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

CANDIDATE NAME									
CENTRE NUMBER						CANDIDATE NUMBER			

7736236717

CHEMISTRY **9701/36**

Advanced Practical Skills 2 **October/November 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 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	
3	
Total	

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1 In this experiment you are to determine the concentration of aqueous potassium manganate(VII), **FB 3**, by titration.

In the titration potassium manganate(VII) is first reacted with acidified potassium iodide to produce iodine. The amount of iodine formed is then determined by titrating the mixture with sodium thiosulfate.

FB 1 is hydrated sodium thiosulfate, $Na_2S_2O_3.5H_2O$.
FB 2 is dilute sulfuric acid, H_2SO_4.
FB 3 is aqueous potassium manganate(VII), $KMnO_4$.
FB 4 is aqueous potassium iodide, KI.
starch indicator

(a) Method

Preparing a solution of FB 1

- Weigh the 250 cm^3 beaker and record the mass in the space below.
- Add all the **FB 1** to the beaker. Weigh the beaker with **FB 1** and record the mass.
- Calculate the mass of **FB 1** used and record this in the space below.
- Add approximately 100 cm^3 of distilled water to the beaker. Stir until all the solid has dissolved.
- Transfer the solution into the 250 cm^3 volumetric (graduated) flask labelled **FB 5**.
- Wash out the beaker thoroughly using distilled water and add the washings to the volumetric flask. Make the solution up to the mark using distilled water.
- Shake the flask thoroughly to mix the solution before using it for your titrations.
- This solution of sodium thiosulfate is **FB 5**.

Titration

- Use the measuring cylinder to add 20 cm^3 of **FB 2** to a conical flask.
- Use the measuring cylinder to add 10 cm^3 of **FB 4** to the same flask.
- Pipette 25.0 cm^3 of **FB 3** into the same flask.
  The colour of the mixture is caused by iodine.
- Fill the burette with **FB 5**.
- Begin each titration **without** adding the starch indicator.
  Add 10 drops of starch indicator when the colour of the mixture becomes (pale) yellow.
  The end-point is when the blue-black colour caused by the starch disappears.
- 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 **FB 5** 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.

suitable value = ... cm^3 of **FB 5** [1]

(c) Calculations

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

(i) Calculate the number of moles of sodium thiosulfate, **FB 1**, that were weighed out. The relative formula mass of hydrated sodium thiosulfate is 248.2.

moles of sodium thiosulfate = ... mol

(ii) Calculate the number of moles of sodium thiosulfate that were present in the volume of **FB 5** calculated in **(b)**.

moles of sodium thiosulfate = ... mol

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[Turn over

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(iii) Iodine produced by the reaction in the conical flask reacts with sodium thiosulfate. Use the equation below to calculate the number of moles of **iodine** that reacted with sodium thiosulfate in **(ii)**.

$$I_2 + 2Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$$

moles of I_2 = ... mol

(iv) The iodine is produced as a result of the oxidation of iodide ions in potassium iodide, **FB 4**, by potassium manganate(VII), **FB 3**.
The ionic equation for this reaction is

$$2MnO_4^- + 16H^+ + 10I^- \rightarrow 2Mn^{2+} + 5I_2 + 8H_2O$$

Calculate the number of moles of potassium manganate(VII), $KMnO_4$, that reacted to produce the iodine in **(iii)**.

moles of $KMnO_4$ = ... mol

(v) Calculate the concentration of potassium manganate(VII), in g dm^{-3}, in **FB 3**.
(A_r : O, 16.0 ; K, 39.1; Mn, 54.9)

concentration of $KMnO_4$ = ... g dm^{-3}
[5]

I	
II	
III	
IV	
V	

(d) (i) State the maximum error in any single reading of the burette.

maximum error = ... cm^3

(ii) Calculate the maximum percentage error in volume of **FB 5** in your **first** accurate titre.

maximum percentage error = ... %
[1]

[Total: 14]

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2 In this experiment you will heat two separate samples of a hydrated salt to drive off the water of crystallisation.
You will then calculate the relative atomic mass of the metal in the salt.

FB 6 is the hydrated salt.
The formula of **FB 6** is **M**$SO_4.7H_2O$, where **M** is the metal.

(a) Method

Record **all** weighings, in an appropriate form, in the space below.

- Record the mass of the empty crucible **without** its lid.
- Add between 2.0 and 2.4 g of **FB 6** into the 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 and carefully for about **two** minutes, with the lid off. Then heat very strongly for a further **three** minutes.
- Put the lid on the crucible and leave it to cool for approximately 10 minutes.

