

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

CANDIDATE NAME					
CENTRE NUMBER		CANDID. NUMBER			
CHEMISTRY			9701/31		
Advanced Pract	ical Skills 1		October/November 2013		
			2 hours		
Candidates ans	wer on the Question Paper.				
Additional Mate	Additional Materials: As listed in the Confidential Instructions				
Write your Cent Give details of t Write in dark blu You may use a Do not use stap DO <b>NOT</b> WRITI	he practical session and labor ue or black pen. soft pencil for any diagrams, les, paper clips, highlighters E IN ANY BARCODES.				
You may lose m	lators may be used.	r working or if you do not use approp	priate units.		
Qualitative Analysis Notes are printed on pages 11 and 12.			Session		

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.

For Exam	For Examiner's Use		
1			
2			
3			
Total			

Laboratory

This document consists of **12** printed pages.



1 In this experiment you are to determine the relative formula mass of an iron(II) salt by titration with potassium manganate(VII).

For Examiner's Use

FA 1 is the iron(II) salt. FA 2 is  $0.0200 \text{ mol dm}^{-3}$  potassium manganate(VII), KMnO<sub>4</sub>. FA 3 is dilute sulfuric acid, H<sub>2</sub>SO<sub>4</sub>.

## (a) Method

## Preparing a solution of FA 1

- Weigh the 250 cm<sup>3</sup> beaker and record the mass in the space below.
- Add all the **FA 1** provided 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.
- Use a measuring cylinder to add approximately 100 cm<sup>3</sup> of **FA 3** to the beaker. Stir until all the solid has dissolved.
- Transfer the solution into the 250 cm<sup>3</sup> volumetric (graduated) flask labelled **FA 4**.
- 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 the iron(II) salt is **FA 4**.

## Titration

- Pipette 25.0 cm<sup>3</sup> of **FA 4** into a conical flask.
- Use a measuring cylinder to add 20 cm<sup>3</sup> of **FA 3** to the flask.
- Fill the burette with **FA 2**.
- Titrate **FA 4** with **FA 2** until the solution changes to a permanent pink colour.
- Perform a rough titration and record your burette readings in the space below.

The rough titre is ...... cm<sup>3</sup>.

- 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.

For Examiner's Use

Ι	
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<sup>3</sup> of **FA 4** required ..... cm<sup>3</sup> of **FA 2** [1]

		-	
(c)	Cal	culations	For Examiner's
		bw your working and appropriate significant figures in the final answer to <b>each</b> step of Ir calculations.	Use
	(i)	Calculate the number of moles of potassium manganate(VII) present in the volume of FA 2 calculated in (b).	
		moles of KMnO <sub>4</sub> = mol	
	(ii)	The half-equation for the reduction of a manganate(VII) ion is:	
	(11)		
		$MnO_{4}^{-} + 8H^{+} + 5e^{-} \rightarrow Mn^{2+} + 4H_{2}O$	
		Give the half-equation for the oxidation of an iron(II) ion to an iron(III) ion.	
		Therefore, 1 mole of manganate(VII) ions reacts with 5 moles of iron(II) ions.	
	(iii)	Calculate the number of moles of iron(II) ions present in 25.0 cm <sup>3</sup> of solution <b>FA 4</b> .	
		moles of $Fe^{2+}$ in 25.0 cm <sup>3</sup> of <b>FA 4</b> = mol	
	(iv)	Calculate the number of moles of iron(II) ions present in 250 cm <sup>3</sup> of solution FA 4.	
		moles of $Fe^{2+}$ in 250 cm <sup>3</sup> of <b>FA 4</b> = mol	
	(v)	In 1 mole of the iron(II) salt, <b>FA 1</b> , there is 1 mole of iron(II) ions.	
		Use the mass of <b>FA 1</b> you weighed out to calculate the relative formula mass of the iron(II) salt.	Ι
			I
			III
			IV
		relative formula mass =[5]	V

(d) (i) A 25 cm<sup>3</sup> pipette is accurate to ±0.06 cm<sup>3</sup>. Calculate the maximum percentage error when the pipette was used to measure use Use

percentage error in measuring FA 4 = .....%

(ii) State the maximum error in the mass of the 250 cm<sup>3</sup> beaker that you recorded in (a).

maximum error = ..... g

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

maximum percentage error = .....%

[Total: 15]

2 In this experiment you are to determine the formula of hydrated barium chloride, **FA 5**, by heating to remove the water of crystallisation. You will heat **two** separate samples. The anhydrous barium chloride does not decompose when heated.

