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

CANDIDATE NAME	
CENTRE NUMBER	
CANDIDATE NUMBER	

3597482834

CHEMISTRY **9701/34**

Advanced Practical Skills 2 **May/June 2012**

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.
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.

Session
Laboratory

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1	
2	
Total	

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

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1 You are to determine the percentage by mass of sodium ethanedioate in a mixture of sodium ethanedioate and ethanedioic acid.

This experiment involves two steps.
In step one, you will carry out a titration to find the amount of acid, $C_2O_4H_2$, present in **FB 3**.
In step two, you will carry out a second titration to find the total amount of ethanedioate ion, $C_2O_4^{2-}$, present in **FB 3**.
Finally, you will use the values found in the two steps to calculate the percentage by mass of sodium ethanedioate in **FB 3**.

FB 1 is 0.100 mol dm^{-3} sodium hydroxide, NaOH.
FB 2 is 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$.
FB 3 is a mixture of aqueous sodium ethanedioate, $C_2O_4Na_2$, and ethanedioic acid, $C_2O_4H_2$.
FB 4 is approximately 2 mol dm^{-3} sulfuric acid.
phenolphthalein indicator

Read through the whole method before starting any practical work.

(a) Method

Step 1

- Fill the burette labelled **FB 1** with **FB 1**.
- Pipette 25.0 cm^3 of **FB 3** into a conical flask.
- Add about three drops of phenolphthalein.
- 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 certain 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 **FB 1** added in each accurate titration.

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Step 2

- Pipette 25.0 cm^3 of **FB 3** into a conical flask.
- Using a measuring cylinder, add about 25 cm^3 of 2 mol dm^{-3} sulfuric acid, **FB 4**, to the flask.
- Place the conical flask on a tripod and gauze and heat to about 80 °C.
- Fill the burette labelled **FB 2** with **FB 2**.
- Use an appropriate method to carefully transfer the hot conical flask onto a white tile under the burette.
- Titrate the mixture in the conical flask with **FB 2** until a permanent pale pink colour is seen. If a permanent brown colour is seen, stop the titration and begin **Step 2** again.
- Perform a **rough titration** and record your burette readings in the space below.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	
IX	
X	
XI	
XII	

The rough titre is ..... cm^3.

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Make certain 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 **FB 2** added in each accurate titration.

[12]

Calculations

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

(b) (i) From your accurate titration results in **Step 1**, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

25.0 cm^3 of **FB 3** required …….. cm^3 of **FB 1**.

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(ii) Write an equation for the reaction between sodium hydroxide and ethanedioic acid to give sodium ethanedioate and water.

.....

(iii) Use your answer from **(i)** to calculate the number of moles of sodium hydroxide, **FB 1**, required to react with 25.0 cm^3 of **FB 3** in **Step 1**.

moles of NaOH = …….. mol

(iv) Use your answer to **(iii)** to determine the number of moles of ethanedioic acid in 25.0 cm^3 of **FB 3**.

moles of $C_2O_4H_2$ in 25.0 cm^3 of **FB 3** = …….. mol
[2]

(c) (i) From your accurate titration results in **Step 2**, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

25.0 cm^3 of **FB 3** required …….. cm^3 of **FB 2**.

(ii) Use your answer from **(i)** to calculate the number of moles of potassium manganate(VII), **FB 2**, required to react with 25.0 cm^3 of **FB 3** in **Step 2**.

moles of $KMnO_4$ = ... mol

(iii) The equation for the reaction between acidified manganate(VII) ions and ethanedioate ions is shown below.

$$2MnO_4^-(aq) + 5C_2O_4^{2-}(aq) + 16H^+(aq) \rightarrow 2Mn^{2+}(aq) + 10CO_2(g) + 8H_2O(l)$$

Calculate the total number of moles of ethanedioate ions in 25.0 cm^3 of **FB 3**.

total moles of $C_2O_4^{2-}$ in 25.0 cm^3 of **FB 3** = …….. mol

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I	
II	
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IV	

(iv) Use your answers to **(b)(iv)** and **(c)(iii)** to calculate the number of moles of ethanedioate ions which came from the sodium ethanedioate dissolved in 25.0 cm^3 of **FB 3**.

moles of $C_2O_4^{2-}$ from $C_2O_4Na_2$ in 25.0 cm^3 of **FB 3** = …….. mol
[4]

(d) (i) Use your answer to **(b)(iv)** to calculate the mass of ethanedioic acid, $C_2O_4H_2$, in 25.0 cm^3 of **FB 3**.
[A_r: H, 1.0; C, 12.0; O, 16.0]
(If you were unable to answer **(b)(iv)**, you may assume that the number of moles of ethanedioic acid is 6.51×10^{-4} mol.)

