

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

CANDIDATE NAME							
CENTRE NUMBER					NDIDATE MBER		



CHEMISTRY 9701/32

Paper 32 Practical Test May/June 2007

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the Instructions to Supervisors

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 are advised to show all working in calculations.

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

For Examiner's Use		
1		
2		
3		
Total		

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

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[Turn over

- You are required to determine the concentration in g dm⁻³ of hydrated ammonium iron(II) sulphate, $(NH_4)_2SO_4$.FeSO₄.6H₂O, in the solution **FB 1**.
 - **FB 1** contains hydrated ammonium iron(II) sulphate.
 - FB 2 is 0.0120 mol dm⁻³ potassium manganate(VII), KMnO₄.

(a) Dilution of FB 1

By using a burette measure between 36.00 cm³ and 37.00 cm³ of **FB 1** into the 250 cm³ graduated flask labelled **FB 3**.

Record your burette readings and the volume of **FB 1** added to the flask in the space below.

Make up the contents of the flask to the 250 cm³ mark with distilled water. Place the stopper in the flask and mix the contents thoroughly by slowly inverting the flask a number of times.

Titration

Fill a second burette with FB 2.

Pipette $25.0\,\mathrm{cm^3}$ of **FB 3** into a conical flask. Use a measuring cylinder to add approximately $10\,\mathrm{cm^3}$ of $1.0\,\mathrm{mol}$ dm⁻³ sulphuric acid, $\mathrm{H_2SO_4}$, and titrate with **FB 2** until the first permanent pink colour remains in the solution.

Perform one rough (trial) titration and sufficient further titrations to obtain accurate results.

Record your titration results in the space below. Make certain that your recorded results show the precision of your working.

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(b)	From your titration results obtain a suitable volume of FB 2 to be used in your calculations. Show clearly how you obtained this volume.
	[1]
Cal	culations
Sho	w your working and appropriate significant figures in all of your calculations.
(c)	Calculate how many moles of KMnO ₄ were run from the burette during the titration.
	mol of KMnO $_4$ were run from the burette. Calculate how many moles of Fe $^{2+}$ ions reacted with the KMnO $_4$ run from the burette. $MnO_4^-(aq) \ + \ 5Fe^{2+}(aq) \ + \ 8H^+(aq) \ \longrightarrow \ Mn^{2+}(aq) \ + \ 5Fe^{3+}(aq) \ + \ 4H_2O(I)$
	mol of ${\rm Fe^{2+}}$ reacted with the ${\rm KMnO_4}$ run from the burette. Calculate the concentration, in mol dm $^{-3}$, of ${\rm Fe^{2+}}$ in FB 3 .

Concentration of Fe²⁺ in **FB 3** = mol dm⁻³.

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[Total: 16]

Calculate the concentration, in mol dm^{-3} , of Fe²⁺ in **FB 1**.

	Concentration of Fe ²⁺ in FB 1 = mol dm ⁻³ .
	Calculate, to 4 significant figures , the concentration of $(NH_4)_2SO_4.FeSO_4.6H_2O$ in FB 1 in g dm ⁻³ . [A_r : Fe, 55.8; H, 1.0; N, 14.0; O, 16.0; S, 32.1]
	FB 1 contains g dm ⁻³ of (NH ₄) ₂ SO ₄ .FeSO ₄ .6H ₂ O. [5]
(d)	A student learns that the solution of the iron(II) salt has been prepared by dissolving the solid in distilled water that has absorbed air from the laboratory. Suggest a way in which the distilled water can be prepared and stored in the laboratory to ensure that it contains a minimum of dissolved air.
(e)	Estimate the error in reading a volume from a burette.
	smallest division on burette scale =cm ³
	estimated error in reading a volume = ±cm ³ [1]
(f)	A titre value is obtained by the difference between final and initial burette readings.
	What is the maximum possible error in obtaining a titre reading?
	estimated maximum error in the titre = \pm
(g)	During one titration a student reads the burette twice. Each reading has an error but the titre has no error. Explain how this can happen.
	[1]

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2 Read through the question before starting any practical work.

You are required to determine the enthalpy change when citric acid reacts with an excess of sodium hydrogencarbonate.

Citric acid, found in citrus fruit such as lemons and limes, is 2-hydroxypropane-1,2,3-tricarboxylic acid.

$$\mathrm{CH_2\text{-}CO_2H}$$
 I $\mathrm{C(OH)\text{-}CO_2H}$ I $\mathrm{CH_2\text{-}CO_2H}$

FB 4 is 0.8 mol dm⁻³ citric acid.

