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EXPERIMENT NO – 1

OBJECT: To determine the alkalinity of the given water sample.

REQUIREMENTS:

APPARATUS: Burette, burette stand, conical flask, Funnel, Dropper etc.

CHEMICALS: Standard N/10 HCI as an intermediate solution, alkaline water.

INDICATOR: Phenolphthalein and Methyl orange (Internal indicator).

THEORY: The alkalinity in water due to the presence of OH^-, HCO^{3-} and CO_3^{-2} ions. These ions can be estimated by titrating them against standard acid solution using phenolphthalein and methyl orange as indicator.

The possibilities of ions present in water are-

1. OH^- Only
2. CO_3^{-2} Only
3. HCO_3^- Only
4. OH^- and HCO_3^-
5. CO_3^{-2} and HCO_3^-

The possibility of OH- and HCO_3^- together is ruled out as they combine to give carbonate Ion

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

On the same basis all the three ions cannot be present at the same time in a water sample.

REACTIONS:

$$\left.\begin{array}{l} \left.\begin{array}{l} OH^- + H^+ \longrightarrow H_2O \\ CO_3^{-2} + H^+ \longrightarrow HCO_3^- \end{array}\right\} [P] \\ HCO_3^- + H^+ \longrightarrow H_2O + CO_2 \end{array}\right\} [M]$$

END POINTS: The end point of reaction one and two is known as phenolphthalein end point [P], here the pink colour of the solution is just disappearing. The methyl orange end point [M] indicates the completion of all the three reactions and here colour change from yellow to pink.

PROCEDURE:

1. Pipette out 10 ml of the alkaline water sample in to a conical flask.
2. Add 2-3 drops of phenolphthalein indicator.
3. Titrate this sample solution against standard N\10 HCI solution with continues shaking until the pink colour just disappears.
4. Note down the reading from the burette, which corresponds to phenolphthalein end point (P).
5. Now add 2-3 drops of methyl orange indicator to the same solution, continue the titration with standard N\10 HCI.

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$N_1 \times 10 = \frac{1}{10} \times 7.2$

$N_1 = 0.072$

Strength $= N_1 \times 50$

$= 3.6$ g/l

$\frac{7.2}{2} \times 1000 = 3600$ mg/L

$\frac{10.1}{2} \times 1000 = 5050$ mg/L

6. Again note down the total volume of N\10 HCl used from. burette, which corresponds to methyl orange end point (M).
7. Repeat the experiments till concordant readings are obtained.

OBSERVATIONS TABLE:

S.No.	Volume of water sample (ml)	Burette readings (ml)			Volume of HCl used (ml)
		Initial	[P] end point = x	[M] end point = (x+y)	
1.	10ml	0.0	7.4	10.2	x = 7.2
2.	10ml	0.0	7.2	10.1	y = 10.1
3.	10ml	0.0	7.2	10.1	
4.	10ml	0.0	7.2	10.1	

CALCULTIONS:

Volume of sample taken = 10 ml

Volume of N\ 10 HCl used (Phenolphthalein end point) = x ml

Volume of N\10 HCl used (Methyl orange end point) = (x + y) ml

Volume of N\ 10 HCl used in presence of Methyl orange = y ml

Now, $N_1 V_2 = N_2 V_2$

(Sample)= (N/10 HCl)

$N_1 \times 10 = N/10 \times x$

$N_1 = x/100$

Strength in terms of $CaCO_3$ = x/100 ×50 gm/litre = x/2 gm / litre

RESULTS: Phenolphthalein alkalinity = x/2 x1000 mg/litre = 3600 mg/L

Methyl orange alkalinity = (x +y)/2 x 1000 mg/litre = 5050 mg/L

PRECAUTIONS:

(1)The apparatus should be cleaned before the start of experiment.

(2)Phenolphthalein end point and methyl orange end point should be noted carefully.

(3)On adding phenolphthalein, if no pink colour appears, it means that no OH^- or CO_3^{-2} are present in the given water sample. In this case add methyl orange in the beginning.

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EXPERIMENT NO – 2

OBJECT: To determine the percentage of available chlorine in the supplied sample of bleaching powder. (Iodo metrically)

REQUIREMENTS:

APPARATUS: Burette, Pipette, Conical flask, Funnel, Mortar and Pestle.

CHEMICALS: Bleaching powder, Potassium iodide, Glacial acetic acid, Sodium thiosulphate as an intermediate solution (HYPO). Distilled water.

INDICATOR: Freshly prepared starch solution.

THEORY: The bleaching powder in actual is a mixture of calcium hypo chloride $Ca(OCl)Cl.3H_2O$, basic calcium chloride $CaCl_2.Ca(OH)_2.H_2O$ & some free calcium hydroxide $Ca(OH)_2$. But the active constituent is calcium hypo chloride, which produce hypo chlorous acid (HOCl) when react with H_2O. The HOCl is responsible for the disinfectant action of bleaching powder.

The amount of Chlorine liberated by the action of dilute mineral acid (H_2SO_4, HNO_3, HCl) on bleaching powder is known as available chlorine. This available Cl_2 is estimated with the help of iodometric titration. In this method the bleaching powder suspension is treated with dilute mineral acid in presence of excess of potassium iodide (KI). The Cl_2 liberated from $CaOCl_2$ react with KI producing on equivalent amount of I_2. This Iodine get adsorb on the surface of starch (used as indicator) forming a deep blue coloured starch iodine adsorbed complex. When it is titrated against hypo, the adsorbed iodine reacts with hypo to form sodium tetra thionate ($Na_2S_4O_6$).

