Centre Number	Candidate Number	Name

SESSION		LABORATORY	

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

CHEMISTRY **9701/03**

Paper 3 Practical Test

May/June 2005

1 hour 15 minutes

Candidates answer on the Question Paper.
Additional Materials: as listed in the Instructions to Supervisors.

READ THESE INSTRUCTIONS FIRST

Write your details, including practical session and laboratory where appropriate, in the boxes provided.
Write in dark blue or black pen in the spaces provided on the Question Paper.
You may use a soft pencil for any diagrams, graphs or rough working.
Do not use staples, paper clips, highlighters, glue or correction fluid.

Answer **all** questions.
The number of marks is given in brackets [ ] at the end of each question or part question.
You are advised to show all working in calculations.
Use of a Data Booklet is unnecessary.
Qualitative Analysis notes are provided on pages 6 and 7.

If you have been given a label, look at the details. If any details are incorrect or missing, please fill in your correct details in the space given at the top of this page.

Stick your personal label here, if provided.

For Examiner's Use	
1	
2	
TOTAL	

This document consists of **7** printed pages and **1** blank page.

SP (SJF3513) S81256/3

UNIVERSITY *of* CAMBRIDGE
International Examinations

[Turn over

1 **FA 1** is a solution containing 5.00 g dm^{-3} of hydrated ethanedioic acid, $H_2C_2O_4.\mathbf{x}H_2O$.
FA 2 is a solution containing 2.37 g dm^{-3} of potassium manganate(VII), $KMnO_4$.
You are also provided with 1.00 mol dm^{-3} sulphuric acid, H_2SO_4.

In the presence of acid, potassium manganate(VII) oxidises ethanedioic acid;

$$2MnO_4^-(aq) + 5H_2C_2O_4(aq) + 6H^+(aq) \rightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

You are to determine the value of **x** in $H_2C_2O_4.\mathbf{x}H_2O$.

(a) Fill the burette with **FA 2**.

Pipette 25.0 cm^3 of **FA 1** into a conical flask. Use the measuring cylinder provided to add to the flask 25 cm^3 of 1.00 mol dm^{-3} sulphuric acid and 40 cm^3 of distilled water.

Heat the solution in the flask until the temperature is just over 65 °C. The exact temperature is not important.

Be careful when handling hot solutions.

Remove the thermometer and carefully place the hot flask under the burette. If the neck of the flask is too hot to hold safely, use a folded paper towel to hold the flask. Run in about 1 cm^3 of **FA 2**. Swirl the flask until the colour of the manganate(VII) ions has disappeared then continue the titration as normal until a permanent pale pink colour is obtained. This is the end point. Record the burette readings in Table 1.1.

If a brown colour appears during the titration, reheat the flask to 65 °C. The brown colour should disappear and the titration can then be completed.

If the brown colour does **not** disappear on reheating, discard the solution and restart the titration.

Repeat the titration as many times as you think necessary to obtain accurate results.

Make certain that the recorded results show the precision of your practical work.

Table 1.1 Titration of FA 1 with FA 2

final burette reading / cm^3 initial burette reading / cm^3				
volume of **FA 2** used / cm^3				

Summary

25.0 cm^3 of **FA 1** reacted with ………………. cm^3 of **FA 2**.

Show which results you used to obtain this volume of **FA 2** by placing a tick (✓) under the readings in Table 1.1.

[7]

You are advised to show full working in all parts of the calculations.

(b) Calculate how many moles of potassium manganate(VII), $KMnO_4$, were run from the burette during the titration.
[A_r: K, 39.1; Mn, 54.9; O, 16.0.]

[2]

(c) Calculate how many moles of ethanedioic acid, $H_2C_2O_4$, reacted with the potassium manganate(VII) run from the burette.

[1]

(d) Calculate the mass of $H_2C_2O_4$ in each dm^3 of **FA 1**
[A_r: H, 1.0; C, 12.0; O, 16.0.]

[3]

(e) Calculate the mass of water in the 5.00 g of $H_2C_2O_4.\mathbf{x}H_2O$.

[1]

(f) Calculate the value of **x**, in $H_2C_2O_4.\mathbf{x}H_2O$.

[1]

[Total: 15]

2 **FA 3** contains **two cations** and **two anions** from those listed on pages 6 and 7.

In all tests, the reagent should be added gradually until no further change is observed, with shaking after each addition.
Record your observations in the spaces provided.
Your answers should include

- details of colour changes and precipitates formed,
- the names of gases evolved and details of the test used to identify each one.

You should indicate clearly at what stage in a test a change occurs.

Marks are **not** given for chemical equations.
No additional or confirmatory tests for ions present should be attempted.

Candidates are reminded that definite deductions may be made from tests where there appears to be no reaction.

Test	*Observations [6]*
(a) To 3 cm depth of **FA 3** in a boiling-tube, add 2 cm depth of dilute sulphuric acid. **Warm** the mixture and leave to stand for several minutes. Continue with test **(b)**. **Care** – mixtures containing precipitates can "bump" when heated and eject the hot acid from the tube.	
Use a teat pipette to transfer 1 cm depth of the solution into a test-tube and add an equal depth of distilled water. Add aqueous sodium hydroxide, drop by drop, until there is no further change.	
(b) To 1 cm depth of **FA 3** in a boiling-tube, add 2 cm depth of aqueous sodium hydroxide. Add a piece of aluminium foil and **warm** the tube. **Care** – solutions containing sodium hydroxide when heated can "bump" and eject the hot alkali from the tube. Remember to complete test **(a)** if you have not yet done so.	
(c) To 3 cm depth of **FA 3** in a boiling-tube, add an equal depth of aqueous ammonia. Filter the solution.	
Add aqueous potassium chromate(VI) to the filtrate.	

Test	*Observations*
(d) To 1 cm depth of **FA 3** in a boiling-tube, add 1 cm depth of aqueous silver nitrate. Warm the mixture and carefully pour away the solution. Wash the precipitate that remains with distilled water and discard the water. Add aqueous ammonia to the washed precipitate.	

Use the information in the Qualitative Analysis Tables on pages 6 and 7 to identify the ions present in **FA 3**. For each ion give one piece of evidence that supports your choice.

Cation 1 present in **FA 3** ……………………………………………….

Evidence for Cation 1

....

.... [1]

Cation 2 present in **FA 3** ……………………………………………….

Evidence for Cation 2

....

.... [1]

Anion 1 present in **FA 3** ……………………………………………….

Evidence for Anion 1

....

.... [1]

Anion 2 present in **FA 3** ……………………………………………….

Evidence for Anion 2

....

.... [1]

[Total: 10]

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 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 acid)
sulphite, SO_3^{2-}(aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in excess dilute strong acid)

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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