Name: .... (2T   )   Date: ....
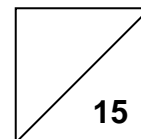
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Experiment 25: **Dry Practice 3**

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Answer **ALL** the questions in the SPACES provided.

To deduce the nature of solutions provided by measuring their pH values.

A series of buffer solutions can be made by mixing varying volumes of equimolar (i.e., of the same concentration) solutions of the salt, NaH_2PO_4 (**FA3)** and the corresponding disodium salt, Na_2HPO_4 (**FA4**) which is equimolar with **FA3**.

The pH of any mixture of **FA3** and **FA4** is given by

$$pH = 6.6 - lg\ V_3 + lg\ V_4$$

where V_3 is the volume of **FA3** in cm^3 used and V_4 is the volume of **FA4** in cm^3 used, and $lg\ V = log_{10}\ V$.

You are provided with:
FA2   solution of NaOH of pH 13;
FA3   sodium salt, NaH_2PO_4;
FA4   disodium salt, Na_2HPO_4, equimolar with **FA3**;
FA5   0.010 mol dm^{-3} hydrochloric acid.

1   Make up the following mixtures of **FA3** and **FA4** using the two burettes provided to measure the volumes. You are then to calculate those pH values which are not given by using the formula for pH given above.

The two solutions should be run into test-tubes clearly marked with the pH value of the mixture you are making.

Volume of **FA3** /cm^3	10	**9**	8	4	2	**1**	0
Volume of **FA4** /cm^3	0	**1**	2	6	8	**9**	10
pH	4.0*	**5.6**	6.0	6.8	7.2	**7.6**	9.0*

*Note that the equation given for pH applies only to mixtures, so you cannot use it for the solutions given for pH 4 and pH 9.

(a)   Calculate the missing pH values.   [2]
   pH = 6.6 – lg V₃ + lg V₄ = 6.6 – lg 9 + lg 1 = 5.6   **[1]**

   pH = 6.6 – lg V₃ + lg V₄ = 6.6 – lg 1 + lg 9 = 7.6   **[1]**

(b) You are also provided with a solution **FA5**, which is 0.010 mol dm^{-3} hydrochloric acid. The solution containing NaOH(aq), **FA2** can be used as a solution of pH 13. Using the measuring cylinder provided, measure 10 cm^3 of each of these solutions into separate test-tubes clearly labelled with their pH value. (You should now have nine labelled test-tubes.)

Add five drops of indicator **I** to each of the labelled solutions, shake and record in the table below the colour of the indicator at each pH. (You may, if you wish, add further drops of indicator to intensify the colours but you **must** add the same number of drops of indicator to each solution.)

Fill in the table with the missing pH values.

pH	**2.0**	4.0	**5.6**	6.0	6.8	7.2	**7.6**	9.0	**13.0**
Colour	**Red**	**Beige**	**Yellow**	**Lime green**	**Light green**	**green**	**Bluish green**	**Blue**	**Purple**

[3]

[1] Recording all the pH that are not given.
[2] Recording all the colours correctly.

2   You are provided with 10 cm^3 of solutions **S1**, **S2**, **S3**, **S4** and **S5** in labelled test-tubes. You are to determine the pH of each of the solutions. Add to each solution the same number of drops of indicator **I** as you used in making your comparison solutions in **1**.

Estimate the pH of each solution by comparing the colour of the solution with the colour of the solutions of known pH. Record your results in the table provided below.

Add 1.0 cm^3 of **FA2** to each solution, shake, and estimate the pH of the resulting solutions.

Solution	pH	pH after adding 1.0 cm^3 of **FA2**
S1	**5.6**	**6.0**
S2	**13.0**	**13.0**
S3	**2.0**	**9.0**
S4	**6.8**	**13.0**
S5	**4.0**	**7.2**

(a) Deduce what you can about the nature of the solutions **S1** to **S5** (e.g. they could be strong or weak acids or bases, or buffer solutions or water).

Give reasons for your answers.
Correct identification of each sample with reasoning [5 x 2 m]

S1: Buffer solution (acid buffer) since the initial pH is 5.6 and that the pH remains fairly constant upon addition of a small amount of the base, NaOH, FA2.

S2: A strong base since the initial pH is 13.0 and on addition of FA2 the pH is still the same.

S3: A weak acid as after addition of a strong base, FA2, the resulting solution is alkaline, pH = 9.0. However, the initial pH is rather low for a weak acid, suggesting that perhaps it is weak acid with relatively high concentration.

