NANYANG JUNIOR COLLEGE
JC2 PRACTICAL PRELIMINARY EXAMINATION
Higher 2

CANDIDATE NAME

CLASS				
CENTRE NUMBER	S			

INDEX NUMBER

CHEMISTRY 9729/04

Paper 4 Practical

14 August 2024

2 hour 30 minutes

Candidates answer on the Question Paper

Additional Materials: As listed in the Confidential Instructions

READ THESE INSTRUCTIONS FIRST

Write your Centre number, index number, name and class 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 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 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	/ 16
2	/ 16
3	/12
4	/ 11
Total	/55

This document consists of **20** printed pages and **4** blank pages.

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

1 An investigation of a gemstone

Spinel minerals form crystals that are often used as gemstones. The general formula of a spinel mineral is $\mathbf{AB_2O_4}$, where **A** and **B** are different metal ions. The presence of different combinations of metal ions accounts for the range of colours found in these gemstones. One example of a spinel gemstone is ***hercynite***, which is blue-green in colour.

As they are essentially composed of metal oxides, spinel crystals may be reacted with strong acids to form solutions containing salts of the metal ions present. The rarity and high cost of spinel gemstones, however, prohibit the preparation of such solutions for this task.

The **FA 1** solution you are supplied with is a mixture of two salts. It is intended to duplicate a solution that could have been produced by reacting hercynite crystals with a strong mineral acid. Consequently, non-metal ions are also present in **FA 1**.

FA 1 is a solution containing the metal ions, **A** and **B**, together with non-metal ions.
FA 2 is a solution of sodium carbonate, Na_2CO_3.
Solid FA 3 is granulated zinc.

You will perform tests to identify the metal ions, **A** and **B**, present in **FA 1**.

You are **not** expected to identify the anions.

You are advised to consider the general formula of the spinel minerals, $\mathbf{AB_2O_4}$, and the likely oxidation states of the metal ions, **A** and **B**, before you start this experiment.

Unless otherwise stated, the volumes given below are approximate and should be estimated rather than measured.

Test and identify any gases evolved.

(a) Carry out the following tests. Carefully record your observations in Table 1.1.

Table 1.1

	test	observations
1	Test the **FA 1** solution using Universal Indicator paper.	
2	Using a measuring cylinder, transfer 10 cm^3 of **FA 1** to a clean boiling tube. Use a dropping pipette to transfer 1 cm^3 of **FA 2** to the boiling tube. Mix the contents of the boiling tube thoroughly. Repeat the transfer of 1 cm^3 of **FA 2** with thorough mixing of the contents in the boiling tube until all of the **FA 2** is used up.	
3	Place about 2 cm^3 of **FA 1** into a test-tube. Carefully add sodium hydroxide, dropwise with shaking, until no further changes are seen. Swirl and filter the mixture, collecting the filtrate in a clean boiling tube. Keep the filtrate for use in Test 6. Wash the residue thoroughly with deionised water. Discard the washings. Keep the residue for use in Test 4.	

<table>
<tr><th colspan="2">test</th><th>observations</th></tr>
<tr><td colspan="3">Note: When aqueous potassium manganate(VII) is added in Test 4, the endpoint is a permanent pale pink colour.</td></tr>
<tr><td>4</td><td>Transfer a spatula load of the residue collected in Test 3 to a clean test-tube.

Carefully add hydrochloric acid, dropwise with shaking, until no further changes are seen.

Add aqueous potassium manganate(VII), dropwise with shaking, until the end-point is reached.

Record the number of drops of aqueous potassium manganate(VII) you have added to reach the end-point.

Keep this mixture for use in Test 5.</td><td></td></tr>
<tr><td>5</td><td>To the final mixture in Test 4, add one piece of FA 3. Gently warm the mixture and set aside for a few minutes. From time to time, observe the mixture. While you are waiting, continue with Test 6.

When no further changes are observed, decant the solution into a second test-tube.

Add a few drops of aqueous potassium manganate(VII) to the second test-tube, with shaking.</td><td></td></tr>
</table>

	test	observations
6	Place about 1 cm^3 of the filtrate collected in Test 3 in a clean test-tube. Carefully add hydrochloric acid, dropwise, until no further change is seen **before** you shake the mixture. This mixture is then use for Test 7.	
Note: You should **NOT** do the following test. Observations have been recorded for you.		
7	Place about 1 cm^3 of the final mixture in Test 6 in a clean test-tube. Add aqueous ammonia, dropwise with shaking, until no further change is seen.	white ppt. insoluble in excess

[8]

Obs points		marks	

(b) By considering your observations in Tests 1 and 2, suggest an explanation for the observations in Test 2 in terms of the chemistry involved.

