CANDIDATE NAME		

CENTRE NUMBER		CANDIDATE NUMBER	

CHEMISTRY 9701/34

Paper 3 Advanced Practical Skills 2 **May/June 2014**

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 an HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.
DO **NOT** WRITE IN ANY BARCODES.

Answer **all** questions.
Electronic calculators may be used.
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 You are to determine the percentage of calcium carbonate in a sample of crushed limestone. You will first react a known mass of the crushed limestone in a known amount of hydrochloric acid, $HCl(aq)$, to make a solution. You may assume that only the calcium carbonate present in the sample will react with the acid.

$$CaCO_3(s) + 2HCl(aq) \rightarrow CaCl_2(aq) + H_2O(l) + CO_2(g)$$

The amount of acid that did not react with the carbonate is then found by a titration using sodium hydroxide. You may assume that no compounds present in the limestone will react with the sodium hydroxide.

$$NaOH(aq) + HCl(aq) \rightarrow NaCl(aq) + H_2O(l)$$
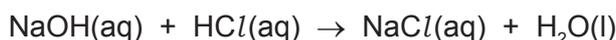
FB 1 is crushed limestone, impure calcium carbonate.
FB 2 is $2.0\,mol\,dm^{-3}$ hydrochloric acid, HCl.
FB 4 is $0.20\,mol\,dm^{-3}$ sodium hydroxide, $NaOH$.
methyl orange indicator

(a) Method

Read through the method before starting any practical work.

Making the solution

- Weigh the container with the limestone, **FB 1**, and record the mass below.
- Tip all the solid **FB 1** into a $250\,cm^3$ beaker.
- Reweigh the container and record the mass.
- Fill the burette with **FB 2**.
- Slowly run between 47.5 and $48.5\,cm^3$ of **FB 2** into the beaker containing **FB 1**.
- Record, in the space below, both your burette readings and the volume of **FB 2** added.
- Stir the mixture carefully until all the solid has reacted.
- Transfer the contents of the beaker into the volumetric flask.
- Rinse the beaker with distilled water and add it to the volumetric flask. Make the solution up to $250\,cm^3$ with distilled water and mix thoroughly. This is solution **FB 3**.

Titration

- Empty and rinse the burette with distilled water.
- Fill the burette with **FB 3** from the volumetric flask.
- Pipette 25.0 cm^3 of **FB 4** into a conical flask.
- Add a few drops of methyl orange indicator.
- 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 3** added in each accurate titration.

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IX	

[9]

(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 **FB 4** required .... cm^3 of **FB 3**. [1]

(vii) From your answer to **(vi)** and the mass of **FB 1** used in **(a)**, calculate the percentage by mass of calcium carbonate in the limestone.
[A_r: C, 12.0; O, 16.0; Ca, 40.1]

percentage of calcium carbonate = ... %

[7]

(d) (i) The maximum error in a single burette reading is $\pm 0.05\,cm^3$.
Student X, carrying out this experiment, recorded that $48.50\,cm^3$ of **FB 2** was added to **FB 1**.
What are the smallest and largest possible volumes of **FB 2** that were added?

smallest volume used = ... cm^3

largest volume used = ... cm^3

(ii) Student Y used an identical mass of **FB 1** but added $47.70\,cm^3$ of **FB 2**. How would the value obtained in **(b)** by student X compare with the value obtained by student Y?
Explain your answer.

....

....

....

[3]

[Total: 20]

2 You are to determine the percentage by mass of calcium carbonate in another sample of the limestone by thermal decomposition. You may assume that none of the other compounds in the limestone sample is affected by heating. The equation for the reaction that occurs is given below.

$$CaCO_3(s) \rightarrow CaO(s) + CO_2(g)$$

FB 5 is crushed limestone, impure calcium carbonate.

(a) Method

Read through the method **before** starting any practical work and prepare a table for your results in the space below.

