ANDERSON SERANGOON JUNIOR COLLEGE

2022 JC 2 PRELIMINARY EXAMINATION

NAME:	()	CLASS: 22 /

CHEMISTRY

Paper 4 Practical

9729/04 25 August 2022 2 hours 30 minutes

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your name, class and index number on all the work you hand in.

Give details of the practical shift and laboratory where appropriate, in the boxes provided. Write in dark blue or black pen.

You may use a 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.

Quantitative Analysis Notes are printed on pages 23 and 24.

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	/ 14
2	/ 21
3	/ 9
4	/ 11
Total	/ 55

This document consists of 22 printed pages and 2 blank pages.

Answer **all** the questions in the spaces provided.

1 Determination of a value for an enthalpy change of solution by an indirect method

- **FA 1** is solid sodium hydrogencarbonate, NaHCO₃.
- $\label{eq:FA2} \textbf{FA2} \quad \text{is 1.50 mol } dm^{-3} \text{ sulfuric acid, } H_2SO_4.$

Sodium hydrogencarbonate dissolves in water according to equation 1.

equation 1 NaHCO₃(s) + aq \rightarrow Na⁺(aq) + HCO₃⁻(aq) ΔH_1

Both solid and aqueous sodium hydrogencarbonate react with sulfuric acid.

equation 2 2NaHCO₃(s) + H₂SO₄(aq) \rightarrow Na₂SO₄(aq) + 2H₂O(l) + 2CO₂(g) ΔH_2

equation 3 2NaHCO₃(aq) + H₂SO₄(aq) \rightarrow Na₂SO₄(aq) + 2H₂O(I) + 2CO₂(g) Δ H₃

In this question, you will perform an experiment to determine a value for ΔH_2 . You will use data provided to calculate ΔH_3 and hence a value for ΔH_1 .

(a) Determination of the molar enthalpy change of reaction, ΔH_2

In this experiment, you will determine the maximum temperature change when a known mass of solid sodium hydrogencarbonate, **FA 1**, reacts with dilute sulfuric acid, **FA 2**.

In an appropriate format in the space provided on page 3, 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.

Procedure

- 1. Weigh the capped bottle containing **FA 1**. Record this mass.
- 2. Place one polystyrene cup inside another polystyrene cup and place both in a glass beaker.
- 3. Use a 25 cm³ measuring cylinder to transfer 25 cm³ of **FA 2** into the polystyrene cup.
- 4. Stir the **FA 2** in the polystyrene cup with the thermometer. Read and record its initial temperature, T_{i} .
- 5. Slip the thermometer through the lid. Carefully transfer all the solid **FA 1** in the bottle to the **FA 2** in the polystyrene cup, in small portions, to avoid too much frothing. Secure the lid onto the cup.
- 6. Use the thermometer to stir the mixture. Observe the temperature until it shows the maximum change from the initial temperature. Record this temperature, T_m .
- 7. Reweigh the empty capped bottle. Record this mass.

Determine the maximum temperature change, ΔT , and the mass of **FA 1** used.

Results

(b) In the following calculations, you should assume that the specific heat capacity of the solution is 4.18 J $g^{-1} K^{-1}$, and the density of the solution is 1.00 g cm⁻³.

3

(i) Use your results from 1(a) to calculate the heat change for your experiment.

heat change =[1]

(ii) Hence, determine a value for ΔH_2 . The sulfuric acid is in excess.

Include the sign of ΔH_2 in your answer.

[A_r: H, 1.0; C, 12.0; O, 16.0; Na, 23.0]

 $\Delta H_2 = \dots \qquad [1]$

The results of an experiment where a solution of 0.690 mol dm⁻³ aqueous sodium hydrogencarbonate, NaHCO₃(aq) was reacted completely with an excess of dilute sulfuric acid, **FA 2**, are shown in Table 1.1.

Table 1.1

volume of NaHCO ₃ (aq) used / cm ³	50.0
initial temperature of NaHCO3(aq) / °C	27.6
volume of FA 2 used / cm ³	25.0
initial temperature of FA 2 / °C	31.2
minimum temperature / °C	28.4

(iii) Use the results given in Table 1.1 and the formula below to calculate the weighted average initial temperature, T_{av} , of the reaction mixture.

The formula for T_{av} is given as

 $T_{av} = \frac{(\text{vol. of FA 2} \times \text{initial temp. of FA 2}) + (\text{vol. of NaHCO}_3 \times \text{initial temp. of NaHCO}_3)}{\text{total volume of reaction mixture}}$

*T*_{av} =[1]

(iv) Hence, calculate a value for ΔH_3 .

