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

CHEMISTRY
Advanced Practical Skills
May/June 2011
2 hours

Candidates answer on the Question Paper.
Additional Materials: As listed in the Instructions to Supervisors

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.

For Examiner’s Use

1
2
3
Total

This document consists of 12 printed pages and 1 insert.
You are to determine the concentration, in mol dm\(^{-3}\), of the aqueous sodium thiosulfate. To do this you will first produce iodine solution by reacting aqueous potassium iodide with aqueous potassium manganate(VII). In this reaction iodide ions are oxidised to iodine by manganate(VII) ions in acidic solution.

\[
2\text{MnO}_4^- (aq) + 10\text{I}^- (aq) + 16\text{H}^+ (aq) \rightarrow 2\text{Mn}^{2+} (aq) + 5\text{I}_2 (aq) + 8\text{H}_2\text{O}(l)
\]

You will then titrate the iodine with aqueous thiosulfate ions, in FB 1.

\[
2\text{S}_2\text{O}_3^{2-} (aq) + \text{I}_2 (aq) \rightarrow \text{S}_4\text{O}_6^{2-} (aq) + 2\text{I}^- (aq)
\]

You are provided with the following.

FB 1 is a solution of sodium thiosulfate, Na\(_2\)S\(_2\)O\(_3\), of unknown concentration.
FB 3 is 0.0050 mol dm\(^{-3}\) potassium manganate(VII), KMnO\(_4\).
FB 4 is 0.10 mol dm\(^{-3}\) potassium iodide, KI.
FB 5 is 1.0 mol dm\(^{-3}\) sulfuric acid, H\(_2\)SO\(_4\).

starch indicator

(a) Method

Dilution

- Fill the burette with FB 1.
- Run between 45.50 cm\(^3\) and 46.50 cm\(^3\) of FB 1 from the burette into the 250 cm\(^3\) graduated (volumetric) flask, labelled FB 2.
- Make the solution up to the mark with distilled water.
- Shake the flask to mix the solution of FB 2.

In the space below record your burette readings and the volume of FB 1 added to the graduated flask.
Titration

- Fill a second burette with FB 2, the diluted sodium thiosulfate.
- Pipette 25.0 cm³ of FB 3 into a conical flask.
- Using a 25 cm³ measuring cylinder, add about 10 cm³ of FB 4.
- Using the same measuring cylinder, add about 10 cm³ of FB 5.
- Titrate the mixture in the flask with FB 2 until the colour is pale yellow.
- Add about 10 drops of starch indicator. A blue-black colour should be seen as the starch reacts with the remaining iodine.
- Continue to add FB 2 until the blue-black colour just disappears leaving a colourless solution.

You should perform a rough titration.
In the space below record your burette readings for this rough titration.

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 an appropriate form below all of your burette readings and the volume of FB 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.

The iodine produced by 25.0 cm³ of FB 3 required ............... cm³ 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 how many moles of MnO$_4^-$ were pipetted into the conical flask.

\[ \text{................. mol of MnO}_4^- \]

(ii) Calculate how many moles of I$_2$ were produced from the number of moles of MnO$_4^-$ calculated in (i).

\[
2\text{MnO}_4^-(aq) + 10\text{I}^- (aq) + 16\text{H}^+(aq) \rightarrow 2\text{Mn}^{2+}(aq) + 5\text{I}_2(aq) + 8\text{H}_2\text{O}(l)
\]

\[ \text{................. mol of I}_2 \]

(iii) Calculate how many moles of S$_2$O$_3^{2-}$ reacted with the I$_2$ in (ii).

\[
2\text{S}_2\text{O}_3^{2-}(aq) + \text{I}_2(aq) \rightarrow \text{S}_4\text{O}_6^{2-}(aq) + 2\text{I}^-(aq)
\]

\[ \text{................. mol of S}_2\text{O}_3^{2-} \]

(iv) Calculate how many moles of S$_2$O$_3^{2-}$ were present in the 250 cm$^3$ graduated (volumetric) flask.

\[ \text{................. mol of S}_2\text{O}_3^{2-} \]

(v) Use your answer to (iv) and the volume of FB 1 that was diluted to calculate the concentration, in mol dm$^{-3}$, of the original solution of sodium thiosulfate, FB 1.

