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 10 and 11.

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.
1 FA 1 is sulfuric acid, H₂SO₄, of approximate concentration 0.7 mol dm⁻³. FA 2 is 0.150 mol dm⁻³ sodium hydroxide. You are also provided with phenolphthalein (indicator).

You will determine the exact concentration of FA 1 by titration.

H₂SO₄(aq) + 2NaOH(aq) → Na₂SO₄(aq) + 2H₂O(l)

(a) Method

Dilution

- Pipette 25.0 cm³ of FA 1 into the 250 cm³ graduated (volumetric) flask labelled FA 3.
- Make the solution up to the mark using distilled water.
- Shake the flask to mix the solution of FA 3.

Titration

- Rinse out the pipette with distilled water and then with FA 3.
- Pipette 25.0 cm³ of FA 3 into a conical flask.
- Add 5 drops of phenolphthalein indicator to the flask. The indicator should remain colourless.
- Fill the burette with FA 2.
- Titrate FA 3 with FA 2, until a permanent pale 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 ........... cm³.

- 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 FA 2 added in each accurate titration.
- Make sure that your recorded results show the precision of your practical work.

(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 3 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 how many moles of NaOH were present in the volume of FA 2 calculated in (b).

............... mol of NaOH

(ii) Calculate how many moles of H₂SO₄ were present in 25.0 cm³ of FA 3.

\[
\text{H}_2\text{SO}_4(aq) + 2\text{NaOH(aq)} \rightarrow \text{Na}_2\text{SO}_4(aq) + 2\text{H}_2\text{O(l)}
\]

............... mol of H₂SO₄

(iii) Calculate how many moles of H₂SO₄ were present in 25.0 cm³ of the undiluted solution FA 1.

............... mol of H₂SO₄

(iv) Calculate the concentration, in mol dm⁻³, of H₂SO₄ in FA 1.

The concentration of H₂SO₄ in FA 1 was ............... mol dm⁻³. [4]

[Total: 12]
2 You will determine, using Hess’ Law, the enthalpy change, ΔH₁, for the reaction of magnesium with oxygen to form magnesium oxide.

\[
\text{Mg(s) + } \frac{1}{2}\text{O}_2(g) \rightarrow \text{MgO(s)}
\]

(a) Reaction of magnesium with sulfuric acid

Method

FA 4 is 0.64 mol dm\(^{-3}\) sulfuric acid.
FA 5 is magnesium turnings. This is supplied in two containers.

You will carry out the experiment twice.

- Support the plastic cup in a 250 cm\(^3\) beaker.
- Using a measuring cylinder, transfer 25 cm\(^3\) of FA 4 into the plastic cup.
- Tilt the beaker so that the bulb of the thermometer is covered by the solution. Measure and record the initial temperature of the solution.
- Carefully, add all the FA 5 from one of the containers into the plastic cup.
- Stir the mixture constantly with the thermometer.
- Record the highest temperature obtained.
- Empty and rinse the plastic cup and dry it with a paper towel.
- Repeat the experiment using the second portion of FA 5.

In the space below, record all your readings in an appropriate form.
Calculate the mean temperature rise.

mean temperature rise = ....................... °C [5]

Calculation

Show your working and express your answers to three significant figures.

(i) Using the mean temperature rise above, calculate the mean heat energy produced in the reaction.
(You may assume that 4.3 J are required to raise the temperature of 1.0 cm\(^3\) of any solution by 1.0°C.)

heat energy produced = ....................... ............
value unit
(ii) Calculate the enthalpy change, $\Delta H_2$, in kJ mol$^{-1}$, for the following reaction.

\[
\text{Mg(s) + H}_2\text{SO}_4(\text{aq}) \rightarrow \text{MgSO}_4(\text{aq}) + \text{H}_2(\text{g})
\]

You should assume that the magnesium in your reaction is in excess.

\[\Delta H_2 = \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \text{kJ mol}^{-1} \quad [2]\]

(b) Reaction of magnesium oxide with sulfuric acid

Method

FA 4 is 0.64 mol dm$^{-3}$ sulfuric acid.
FA 6 is magnesium oxide.

- Using a measuring cylinder, transfer 50 cm$^3$ of FA 4 into a 250 cm$^3$ beaker.
- Place the beaker on a tripod and gauze, and heat gently until the temperature of the acid reaches 45 °C–60 °C.
- Support a plastic cup in a 250 cm$^3$ beaker.
- Transfer all the solution of hot FA 4 into the plastic cup.
- Stir and record the temperature of hot FA 4.
- Immediately add all the FA 6 to the FA 4 in the plastic cup.
- Stir the mixture constantly with the thermometer.
- Record the highest temperature obtained.

