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

CANDIDATE NAME	
CENTRE NUMBER	
CANDIDATE NUMBER	

0014911874

CHEMISTRY **9701/33**

Advanced Practical Skills 1 **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 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

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1	
2	
Total	

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

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1 You are to determine the percentage purity of a sample of calcium carbonate.

This experiment involves three steps.
In step one, you will react the impure calcium carbonate with an excess of acid.
In step two, you will carry out a titration to find the amount of acid you used in step one.
In step three, you will carry out a second titration to find how much (excess) acid remained following the reaction in step one.
Finally, you will use the values found in the three steps to calculate the percentage purity of the calcium carbonate.
Assume the impurity in the calcium carbonate will not react with acid or alkali.

FA 1 is 0.100 mol dm^{-3} sodium hydroxide, NaOH.
FA 2 is approximately 1 mol dm^{-3} hydrochloric acid, HC*l*.
FA 3 is **FA 2** diluted by a factor of 10, approximately 0.1 mol dm^{-3} hydrochloric acid, HC*l*.
FA 4 is a sample of impure calcium carbonate, $CaCO_3$.
methyl orange indicator

Read through the whole method before starting any practical work.

(a) Method

Step 1

- Fill the burette labelled **FA 2** with **FA 2**.
- Run 50.00 cm^3 of **FA 2** into a 250 cm^3 beaker.
- Weigh the tube containing the impure calcium carbonate, **FA 4**.
- Tip the contents of the tube, in small portions, into the acid to avoid acid spray. Stir the mixture and leave the stirring rod in the beaker.
- Reweigh the tube containing any residue.
- Record the weighings and the mass of **FA 4** added in a suitable form below.

Step 2

- Fill the burette labelled **FA 1** with **FA 1**.
- Pipette 25.0 cm^3 of **FA 3** into a conical flask.
- Add methyl orange indicator.
- Perform a **rough titration** and record your burette readings in the space below.

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

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- 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 **FA 1** added in each accurate titration.

Step 3

- Stir the mixture from **Step 1** again to ensure that all the solid has dissolved.
- Transfer the solution to the 250 cm^3 graduated (volumetric) flask labelled **FA 5**.
- Rinse the beaker twice with a little distilled water and add the washings to the graduated flask.
- Make the solution up to 250 cm^3 with distilled water. Ensure that the contents of the flask are thoroughly mixed.
- Transfer 25.0 cm^3 of this solution, **FA 5**, into a second conical flask using a second pipette.
- 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 **FA 1** added in each accurate titration.

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Calculations

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

The equation for the reaction between sodium hydroxide and hydrochloric acid is shown below.

$$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$$

(b) (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 **FA 3** required …….. cm^3 of **FA 1**.

(ii) Use your answer from **(i)** to calculate the number of moles of sodium hydroxide, **FA 1**, required to react with 25.0 cm^3 of **FA 3** in **Step 2**.

moles of NaOH = ... mol

(iii) Use your answer from **(ii)** to determine the number of moles of hydrochloric acid in 25.0 cm^3 of **FA 3**.

moles of HCl in 25.0 cm^3 of **FA 3** = ... mol

(iv) **FA 3** was produced by diluting **FA 2**. Calculate the number of moles of hydrochloric acid in 50.00 cm^3 of **FA 2**.

moles of HCl in 50.00 cm^3 of **FA 2** = ... mol
[2]

(c) (i) From your accurate titration results in **Step 3**, obtain a suitable value to be used in your calculations. Show clearly how you have obtained this value.

25.0 cm^3 of **FA 5** required …….. cm^3 of **FA 1**.

(ii) Use your answer from **(i)** to calculate the number of moles of sodium hydroxide, **FA 1**, required to react with 25.0 cm^3 of **FA 5** in **Step 3**.

moles of NaOH = ... mol

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(iii) Use your answer from **(ii)** to determine the number of moles of hydrochloric acid in 25.0 cm^3 of **FA 5**.

moles of HCl in 25.0 cm^3 of **FA 5** = ... mol

(iv) Use your answer from **(iii)** to determine the number of moles of hydrochloric acid in 250 cm^3 of **FA 5**.

moles of HCl in 250 cm^3 of **FA 5** = ... mol
[4]

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(d) (i) Write an equation for the reaction between calcium carbonate and hydrochloric acid.

.....