While you are waiting for the crucible to cool, start work on Question 3.

- When the crucible is cool, **remove the lid**, and weigh the crucible with the residue.
- Record the mass of anhydrous **M**SO_4 remaining in the crucible after heating and therefore calculate the mass of water lost.

- To prepare for the second experiment, use a spatula to remove the solid residue from the crucible into the beaker labelled **waste**.
- Reweigh the empty crucible **without** its lid.
- Carry out the experiment again. This time use between 2.5 and 2.9 g of **FB 6**.

[6]

I	
II	
III	
IV	
V	
VI	

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(b) Calculation

Show your working and give your answers to **three** significant figures.

(i) State **and** explain which of your two experiments in **(a)** is likely to be the more accurate.

.....

.....

.....

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

moles of H_2O = ... mol

(iii) Complete the equation for the removal of water from hydrated **FB 6**.
Include state symbols.

MSO_4.7H_2O (.....) → .....(.....) + ...(.....)

(iv) Using your answer to **(ii)**, calculate the number of moles of anhydrous **M**SO_4 produced in the **more accurate** experiment.

moles of **M**SO_4 = ... mol

(v) Use the mass of anhydrous **M**SO_4 produced in the **more accurate** experiment to calculate the relative formula mass of **M**SO_4.

relative formula mass of **M**SO_4 = ...

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(vi) Calculate the relative atomic mass of **M**.
(A_r : O, 16.0; S, 32.1)

(If you were unable to calculate the relative formula mass of anhydrous **M**SO_4 you may assume that it was 126.3. This is not the correct value.)

A_r of **M** = ...

(vii) The relative atomic masses of some of the cations on page 11 are given below.
(A_r : Mg, 24.3; Ca, 40.0; Fe, 55.8; Cu, 63.5; Mn, 54.9; Zn, 65.4)

M is a cation of one of the elements listed above.
Suggest the identity of **M** and justify your answer.

.....

.....

(viii) Suggest why it was **not** necessary to include the cations aluminium and chromium from page 11 in the list of relative atomic masses in **(vii)**.

.....

.....

.....
[8]

(c) The crucible was cooled with the lid on to prevent absorption of water vapour from the air. Suggest a better way of preventing water vapour being absorbed during cooling.

.....

.....
[1]

[Total: 15]

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[Turn over

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

(a) You will carry out further tests on the ions in **FB 6**.

- Put a spatula measure of **FB 6** into a test-tube.
- Half fill the test-tube with distilled water and stir until the solid dissolves.
- Use a 1 cm depth of the solution of **FB 6** in separate test-tubes for the tests you will carry out.

(i) Add aqueous sodium hydroxide to **FB 6** solution.
Add aqueous ammonia to **FB 6** solution.

Record your observations below.

(ii) Carry out a test of your choice to show that sulfate ions are present in **FB 6**.

reagent(s) used .....

observation(s) .....

.....

(iii) Give the **ionic** equation for the reaction in test **(ii)**.

.....

[4]

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(b) **FB 7**, **FB 8** and **FB 9** are aqueous solutions, each containing one cation and one anion. None of the cations and none of the anions in **FB 7**, **FB 8** and **FB 9** are identical.

(i) Add a 2 cm magnesium strip to a 2 cm depth of each solution in a clean test-tube.
Mix pairs of solutions as shown so that you can complete the table shown below.
Use 1 cm depths of solutions in clean test-tubes.
Record your observations in the table.

	FB 7	**FB 8**	**FB 9**
add a 2 cm strip of magnesium ribbon			
FB 7			[Keep this mixture for use in test **(iii)**]
FB 8			

(ii) The anion present in **FB 7** is the sulfate ion.
Identify **FB 7**, giving evidence from your observations.

FB 7 is ..... .

evidence .....

.....

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(iii) Add a 1 cm depth of aqueous hydrogen peroxide, **FB 10**, to the mixture of **FB 7** and **FB 9** that you kept from **(i)**. Then add three drops of starch.
Record your observation(s).
Identify the coloured chemical produced when hydrogen peroxide was added to the mixture of **FB 7** and **FB 9** and name the anion present in **FB 9**.

observations .....

.....

chemical produced .....

anion in **FB 9** .....

(iv) Give the chemical formula of the substance you observed when solutions **FB 8** and **FB 9** were mixed.

.....

[7]

[Total: 11]

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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 $\rightarrow$ (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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