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EXPERIMENT - 1

Object :- To prepare and standardize the solution of NaOH (N/10) against standard solution of Oxalic acid (N/5).

APPARATUS/CHEMICALS : Burette, Pipette, conical flask, NaOH solution, phenolphthalein indicator, Oxalic acid (N/5).

THEORY :

Standard solution is one in which amount of a substance is present in definite volume of the solution, whose concentration is known called as standard solution.

Types of solution

1) Primary standard solution
2) Secondary standard solution

PRIMARY STANDARD SOLUTION :-

The substance whose standard solution is prepare by dissolving directly its definite amount and then dissolving in definite volume of solvent or solution is

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known as a primary standard substance and the solution is called as primary standard solution.

SECONDARY STANDARD SOLUTION:

The substance whose solution cannot be prepared directly by weighing its definite amount and then dissolving in definite volume of solvent is called secondary standard substance.

The common secondary standard substances are alkali hydroxide, inorganic acids and $KMnO_4$, etc.

NORMALITY (N):

The normality of a solution is the number of gram-equivalent of the solute per litre of the solution.

$$N = \frac{\text{No. of grams equivalent of solute}}{\text{Volume of the solution in 1000mL}}$$

PROCEDURE:

1. Prepare NaOH solution
2. Rinse and fill the burette with N/5 oxalic acid solution.
3. Pitteptte out 10ml of above prepare NaOH solution in a conical flask.
4. Edd 2-3 drop of phenolphathelein indicator solution

OBSERVATION TABLE :-

S. No.	VOL. OF NaOH SOLN (V_1)	BURETTE READING		VOL OF Std. SOLN OF OXALIC ACID (V_1)
		INITIAL	FINAL	
1)	10ml	0.0	9.5	9.5 ml
2)	10ml	10.0	19.4	9.4 ml
3)	10ml	20.0	29.3	9.3ml
4)	10ml	30.0	39.4	9.4ml

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5. Add oxalic acid solution from burette drop by drop into it till pink colour disappear.
6. Note down the reading.

OBSERVATION:-

Wt of the empty weighing tube (W_1) gm.

Wt of the weighing tube with substance (W_2) gm.

Wt of the substance ($W_2 - W_1$) gm

CALCULATION:-

N_2V_2 (oxalic acid) = N_1V_1 (NaOH)

$N_1V_1 = N_2V_2$

$N_1 \times 10 = 0.1 \times 9.4$

$N_1 = \frac{0.1 \times 9.4}{10}$

$N_1 = 0.094$

$\frac{N}{5} \times 9.4 = N_1 \times 10$

$N_1 = \frac{0.2 \times 9.4}{10}$

$= \frac{1.88}{10}$

$N_1 = 0.188$

RESULT:-

Standard solution of N/10 NaOH solution is prepared. 0.188

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PRECAUTION:-

Solution should be make up to desired volume after complete dissolving the solute.

During titration the solution should be stirred thoroughly.

Do not take mean of burette readings.

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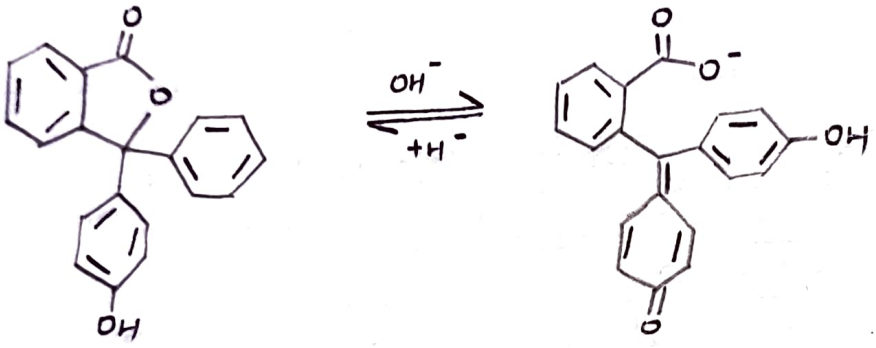
EXPERIMENT-2

REACTION :-

$$OH^{\ominus} + H^{\oplus} \longrightarrow H_2O \quad] P$$
$$CO_3^{2-} + H^{\oplus} \longrightarrow HCO_3^{2-} \quad] M$$
$$HCO_3^{2-} + H^{\oplus} \longrightarrow H_2O + CO_2$$