S4: Water as the initial pH is about neutral, pH = 6.8 and on addition of FA2 the pH goes up to 13.0.

S5: A strong acid as it is neutralised by the strong base, FA2 to give an almost neutral solution pH = 7.2. However, the initial pH is rather high for a strong acid, suggesting that perhaps it is a strong acid with relatively low concentration.
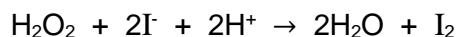
[10]

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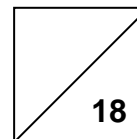
Experiment 26: **Dry Practice 4**

Answer **ALL** the questions in the SPACES provided.

You are required to investigate how the rate of the reaction between hydrogen peroxide and iodide ions in acid solution,

$$H_2O_2 + 2I^- + 2H^+ \rightarrow 2H_2O + I_2$$

depends on the concentration of the reactants.

You will require
(i) Solution **FA2** containing 12 g dm^{-3} of sodium thiosulfate pentahydrate;
(ii) Solution **FA3** containing 25 g dm^{-3} of potassium iodide;
(iii) Solution **FA4**, which is approximately 0.1 mol dm^{-3} hydrogen peroxide, with a 25 cm^3 pipette to measure it;
(iv) a solution of sulfuric acid of approximate concentration of 1 mol dm^{-3} and a measuring cylinder with which to measure 20 cm^3 portions;
(v) starch indicator and a measuring cylinder with which to measure 10 cm^3 portions;
(vi) a supply of deionised water, and a 50 cm^3 measuring cylinder.

If hydrogen peroxide, potassium iodide, and an acid are mixed in the presence of starch and some sodium thiosulfate, the iodine liberated will react with thiosulfate ions until they are all used up. When no thiosulfate ions are left, any further iodine formed will react with starch and the blue-black colour of the starch-iodine complex will appear. The time taken for the blue-black colour to appear with a constant quantity of sodium thiosulfate present will depend on the rate of formation of the iodine.

You are required to measure the time taken for the experiments in which the concentrations of the reactants is varied.

Method
1   Pipette 25.0 cm^3 of hydrogen peroxide solution, **FA4** into a conical flask. Add 20 cm^3 of 1 mol dm^{-3} sulfuric acid, 10 cm^3 of starch solution, and 25 cm^3 of deionised water, measured as accurately as possible with the vessels provided.
2   Run in from a burette 10.00 cm^3 of sodium thiosulfate, **FA2**.
3   Measure 50 cm^3 of potassium iodide, **FA3** into a small beaker, and add it, in one portion, to the mixture in the conical flask, noting the time as you do so.
4   Swirl the flask gently to ensure good mixing, and measure the time taken to the nearest second, for the blue-black colour to appear.
5   If no colour appears after two minutes, you have made a mistake in mixing, so try again.
6   Record your result for this experiment in the table below.
7   Repeat the experiment described above, using the volumes of solution (in cm^3) shown in the last three lines of the table below.

8   Record the times taken, t (to nearest seconds) in the table and then evaluate the product (V x t) of volume V of potassium iodide and the time, t and record your answers in the table.

Note that the time of two minutes maximum is given as a guide for the first experiment only.

Volume of H_2O_2 /cm^3	Volume of H_2SO_4 /cm^3	Volume of Starch /cm^3	Volume of Water /cm^3	Volume of **FA2** /cm^3	Volume of KI (V) /cm^3	Time (t) /s	Product (V x t) /cm^3 s
25.0	20	10	25	10.00	50	**24**	**1200**
25.0	20	10	35	10.00	40	**31**	**1240**
25.0	20	10	45	10.00	30	**41**	**1230**
25.0	20	10	55	10.00	20	**62**	**1240**

[5]

Answer the following questions about the experiment you have carried out.

(a) Why is the volume of water used in each experiment varied?
To keep the total volume of the reaction mixture constant at 140 cm^3.

[1]

(b) Does the product of volume and time ((V x t) gradually increase, or gradually decrease, or remain approximately constant, or vary in a non-uniform way?
Remains approximately constant.

[1]

(c) How does the concentration of iodide ion in the reaction mixture depend on the volume of potassium iodide solution, **FA3** used in each experiment?
As the total volume of the reaction mixture is kept constant, the concentration of the iodide ion is directly proportional to the volume of potassium iodide solution;
i.e. [I⁻] $\propto V$

[1]

(d) How is the rate of reaction related to the time taken for the appearance of the blue-black colour?

Rate is inversely proportional to time; i.e. rate $\propto \dfrac{1}{t}$

[1]

(e) How does the rate of reaction depend on the concentration of the iodide ion?
As (V x t) is approximately constant, if follows that the rate of reaction is directly proportional to [I⁻].

