SINHGAD ACADEMY OF ENGINEERING
CHEMISTRY ENGINEERING DEPARTMENT

Date:

1. HARDNESS OF WATER

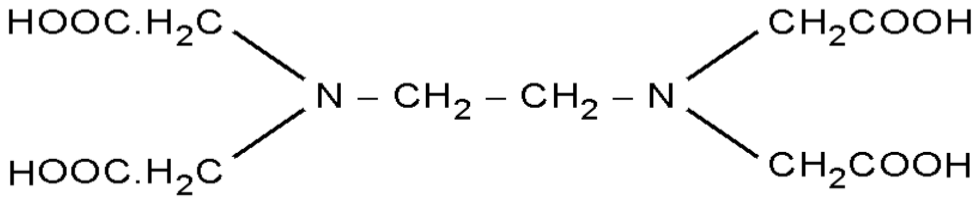
AIM: To determine total hardness of the given sample of water by using EDTA method.

APPARATUS: Titration apparatus.

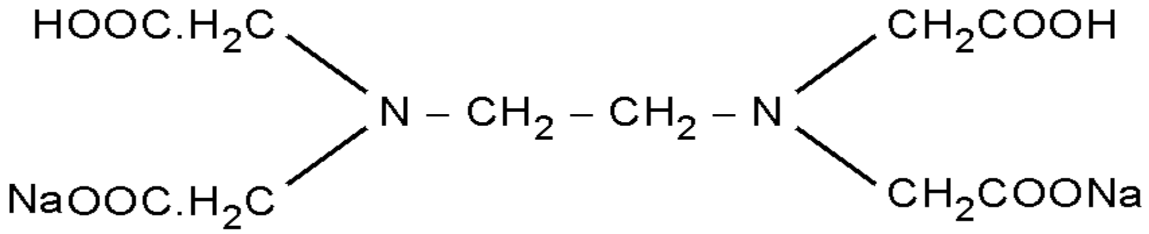
CHEMICALS: EDTA, Buffer solution, EBT indicator, $ZnSO_4$, Hard water sample

PRINCIPLE: The total hardness of water is mainly due to the dissolved salts of calcium and magnesium. These Ca^{2+} and Mg^{2+} ions readily form complexes with EDTA (Ethylene Diamine Tetraacetic acid). EDTA is tetraprotic (tetrabasic) salt. And its structure is

Structure of EDTA :-



Structure of Na2EDTA (disodium salt of ethylene diamine tetra acetic acid :-

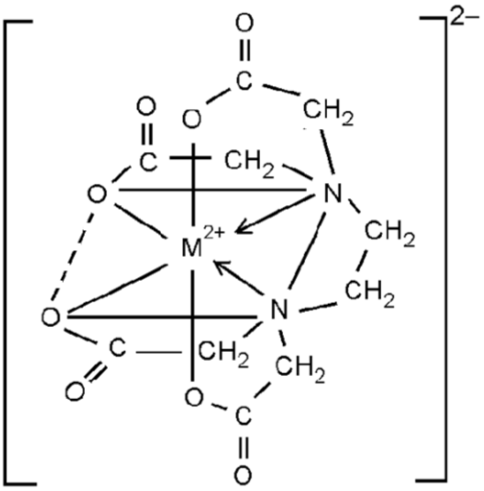


It is usually written as H_4Y. Actually the disodium of EDTA (Na_2H_4Y) is used in the titration and called as EDTA. Both nitrogen and oxygen atoms in EDTA are electron donors, hence it forms chelate complexes with many metal ions. It reacts with metal ions in the molar ratio of 1:1. The reaction between divalent metal ion; (e.g. $Ca^{2+,}$ $Mg^{2+,}$ $Zn^{2+)}$ can be represented as :

$$M^{2+} + H2Y2^{-} \rightleftharpoons (MY)^{2-} + 2\,H^{+}$$

Most of the cations form stable and soluble complexes with EDTA. Since H^+ ions are produced during the formation of a complex, the stability of EDTA complexes depends upon the pH of the solution The structure of the metal EDTA complex is shown below.

- 3D structure M-EDTA complex ion (Hexadentate)



The quantitative estimation of the metal ions is carried out by titration their solution against EDTA using suitable indicator at proper pH of the solution. These titrations are usually called complexometric. The water sample having Ca^{2+} and Mg^{2+} ions is titrated against EDTA solution using Erichrome Black –T as an indicator. This gives total harness of the given sample of water.

