NATIONAL JUNIOR COLLEGE
SH 2 Year – End Practical Examination
Higher 2

CANDIDATE NAME	

SUBJECT CLASS		REGISTRATION NUMBER	

CHEMISTRY 9729/04

Paper 4 Practical **Tuesday 20 August 2019**

Candidate answer on the Question paper. **2 hours 30 minutes**

READ THE INSTRUCTIONS FIRST
Write your identification number and name. Circle 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, highlighters, 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, submit the question paper.
The number of marks is given in brackets [ ] at the end of each question or part question.

Shift			
1	**2**	**3**	
Laboratory			
CM41	**CM42**	**CM43**	**CM44**
PH31	**PH32**	**PH33**	
BI23	**BI24**		

For Examiner's Use	
1	**/ 12**
2	**/ 21**
3	**/ 14**
4	**/ 8**
Total	**/ 55**

This document consists of **19** printed pages and **1** blank page.

1 Determination of the solubility product of calcium iodate(V)

When solid calcium iodate(V) is added to water to form a saturated solution, an equilibrium between the undissolved salt and its aqueous ions is established.

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

The solubility product, K_{sp} , of calcium iodate(V) is expressed as follows.

$$K_{sp} = [Ca^{2+}(aq)]\,[IO_3^-(aq)]^2$$

The solubility product can be found by determining the equilibrium concentration of IO_3^- ions in a saturated solution of calcium iodate(V) through a redox titration.

Iodate(V) ions, IO_3^-, react with iodide ions according to the following equation.

$$IO_3^-\,(aq) + 5I^-(aq) + 6H^+\,(aq) \longrightarrow 3I_2\,(aq) + 3H_2O\,(l)$$

The iodine produced in this reaction may be titrated against sodium thiosulfate using starch solution as indicator. Thiosulfate ions react with iodine according to the following equation:

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

In this question, you will prepare a saturated solution of calcium iodate(V) to carry out the redox titration. Using the experimental results, you will determine the solubility product of calcium iodate(V).

You are provided with:
FA 1 is solid calcium iodate(V), $Ca(IO_3)_2$

FA 2 is 0.05 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$

FA 3 is 0.50 mol dm^{-3} potassium iodide, KI

FA 4 is 0.50 mol dm^{-3} sulfuric acid, H_2SO_4

FA 5 is starch indicator

Preparation of a saturated solution of calcium iodate (V), FA 6

(a) Procedure

1. Use a 100 cm^3 measuring cylinder to transfer 80.0 cm^3 of deionised water into a 250 cm^3 beaker.
2. Transfer all **FA 1** from the weighing bottle into the beaker. Stir for 1 to 2 minutes and leave to stand for **5 minutes**. There will be some $Ca(IO_3)_2$ solids left undissolved.
3. Use a 0.2 ^oC division thermometer, record the temperature of the solution in the beaker.
4. To remove the undissolved solids, filter the saturated solution into another clean and **dry** 250 cm^3 beaker using a **dry** filter paper and **dry** funnel. Label this solution as **FA 6**.

Titration

(b) **Procedure**

(i) **1.** Fill a burette with **FA 2**.

2. Pipette 10.0 cm^3 of **FA 6** into a conical flask.

3. Use a 10 cm^3 measuring cylinder to add 5.0 cm^3 of **FA 3**, followed by 5.0 cm^3 of **FA 4** to the same conical flask and swirl quickly.

4. Titrate the iodine in the conical flask with **FA 2** until the brown colour of the iodine becomes pale yellow.

5. Add about 10 drops of starch solution to the flask and continue adding **FA 2** until the blue-black colour just disappears.

6. Repeat the titration as many times as you think necessary to obtain consistent results. Record your titration results in the space below.

Results

[4]

(ii) From your titres, obtain a suitable volume of **FA 2** to be used in your calculations. Show clearly how you obtained this volume.

