



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

CANDIDATE  
NAME

CENTRE  
NUMBER

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CANDIDATE  
NUMBER

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**CHEMISTRY**

**9701/33**

Advanced Practical Skills 1

**May/June 2013**

**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.  
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 12 and 13.  
A Periodic Table is printed on page 16.

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 **14** printed pages and **2** blank pages.

- 1 You are to determine the enthalpy change of the reaction between hydrochloric acid and sodium hydroxide by adding various volumes of acid and alkali and measuring the change in temperature.

**FA 1** is  $0.950 \text{ mol dm}^{-3}$  hydrochloric acid,  $\text{HCl}$ .

**FA 2** is aqueous sodium hydroxide,  $\text{NaOH}$ .

**(a) Method**

- Support the plastic cup in a  $250 \text{ cm}^3$  beaker.
- Using a measuring cylinder, transfer  $25 \text{ cm}^3$  of **FA 1** into the cup and measure the temperature of the acid. Tilt the cup if necessary to cover the bulb of the thermometer.
- Record this initial temperature.

initial temperature of **FA 1** = .....  $^{\circ}\text{C}$

- Use a second measuring cylinder to transfer  $10 \text{ cm}^3$  of **FA 2** and  $25 \text{ cm}^3$  of water into a  $100 \text{ cm}^3$  beaker.
- Add this mixture to the plastic cup and stir.
- Measure the maximum temperature reached and record this maximum temperature in the table below.
- Rinse out the plastic cup and shake it to remove excess water.
- Repeat the experiment, using the volumes of **FA 1**, **FA 2** and water shown in the table. Record the maximum temperature for each experiment.

volume <b>FA 1</b> / $\text{cm}^3$	volume <b>FA 2</b> / $\text{cm}^3$	volume water / $\text{cm}^3$	maximum temperature / $^{\circ}\text{C}$
25	10	25	
25	15	20	
25	20	15	
25	25	10	
25	30	5	
25	35	0	

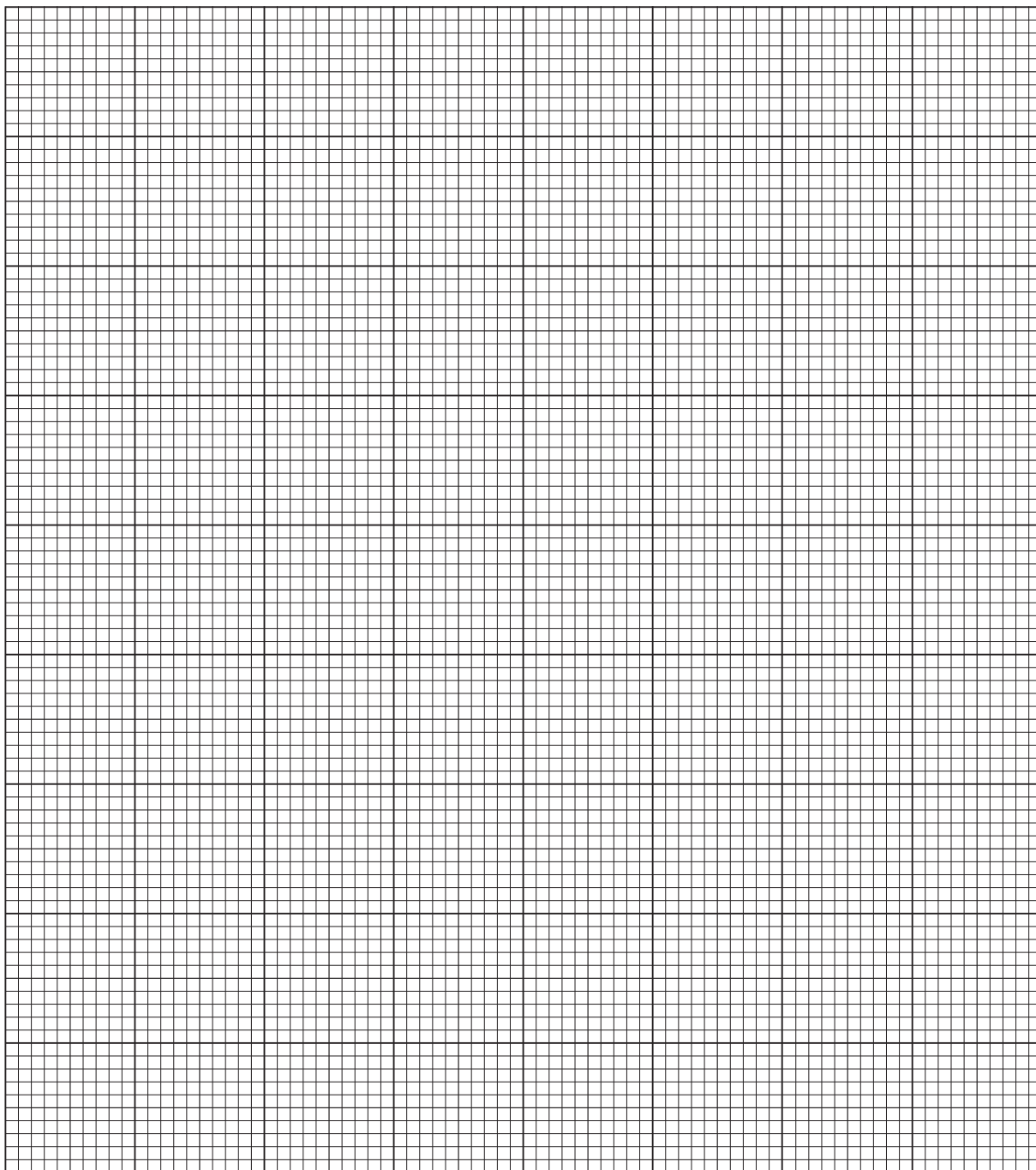
You are going to plot a graph using these results to find the volume of **FA 2** that gives the greatest maximum temperature.