This experiment involves following steps

(A) Preparation standard 0.01 M $ZnS0_4$ solution.
(B) Standardization of give EDTA solution.
(C) Estimation of total harness of water sample.

PROCEDURE :

Part A: Standardization of given EDTA solution

1) Fill the burette with approximate 0.01 M EDTA solution.
2) Take 10 ml of 0.01 M $ZnSO_4$ solution by pipette in a 100 ml conical flask.
3) Add 5 ml buffer solution (pH = 10) with the help of a cylinder. Then add 5 drops of Erichrome Black T indicator. Shake the flask. Wine red colour is developed.
4) Add from the burette EDTA solution drop by drop till wine red colour of the solution changes to blue. This is the endpoint of titration.
5) Record the burette reading. Take three such readings. Note the constant burette reading (X ml).
6) From the burette readings find the molarity of EDTA.

Part B: Determination of total hardness

1) Fill the burette with standardized EDTA solution.

2) Take 10 ml hard water sample solution by pipette in a 100 ml conical flask
3) Add 5 ml buffer solution (pH = 10) with the help of a cylinder. Then add 5 drops of Erichrome Black T indicator. Shake the flask. Wine red colour is developed.
4) Add from the burette EDTA solution drop by drop till wine red colour of the solution changes to blue. This is the endpoint of titration.
5) Record the burette reading. Take three such readings. Note the constant burette reading (Y ml).
6) From the burette readings find the total hardness of given water sample.

OBSERVATION TABLE:

Part A : Standardization of EDTA solution

Given : 0.01 M $ZnSO_4$ solution
To find : Exact molarity of EDTA solution
Burette Solution : EDTA solution
Pipette Solution : 0.01 M $ZnSO_4$ solution
Buffer solution : (pH =10) 5 ml
Indicator : Erichrome Black T
Endpoint : Wine red to blue

EQUATION:

$$Na_2EDTA + Zn^{2+} \longrightarrow [EDTA\ (Zn^{2+})\ complex]^{2-} + 2\ Na^{+}$$

Reading in ml	Pilot Reading	I (ml)	II (ml)	III (ml)	C.B.R. (X ml)
Initial					
Final					
Difference					

CALCULATIONS:

$$EDTA = ZnSO_4$$
$$M_1V_1 = M\,V$$

∴ Exact Molarity of EDTA (Z) = (use in calculation)

OBSERVATION TABLE:

Part C: Determination of total hardness

Given : Standardized EDTA solution
To find : Total hardness of water
Burette Solution : EDTA solution
Pipette Solution : Hard water sample solution (Vml)
Buffer solution : (pH =10) 5 ml
Indicator : Erichrome Black T
Endpoint : Wine red to blue

EQUATION:

$Na_2EDTA + M2^+ (Ca^{2+}, Mg^{2+)} \longrightarrow EDTA\ (M^{2+)}\ complex]^{2-} + 2\ Na^+ + 2H2^+$

Reading in ml	Pilot Reading	I (ml)	II (ml)	III (ml)	C.B.R. (Y ml)
Initial					
Final					
Difference					

CALCULATIONS:

The hardness of water is usually expressed in terms of calcium carbonate. The reaction is,

$Na_2EDTA + Ca2^+ \longrightarrow [EDTA\ (Ca^{2+)}\ complex]^{2-} + 2\ Na^+ + 2H2^+$

Observations:

1) Y = Volume of EDTA required
2) V = Volume of water sample
3) Z = Molarity of EDTA

Total hardness = $\frac{Y}{V}$ x z x 10^5 ppm of $CacO_3$

Total hardness of given water sample: ________________ ppm of CacO3

RESULT TABLE:

1.	Exact molarity of EDTA solution	_________ M
2.	Total hardness of the given sample of water in mg/lit	_________mg/lit of $CaCO_3$
3.	Total hardness of the given sample of water in ppm	________ ppm of $CaCO_3$

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02. ALKALINITY OF WATER

AIM: To determine total alkalinity of water sample

APPARATUS: Titration apparatus

CHEMICALS: HCl, methyl orange, phenolphthalein.

THEORY:

A natural water may be alkaline due to presence of hydroxides, bicarbonates and carbonates compounds dissolved in water.

