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

CANDIDATE NAME	

CENTRE NUMBER							CANDIDATE NUMBER					

CHEMISTRY **9701/31**

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.

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 and **4** blank pages.

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For Examiner's Use

1 The reaction between sulfuric acid and sodium hydroxide is exothermic.

$$H_2SO_4(aq) + 2NaOH(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$$

By measuring the temperature changes that occur when different volumes of the acid are added to a fixed volume of the alkali, it is possible to determine the neutralisation point. This is the point at which just enough acid has been added to react with all the alkali present. The aim of the investigation is to determine the concentration of the sulfuric acid.

FA 1 is 2.00 mol dm^{-3} sodium hydroxide, NaOH.
FA 2 is dilute sulfuric acid, H_2SO_4.

Read through the instructions carefully and prepare a table for your results before starting any practical work.

(a) Method

- Support a plastic cup in a 250 cm^3 beaker.
- Use a pipette to transfer 25.0 cm^3 of **FA 1** into the plastic cup.
- Record the temperature of **FA 1**, T_1, in the space below.

T_1 = ………… °C

- Fill the burette labelled **FA 2** with **FA 2**.
- Add 5.00 cm^3 of **FA 2** from the burette to the plastic cup.
- Stir the mixture thoroughly and record the temperature of the solution.
- Add a further 5.00 cm^3 of **FA 2** to the plastic cup and again record the temperature.
- Repeat the addition of 5.00 cm^3 portions of **FA 2** until you have added a total of 50.00 cm^3 of **FA 2** to the plastic cup. Measure the temperature after each addition.
- Record in your table below the total volume of **FA 2** added and the temperature of the solution after each addition.

I	
II	
III	
IV	
V	

[5]

For Examiner's Use

(b) After each addition of acid, the temperature rise, ΔT, is given by,

ΔT = temperature recorded – T_1.

The total volume of solution in the plastic cup, V_T is given by,

V_T = volume of **FA 2** + volume of **FA 1**.

The heat given out by the reaction is proportional to the temperature rise, ΔT, multiplied by the total volume of solution in the plastic cup, V_T.

Use your experimental results to complete the following table.
You should include:

- the volume of **FA 2**
- the total volume in the plastic cup, V_T
- the temperature of the solution
- the temperature rise, ΔT
- the total volume × the temperature rise, ($V_T \times \Delta T$)

[1]

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(c) (i) On the grid below, plot the values of ($V_T \times \Delta T$) on the y-axis against the volume of **FA 2** on the x-axis.

I	
II	
III	
IV	

For Examiner's Use

(ii) Draw a straight line of best fit through the points where the values of ($V_T \times \Delta T$) are increasing. Draw a second straight line of best fit through the points where the values of ($V_T \times \Delta T$) are decreasing.

(iii) From your graph, determine the volume of **FA 2** where the two lines of best fit intersect.

volume of **FA 2** = ……………………………………….. cm^3
[5]

(d) The value you recorded in **(c)(iii)** is the volume of **FA 2** which is needed to neutralise 25.0 cm^3 of **FA 1**. In the following calculations you will determine the concentration of **FA 2**.

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

(i) Calculate how many moles of sodium hydroxide are contained in 25.0 cm^3 of **FA 1**.

moles of NaOH = ………………… mol

(ii) Calculate how many moles of sulfuric acid would react with the number of moles of NaOH in **(i)**.

moles of H_2SO_4 = ………………… mol

(iii) Calculate the concentration of **FA 2**.

concentration of **FA 2** = …………………….. mol dm^{-3}
[3]

(e) Other than heat losses from the plastic cup to the surroundings, suggest an additional source of error in this experiment and how this error could be reduced.

....

....

.... [1]

[Total: 15]

For Examiner's Use

2 A second way to determine the concentration of an acid is by volumetric titration. In this experiment you will first dilute the sample of **FA 2** that you used in **Question 1** and then titrate this diluted solution using aqueous sodium hydroxide.

$$H_2SO_4(aq) + 2NaOH(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$$

FA 2 is dilute sulfuric acid, H_2SO_4.
FA 3 is 0.150 mol dm^{-3} sodium hydroxide, NaOH.
distilled water

(a) Method

Dilution of FA 2

- Use the burette labelled **FA 2** to transfer 25.00 cm^3 of **FA 2** into the 250 cm^3 graduated (volumetric) flask, labelled **FA 4**.
- Make up the contents of the flask to the 250 cm^3 mark with distilled water.
- Stopper the flask and mix the contents thoroughly. This is solution **FA 4**.

Titration

- Fill the burette labelled **FA 3** with **FA 3**.
- Use a clean pipette to transfer 25.0 cm^3 of **FA 4** into a conical flask.
- Add to the flask a few drops of the acid-base indicator provided.
- Titrate the acid in the flask with the alkali, **FA 3**.

You should perform a rough titration.
In the space below record your burette readings for this rough titration.

The rough titre is …………………… cm^3.

- Carry out as many accurate titrations as you think necessary to obtain consistent results.
- Record, in a suitable form below, all of your burette readings and the volume of **FA 3** added in each accurate titration. Make certain that any recorded results show the precision of your practical work.

I	
II	
III	
IV	
V	

[5]

For Examiner's Use

(b) From your titration results obtain a suitable value to be used in your calculation. Show clearly how you have obtained this value.

25.0 cm^3 of **FA 4** required ………….. cm^3 of **FA 3**.
[1]

(c) **(i)** Calculate how many moles of NaOH are contained in the volume recorded in **(b)**.

moles of NaOH = ………………… mol

(ii) Hence, calculate how many moles of H_2SO_4 are contained in 25.0 cm^3 of **FA 4**.

moles of H_2SO_4 = ………………… mol

(iii) Calculate the concentration of the sulfuric acid, **FA 2**.

I	
II	
III	

concentration of **FA 2** = …………………….. mol dm^{-3}
[3]

(d) You have used two methods to determine the concentration of the sulfuric acid in **FA 2**. Use your answers to **1(d)(iii)** and **2(c)(iii)** to calculate the difference in these values as a percentage of the value found by the volumetric titration method.

percentage difference = …………………….. %
[1]

[Total: 10]

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) **FA 5**, **FA 6**, **FA 7** and **FA 8** are aqueous solutions each of which contains a single cation and a single anion. Some of the ions present are listed below.

Pb^{2+} Cl^{-} CO_3^{2-} CrO_4^{2-}

By observing the reactions that occur when pairs of the solutions are mixed together, you will be able to identify which solution contains which of these ions.

Use a 1 cm depth of each solution in a test-tube and record your observations in the following table.

	FA 6	FA 7	FA 8
FA 5			
FA 6			
FA 7			

[8]

For Examiner's Use

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

(b) From your observations deduce which solution contains each of the following ions.

ion	Pb^{2+}	Cl^{-}	CO_3^{2-}	CrO_4^{2-}
solution				

[2]

I	
II	

(c) Identify another ion that is present in one of the solutions. Explain your reasoning.

ion ....

explanation ....

.... [1]

For Examiner's Use

(d) **(i)** If chloride ions, Cl^-, were to be replaced with bromide ions, Br^-, in one of the solutions, would it make any difference to the observations you made in **(a)**? Explain your answer.

....

....

....

(ii) **FA 9** is an aqueous solution containing either chloride ions or bromide ions. Select a pair of reagents to identify which anion is present.

reagents ....

Carry out this test and record your observations and conclusion.

observations ....

....

The anion in **FA 9** is ....

[4]

[Total: 15]

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.]

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 $\rightarrow$ (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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