10.0 cm^3 of **FA 6** produced an amount of iodine which required ……...… cm^3 of **FA 2.**
[1]

(c) Calculations

Show your working and appropriate significant figures in the final answer to **each** part.

(i) Calculate the number of moles of $S_2O_3^{2-}$ in the volume of **FA 2** recorded in **1b(ii)**, and hence determine the molar concentration of IO_3^- ions in **FA 6**.

number of moles of $S_2O_3^{2-}$ =……………………………..

concentration of IO_3^- ions in **FA 6** =……………………………..

[2]

(ii) Determine the molar concentration of Ca^{2+} in **FA 6** and hence, calculate the solubility product, K_{sp}, of calcium iodate(V), stating the units.

concentration of Ca^{2+} ions in **FA 6** = ……………………………..

solubility product of $Ca(IO_3)_2$ = ……………………………..

[2]

(d) Explain why it was necessary to use the **dry** apparatus (e.g. beaker, filter paper and funnel) in the preparation of **FA 6**, and what effect failing to do it would have on the titre values?

………..

………[1]

(e) A literature value for the solubility product of calcium iodate(V) is found to be 6.15×10^{-6} at 20 °C.

State a possible reason for the difference in the K_{sp} value that you have calculated and suggest an improvement that might allow a value closer to the literature value to be obtained.

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………[2]

[Total: 12]

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2 **Determination of the enthalpy change of neutralisation, ΔH_n**

FA 7 is a solution of HC*l* of unknown concentration

FA 8 is a solution of 1.0 mol dm^{-3} of NaOH

In this question, you will carry out a series of experiments where different volumes of **FA 7** and **FA 8** are mixed together.

You will determine the temperature change of the reaction mixture, ΔT, of each experiment and then analyse your results graphically in order to determine the

- concentration of HC*l* in **FA 7**
- maximum temperature change, ΔT_{max}
- value for the enthalpy change of neutralisation, ΔH_n

(a) **Determining the change in temperature for a series of reactions between FA 7 and FA 8**

(i) **Experiment 1**

1. Place a Styrofoam cup inside a second Styrofoam cup held in a 250 cm^3 beaker to prevent it from tipping over.

2. Using a measuring cylinder, transfer 20.0 cm^3 of **FA 7** into the Styrofoam cup.

3. Measure the temperature of **FA 7** in the Styrofoam cup. Record the initial temperature of the solution of **FA 7** as $\mathbf{T_{FA\,7}}$.

4. Rinse and dry the thermometer.

5. Measure 50.0 cm^3 of **FA 8** using another measuring cylinder. Record the initial temperature of the solution of **FA 8** as $\mathbf{T_{FA\,8}}$.

6. Transfer the **FA 8** into the cup containing **FA 7**. Stir and record the maximum temperature of the reaction as $\mathbf{T}_{max}$.

7. Rinse and dry the Styrofoam cup and thermometer.

Experiment 2

Repeat steps 2 to 7 using 50.0 cm^3 of **FA 7** and 20.0 cm^3 **FA 8**.

Experiments 3 to 6

Carry out **four** further experiments to investigate how the $\mathbf{T}_{max}$ changes with different volumes of **FA 7** and **FA 8**. In each case, the total volume of the reaction mixture must be 70.0 cm^3 and the volume of each reagent should not be less than 20.0 cm^3.

In an appropriate format in the space provided on next page, record

- all measurements of volumes used,
- all temperatures measured, $T_{weighted\ initial}$ and ΔT

$\Delta\mathbf{T} = \mathbf{T}_{max} - \mathbf{T}_{weighted\ initial}$

$$\mathbf{T}_{weighted\ initial} = \frac{(\text{Volume of FA 7} \times T_{\text{FA 7}}) + (\text{Volume of FA 8} \times T_{\text{FA 8}})}{\text{Volume of FA 7} + \text{Volume of FA 8}}$$

Results

[4]

(ii) Plot a graph of ΔT (y–axis) against volume of **FA 7** used (x–axis) on the grid in Fig. 2.1 using the data you have obtained in **2a(i)**.