Hydroxides : NH_4OH, NaOH, Ca $(OH)_2$
Bicarbonates : $Ca(HCO_3)_2$, $Mg(HCO_3)_2$, $Fe(HCO_3)_2$, $NaHCO_3$ etc.
Carbonates : $MgCO_3$, $FeCO_3$, $Na2CO_3$

Hydroxides and carbonates are stronger bases than bicarbonates.

When alkaline water is titrated with a strong acid, first all ^{-}OH get neutralized, then all the CO_3^{-2} ions are half neutralized to HCO_3^{-}. Till this stage, pH of mixture decreased to about 8.2 and completion of this stage is indicated by change in colour of phenolphthalein. On continued addition of acid during titration, all the HCO_3^{-} in the titration mixture (produced by half neutralization of CO_3^{-2} and present from beginning) get neutralized and completion of this stage, is indicated by methyl orange colour change at about 3.7 pH.

$$\left.\begin{array}{ll} \text{I)} & \left.\begin{array}{l} ^{-}OH + H^{+} \longrightarrow H_2O \\ CO_3^{-2} + H^{+} \longrightarrow HCO_3^{-} \end{array}\right\} \mathbf{P} \\ \\ \text{II)} & HCO_3 + H^{+} \longrightarrow H_2O + CO_2 \end{array}\right\} \mathbf{M}$$

PROCEDURE:

1) Fill the burette with 0.1N HCl solution
2) Take 10 ml of water sample by pipette in a 100 ml conical flask.
3) Take 10 ml of water sample in a conical flask and add 2 drops of phenolphthalein indicator in it. The colour of solution turns pink.
4) Titrate this sample against standard strong acid i.e. 0.1 N HCl solution from burette , till pink colour changes to colourless. This is endpoint of titration. Record burette reading as X_1 ml. This is the reading of phenolphthalein alkalinity of sample.
5) Add 4-5 drops of methyl orange indicator into the same titrating mixture and continue the titration till yellow colour of mixture changes to orange. Note down the burette reading as Y_1 ml. This is endpoint for total alkalinity of water sample. This is methyl orange i.e. total alkalinity of sample.

6) Repeat same procedure for two more times to find constant burette reading.

OBSERVATION TABLE

Burette no. 1 : Water sample
Burette no. 2 : 0.1N HCl solution
Indicators : 1) Phenolphthalein
2) Methyl orange
Endpoints : 1) Pink to colourless
2) Yellow to Orange

(A) Phenolphthalein Alkalinity :

Reading	I	II	III	C.B. R.
(Water sample)	10 ml	10 ml	10 ml	X= --------- ml
(0.1N HCl)	X_1 =	X_2 =	X_3 =	

(B) Methyl Orange Alkalinity :

Reading	I	II	III	C.B. R.
(Water sample)	10 ml	10 ml	10 ml	Y= --------- ml
(0.1N HCl)	Y_1 =	Y_2 =	Y_3 =	

CALCULATION :-

(A) Phenolphthalein Alkalinity:

$$P = \frac{V1 \, x \, z \, x \, 50 \, x \, 1000}{V}$$

∴ Phenolphthalein Alkalinity in given water sample P = ___________ ppm of $CaCO_3$

(B) Methyl Orange Alkalinity i.e. Total Alkalinity :

$$P= \frac{V2 \, x \, z \, x \, 50 \, x \, 1000}{V}$$

∴ **Methyl Orange Alkalinity** = -------------------- (B * 1000) mg of CaCO3 / ppm of $CaCO_3$

∴ Total Alkalinity in given water sample = M = -------------- ppm $CaCO_3$

The possible combinations of alkalinities in water are:

- i) Only ^{-}OH
- ii) Only CO_3^{-2}
- iii) Only HCO_3^{-}
- iv) ^{-}OH and CO_3^{-2} together
- v) CO_3^{-2} and HCO_3^{-} together

The amount of alkalinities due to the ^{-}OH, CO_3^{-2}, HCO_3^{-} are calculated from following table.

Sr. No.	Alkalinity	Quantity of ^{-}OH	Quantity of CO_3^{-2}	Quantity of HCO_3^{-}
1	P = 0	0	0	M
2	P = ½ M	0	2P	0
3	P = M	P	0	0
4	P < ½ M	0	2P	M – 2P

5	P > ½ M	(2P –M)	2(M-P)	0

CALCULATIONS:-

Now calculate type of alkalinity for sample using above table

RESULT TABLE:

Type of alkalinity detected		**Amount of alkalinity**
1		
2		

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3. 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, calomel and glass electrode, burette, pipette, stirrer etc.