REACTIONS:

$$CaOCl_2\ 2HCl \longrightarrow CaCl_2 + H_2O + Cl_2$$

$$Cl_2 + H_2O \longrightarrow HOCl + HCl$$

$$CaOCl_2 + 2CH_3COOH \longrightarrow (CH_3COO)_2Ca + H_2O$$

$$2KI + Cl_2 \longrightarrow I_2 + 2KCl$$

$$Starch + I_2 \longrightarrow \text{Starch Iodine adsorbed complex (deep blue)}$$

$$2Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI$$

(Sodium tetra thionate)

PROCEDURE:

1. Pipette out 10ml of given water sample in a conical flask.
2. Add about half test tube KI solution and half test tube of glacial acetic acid to this solution.
3. Titrate the liberated iodine against standard sodium thiosulphate solution with continuous shaking till the appearance of light yellow colour.
4. Now add about 7-8 drops of freshly prepared starch $(C_6H_{10}O_5)n$ solution to it blue colour appears.
5. Titrate again the solution with sodium thiosulphate (Hypo) solution till the disappearance of blue colour (End Point).
6. Note down the volume of Hypo use from the burette.
7. Repeat the experiments till concordant readings are obtained.
8. Weight of Bleaching Powder (W).....gm

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$N_1 \times 10 = \frac{1}{60} \times 6.4$

$N_1 = 0.01067$

Amt. of chlorine $= N_1 \times 35.5 = 0.3787$

% of avl. chlorine $= \frac{N_1 \times 35.5 \times 100}{W} = \frac{0.3787 \times 100}{1.25}$

$= 30.29\%$

END POINT : The deep blue colour is disappeared from solution.

OBSERVATION TABLE:

S.No.	Volume of Water Sample (ml)	Burette Readings(ml)		Volume of Hypo used (ml)
		Initial	Final	
1.	10	0.0	6.7	6.4 V_2 ml
2.	10	0.0	6.5	
3.	10	0.0	6.4	
4.	10	0.0	6.4	

CALCULATIONS:

Volume of Bleaching powder sample taken = 10ml (V_1)
Concordant volume of titration of Hypo used = V_2ml

$N_1 \times V_1 = N_2 \times V_2$
(Sample) (Hypo)

$N_1 \times 10 = N_2 \times V_2$

$N_1 = \frac{N_2 \times V_2}{10}$

Amount of Chlorine $= N_1 \times 35.5 =$ 0.3787 gm/litre

% of avilableChlorine $= \frac{N_1 \times 35.5 \times 100}{W} =$ 30.29%

RESULTS: The % of available chlorine in the given bleaching powder sample is 30.29

PRECAUTIONS:

1. The apparatus should be cleaned with distilled water.
2. The end point of the titration should be carefully noted
3. All the reagents should be freshly prepared
4. After acetic acid titration must be started immediately, as chlorine is librated instantly.

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EXPERIMENT NO – 3

OBJECT: To determine the temporary and permanent hardness in the given water sample using EDTA as standard solution by complexometric titration.

APPARATUS: Conical flask, burette, pipette, beaker, measuring flask.

REAGENTS: 0.1N EDTA solution, buffer solution of pH 10, standard hard water, supplied water sample and boiled water sample.

INDICATOR: Eriochrome Black-T

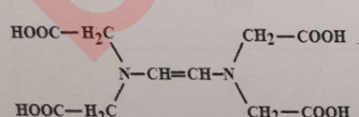
PRINCIPLE: Temporary hardness of water is due to the presence of soluble salts of bicarbonates of calcium and magnesium, while the permanent hardness of water is due to the presence of sulphates, chlorides, nitrates etc. of calcium, magnesium and other heavy metals. Temporary hardness of water can be removed by boiling the water sample while permanent hardness cannot be removed by mere boiling.

Estimation of total hardness by EDTA method is based on the complexometric titration employing a metal ion indicator. In EDTA method, a known volume of given hard water is titrated against EDTA solution using Eriochrome Black-T indicator.

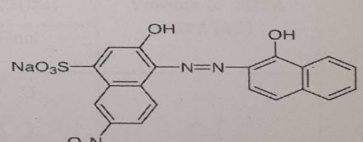
Initially when Eriochrome Black-T indicator is added to the hard water sample, it forms a loose wine red colored complex with Ca^{2+} /Mg^{2+} metal ions present in hard water.

Further when EDTA is added, the loose wine red colored complex completely breaks down and EDTA combines with Ca^{2+} /Mg^{2+} to forms a stable colorless EDTA Metal ion complex and blue Eriochrome Black-T is released, so the solution now appears blue in color.