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(c) **(i)** By considering your observations in Tests 3, 6 and 7, identify the metal-containing complex ion present in the filtrate collected in Test 3.

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

(ii) Identify the residue collected in Test 3.

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

(iii) **FA 1** is intended to duplicate a solution that could have been produced by dissolving hercynite in a strong acid.

Suggest a formula for hercynite.

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

(d) Explain the following reactions involving the metal ions in the reaction mixture used in Test 5.

- reaction between granulated zinc and resultant mixture from Test 4
- reaction between solution in the second test-tube and aqueous potassium manganate(VII)

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

(e) Explain, in terms of the chemistry involved, and with the aid of relevant equations, your observations in Test 6.

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[Total: 16]

2 Determination of the molar enthalpy change of a reaction by an indirect method

It is not possible to measure experimentally the enthalpy change, ΔH_1, for the following reaction as it does not take place in the laboratory.

equation 1 $Na_2CO_3(s) + CO_2(g) + H_2O(l) \rightarrow 2NaHCO_3(s)$ ΔH_1

Sodium carbonate and sodium hydrogencarbonate each react with hydrochloric acid.

equation 2 $Na_2CO_3(s) + 2HCl(aq) \rightarrow 2NaCl(aq) + H_2O(l) + CO_2(g)$ $\Delta H_2 = -36\ kJ\ mol^{-1}$

equation 3 $NaHCO_3(s) + HCl(aq) \rightarrow NaCl + CO_2 + H_2O$ ΔH_3

In this question, you will perform an experiment to determine a value for ΔH_3. You will use the results of the experiments you have carried out, data provided for ΔH_2 and an energy cycle to calculate a 'theoretical' value for ΔH_1.

You are provided with:
- **FA 4**, 2.00 mol dm^{-3} hydrochloric acid, HCl
- solid **FA 5**, $NaHCO_3$.

(a) (i) Determination of the molar enthalpy change of reaction, ΔH_3

In this experiment, you will measure the temperature of the contents of a polystyrene cup at timed intervals, both before and after solid $NaHCO_3$ is added. You will analyse your results graphically in order to determine an accurate value for the temperature change of the mixture, caused by the reaction between solid $NaHCO_3$ and HCl.

You will use this value to calculate the heat change, q, for the experiment and hence determine a value for the molar enthalpy change of reaction, ΔH_3.

In an appropriate format in the space provided on page 8, prepare tables in which to record results 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 the nearest 0.5 min.

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

Procedure

1. Weigh the capped bottle containing solid **FA 5**. Record the mass in your table on page 8.

2. Place one polystyrene cup inside a second polystyrene cup. Place these in a glass beaker to prevent them from tipping over.

3. Use a measuring cylinder to transfer 50 cm^3 of **FA 4** into the first polystyrene cup. Cover the cup with a lid.

4. Carefully stir the solution of **FA 4** in the cup with the thermometer. Read and record the temperature, *T*. Start the stopwatch (*t* = 0.0 min). The stopwatch must be left to run for the rest of the experiment.

5. Continue to stir the solution. Read and record *T* every minute.

6. At **exactly** three minutes, remove the lid and transfer the solid **FA 5** cautiously to the polystyrene cup in three separate lots, taking care that the mixture does not overflow. Replace the lid. Stir the mixture but do not read *T*.

7. Continue to stir the mixture. Read and record *T* at *t* = 3.5 min.

8. Continue to stir the mixture. Read and record *T* at *t* = 4.0 min and every minute until *t* = 9.0 min.

9. Reweigh the emptied bottle with its cap. Record this mass in your table below.

Results

[4]

(ii) Plot a graph of temperature, *T*, on the *y-axis*, against time, *t*, on the *x*-axis on the grid in Fig. 2.1.

Draw a best-fit straight line taking into account all of the points before *t* = 3.0 min.