CHEMICALS: 0.1 N (approx) of a given weak acid, 0.5N (exact) NaOH, 0.05M potassium hydrogen phthalate (pH=4)

THEORY: Dissociation of a weak acid e.g. CH_3COOH may be represented by the equation

$$CH_3COOH \rightleftharpoons CH_3COO^- + H^+$$

When the acid is half neutralised, the concentration of acid and the concentration of its salt in the solution will be equal. For a weak acid the concentration of undissociated acid molecules can be assumed to be unchanged by a small fraction which dissociates to provide the hydrogen ions. The salt can be regarded as fully dissociated and hence the concentration of CH_3COO^- ions can be assumed to be equal to that of the salt formed up to any stage of titration.

For the half neutralization stage, [CH3COOH] = [CH3COO]

$$\mathbf{K_a} = \frac{[CH_3COO^-]\,[H^+]}{[CH3COOH]}$$

$$\mathbf{K_a} = \frac{[CH_3COO^-]}{[CH3COOH]} \text{ X } [H^+]$$

$$\therefore K_a \approx [H^+]$$

$$\therefore -\log K_a = -\log [H^+]$$

$$pK_a = pH$$

Thus the dissociation constant can be evaluated either by knowing the $[H^+]$ or the pH value for the half neutralized solution of the acid.

Procedure:

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 temperature dial to 25^0C.
3) With the help of special cable provided connect the electrode terminal and 7 pH.
4) Now remove the wire from 7 pH and connect to 4.2 pH Adjust ‘SLOPE’ to read 4.20.
5) Now remove the wire from 4.20 pH and connect to 9.20 pH. The reading displayed must be 9.20.
6) If the above procedure works then the instrument is ‘OK’.
7) If not report it to instructor.

PART – II Determination the pK$_a$ value of weak acid

(1) Standardize the pH meter using 0.05 M potassium hydrogen phthalate solution.

(2) Take 20 ml of the .given weak acid and about 50 ml of water in 100 ml beaker.

(3) Place the stirrer and calomel - glass electrodes into the beaker. See that the electrodes are properly dipped into the solution.

(4) Fix burette containing the strong base i.e. O.5N NaOH above the beaker.

(5) Measure the pH of the acid solution.

(6) Add O.2 of O.5N NaOH from the burette to the acid solution. Stir well and note the pH of the solution.

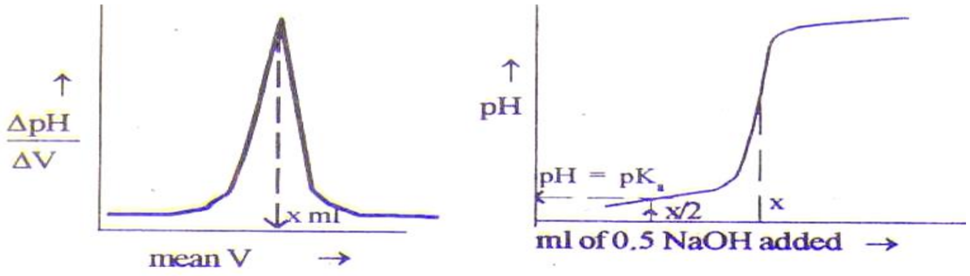
(7) Repeat the procedure by adding 0.3 ml NaOH each time. Record the pH of the solution at each time, continue the addition of NaOH solution till the end point exceeds by 2 ml.

OBSERVATION:

Obs. No.	Volume of 0.5N NaOH added V(ml)	pH	ΔpH	ΔV	$\frac{\Delta pH}{\Delta V}$	Mean V
1.						
2.						
3.						
4.						
5.						
6.						
7.						
8.						
9.						
10.						
11.						
12.						
13.						

14.						
15.						
16.						
17.						
18.						
19.						
20.						
21.						
22.						
23.						
24.						
25.						
26.						
27.						

Graph: Plot the following graphs.



(1) $\Delta pH / \Delta V$ against mean V
(2) pH against volume of NaOH added.

CALCULATIONS:-

From the graph (1) note the equivalence point of the titration as X ml. Find the half neutralization point as X/2 ml.