1. EBT + Ca^{2+}/ Mg^{2+} (Blue) ⟶ [EBT – Ca / Mg] Complex (Wine red)
2. EDTA + Ca^{2+}/ Mg^{2+} ⟶ [EDTA – Ca / Mg] Complex (Colorless, stable)
3. EDTA+ EBT– Ca / Mg ⟶ [EDTA – Ca / Mg] + EBT
   Complex (Blue)



Structure of EDTA



Structure of Eriochrome Black —T

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OCEDURE:

1. Take 10ml. of standard hard water in a conical flask. Add 2ml of buffer solution and 3 drops of Eriochrome Black-T indicator, the colour of the solution turns wine red. Now titrate the solution against EDTA till the color changes from wine red to blue. Repeat the titration for two concordant readings
2. Take 10 ml of the unknown water sample and titrate it by the same procedure as mentioned above.
3. Take 10 ml of the boiled water sample and titrate it by the same procedure as mentioned above.

BSERVATIONS:

A] Titration with standard hard water sample:

S.No.	Volume of Water Sample (ml)	Burette Readings(ml)		Volume of EDTA used (ml)
		Initial	Final	
1.	10	0.0	8.2	
2.	10	0.0	7.9	7.9 v_1 ml
3.	10	0.0	7.9	
4.	10	0.0	7.9	

[B] Titration with given hard water sample:

S.No.	Volume of Water Sample (ml)	Burette Readings(ml)		Volume of EDTA used (ml)
		Initial	Final	
1.	10	0.0	8.3	
2.	10	0.0	8.1	8.1 v_2 ml
3.	10	0.0	8.1	
4.	10	0.0	8.1	

[C] Titration with boiled water sample:

S.No.	Volume of Water Sample (ml)	Burette Readings(ml)		Volume of EDTA used (ml)
		Initial	Final	
1.	10	0.0	5.3	
2.	10	0.0	5.2	5.1 v_3 ml
3.	10	0.0	5.1	
4.	10	0.0	5.1	

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Total Hardness

$8.1 \times \frac{10}{7.9} = 10.25$

$\frac{8.1}{7.9} \times \frac{10}{10} = 1.025$

Total hardness = 1.025×1000

= 1025 mg/l

= 1025 ppm

Permanent hardness = $\frac{5.1}{7.9} \times 1000 = 645$ ppm

Temporary hardness = Total - Permanent

= 1025 - 645

= 380 ppm

CALCULATIONS:-

(A)

1 lit. Solution = 1gm. $CaCO_3$

1000 ml. solution = 1000 mg. of $CaCO_3$

1 ml. solution = 1 mg. of $CaCO_3$

1 ml of standard hard solution = 1 mg $CaCO_3$

v_1 ml of EDTA = 10 ml of standard hard water

= 10 mg $CaCO_3$

So 1 ml of EDTA = $10/v_1$ mg. $CaCO_3$

(B) Total Hardness : -

10 ml of given hard water = v_2 ml EDTA

$= v_2 \times \frac{10}{V_1}$ mg. $CaCO_3$

1 ml of given hard water $= \frac{V_2}{V_1} \times \frac{10}{10}$ mg. $CaCO_3$

$= \frac{V_2}{V_1}$ mg. $CaCO_3$

1000 ml of given hard water $= \frac{V_2}{V_1} \times 1000$ mg. $CaCO_3$

Total hardness $= \frac{V_2}{V_1} \times 1000$ mg. $CaCO_3$

$= \frac{V_2}{V_1} \times 1000$ mg. $CaCO_3$

= 1025 ppm

(C) Permanent Hardness :-

10 ml of boiled water = v_3 ml EDTA

Permanent hardness $= \frac{V_3}{V_1} \times 1000$ mg. $CaCO_3$

= 645 ppm

(D) Temporary Hardness :-

Temporary hardness = Total hardness - Permanent hardness

= 380 ppm

RESULT: - The given water sample contains.

Total hardness = 1025 ppm

Permanent hardness = 645 ppm

Temporary hardness = 380 ppm.

PRECAUTIONS:-

(1) The glass wares should he properly rinsed with distilled water.

(2) The reaction mixture should be shaken properly.

(3) The amount of indicator should be same in all the titrations.

(4) The pH (10+/- 0.1) should be maintained during titration.

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EXPERIMENT NO- 4

OBJECT :

To determine the viscosity of given liquid with respect to water at room temperature by Ostwald's viscometer.

REQUIREMENTS : Ostwald's viscometer, R.D. bottle, rubber tube, stopwatch, beaker, distilled water, experimental liquid.

THEORY :

The measurement of viscosity by viscometer is based on the poiseuille's equation,

$$\eta = \frac{\pi r^4 t P}{8Vl} \quad \text{.....(1)}$$

where, P= hydrostatic pressure of the liquid, V= volume of the liquid of viscosity 'η' flowing in time 't', through the capillary tube of radius 'r' and length 'l'. If t1 and t2 are times required to flow for equal volumes of two liquids through the same length of the capillary tube, then from equation (1) , we have

$$\frac{\eta_1}{\eta_2} = \frac{P_1 t_1}{P_2 t_2} \quad \text{.....(2)}$$

The pressure of the liquid = $h\rho g$

Since, for the two liquids, h and g are the same,

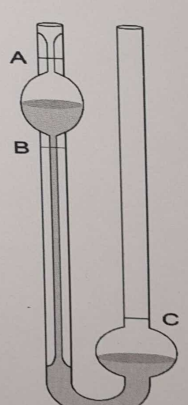
Hence,

$$\frac{\eta_1}{\eta_2} = \frac{\rho_1 t_1}{\rho_2 t_2} \quad \text{.....(3)}$$

Where, ρ_1 and ρ_2 are the densities of two liquids and η_1 and η_2 are their viscosities.

