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

CANDIDATE NAME	

CENTRE NUMBER						CANDIDATE NUMBER					

CHEMISTRY **9701/31**

Paper 31 Advanced Practical Skills **May/June 2008**

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 are advised to show all working in calculations.
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	
Total	

This document consists of **11** printed pages and **1** blank page.

SPA (NF/KN) T58586/1

UNIVERSITY *of* CAMBRIDGE
International Examinations

[Turn over

1 **Read through question 1 before starting any practical work.**

You are provided with the following reagents.

- **FA 1** containing 15.68 g dm^{-3} of hydrated ammonium iron(II) sulphate $(NH_4)_2SO_4.FeSO_4.6H_2O$
- **FA 2**, 0.015 mol dm^{-3} potassium manganate(VII), $KMnO_4$
- **FA 3** containing 0.025 mol dm^{-3} of a reagent **X**
- 1.0 mol dm^{-3} sulphuric acid, H_2SO_4

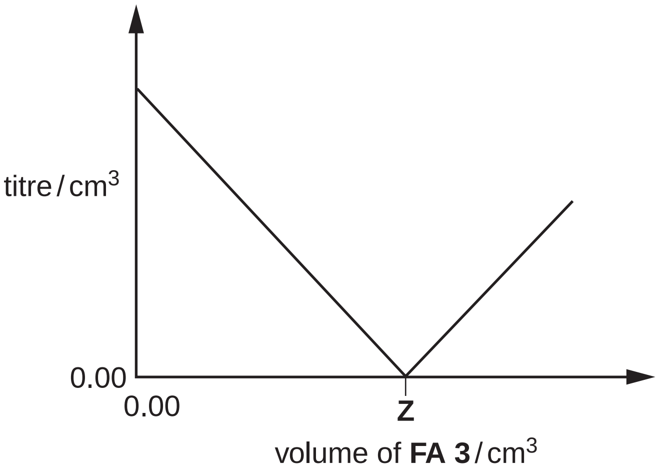
Iron(II) ions, Fe^{2+}, are oxidised by acidified manganate(VII) ions.

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

$$Fe^{2+}(aq) \rightarrow Fe^{3+}(aq) + e^-$$

Reagent **X** oxidises Fe^{2+} to Fe^{3+} and is also oxidised by acidified MnO_4^-.

If varying volumes of **FA 3**, containing reagent **X**, are added to 25.0 cm^3 of **FA 1** in the presence of H_2SO_4 and the mixtures are titrated against **FA 2**, a graph of the results can be drawn as shown.



You are to determine experimentally

- **Z**, the exact volume of **FA 3** which reacts with 25.0 cm^3 of **FA 1**,
- the mole ratio for the reaction of **FA 1** with reagent **X**.

(a) Method

- Fill a burette with **FA 2**.
- Pipette 25.0 cm^3 of **FA 1** into a conical flask.
- Use a measuring cylinder to add approximately 10 cm^3 of 1.0 mol dm^{-3} H_2SO_4 to the solution in the flask.
- Titrate the **FA 1** in the flask with **FA 2** until the first permanent pink colour remains in the solution.

The end-point should be found after the addition of approximately 13 cm^3 of **FA 2**.
One titration, performed accurately, will be sufficient.

You are reminded that just before the end-point the pink colour from a single drop of **FA 2** spreads through the whole of the solution before disappearing.

Record your titration results in the space below.

[1]

(b) **Method**

- Fill the second burette with **FA 3**.
- Empty and rinse the conical flask used in part **(a)**.
- Pipette 25.0 cm^3 of **FA 1** into the conical flask and add 10 cm^3 of H_2SO_4 using a measuring cylinder.
- Run 12.00 cm^3 of **FA 3** from the second burette into the flask.
- Titrate against **FA 2** until the first permanent pink colour remains in the solution.

The end-point should be found after the addition of approximately 5 cm^3 of **FA 2**.
One titration, performed accurately, will be sufficient.

You are reminded that just before the end-point the pink colour from a single drop of **FA 2** spreads through the whole of the solution before disappearing.

Record your titration results in the space below.

[1]

(c) The volume of **FA 3** added to the flask in **(b)** reacts with some but not all of the **FA 1** present. Calculate the difference between the titres obtained in parts **(a)** and **(b)**. Use this difference and the volume of **FA 3** added to the flask in **(b)** to calculate the volume of **FA 3** that you would expect to react with all of the Fe^{2+} ions in 25.0 cm^3 of **FA 1**.

volume of **FA 3** = ... cm^3
[1]

(d) The value you have obtained in **(c)** is an **approximate** value of **Z**. You are to perform four more titrations, **each** with a **different** volume of **FA 3** added to 25.0 cm^3 of **FA 1**, in order to plot a graph of the form shown on page 2 and to obtain an exact value for **Z**.