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

Extrapolate (extend) both lines to *t* = 3.0 min. [3]

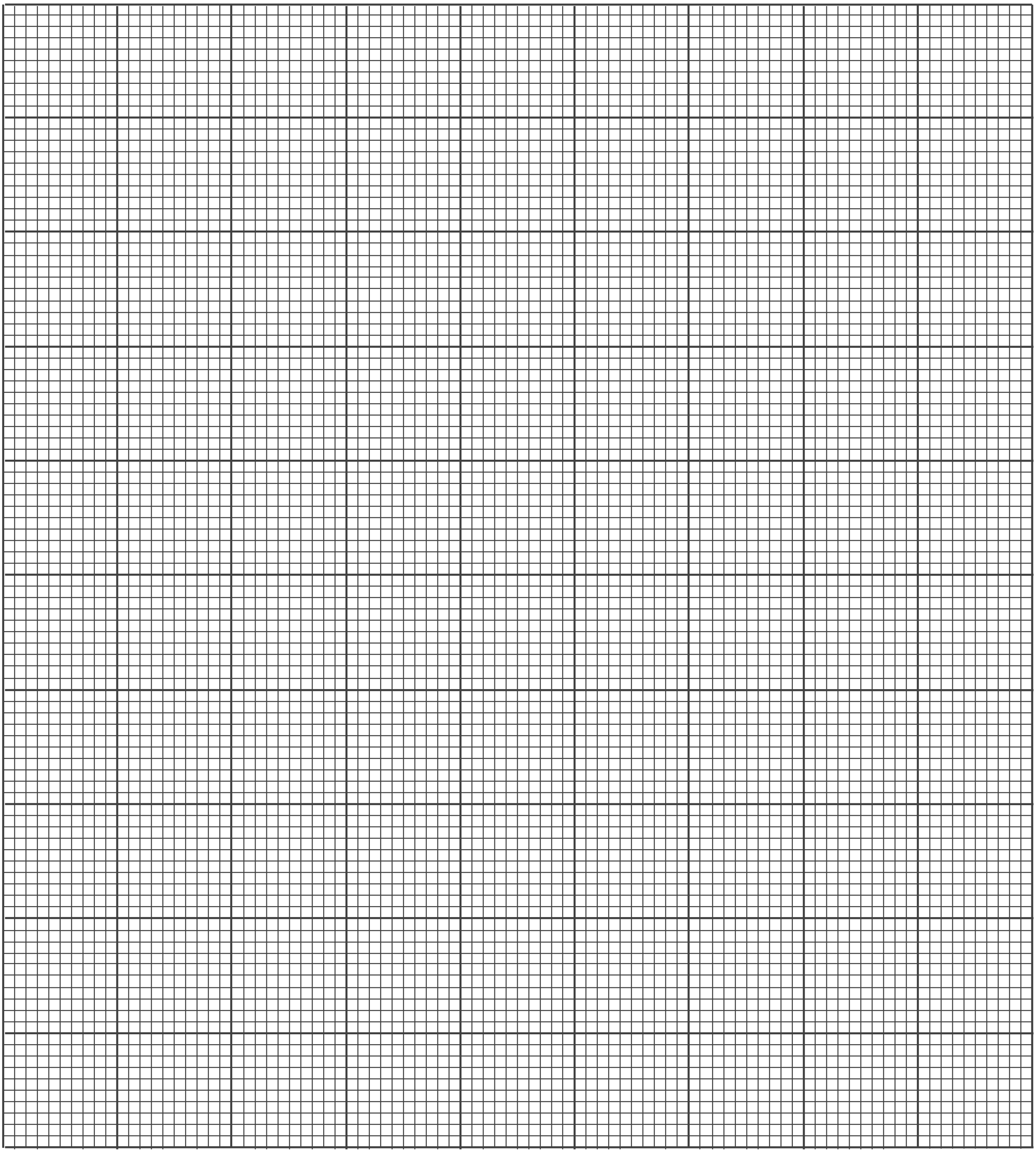

Fig. 2.1

(iii) From your graph, read the minimum temperature, T_{min}, and the maximum temperature, T_{max}, at t = 3.0 min. Record these values in the spaces provided.

Calculate the temperature change, ΔT, at t = 3.0 min.

T_{min} = ……………………………..

T_{max} = ……………………………..

ΔT = ……………………………..

[2]

(iv) Calculate the heat change, q, for your experiment using the value you calculated in **(a)(iii)**.

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

q = …………………………….. [1]

(v) Determine the molar enthalpy change, ΔH_3, for the reaction in equation 3.

Include the sign of ΔH_3 in your answer.
[A_r: Na, 23.0, H, 1.0, C, 12.0, O, 16.0]

ΔH_3 = ……………………………. [2]

(b) Use the value of ΔH_2 given and your value of ΔH_3 calculated in **(a)(v)**, calculate a value for ΔH_1 for the reaction in equation 1.

ΔH_1 = ……………………………………[2]

(c) A student performed the same experiment using 40 cm^3 of **FA 4**.

Explain how this will affect the temperature change per gram of **FA 5** and hence the accuracy of the experiment.

