ANDERSON SERANGOON JUNIOR COLLEGE

2020 JC 2 PRELIMINARY EXAMINATION

NAME:____________________________ (   ) **CLASS:** 20 /____

CHEMISTRY **9729/04**

Paper 4 Practical

27 August 2020

2 hours 30 minutes

Candidates answer on the Question Paper.

Additional Materials: As listed in the Confidential Instructions

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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 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 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	**/ 14**
2	**/ 18**
3	**/ 11**
4	**/ 12**
Total	**/ 55**

This document consists of **20** printed pages.

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

1 Determination of the oxidation number of iodine in a compound

In this experiment you will determine the oxidation number of iodine in one of its compounds by titration.

You are provided with the following:

FA 1 is a 0.0197 mol dm^{-3} solution of the iodine–containing compound.
FA 2 is dilute sulfuric acid, H_2SO_4.
FA 3 is aqueous potassium iodide, KI.
FA 4 is 0.105 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$. (This solution is also used in **Question 2**.)
starch indicator

FA 1 reacts with excess acidified potassium iodide to produce iodine, I_2. This iodine is then titrated with aqueous sodium thiosulfate using starch indicator.

(a) Titration of iodine formed against FA 4

1. Fill the burette with **FA 4**. (You will use the **same** burette for **Question 2**.)
2. Pipette 25.0 cm^3 of **FA 1** into a conical flask.
3. Using a 25 cm^3 measuring cylinder, add 10 cm^3 of **FA 2** to the same conical flask.
4. Using the same measuring cylinder, add 20 cm^3 of **FA 3** to the mixture in the conical flask. The mixture will now be a red–brown colour, due to iodine produced.
5. Add **FA 4** from the burette until the mixture becomes light brown.
6. Then add 10 drops of starch indicator. The mixture will change to a dark blue colour.
7. Continue titrating until the mixture becomes colourless. This is the end–point.
8. Carry out as many accurate titrations as you think necessary to obtain consistent results.
9. Record in a suitable form below all of your burette readings and the volume of **FA 4** added in each accurate titration.

(i) Results

[5]

(ii) From your accurate titration results, obtain a suitable value for the volume of **FA 4** to be used in your calculations. Show clearly how you obtained this value.

The iodine produced required …………………………….. cm^3 of **FA 4**. [1]

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

(i) Calculate the number of moles of sodium thiosulfate in the volume of **FA 4** calculated in **(a)(ii)**.

moles of $Na_2S_2O_3$ = …………………………….. [1]

(ii) The equation for the reaction of iodine with sodium thiosulfate is shown.

$$I_2(aq) + 2Na_2S_2O_3(aq) \rightarrow Na_2S_4O_6(aq) + 2NaI(aq)$$

Calculate the number of moles of iodine that reacted with the sodium thiosulfate calculated in **(b)(i)**.

moles of I_2 = …………………………….. [1]

(iii) Use the information on page 2 to calculate the number of moles of iodine–containing compound in the 25.0 cm^3 of **FA 1** used in each titration.

moles of iodine–containing compound **FA 1** = …………………………….. [1]

(iv) Use your answers to **(b)(ii)** and **(b)(iii)** to calculate the number of moles of iodine produced when 1 mole of the iodine–containing compound in **FA 1** reacts with excess **FA 3**.
Give your answer as an integer.

moles of I_2 = …………………………….. [1]

(v) The anion in **FA 1** is IO_x^- where x is the number of oxygen atoms present in the formula.

Use your answer to **(b)(iv)** to balance the ionic equation for the reaction between **FA 1** and **FA 3** under acidic conditions.
Hence deduce the value of x in the formula IO_x^-.

$IO_{....}^-$ + ... I^- + ... H^+ → ...... I_2 + ... H_2O

x = …………………………….. [2]

(vi) Calculate the oxidation state of iodine in **FA 1**.
(If you were unable to calculate x in **(b)(v)**, assume that x = 4.)

oxidation state of iodine = …………………………….. [2]

[Total: 14]

Question 2 starts on the next page.

2 To investigate the effect of concentration changes on the rate of a reaction.

FA 5 is 0.0500 mol dm^{-3} acidified iron(III) chloride, $FeCl_3$.
FA 6 is 0.0500 mol dm^{-3} potassium iodide, KI.

Iron(III) ions oxidise iodide ions, I^-, to iodine, I_2 as shown in equation 1.

equation 1 $2Fe^{3+}(aq) + 2I^-(aq) \rightarrow 2Fe^{2+}(aq) + I_2(aq)$

In this experiment you will investigate how the rate of this reaction is affected by the concentration of Fe^{3+} ions. To do this you will add thiosulfate ions, $S_2O_3^{2-}$, and starch indicator to a mixture of $Fe^{3+}(aq)$ and $I^-(aq)$. The iodine produced by the reaction reacts immediately with the thiosulfate ions and is reduced back to iodide as shown in equation 2.

