

Department of Engineering Sciences

Experiment No. : -----

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Estimation of Total Hardness of a given sample of water by EDTA method.

Aim : To determine the total hardness of water sample by EDTA method.

Apparatus : Volumetric flask, conical flask, beaker, measuring cylinder, pipette (10 ml),
Burette, etc.

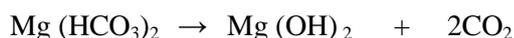
Chemicals : Std. ZnSO₄, 0.05N EDTA solution, buffer solution (pH-10), Eriochrome black –T
indicator, hard water sample, etc.

Theory : Water sample that does not produce lather readily with soap is known as hard water. On heating it leaves deposits of scales on the walls of the container. The total hardness is due to the presence of dissolved bicarbonates, sulphates, chlorides, and nitrates of magnesium and calcium.

The relation between the type of water sample and degree of hardness can be given by following table.

Hardness description of water sample	Hardness as ppm of CaCO ₃ ppm
Soft	0-75
Moderately hard	75-150
Hard	150-300
Very hard	above 300

The hardness that can be easily removed on heating is known as temporary hardness .It is due to bicarbonates of calcium and magnesium which get decomposed on heating as insoluble carbonates and hydroxides which can be removed by filtration.



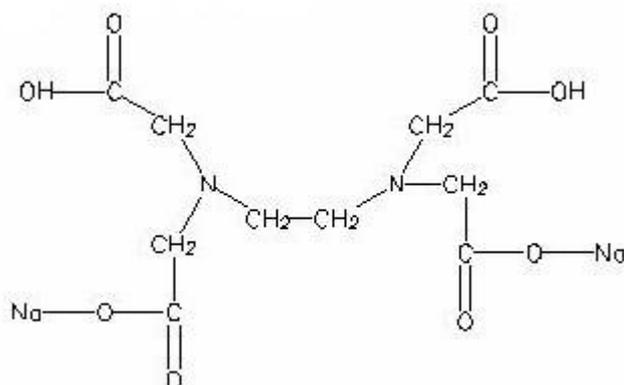
This hardness is also known as bicarbonates hardness. The hardness which cannot be removed by heating is known as permanent hardness or non-carbonate hardness. The sum of temporary and permanent hardness is known as total hardness.

To determine suitability of water for domestic and industrial purpose, type of hardness and hence magnitude of hardness is important. To determine total hardness of given water sample, disodium salt of ethylenediaminetetraacetic acid is used as it forms strong 1:1 complex with divalent metal ions.

EDTA Structure:-

Di-sodium salt of EDTA:-

The disodium salt of EDTA ionizes in water as



The anion is a strong chelating agent and can be represented as H_2Y^{2-}

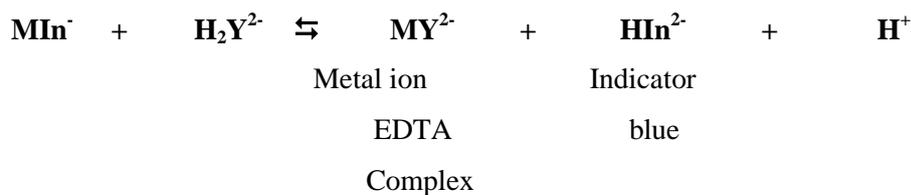
The EDTA forms a stable complex in basic medium, thus alkaline buffer of NH_4OH and NH_4Cl of $pH=10$ are used.

The complex made by H_2EDTA^{2-} with Ca^{2+} ion

In this Complexometric titration, Eriochrome black-T is used as an indicator. This indicator forms less stable wine-red coloured complex with metal ion (less stable w.r.t. metal ion-EDTA complex), which dissociates on titration with EDTA solution. On dissociation, a strong 1:1 metal ion-EDTA complex is formed and indicator is set free which gives blue colour to the solution.



Metal	Indicator	Metal- indicator
ion	blue	complex
		Wine red

**Procedure:-****Part A: preparation of standard 0.05 N ZnSO₄.7H₂O solution.**

1. Weigh accurately 2.51g of pure zinc sulphate on a watch and transfer it into 350 ml beaker.
- 2) Dissolve it in little distilled water; take washings of watch glass also.
- 3) Transfer it into volumetric flask, take washings of beaker also.
- 4) Dilute the solution up to the mark with distilled water.
- 5) Take out all solution in a beaker and stir well to make homogeneous.

Part B: standardization of EDTA solution.

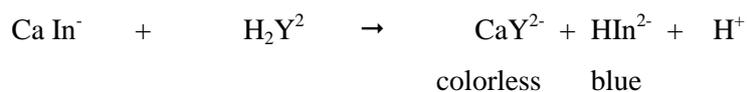
1. Fill the burette with approximately 0.05N EDTA solution.
2. Pipette out 10ml of above standard ZnSO₄ solution in a conical flask and add 2 ml of buffer solution (pH 10) by measuring cylinder and add 3 drops of Eriochrome black T indicator. At the end point, colour changes from wine red to blue.
3. Repeat for three time and note the constant burette reading as 'X' ml.
4. By using this reading, find out exact normality of EDTA solution

Part C: To find out total hardness of given water sample

1. Fill the burette with EDTA solution (N₁ normal).
2. Pipette out 10 ml of water sample in a conical flask, add 2 ml of buffer (pH 10) solution by measuring cylinder and 3 drops of Eriochrome black-T indicator.
3. Titrate the solution against EDTA solution.
4. At the end point, colour changes from wine red to blue.
5. Repeat for 3 times and note constant reading 'Y' ml.
6. By using this reading, calculate total hardness of water sample.

