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

CANDIDATE NAME

CENTRE NUMBER

CANDIDATE NUMBER

5651142458

CHEMISTRY **9701/33**

Advanced Practical Skills 1 **October/November 2013**

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.
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.

Session
Laboratory

For Examiner's Use	
1	
2	
3	
Total	

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

UNIVERSITY *of* CAMBRIDGE
International Examinations

[Turn over

For Examiner's Use

1 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_2SO_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.

I	
II	
III	
IV	
V	
VI	
VII	

[7]

(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 cm^3 of **FA 4** required .... cm^3 of **FA 2** [1]

For Examiner's Use

(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)**.

moles of $KMnO_4$ = ... mol

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

$$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$$

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**.

moles of Fe^{2+} in 25.0 cm^3 of **FA 4** = ... mol

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

moles of Fe^{2+} in 250 cm^3 of **FA 4** = ... 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.

relative formula mass = ...

[5]

I	
II	
III	
IV	
V	

For Examiner's Use

(d) (i) A 25 cm^3 pipette is accurate to $\pm0.06\,cm^3$.
Calculate the maximum percentage error when the pipette was used to measure solution **FA 4**.

percentage error in measuring **FA 4** = ... %

(ii) State the maximum error in the mass of the 250 cm^3 beaker that you recorded in **(a)**.

maximum error = ... g

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

maximum percentage error = ... %
[2]

[Total: 15]

For Examiner's Use

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, $BaCl_2.\mathbf{x}H_2O$

(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**.

I	
II	
III	
IV	
V	
VI	

[6]

For Examiner's Use

(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)

moles of H_2O = ... mol

(ii) Calculate the **mean** number of moles of anhydrous barium chloride produced in the experiments.
(A_r : Ba, 137; Cl, 35.5)

moles of $BaCl_2$ = ... mol

(iii) Calculate the value of **x** in the formula of hydrated barium chloride, $BaCl_2.\mathbf{x}H_2O$.

x = ...

[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.

[3]

[Total: 12]

For Examiner's Use

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.

test	*observations*
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.	
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.	

For Examiner's Use

(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.

[7]

[Turn over

For Examiner's Use

(b) **FA 7**, **FA 8** and **FA 9** are aqueous solutions. Each contains **one** cation and **one** anion. **FA 3** is dilute sulfuric acid, H_2SO_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.

	FA 7	**FA 8**	**FA 9**
FA 3			
FA 7			
FA 8			

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

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 $\rightarrow$ (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