CHEMISTRY
Advanced Practical Skills
October/November 2010
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 13 and 14.

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

3

Total

This document consists of 12 printed pages and 4 blank pages.
There are three questions on this paper. Question 2 should not be the last question attempted.

1 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, Fe\(_2\)(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).

\[
2\text{KMnO}_4(aq) + 8\text{H}_2\text{SO}_4(aq) + 10\text{FeSO}_4(aq) \rightarrow 5\text{Fe}_2(\text{SO}_4)_3(aq) + 2\text{MnSO}_4(aq) + \text{K}_2\text{SO}_4(aq) + 8\text{H}_2\text{O}(l)
\]

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

25.0 cm$^3$ of FB 1 required .............. cm$^3$ 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$^{-3}$ KMnO$_4$.

[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 KMnO$_4$ were present in the volume calculated in (b).

....................... mol of KMnO$_4$.

(iii) Calculate how many moles of iron(II) sulfate, FeSO$_4$, reacted with the potassium manganate(VII) in (ii).

2KMnO$_4$(aq) + 8H$_2$SO$_4$(aq) + 10FeSO$_4$(aq) → 5Fe$_2$(SO$_4$)$_3$(aq) + 2MnSO$_4$(aq) + K$_2$SO$_4$(aq) + 8H$_2$O(l)

............................... mol of FeSO$_4$ reacted with the potassium manganate(VII).
(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 Fe$_2$(SO$_4$)$_3$.

Calculate the percentage, by mass, of FeSO$_4$ in this mixture.

The mixture contains .................... % FeSO$_4$.

[5]

[Total: 13]
2 FB 3 is a mixture containing anhydrous sodium carbonate, $\text{Na}_2\text{CO}_3$, and sodium hydrogen carbonate, $\text{NaHCO}_3$.

When heated, sodium hydrogen carbonate decomposes.

$$2\text{NaHCO}_3(\text{s}) \rightarrow \text{Na}_2\text{CO}_3(\text{s}) + \text{CO}_2(\text{g}) + \text{H}_2\text{O}(\text{g})$$

Anhydrous sodium carbonate does not decompose when heated.

You are to determine if sodium hydrogen carbonate is the major component, by mass, of the mixture in FB 3.

(a) Method – Read through the instructions before starting any practical work.

- Weigh and record the mass of an empty boiling-tube.
- Tip the contents of the tube labelled FB 3 into the weighed boiling-tube. Reweigh and record the mass of the boiling-tube and FB 3.
- Gently heat the FB 3 in the boiling-tube for 2 minutes then heat strongly for a further 2 minutes. Take care not to lose any solid from the tube during heating.
- Warm the upper parts of the boiling-tube to evaporate any water that may have condensed while heating the solid.
- Place the hot tube on a heat-proof mat and leave to cool.
- You are advised to continue with part (d) of this question or to start another question while the tube cools.
- When cool, reweigh the boiling-tube and the residual sodium carbonate.
- Reheat, cool and reweigh the tube until you are satisfied decomposition is complete.

Results

In an appropriate form, in the space below, record all of your balance readings, the mass of FB 3 heated, the mass of residual sodium carbonate and the mass loss on heating.
Calculations

Do not use your experimental results in part (i)

(b) (i) Use the equation for the decomposition of NaHCO₃ on heating to calculate the theoretical ratio of \( \frac{\text{mass of NaHCO}_3}{\text{mass loss on heating}} \).

\[
2\text{NaHCO}_3(s) \rightarrow \text{Na}_2\text{CO}_3(s) + \text{CO}_2(g) + \text{H}_2\text{O}(g)
\]

\[M_r: \text{NaHCO}_3, 84.0; \text{CO}_2, 44.0; \text{H}_2\text{O}, 18.0\]

Theoretical ratio = ...........................................

(ii) Use the following expression to calculate the mass of NaHCO₃ in the sample of FB 3 that was heated.

Theoretical ratio from (b(i)) \( \times \) experimental mass loss from (a)

\[
\text{mass of NaHCO}_3 = \text{........................................... g}
\]

(iii) Tick the appropriate box in the table below.

\[
\begin{array}{|c|c|}
\hline
\text{NaHCO}_3 & \text{is} & \text{the major component, by mass, in FB 3} \\
\hline
\text{NaHCO}_3 & \text{is not} & \text{the major component, by mass, in FB 3} \\
\hline
\end{array}
\]

Justify your answer with supporting evidence.

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

[2]
(c) Do not carry out your suggestions.