Note: V x t = constant = c
$t = c/V$
$\dfrac{1}{t} = \left(\dfrac{1}{c}\right) V$ So, rate = $\left(\dfrac{1}{c}\right)$ [I⁻]   as rate $\propto \dfrac{1}{t}$ and [I⁻] $\propto V$ and $\left(\dfrac{1}{c}\right)$ is a constant.

Therefore, rate $\propto$ [I⁻]

[1]

(f) Hence, what is the order of reaction with respect to the concentration of the iodide ion?
Order of reaction = 1

[1]

5

Carry out one further experiment similar to those present in **Method 1 to 8** which will enable you to deduce how the rate of the reaction between iodide ions and hydrogen peroxide in acid solution depends on the concentration of hydrogen peroxide. Record the volumes of the solutions you use and the time you measure in the table below.

Volume of H_2O_2 /cm^3	Volume of H_2SO_4 /cm^3	Volume of Starch /cm^3	Volume of Water /cm^3	Volume of **FA2** /cm^3	Volume of KI (V) /cm^3	Time (t) /s
15.0	20	10	45	10.00	40	52

[1]

(g) Deduce from your results how the rate of the reaction depends on the concentration of hydrogen peroxide.

Volume of H_2O_2 (V) /cm^3	Volume of H_2SO_4 /cm^3	Volume of Starch /cm^3	Volume of Water /cm^3	Volume of **FA2** /cm^3	Volume of KI /cm^3	Time (t) /s	Product (V' x t) /cm^3 s
15.0	20	10	45	10.00	40	52	780
25.0	20	10	35	10.00	40	31	775

To deduce the rate of reaction wrt concentration of H_2O_2, vary the volume of H_2O_2 but keeping the volume of the other reagents constant. In addition, also vary the volume of water so as to keep the total volume of the reaction mixture fixed at 140 cm^3.

Since, the product (V' x t) is approximately constant, it follows that the rate of reaction is directly proportional to [H_2O_2].
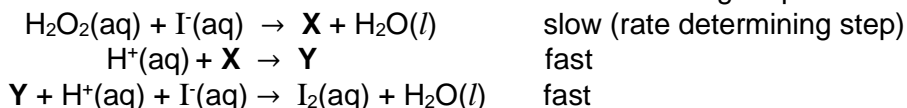
(The above conclusion can also be arrived at by the use of the usual mathematical method.)
[2]

(h) What is the order of reaction with respect to the concentration of the hydrogen peroxide?
Order of reaction = 1
[1]

(i) Hence, write out the rate equation for the reaction between hydrogen peroxide and iodide ions in acid solution, using k to denote the rate constant.
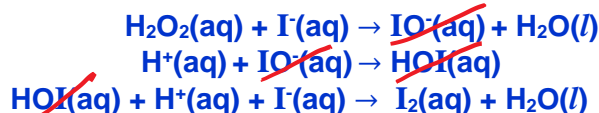**Rate = k [H_2O_2] [I^-]**

[1]

(j) A proposed mechanism for the reaction consists of the following steps:

$$H_2O_2(aq) + I^-(aq) \rightarrow X + H_2O(l) \quad \text{slow (rate determining step)}$$
$$H^+(aq) + X \rightarrow Y \quad \text{fast}$$
$$Y + H^+(aq) + I^-(aq) \rightarrow I_2(aq) + H_2O(l) \quad \text{fast}$$

Suggest species for **X** and **Y**, which are intermediates.
X = IO^-(aq) and Y = HOI(aq)

[2]

$H_2O_2(aq) + I^-(aq) \rightarrow$ ~~IO^-(aq)~~ $+ H_2O(l)$
$H^+(aq) +$ ~~IO^-(aq)~~ $\rightarrow$ ~~HOI(aq)~~
~~HOI(aq)~~ $+ H^+(aq) + I^-(aq) \rightarrow I_2(aq) + H_2O(l)$

$H_2O_2(aq) + 2I^-(aq) + 2H^+(aq) \rightarrow I_2(aq) + 2H_2O(l)$ (overall equation)

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Some Important Pointers in Qualitative Analysis (QA)

Students are often unsure as to when should one should test for the liberation of gases in QA. Let us first examine the test for gases as given in the QA Notes provided in the Paper 4 (Practical Paper) at A Level.