Fig. 2.1 [3]

(iii) Draw two best-fit straight lines, the first should be drawn using the plotted points where ΔT is increasing and the second should be drawn using the plotted points where ΔT is decreasing. Extrapolate (extend) both lines until they intercept.

[1]

(iv) From your graph in Fig. 2.1, determine the maximum temperature change, ΔT_{max}, and the volume, V_{max}, of **FA 7** required to obtain this.

ΔT_{max} = …………………………..^{o}C $V_{\mathbf{max}}$ = ……………………………..cm^3 [2]

(b) **Calculation**
Show your working and appropriate significant figures in the final answer to **each** part.

Using your answers in **2a(iv)**, calculate

(i) the concentration, in mol dm^{-3}, of HC*l* in **FA 7**.

concentration of HC*l* in **FA 7** = …………………………….. [2]

(ii) the heat change for the neutralisation reaction at $\Delta T_{max.}$.

You may assume that the specific heat capacity of the reaction mixture is 4.18 J g^{-1} K^{-1} and that the density is 1.00 g cm^{-3}.

heat change = …………………………….. [1]

(iii) Hence, calculate the enthalpy change of neutralisation, $\Delta \boldsymbol{H_n}$.

$\Delta \boldsymbol{H_n}$ = …………………………….. [1]

(c) In another similar experiment, 1.0 mol dm^{-3} aqueous ammonia was used instead of 1.0 mol dm^{-3} NaOH. A graph of change in temperature, ΔT, against volume of **FA 7** was plotted.

Suggest how would the ΔT_{max} and volume, V_{max}, of **FA 7** from this experiment be compared to your result obtained in **2a(iv)** .

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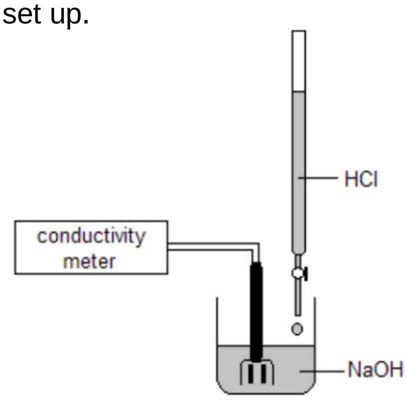
(d) A student employed another method to determine the concentration of the HC*l* in **FA 7** through the use of conductometry. Conductometry is the measurement of the electrolytic conductivity to monitor the progress of a chemical reaction due to ions being produced or reacted away. The equivalence point can then be determined for the acid – base reaction by plotting the conductivity against the volume of the HC*l* added.

$$H^+ + OH^- \longrightarrow H_2O$$

The following apparatus were set up.



The student measured the conductivity of the solution in the beaker with each addition of a small fixed volume of HC*l* and obtained the following graph.

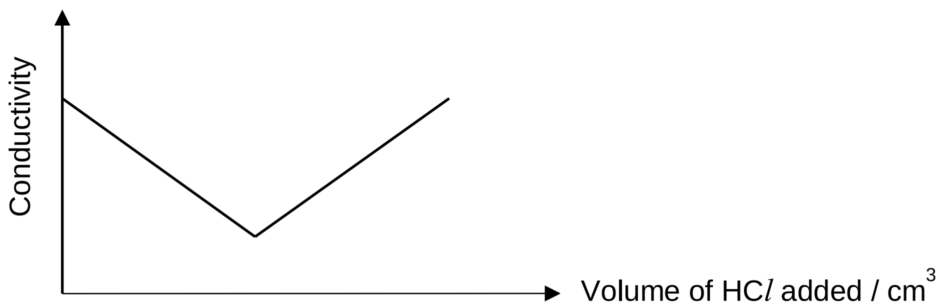


Fig. 2.2

(i) Indicate on the graph (Fig. 2.2), the point which marks the volume of HC*l* needed for complete reaction. [1]

(ii) Explain the shape of the graph in Fig. 2.2.