Suggest two ways in which you could show that sodium carbonate does not decompose on heating.

(i) ........................................................................................................................................
........................................................................................................................................

(ii) ...................................................................................................................................... [2]
........................................................................................................................................
........................................................................................................................................

(d) A student is asked to weigh, with maximum precision, a solid.

The three balances available are:

- balance A, reading to 1 decimal place,
- balance B, reading to 2 decimal places,
- balance C, reading to 3 decimal places.

The smallest division on a burette is 0.1 cm³.
The maximum error in a single burette reading is ±0.05 cm³.

Balance readings can be treated in the same way.

Complete the following table.

<table>
<thead>
<tr>
<th>balance</th>
<th>maximum error for a single balance reading / g</th>
<th>maximum % error when weighing:</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>±</td>
<td>9.0 g of solid =</td>
</tr>
<tr>
<td>B</td>
<td>±</td>
<td>4.00 g of solid =</td>
</tr>
<tr>
<td>C</td>
<td>±</td>
<td>0.500 g of solid =</td>
</tr>
</tbody>
</table>

[2]

[Total: 12]
3 FB 4, FB 5, FB 6 and FB 7 are aqueous solutions each containing one of the ions $\text{Al}^{3+}$, $\text{NH}_4^+$, $\text{Mg}^{2+}$, $\text{Mn}^{2+}$.

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

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.

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

<table>
<thead>
<tr>
<th>test</th>
<th>observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>(i)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>To 1 cm depth of solution in a test-tube add 1 cm depth of aqueous sodium hydroxide. Swirl the tube, then add a further 2 cm depth of aqueous sodium hydroxide.</td>
</tr>
<tr>
<td>(ii)</td>
<td>If a precipitate remains at the end of test (i) leave the test-tube and contents to stand for a few minutes.</td>
</tr>
<tr>
<td>(iii)</td>
<td>If no precipitate formed at all in test (i) tip the contents of the tube into a boiling-tube and warm gently. Care: heated solutions containing sodium hydroxide are liable to be ejected from the tube.</td>
</tr>
</tbody>
</table>

In tests (ii) and (iii) put a cross in any boxes where the test is not carried out.
(iv) To 1 cm depth of solution in a test-tube add 1 cm depth of aqueous ammonia. Swirl the tube, then add a further 2 cm depth of aqueous ammonia.

(b) Use the Qualitative Analysis Notes on page 13 to identify the cation present in each of the solutions. Complete the table below to identify each ion and to give supporting evidence from your observations.

<table>
<thead>
<tr>
<th>solution</th>
<th>cation</th>
<th>supporting evidence</th>
</tr>
</thead>
<tbody>
<tr>
<td>FB 4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>FB 5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>FB 6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>FB 7</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Rinse and re-use test-tubes where possible.
(c) Carry out the following tests on the solution you have identified as containing Al\(^{3+}\) ions and record your observations in the spaces provided.

<table>
<thead>
<tr>
<th></th>
<th>observation</th>
</tr>
</thead>
<tbody>
<tr>
<td>(i)</td>
<td>Add aqueous sodium iodide</td>
</tr>
<tr>
<td>(ii)</td>
<td>Add dilute sulfuric acid</td>
</tr>
</tbody>
</table>

Explain how your results confirm the presence of Al\(^{3+}\) and eliminate any other ion.

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

(d) What other cation listed in the Qualitative Analysis Notes on page 13 would give similar results to Al\(^{3+}\) in (a)?

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

(e) Carry out the following tests and make careful observations of all that happens in each experiment. Complete the table.

<table>
<thead>
<tr>
<th>test</th>
<th>observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>(i)</td>
<td>To 1 cm depth of aqueous silver nitrate in a test-tube add 1 cm depth of aqueous sodium chloride. Keep the tube for comparison with the observations in test (ii).</td>
</tr>
<tr>
<td>(ii)</td>
<td>Repeat test (i). To 1 cm depth of aqueous silver nitrate in a test-tube add 1 cm depth of aqueous sodium chloride, then add 1 cm depth of aqueous sodium iodide and shake the tube. <strong>Do not repeat your observations from test (i)</strong></td>
</tr>
</tbody>
</table>

(f) Suggest an explanation for your observations when aqueous sodium iodide is added in test (e)(ii).