**Before** you plot the graph, choose two further volumes of **FA 2** that will allow you to find more precisely the volume that gives the greatest maximum temperature.

Record the volumes you choose, carry out the experiments and record the corresponding maximum temperatures, in the table. [2]

- (b) (i) On the grid below, plot the maximum temperature on the y-axis against the volume of **FA 2** on the x-axis.

For  
Examiner's  
Use



- (ii) Draw two straight lines of best fit on your graph, one to show where the temperature was increasing and the other after the greatest maximum temperature had been reached.
- (iii) Using your graph and the initial temperature recorded in (a), determine the maximum temperature **change** that could occur when 25 cm<sup>3</sup> of **FA 1** react with **FA 2**.

maximum temperature **change** = ..... °C  
[5]

**(c) Calculation**

- (i)** Calculate the energy needed to produce the temperature change in **(b)(iii)**.  
(Assume that 4.3J of heat energy changes the temperature of 1.0 cm<sup>3</sup> of solution by 1.0 °C.)

energy needed = ..... J

- (ii)** Calculate the number of moles of HCl used in each experiment.

moles of HCl = ..... mol

- (iii)** Calculate the enthalpy change, in kJ mol<sup>-1</sup>, when 1 mole of HCl reacts with NaOH.

enthalpy change = ..... kJ mol<sup>-1</sup>  
(sign) (value) [3]

[Total: 10]

- 2 The identity of a metal, M, can be found by titrating a solution of its carbonate with hydrochloric acid of known concentration.

For  
Examiner's  
Use

**FA 3** is a solution of the metal carbonate,  $M_2CO_3$ , of concentration  $6.90 \text{ g dm}^{-3}$ .

You are to dilute the hydrochloric acid that you used in **Question 1** and then titrate the carbonate solution with this acid.

**(a) Method**

**Dilution of the acid**

- Pipette  $25.0 \text{ cm}^3$  of **FA 1** into the  $250 \text{ cm}^3$  volumetric (graduated) flask labelled **FA 4**.
- Add distilled water to make the total volume  $250 \text{ cm}^3$ .
- Stopper the flask and mix the contents thoroughly.

**Titration**

- Fill the burette with diluted hydrochloric acid, **FA 4**.
- Use a clean pipette to transfer  $25.0 \text{ cm}^3$  of **FA 3** into a conical flask.
- Titrate **FA 3** with **FA 4** using the indicator provided.
- Perform a rough titration and record your burette readings in the space below.