PROCEDURE:

1. Thoroughly clean the Ostwald's viscometer with chromic acid and wash finally with distilled water.
2. A known volume of water is introduced into the bulb-C of the viscometer.
3. The water is then gently sucked into the bulb-A.
4. Allow the water to flow back and note the time (t_2) required for the water to flow through the capillary from the mark A to B.
5. Repeat it for three times.
6. Similarly note down the time of flow of the given liquid (t_1)
7. Again take three concordant readings.
8. Density of water and the liquid is determined by R.D. bottle.



OSTWALD'S VISCOMETER

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$$\rho_1 = \frac{w_2 - w_1}{\text{Vol. of RD bottle}} = \frac{35.34 - 11.47}{25} = 0.9548$$

$$\rho_2 = \frac{w_3 - w_1}{\text{Vol of RD bottle}} = \frac{34.33 - 11.47}{25} = 0.9144$$

$$\eta_1 = \frac{\rho_1 t_1}{\rho_2 t_2} \times \eta_2 = \frac{0.9548 \times 10}{0.9144 \times 16} \times 0.01 = 0.0117 \text{ poise}$$

OBSERVATIONS –

1. Viscosity of water (η_2) = 0.01 poise.
2. Time of flow between A & B.

S.No.	Given liquid		Water	
	Time of flow	Mean (t_1)	Time of flow	Mean (t_2)
1	18 sec		16 sec	
2	18 sec	18 sec	16 sec	16 sec
3	18 sec		16 sec	

3. Density measurement of the liquid (ρ_1) & the water (ρ_2)

Mass of empty R.D. bottle = 11.47 w_1

Mass of R.D. bottle + given liquid = 35.34 w_2

Mass of R.D. bottle + distilled water = 34.33 w_3

Vol. of R.D. bottle = 25 ml

CALCULATIONS –

(i) Density of Liquid ($\rho 1$) $= \frac{w2 - w1}{\text{Vol. of R.D. bottle}} = 0.9548$

(ii) Density of Water ($\rho 2$) $= \frac{w3 - w1}{\text{Vol. of R.D. bottle}} = 0.9144$

Relative viscosity of the given liquid $= \frac{\eta_1}{\eta_2} = \frac{\rho_1 t_1}{\rho_2 t_2}$

Absolute viscosity of the given liquid (η_1) $= \frac{\rho_1 t_1}{\rho_2 t_2} \times \eta 2 = 0.0117$ poise

RESULT - The viscosity of the given liquid ($\eta 1$) = 0.0117 poise.

PRECAUTIONS –

1. Before use the viscometer should be cleaned & dried.
2. Viscometer must be kept in vertical position.
3. Observe accurately the meniscus of liquid & water as they pass through the upper & lower marks.
4. Same volumes of the water & of the liquid should be taken in viscometer.

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EXPERIMENT NO- 5

OBJECT:

To determine the surface tension of a given liquid at room temperature using stalagmometer by drop number method.

REQUIREMENTS:

Stalagmometer, specific gravity bottle, a small rubber tube with a screw pinchcock, distilled water, experimental liquid.

THEORY:

In the drop number method, the number of drops formed by equal volumes of two liquids is counted. Let m_1 and m_2 are the mass of one drop of each of the liquid having densities d_1 and d_2, respectively. If n_1 and n_2 is the number of drops formed by volume (V) of the two liquids, then their surface tensions are related as:

$$\frac{\gamma_1}{\gamma_2} = \frac{d_1}{d_2} \times \frac{n_2}{n_1}$$

One of the liquid is water, its surface tension and density are known. Then, the surface tension of the given liquid can be calculated.

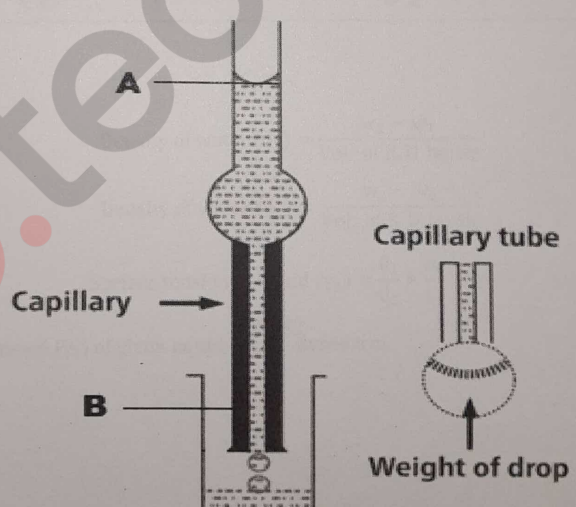
PROCEDURE:

(1)Clean the stalagmometer with chromic acid mixture, wash with water and dry it

(2)Attach a small piece of rubber tube having a screw pinchcock at the upper end of the stalagmometer.

(3) Immerse the lower end of the stalagmometer in distilled water and suck the water 1-2 cm above the mark 'A'. Adjust the pinchcock so that 10-15 drops fall per minute.