Observation:

Solution in Burette : EDTA Solution.
 Solution in Conical flask : 0.05 N ZnSO₄
 Indicator : Eriochrome Black T

**Observation Table:-**

Readings in ml	I	II	III	Constant reading
Initial				
Final				
Difference				

Calculations:-

$$\text{Total Hardness} = \frac{N_1 \times V_1 \times 50 \times 1000}{V} \text{ ppm of CaCO}_3$$

Where

N_1 = Normality of EDTA

V = Volume of H_2O = 25 ml

V_1 = Mean end point

50 = Equi.wt. of CaCO_3

D) Permanent hardness of given water sample.

1. Take approx. 400ml water in beaker. Boil that water for 20 min. Cool & filter it.
2. Transfer this water in 250 ml volumetric flask & dilute up to mark with distilled water. Now we have to find out hardness of this water.

- Wash, rinse and fill burette -1 with std. EDTA solution.
- Wash, rinse and fill burette -2 with boiled hard water sample.
- Take 10 ml boiled hard water sample into conical flask & add to it 2 ml buffer solution & 3 drops EBT as an indicator. Color becomes wine red.
- Titrate this solution against EDTA from burette-1 till color changes to blue. Note down this burette reading as V_2 ml.
- Repeat the same procedure twice to obtain two readings of V_2 .

Observations:-**Burette 1** :- Std N_1 EDTA solution**Burette 2** :-Boiled water sample**Given** :-Conc. of EDTA solution**To find** :-Hardness of water sample**Indicator** :-Erichrome black T**End-Point** :- Wine red to blue**Reaction** :- $M^{+2} + EBT \rightarrow [M- EBT]^{+2}$ **Observation Table:-**

Reading	I	II	III	Mean
V_2				

Calculation:-

$$\text{Permanent Hardness} = \frac{N_1 \times V_2 \times 50 \times 1000}{V} \quad \text{ppm of CaCO}_3$$

$$\text{Temporary hardness} = \text{Total Hardness} - \text{Permanent hardness}$$

Results:-

1	Exact Normality of EDTA	N
2	Total hardness of water	ppm CaCO ₃ equi.
3	Permanent hardness of water	ppm CaCO ₃ equi.
4	Temporary hardness of water	ppm CaCO ₃ equi.

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Determination of alkalinity of given sample of water.

Aim : Determination of alkalinity of given sample of water.

Apparatus : Pipette, Conical flask, Burette etc.

Chemicals : Water sample, H₂SO₄ solution, phenolphthalein indicator methyl Orange indicator etc.

Theory : Pure water is neutral. Its pH is supposed to be 7. But because of dissociation or hydrolysis of different minerals present in water, conc. of OH⁻ ions in water increases, this makes the water alkaline. Alkalinity of water means total content of OH⁻, HCO₃⁻, CO₃²⁻, in water, which make the water alkaline. In boiler for steam generation, water having high alkalinity leads to caustic embrittlement, precipitation of sludges and deposition of scales. These alkalinities can be estimate volumetrically by titrating water sample against standard acid using methyl orange and phenolphthalein indicators.

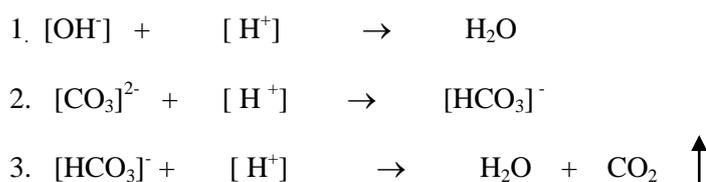
The possible combination of alkalinities in water is:

- 1) Only OH⁻
- 1) Only CO₃²⁻
- 2) Only HCO₃⁻
- 3) OH⁻ and CO₃²⁻ together
- 4) CO₃²⁻ and HCO₃⁻ together

The amount of alkalinities due to the OH⁻, CO₃²⁻, HCO₃⁻ are Calculated from following table.

Alkalinity	Quantity of OH ⁻	Quantity of CO ₃	Quantity of HCO ₃
P = 0	0	0	M
P = ½ M	0	2P	0
P = M	P	0	0
P < ½ M	0	2P	M-2 P
P > ½ M	(2P - M)	2(M-P)	0

Reaction:-



Procedure:-

- 1) First check the pH of water sample with pH Paper.
- 2) Pipette out 10 ml of water sample in a conical flask.
- 3) If pH of the water sample is above 8.5 units then titrate it against 0.1 N HCl using phenolphthalein as an indicator, till pink colour vanishes. Let this reading be V_1
- 4) To this solution add 2 to 3 drops of methyl orange as an indicator and continue the titration till light yellow colour changes to red.
- 5) Let this reading be V_2 from the beginning.
- 6) This procedure is repeated for different sample of water.

Observation Table: - Phenolphthalein Indicator.

Reading	I ml	II ml	III ml	C.B.R.
Initial				
Final				
Difference				

$V_1 =$ ml

Observation Table: - Methyl Orange Indicator.

Reading	I ml	II ml	III ml	C.B.R.
Initial				
Final				
Difference				

$V_2 =$ ml

Calculation:-

This corresponds to 10 ml of water sample.

$$\text{Then phenolphthalein alkalinity } P = \frac{Z \times V_1 \times 50 \times 1000}{V}$$

$$\text{Methyl Orange alkalinity } M = \frac{Z \times V_2 \times 50 \times 1000}{V}$$

Where Z = Normality of HCl

V = Volume of H₂O sample

V₁ = First end point (phenolphthalein)

V₂ = Second end point (methyl orange)

50 = Equi.wt. Of CaCO₃

From the values of P & M calculate conc. of [OH]⁻, [CO₃²⁻] & [HCO₃⁻].

