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

CANDIDATE NAME	

CENTRE NUMBER						CANDIDATE NUMBER					

2059021505

CHEMISTRY **9701/35**

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 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

For Examiner's Use	
1	
2	
3	
Total	

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

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For Examiner's Use

You are advised to begin work on question 2, and return to question 1 later.

1 In this experiment you are to investigate the reaction between phosphoric acid and sodium hydroxide in order to determine the chemical equation.

FA 1 is an aqueous solution of phosphoric acid, H_3PO_4.
FA 2 is 0.115 mol dm^{-3} sodium hydroxide, NaOH.
phenolphthalein indicator

(a) Method

Dilution

- Weigh the 100 cm^3 beaker provided. Record the mass in the space below.
- Use the measuring cylinder to add about 10 cm^3 of **FA 1** to the beaker. Weigh the beaker with **FA 1** and record the mass.
- Calculate the mass of **FA 1** used and record this in the space below.
- Pour the **FA 1** from the beaker into the 250 cm^3 graduated (volumetric) flask provided, labelled **FA 3**.
  Wash the beaker twice with small quantities of water and add these washings to the volumetric flask.
- Make the solution up to the mark using distilled water. This diluted solution of phosphoric acid is **FA 3**.
- Ensure that the contents of the flask are thoroughly mixed before using **FA 3** for your titrations.

Titration

- Fill the burette with **FA 2**.
- Pipette 25.0 cm^3 of **FA 3** into a conical flask.
- Add 5 drops of phenolphthalein indicator to the flask. The indicator should remain colourless.
- Titrate **FA 3** with **FA 2** until the indicator changes to a permanent pale pink colour.
- 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 sure 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 2** 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 have obtained this value.

25.0 cm^3 of **FA 3** required .... cm^3 of **FA 2**.
[1]

(c) Calculations

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 were present in the volume of **FA 2** calculated in **(b)**.

moles of NaOH = ...mol

(ii) The phosphoric acid, **FA 1**, that you weighed out contained 8.40 % by mass of H_3PO_4. Calculate the mass of H_3PO_4 that you weighed out.

mass of H_3PO_4 = ... g

(iii) Calculate how many moles of H_3PO_4 were present in 25.0 cm^3 of the diluted solution, **FA 3**.
(A_r : H, 1.0; O, 16.0; P, 31.0)

moles of H_3PO_4 = ... mol

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(iv) Use your answers to **(i)** and **(iii)** to calculate how many moles of NaOH react with 1 mole of H_3PO_4.
Give your answer to the nearest whole number.

moles of NaOH = ... mol

(v) When NaOH reacts with H_3PO_4, the salt formed could be NaH_2PO_4, Na_2HPO_4 or Na_3PO_4.
Use your answer to **(iv)** to deduce which one of these three salts was the major product formed during the titration.
Write the equation for the reaction of NaOH with H_3PO_4 to produce this salt.

[5]

(d) **(i)** A 25 cm^3 pipette is accurate to $\pm 0.06\ cm^3$.
Calculate the maximum percentage error when the pipette was used to measure solution **FA 3**.

percentage error in measuring **FA 3** = ... %

(ii) State the maximum error in the mass of the 100 cm^3 beaker used in the dilution of **FA 1**.

maximum error = ... g

(iii) Calculate the maximum percentage error in the mass of **FA 1** used.

maximum percentage error = ... %
[2]

[Total: 15]

For Examiner's Use

2 In this experiment you are to heat a hydrated salt, **FA 4**, to remove the water of crystallisation. You will then calculate the relative formula mass of the anhydrous salt.
The formula of **FA 4** is $MX_2.2H_2O$, where **M** is a metal and **X** is a halogen.

(a) Method

You will carry out the following experiment twice.
Record all weighings for the first and second experiments in an appropriate form in the space below.

- Record the mass of an empty crucible without its lid.
- Add 2.6 – 3.4 g of **FA 4** into the weighed crucible. Record the mass of the crucible and its contents.
- Use a pipe-clay triangle to support the crucible and contents on a tripod.
- Heat the crucible and its contents gently for about one minute with the lid off. Then heat strongly for a further **four** minutes.
- Put the lid on the crucible and leave to cool for several minutes.

While you are waiting for the crucible to cool, start work on Question 1.