The concentration of Na$_2$S$_2$O$_3$ in FB 1 was ................. mol dm$^{-3}$. [6]
(d) The maximum error for a 25 cm³ pipette commonly used in schools is ±0.06 cm³. The maximum individual error in any single burette reading is ±0.05 cm³.

Calculate each of the following.

The maximum percentage error in the volume of FB 3 pipetted into the conical flask

maximum percentage error in the pipetted volume of FB 3 = ……………… %

The maximum percentage error in the titre calculated in (b)

maximum percentage error in the titre volume = ……………… %

[1]

[Total: 15]
2 You are to investigate how the rate of the following reaction varies with the concentration of sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$.

$$\text{Na}_2\text{S}_2\text{O}_3(\text{aq}) + \text{H}_2\text{SO}_4(\text{aq}) \rightarrow \text{S(s)} + \text{Na}_2\text{SO}_4(\text{aq}) + \text{SO}_2(\text{g}) + \text{H}_2\text{O}(\text{l})$$

The rate can be found by measuring how long it takes for the solid sulfur formed to obscure the printing on the insert provided.

Care should be taken to avoid inhalation of $\text{SO}_2(\text{g})$ that is given off during this reaction.

**FB 5** is 1.0 mol dm$^{-3}$ sulfuric acid, $\text{H}_2\text{SO}_4$.

**FB 6** is 0.10 mol dm$^{-3}$ sodium thiosulfate $\text{Na}_2\text{S}_2\text{O}_3$.

Read through the instructions carefully and prepare a table for your results on page 7 before starting any practical work.

(a) Method

- Using the 50 cm$^3$ measuring cylinder transfer 45 cm$^3$ of **FB 6** into a 100 cm$^3$ beaker.
- Using the 25 cm$^3$ measuring cylinder measure 10 cm$^3$ of **FB 5**.
- Tip the **FB 5** into the **FB 6** in the beaker and immediately start timing.
- Stir the mixture once with a glass rod and place it on top of the printed insert.
- View the printed insert from above so that it is seen through the mixture.
- Record the time, to the nearest second, when the printing on the insert just disappears.

- Empty and rinse the beaker. Shake out as much of the water as possible and dry the outside of the beaker.
- You will repeat the experiment to find out how the time for the printing on the insert to disappear changes when a different volume of **FB 6** is used.
- Using the 50 cm$^3$ measuring cylinder transfer 20 cm$^3$ of **FB 6** and 25 cm$^3$ of distilled water into the 100 cm$^3$ beaker.
- Using the 25 cm$^3$ measuring cylinder add 10 cm$^3$ of **FB 5** to the mixture and immediately start timing.
- Stir the mixture once with a glass rod and place it on top of the printed insert.
- View the printed insert from above so that it is seen through the mixture.
- Record the time, to the nearest second, when the printing on the insert just disappears.

- Select suitable volumes of **FB 6** and distilled water for two further experiments to investigate the effect of volume of sodium thiosulfate on the time taken for the printing on the insert to just disappear.

Calculate the values of $1$/time, where time is in seconds, to an appropriate number of significant figures.
In the space below, record, in an appropriate form, all measurements of volume, time, and your calculated values of 1/time.

(b) Why was the total volume of solution kept constant in the experiments?
..........................................................................................................................................
..........................................................................................................................................
..........................................................................................................................................
[1]

(c) It may be assumed that the rate of reaction is proportional to 1/time.
Draw a conclusion from your results about the relationship between the concentration of sodium thiosulfate used and the rate of reaction.
Explain your answer.
..........................................................................................................................................
..........................................................................................................................................
..........................................................................................................................................
[1]

(d) In the four experiments, which value of the time measured had the greatest error?
Explain your answer.
..........................................................................................................................................
..........................................................................................................................................
..........................................................................................................................................
[1]

(e) How could the procedure be adapted to find the effect of changing the concentration of acid on the rate of reaction?
..........................................................................................................................................
..........................................................................................................................................
..........................................................................................................................................
[1]

[Total: 9]
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 full name or correct formula of the reagents must be given.

There are two parts to this question. In the first part you will analyse three metal salts, FB 7, FB 8 and FB 9, to identify the cation in each. In the second part you will carry out a series of tests on a different salt, FB 10, to identify the anion.