Sr. NO.	Alkalinity type	[OH] ⁻ ppm	[CO ₃ ²⁻] ppm	[HCO ₃ ⁻] ppm
1	P = 0	0	0	M
2	P = M	M	0	0
3	2P = M	0	2P	0
4	2P > M	2P - M	2(M - P)	0
5	2P < M	0	2P	M - 2P

Results:-

1	Conc. Of [OH] ⁻	ppm of CaCO ₃
2	Conc. Of [CO ₃ ²⁻]	ppm of CaCO ₃
3	Conc. Of [HCO ₃ ⁻]	ppm of CaCO ₃

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Determination of dissociation constant of weak acid (acetic acid) using pH meter.

Aim : Determination of dissociation constant of weak acid (acetic acid) using pH meter

Apparatus : pH meter, Burette, Beaker, Pipette, Conical flask, volumetric flask.

Chemicals : Acetic Acid, 0.1N NaOH, Phenolphthalein etc.

Theory : All acids and alkalis dissociate in aqueous solutions to give hydrogen or hydroxyl ions. Some acids such as HCl, H₂SO₄, HNO₃ are completely ionized in aqueous solutions while CH₃COOH, HCOOH ionize to extent only. Hence they are called strong and weak acids respectively. The acidity of a solution depends on the concentration of hydrogen ions produced by the acid.

The acidic and alkaline nature of a solution can be expressed in terms of hydrogen ion concentration. The water weakly ionizes into hydrogen and hydroxyl ions. The ionic product of water, $K_w = (H^+) (OH^-)$ for pure water $(H^+) = (OH^-) = 10^{-7}$

Sorensen proposed pH scale to express acidity of any solution. He defined pH of a solution as negative logarithm of hydrogen ion concentration.

$$pH = \log_{10}(H^+)$$

The pH scale has values between 0 and 14.

H 10^0 10^{-1} 10^{-2} 10^{-3} 10^{-4} 10^{-5} 10^{-6} 10^{-7} 10^{-8} 10^{-9} 10^{-10} 10^{-11} 10^{-12} 10^{-13} 10^{-14}

pH 1 2 3 4 5 6 7 8 9 10 11 12 13 14

The pH of a solution can be accurately measured with the help of pH meter.

Principal of pH Meter:-

Immersing glass electrode in the experimental solution and coupling it with a saturated calomel electrode can measure the emf of the cell. Combined electrodes – having reference electrode and glass electrode - are very common these days. They are convenient to use in laboratory. Instruments called pH meters are available in which the galvanometer readings are directly scaled to read pH values.

The glass electrode acts as the negative electrode and the calomel electrode acts as positive electrode.

Procedure:-

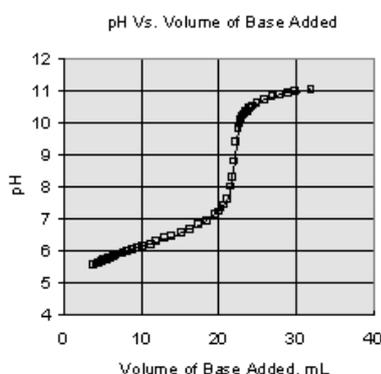
Measurement of pH of unknown sample consists of two steps. First step is to standardize the pH meter against a known standard buffer at room temperature. Then in second step the pH of unknown solution is measured.

Part I: - Standardization of pH meter.

1. Switch on the instrument by turning the control 'ON'. Allow it to warm up for 10 minutes.
2. Adjust the temperature dial to 25⁰C.
3. With the help of special cable provided connect the electrode terminal and 7 pH terminal. Adjust Asymm. Pot. Knob to read 7.0.
4. Now remove the wire from 7 pH and connect to 4.2 pH. Adjust 'SLOPE' to read 4.2.
5. Now remove the wire from 4.2 pH and connect to 9.2 pH. The reading displayed must be 9.2.
6. If the above procedure works then the instrument is 'OK'.
7. If not report it to instructor.

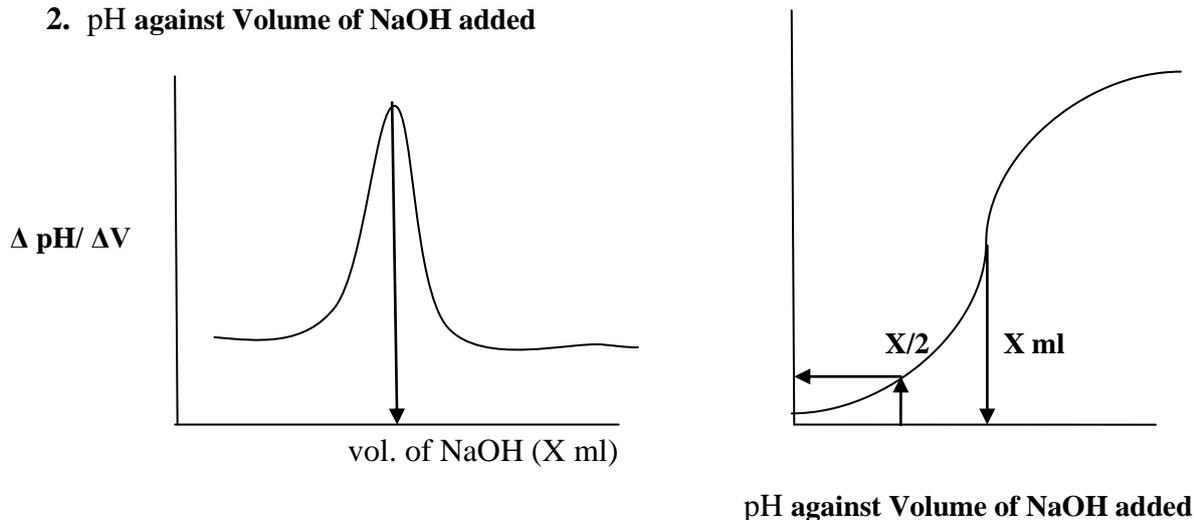
Part II: Acid base titration using pH meter