 $\Delta H_3 = \dots \qquad [4]$

(c) Use your answers from 1(b)(ii) and 1(b)(iv) to calculate a value for ΔH_1 for the reaction shown in equation 1.

If you are not able to determine a value for 1(b)(ii) and/or 1(b)(iv), you may use x and y to represent the respective enthalpy changes and proceed with this part of the question.

 $\Delta H_1 = \dots [2]$

[Total: 14]

2 To determine the order of reaction with respect to the concentration of iodine in the iodination of propanone reaction

FA 3 is 1.00 mol dm⁻³ sulfuric acid, H_2SO_4 . **FA 4** is 1.00 mol dm⁻³ propanone, CH_3COCH_3 . **FA 5** is an aqueous solution of iodine, I_2 . **FA 6** is 0.0100 mol dm⁻³ sodium thiosulfate, $Na_2S_2O_3$. **FA 7** is 0.50 mol dm⁻³ sodium hydrogencarbonate, NaHCO₃.

You are also provided with a starch indicator.

The equation in reaction 1 represents the reaction between CH₃COCH₃ and I₂.

reaction 1: $CH_3COCH_3(aq) + I_2(aq) \rightarrow CH_3COCH_2I(aq) + HI(aq)$

This reaction is first order with respect to both CH₃COCH₃ and H⁺ ions.

You are to investigate the order of reaction with respect to I₂.

A reaction mixture containing an acidified solution of CH_3COCH_3 and I_2 is first prepared. At timed intervals, aliquots (portions) of this reaction mixture will be removed and quenched using excess NaHCO₃.

It is **not** essential that you complete the titration of one aliquot before extracting the next one from the reaction mixture. However, you **must** ensure that each aliquot is mixed **immediately** with NaHCO₃.

The remaining amount of I_2 at different times can then be determined by titration against $Na_2S_2O_3$. $Na_2S_2O_3$ reacts with I_2 as shown in the equation in reaction 2.

reaction 2: $2S_2O_3^{2-}(aq) + I_2(aq) \rightarrow 2I^{-}(aq) + S_4O_6^{2-}(aq)$

The required order of reaction can be obtained by graphical analysis of your results.

(a) Preparing and titration of the reaction mixture

Notes:

You will perform each titration **once** only. Great care must be taken that you do not exceed the end-point.

Once you have started the stopwatch, it must continue running for the duration of the experiment. You must **not** stop the stopwatch until you have finished this experiment.

You should aim to transfer your first aliquot approximately four minutes after starting the reaction.

You should aim **not** to exceed a maximum reaction time of 20 minutes for this experiment.

In an appropriate format in the space provided on page 8, prepare a table in which to record for each aliquot

- the time of transfer, *t*, in minutes and seconds,
- the decimal time, t_d , in minutes, to 0.1 min, for example, if t = 4 min 33 s then $t_d = 4 \text{ min } + 33/60 \text{ min} = 4.6 \text{ min}$,
- the burette readings and the volume of **FA 6** added.

Question 2 continues on the next page.

Safety:

Propanone is flammable. Transfer your titrated solutions into the **waste** bottle for later disposal. Keep this bottle stoppered when not in use.

Keep the conical flask labelled **reaction mixture** stoppered except when removing aliquots.

- 1. Fill a burette with **FA 6**.
- 2. Using a 25 cm³ measuring cylinder, add the following to a 100 cm³ beaker.
 - 25.0 cm³ of **FA 3**
 - 25.0 cm³ of **FA 4**
- 3. Using a 100 cm³ measuring cylinder, transfer 50.0 cm³ of **FA 5** into the 250 cm³ conical flask, labelled **reaction mixture.**
- 4. Pour the contents of the 100 cm³ beaker into this 250 cm³ conical flask. Start the stopwatch, **insert the stopper** and swirl the mixture thoroughly to mix its contents.
- 5. Using a 10 cm³ measuring cylinder, measure 10.0 cm³ of **FA 7** into a second conical flask.
- 6. At approximately 4 minutes, using a 10.0 cm³ pipette, remove a 10.0 cm³ aliquot of the reaction mixture. **Immediately** transfer this aliquot into the second conical flask containing **FA 7** and swirl the mixture thoroughly.

Note the time of transfer, *t*, to the nearest second, when half of the reaction mixture has been dispensed from the pipette. Replace the stopper in the reaction flask.

- 7. Titrate the iodine in the second conical flask with **FA 6**. When the colour of the solution turns pale yellow, add about 1 cm³ of starch indicator. The solution will turn blue–black. The end–point is reached when the blue–black colour just disappears. Record your results.
- 8. Empty the contents of this conical flask into the waste bottle. Wash this conical flask thoroughly with water.
- 9. Repeat steps 5 to 8 until a total of **five** aliquots have been titrated and their results recorded.