(a) FB 7, FB 8 and FB 9 each contain one of aluminium ions, lead ions or zinc ions. By reference to the Qualitative Analysis Notes on pages 11 and 12, select two appropriate reagents to perform tests to identify the cations present.

reagent ...............................................................................................................................

Record the tests performed and the results of those tests in an appropriate form in the space below.

(b) From your observations, identify the cations present in FB 7, FB 8 and FB 9. State the minimum evidence to support each of your choices.

FB 7 contains .................................... cations.

evidence ................................................................................................................................

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

[5]
FB 8 contains …………………… cations.
evidence …………………………………………………………………………………………………………. 
…………………………………………………………………………………………………………………………..

FB 9 contains …………………… cations.
evidence …………………………………………………………………………………………………………… 
……………………………………………………………………………………………………………………………..

[c] You are provided with a solid sample of FB 10 which is a metal salt. Use this sample to perform the experiments described below. Record all your observations in the table.

<table>
<thead>
<tr>
<th>test</th>
<th>observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>(i)</td>
<td>Place a spatula measure of solid FB 10 into a dry hard-glass tube. Hold the test-tube in a holder. Heat the test-tube gently at first and then very strongly for several minutes. Allow the test-tube to cool, and then half fill it with distilled water. Dissolve the solid residue. This is FB 11. Retain this for tests (iv) and (v). While you are waiting for the test-tube to cool, continue with experiment (c)(ii) and (iii).</td>
</tr>
<tr>
<td>(ii)</td>
<td>Pour a 2 cm depth of aqueous copper(II) sulfate in a test-tube. Add a spatula measure of solid FB 10.</td>
</tr>
<tr>
<td>(iii)</td>
<td>Pour a 2 cm depth of aqueous aluminium sulfate into a test-tube. Add a spatula measure of solid FB 10.</td>
</tr>
<tr>
<td><strong>test</strong></td>
<td><strong>observations</strong></td>
</tr>
<tr>
<td>----------</td>
<td>------------------</td>
</tr>
</tbody>
</table>
| (iv) Pour a 2 cm depth of solution **FB 11** into a test-tube.  
Add the same volume of aqueous barium chloride.  
Then, using a dropping pipette, add dilute hydrochloric acid until no further change is seen. | |
| (v) Pour 1 cm depth of **FB 11** into a test-tube and add an equal depth of aqueous copper(II) sulfate.  
Transfer the contents of the test-tube into an evaporating dish and place it over a Bunsen burner on a tripod and gauze. Heat strongly until all the water has been driven off and no further change is seen. | |

(d) Consider your observations in (c) and answer the following questions. In each case, provide evidence from your observations to support your conclusions.

(i) Identify the anion present in **FB 11** and state evidence to support your choice.

**FB 11** contains the ………………… anion.

evidence …………………………………………………………………………………………………

(ii) Suggest what type of reaction occurs as **FB 10** is converted into **FB 11**.

…………………………………………………………………………………………………………

(iii) Suggest an explanation for what you observed in (c)(iii).

…………………………………………………………………………………………………………

[3]