1. Fill the burette with 0.1 N NaOH.
2. Pipette out 25 ml of given acid solution in a 250 ml beaker and place the magnetic needle in it.
3. Place the beaker on the magnetic stirrer.
4. Wash the pH electrode with distilled water and wipe carefully with filter paper. Dip the electrode in the acid solution of the beaker. 'On' the magnetic stirrer taking care that the needle will not hit the electrode. Note the pH of the solution on the digital display.
5. Arrange the position of the burette such that the solution from burette will fall in the beaker containing acid solution.
6. Add 5 ml of NaOH from the burette to the solution of beaker and record the pH on the digital display.
7. Record the pH of the reaction mixture for each addition of base aliquot as mentioned in the observation table.
8. Plot a graph as given below.



Plot the following graph

1. $\Delta \text{pH} / \Delta V$ against mean V
2. pH against Volume of NaOH added



pH vs ml of NaOH added

From the graph determine the end point of titration. X ml Find half neutralization, X/2 ml

At the half neutralization, X/2 ml find pH of the solution from plot 2

The pH value at half neutralization, point is the pKa value of acid

Calculation:

Thus at the point of half-neutralization, $K_a = [\text{H}_3\text{O}^+]$ or $\text{pKa} = \text{pH}$, where

$$\text{pKa} = -\log K_a,$$

$$K_a = \text{Antilog}(-\text{pKa})$$

Acid	M	K_{a1}	pKa_1
Acetic	60.05	1.80×10^{-5}	4.74

Results:-

The dissociation constant (K_a) for acetic acid is

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To determine maximum wavelength of absorption of CuSO₄, verify Beer's law and find unknown concentration in given sample.

Aim : To determine maximum wavelength of absorption of CuSO₄, verify Beer's law and find unknown concentration in given sample.

Apparatus : Volumetric flask, Colorimeter.

Chemical : 0.1 M CuSO₄ solutions, Ammonia, distilled water.

Theory : Colorimetry deals with measurement of colour intensity. The colour of a substance is due to absorbance of light waves of certain wavelengths. If solution does not absorb light it is transparent and colorless if it absorbs light completely black. The absorption of light by solution results in excitation of electrons in its molecule. The advantage of colorimetric analysis is that it requires much less time and it is more accurate than chemical analysis. Colorimetry is used to determine only low concentration usually less than 2 %.

Fundamental laws of colorimetry:

When light (monochromatic or Heterochromatic) is incident upon a homogenous medium a part of radiant power of incident light (Po) is reflected (Pv) a part is absorbed by medium Pa and remainder transmitted P.

$$P_o = P_r + P_a + P$$

Po – Radiant power of incident light.

Pr – Radiant power of reflected light.

Pa – Radiant power of absorbed light.

P – Radiant power of transmitted light.

If a comparison cell or the same cell is used during analysis the value of Pr, which is very small $\approx 4\%$ can be eliminated for air glass interfaces $P_o = P_a + P$.

Colorimetry is based on following fundamental laws:

1. Lambert's law

2. Beer's law

Combined form of these laws is called as Beer Lambert's law.

1) Beer's Law:-

When a beam of light is allowed to pass through a transparent medium the rate of decrease of radiant power with the concentration of medium is directly proportional to radiant power i.e absorbance of solution is directly proportional to concentration of solution.

2) Lambert's Law:-

This law can be stated as when a beam of monochromatic light is allowed to pass through a transparent medium the rate of decrease of radiant power with thickness of the medium is directly proportional to thickness of medium or path length.

3) Beer-Lambert's Law

The Lambert's law states that

Absorbance = constant x thickness of medium

The Beer's law states that

Absorbance = constant x concentration of medium.

The combined law states that,

Absorbance = constant x thickness of medium x concentration of medium

i.e $A = \text{constant} \times a \times c$.

Thus laws states that,

1) When a beam of monochromatic light is allowed to pass through a transparent medium, the absorption of medium is directly proportional to thickness & concentration of medium ($A=bc$). when concentration is expressed g/l and thickness in cm, the constant in above equation is called as absorption and represented by the symbol 'a'. Thus $A = a b c$.

When the concentration is expressed in mol/lit and thickness in cm, the constant in above equation is called as absorptivity and represented by symbol 'ε'.

Thus $A = \epsilon bc$.

$A = \epsilon$ when $b= 1$ cm and $c= 1$ mol/lit.

The molar absorptivity is absorption of solution measured in a cell of one mole per lit. It is a constant independent of concentration, length of path & radiant power of incident light.

Now for the given thickness of medium & system the relation $A = \text{constant} \times c$ or $A \propto c$ and therefore the Beer –Lambert's law can also stated as.

2) For the given system and the thickness of the medium. The absorbance of solution is directly proportional to concentration of an absorbing species.

3) **Procedure:-****Part I:- Preparation of different conc. of 0.01 M CuSO₄ .NH₃ Solution****Observation Table:-**

Flask No.	Conc. Of CuSO ₄	0.01 M CuSO ₄ solution for dilution	NH ₃ added (ml)	Total volume after dilution
1	0.006	60 ml	25 ml	100 ml
2	0.004	40 ml	25 ml	100 ml
3	0.002	20 ml	25 ml	100 ml
4	0.001	10 ml	25 ml	100 ml
5	Unknown	Given volume	25 ml	100 ml

Part II:- Determination λ_{\max}

To select filter , use 0.006 M CuSO₄ -NH₃ complex solution from above solution and blank solution as liquor ammonia diluted to 100 ml using distilled water try all the filters available in laboratory & select one with which the experimental solution has maximum absorbance call the wave length of this filter has λ_{\max} .

