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

CANDIDATE NAME

CENTRE NUMBER ☐☐☐☐☐

CANDIDATE NUMBER ☐☐☐☐

7264067875

CHEMISTRY **9701/33**

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.

Session
Laboratory

For Examiner's Use	
1	
2	
3	
Total	

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

[Turn over

1 **FA 1** is an iron salt in which all the iron is present as Fe^{2+} cations. You will work out the percentage of iron in this salt by titrating a solution of this salt with a standard solution aqueous potassium manganate(VII).

FA 1 is an unknown iron(II) salt.
FA 2 is 1.00 mol dm^{-3} sulfuric acid.
FA 3 is 0.0100 mol dm^{-3} potassium manganate(VII).

(a) Method

Weighing out the salt

- Weigh the tube containing **FA 1**.
- Tip the contents of the tube into a 250 cm^3 beaker.
- Re-weigh the empty tube.
- Record all your readings in a suitable form in the space below.

Preparing the solution

- To the salt in the beaker use a measuring cylinder to add approximately 200 cm^3 of **FA 2** and stir until the salt has dissolved.
- Pour the contents of the beaker carefully into the 250 cm^3 graduated (volumetric) flask using the small funnel.
- Rinse the contents of the beaker twice with a little distilled water and add these washings to the graduated flask.
- Fill the graduated flask to the line with distilled water. Shake carefully to ensure adequate mixing.

Titration

- Fill the burette with **FA 3**.
- Pipette 25.0 cm^3 of the solution of **FA 1** from the graduated flask into a conical flask.
- Titrate the solution of **FA 1** in the flask with **FA 3** until the first appearance of a permanent pink colour.

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 are 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 **FA 3** added in each accurate titration.

For Examiner's Use

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 the solution of **FA 1** required .... cm^3 of **FA 3**.
[2]

(c) Calculations

For Examiner's Use

Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

(i) Calculate how many moles of $MnO_4^-(aq)$ were present in the volume of **FA 3** calculated in **(b)**.

moles of $MnO_4^-(aq)$ = ... mol

(ii) Use the following equation to calculate how many moles of $Fe^{2+}(aq)$ were present in the conical flask.

$$MnO_4^-(aq) + 8H^+(aq) + 5Fe^{2+}(aq) \rightarrow Mn^{2+}(aq) + 5Fe^{3+}(aq) + 8H_2O(aq)$$

moles of $Fe^{2+}(aq)$ in the conical flask = ... mol

(iii) Calculate the number of moles of Fe^{2+} in your weighed sample of **FA 1**.

moles of Fe^{2+} in the weighed sample = ... mol

I	
II	
III	
IV	
V	

(iv) Calculate the percentage of iron in **FA 1**.
[A_r: Fe, 55.8]

the percentage of iron in **FA 1** = ... %
[5]

(d) There are a number of sources of potential error in this experiment. One of these involves the readings taken using the balance.

(i) State the maximum individual error in any single balance reading.

maximum individual error = ... g

(ii) Calculate the maximum percentage error in the mass of **FA 1** used in your experiment.

maximum percentage error = ... % [2]

[Total: 16]

2 **FA 4** is an **impure** sample of hydrated magnesium sulfate, $MgSO_4.7H_2O$. When heated the water of crystallisation is driven off to leave anhydrous magnesium sulfate, $MgSO_4$. The impurity does not give off water when heated. By determining how much water is present in the impure sample, the percentage purity can be calculated.

For Examiner's Use

(a) Method

- Weigh a clean dry crucible.
- Empty all of the **FA 4** into the crucible.
- Reweigh the crucible and its contents.
- Support the crucible in the pipe-clay triangle on top of a tripod.
- Heat the crucible gently for about 1 minute and then more strongly for a further 4 minutes.
- Allow the crucible to cool. You should start question 3 while cooling is taking place.
- When the crucible is cool enough to handle, reweigh the crucible and its contents.
- Repeat the cycle of heating and weighing as many times as you think necessary.

In the space below, record, in an appropriate form, all your weighings and include the mass of **FA 4** used and the mass of water that was lost.

I	
II	
III	
IV	
V	

[5]

(b) Calculations

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

(i) Using the mass of water that was lost on heating, calculate the mass of $MgSO_4.7H_2O$ that was present in the initial sample of **FA 4**.
[A_r: H, 1.0; O, 16.0; Mg, 24.3; S, 32.1]

mass of $MgSO_4.7H_2O$ = ... g [1]

(ii) Calculate the percentage by mass of $MgSO_4.7H_2O$ in **FA 4**.

percentage by mass of $MgSO_4.7H_2O$ in **FA 4** = ... % [1]

(c) Suggest an improvement to the practical procedure that would give a more accurate value for the percentage by mass of $MgSO_4.7H_2O$ in **FA 4**.

....

....

.... [1]

[Total: 8]

Qualitative Analysis

3 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.

FA 5 is a sodium salt.
FA 6 is a salt containing a single cation and a single anion from those listed in the Qualitative Analysis Notes on pages 11 and 12.
FA 7 is an aqueous solution of an unknown compound.

By carrying out specific tests you will identify some of the ions in these.

(a) Put a spatula measure of **FA 5** into a boiling tube. Heat it gently for a few minutes and then strongly until no further changes are seen.
Record your observations at each stage, in the space below.

[2]

Leave the boiling tube to cool. Do not discard the contents as they will be used later in the question.

(b) Put a spatula measure of **FA 6** into a test-tube. Half fill the test-tube with distilled water and dissolve the solid. You will use this solution to carry out the following tests.

- To a 1 cm depth of a solution of **FA 6** in a boiling tube, add 0.5 cm depth of aqueous sodium hydroxide using a teat pipette. Heat the mixture carefully.
- To a 1 cm depth of a solution of **FA 6** in a test-tube, add aqueous ammonia.
- To a 1 cm depth of a solution of **FA 6** in a test-tube, add aqueous barium chloride or barium nitrate.
- To a 1 cm depth of a solution of **FA 6** in a test-tube, add aqueous silver nitrate, followed by aqueous ammonia.

Record your observations for each of the tests in the space below.
Identify the ions present in **FA 6**.

FA 6 contains .... and .... [6]

(c) Put a small spatula measure of **FA 6** into a boiling tube. Taking great care, add **5 drops** of concentrated sulfuric acid.
CARE: Concentrated sulfuric acid is very corrosive.

Once you have made your observations fill the boiling tube with water.
Record your observations in the space below.

What type of chemical reaction occurs between **FA 6** and sulfuric acid?
Justify your answer.

....

.... [3]

(d) To the residue in the boiling tube from **(a)**, slowly and carefully add **FA 7** to a depth of about 5 cm.
Divide this solution equally into two test-tubes.

- To one test-tube add 5 drops of aqueous lead nitrate.
- To the other test-tube add 5 drops of aqueous silver nitrate.

Record your observations for each test in the space below.

[2]

(e) Use the information in the Qualitative Analysis Notes on pages 11 and 12 to select one test to confirm the identity of the cation in **FA 7** and one test to confirm the identity of the anion in **FA 7**.
Carry out both tests and record your observations for each of the tests in the space below.
Identify the ions present in **FA 7**.

FA 7 contains .... and .... [3]

[Total: 16]

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.]

[Turn over

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

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