Results

(b) (i) On the grid in Fig. 2.1, plot a graph of the volume of **FA 6** added, on the *y*-axis, against decimal time, t_d , on the *x*-axis.

Draw the most appropriate best–fit line taking into account all of your plotted points. Extrapolate (extend) this line to $t_d = 0.0$ min.



Fig. 2.1

[4]

(ii) Deduce the order of reaction with respect to the $[I_2]$ in reaction 1. Explain your answer.

(iii) Calculate the gradient of the line, showing your working clearly on the graph.

(iv) The y-intercept of your line gives the volume of sodium thiosulfate required to completely react with the iodine present if an aliquot is taken at $t_d = 0.0$ min.

Read from your graph and record this volume of FA 6, V_{max} . Use this value to calculate the concentration of iodine in FA 5.

volume of **FA 6**, *V*_{max} =cm³

concentration of iodine in **FA 5** =mol dm^{-3} [3]

(v) Use your answer to part 2(b)(iii) together with your value for V_{max} to estimate t_{max} , the time at which all the iodine in your reaction mixture would have reacted completely.

*t*_{max} =min [1]

(c) Use the information provided on page 6 and your answer to **2(b)(ii)**, write an expression for the rate equation for reaction 1. Include the units for rate in your answer.

Rate =[1]

(d) A student performed a modified experiment of the one that you performed in 2(a). Instead of using 25.0 cm³ of FA 4, the student used a 25.0 cm³ solution prepared from equal volumes of FA 4 and water.

On the axes in Fig. 2.2, sketch the graph you have obtained in **2(b)(i)** and the graph you would expect to obtain from the modified experiment conducted by the student. Label both graphs clearly.

Explain your answer.

Fig. 2.2

	expla	anation
		[2]
(e)	In ste flask	ep 6 of the experimental procedure, the aliquot is transferred into the second conical containing FA 7 .
	(i)	Explain why it is necessary to add FA 7 , and how the titre values will be affected with the omission of FA 7 .
		[1]
	(ii)	A small temperature change is observed upon the transfer of the aliquot into the second conical flask. Suggest, with a reason, how the temperature would change.
		[1]



(f) The following steps represent a possible mechanism for reaction 1.

3 Investigation of the chemistry of some vanadium ions

FA 8 contains vanadate(V) ions, VO_3^- , of concentration 0.50 mol dm⁻³.

FA 9 is a metal.

FA 10 is a solution made by reacting solid FA 9 with dilute sulfuric acid.

You are also provided with **FA 3**, 1.00 mol dm⁻³ of sulfuric acid, H₂SO₄ used in **Q2**.

Ammonium vanadate(V), NH₄VO₃, is a crystalline solid that is slightly yellow in colour. It has a relatively low solubility in water at room temperature.

You are provided with a solution, **FA 8**, produced by warming solid NH_4VO_3 with aqueous sodium hydroxide. In this reaction, a more soluble salt is formed and the anion is unchanged.

(a) Write an equation for the reaction between ammonium vanadate(V) and sodium hydroxide to produce **FA 8**.

Describe a simple test that can be performed to confirm the presence of one of the products of the reaction between ammonium vanadate(V) and sodium hydroxide.

equation	
test	
	[2]

(b) When FA 8 is acidified and reacted with FA 9, a series of colour changes can be observed during the reaction.

You will carry out tests to study the reactions of acidified **FA 8** with **FA 9**. Record your observations in Table 3.1.

In these reactions, you do **not** need to test for any gas that is evolved.

	tests	observations
(i)	Using a 50 cm ³ measuring cylinder, measure about 10 cm ³ of FA 8 into a 250 cm ³ beaker.	
	Using the same measuring cylinder, measure about 40 cm ³ of FA 3 to the same beaker.	
	The resulting solution obtained is solution W , which will be used in 3(b)(ii) and 3(c) .	
(ii)	Transfer about 30 cm ³ of solution W into a 100 cm ³ conical flask, using the same measuring cylinder used in 3(b)(i) .	
	FA 9 provided, a small spatula measure each time, into the conical flask. Swirl after each addition.	
	You may then leave the mixture to stand, swirling the flask from time to time and observe the solution until no further changes are seen.	
While	e you are waiting, continue with th	ne test in 3(c).

Table 3.1

[2]

(c) Reaction of solution **W** with tin powder gives different observations from those observed with **FA 9**. Record your observations in Table 3.2.

