Cambridge International Examinations
Cambridge International Advanced Subsidiary and Advanced Level

CANDIDATE NAME	

CENTRE NUMBER							CANDIDATE NUMBER				

6348483408

CHEMISTRY **9701/33**

Paper 3 Advanced Practical Skills 1 **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 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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CAMBRIDGE International Examinations

[Turn over

1 You are to determine, by titration, the change in oxidation number of a transition metal ion, **M**$^{2+}$, when reacted with acidified potassium manganate(VII).

FA 1 is 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$.
FA 2 is 0.0530 mol dm^{-3} transition metal salt, **M**SO_4.
FA 3 is 1.0 mol dm^{-3} sulfuric acid, H_2SO_4.

(a) Method

- Fill the burette with **FA 1**.
- Pipette 25.0 cm^3 of **FA 2** into the conical flask.
- Use the measuring cylinder to add 25 cm^3 of **FA 3** into the conical flask.
- Carry out a **rough titration** and record your burette readings in the space below. Add **FA 1** until the contents of the flask turn a permanent pale pink colour.

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 below, in a suitable form, all of your burette readings and the volume of **FA 1** 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 2** required .... cm^3 of **FA 1**. [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 potassium manganate(VII) present in the volume of **FA 1** calculated in **(b)**.

moles of $KMnO_4$ = ... mol

(ii) Calculate the number of moles of **M**SO_4 in 25.0 cm^3 of **FA 2**.

moles of **M**SO_4 in 25.0 cm^3 = ... mol

(iii) Use your answers to **(i)** and **(ii)** to calculate the number of moles of **M**SO_4 that react with 1 mole of $KMnO_4$.

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

(iv) Two possible equations for the reaction of acidified $KMnO_4$ with **M**SO_4 are below.

equation 1 $2KMnO_4 + 10\mathbf{M}SO_4 + 8H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5\mathbf{M}_2(SO_4)_3 + 8H_2O$

equation 2 $2KMnO_4 + 5\mathbf{M}SO_4 + 8H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5\mathbf{M}(SO_4)_2 + 8H_2O$

State and explain which of these two equations is consistent with your answer to **(iii)**.

....

....

(v) Use your answer to **(iv)** to state the oxidation number of the transition metal **M** in the product of the reaction.

....

[5]

[Total: 13]

I	
II	
III	
IV	
V	

2 You will determine the enthalpy change, ΔH, for the reaction between magnesium and dilute sulfuric acid. The equation for the reaction is given below.

$$Mg(s) + H_2SO_4(aq) \rightarrow MgSO_4(aq) + H_2(g)$$

FA 3 is 1.00 mol dm^{-3} sulfuric acid, H_2SO_4.
two different coiled lengths of magnesium ribbon, Mg.

(a) Method

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

- Weigh the shorter piece of magnesium ribbon and record its mass.
- Support the plastic cup in the 250 cm^3 beaker.
- Use the measuring cylinder to transfer 50 cm^3 of **FA 3** into the plastic cup.
- Place the thermometer in the **FA 3** in the plastic cup and record the initial temperature.
- Add the shorter piece of magnesium ribbon into the plastic cup. Ensure that all of the magnesium is in contact with the acid. (**Care**: acid spray may occur.)
- Stir the mixture and record the maximum temperature.
- Empty and rinse the plastic cup. Shake out any excess water.
- Repeat the experiment using the longer piece of magnesium ribbon and record all your data.

Results

[4]

(b) Calculations

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

(i) Show by calculation that the sulfuric acid, **FA 3**, was used in excess in both experiments. (A_r: Mg, 24.3)

(ii) State an observation which confirms that the sulfuric acid, **FA 3**, was in excess.

....

(iii) Calculate the heat energy produced when the **shorter** piece of magnesium was added to **FA 3**.
(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm^3 of solution by 1.0 °C.)

heat energy produced = ... J

(iv) Calculate the enthalpy change, in kJ mol^{-1}, for the reaction between the **shorter** piece of magnesium and the sulfuric acid.

enthalpy change = ... kJ mol^{-1}
(sign) (value)

(v) Calculate the heat energy produced when the **longer** piece of magnesium was added to **FA 3**.
(Assume that 4.3 J of heat energy changes the temperature of 1.0 cm^3 of solution by 1.0 °C.)

heat energy produced = ... J

(vi) Calculate the enthalpy change, in kJ mol^{-1}, for the reaction between the **longer** piece of magnesium and the sulfuric acid.

enthalpy change = ... kJ mol^{-1}
(sign) (value)

[5]

(c) **(i)** What is the maximum error in a reading of the thermometer used in this experiment?

maximum error = ... °C.

(ii) Which of your temperature changes has the higher percentage error?

....

(iii) Calculate this maximum percentage error.

maximum percentage error in the temperature change = ... %

[1]

(d) Apart from errors due to heat loss and thermometer readings, suggest another significant source of error in this experiment. State what improvement could be made to the procedure to reduce this error.

....

....

.... [2]

[Total: 12]

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 4**, **FA 5** and **FA 6** are solutions, each containing one transition metal ion. One of the solutions also contains the ammonium ion. All the cations present are listed in the Qualitative Analysis Notes on page 10.

(i) Carry out the following tests on the three solutions.

I	
II	
III	
IV	

test	*observations*
To a 1 cm depth of **FA 4** in a test-tube, add **FA 1**, aqueous potassium manganate(VII), dropwise.	
To a 1 cm depth of **FA 5** in a test-tube, add **FA 1**, aqueous potassium manganate(VII), dropwise.	
To a 1 cm depth of **FA 6** in a test-tube, add **FA 1**, aqueous potassium manganate(VII), dropwise.	

(ii) State which solution(s) contain ions which have been oxidised.

....

[4]

(b) (i) Select a reagent or reagents to identify **all** the cations present in the three solutions.

reagent(s) ....

Carry out experiments using your reagent(s) on each of **FA 4**, **FA 5** and **FA 6** and record your observations in a suitable form in the space below.

I	
II	
III	
IV	
V	
VI	
VII	
VIII	

(ii) Use your observations to identify the cations present in the three solutions.

FA 4 contains ....

FA 5 contains ....

FA 6 contains ....

[8]

(c) Each of the solutions **FA 4**, **FA 5** and **FA 6** contains either a chloride or a sulfate ion.

(i) Choose a reagent or reagents to identify which solution(s) contain **chloride** ions.

reagent(s) ....

Use your reagent(s) to carry out a test on each of **FA 4**, **FA 5** and **FA 6** and record your results in the space below.

(ii) State which solution(s) contain a chloride ion.

....

[3]

[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
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 $\rightarrow$ (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

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