Name: Centre/Index Number: Class:



H2 CHEMISTRY

9729/04

Paper 4 Practical

25 August 2022 2 hours 30 minutes

Candidates answer on the Question Paper.

READ THESE INSTRUCTIONS FIRST

Write your centre number, index number, name and class at the top of this page.

Give details of the practical shift 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.

Answer all questions in the spaces provided on the Question Paper.

The use of an approved scientific calculator is expected, where appropriate.

You may lose marks if you do not show your working or if you do not use appropriate units.

Qualitative Analysis Notes are printed on pages 19 and 20.

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.

Shift		
Laboratory		

For Examiner's Use	
1	16
2	16
2	10
3	14
4	15
Total	55

Answer all questions in the spaces provided.

1 Determination of solubility product, K_{sp} , of magnesium carbonate

The solubility of solid magnesium carbonate, MgCO₃, in water is low. In this experiment, you will determine the solubility product, K_{sp} , of magnesium carbonate using volumetric analysis.

$$MgCO_3(s) \rightleftharpoons Mg^{2+}(aq) + CO_3^{2-}(aq)$$

A saturated solution of magnesium carbonate was prepared by collecting the filtrate after mixing aqueous solutions of magnesium sulfate and sodium carbonate.

You will then determine the amount of carbonate ions left in the filtrate using hydrochloric acid.

FA 1 is a saturated solution of magnesium carbonate, MgCO $_3$. **FA 2** is 0.003 mol dm $^{-3}$ hydrochloric acid, HCl. methyl orange indicator

Note:

- Read the procedures given in part (a) and part (b)(i).
- Part (a) has been done for you. You are not required to carry out part (a), but you will still
 need to read part (a) to know how FA 1 is prepared.

(a) Preparation of FA 1

- 1. Use a measuring cylinder to transfer 20 cm³ of 0.100 mol dm⁻³ magnesium sulfate to a clean and dry 250 cm³ beaker.
- 2. Use a measuring cylinder to transfer 50 cm³ of 0.0400 mol dm⁻³ sodium carbonate to the same beaker. A white precipitate forms.
- 3. Stir the mixture thoroughly. Leave this mixture to stand for 15 minutes to allow equilibrium to be reached.
- 4. Filter the reaction mixture into a dry 250 cm³ conical flask, using dry filter funnel and filter paper. The filtrate is labelled as **FA 1**. Do not wash the white precipitate with water.

(b) (i) Titration of FA 1 against FA 2

- 1. Fill a burette with FA 2.
- 2. Pipette 10.0 cm³ of **FA 1** into a 100 cm³ conical flask.
- 3. Add a few drops of methyl orange indicator to the conical flask.
- 4. Titrate the solution in the conical flask with **FA 2**. The end-point for this titration is reached when the solution changes colour from yellow to orange.
- 5. Record all burette readings, to an appropriate level of precision, in the space provided on page 3.
- 6. Repeat steps 1 to 5 until consistent results are obtained.

Titration results

(ii)	Calculate the total amount of CO ₃ ²⁻ ions present in the total volume of filtrate prepared in (a) .
	total amount of CO ₃ ²⁻ ions = mol [1]
(iii)	Hence, calculate the amount of CO ₃ ²⁻ ions precipitated as MgCO ₃ .
	amount of CO_3^{2-} ions precipitated as $MgCO_3 = \dots mol$ [2]
(iv)	Deduce the amount of Mg^{2+} ions removed by precipitation, in step 3 of the procedure in (a) . Hence, calculate the amount of Mg^{2+} ions left in FA 1 .
	amount of Mg ²⁺ ions removed by precipitation = mol
	amount of Mg ²⁺ ions left = mol [2]

	(v)	Write an expression for the solubility product, \textit{K}_{sp} , of magnesium carbonate. Include units in your answer.
		\mathcal{K}_{sp} of MgCO ₃ =
		units =[1]
	(vi)	Calculate a value for the solubility product, $K_{\!sp}$, of magnesium carbonate.
		$K_{sp} =$ [1]
(d)		dent follows the procedures described and obtained a higher K_{sp} value compared to the ture value of 6.82×10^{-6} at 25 °C.
	Give	a possible explanation for the higher $K_{\!\scriptscriptstyle \mathrm{Sp}}$ value obtained.
		[1]
		[Total: 16]

2 Planning

Avogadro's constant, L, is defined as the number of particles in one mole of a substance. The currently accepted value is 6.02×10^{23} mol⁻¹.