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

The gases that are of concern are four gases, CO_2, O_2, H_2 and NH_3. The point to note is that a test must be carried for the identity of the gases evolved. For e.g. if you are asked to add an acid to a carbonate, effervescence is observed indicating CO_2 gas liberated. Not enough to state "effervescence" only! A test such as "gas liberated gives a white ppt with limewater" has to be performed and recorded! The gas also needs to be identified as "CO_2".

Dry Test
Heat specimen in a **DRY** test tube. Heat until no change. For a dry test, only 2 gases are of concern to us. It is either **O_2 or CO_2** gas liberated.

1 If CO_2 is liberated, suspect CO_3^{2-}, e.g. $CaCO_3(s) \rightarrow CaO(s) + CO_2(g)$
2 If O_2 is liberated, suspect oxygen containing compound, e.g. ClO_3^- or IO_3^-, etc. (actual identity is not required). $2KClO_3(s) \rightarrow 2KCl(s) + 3O_2(g)$

   Then take note of the colour of the residue, if any.

3 In addition, if charring occurs, suspect organic substances, e.g. $CH_3CO_2^-$, HCO_2^-, $C_6H_5CO_2^-$, $C_2O_4^{2-}$, etc. (actual identity is not required). Then follow up by testing for any organic vapour/gas liberated by use of a lighted splint. If vapour/gas liberated burns with a luminous (yellow) flame, suspect organic vapour/gas.

   Very likely propanone, CH_3COCH_3 vapour liberated. Suspect $CH_3CO_2^-$
   e.g. $Ca(CH_3CO_2)_2(s) \rightarrow CaCO_3(s) + CH_3COCH_3(g)$

   Refer to Practical Experiment 24.

Wet Test

Prepare an aqueous solution of sufficient concentration (test solution) as instructed, and add the usual bench reagents as instructed too, to the test solution until excess.
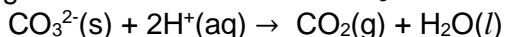
Note precipitation, change of colour and so on.

Test for **gases** only
(i) When asked to add acids to solids (or adding carbonates to unknown).
(ii) When asked to heat.

1 If effervescence is observed, e.g. action of acids on solids (or adding carbonates to unknown), go for CO_2 using limewater. Presence of CO_3^{2-}.
$$CO_3^{2-}(s) + 2H^+(aq) \rightarrow CO_2(g) + H_2O(l)$$

2 If effervescence is observed, e.g. action of acids on black/grey powder, go for H_2 using a wooden splint. The black/grey powder is likely to be a reactive metal such as Zn or Mg or Al.
$$Zn(s) + 2H^+(aq) \rightarrow H_2(g) + Zn^{2+}(aq)$$
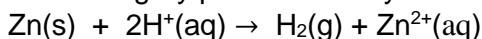
3 If asked to add NaOH(aq) to test solution and <u>heat</u>, test for NH_3 gas using a damp red litmus paper, placed over the mouth of the test-tube. NH_4^+ is suspected to be present.
$$NH_4^+ + OH^-(aq) \rightarrow NH_3(g) + H_2O(l)$$
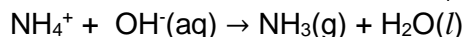
The idea of heating is to drive out any soluble gas such as NH_3 as the solubility of gases decreases with increasing temperature. For insoluble or sparingly soluble gases such as O_2 and H_2, effervescence will be observed without heating!

4 If asked to add H_2O_2(aq) to test solution, test for O_2 gas using a glowing splint. Effervescence will be observed. Apart from the oxidising property of H_2O_2(aq) it will also undergo decomposition, spontaneously.
$$2H_2O_2(aq) \rightarrow O_2(g) + 2H_2O(l)$$
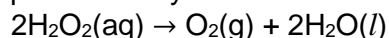
Note that for the wet test, only 4 gases are of relevance, namely, **O_2, CO_2, H_2 and NH_3**. An understanding of when to test for the relevant gas is important as described above. Do not test for gases each and every time a test is given! It is a complete waste of time! You need to know what you are doing!

Remember, for wet tests, the observed ppt formed, and its solubility in excess reagents, change of colours of solutions and so on, are more important in order to arrive at the identity of an unknown sample given.

In the light of this, it is important to be familiar with the QA Notes provided on the last few pages of Paper 4. In addition, also go through the detailed explanatory notes on the reactions involving the stated cations and anions with respect to the QA Notes. This is found in your **Practical Handbook 4**. Of course, you will not be asked to explain the reactions given but it is good to be able to understand the reactions.

Finally, take note that also you will not be asked to carry any systematic analysis to find the identity of unknown substances given. You will only be asked to carry out prescribed tests and then to note the observations. And then to draw conclusions based on the observations.