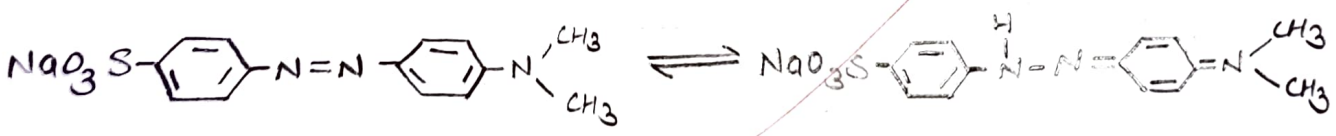
$$OH^{\ominus} + HCO_3^{2-} \longrightarrow H_2O + CO_3^{2-}$$

PHENOLPHTHALEIN STRUCTURE :-



	ACIDIC MEDIUM	ALKALINE MEDIUM
1)	COLOURLESS	(PINK COLOUR)
2)	BENZENOID STRUCTURE	(QUINONOID STRUCTURE)

METHYL ORANGE :-



(BENZENOID STRUCTURE) (QUININOID STRUCTURE)

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EXPERIMENT - 2

OBJECT :- To determine the constituent and amount of alkalinity in the given water sample by titrating it against standard HCl solution (N/20) using phenolphthalein and methyl orange as an indicators.

CHEMICALS/APPARATUS :- N/20 HCl solution, phenolphthalein and methyl orange indicators, sample solution, burette, pipette, beaker, funnel.

THEORY:

The alkalinity in water is due to the presence of hydroxyl ion (OH^-), carbonate ion (CO_3^{2-}) and bicarbonate ion (HCO_3^-) present in the given sample of water. These can be estimated separately by titrating against standard acid (N/20 HCl) using phenolphthalein and methyl orange as indicators.

PROCEDURE:

1) Rinse and fill the burette with N/20 HCl
2) Pipette out 10ml of water sample into a conical flask, add 1-2 drops of phenolphthalein indicator.
3) Add HCl from burette drop by drop till the pink colour just disappear.

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CALCULATION :-

$$N_1V_1 = N_2V_2$$
$$(H_2O) \quad (HCl)$$
$$N \times 10 = \frac{1}{20} \times 2P$$
$$N_1 = \frac{1}{200} \times 2 = 0.01$$

FOR CO_3^{2-}

$$2 \times 1 \times N/20 \times 50 \times 1000$$
$$2 \times 0.05 \times 50 \times 1000$$
$$0.10 \times 5 \times 10^4$$
$$1 \times 10^{-2} \times 5 \times 10^4$$
$$5 \times 10^2 \Rightarrow 500 \text{ mg/L or ppm}$$

FOR HCO_3^{2-}

$$2.1 \times 2 \times 0.05 \times 50 \times 1000$$
$$4.2 \times 0.05 \times 5 \times 10^4$$
$$0.21 \times 5 \times 10^4$$
$$1.05 \times 10^4$$
$$10500 \text{ mg/L or ppm.}$$

4) Now add 1-2 drops of methyl orange indicator to above solution. A yellow colour will appear.
5) Add N/20 HCl drop by drop to it till the yellow colour just changes to red.
6) Repeat this titration until two concordant reading are obtained.

CALCULATION:

FOR CO_3^{2-}

$$N_1V_1 = N_2V_2$$

(For Water Sample) = (For HCl)

Volume of HCl used (V_2) = 2P

Strength = N_1 X equivalent wt of CO_3^{2-} X 1000 ppm

Alkalinity = N_1 X equivalent wt. of $CaCO_3$ X 1000 ppm

FOR HCO_3^-

$$N_1V_1 = N_2V_2$$

(For water sample) = (For HCl)

Volume of HCl (V_2) = (M-P)

Strength = N_1 X equivalent wt of HCO_3^{2-} X 1000 ppm

Alkalinity = N_1 X equivalent wt of $CaCO_3$ X 1000 ppm

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OBSERVATION TABLE

Sr. No.	Volume of sample solutn.	Burette reading				Vol of HCl with phenolphth.	Vol of HCl with methly orange
		Phenolphthalien		Methly orange			
		Initial	Final	Initial	Final		
1)	10	0	1	1	3.1	0.2	1.4
2)	10	0	1.1	1.1	3.0	0.2	1.4

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RESULT:

The alkalinity due to presence of CO_3^{2-} is 500 ppm.

The alkalinity due to presence of HCO_3^{2-} is 10500 ppm

The alkalinity due to presence of OH^- is - ppm.

PRECAUTIONS:

Phenolphthalein indicator should be added first and then methyl orange.

The volume of indicator should same in all the titrations.

The reaction mixture should be shaken properly

Do not take mean of burette readings.