(ii) Calculate the number of moles of hydrochloric acid that reacted with calcium carbonate in **FA 4** using the following expression.

moles of HCl = **(b)(iv)** – **(c)(iv)**

= ..... mol

(iii) Use your answers from **(i)** and **(ii)** to calculate the mass of $CaCO_3$ in **FA 4**.
[A_r: C, 12.0; O, 16.0; Ca, 40.1]
(If you were unable to answer **(d)(ii)**, you may assume that the number of moles of hydrochloric acid that reacted with calcium carbonate was 0.0351 mol.)

mass of $CaCO_3$ in **FA 4** = ... g

(iv) Calculate the percentage purity by mass of the calcium carbonate in **FA 4**.

The percentage purity by mass of calcium carbonate is ..... %
[4]

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

maximum error in a burette reading = ... cm^3

(ii) Calculate the maximum percentage error for one of your accurate titres in **Step 3**. Show your working.

maximum percentage error = ... %
[2]

(f) A student decided to use a larger mass of **FA 4**. State and explain whether this alteration will improve the accuracy of the percentage purity obtained.

.....

..... [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.

Half fill a 250 cm^3 beaker with water. Heat to approximately 80 °C, then stop heating. You will need this as a hot water bath in **(a)(viii)**.

(a) Compounds **FA 6**, **FA 7** and **FA 8** are salts containing the same transition metal but in three different oxidation states. You are provided with solutions of **FA 6** and **FA 7** and a solid sample of **FA 8**. Carry out the experiments described below and record your observations in the table.

test	*observations*
(i) To 1 cm depth of **FA 6** in a test-tube add about 5 cm depth of dilute sulfuric acid and mix the two solutions. Use this mixture for tests **(ii)** and **(iii)**.	**no observation required**
(ii) To 1 cm depth of hydrogen peroxide in a test-tube add 10 drops of acidified **FA 6** from **(i)**.	
(iii) To 1 cm depth of potassium iodide in a test-tube add 1 cm depth of acidified **FA 6** from **(i)**.	

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test	*observations*
(iv) To 1 cm depth of **FA 7** in a test-tube add 1 cm depth of aqueous sodium hydroxide and allow to stand for a few minutes.	
(v) To 1 cm depth of **FA 7** in a test-tube add aqueous ammonia until in excess.	
(vi) To 1 cm depth of **FA 7** in a test-tube add 1 cm depth of **FA 6** and allow to stand for a few minutes.	
(vii) To 1 cm depth of hydrogen peroxide in a test-tube add a small spatula measure of **FA 8**.	
(viii) Place a small spatula measure of **FA 8** in a test-tube and add about 10 drops of concentrated hydrochloric acid **with care**. Place the tube in the hot water bath. Test any gas produced with damp litmus paper. When you have made your observations, fill the test-tube with cold water to stop any further reaction.	

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[6]

(b) (i) From your observations in **(a)** suggest the identity of the transition metal contained in **FA 6**, **FA 7** and **FA 8**.

The transition metal present in the three compounds is .....

Explain how your observations support your conclusion.

.....

.....

.....

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

.....

(iii) Give the oxidation state of the transition metal in **FA 7**.

.....

The oxidation state of the transition metal in **FA 6** is +7.

Suggest an oxidation state of the transition metal in the product formed in **(a)(vi)**.

.....

[3]

(c) Aqueous solutions **FA 9** and **FA 10** both contain halide ions.

(i) Use the Qualitative Analysis Notes on page 12 to select two reagents which, **used together**, identify the halide ions in **FA 9** and **FA 10**.

The first reagent is .....

and this is followed by .....

(ii) Use your chosen reagents to carry out tests on **FA 9** and **FA 10**.
Record your results in an appropriate form in the space below.

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(iii) From the results of the tests in **(ii)**, state which halide each solution contains.

FA 9 contains .....

FA 10 contains .....

(iv) Halides can also be identified by reaction with concentrated sulfuric acid. The acid can act as an oxidising agent.
State what you would expect to **see** if concentrated sulfuric acid was added to a solid sample of **FA 9** and **FA 10**.
Do not carry out these experiments.

expected observation with **FA 9** .....

.....

expected observation with **FA 10** .....

.....

(v) Solutions containing the copper(II) ion react with concentrated hydrochloric acid.
To a 1 cm depth of aqueous copper(II) sulfate in a test-tube, add an equal volume of concentrated hydrochloric acid **with care**.

observation .....

.....
[6]

[Total: 15]

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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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