- Weigh the empty crucible and record the mass in your table.
- Transfer **all** the **FB 5** into the crucible.
- Weigh the crucible with **FB 5** and record the mass.
- Place the crucible on the pipe-clay triangle.
- Heat the crucible gently for about one minute and then strongly for three minutes.
- Remove the Bunsen burner and allow the crucible to cool.
- While the crucible is cooling start working on another question.
- Reweigh the cooled crucible with contents and record the mass.
- Reheat the crucible strongly for three minutes, cool and reweigh. Record the mass.
- Record the mass of **FB 5** used and the mass of solid remaining after the second heating.

[2]

(b) Calculations

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

(i) From your results in **(a)**, calculate the total mass of carbon dioxide lost on heating **FB 5**.

mass of CO_2 lost = ... g

(ii) Use your answer to **(i)** to calculate the mass of calcium carbonate present in the sample of **FB 5** heated.
[A_r: C, 12.0; O, 16.0; Ca, 40.1]

mass of $CaCO_3$ = ... g

(iii) Calculate the percentage by mass of calcium carbonate in the limestone.

percentage of calcium carbonate = ... %

[3]

(c) **FB 5** and **FB 1** are samples of the same limestone. You have determined the percentage of calcium carbonate in both **Questions 1** and **2** using two different procedures.

(i) Which procedure is the less accurate? Explain your answer.

....

....

(ii) Suggest a change to the less accurate practical procedure that would improve the accuracy and explain your answer.

....

....

....

[2]

[Total: 7]

3 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 name or correct formula of the element or compound must be given.**

(a) A different sample of limestone was reacted with dilute nitric acid to give solution **FB 6**.
This sample of limestone contained calcium carbonate, $CaCO_3$, and one other salt.
This additional salt contains a single cation and a single anion from those listed on pages 11 and 12. By carrying out the following tests you will be able to suggest identities of the additional ions.

test	observations
(i) To a 1 cm depth of **FB 6** in a test-tube add aqueous ammonia.	
(ii) To a 1 cm depth of **FB 6** in a test-tube add a 1 cm depth of aqueous silver nitrate.	
(iii) To a 1 cm depth of **FB 6** in a test-tube add a 1 cm depth of aqueous barium chloride or barium nitrate.	

(iv) Suggest all possible identities for the ions present in **FB 6**, apart from Ca^{2+} and NO_3^-.

....

(v) Select a reagent to use in a further test on **FB 6** to confirm that one of the cations you have listed in **(iv)** is **not** present in **FB 6**. Carry out your test and complete the table.

test	observations	conclusion
To a 1 cm depth of **FB 6** in a test-tube add		

I	
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VI	

[6]

(b) You are provided with a solid, **FB 7**. By carrying out the following tests you will be able to identify three of the ions present in **FB 7**.

test	observations
(i) Place a spatula measure of **FB 7** in a hard-glass test-tube. Heat gently at first, then	
heat more strongly until no further change is seen, then	
allow the tube to cool.	

	test	observations
(ii)	Place a spatula measure of **FB 7** in a test-tube. Add about a 5 cm depth of dilute nitric acid. You will use the solution formed for tests **(iii)** to **(v)**.	
(iii)	To a 1 cm depth of the solution in a test-tube add a few drops of aqueous potassium manganate(VII), then add a few drops of starch solution.	
(iv)	To a 1 cm depth of the solution in a test-tube add a few drops of aqueous silver nitrate.	
(v)	To a 1 cm depth of the solution in a test-tube add aqueous ammonia.	

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Use the Qualitative Analysis Notes on pages 11 and 12 to identify **three** of the ions present.

....

[7]

[Total: 13]

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
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

2   Reactions of anions

ion	reaction
carbonate, CO_3^{2-}	CO_2 liberated by dilute acids
chloride, Cl^-(aq)	gives white ppt. with Ag^+(aq) (soluble in NH_3(aq));
bromide, Br^-(aq)	gives cream ppt. with Ag^+(aq) (partially soluble in NH_3(aq));
iodide, I^-(aq)	gives yellow ppt. with Ag^+(aq) (insoluble in NH_3(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) (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 manganate(VII) from purple to colourless