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

When all the thiosulfate has reacted, the iodine remaining in solution turns the starch indicator blue–black. The rate of reaction can be determined by timing how long it takes for the reaction mixture to turn blue–black.

(a) Preparation of FA 7 by dilution of FA 4

FA 7 which contains 0.00500 mol dm^{-3} of sodium thiosulfate, $Na_2S_2O_3$, can be prepared by diluting **FA 4** from **Question 1** in a 250 cm^3 graduated flask.

(i) The concentration of sodium thiosulfate, $Na_2S_2O_3$, in **FA 4** is 0.105 mol dm^{-3}.

Show, by calculation, that the volume of **FA 4** needed to prepare this diluted solution in the 250 cm^3 graduated flask is 11.90 cm^3.

[1]

(ii) Using the burette from **Question 1**, measure 11.90 cm^3 of **FA 4** into a 250 cm^3 graduated flask labelled **FA 7**. Make up to the mark with distilled water.

Record the volume of **FA 4** added to the flask in the space provided.

volume of **FA 4** added = …………………………….. cm^3

(b) Experiment 1

1. Fill the burette labelled **FA 5** with **FA 5**.
2. Run 20.00 cm^3 of **FA 5** into a 100 cm^3 beaker.
3. Using the **same** measuring cylinder, add the following to the **second** 100 cm^3 beaker:
   - 10 cm^3 of **FA 6**
   - 20 cm^3 of **FA 7**
   - 10 cm^3 of starch solution
4. Add the contents of the first beaker to the second beaker and start timing immediately.
5. Stir the mixture once and place the beaker on a white tile.
6. Stop timing as soon as the solution turns blue–black. Ignore any colour changes that occur before the intense blue–black colouration.
7. Record this reaction time, to the nearest second, in the space provided on page 8.
8. Rinse both beakers and shake dry. Rinse and dry the glass rod.

Experiment 2

1. Run 10.00 cm^3 of **FA 5** into a 100 cm^3 beaker.
2. Using another measuring cylinder, add 10.0 cm^3 of distilled water into the beaker containing **FA 5**.
3. Using the measuring cylinder from Experiment 1, add the following to the **second** 100 cm^3 beaker:
   - 10 cm^3 of **FA 6**
   - 20 cm^3 of **FA 7**
   - 10 cm^3 of starch solution
4. Add the contents of the first beaker to the second beaker and start timing immediately.
5. Stir the mixture once and place the beaker on a white tile.
6. Stop timing as soon as the solution turns blue–black. Ignore any colour changes that occur before the intense blue–black colouration.
7. Record this reaction time, to the nearest second, in the space provided on page 8.
8. Rinse both beakers and shake dry. Rinse and dry the glass rod.

Experiment 3 to 5

Carry out **three** further experiments to investigate how the reaction time changes with different volumes of **FA 5**.

Remember that the combined volume of **FA 5** and distilled water must always be 20.00 cm^3.

Do **not** carry out an experiment using 15.00 cm^3 of **FA 5**.
Do **not** use a volume of **FA 5** that is less than 5.00 cm^3.

Record all your results in a single table. You should include the volume of **FA 5**, the volume of distilled water and the reaction time.

The relative rate for the reaction is given by the following expression.

$$\text{relative rate} = \frac{1000}{\text{reaction time in seconds}}$$

Use this expression to calculate the relative rate for each of your experiments and record the values, to 3 significant figures, in your results table.

(i) Results

[5]

(ii) Plot a graph of relative rate against the volume of **FA 5** used on the grid in Fig 2.1. Include the origin in your plot. Draw a best–fit line. Label any points you consider anomalous.

Fig 2.1

[3]

(c) From your graph, what conclusion can you make about the relationship between the relative rate for the reaction and the volume of **FA 5** used? Explain your answer.

…………………………………………………………………………………………………

…………………………………………………………………………………………………

…………………………………………………………………………………………………

…………………………………………………………………………………………………[2]

(d) Explain why a small and fixed amount of sodium thiosulfate is required for this reaction.

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…………………………………………………………………………………………………

…………………………………………………………………………………………………

…………………………………………………………………………………………………[2]

(e) A student carried out the same experiment but used 15.00 cm^3 of **FA 5**. The student recorded a value for the reaction time of 28 s.