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

[Total: 15]
## 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. soluble in excess</td>
<td>white ppt. insoluble in excess</td>
</tr>
<tr>
<td>ammonium, NH₄⁺(aq)</td>
<td>no ppt. 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 giving dark green solution</td>
<td>grey-green ppt. insoluble in excess</td>
</tr>
<tr>
<td>copper(II), Cu²⁺(aq)</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), Fe²⁺(aq)</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), Fe³⁺(aq)</td>
<td>red-brown ppt. insoluble in excess</td>
<td>red-brown ppt. insoluble in excess</td>
</tr>
<tr>
<td>lead(II), Pb²⁺(aq)</td>
<td>white ppt. soluble in excess</td>
<td>white ppt. insoluble in excess</td>
</tr>
<tr>
<td>magnesium, Mg²⁺(aq)</td>
<td>white ppt. insoluble in excess</td>
<td>white ppt. insoluble in excess</td>
</tr>
<tr>
<td>manganese(II), Mn²⁺(aq)</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, Zn²⁺(aq)</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, ( \text{CO}_3^{2-} )</td>
<td>( \text{CO}_2 ) liberated by dilute acids</td>
</tr>
<tr>
<td>chromate(VI), ( \text{CrO}_4^{2-} ) (aq)</td>
<td>yellow solution turns orange with ( \text{H}^+(\text{aq}) ); gives yellow ppt. with ( \text{Ba}^{2+}(\text{aq}) ); gives bright yellow ppt. with ( \text{Pb}^{2+}(\text{aq}) )</td>
</tr>
<tr>
<td>chloride, ( \text{Cl}^- ) (aq)</td>
<td>gives white ppt. with ( \text{Ag}^+(\text{aq}) ) (soluble in ( \text{NH}_3(\text{aq}) )); gives white ppt. with ( \text{Pb}^{2+}(\text{aq}) )</td>
</tr>
<tr>
<td>bromide, ( \text{Br}^- ) (aq)</td>
<td>gives cream ppt. with ( \text{Ag}^+(\text{aq}) ) (partially soluble in ( \text{NH}_3(\text{aq}) )); gives white ppt. with ( \text{Pb}^{2+}(\text{aq}) )</td>
</tr>
<tr>
<td>iodide, ( \text{I}^- ) (aq)</td>
<td>gives yellow ppt. with ( \text{Ag}^+(\text{aq}) ) (insoluble in ( \text{NH}_3(\text{aq}) )); gives yellow ppt. with ( \text{Pb}^{2+}(\text{aq}) )</td>
</tr>
<tr>
<td>nitrate, ( \text{NO}_3^- ) (aq)</td>
<td>( \text{NH}_3 ) liberated on heating with ( \text{OH}^-(\text{aq}) ) and ( \text{Al} ) foil</td>
</tr>
<tr>
<td>nitrite, ( \text{NO}_2^- ) (aq)</td>
<td>( \text{NH}_3 ) liberated on heating with ( \text{OH}^-(\text{aq}) ) and ( \text{Al} ) foil; NO liberated by dilute acids (colourless ( \text{NO} ) → (pale) brown ( \text{NO}_2 ) in air)</td>
</tr>
<tr>
<td>sulfate, ( \text{SO}_4^{2-} ) (aq)</td>
<td>gives white ppt. with ( \text{Ba}^{2+}(\text{aq}) ) or with ( \text{Pb}^{2+}(\text{aq}) ) (insoluble in excess dilute strong acid)</td>
</tr>
<tr>
<td>sulfite, ( \text{SO}_3^{2-} ) (aq)</td>
<td>( \text{SO}_2 ) liberated with dilute acids; gives white ppt. with ( \text{Ba}^{2+}(\text{aq}) ) (soluble in excess dilute strong acid)</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, ( \text{NH}_3 )</td>
<td>turns damp red litmus paper blue</td>
</tr>
<tr>
<td>carbon dioxide, ( \text{CO}_2 )</td>
<td>gives a white ppt. with limewater (ppt. dissolves with excess ( \text{CO}_2 ))</td>
</tr>
<tr>
<td>chlorine, ( \text{Cl}_2 )</td>
<td>bleaches damp litmus paper</td>
</tr>
<tr>
<td>hydrogen, ( \text{H}_2 )</td>
<td>&quot;pops&quot; with a lighted splint</td>
</tr>
<tr>
<td>oxygen, ( \text{O}_2 )</td>
<td>relights a glowing splint</td>
</tr>
<tr>
<td>sulfur dioxide, ( \text{SO}_2 )</td>
<td>turns acidified aqueous potassium dichromate(VI) from orange to green</td>
</tr>
</tbody>
</table>