In the space below, record all your readings in an appropriate form.
Calculation

Show your working and express your answers to **three** significant figures.

(i) Calculate the heat energy produced in the reaction.
(You may assume that 4.3 J are required to raise the temperature of 1.0 cm³ of any solution by 1.0 °C.)

\[ \text{heat energy produced} = \ldots \ldots \ldots \text{value} \text{ unit} \]

(ii) Calculate the enthalpy change, \( \Delta H_3 \), in kJ mol\(^{-1} \), for the following reaction.
\[ \text{MgO(s) + H}_2\text{SO}_4(\text{aq}) \rightarrow \text{MgSO}_4(\text{aq}) + \text{H}_2\text{O(l)} \]
You should assume that the magnesium oxide in your reaction is in excess.

\[ \Delta H_3 = \ldots \ldots \ldots \text{kJ mol}^{-1} \]

(iii) The enthalpy change for the following reaction is \(-286\) kJ mol\(^{-1} \).
\[ \text{H}_2(\text{g}) + \frac{1}{2}\text{O}_2(\text{g}) \rightarrow \text{H}_2\text{O(l)} \quad \Delta H = -286\text{kJ mol}^{-1} \]
Use the Hess’ Law cycle given below to calculate \( \Delta H_1 \), the enthalpy change for the reaction of magnesium with oxygen.

\[ \Delta H_1 = \ldots \ldots \ldots \text{kJ mol}^{-1} \quad [3] \]

(c) Suggest one improvement to the method by which heat losses from your apparatus could have been reduced.

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

[Total: 14]
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

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 on pages 10 and 11.

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.

<table>
<thead>
<tr>
<th>test</th>
<th>observation(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Add 5 drops of aqueous barium chloride (or barium nitrate) to your solution of <strong>FA 7</strong>.</td>
<td></td>
</tr>
<tr>
<td>Add 5 drops of aqueous silver nitrate to your solution of <strong>FA 7</strong>.</td>
<td></td>
</tr>
</tbody>
</table>
(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 on pages 10 and 11 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.

(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.

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

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

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

...........................................................................................................................................
(b) FA 8 contains one cation from those listed on page 10 and 11.

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 cm$^3$ 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.

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

(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.

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

[Total: 14]
Key: [ppt. = precipitate]

1 Reactions of aqueous cations

<table>
<thead>
<tr>
<th>ion</th>
<th>NaOH(aq)</th>
<th>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</td>
<td>green ppt. turning brown on contact with</td>
</tr>
<tr>
<td></td>
<td>air</td>
<td>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</td>
<td>off-white ppt. rapidly turning brown on</td>
</tr>
<tr>
<td></td>
<td>contact with air</td>
<td>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, $\text{CO}_3^{2-}$</td>
<td>$\text{CO}_2$ liberated by dilute acids</td>
</tr>
<tr>
<td>chromate(\text{VI}), $\text{CrO}_4^{2-}$ (aq)</td>
<td>yellow solution turns orange with $\text{H}^+$ (aq); gives yellow ppt. with $\text{Ba}^{2+}$ (aq); gives bright yellow ppt. with $\text{Pb}^{2+}$ (aq)</td>
</tr>
<tr>
<td>chloride, $\text{Cl}^-$ (aq)</td>
<td>gives white ppt. with $\text{Ag}^+$ (aq) (soluble in NH$_3$(aq)); gives white ppt. with $\text{Pb}^{2+}$ (aq)</td>
</tr>
<tr>
<td>bromide, $\text{Br}^-$ (aq)</td>
<td>gives cream ppt. with $\text{Ag}^+$ (aq) (partially soluble in NH$_3$(aq)); gives white ppt. with $\text{Pb}^{2+}$ (aq)</td>
</tr>
<tr>
<td>iodide, $\text{I}^-$ (aq)</td>
<td>gives yellow ppt. with $\text{Ag}^+$ (aq) (insoluble in NH$_3$(aq)); gives yellow ppt. with $\text{Pb}^{2+}$ (aq)</td>
</tr>
<tr>
<td>nitrate, $\text{NO}_3^-$ (aq)</td>
<td>NH$_3$ liberated on heating with OH$^-$ (aq) and Al foil</td>
</tr>
<tr>
<td>nitrite, $\text{NO}_2^-$ (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, $\text{SO}_4^{2-}$ (aq)</td>
<td>gives white ppt. with $\text{Ba}^{2+}$ (aq) or with $\text{Pb}^{2+}$ (aq) (insoluble in excess dilute strong acids)</td>
</tr>
<tr>
<td>sulfite, $\text{SO}_3^{2-}$ (aq)</td>
<td>SO$_2$ liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}$ (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(\text{VI}) from orange to green</td>
</tr>
</tbody>
</table>