The Avogadro's constant, *L*, can be determined through the electrolysis of acidified aqueous potassium iodide.

During electrolysis, the amount of material discharged at each of the electrodes depends solely on the amount of current that has passed through the system.

A student conducted the electrolysis experiment. When a current was passed through a solution of acidified aqueous potassium iodide, hydrogen gas was produced at one electrode, while iodine was produced at the other electrode.

The amount of hydrogen produced can be determined from the volume of hydrogen gas collected by downward displacement of water. The amount of iodine produced can be determined by titration of a portion of the resultant solution with aqueous sodium thiosulfate.

$$I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$$

(a)	Explain why the volume of gas collected was higher than the theoretical volume of hydrogen gas that can be produced in the electrolysis.
	[1]

(b) (i) Plan an investigation to determine the amount of iodine produced in the electrolysis of acidified aqueous potassium iodide, if a current of 0.50 A is passed for 10 minutes, and hence determine a value for the Avogadro's constant, *L*.

You may assume that you are provided with:

- 500 cm³ of 0.500 mol dm⁻³ acidified aqueous potassium iodide,
- 0.0100 mol dm⁻³ aqueous sodium thiosulfate.
- ammeter (instrument used to measure current)
- wires and crocodile clips
- adjustable DC power source (battery)
- graphite rods
- the equipment normally found in a school or college laboratory.

In your plan you should include brief details of:

- a fully labelled diagram of the electrolytic set—up, showing how you would collect and measure the volume of hydrogen gas,
- the apparatus you would use.
- the procedure you would follow for the electrolysis of aqueous potassium iodide.

You need not include details of:

- the adjustment of the current setting of the power source to 0.50 A,
- the titration of the resultant solution with aqueous sodium thiosulfate.

(ii) Table 2.1 gives information about the titration of the resultant solution with aqueous sodium thiosulfate.

Table 2.1

total volume of resultant solution after electrolysis / cm ³	500
volume of portion of resultant solution used in titration / cm ³	25.0
volume of sodium thiosulfate used to reach end-point / cm³	V

By means of calculations, show how you would use the information in Table 2.1 to determine the Avogadro's constant, L, in terms of V. Show your working clearly.

[electronic charge, $e = -1.60 \times 10^{-19}$ C]

(c)	A student suggested that the percentage error in titre value can be reduced if $0.0500~\rm mol~dm^{-3}$ aqueous sodium thiosulfate was used instead.
	Do you agree with this student? Explain your answer.
	[41]

[Total: 10]

[3]

3 Determination of a value for the enthalpy change of reaction between CO₂ and KOH via Hess' Law

The reaction between KHCO₃(s) and HCl(aq) is an endothermic process.

$$KHCO_3(s) + HCl(aq) \rightarrow KCl(aq) + CO_2(q) + H_2O(l)$$
 ΔH_1

Using ΔH_1 and the standard enthalpy change of neutralisation, you will be able to determine the enthalpy change, ΔH_2 , of the following reaction between CO₂ and KOH via Hess' Law.

$$CO_2(g) + KOH(ag) \rightarrow KHCO_3(s)$$
 ΔH_2

FA 3 is solid potassium bicarbonate, KHCO₃.

FA 4 is 2.00 mol dm $^{-3}$ hydrochloric acid, HCl.

(a) Determination of ΔH_1

In this experiment, you will be adding KHCO₃(s) to HCl(aq) in a Styrofoam cup. You will then measure the temperature of the reaction mixture over time. Plotting an appropriate graph using the data obtained will allow you to determine ΔH_1 .

In an appropriate format in the space provided on page 10, prepare tables in which to record for your experiment:

- all weighings to an appropriate level of precision,
- all values of temperature, *T*, to an appropriate level of precision,
- all values of time, t, recorded to 0.5 min.

It is important that you measure each temperature at the specified time.

Procedure

- 1. Weigh the capped bottle containing solid **FA 3**.
- 2. Place a clean dry Styrofoam cup inside a second Styrofoam cup, then place these in a glass beaker.
- 3. Using a measuring cylinder, add 40 cm³ of **FA 4** into the first Styrofoam cup.
- 4. Stir the solution in the cup with the thermometer. Read and record its temperature, T (time, t = 0.0 min).
- 5. Continue to stir the solution. Read and record *T* every 0.5 min.
- 6. At exactly 2.0 minutes, transfer all the solid **FA 3** into the Styrofoam cup in three portions, taking extra care that the mixture that does not overflow. Stir the mixture but do not read *T*.
- 7. Continue to stir the mixture. Read and record T at t = 2.5 min.
- 8. Continue stirring and reading T every 0.5 min until t = 7.0 min.
- 9. Reweigh the emptied bottle and its cap.