(4)Clamp the stalagmometer. Allow the water drops to fall and start counting the number of drops when the meniscus crosses the upper mark A and stop counting when the meniscus passes mark B.



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$$d_1 = \frac{42.07 - 10.81}{31} = 1.0083$$

$$d_2 = \frac{41.03 - 10.81}{31} = 0.9748$$

$$\gamma_1 = \frac{d_1}{d_2} \times \frac{n_2}{n_1} \times \gamma_2 = \frac{1.0083}{0.9748} \times \frac{22}{20} \times 71.18$$

$$= 80.983 \text{ dynes/cm}$$

(5)Repeat the exercise to take three to four readings.

(6)Rinse the stalagmometer with alcohol and dry it.

(7)Suck the given liquid in the stalagmometer and count the number of drops as in case of water.

(8)Take a clean dry weighing bottle. Weigh it with water as well as with liquid.

(9)Note the temperature of water taken in a beaker.

OBSERVATIONS:

(1) Room temperature (t) = 30 °C.

(2) Surface tension of water (γ_2) = 71.18 dynes/cm.

(3)Density measurement of the liquid (d_1) and the water (d_2)

Mass of empty R.D. bottle (w_1) = 10.81 gm

Mass of R.D. bottle + given liquid (w_2) = 42.07

Mass of R.D. bottle + distilled water (w_3) = 41.03

Vol. of R.D. bottle = 31 ml

(4) Drop Count between A & B:

S. No.	Given liquid		Water	
	No. of Drops	Mean (n_1)	No. of Drops	Mean (n_2)
1	20	20	22	22
2	20		22	
3	20		22	

CALCULATIONS:

$$\text{Density of water } (d_1) = \frac{w_2 - w_1}{\text{Vol. of R. D. bottle}}$$

$$\text{Density of water } (d_2) = \frac{w_3 - w_1}{\text{Vol. of R. D. bottle}}$$

$$\text{Surface tension of liquid } (\gamma_1) = \frac{d_1}{d_2} \times \frac{n_2}{n_1} \times \gamma_2$$

RESULT: The surface tension (γ_1) of given liquid is 80.983 dynes/cm.

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EXPERIMENT NO. 6

OBJECT -: Preparation of Urea Formaldehyde resin

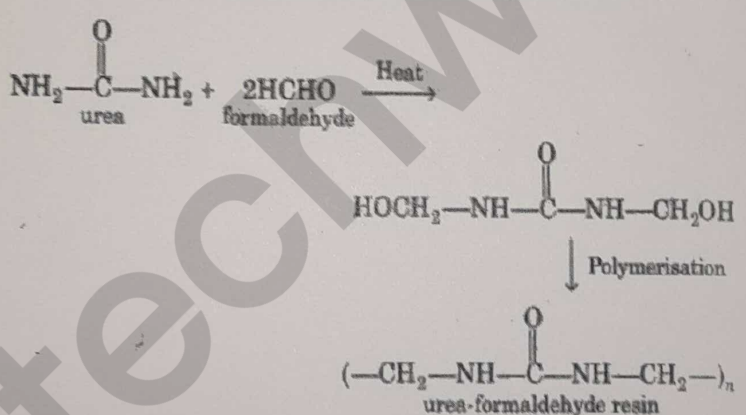
APPARATUS REQUIRED -: Beaker, Glass rod, Funnel etc.

CHEMICALS REQUIRED -: Urea, Formaldehyde (40%), Conc. H_2SO_4

THEORY -: Amino resins are condensation products obtained by the reaction of urea or melamine with formaldehyde. Resins formed by the condensation of formaldehyde with other nitrogen bearing compounds such as aniline and amides also belong to this group. It is commercially important resin. Which is formed by the condensation polymerization reaction of urea with aqueous formaldehyde. Such resins find use in packaging, accessories, unbreakable dishes, water tumblers, buttons, clock cases etc.

It is water soluble and hence is used as sizing agents and textile finishing resin. It is also used in the paper industry to improve the wet strength of paper. In the plywood industry used as adhesive.

The structure of Urea formaldehyde resin is as given below:



PROCEDURE -:

(1) Place 5 ml of 40% Formaldehyde solution in 100ml beaker

(2) Add about 3 gms of urea while stirring until a saturated solution is obtained.

(3) Add a few drops of conc. Sulphuric acid with stirring continuously during the addition.

(4) All of a sudden a voluminous white solid mass appears in the beaker.

(5) When the reaction is complete wash the residue with water and dry the product and calculate the yield of the product formed.

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RESULT -: The Yield of obtained urea formaldehyde resin is = 6.65 grams

PRECAUTIONS -:

(1) The reaction is sometimes vigorous and it is better to be little away from the beaker while addition of Conc. H_2SO_4 and until the reaction is complete.

(2) The reaction should be carried out in fume cupboard.