The rough titre is .....  $\text{cm}^3$ .

- 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 4** added in each accurate titration.

I	
II	
III	
IV	
V	
VI	
VII	

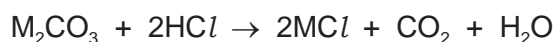
[7]

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

25.0 cm<sup>3</sup> of **FA 3** required ..... cm<sup>3</sup> of **FA 4**  
[1]

**(c) Calculation**

The equation for the reaction between hydrochloric acid and the metal carbonate is given below.



- (i) Calculate the number of moles of hydrochloric acid present in the volume in (b).

moles of HCl = ..... mol

- (ii) Hence, calculate the number of moles of M<sub>2</sub>CO<sub>3</sub> present in 25.0 cm<sup>3</sup> of **FA 3**.

moles of M<sub>2</sub>CO<sub>3</sub> = ..... mol

- (iii) Calculate the concentration of M<sub>2</sub>CO<sub>3</sub> in **FA 3** in mol dm<sup>-3</sup>.

concentration of M<sub>2</sub>CO<sub>3</sub> = ..... mol dm<sup>-3</sup>

- (iv) Use your answer to (iii), and the fact that **FA 3** contains 6.90 g dm<sup>-3</sup>, to determine the relative atomic mass, *A<sub>r</sub>*, of M.

*A<sub>r</sub>* of M = .....

- (v) Use your answer to (iv) and the Periodic Table on page 16 to suggest the identity of M.

M is ..... [5]

(d) The concentration of a carbonate solution could be found using either the method in **Question 1** or that in **Question 2**.

(i) Suggest, and explain, which of the methods is more accurate.

.....  
.....

(ii) For the method that you think is less accurate, suggest an improvement to the practical procedure that could be made to improve the accuracy.

.....  
..... [2]

[Total: 15]

### 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) You are provided with a solid, **FA 5**. **FA 5** is a mixture that contains two anions and two cations.

To all your sample of **FA 5** in a boiling tube add 3 cm depth of distilled water. Shake the tube and filter the contents. Keep the solid residue for tests in (b) and the filtered solution for tests in (c).

- (b) (i) Open up the filter paper and scrape the residue into a boiling tube. Add dilute nitric acid,  $\text{HNO}_3$ , using a dropping pipette until the solid **just** disappears. Record your observations and keep the solution for tests in (ii).

observations .....

.....

- (ii) Divide the solution from test (i) equally into **three** test-tubes.

To the first test-tube add aqueous sodium hydroxide,  $\text{NaOH}$ , until in excess. Record your observations.

observations .....

.....

Which cations, from those listed in the Qualitative Analysis Notes on page 12, would give these observations?

.....



- (iii) You are to devise tests that will positively identify which one of the cations you have suggested in (ii) is present. For each of the possible ions you should indicate the test and the expected result for each test in a suitable table in the space below.

Use the solutions in the second and third test-tubes to carry out these tests and record your observations in the space below.

Identify the cation present.

The cation present is .....

[7]

- (c) To 1 cm depth of filtered solution from (a) in a test-tube add 1 cm depth of dilute nitric acid followed by a few drops of aqueous silver nitrate. Record your observation.

observation .....

Which further reagent could be added to this test-tube to help you to confirm the nature of the anion present?

reagent .....

Carry out a test using this additional reagent. Record your observation and conclusion about the anion present.

observation .....

The anion present is ..... [2]

- (d) Using your observation in (b)(i) state which other anion is present in FA 5.

The anion present is ..... [1]

- (e) Solutions **FA 6** and **FA 7** each contain one of the ions sulfite,  $\text{SO}_3^{2-}$ , sulfate,  $\text{SO}_4^{2-}$ , nitrite,  $\text{NO}_2^-$ , or nitrate,  $\text{NO}_3^-$ .

- (i) Carry out the tests in the table below to identify which ion is present in each solution.

test	observations	
	FA 6	FA 7
To 1 cm depth of solution in a <b>boiling</b> tube, add a small piece of aluminium foil and 1 cm depth of aqueous sodium hydroxide. Warm the mixture <b>with care</b> .		
To 1 cm depth of solution in a test-tube, add a few drops of aqueous barium chloride or barium nitrate, then		
add dilute hydrochloric acid.		
To 1 cm depth of solution in a test-tube, add 1 cm depth of dilute hydrochloric acid.		