(i) Use your graph to determine the expected reaction time, to the nearest second, you would have to record if you had carried out an experiment using 15.00 cm^3 of **FA 5**.

Show the construction lines on your graph and show your working in the calculation.

expected reaction time = …………………………….. s [2]

(ii) Calculate the difference between your value and that of the student and express it as a percentage of the student's value.

percentage difference = …………………………….. % [1]

(f) Kinetics studies have shown that the reaction in equation 1 is second order with respect to iodide ions and third order overall.

Steps 1 to 3 represent a possible mechanism of the reaction.

step 1 $Fe^{3+} + I^- \rightleftharpoons [FeI]^{2+}$

step 2 $[FeI]^{2+} + I^- \rightarrow Fe^{2+} + I_2^-$

step 3 $2I_2^- \rightarrow I_2 + 2I^-$

(i) State which step is the rate–determining step.

……………………………………………………………………………………………..[1]

(ii) The species in the steps shown have various roles. They can be reactants, products, catalysts or intermediates.

Suggest, with a reason, the role of $[FeI]^{2+}$ ions in the mechanism shown above.

………………………………………………………………………………………………..

………………………………………………………………………………………………..

……………………………………………………………………………………………..[1]

[Total: 18]

3 Investigation of some inorganic reactions

At each stage of any test you are to record details of the following:

- colour changes seen;
- the formation of any precipitate and its solubility in an excess of the reagent added;
- the formation of any gas and its identification by a suitable test.

You should indicate clearly at what stage in a test a change occurs. If there is no observable change, write **no observable change**.

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured. Rinse and reuse test–tubes, where possible.

No additional tests for ions present should be attempted.

(a) **FA 8** is a solution containing two cations. The two cations have **different** oxidation states.

(i) Carry out the following tests and record your observations in Table 3.1.

Table 3.1

test	observations
To a portion of the solution **FA 8**, add aqueous sodium hydroxide, until no further change is seen.	
Filter the mixture and add dilute sulfuric acid to the filtrate.	
Wash the residue by pouring distilled water through it. Discard the washings and then pour dilute aqueous ammonia through the residue.	

[3]

(ii) Consider your observations in Table 3.1.

The cations in **FA 8** are ……………….. and ………………… [2]

(b) **FA 9** is a solution which contains two of the following anions: carbonate, chloride, bromide, iodide, sulfate(VI).

You are to introduce dilute nitric acid into **FA 9** prior to conducting further tests on the suspected anions present.

(i) To 2 cm depth of **FA 9** in a test–tube, add dilute nitric acid.

Record your observations and deduce the identity of the anion present.

Keep this solution for **(b)(iii)**.

observations ……………………………………………………………………………………

……………………………………………………………………………………………………

identity of anion …………………………………………………………………………[1]

(ii) By reference to the Qualitative Analysis Notes on pages 19 and 20 and using only the bench reagents provided, describe **two different** tests which will allow you to identify the other anion in **FA 9**.

test 1 ……………………………………………………………………………………………

……………………………………………………………………………………………………

……………………………………………………………………………………………………

test 2 ……………………………………………………………………………………………

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……………………………………………………………………………………………………

[2]

(iii) Perform the tests you described in **(b)(ii)** using the solution obtained from **(b)(i).**

Record your observations and hence deduce the identity of the other anion present in **FA 9**.

test 1 ……………………………………………………………………………………………

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test 2 ……………………………………………………………………………………………

……………………………………………………………………………………………………

identity of the other anion ………………………………………………………………...

[3]

[Total: 11]

4 Planning

When a monobasic acid, HX(aq) is mixed with aqueous potassium hydroxide, KOH(aq), the reaction releases heat causing a rise in the temperature of the solution.

$$HX(aq) + KOH(aq) \rightarrow KX(aq) + H_2O(l) \qquad \Delta H_1 < 0$$

A series of experiments can be performed where increasing volumes of HX(aq) and decreasing volumes of KOH(aq) are mixed and the temperature rise, ΔT, for each experiment is determined.

In each of the experiments using different volumes of HX(aq) and KOH(aq), the total volume has to be kept constant. Since the total volume of mixture remains the same, the temperature rise, ΔT, is a direct measure of the heat given out by the reaction. The maximum amount of heat is evolved when all the acid present is exactly neutralised by all the alkali present.

Plotting a graph of ΔT against the volume of HX used will give 2 straight lines of best–fit.

Extrapolation of the two straight lines will produce a point of intersection from which the concentration of HX(aq) and the enthalpy change, ΔH_1, of the exothermic reaction between HX(aq) and KOH(aq) can be determined.