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EXPERIMENT NO. 7

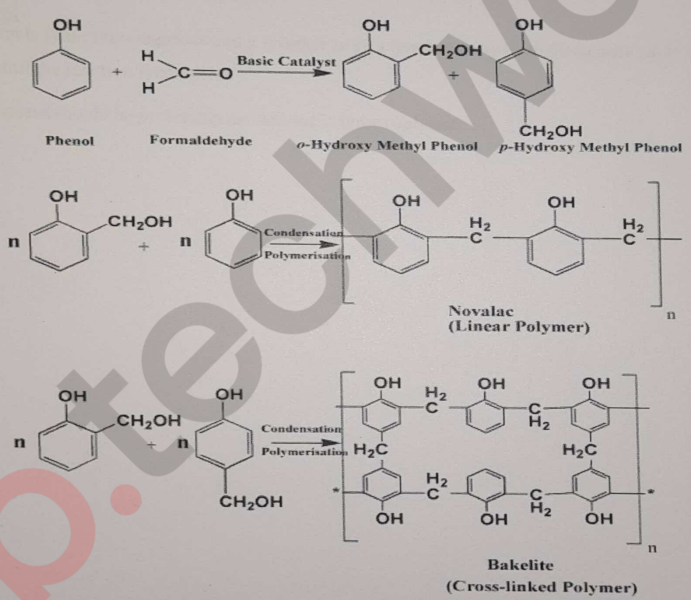
OBJECT -: Preparation of Phenol Formaldehyde resin (Bakelite)

APPARATUS REQUIRED -: Beaker, Glass rod, Funnel, Filter paper etc.

CHEMICALS REQUIRED -: Glacial acetic acid, Formaldehyde (40%), phenol, Conc. HCl

THEORY -: Phenolic resins are condensation polymerization products of phenolic derivatives (like phenol) with aldehyde (like formaldehyde, furfural). Most important member of this class is bakelite or phenol-formaldehyde resin. It is prepared by condensing phenol with formaldehyde in presence of acidic/alkaline catalyst.

The structure of Phenol formaldehyde resin is as given below:



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PROCEDURE -:

(1) Place 5 ml glacial acetic acid in 100ml beaker

(2) Add about 3 ml of 40% Formaldehyde solution.

(3) Add 3ml of phenol

(4) Warp a cloth or a towel loosely round the beaker and add few ml of conc. HCl into the mixture carefully. Within 5 minutes a large mass of pink plastic is formed

(5) The residue obtained is washed several times with distilled water and filtered product is dried and yield is calculated.

Result -: The Yield of the Bakelite is =.....7.68..... gram

Precautions -:

(1)The reaction is sometimes vigorous and it is better to be a few feet away from the beaker while addition of Conc. HCl and until the reaction is complete.

(2)The Experiment should be preferably carried out in fume cupboard.

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EXPERIMENT NO 8

OBJECT: Prepare N/30 Ferrous ammonium sulphate (FAS) and find out the strength and ferrous content in supplied sample of iron solution by titrimetric analysis against standard potassium dichromate solution ($K_2Cr_2O_7$) using potassium ferricyanide$K_3[Fe(CN)_6]$ as an external indicator.

REQUIREMENT:

APPARATUS: Burette, Burette Stand, Pipette, Beaker, Conical flask, Funnel, Glass rod, Tile, Test tube, Measuring flask, Dropper etc.

REAGENTS: Potassium dichromate as intermediate solution, Potassium ferricyanide as external indicator, dilute sulphuric acid and ferrous ammonium sulphate solution

THEORY: $K_2Cr_2O_7$ oxidise ferrous sulphate into ferric sulphate in the presence of Dilute H_2SO_4. The $K_3[Fe(CN)_6]$ used as external indicator gives a greenish blue colour due to the formation of ferrous Ferricyanide $Fe_3[Fe\ (CN)_6]_2$

REACTION:

$$K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$$

$$[2FeSO_4 + H_2SO_4 + (O) \rightarrow Fe_2(SO_4)_3 + H_2O\ ]\times 3$$

$$K_2Cr_2O_7 + 6FeSO_4 + 7H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$$

$$2K_3[Fe(CN)_6] + 3FeSO_4 \rightarrow Fe_3[Fe(CN)_6]_2 + 3K_2SO_4$$

(ferroferricyanide)

(Greenish blue)

PROCEDURE :

1. Pipette out 10 ml of standard iron solution in a conical flask.
2. Add 1/2 Test tube of dilute H_2SO_4 now put 7-8 drops of potassium ferricyanide indicator on white glazed tile.
3. Titrate the solution against standard N/10 $K_2Cr_2O_7$ solution, ml wise.
4. Now take 1 drop of the solution from the conical flask, touch it on the potassium ferricyanide drop on the white tile. If a greenish blue colour appears, then it indicates that the all ferrous ions are not oxidized to ferric ions.
5. Continue titration with N/10 $K_2Cr_2O_7$ solution till no change in the colour of indicator drop. This indicates the end point.
6. Note down the volume of $K_2Cr_2O_7$ used from burette and repeat the process till concordant reading obtained.

END POINT: When there is no change in colour of indicator.