At half neutralization point (X/2 ml.) find the pH of the solution from the plot 2. The pH value at half neutralization point is the pK_a value of the acid.

Now, $pK_a = -\log K_a$

$\mathbf{K_a} = \text{antilog}(-pK_a)$

RESULT TABLE:

1.	Equivalence point of the titration	--------------- ml
2.	pK_a of the given weak acid	
3.	Dissociation constant (K_a) of the acid	

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4. CONDUCTOMETRIC TITRATION

AIM : - To determine the end point of conductometric titration of strong acid and strong base

APPARATUS: - Conductivity meter, conductivity cells, Beakers, Pipette, Burette.

CHEMICALS:- HCl (0.1N), Acetic acid Solution (0.1N), NaOH Solution, (0.1N)

PRINCIPLE: -

Solution of electrolytes conducts electricity due to the presence of ions. Since Specific conductance of a solution is proportional to the concentration of ions in it Conductance of the solution is measured during titration. When the sodium hydroxide is added slowly from the burette to the solution HCl (strong acid) gets neutralized. Since the fast moving H^+ ions are replaced by slow moving Na^+ ions, decrease in conductance take place until the end point

Reaction

$$H^+ + CI^- + Na^- + OH^- \longrightarrow Na^+ Cl^- + H_2O$$

Equation

1ml 1N NaoH = 36.5 mg of HCl

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 decrees 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 NaOH added, the end point can be calculated.

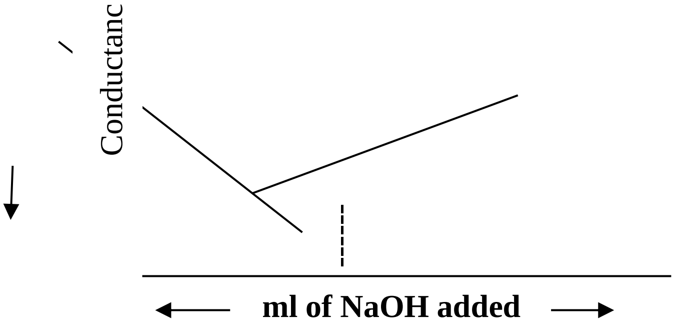
PROCEDURE: -

1. The burette is washed well with water and rinsed with the given standard NaOH solution.
2. It is then filled with NaOH solution upto the zero level.
3. 20 ml of the given acid (HCL) is pipette out into a clean 100 ml beaker.
4. The conductivity cell is placed in it and then diluted to 50 ml by adding water, so that the electrodes are well immersed in the solution.
5. Now 1 ml of NaOH from the burette is added to the solution taken in the beaker.
6. Mixture of solution stirred for some time and then conductivity is measured.
7. This process is repeated until at least five readings are taken after the end point (A) has been reached.
9. After the endpoint, again NaOH is gradually added, which causes increase in conductance.
10. Now the graph is plotted between the volume of NaOH Vs conductivity. From the graph the first end point (A) and second end point (B) are noted.

OBSERVATION TABLE:

Sr. No.	Volume of NaOH added (ml)	Conductance
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		

GRAPH: Plot the graph of conductance Vs ml of NaOH added.



CONCLUSION:

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5. DETERMINE λmax AND CONCENTRATION OF GIVEN SOLUTION OF $CuS0_4$

AIM: - To determine λmax and concentration of given solution of $CuS0_4$.

THEORY: - Lambert- Beers Law states, for the given system and the thickness of the medium, the absorbance of the solution is directly proportional to the concentration of an absorbing species.

APPARATUS: Colorimeter

CHEMICALS: $CuSo_4$, NH_3 etc.

PROCEDURE:

A) Prepare various solutions as per the table given below.

Flask No.	Cone of $CuS0_4$ Solution (M)	0.01 M $CuS0_4$ Solution taken for dilution (ml)	Liquor NH_3 added (ml)	Total volume after dilution(ml)
1	0.001	10 ml	12 ml	100 ml

2	0.002	20ml	12 ml	100 ml
3	0.004	40 ml	12 ml	100 ml
4	0.006	60 ml	12 ml	100 ml
5	Unknown	Given volume	12 m	100 ml

B) Selection of λ max (wavelength of maximum absorption)

It is found that solution does not absorb various incident monochromatic wavelengths.