- When the crucible is cool, **remove the lid** and weigh the crucible with the anhydrous residue, MX_2.
- Repeat the experiment using the second crucible. This time add 1.6 – 2.4 g of **FA 4**.

I	
II	
III	
IV	
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VI	

[6]

(b) Calculation

Show your working and express your answers to **each** step to **three** significant figures.

(i) Calculate the number of moles of water removed from the hydrated salt in the **first** experiment.
(A_r : H, 1.0; O, 16.0)

moles of H_2O = ... mol

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(ii) Complete the equation for the removal of water from hydrated **FA 4**.
Include state symbols.

$MX_2.2H_2O$(.....) $\rightarrow$ ....(.....) + ...(.....)

(iii) Use your equation from **(ii)** and your answer to **(i)** to calculate the number of moles of anhydrous MX_2 produced in the **first** experiment.

moles of MX_2 = ... mol

(iv) Use your results from the **first** experiment to calculate the mass of anhydrous MX_2 produced.

mass of MX_2 = ... g

(v) Calculate the relative formula mass of MX_2.

relative formula mass of MX_2 = ...
[4]

(c) **(i)** Suggest how the experimental procedure could be modified to ensure that all of the water of crystallisation had been removed by heating hydrated **FA 4**.

....

....

....

(ii) The crucible was cooled with the lid on to prevent absorption of water vapour from the air. Suggest an alternative way of preventing water vapour being absorbed.

....

....
[2]

[Total: 12]

For Examiner's Use

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.
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) **FA 4** contains a halide ion.
FA 5 is a solution of a mixture of two salts. It contains **one cation** and **two anions**.

- Place a spatula measure of **FA 4** into a boiling tube.
- Half fill the boiling tube with distilled water and stir until the solid dissolves. Use this aqueous solution of **FA 4** as instructed in the following tests.

(i) Use aqueous silver nitrate and aqueous ammonia to identify the halide ion present in **FA 4**.
Carry out the same test on **FA 5**.
Use a 1 cm depth of solution in a test-tube for each test that you carry out.

Record your observations **and** conclusions in an appropriate form in the space below.

(ii) Write the **ionic** equation for the reaction of the halide ion in **FA 4** in test **(i)**.

....

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(iii) Use your conclusion from **3(a)(i)** and your answer to **2(b)(v)** to calculate the relative atomic mass of metal **M** in **FA 4**.
(A_r : F, 19.0; C*l*, 35.5; Br, 79.9; I, 127)

(If you were unable to calculate the relative formula mass of anhydrous MX_2 in **2(b)(v)** assume that it was 222, so that you are able to carry out this calculation).

A_r of **M** = ...

(iv) The relative atomic masses of some of the cations on page 10 are given below.
(A_r: Mg, 24.3; Ca, 40.0; Fe, 55.8; Cu, 63.5; Mn, 54.9; Zn, 65.4; Ba, 137; Pb, 207)

M is one of the cations listed above.
Suggest the identity of **M** and justify your answer.

....

....

(v) Suggest why it was not necessary to include the cations aluminium and chromium from page 10 in the list of relative atomic masses in **(iv)**.

....

....
[7]

(b) (i) Perform the experiments described below and record your observations in the table Record your observations in the table below.

test	*observation*
To 1 cm depth of aqueous **FA 4** in a test-tube, add aqueous ammonia.	
To 1 cm depth of **FA 5** in a test-tube, add aqueous ammonia.	

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(ii) Use **only** the results from **(i)** to identify the **two** possible cations that would have resulted in your observations for **FA 4**.

FA 4 contains .... or ....

(iii) Use the results from **(i)** to identify the cation in **FA 5**.

FA 5 contains..

(iv) Use the information on pages 10 and 11 to select **one** reagent, other than sodium hydroxide, to distinguish between the pair of possible cations in **FA 4** identified in **(ii)**.
Carry out the test with your selected reagent, using a 1 cm depth of **FA 4**.
From **this** test, identify the cation in **FA 4**.

reagent ....

observation ....

FA 4 contains ....

(v) Perform the tests described below and record your observations.
Identify the second anion in **FA 5**.

test	*observation*
To 1 cm depth of aqueous **FA 4** in a test-tube, add 1 cm depth of **FA 5**.	
Then add 2 cm depth of dilute hydrochloric acid.	

FA 5 contains ....

[6]

[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
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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