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(iii) With reference to your experiment in **2(a)**, suggest which method (calorimetry or conductometry) is more accurate for the determination of the concentration of HCl in **FA 7**.

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[Total: 21]

3 Identification of ions

FA 9 is a solution containing a mixture of two salts.

You are to perform the tests described in Table 3.1 and record your observations in the table.

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.

The volumes given below are approximate and should be estimated rather than measured, unless otherwise stated. If there is no observable change, write **no observable change**.

No additional or confirmatory tests for ions present should be attempted.

Table 3.1

	Test	*Observation*
(a)	Place about 3 cm depth of **FA 9** in a test-tube, add equal volume of aqueous sodium hydroxide. Warm the contents in the tube.	
(b)	Filter the mixture from **3(a)** and collect the filtrate in a test-tube. Label the filtrate as **FA 10** and retain it for tests **3(c)** and **3(d)**. Leave the residue in the filter paper and observe it again after 5 minutes.	
(c)	Place 2 cm depth of **FA 10** in a test-tube and add nitric acid until no further change is seen. Label the solution as **FA 11** and retain it for test **3(e)**.	
(d)	Place 1 cm depth of **FA 10** in a test-tube. Add half a spatula of zinc powder. Warm the contents in the tube cautiously.	

	Test	*Observation*
(e)	Place 2 cm depth of **FA 11** in a test-tube, add equal depth of aqueous ammonia. Add excess dilute nitric acid followed by aqueous silver nitrate.	
(f)	Place 1 cm depth of **FA 9** in a test-tube. Add dilute hydrochloric acid followed by aqueous barium chloride.	

[6]

(g) **(i)** Identify the **two** cations and **two** anions present in **FA 9**.

cation 1: ………………………… cation 2: …………………………

anion 1: ………………………… anion 2: …………………………

[4]

(ii) Write equations, with state symbols, to illustrate the observations in **3(c)**.

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

(iii) Predict what will happen when solid ammonium nitrate is added to **FA 9** followed by aqueous ammonia.

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[Total : 14]

4 Planning

Determination of the hardness of water in a sample.

Hard water is water that has high mineral content and is formed when water percolates through deposits of limestone, chalk and gypsum, which are largely made of calcium carbonates, bicarbonates and sulfates. The hardness of water is measured in terms of the molar concentration of Ca^{2+} in mol dm^{-3}.

In a particular sample of water, the hardness is caused by the presence of calcium chloride. The amount of Ca^{2+}, and hence the concentration of Ca^{2+}, can be determined by precipitation with an excess of oxalic acid.

$$Ca^{2+}(aq) + C_2O_4^{2-}(aq) \rightleftharpoons CaC_2O_4(s)$$

You are to design an experiment to determine the hardness of water in a sample.

You are provided with

- sample of water with estimated hardness of 0.02 mol dm^{-3}
- 0.1 mol dm^{-3} of oxalic acid $H_2C_2O_4$
- electronic balance
- common laboratory apparatus

(a) Given the K_{sp} of calcium oxalate is 2.7×10^{-9} mol^2 dm^{-6}, predict what will be observed when 25.0 cm^3 of the water sample is mixed with equal volume of the 0.1 mol dm^{-3} of oxalic acid solution.

[2]

(b) Your plan should include a step-by-step description of the method, including

- the sequence of steps to ensure that the maximum amount of precipitate is recovered,
- the use of appropriate apparatus,
- the recordings using the letters A, B, C etc to represent the measurement taken and present it with an appropriate table format,
- the processing of the results to determine the molar concentration of Ca^{2+} in the water sample.

[A_r: C, 12.0; Ca, 40.1; H, 1.0; O, 16.0]

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[Total: 8]

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

anion	*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 A*l* foil
nitrite, NO_2^- (aq)	NH_3 liberated on heating with OH^-(aq) and A*l* 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, 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