FILTER NO	WAVE LENGTH IN n.m.	ABSORBANCE
	400	
	420	
	500	
	550	
	600	
	620	
	670	
	700	

Part II:- To find out Absorbance for known and unknown solution.

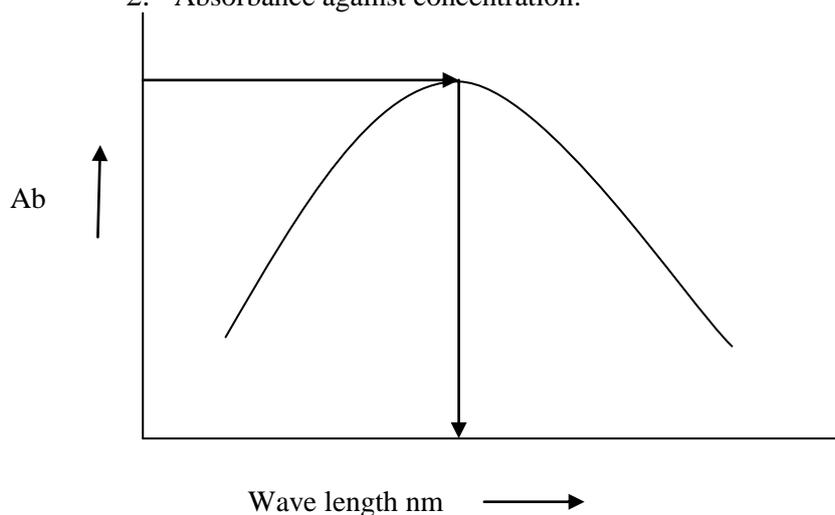
1. Place the appropriate filter in the filter compartment.
2. Take reference (blank solution- 25 ml NH_3 diluted to 100 ml d/w.) in cuvette, clean it from outside and insert it into sample compartment.
3. Adjust the meter reading to 100 % transmittance with the help of coarse and fine set knob.
4. Remove the cuvette, wash it with distilled water. Put 0.06 M $\text{CuSO}_4 \cdot \text{NH}_3$ complex solution from flask no.1 in it. Clean it from outside and % transmittance of the solution on metal scale.
5. Repeat the procedure remaining known and unknown solution to record their absorbance value.

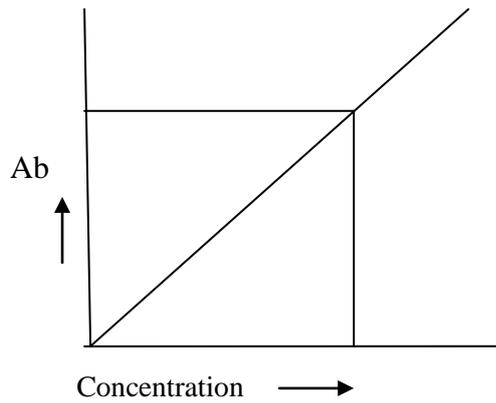
Observation Table:-

Flask No	Conc. Of CuSO_4	Absorbance or (A) Optical density
1	0.006	
2	0.004	
3	0.002	
4	0.001	
5	Unknown	

Graph

1. Absorbance against Wave length
2. Absorbance against concentration.





By knowing the absorbance of unknown solution the corresponding concentration value can be determine graphically.

Result:-

1. Maximum wavelength of absorption of CuSO_4 _____
2. Concentration of Unknown CuSO_4 solution _____ ml.

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Titration of mixture of weak acid and strong acid with strong base using Conductometer.

Aim : Titration of mixture of weak acid and strong acid with strong base using Conduct meter.**Apparatus** : Conductivity meter, conductivity cells, Beakers, Pipette, Burette**Chemicals** : Oxalic acid Solution (0.1N), NaOH Solution, HCl, Phenolphthalein Indicator

Theory : The conductivity of HCl is very high due to mobility of H^+ ions in it. When a solution of strong alkali is added to the solution of strong acid, the highly conducting H^+ ions are replaced by Na^+ ions is considerably smaller than H^+ and hence addition of alkali to the acid always accompanied by a decrease in conductivity. When all the H^+ are replaced that is after the end point, further addition of NaOH results in increase in conductivity. From the plot of the conductivity Vs ml of NaOH added, the end point can be calculated

Procedure: -**Determination of Strength of Given Hydrochloric acid & Acetic acid Solution:-**

10 ml of given unknown HCl solution is taken in a 100 ml beaker. To this 10ml of unknown Acetic Acid solution is added, the electrode of the cell is dipped in the solution. The conductivity of solution is noted before adding the alkali. The standardized sodium hydroxide is added from a burette with 1ml of increment and by shaking thoroughly, the contents of the beaker the conductivity values are noted. The values of observed conductivities are plotted against volume of sodium hydroxide. The concentration of hydrochloric acid is determined using the end point from the graph.

Precautions: after 2 each addition of titrant from the burette the solution should be thoroughly stirred and then the reading should be taken.

Determination of equivalent point of HCl and CH₃COOH using NaOH as a titrant :-

Sr.No.	Volume of NaOH	Conductivity (ohm ⁻¹)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		

Calculations:

- The Normality of the **HCl** acid can be determined by using normality equation

$$\begin{array}{cc} \textit{Acid} & \textit{Base} \\ N_1 V_1 = & N_2 V_2 \end{array}$$

where, N₁ = Normality of the acid

N₂ = Normality of base.