One titration, performed accurately, will be sufficient for each volume of **FA 3** added.

The volume of **FA 3** you have obtained in **(c)** will help you to choose suitable volumes of **FA 3** to be added for each titration.
(If you were unable to calculate the volume of **FA 3** in **(c)** assume that the value lies in the range 19.0 cm^3 to 21.0 cm^3.)

Remember

- you should not use more than 40.0 cm^3 of **FA 3** for any single titration,
- you already have data for titrations with no **FA 3** added and with 12.00 cm^3 added.

Prepare a table in the space below and use it to record the titration results for each volume of **FA 3** added. Include in your table the titre values from parts **(a)** and **(b)**.

[6]

(e) Use the grid on the opposite page to plot a graph of titre against volume of **FA 3** added.

Draw **two** straight lines through the plotted points to find **Z**, the volume of **FA 3** that just reacts with the Fe^{2+} ions in 25.0 cm^3 of **FA 1**.

Z, the volume of **FA 3** read from the graph is .... cm^3.

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[7]

(f) Circle on the graph **one** point where a repeat titration might be appropriate and justify you decision. If you do not think that any titration needs to be repeated, explain why you have come to that conclusion.

....

....

....
[1]

Calculations

Show your working and appropriate significant figures in **all** of your calculations. [2]

(g) Calculate how many moles of Fe^{2+} ions were pipetted into the flask.
[A_r: Fe, 55.8; H, 1.0; N, 14.0; O, 16.0; S, 32.1]

.... mol of Fe^{2+} were pipetted into the flask. [2]

(h) Calculate how many moles of **X** are present in **Z**, the volume of **FA 3** read from your graph.

.... mol of **X** were present in .... cm^3 of **FA 3**. [1]

(i) Calculate, to 3 significant figures, the number of moles of Fe^{2+} ions that react with 1 mol of **X**.

[2]

[Total: 24]

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2 The three solutions **FA 4**, **FA 5**, and **FA 6 each** contain **one** of the following.

aluminium sulphate, $Al_2(SO_4)_3$
ammonium iodide, NH_4I
zinc nitrate, $Zn(NO_3)_2$

(a) Use the information on page 12 to select two suitable reagents to use to discover which solution contains iodide ions.

Record, in the space below, the reagents used and the observations made.

From these tests, solution **FA** .... contains iodide ions. [5]

You are to perform the tests given in the table opposite on each of **FA 4**, **FA 5** and **FA 6** to identify, where possible, the cation and anion present in each solution.

Record details of colour changes seen, the formation of any precipitate and the solubility of any such precipitate 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 table.
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.

test	observations with **FA 4**	observations with **FA 5**	observations with **FA 6**
(b) To 1 cm depth of solution in a test-tube, add aqueous sodium hydroxide drop-by-drop until it is in excess.			
(c) To 1 cm depth of solution in a test-tube, add aqueous ammonia drop-by-drop until it is in excess.			
(d) To 1 cm depth of solution in a test-tube, add aqueous barium chloride, then			
add dilute hydrochloric acid.			
(e) To 1 cm depth of solution in a boiling-tube add 2 cm depth of water and 1 cm depth of aqueous lead(II) nitrate, then			
if a precipitate has formed, cautiously warm until the solution boils, then			
cool the tube by standing it in a beaker of cold water.			

[6]

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(f) For each of the solutions **FA 4**, **FA 5**, and **FA 6**, summarise the evidence from the tests performed to identify the cations and anions present.
State clearly where a cation or an anion has **not** been specifically identified.

FA 4 contains ....

supporting evidence

....

....

....

FA 5 contains ....

supporting evidence

....

....

....

FA 6 contains ....

supporting evidence

....

....

....

[4]

(g) When testing a solution containing both NH_4I and $Zn(NO_3)_2$, suggest why a student should identify the NH_4^+ ion before attempting to identify the NO_3^- ion. The Qualitative Analysis Notes on pages 11 and 12 should help you to answer this.

....

....

....

....

....

....[1]

[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)	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. insoluble in excess	green ppt. 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. insoluble in excess	off-white ppt. 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 pale 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 A*l* foil
nitrite, NO_2^-(aq)	NH_3 liberated on heating with OH^-(aq) and A*l* foil; NO liberated by dilute acids (colourless NO → (pale) brown NO_2 in air)
sulphate, SO_4^{2-} (aq)	gives white ppt. with Ba^{2+}(aq) or with Pb^{2+}(aq) (insoluble in excess dilute strong acids)
sulphite, 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
sulphur dioxide, SO_2	turns potassium dichromate(VI) (aq) from orange to green

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