[Total: 16]
Qualitative Analysis Notes

**Key:** \( [ \text{ppt.} = \text{precipitate} ] \)

## 1 Reactions of aqueous cations

<table>
<thead>
<tr>
<th>ion ( \text{aq} )</th>
<th>reaction with NaOH(aq)</th>
<th>reaction with NH(_3)(aq)</th>
</tr>
</thead>
<tbody>
<tr>
<td>aluminium, ( \text{Al}^{3+} )</td>
<td>white ppt. soluble in excess</td>
<td>white ppt. insoluble in excess</td>
</tr>
<tr>
<td>ammonium, ( \text{NH}_4^+ )</td>
<td>no ppt. ammonia produced on heating</td>
<td>-</td>
</tr>
<tr>
<td>barium, ( \text{Ba}^{2+} )</td>
<td>no ppt. (if reagents are pure)</td>
<td>no ppt.</td>
</tr>
<tr>
<td>calcium, ( \text{Ca}^{2+} )</td>
<td>white ppt. with high ([\text{Ca}^{2+}(\text{aq})])</td>
<td>no ppt.</td>
</tr>
<tr>
<td>chromium(III), ( \text{Cr}^{3+} )</td>
<td>grey-green ppt. soluble in excess giving dark green solution</td>
<td>grey-green ppt. insoluble in excess</td>
</tr>
<tr>
<td>copper(II), ( \text{Cu}^{2+} )</td>
<td>pale blue ppt. insoluble in excess</td>
<td>blue ppt. soluble in excess giving dark blue solution</td>
</tr>
<tr>
<td>iron(II), ( \text{Fe}^{2+} )</td>
<td>green ppt. turning brown on contact with air insoluble in excess</td>
<td>green ppt. turning brown on contact with air insoluble in excess</td>
</tr>
<tr>
<td>iron(III), ( \text{Fe}^{3+} )</td>
<td>red-brown ppt. insoluble in excess</td>
<td>red-brown ppt. insoluble in excess</td>
</tr>
<tr>
<td>lead(II), ( \text{Pb}^{2+} )</td>
<td>white ppt. soluble in excess</td>
<td>white ppt. insoluble in excess</td>
</tr>
<tr>
<td>magnesium, ( \text{Mg}^{2+} )</td>
<td>white ppt. insoluble in excess</td>
<td>white ppt. insoluble in excess</td>
</tr>
<tr>
<td>manganese(II), ( \text{Mn}^{2+} )</td>
<td>off-white ppt. rapidly turning brown on contact with air insoluble in excess</td>
<td>off-white ppt. rapidly turning brown on contact with air insoluble in excess</td>
</tr>
<tr>
<td>zinc, ( \text{Zn}^{2+} )</td>
<td>white ppt. soluble in excess</td>
<td>white ppt. 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, CO$_3^{2-}$</td>
<td>CO$_2$ liberated by dilute acids</td>
</tr>
<tr>
<td>chromate(VI), CrO$_4^{2-}$ (aq)</td>
<td>yellow solution turns orange with H$^+$ (aq); gives yellow ppt. with Ba$^{2+}$ (aq); gives bright yellow ppt. with Pb$^{2+}$ (aq)</td>
</tr>
<tr>
<td>chloride, Cl$^-$ (aq)</td>
<td>gives white ppt. with Ag$^+$ (aq) (soluble in NH$_3$ (aq)); gives white ppt. with Pb$^{2+}$ (aq)</td>
</tr>
<tr>
<td>bromide, Br$^-$ (aq)</td>
<td>gives cream ppt. with Ag$^+$ (aq) (partially soluble in NH$_3$ (aq)); gives white ppt. with Pb$^{2+}$ (aq)</td>
</tr>
<tr>
<td>iodide, I$^-$ (aq)</td>
<td>gives yellow ppt. with Ag$^+$ (aq) (insoluble in NH$_3$ (aq)); gives yellow ppt. with Pb$^{2+}$ (aq)</td>
</tr>
<tr>
<td>nitrate, NO$_3^-$ (aq)</td>
<td>NH$_3$ liberated on heating with OH$^-$ (aq) and Al foil</td>
</tr>
<tr>
<td>nitrite, NO$_2^-$ (aq)</td>
<td>NH$_3$ liberated on heating with OH$^-$ (aq) and Al foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO$_2$ in air)</td>
</tr>
<tr>
<td>sulfate, SO$_4^{2-}$ (aq)</td>
<td>gives white ppt. with Ba$^{2+}$ (aq) or with Pb$^{2+}$ (aq) (insoluble in excess dilute strong acids)</td>
</tr>
<tr>
<td>sulfite, SO$_3^{2-}$ (aq)</td>
<td>SO$_2$ liberated with dilute acids; gives white ppt. with Ba$^{2+}$ (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, NH$_3$</td>
<td>turns damp red litmus paper blue</td>
</tr>
<tr>
<td>carbon dioxide, CO$_2$</td>
<td>gives a white ppt. with limewater (ppt. dissolves with excess CO$_2$)</td>
</tr>
<tr>
<td>chlorine, Cl$_2$</td>
<td>bleaches damp litmus paper</td>
</tr>
<tr>
<td>hydrogen, H$_2$</td>
<td>&quot;pops&quot; with a lighted splint</td>
</tr>
<tr>
<td>oxygen, O$_2$</td>
<td>relights a glowing splint</td>
</tr>
<tr>
<td>sulfur dioxide, SO$_2$</td>
<td>turns acidified aqueous potassium dichromate(VI) from orange to green</td>
</tr>
</tbody>
</table>