V₁ = Volume of acid taken for the titration

V₂ = Volume of base added till the equivalence point (Taken from the plotted Graph).

$$N_1 = \frac{N_2 V_2}{V_1}$$

$$\begin{aligned} \text{Strength of the HCl} &= N_1 \times \text{Equivalent weight of the HCl acid gm/l (36.5)} \\ &= \quad \quad \quad \text{gm/l} \end{aligned}$$

2. The Normality of the CH_3COOH acid can be determined by using normality equation

$$\begin{array}{cc} \textit{Acid} & \textit{Base} \\ N_1 V_1 & = N_2 V_2 \end{array}$$

where, N_1 = Normality of the acid

N_2 = Normality of base.

V_1 = Volume of acid taken for the titration

V_2 = Volume of base added till the equivalence point (Taken from the plotted Graph).

$$N_1 = \frac{N_2 V_2}{V_1}$$

$$\begin{aligned} \text{Strength of the } \text{CH}_3\text{COOH} &= N_1 \times \text{Equivalent weight of the acid gm/l (60)} \\ &= \quad \quad \quad \text{gm/l} \end{aligned}$$

Result:

The strength of the given Hydrochloric acid solution is ----- gm/l.

The strength of the given Acetic acid solution is ----- gm/l.

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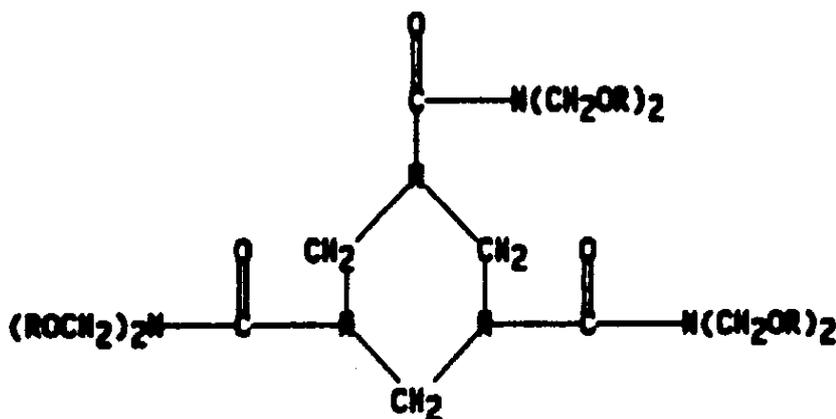
Preparation of urea formaldehyde resin and its characterization.

Aim : Preparation of Urea formaldehyde resin.**Apparatus** : 1) Beaker 2) Glass rod 3) watch glass**Chemicals** : 1) 40% Formaldehyde 2) Urea 3) conc.H₂SO₄**Procedure** :-

1. Take 20 ml of 40% formaldehyde solution in a 100 ml beaker.
2. Add about 1 gm of Urea with constant stirring till saturated solution is obtained.
3. Pour little quantity of this saturated solution on a watch glass.
4. Add few drops of conc. H₂SO₄ in this solution carefully.
5. Within 2 minutes a white solid mass appears in the watch glass.
6. Wash the white solid with water and dry it in the folds of filter paper.
7. Place it in plastic bag and staple to your sheet.

Observation table:-

1	Colour of polymer prepared	
2	Weight of plastic beaker W ₁ g	
3	Weight of plastic beaker with product W ₂ g	

Reaction:-**Calculation:-**

$$\text{Weight of polymer} = W = (W_2 - W_1) = \quad \text{g}$$

Characterization:-

Perform following characteristic tests.

- 1) **Flame test:** Hold a piece of polymer in pair of tongs on flame.

Observation: Product burns with sooty flame i.e. polymer is aromatic.

- 2) **Solubility test:**

Crush the polymer formed using glass rod. Take little quantity of the Polymer in the test tube and add water to it. Warm if required (Do not boil)

Observation: Polymer is water insoluble

Result Table :-

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Determination of molecular weight of macromolecule polystyrene by viscosity measurements.

Aim: To determine the molecular weight of a high polymer using its solution with different concentrations.

Apparatus: Ostwald's viscometer, stop watch, pipette, Iron stand.

Chemicals: Pure solvent, 0.1%, 0.2%, 0.3%, 0.4%, 0.5% polymer solutions, Acetone.

Theory:

Viscosity measures the resistance of flow offered by fluid layers to each other during the flow. The coefficient of viscosity (η) is the force required per unit area of the layer to maintain a unit difference of velocity between two parallel layers of the liquid one centimeter apart at the given temperature. It is measured in 'poise' in CGS unit. Viscosity determinations are very much important in the study of **high polymers**. Using simple viscosity measurements an average molecular weight of the polymer may be determined. According to **Poiseuille's** equations,

$$V = \frac{\pi \rho r^4 t}{8 \eta I} \quad \text{or} \quad \eta = \frac{\pi \rho r^4 t}{8 V I} \dots\dots\dots 1$$

Where

(η). = coefficient of viscosity (poise)

P = difference in pressures at the two ends of the tube to maintain uniform rate of flow.

r = radius of the capillary tube (cm)

t = time (second) required to flow 'V' volume.

v = volume (ml) of the liquid flowing out of the capillary in time 't' seconds.