FB 5 is solid sodium hydrogencarbonate, NaHCO₃.

(a) Citric acid is a triprotic (tribasic) acid – one mole of the acid reacts with three moles of sodium hydrogencarbonate.

Calculate the minimum mass of sodium hydrogencarbonate that will react with all of the acid in 50.0 cm³ of **FB 4**.

mass of
$$NaHCO_3 = \dots g[1]$$

(b) Method

Follow the instructions below to determine the enthalpy change for the reaction. You will carry out the experiment twice.

- Weigh the empty weighing bottle.
- Weigh the bottle with between 11.5 g and 12.0 g, an excess, of **FB 5**.
- Support the plastic cup in the 250 cm³ beaker and pipette into it 50.0 cm³ of **FB 4**.
- Measure and record the steady temperature of the **FB 4** in the plastic cup.
- Add the **FB 5** from the weighing bottle, a little at a time, to the plastic cup.
- Stir and record the lowest temperature reached.
- Reweigh the empty weighing bottle.

In an appropriate form at the top of the next page record

- all measurements of mass and temperature,
- the temperature fall, ΔT .

Empty and rinse the plastic cup.

Repeat the experiment and calculate the mean value of ΔT .

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Results

The mean value of ΔT is°C.

[6]

(c) Calculate the enthalpy change of reaction using the following expression.

$$\Delta H_{\rm reaction} = {\rm mean} \ \Delta T \ \times \ 4.3 \ {\rm kJ} \ {\rm mol}^{-1}$$

Your answer should include the appropriate sign.

$$\Delta H_{\text{reaction}} = \dots kJ \text{ mol}^{-1} [1]$$

[Total: 8]

3 You are provided with three solutions, FB 6, FB 7 and FB 8, each containing one cation and one anion.

One or more of the solutions contains a halide ion. One or more of the solutions contains a sulphate or sulphite ion.

Identification of the anions in FB 6, FB 7 and FB 8

- (a) By reference to the Qualitative Analysis Notes on page 12 you are to select and use
 - (i) one reagent to precipitate any halide ion that is present,
 - (ii) a second reagent to confirm the identity of any halide ion present.

Because the solutions are coloured you will need to remove traces of solution from the precipitates.

Record the tests performed, the practical procedures used and the observations made for each of the solutions.

Present this information as clearly as possible in a suitable format in the space below.

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Use your observations to identify any halide ions present in the solutions FB 6 , FE and FB 8 and state which ion is present in which solution.	3 7
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[7]

- (b) Select reagents and carry out tests
 - (i) to show which of the solutions contains a sulphate ion or a sulphite ion, and
 - (ii) to establish which of these ions is present.

Record your tests and observations below.

State which of the ions, sulphate or sulphite, is present in which of the solutions FI FB 7 and FB 8 and explain how you reached this conclusion from your tests above.	
	[3]

Identification of the cations in FB 6, FB 7 and FB 8

(c) Using aqueous sodium hydroxide and aqueous ammonia it is possible to identify two of the cations present and to draw some conclusions about the nature of the remaining cation.

Carry out tests with these reagents, recording details of what you did and observed in a suitable format in the space below.

(d)	Explain how your observations in (c) identify two of the cations present and which of the solutions contain those cations.
	The cation contained in solution FB is
	explanation
	The cation contained in solution FB is
	explanation
	What conclusion of a general nature about the third cation can you draw from your observations in (c) ?
	[2]

[Total: 16]

Key: [ppt. = precipitate.]

1 Reactions of aqueous cations

	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)	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. insoluble in excess	green ppt. 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. insoluble in excess	off-white ppt. 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 pale 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 A <i>l</i> 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)
sulphate, SO ₄ ²⁻ (aq)	gives white ppt. with Ba ²⁺ (aq) or with Pb ²⁺ (aq) (insoluble in excess dilute strong acid)
sulphite, SO ₃ ²⁻ (aq)	SO ₂ liberated with dilute acids; gives white ppt. with Ba ²⁺ (aq) (soluble in excess dilute strong acid)

3 Tests for gases

gas	test and test results
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)
chloride, Cl ₂	bleaches damp litmus paper
hydrogen, H ₂	"pops" with a lighted splint
oxygen, O ₂	relights a glowing splint
sulphur dioxide, SO ₂	turns potassium dichromate(VI) (aq) from orange to green

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