**FA 5** is hydrated barium chloride,  $BaCl_2$ .**x**H<sub>2</sub>O

### (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 **FA 5** 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 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 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 barium chloride remaining in the crucible after heating and the mass of water lost.
- To prepare for the second experiment, use a spatula to remove the 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 1.5 and 1.9 g of **FA 5**.

Ι	
II	
III	
IV	
V	
VI	

For

Examiner's

Use

[6]

[3]

[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. 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** is hydrated barium chloride.

**FA 6** is the same iron(II) salt used in Question 1. It contains **one other** cation and **one** anion.

(i) Place a small spatula measure of **FA 6** into a test-tube. Dissolve the solid in about a 5 cm depth of distilled water. Use the solution for the following tests.

test	observations
To a 1 cm depth of aqueous <b>FA 6</b> in a boiling tube, add aqueous sodium hydroxide until no further change occurs, then	
heat the mixture carefully.	
Dissolve a few crystals of <b>FA 5</b> in a 1 cm depth of distilled water in a test-tube. Add a 1 cm depth of <b>FA 6</b> , then	
add excess dilute hydrochloric acid to the mixture.	

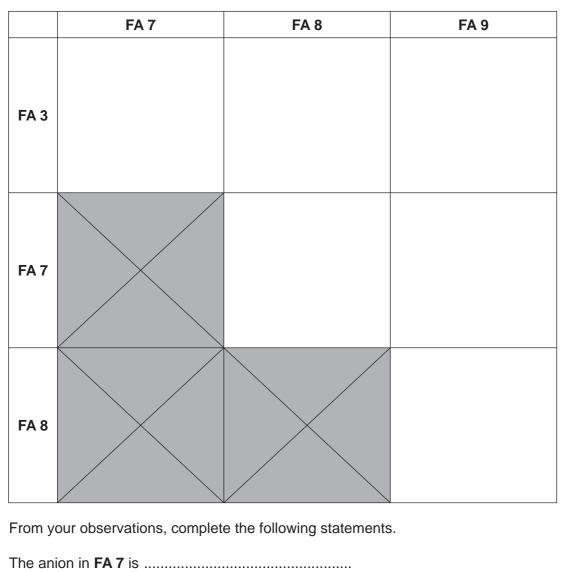
For Examiner's Use

Identify the ions present in FA 6.	For Examiner's
cations: Fe <sup>2+</sup> and anion:	Use
Give the ionic equation for the reaction of iron(II) ions with hydroxide ions.	
Place a <b>small</b> spatula measure of <b>FA 6</b> into a <b>hard-glass</b> test-tube. Heat gently, then strongly, until no further change is observed. Record your observations in the space below.	
	cations: Fe <sup>2+</sup> and anion: Give the ionic equation for the reaction of iron(II) ions with hydroxide ions. Place a <b>small</b> spatula measure of <b>FA 6</b> into a <b>hard-glass</b> test-tube. Heat gently, then strongly, until no further change is observed.

[7]

- For Examiner's Use
- (b) FA 7, FA 8 and FA 9 are aqueous solutions. Each contains one cation and one anion. FA 3 is dilute sulfuric acid,  $H_2SO_4$ .

Mix pairs of solutions so that you can complete the table below. For each test, use 1 cm depths of each solution in clean test-tubes. Record your observations in the table.



 	_	<b>•</b> •						

The cation in **FA 8** is .....

The anion in FA 9 is .....