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

[Total: 16]

3 Analysis of a redox reaction between hydroxylamine and iron(III) ion

A redox reaction takes place between hydroxylamine, NH_2OH, and iron(III) ion, Fe^{3+}, in acidic conditions. Iron(III) ion is reduced to an iron(II) ion, Fe^{2+}. The reaction is slow at room temperature but is completed in a few minutes at 100 °C.

The reaction is shown by one of the following equations.

equation 1 $NH_2OH(aq) + Fe^{3+}(aq) \rightarrow Fe^{2+}(aq) + H^+(aq) + \frac{1}{2}N_2(g) + H_2O(l)$

equation 2 $NH_2OH(aq) + 2Fe^{3+}(aq) \rightarrow 2Fe^{2+}(aq) + \frac{1}{2}N_2O(g) + 2H^+(aq) + \frac{1}{2}H_2O(l)$

equation 3 $NH_2OH(aq) + 3Fe^{3+}(aq) \rightarrow 3Fe^{2+}(aq) + NO(g) + 3H^+(aq)$

You will carry out a titration to determine which of the three suggested equations best represents the reaction. Iron(II) ions formed in the reaction with hydroxylamine are oxidised by manganate(VII) ions.

$MnO_4^-(aq) + 5Fe^{2+}(aq) + 8H^+(aq) \rightarrow Mn^{2+}(aq) + 5Fe^{3+}(aq) + 4H_2O(l)$

FA 6 is 0.0200 mol dm^{-3} potassium manganate(VII), $KMnO_4$.
FA 7 is a solution prepared by boiling a 1.00 dm^3 aqueous mixture containing 3.30 g of hydroxylamine hydrochloride, $NH_2OH•HCl$, excess iron(III) chloride, $FeCl_3$, and excess sulfuric acid. Any water lost by evaporation was replaced after cooling.
FA 8 is dilute sulfuric acid.

Assume that one mole of hydroxylamine hydrochloride gives one mole of hydroxylamine in solution.

(a) (i) Procedure

1. Fill the burette with **FA 6**.
2. Pipette 25.0 cm^3 of **FA 7** into a conical flask.
3. Use a 10 cm^3 measuring cylinder, add 10 cm^3 of **FA 8** into the same conical flask.
4. Titrate the solution in the conical flask with **FA 6**. The end-point is reached when a permanent pale pink colour is obtained.
5. Record your titration results to an appropriate level of precision in the space on page 13.
6. Repeat steps 2 to 5 until consistent results are obtained.

Titration results

[3]

(ii) From your titrations, obtain a suitable volume of **FA 6** to be used in your calculations. Show clearly how you obtained this volume.

volume of **FA 6** = ……………………….. [3]

(b) **(i)** Calculate the amount in moles of potassium manganate(VII) present in the volume of **FA 6** in **(a)(ii)**.

amount of potassium manganate(VII) = ……………………. mol [1]

(ii) Use your answer in **(b)(i)** to calculate the amount in moles of iron(II) ions in 25.0 cm^3 of solution **FA 7**.

amount of iron(II) ions = …………………… mol [1]

(iii) Calculate the amount in moles of hydroxylamine hydrochloride that has reacted with the **FA 7** pipetted into the conical flask. Show your working.
[A_r: N, 14.0; H, 1.0; O, 16.0; Cl, 35.5]

amount of hydroxylamine hydrochloride = …………………………. mol [2]

(iv) Use your answer in **(b)(iii)** to deduce which of the three suggested equations corresponds to your results. Show your working.

The correct equation number is ………………………. [2]

[Total: 12]

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4 Planning

Baker's yeast is a useful enzyme which can be used to catalyse the decomposition of hydrogen peroxide.

$$H_2O_2(aq) \rightarrow H_2O(l) + \frac{1}{2}O_2(g)$$

You are provided with

apparatus:
- 100 cm^3 conical flask, fitted with rubber bung and rubber tubing
- gas syringe (possible capacities of 10, 50 or 100 cm^3)
- stopwatch
- all other common laboratory equipment

reagents:
- 50 cm^3 of yeast suspension
- 30 cm^3 of 3% (by weight) hydrogen peroxide
- distilled water

When 8.0 cm^3 of the yeast suspension, 4.0 cm^3 of H_2O_2 and 18.0 cm^3 of distilled water were mixed, 10 cm^3 of oxygen gas was produced in 90 s.

Use the above information and the reagents and apparatus provided to design an experiment to determine the orders of reaction with respect to yeast and hydrogen peroxide respectively.