- (ii) From your observations, identify the anion present in each solution.

**FA 6** contains .....

**FA 7** contains .....

- (iii) What type of reaction takes place when a positive observation is seen with aluminium foil and aqueous sodium hydroxide in (i)?

.....  
[5]

[Total: 15]

## Qualitative Analysis Notes

Key: [ppt. = precipitate]

## 1 Reactions of aqueous cations

ion	reaction with	
	NaOH(aq)	NH <sub>3</sub> (aq)
aluminium, Al <sup>3+</sup> (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, NH <sub>4</sub> <sup>+</sup> (aq)	no ppt. ammonia produced on heating	–
barium, Ba <sup>2+</sup> (aq)	no ppt. (if reagents are pure)	no ppt.
calcium, Ca <sup>2+</sup> (aq)	white ppt. with high [Ca <sup>2+</sup> (aq)]	no ppt.
chromium(III), Cr <sup>3+</sup> (aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess
copper(II), Cu <sup>2+</sup> (aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution
iron(II), Fe <sup>2+</sup> (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 <sup>3+</sup> (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess
lead(II), Pb <sup>2+</sup> (aq)	white ppt. soluble in excess	white ppt. insoluble in excess
magnesium, Mg <sup>2+</sup> (aq)	white ppt. insoluble in excess	white ppt. insoluble in excess
manganese(II), Mn <sup>2+</sup> (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 <sup>2+</sup> (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

<i>ion</i>	<i>reaction</i>
carbonate, $\text{CO}_3^{2-}$	$\text{CO}_2$ liberated by dilute acids
chromate(VI), $\text{CrO}_4^{2-}(\text{aq})$	yellow solution turns orange with $\text{H}^+(\text{aq})$ ; gives yellow ppt. with $\text{Ba}^{2+}(\text{aq})$ ; gives bright yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
chloride, $\text{Cl}^-(\text{aq})$	gives white ppt. with $\text{Ag}^+(\text{aq})$ (soluble in $\text{NH}_3(\text{aq})$ ); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
bromide, $\text{Br}^-(\text{aq})$	gives cream ppt. with $\text{Ag}^+(\text{aq})$ (partially soluble in $\text{NH}_3(\text{aq})$ ); gives white ppt. with $\text{Pb}^{2+}(\text{aq})$
iodide, $\text{I}^-(\text{aq})$	gives yellow ppt. with $\text{Ag}^+(\text{aq})$ (insoluble in $\text{NH}_3(\text{aq})$ ); gives yellow ppt. with $\text{Pb}^{2+}(\text{aq})$
nitrate, $\text{NO}_3^-(\text{aq})$	$\text{NH}_3$ liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil
nitrite, $\text{NO}_2^-(\text{aq})$	$\text{NH}_3$ liberated on heating with $\text{OH}^-(\text{aq})$ and Al foil; $\text{NO}$ liberated by dilute acids (colourless $\text{NO} \rightarrow$ (pale) brown $\text{NO}_2$ in air)
sulfate, $\text{SO}_4^{2-}(\text{aq})$	gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ or with $\text{Pb}^{2+}(\text{aq})$ (insoluble in excess dilute strong acids)
sulfite, $\text{SO}_3^{2-}(\text{aq})$	$\text{SO}_2$ liberated with dilute acids; gives white ppt. with $\text{Ba}^{2+}(\text{aq})$ (soluble in excess dilute strong acids)

## 3 Tests for gases

<i>gas</i>	<i>test and test result</i>
ammonia, $\text{NH}_3$	turns damp red litmus paper blue
carbon dioxide, $\text{CO}_2$	gives a white ppt. with limewater (ppt. dissolves with excess $\text{CO}_2$ )
chlorine, $\text{Cl}_2$	bleaches damp litmus paper
hydrogen, $\text{H}_2$	“pops” with a lighted splint
oxygen, $\text{O}_2$	relights a glowing splint
sulfur dioxide, $\text{SO}_2$	turns acidified aqueous potassium dichromate(VI) from orange to green

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## The Periodic Table of the Elements

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