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Wt. of Salt = $\frac{NEV}{1000} = \frac{1}{30} \times \frac{392.12 \times 100}{1000} = 1.307$ gm

Strength = $\frac{\text{weight of salt} \times 10 \times V_2}{V_1} = \frac{1.307 \times 10 \times 9.8}{10.2}$

$= 12.56$ gm/L

Amt. of ferrous content = $\frac{56 \times \text{strength}}{392.12} = \frac{56 \times 12.56}{392.12}$

$= 1.793$ gm

OBSERVATIONS:

[A] For known Solution:

S.No.	Volume of Water Sample (ml)	Burette Readings(ml)		Volume of $K_2Cr_2O_7$ used (ml)
		Initial	Final	
1.	10	0.0	10.4	10.2 V_1 ml
2.	10	0.0	10.2	
3.	10	0.0	10.2	
4.	10	0.0	10.2	

[B] For unknown Solution:

S.No.	Volume of Water Sample (ml)	Burette Readings(ml)		Volume of $K_2Cr_2O_7$ used (ml)
		Initial	Final	
1.	10	0.0	9.9	9.8 V_2 ml
2.	10	0.0	9.8	
3.	10	0.0	9.8	
4.	10	0.0	9.8	

CALCULATION:

Weight of Salt = NEV/ 1000 = 1.307 gm-

Strength of supplied solution = $\frac{\text{weight of salt} \times 10 \times (V_2)}{(V_1)}$

Strength = 12.56 gm/ litre

Amount of ferrous content = $\frac{56 \times \text{Strength}}{392.12}$

Ferrous content = 1.793 gm.

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RESULTS: Strength of supplied water solution is 12.56 gm/litre

Ferrous content in supplied iron solution is 1.793 gm

PRECAUTIONS:

1. In acidic medium $K_2Cr_2O_7$ reacts as an oxidising agent therefore, small amount of dilute H_2SO_4 is added in Mohr's salt solution.
2. The glass apparatus should be cleaned properly with distilled water.
3. The volume of solution taken out for the checking of end point should be very small.

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EXPERIMENT NO - 9

OBJECT: Determination of iron concentration in sample of water by colorimetry using KCNS as colour developing agent.

APPARATUS: Colorimeter, Burette, Pipette, Measuring Cylinder and Beaker, test tubes, Cuvette

CHEMICALS: 0.1% Fe^{3+} -ion solution (with excess HCl), 20% KCNS in distilled water.

THEORY:
Spectroscopy is that branch of science which deals with the interaction of matter with electromagnetic radiations, which are different waves of energy. It covers a wide range of wavelengths or energies and visible light is part of electromagnetic radiations. When monochromatic visible light falls on the homogeneous medium, the intensity of transmitted light is less than that of incident light. A part of incident light has been absorbed.

$$Io=Ia+It$$

Io- intensity of incident light, Ia- intensity of absorbed light, It- intensity of transmitted light

A Spectrometer is a device which detects the percentage transmittance of light radiation, when the light of certain intensity and frequency range is passed through the sample. It is based on Lambert Beer Law which states that "when a monochromatic light is passed through the sample medium, the intensity of that light decreases exponentially with the increase in concentration and thickness of absorbing medium."

$$It=Io\ e^{-ct}$$

Absorbance or optical density is defined as:

$A=\log Io/It= €CT$

A- Absorbance

€- Molar Extinction coefficient or Molar absorptivity

T- Thickness of medium

C- Concentration of the absorbing medium

If the same sample cell i.e. T is constant is used for measurement of absorbance of solution having different concentrations then extent of absorbance (A) is directly proportional to the concentration (C) of the medium.

Thus, if a graph is plotted between A and C, we get a straight line in compliance with Lambert Beer Law. This is known as calibration curve.

For measuring the concentration of given solute, the calibration curve is obtained by measuring the absorbance of standard solution of different concentration. This calibration curve is then used to measure the conc., of unknown solution.

PROCEDURE:

a) **Setting of colorimeter**:

Insert a cuvette containing distilled water in the sample holder adjust the control knob to get zero absorbance

b) **Determination of λmax**:

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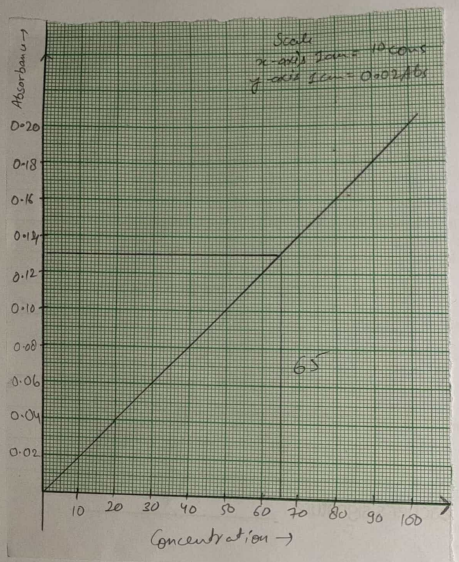

Insert the cuvette containing given colored solution and note the absorbance reading at different wavelengths or filters. At a specific wavelength maximum absorbance will be observed with given coloured solution

c) **Determination of concentration of sample solution**:

Once λ max is fixed. Prepare the following solution from given sample solution and distilled water using a measuring cylinder

Solution. No.	% Conc. of given sample solution	Absorbance at λmax
I	10	0.02
II	20	0.04
III	30	0.06
IV	40	0.08
V	50	0.10
VI	60	0.12
VII	70	0.14
VIII	80	0.16
IX	90	0.18
X	100	0.20
Unknown solution	65	0.13

Note down the value of absorbance of above prepared solutions in series. Plot a graph of values of absorbance so obtained and respective concentration should be a straight line. Now note down the absorbance of unknown concentration solution.