In your experiment, you should perform a total of five experimental runs (including the above run) to measure the volume of oxygen gas produced at regular intervals in each run. The volume of yeast suspension used should be varied in all the runs while keeping the volume of H_2O_2 constant at 4.0 cm^3. One of the experimental runs should be conducted to allow the reaction to go to completion.

(a) Calculate the maximum volume of oxygen gas produced for an experimental run if the reaction was allowed to go to completion. In your calculations, assume that the density of H_2O_2 is 1.00 g cm^{-3} and the experiment is conducted under r.t.p. conditions.
[A_r: H, 1.0; O, 16.0 and V_m = 24 dm^3 mol^{-1} at r.t.p.]

maximum volume of oxygen gas produced = …………………………..

Hence, state the appropriate capacity of the gas syringe to be used.

capacity of gas syringe = …………………………..

[2]

(b) Plan an experiment to collect sufficient data to plot suitable graphs to prove that the reaction is first order with respect to hydrogen peroxide and first order with respect to yeast.

Your plan should include details of:

- a table to tabulate the volumes of each of the reagents that you will be using for each run of the experiment
- the apparatus you would use
- the procedure you would follow
- the measurements you would take

You may find it useful to indicate in your procedure the experimental run that would be conducted to allow the reaction to go to completion and the measurement to be taken when the reaction has reached completion.

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[5]

(c) **(i)** Sketch a graph you would expect to show how the volume of O_2 will vary with time on Fig. 4.1. Using the maximum volume of oxygen gas calculated in **(a)**, describe how you would use your graph to prove that the reaction is first order with respect to hydrogen peroxide.

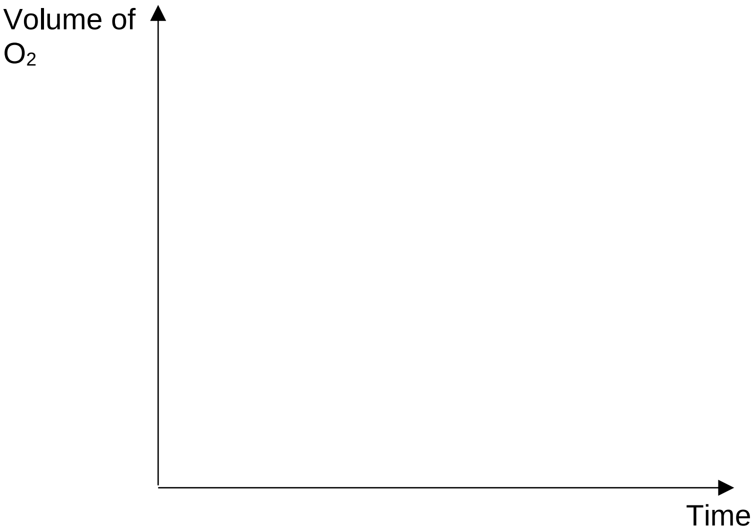

Fig. 4.1

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…………………………………………………………………………………………….

……………………………………………………………………………………… [2]

(ii) In Fig. 4.1 above, show how the initial rate can be determined. Hence, sketch the graph you would expect on Fig. 4.2 to prove that the reaction is first order with respect to yeast.

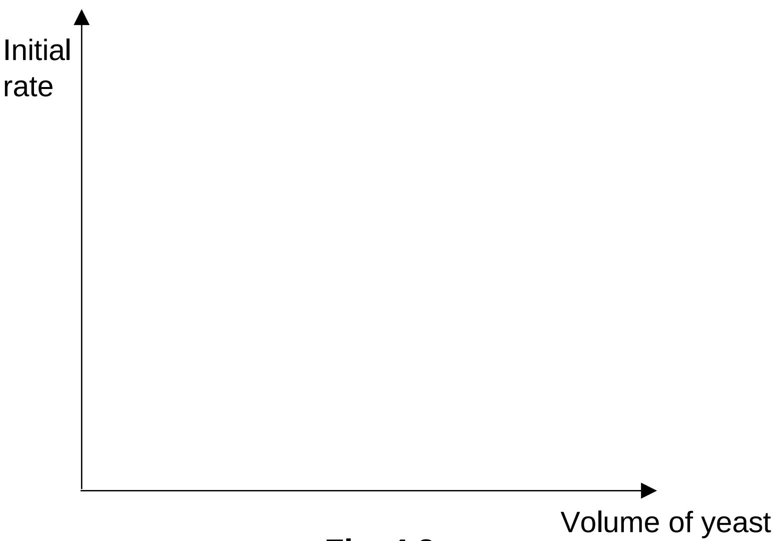

Fig. 4.2

[2]

[Total: 11]

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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