(i) Results

(ii) Plot a graph of temperature, *T*, on the y-axis, against time, *t*, on the axis, on the grid in Fig. 3.1.

Draw a best-fit straight line taking into account all of the points before t = 2.0 min.

Draw another best-fit straight line taking into account all of the points after the temperature of the mixture has started to increase steadily.

Extrapolate both lines to t = 2.0 min.

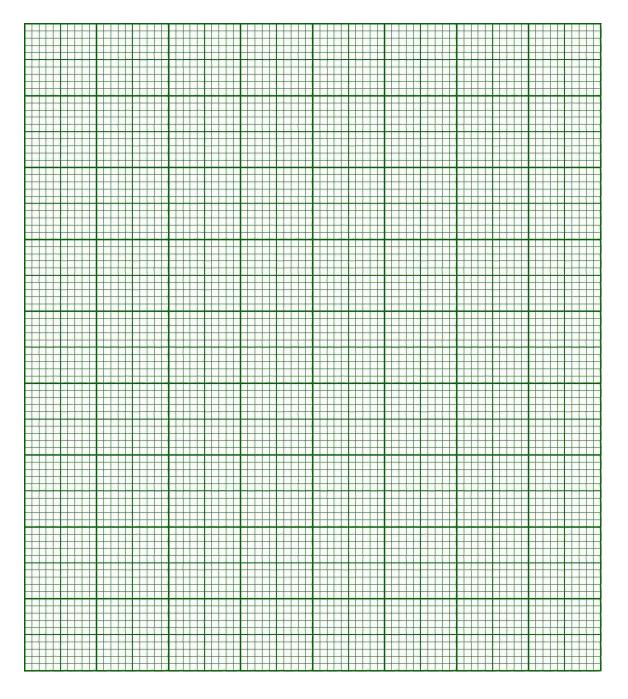


Fig 3.1

(iii)	From your graph, read the minimum temperature, T_{\min} , and the maximum temperature, T_{\max} , at 2.0 min. Record these values in the spaces provided below. Deduce the temperature change, ΔT , at $t=2.0$ min.	
	$T_{min} = \dots$	
	$T_{max} = \dots$	
	$\Delta T = \dots$ [1]	
(iv)	Calculate the heat change, q , using the values you deduced in (a)(iii).	
	You should assume that the specific heat capacity of the solution is 4.18 J $\rm g^{-1}$ K $^{-1}$, and that the density of the solution is 1.00 g cm $^{-3}$.	
	<i>q</i> = [1]	
(v)	Hence, determine the enthalpy change of reaction, ΔH_1 .	
	[A _r : K, 39.1; H, 1.0; C, 12.0; O, 16.0]	
	$\Delta H_1 = \dots $ [2]	
(vi)	Calculate the percentage error associated with the measurement of the volume of FA 4 .	
	percentage error = [1]	

		13	
	(vii)	Suggest the effect that using 80 cm ³ , rather than 40 cm ³ , of HC l would have or value for ΔT . Hence, deduce and explain the effect this will have on the value for	
			[1]
b)	Dete	ermination of Δ <i>H</i> ₂	
	The –57.3	enthalpy change of neutralisation between a strong acid and a strong bas $.3~{\rm kJ~mol^{-1}}.$	e is
		ng this information and your answer in (a)(v) , draw an appropriate energy cyclermine ΔH_2 , the enthalpy change of following reaction.	e to
		$CO_2(g) + KOH(aq) \rightarrow KHCO_3(s)$ ΔI	H_2
		$\Delta H_2 = \dots$. [2]
		[Total	: 14]

4 Inorganic Analysis

(a) FA 5 contains two cations and two anions.

FA 6 is a solution of ammonium vanadate(V), NH₄VO₃.

You are to perform the tests described in Table 4.1 and record your observations in the table. Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured.

In all tests, the reagents should be added gradually until no further change is observed unless you are instructed otherwise.

You should indicate clearly at which stage in a test a change occurs, recording your observations alongside the relevant tests. Your answers should include

- · details of colour changes and precipitates formed,
- the identities of gases evolved and details of the test used to identify each gas, **except** tests (a)(iv), (vi) and (vii).

If there is no observable change, write **no observable change**.