The volume of HX(aq) used should be at least 10.00 cm^3 and the total volume of the reaction mixture should be kept constant at 50.00 cm^3 for all experiments.

(a) Using the information given, you are required to write a plan to determine the concentration of HX(aq) and the enthalpy change, ΔH_1, of the exothermic reaction between HX(aq) and KOH(aq).

You may assume that you are provided with:

- strong monobasic acid, HX(aq), of unknown concentration,
- 2.0 mol dm^{-3} potassium hydroxide, KOH(aq),
- Styrofoam cups and lids,
- the 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, including suggested volumes of KOH(aq) and HX(aq), in a suitable table,
- the procedure you would follow,
- the measurements you would make,
- the tabulation of the results.

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……………………………………………………………………………………………………[5]

(b) Sketch on Fig 4.1 the graph you would expect to obtain from your results.

Indicate clearly on your sketch how you would determine:

- $\boldsymbol{V}_{\text{neut}}$, the volume of HX(aq) needed to just completely neutralise (50 – $\boldsymbol{V}_{\text{neut}}$) cm^3 of KOH(aq).
- $\Delta\boldsymbol{T}_{\text{max}}$, the maximum temperature rise when stoichiometric amount of HX(aq) and KOH(aq) reacted.

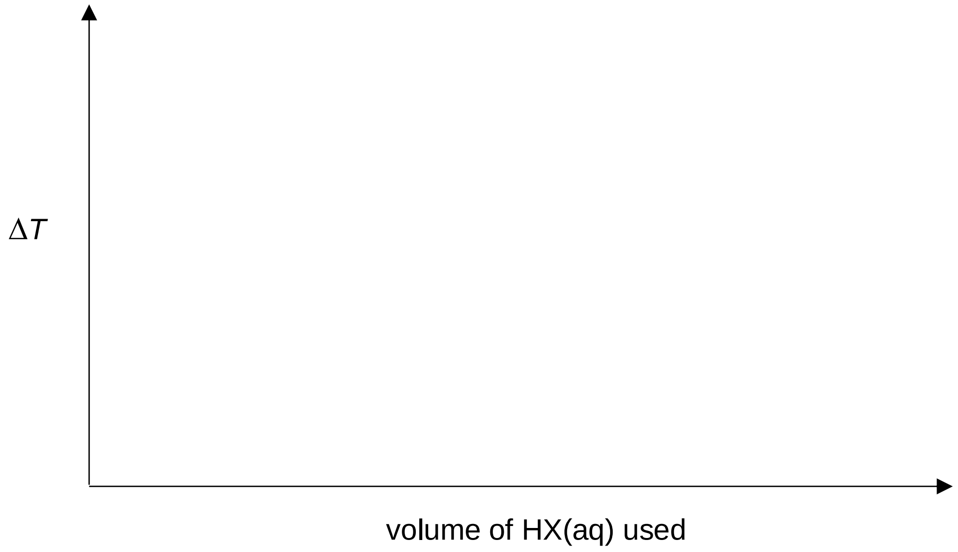

Fig 4.1

[2]

(c) Outline how you would use your answers from **(b)** to determine

(i) the concentration, in mol dm^{-3}, of HX(aq).

You may show your results in terms of $\boldsymbol{V}_{\text{neut}}$.

[1]

(ii) the enthalpy change of reaction, ΔH_1, for this reaction.

$$HX(aq) + KOH(aq) \rightarrow KX(aq) + H_2O(l) \qquad \Delta H_1$$

Express your answers in terms of ΔT_{max}

You should assume that the specific heat capacity of the final solution is 4.18 J g^{-1} K^{-1} and its density is 1.00 g cm^{-3}.

[2]

(d) A similar exothermic reaction will take place between a monobasic **weak** acid, HY(aq) and KOH(aq).

Explain how each of the following

- $\boldsymbol{V}_{\text{neut}}$**(HY)**, the volume of HY(aq) needed to just completely neutralise (50 – $\boldsymbol{V}_{\text{neut}}$**(HY)**) cm^3 of KOH(aq).

- $\Delta \boldsymbol{T}_{\text{max}}$**(HY)**, the maximum temperature rise when stoichiometric amount of HY(aq) and KOH(aq) reacted.

would differ from the values obtained in **(b)** when the same experiment you have described in **(a)** is repeated using a monobasic weak acid, HY(aq) of the same concentration.

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....

....[2]

[Total: 12]

Qualitative Analysis Notes

[ppt. = precipitate]

(a) Reactions of aqueous cations

cation	***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. 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, $Cl^-(aq)$	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, $NO_3^-(aq)$	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 → (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 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, 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 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