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EXPERIMENT-3

OBJECT :- To determine the temporary, permanent and total hardness of water in a given sample of water by titrating it against standard EDTA solution (N/20) using Eriochrome Black-T as an internal indicator.

APPARATUS/CHEMICALS :- Burette, pipette, concical flask, beaker, measuring cylinder, tripod stand, wire gauze, funnel, filter paper, dropper, standard EDTA solution ammonium buffer solution, Eriochrome Black-T indicator, Hard water.

PRINCIPLE :- When Eriochrome Black-T (indicator) is added to hard water solution at around 10pH, it gives wine red coloured complex is titrated againt EDTA solution. The complex change from wine red to original blue color showing the end point.

EDTA complex with divalent metal cation ($M = Ca^{2+}, Mg^{2+}$) The indicator used is a complex organic compound commercially known as Eriochrome Black-T.

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S.No.	Vol of hard water	BURETTE READING		Vol of EDTA
		INITIAL	FINAL	
1)	10ml	0	8.5	8.5ml
2)	10ml	0	8.5	8.5ml.

S.No.	Vol of hard water	BURETTE READING		Vol of EDTA
		INITIAL	FINAL	
1)	10ml	0	4.5 ml	4.5
2)	10ml.	0	4.5ml	4.5

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PROCEDURE :-

Rinse the burette with N/20 EDTA soln.

Pipette out 10ml of hard water into a conical flask Add 3ml of basis buffer solution.

Add 2-3 drop of indicator Erichrome Black-T. The colour of the soln becomes wine red.

Add drop by drop EDTA soln, wine red to blue colour

Repeat this titration until two concordant reading are obtained.

Take 50ml of hard water in a beaker ~~and~~ and boil it until the volume is reduces to nearby half

Filter it and make it to 50ml by adding distilled water.

Pipette out 10ml of this water and titrate until two concordant reading are obtained.

CALCULATION :-

$$N_1V_1 = N_2V_2$$

(H_2O) (EDTA)

$$N \times 10 = \frac{1}{20} \times 8.5$$

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$N_1 = \frac{0.425}{10}$

$x = \boxed{N_1 = 0.0425}$

∴ Total hardness = 0.0425

$N_1V_1 = N_2V_2$

$N_1 \times 10 = 0.05 \times 4.5$

$N_1 = \frac{0.2250}{10}$

$y = \boxed{N_1 = 0.0225}$

∴ Permanent hardness = 0.0225

Temporary hardness = 0.0425 − 0.0225

= 0.02

RESULTS:-

Total hardness = 0.0425 mg/litre or ppm

Permanent hardness = 0.0225 mg/litre or ppm

Temporary hardness = 0.02 mg/litre or ppm.

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PRECAUTION :-

The glasswares should be properly rinsed with distilled water.

The reaction mixture should be shaken properly.

The end point should be noted correctly.

The pH should be maintained during titration.

The amount of indicator should be same in all titrations.

Do not take mean of burette readings.

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EXPERIMENT-8

OBJECT :- To determine the % of moisture in a given sample of coal by proximate analysis.

MATERIAL REQUIRED :- Coal, Analytical balance, Electric oven, Desiccator, silica crucible, long legged tong, etc.

THEORY :- Proximate analysis is the simplest type of analysis and it informs about the practical utilization of coal. The analysis since the result have no absolute significance.

MOISTURE coal contain two type of moisture :-

Free or surface moisture :- The moisture which is that lost in air drying is called as free moisture or surface moisture.

Internal moisture :- The internal moisture is that that type of moisture which is retained by air dried coal. It is rough indicator of the coal maturity

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OBSERVATION :-

wt of crucible + dry coal = 15.26gm

wt of crucible + wet coal = 19.49gm

wt of crucible + coal sample = 18.98gm

wt of coal sample = 4.23gm

% of moisture = 2 %

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OBERSERVATION AND CALCULATIONS :-

Weight of empty crucible $= x_1$ gm $= 15.26$ gm

Weight of crucible + coal sample $= x_2$ gm $= 19.49$ gm

Weight of crucible + coal sample $= x_3$ gm $= 18.98$ gm

$\therefore$ Weight of coal sample $= (x_2 - x_1)$ gm. $= 4.23$ gm

$\therefore$ Weight of moisture $= (x_2 - x_3)$ gm $= 0.51$ gm

$$\% \text{ of moisture} = \frac{(x_2 - x_3) \times 100}{(x_2 - x_1)}$$

$$= 0.0425 - 0.0225$$

$$= 0.02.$$

RESULT :-

Moisture content in given coal sample is 2 %.

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