2$ liberated by dilute acids |
| chromate(VI), $CrO_4^{2-}(aq)$ | yellow solution turns orange with $H^+(aq)$; gives yellow ppt. with $Ba^{2+}(aq)$; gives bright yellow ppt. with $Pb^{2+}(aq)$ |
| chloride, $Cl^-(aq)$ | gives white ppt. with $Ag^+(aq)$ (soluble in $NH_3(aq)$); gives white ppt. with $Pb^{2+}(aq)$ |
| bromide, $Br^-(aq)$ | gives cream ppt. with $Ag^+(aq)$ (partially soluble in $NH_3(aq)$); gives white ppt. with $Pb^{2+}(aq)$ |
| iodide, $I^-(aq)$ | gives yellow ppt. with $Ag^+(aq)$ (insoluble in $NH_3(aq)$); gives yellow ppt. with $Pb^{2+}(aq)$ |
| nitrate, $NO_3^-(aq)$ | $NH_3$ liberated on heating with $OH^-(aq)$ and $Al$ foil |
| nitrite, $NO_2^-(aq)$ | $NH_3$ liberated on heating with $OH^-(aq)$ and $Al$ foil; NO liberated by dilute acids (colourless NO → (pale) brown $NO_2$ in air) |
| sulfate, $SO_4^{2-}(aq)$ | gives white ppt. with $Ba^{2+}(aq)$ or with $Pb^{2+}(aq)$ (insoluble in excess dilute strong acids) |
| sulfite, $SO_3^{2-}(aq)$ | $SO_2$ liberated with dilute acids; gives white ppt. with $Ba^{2+}(aq)$ (soluble in excess dilute strong acids) |

## 3 Tests for gases

| gas | test and test result |
|---|---|
| ammonia, $NH_3$ | turns damp red litmus paper blue |
| carbon dioxide, $CO_2$ | gives a white ppt. with limewater (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 dichromate(VI) from orange to green |