OBSERVATION TABLE:

Solution	I	II	III	IV	V	VI	VII	VIII	IX	X
Given Sample (ml)	1	2	3	4	5	6	7	8	9	10
Distilled water (ml)	9	8	7	6	5	4	3	2	1	0
% concentration	10	20	30	40	50	60	70	80	90	100

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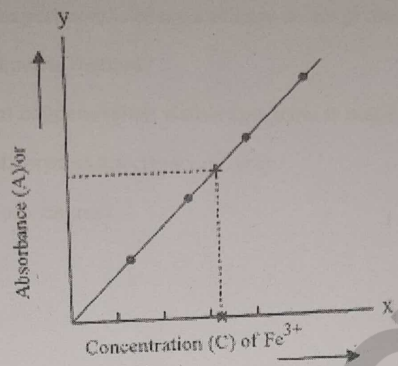
GRAPH:



CALCULATIONS: Concentration of unknown solution from graph= A gm/litre

392 gm of given sample contains=56gm of iron

A gm of given sample contains=56/392×Agm/litre

= (56xA×1000)/392mgm/litre

RESULT: The concentration of Fe (III) in the given sample is obtained from the graph & calculated to be 65 ppm.

PRECAUTIONS:

1) For the preparation of calibration curve, dilute solutions of unknown concentration should be used

2) λmax should be carefully observed

3) Cuvette should be handled carefully as it is fragile

4) Cuvette should be clean and dry from outside

5) Colorimeter should be switched on 15 minutes before the start of experiment

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EXPERIMENT NO – 10

OBJECT: Determine the chloride content in given water sample by Mohr's method.

REQUIREMENTS:

APPARATUS: Burette, conical flask, Pipette, Beaker, Funnel, Dropper etc.

CHEMICAL: Standard N/30 silver nitrate as an intermediate solution.

INDICATOR: Potassium chromate (K_2CrO_4) as an internal indicator.

THEORY: The chlorides are generally present in water in the form of NaCl, $CaCl_2$, $MgCl_2$ etc. These chlorides can be estimated by titrating against standard $AgNO_3$ solution using K_2CrO_4 as indicator. The $AgNO_3$ first react with chlorides present in water forming white colour precipitate as AgCl, the excess drop of $AgNO_3$ react with K_2CrO_4 forming brick red coloured precipitate of Ag_2CrO_4. As the Ksp of AgCl is greater than the Ksp of Ag_2CrO_4, hence AgCl will first precipitate. If chloride ions are present in excess, they can replace chromate ion from Ag_2CrO_4

REACTIONS:

$$NaCl + AgNO_3 \longrightarrow NaNO_3 + AgCl\downarrow$$

$$K_2CrO_4 + 2AgNO_3 \longrightarrow \underset{\text{(Brick red)}}{Ag_2CrO_4} + 2KNO_3\downarrow$$

When chloride is in excess: $Ag_2CrO_4\downarrow + 2Cl \longrightarrow 2AgCl + CrO\text{-}_4$

PROCEDURE:

1. Pipette out 10 ml of water sample in a conical flask.
2. Add 3-4 drops of K_2CrO_4 indicator solution.
3. Titrate it against standard N/30 $AgNO_3$ solution till the brick red colour of Ag_2CrO_4 persists
4. Note down the volume of $AgNO_3$ solution used from burette.
5. Repeat the process till concordant readings are obtained.

OBSERVATION TABLES:

[A] Titration with given water sample:

S.No.	Volume of Water Sample (ml)	Burette Readings(ml)		Volume of $AgNO_3$ used (m1)
		Initial	Final	
1.	10	0.0	9.7	
2.	10	0.0	9.6	9.6
3.	10	0.0	9.6	X_1 ml
4.	10	0.0	9.6	

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Actual vol. of $AgNO_3$ used = 9.6 − 0.2 = 9.4 ml

$N_1 \times 10 = \frac{1}{30} \times 9.4$

$N_1 = 0.03133$ g/L

Strength = 0.03133 × 35.5 = 1.112

chloride content = 0.03133 × 35.5 × 1000

= 1112 mg/L

[B] Titration with distilled water (Blank titration):

S.No.	Volume of Water Sample (ml)	Burette Readings(ml)		Volume of $AgNO_3$ used (m1)
		Initial	Final	
1.	10	0.0	0.3	0.2 X_2 ml
2.	10	0.0	0.2	
3.	10	0.0	0.2	
4.	10	0.0	0.2	

CALCULATIONS:

Volume of given water sample taken = 10 ml

Actual volume of AgNO3 used = $X_1 - X_2$ = x ml

$N_1 \times V_1 = N_2 \times V_2$

$N_1 \times 10 = N_2 \times x$

$N_1 = (N_2 \times x)/10 = (1/30) \times x/10$

Strength of chloride ions = $(x \times 35.5)/30 \times 10$ = 1.112 gm/litre

or Chloride content = $(x \times 35.5 \times 1000)/30 \times 10$ mg/litre

RESULT: The chloride content in the given water sample is 1112 mg/litre.

PRECAUTIONS:

1. The glass apparatus should be cleaned with distilled water.
2. The reaction content should be continuously shaken during titration.
3. Equal volume of water sample and distilled water (for blank correction) should be taken.

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