Table 4.1

	test	observations
(i)	Using a measuring cylinder, add 2 cm ³ of FA 5 into a boiling tube. Add aqueous sodium hydroxide slowly, with shaking, until no further change is seen.	
	Filter the mixture. Retain the filtrate for tests (ii) and (iii). Retain the residue for test (iv).	
(ii)	Add 1 cm depth of the filtrate from test (i) into a boiling tube. Add one piece of aluminium foil and warm gently.	
(iii)	Add 1 cm depth of filtrate from test (i) into a test-tube. Add an equal volume of barium nitrate followed by excess dilute nitric acid.	
(iv)	Add 1 cm ³ of hydrogen peroxide to the residue from test (i).	

	test	observations
(v)	Using a measuring cylinder, transfer 2 cm ³ of FA 6 into a boiling tube.	
	Add 10 cm ³ of dilute sulfuric acid and swirl the mixture gently.	
	The resultant yellow solution is FA 7, which is an acidified solution of VO_2^+ .	
(vi)	Using a spatula, add a very small quantity of zinc powder to the boiling tube containing FA 7 solution from test (v) . Swirl the mixture gently and record your observations.	
	Continue to add more zinc powder in small quantities with swirling, until no further colour change is observed. Record all colour changes observed.	
	Filter the mixture and retain the filtrate for test (vii).	
(vii)	To 1 cm depth of the filtrate from test (vi), add an equal volume of aqueous hydrogen peroxide.	

[7]

(1)	present in FA 5 .
	cations
	anions[2]
	(1)

(ii) Table 4.2 shows some standard electrode potentials.

Table 4.2

electrode reaction	<i>E</i> [⊕] /V
$VO_2^+ + 2H^+ + e^- \rightleftharpoons VO^{2+} + H_2O$	+1.00
$VO^{2+} + 2H^+ + e^- \rightleftharpoons V^{3+} + H_2O$	+0.34
$V^{3+} + e^- \rightleftharpoons V^{2+}$	-0.26
V ²⁺ + 2e ⁻ ⇌ V	-1.20
Zn ²⁺ + 2e⁻ ⇌ Zn	-0.76

State a (a)(vi).	nd	expla	in w	/hich	ion	is	resp	ons	ible	for	the	final	colo	ur c	of the	e filt	rate	in	test
																			[2]

(c) A student performed an analysis using aqueous cobalt(II) chloride, $CoCl_2$. The test and observations are recorded in Table 4.3.

Table 4.3

test	observations
To 1 cm depth of $CoCl_2(aq)$ in a test-tube, add aqueous ammonia dropwise, with shaking, until no further change is seen.	Blue-green ppt formed is partially soluble in excess NH ₃ (aq) to form a pale brown solution.
Then add about 1 cm depth of aqueous hydrogen peroxide and shake the mixture thoroughly.	Blue-green ppt turned brown. Effervescence observed. Gas relights glowing splint. Gas is O ₂ .

(i)	Aqueous $CoCl_2$ contains $[Co(H_2O)_6]^{2+}$ ions.
	Write an equation for the formation of the blue-green precipitate.
	[1]

(ii)	Using your answer in (c)(i) , account for the partial dissolution of the blue-green precipitate to form a pale brown solution when aqueous ammonia was added in excess.
	[2]
(iii)	With reference to changes in oxidation state, explain the formation of the brown precipitate upon the addition of aqueous hydrogen peroxide.
	[1]
	[Total: 15]

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Qualitative Analysis Notes [ppt. = precipitate]

(a) Reactions of aqueous cations

antin n	reaction with							
cation	NaOH(aq)	NH₃(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., 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						

(b) Reactions of anions

ion	reaction					
carbonate, CO ₃ ²⁻	CO ₂ liberated by dilute acids					
chloride, C <i>l</i> ⁻ (aq)	gives white ppt. with Ag ⁺ (aq) (soluble in NH ₃ (aq))					
bromide, Br ⁻ (aq)	gives pale 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)	NH₃ 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)					
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 dilute strong acids)					

(c) Tests for gases

gas	test and test result
ammonia, NH₃	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 aqueous acidified potassium manganate(VII) from purple to colourless

(d) Colour of halogens

halogen	colour of element	colour in aqueous solution	colour in hexane
chlorine, Cl_2	greenish yellow gas	pale yellow	pale yellow
bromine, Br ₂	reddish brown gas / liquid	orange	orange-red
iodine, I ₂	black solid / purple gas	brown	purple