For two liquids of the same volume and with the use of same viscometer the equation can be written as

$$\eta_1 = \frac{\pi \rho r^4 t}{8 V I} \quad \text{and} \quad \eta_2 = \frac{\pi \rho r^4 t}{8 V I}$$

$$\frac{\eta_1}{\eta_2} = \frac{p_1 t_1}{p_2 t_2} = \frac{d_1 t_1}{d_2 t_2} \dots\dots\dots 2$$

As pressure is directly proportional to the density of the liquid ($P \propto d$)
 By comparing the viscosities of pure solvent and the solution of polymer in the same solvent, the molecular weight of high polymer is given by following expression-

$$\frac{[\eta_1 / \eta_0 - 1]}{C} = KM^\alpha \dots\dots\dots 3$$

- Where M = Molecular weight of high polymer
- K = Constant for a given polymer for the particular solvent and temperature
- α = Function of the geometry of the molecules.
- C = Concentration of polymer in g 100 ml⁻¹ solution.
- (η) = Viscosity of solution of high polymer.
- (η_0) = Viscosity of pure solvent

As we know

$$[\eta_1 / \eta_0 - 1] = \eta_{SP} = \text{specific viscosity}$$

$$\frac{\eta_{SP}}{C} = KM^\alpha$$

This equation is valid only for very dilute solution (less than 1 %)

The graph of η_{SP}/C against 'C' is extrapolated to zero concentration. The extrapolated value is known as the **intrinsic viscosity (η)**.

The intrinsic viscosity (η) can also be determined from the plot of $2.303/C \log \eta / \eta_0$ against C. The plot is straight line giving intercept on y- axis. The intercept value describes intrinsic viscosity (η). Viscosity of a polymer solution depends on concentration and size (i.e., molecular weight) of the dissolved polymer. By measuring the solution viscosity we should be able to get an idea about molecular weight. Viscosity techniques are very popular because they are experimentally simple. The molecular weight of polymer is given by following equation known as Mark-Houwink expression.

$$(\eta) = KM^\alpha \dots\dots\dots 4$$

where a is some constant that depends on the polymer-solvent pair and on temperature. Values of a are typically between 0.5 and 0.8.

The value of k and α , are listed below.

For Polystyrene K = 2 x 10⁻⁴ and α = 0.4

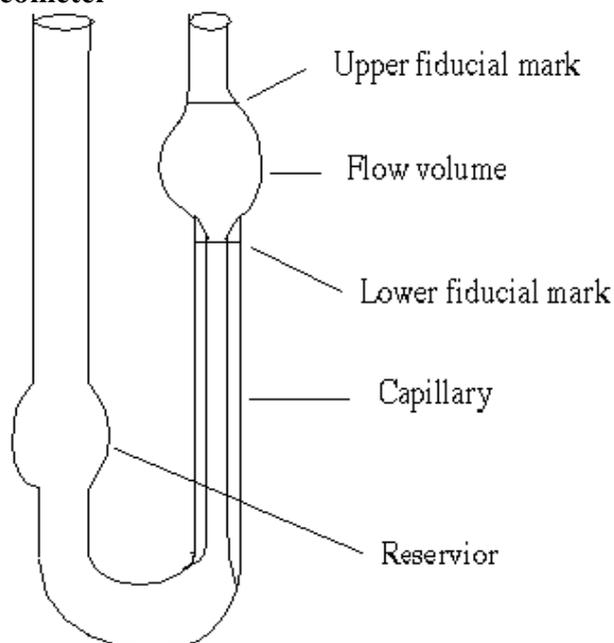
Procedure : Perform the experiment in two parts:

Part I: To determine the time of flow for a given pure solvent.

- Take a clean and dry viscometer and clamp it in a perfectly vertical position to an iron stand.
- Keeps the viscometer in a thermostat or beaker containing water in order to retain constant temperature.
- Introduce exactly 20 ml of pure solvent (water) to the viscometer from clamp side.
- Suck the liquid by means of rubber tube, above the mark 'X'.
- Release the liquid and start the stopwatch when liquid leaves upper mark 'X'.
- Stop the stopwatch when liquid reaches lower mark 'Y'.
- Take three time of flow (in seconds) for a liquid flowing from 'X' to 'Y'.
- Record three readings; Remove the pure solvent from the viscometer.
- Then wash the viscometer carefully with acetone. Dry it by passing current of hot air (hair dryer can be used).

Part II: To determine the time of flow for a given solution of polymer.

- Introduce exactly 20 ml of the 0.1% solution of unknown polymer in to the viscometer from clamp side.
- Repeat the procedure from (4) to (6) as given in part I
- Record the time of flow (in seconds) for 0.1% solution of polymer.
- Similarly repeat the procedure for 0.2%, 0.3%, 0.4%, 0.5% s solutions of polymer and the time of flow.

Figure: Ostwald's Viscometer**Observation Table-Part-I:**

Solvent	Time of flow (seconds)			Mean Time t_0 (seconds)
	1	2	3	

Observation Table-Part-II:

Conc. of polymer	Time 't' (sec)			$t / t_0 = \eta / \eta_0$	$\eta / \eta_0 - 1 = \eta_{SP}$	η_{SP}/C	$(2.303 / C) \times \log \eta / \eta_0$
	1	2	Mean				
0.1%							
0.2%							
0.3%							
0.4%							
0.5%							

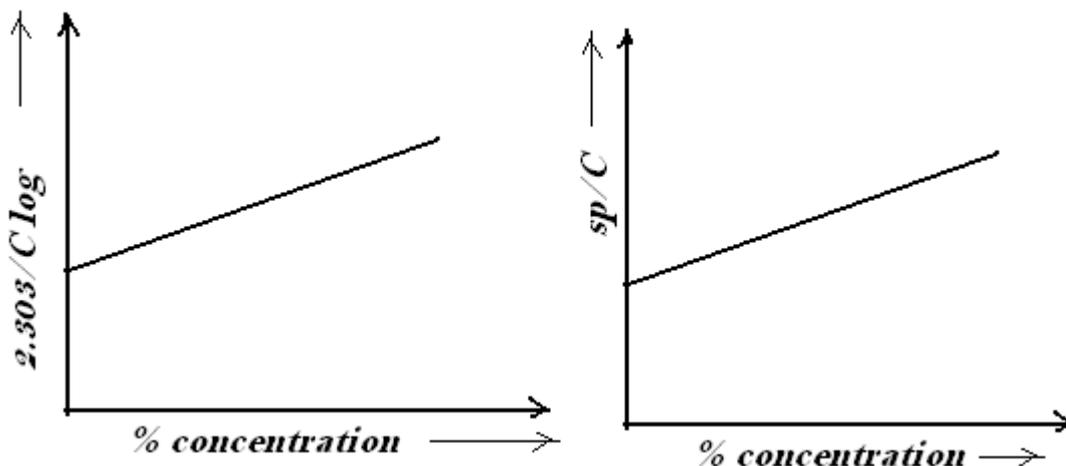
Graphs:

- Plot the graph of η_{SP}/C against % concentration of polymer. Find out intrinsic viscosity (η) by extrapolation to zero concentration.
- Also plot the graph of $(2.303/C) \log \eta/\eta_0$ against % concentration of polymer. Find out intrinsic viscosity (η) by extrapolation to zero concentration. Ask for values of K and α .
Thus knowing the values of (η) intrinsic viscosity of the given polymer from the graph and the constant k and α (supplied), the molecular weight of high polymer can be calculated by using the formula,

$$\begin{aligned}
 (\eta) &= KM^\alpha \text{ i.e.} \\
 M^\alpha &= (\eta) / K \\
 \alpha \log M &= \log (\eta) / K \\
 \log M &= [\log (\eta) / K] / \alpha \\
 M &= \text{antilog} [\log (\eta) / K] / \alpha
 \end{aligned}$$

Knowing (η), K, and α , find log M and then molecular weight of Polymer M.

Where $\alpha = 0.4$ and $K = 2 \times 10^{-4}$ for Polystyrene.

**Calculations:**

1) **From Graph 1 :**

$$\log M = [\log (\eta) /K] / \alpha$$

1) **From Graph 2 :**

$$\log M = [\log (\eta) /K] / \alpha$$

Result Table:

Molecular Weight	From graph(I)	From graph(II)
Polystyrene		

Questions:-

- 1) Define Viscosity.
- 2) Why polymer solutions viscous in nature? Which apparatus is used to determine Viscosity?

Department of Engineering Sciences

Experiment No. : -----

Date of Performance: -----

Division: -----

Roll No. :-----

Proximate analysis of coal.

Aim : Estimation of moisture, volatile matter and ash content in a given sample of coal.

Apparatus : Coal, Silica crucible, oven, weighing apparatus, burner etc.

Theory : In order to certain the quantity of coal proximate and ultimate analysis of coal is done. Determine proximate analysis is determination of moisture content, volatile matter, ash content and fixed carbon content. Moisture content is the loss in weight of coal when heated in crucible at 110⁰C for one hour. During Burning of coal, moisture evaporates and is removed as steam .same amount of heat is required for this which is taken during burning of fuel. Calorific fuel is reduced due to presence of moisture of fuel. Ash content of coal is the weight of residue left after burning known quantity of dry coal in coal crucible at 700⁰ C in muffle furnace's reduces the heating value of coal .volatile matter evolve and its composition depend on quality of coal and also on temperature, rate of heating and time for which heating is continued for comparable results. Conditions specified by ASTM are followed. Volatile matter is percentage loss in weight of coal when it is heated in absence of air for exactly 7 minutes at 950 ± 20⁰C in a crucible of standard dimension. The coal is with high volatile matter are not suitable for production of coal gas. The furnace design depends upon volatile matter. Quality of coal varies with its volatile matter.

Procedure:-

Part A: Moisture content

Take some known quantity of powdered coal sample in previously weighed silica crucible. Heat the sample in a electric oven at about 100 to 110 °C for about one hour. Take out the crucible cool it in desiccators and weigh it. Repeat the procedure of heating cooling and weighing the crucible till constant weight is obtained. Note down the weight.

Part B: Ash content

Take some known amount of powdered dry coal sample in a previously weighed silica crucible. Heat the crucible in a muffle furnace at 700 to 800 °c .Stirrer the residue and ignite it for half an hour at same temperature .Moisten it with two drops of alcohol continue ignition for another is minutes, cool in desiccators and weight repeat ignition cooling and weighting till constant .weight is obtained.

Calculations : Part A: Moisture Content

S.N.	Description	Value	Unit
1	Weight of empty crucible		W ₁ g
2	Weight of crucible & coal		W ₂ g
3	Weight of coal		(W ₂ -w ₁) g
4	Weight of crucible & coal ,coal after heating		W ₃ g
5	Weight of coal after heating		(W ₃ - W ₁) g

Loss in weight due to heating = moisture constant of sample

$$= (W_2 - W_1) - (W_3 - W_1) \text{ g}$$

$$= (W_2 - W_3) \text{ g}$$

=

=

% moisture = (loss in weight / weight of coal) X 100

$$= \frac{(W_2 - W_3)}{(W_1 - W_1)} \times 100$$

=

Part B: - Ash Content

S.N.	Description	Value	Unit
1	Weight of empty crucible		Y ₁ g
2	Weight of crucible with dry coal		Y ₂ g
3	Weight of dry coal		Y ₂ -Y ₁ g
4	Weight of crucible & ash left		Y ₃ g

5	Weight of ash		$Y_3 - Y_1$ g
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% of Ash = (weight of ash formed / weight of dry coal) x 100

$$= \frac{(Y_3 - Y_1)}{(Y_2 - Y_1)} \times 100$$

Results:-

1.	Percentage of moisture	
2.	Percentage of ash	