[6]

[Total: 13]

# **Qualitative Analysis Notes**

## *Key:* [*ppt.* = *precipitate*]

# 1 Reactions of aqueous cations

	reaction with						
ion	NaOH(aq)	NH <sub>3</sub> (aq)					
aluminium, A <i>l</i> ³+(aq)	white ppt. soluble in excess	white ppt. insoluble in excess					
ammonium, NH₄⁺(aq)	no ppt. ammonia produced on heating	_					
barium, Ba²⁺(aq)	no ppt. (if reagents are pure)	no ppt.					
calcium, Ca²⁺(aq)	white ppt. with high [Ca <sup>2+</sup> (aq)]	no ppt.					
chromium(III), Cr³⁺(aq)	grey-green ppt. soluble in excess giving dark green solution	grey-green ppt. insoluble in excess					
copper(II), Cu²+(aq)	pale blue ppt. insoluble in excess	blue ppt. soluble in excess giving dark blue solution					
iron(II), Fe²+(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 <sup>3+</sup> (aq)	red-brown ppt. insoluble in excess	red-brown ppt. insoluble in excess					
lead(II), Pb²+(aq)	white ppt. soluble in excess	white ppt. insoluble in excess					
magnesium, Mg²+(aq)	white ppt. insoluble in excess	white ppt. insoluble in excess					
manganese(II), Mn²+(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, Zn²+(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 <sub>3</sub> <sup>2–</sup>	CO <sub>2</sub> liberated by dilute acids
chromate(VI), CrO <sub>4</sub> <sup>2–</sup> (aq)	yellow solution turns orange with H <sup>+</sup> (aq); gives yellow ppt. with Ba <sup>2+</sup> (aq); gives bright yellow ppt. with Pb <sup>2+</sup> (aq)
chloride, C <i>l</i> ⁻(aq)	gives white ppt. with Ag <sup>+</sup> (aq) (soluble in $NH_3(aq)$ ); gives white ppt. with Pb <sup>2+</sup> (aq)
bromide, Br⁻(aq)	gives cream ppt. with Ag <sup>+</sup> (aq) (partially soluble in NH <sub>3</sub> (aq)); gives white ppt. with Pb <sup>2+</sup> (aq)
iodide, I⁻(aq)	gives yellow ppt. with Ag <sup>+</sup> (aq) (insoluble in NH <sub>3</sub> (aq)); gives yellow ppt. with Pb <sup>2+</sup> (aq)
nitrate, NO <sub>3</sub> ⁻(aq)	$NH_3$ liberated on heating with $OH^-(aq)$ and $Al$ foil
nitrite, NO₂⁻(aq)	NH <sub>3</sub> liberated on heating with OH <sup>-</sup> (aq) and A <i>l</i> foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO <sub>2</sub> in air)
sulfate, SO <sub>4</sub> <sup>2-</sup> (aq)	gives white ppt. with Ba <sup>2+</sup> (aq) or with Pb <sup>2+</sup> (aq) (insoluble in excess dilute strong acids)
sulfite, SO <sub>3</sub> ²⁻(aq)	$SO_2$ liberated with dilute acids; gives white ppt. with Ba <sup>2+</sup> (aq) (soluble in excess dilute strong acids)

## 3 Tests for gases

gas	test and test result
ammonia, NH <sub>3</sub>	turns damp red litmus paper blue
carbon dioxide, CO <sub>2</sub>	gives a white ppt. with limewater (ppt. dissolves with excess CO <sub>2</sub> )
chlorine, $Cl_2$	bleaches damp litmus paper
hydrogen, H <sub>2</sub>	"pops" with a lighted splint
oxygen, O <sub>2</sub>	relights a glowing splint
sulfur dioxide, SO <sub>2</sub>	turns acidified aqueous potassium dichromate(VI) from orange to green

Permission to reproduce items where third-party owned material protected by copyright is included has been sought and cleared where possible. Every reasonable effort has been made by the publisher (UCLES) to trace copyright holders, but if any items requiring clearance have unwittingly been included the publisher will be pleased to make amends at the earliest possible opportunity.

University of Cambridge International Examinations is part of the Cambridge Assessment Group. Cambridge Assessment is the brand name of University of Cambridge Local Examinations Syndicate (UCLES), which is itself a department of the University of Cambridge.