mass of ethanedioic acid = ... g

(ii) Use your answer to **(c)(iv)** to calculate the mass of sodium ethanedioate, $C_2O_4Na_2$ in 25.0 cm^3 of **FB 3**.
[A_r: C, 12.0; O, 16.0; Na, 23.0]
(If you were unable to answer **(c)(iv)**, you may assume that the number of moles of sodium ethanedioate is 4.13×10^{-4} mol.)

mass of sodium ethanedioate = ... g

I	
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III	
IV	

(iii) Calculate the percentage by mass of sodium ethanedioate present in **FB 3**.

Percentage by mass of sodium ethanedioate present is ..... %.
[4]

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[Turn over

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(e) **(i)** What is the maximum error in a single burette reading?

maximum error = ... cm^3

(ii) A student suggested that using a burette to measure the 25.0 cm^3 of acid would give a more accurate result than using a pipette. The percentage error of a 25.0 cm^3 pipette is 0.24 %. Is the student correct? Explain your answer.

.....

.....

.....
[2]

(f) A student decided to use a 25.0 cm^3 pipette instead of a measuring cylinder to measure the volume of **FB 4** in **Step 2**.
State and explain whether this alteration will improve the accuracy of the calculation of the percentage by mass of sodium ethanedioate in the mixture.

.....

.....

..... [1]

[Total: 25]

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2 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.
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 reagent must be given.

(a) Compounds **FB 5**, **FB 6** and **FB 7** contain the same non-metal but in three different oxidation states. You are provided with solid samples of **FB 5**, **FB 6** and **FB 7**. Carry out the tests described below and record your observations in the table.

test	*observations*
(i) To 1 cm depth of dilute hydrochloric acid in a test-tube add a small spatula measure of **FB 5**.	
(ii) To 1 cm depth of dilute sulfuric acid in a boiling tube add the same depth of aqueous potassium iodide. Add a small spatula measure of **FB 5**.	
(iii) To 1 cm depth of dilute sulfuric acid in a test-tube add about ten drops of aqueous potassium manganate(VII). Add a small spatula measure of **FB 5**.	

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test	observations
(iv) Place a small spatula measure of **FB 6** into a hard glass test-tube. Heat the contents gently.	
(v) Place a small spatula measure of **FB 6** into a boiling tube. Dissolve the solid in 1 cm depth of distilled water. Add 1 cm depth of aqueous sodium hydroxide. Warm the mixture **with care**.	
(vi) Place a small spatula measure of **FB 7** into a hard glass test-tube. Heat the contents gently at first, then heat more strongly. Allow to stand for a few minutes	
(vii) Place a small spatula measure of **FB 7** into a boiling tube. Dissolve the solid in about 1 cm depth of distilled water. Add 1 cm depth of aqueous sodium hydroxide. Warm the mixture **with care**.	

I	
II	
III	
IV	
V	
VI	

[6]

(b) (i) From your observations in **(a)**, identify the non-metal present in **FB 5**, **FB 6** and **FB 7**.

.....

(ii) Suggest the oxidation state of the non-metal in **FB 5** and **FB 6**.

The oxidation state of the non-metal in **FB 5** is .....

The oxidation state of the non-metal in **FB 6** is .....

(iii) Suggest the type of reaction occurring in **(a)(iii)**.

.....

[3]

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(c) Solid compounds containing Fe^{2+} and Ni^{2+} are usually green. One of these ions is present in **FB 8** and the other in **FB 9**. Both **FB8** and **FB9** are aqueous solutions.

(i) Use the Qualitative Analysis Notes on page 10 to select two reagents that, **used in separate tests**, could identify the presence of the Fe^{2+} ion.

The reagents are .....

and .....

(ii) Use your chosen reagents to carry out tests on **both FB 8** and **FB 9**.
Record your results in an appropriate form in the space below.

(iii) From the results of the tests in **(ii)**, state which solution contains the iron(II) ions.

Fe^{2+} ions are contained in solution ..... .

Explain how your observations support your conclusion.

.....

.....

(iv) Aqueous EDTA is a reagent used to identify some transition metals. To 1 cm depth of the solution containing the nickel(II) ion, add 1 cm depth of aqueous EDTA.

observation

.....

.....

(v) State what you would expect to **see** if acidified potassium manganate(VII) was added to a sample of the solution containing the iron(II) ion.
Do not carry out this experiment.

expected observation .....

.....

[6]

[Total: 15]

I	
II	
III	
IV	
V	
VI	

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[Turn over

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.]

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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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