Та	ble	3.2

test	observations
Transfer about 10 cm ³ of solution W into a boiling tube using the same measuring cylinder used in 3(b)(i) .	
Add all the tin powder in the weighing bottle into the boiling tube and shake the mixture.	
Gently warm the boiling tube. You should not allow the mixture in the boiling tube to boil or heat to dryness.	
To view the colour of the solution clearly, you may find it necessary, from time to time, to allow the tin to settle.	
Observe the mixture until no further changes are seen.	
	[1]

(d) (i) You will perform tests to identify the metal cation present in **FA 10** and hence the identity of **FA 9**.

Carry out the following tests. Record your observations in Table 3.3.

tests	observations
1 To about 1 cm depth of FA 10 in a test–tube, add aqueous sodium hydroxide until no further changes are observed.	
2 To about 1 cm depth of FA 10 in a test–tube, add aqueous ammonia until no further changes are observed.	
	כו

Table 3.3

[1]

(iii) Write equations to explain the recorded observations for test **2** in Table 3.3.

(ii) Identity of FA 9:

.....

.....[1]

[Total: 9]

4 Planning

Calcium iodate(V), Ca(IO₃)₂, has low solubility of 6.2×10^{-3} mol dm⁻³ in water at 20 °C. When calcium iodate (V) is added to water, the following equilibrium is established.

$$Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2IO_3^{-}(aq)$$

You are to plan an experiment to determine the solubility product, K_{sp} , of Ca(IO₃)₂(s) by first preparing a saturated solution of Ca(IO₃)₂. A saturated solution is one in which no more solid can dissolve at a particular temperature. In a saturated solution with undissolved Ca(IO₃)₂(s), the above equilibrium is established after about one hour.

The mixture is then filtered and the amount of iodate(V) ions, IO_3^- , in the filtrate is determined as described below.

10 cm³ of potassium iodide (in excess) and 10 cm³ of sulfuric acid are added to 25 cm³ of the filtrate containing IO_3^- ions. Iodine is liberated, as shown in equation 4.

equation 4 $IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow 3I_2(aq) + 3H_2O(I)$

The liberated iodine is then titrated with a standard solution of sodium thiosulfate, as shown in equation 5.

equation 5 $2S_2O_3^{2-}(aq) + I_2(aq) \rightarrow 2I^{-}(aq) + S_4O_6^{2-}(aq)$

(a) Calculate the minimum mass of $Ca(IO_3)_2(s)$ that is required to prepare a saturated solution of $Ca(IO_3)_2$ in 100 cm³ of deionised water at 20°C.

[molar mass of $Ca(IO_3)_2 = 389.9 \text{ g mol}^{-1}$]

(b) Plan a procedure to prepare a saturated solution of $Ca(IO_3)_2$ in 100 cm³ of deionised water **and** an investigation to determine the solubility product of the salt at 20°C.

You may assume that you are provided with:

- deionised water
- solid calcium iodate(V), Ca(IO₃)₂
- 0.100 mol dm⁻³ sodium thiosulfate, Na₂S₂O₃
- aqueous potassium iodide, KI
- 1.00 mol dm⁻³ dilute sulfuric acid, H₂SO₄
- starch indicator
- filter funnel and filter paper
- the apparatus and equipment normally found in a school or college laboratory.

In your plan you should include brief details of:

- the apparatus you would use
- the quantities you would use
- the procedure you would follow
- the measurements you would make
- how you would ensure that an **accurate** and **reliable** value of K_{sp} is obtained.

 • • •

19

.....

[6]

(ii) Outline how you would use your titration results to calculate the K_{sp} of Ca(IO₃)₂. You may wish to use V dm³ as your average titre volume.

[3]

[Total: 11]

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Qualitative Analysis Notes [ppt. = precipitate]

(a) Reactions of aqueous cations

ootion	reaction with		
cation	NaOH(aq)	NH ₃ (aq)	
aluminium, A <i>l</i> ³+(aq)	white ppt. soluble in excess	white ppt. insoluble in excess	
ammonium, NH₄⁺(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), Cr³⁺(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, 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, Zn ²⁺ (aq)	white ppt. soluble in excess	white ppt. soluble in excess	

(b) Reactions of anions

anion	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 NH₃(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, NO₃⁻(aq)	NH ₃ liberated on heating with OH ⁻ (aq) and A <i>l</i> foil	
nitrite, NO₂⁻(aq)	NH ₃ liberated on heating with OH ⁻ (aq) and A <i>l</i> foil NO liberated by dilute acids (colourless NO \rightarrow (pale) brown NO ₂ in air)	
sulfate, SO₄²⁻(aq)	gives white ppt. with Ba ²⁺ (aq) (insoluble in excess dilute strong acids)	
sulfite, SO ₃ ^{2–} (aq)	SO ₂ liberated on warming 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