But the certain wavelength is absorbed maximum known as λ max.

Sr. No.	Filter (Wavelength)	Absorbance
1	400 nm	
2	420 nm	
3	470 nm	
4	500 nm	
5	530 nm	
6	620 nm	
7	660 nm	
8	700 nm	

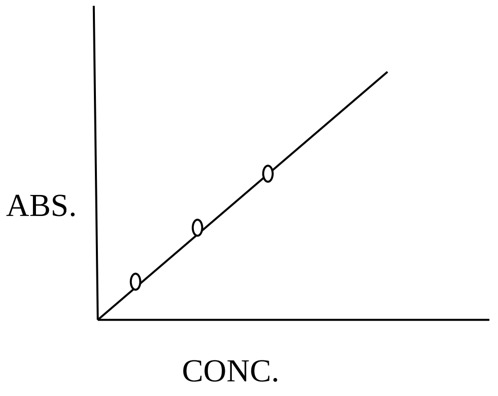
1. Before instrument is switched ON, check that required filter is rotated in the instrument.
2. Allow the instrument to warm up for 15-20 minutes.
3. Insert the test tube filled with solvent (Blank) in its place.
4. Do not disturb the test tube. Adjust 100% (Coarse and line) knobs till display reads 100.

5. Record absorbance for solution from flasks 1, 2,3,4,5 with disturbing adjustment done.
6. Plot the graph or absorbance against concentration.
7. Determine graphically the concentration of unknown solution.

OBSERVATIONS:

SR. NO.	FLASK NO.	CONCENTRATION	ABSORBANCE
1			
2			
3			
4			
5			

PLOT A GRAPH: ABSORBANCE VS CONCENTRATION



RESULTS:

1. Graph is a straight line passing through origin: Lambert -Beer's Law verified.

2. Concentration of given unknown solution is: _______________

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06. PREPERATION OF POLYMER

Aim: Preparation of urea formaldehyde and its characterization

A] Preparation of urea formaldely

Theory:

Ph

enolic resins are condensation polymerization products of phenolic derivatives with aldehydes.urea formaldehyde resin are prepared by the condensation reaction between urea and neutral or alkaline conditions.

Apparatus:- Glass rod, Watch glass, Beaker

Chemicals: 40% formaldehyde, urea, conc. H_2SO_4

Procedure:

1. Take 10 ml of 40% formaldehyde solution in a 100 ml beaker.
2. Add about 5gms 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. H_2SO_4 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.

Reaction

Test for characterization of Urea formaldehyde

Substance + cold water, shake
→ (A) Water soluble / miscible
→ (B) Water Insoluble / immiscible

Substance i.e. Urea formaldehyde is ______________________________

Type Determination of Substances

Sr. No.	Test	Observation	Inference
1	10 mg substance + 10 drops 10% $NaHCO_3$	Soluble with effervescence of CO_2 and reprecipitated by conc. HCl	Acid is present and confirmed.
2.	10 mg of substance + 10 drops 10% NaOH	Completely soluble and reprecipitated with conc. HCl	Phenol is present and confirmed
3	10 mg substance + 10 drops of 1:1 HCl & shake	Completely soluble and reprecipitated with 10 % NaOH	Base is present and confirmed
4	If all the tests (1), (2), (3) are negative	Then →	∴ Substance is Neutral.

Conclusion : Type of the substance is Acidic / Phenolic / Basic / Neutral

Type of the substance is ☐

Detection of Saturation/Unsaturation

Sr. No.	Test	Observation	Inference

1	**$KMnO_4$ Test :** 10 mg substance + 10 droops 10 % Na_2CO_3 + 4 drops of dilute $KMnO_4$ and shake well	i) Pink colour of $KMnO_4$ disappears ii) No decolourisation (Pink colour persists)	∴ Unsaturated compound present. ∴ Saturated compound present

Detection of Aliphatic/Aromatic Nature

Sr. No.	Test	Observation	Inference
1	Heat a little (10 mg) substance on a clean copper gauze	i) Sooty flame ii) Non sooty flame	∴ Aromatic compound present ∴ Aliphatic compound present

Conclusion : Polymer compound is :

(i) Acidic/ Phenolic / Basic / Neutral

(ii) Saturated / Unsaturated

(iii) Aromatic / Aliphatic

Result Table:

Polymer compound is____

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07. DETERMINATION OF MOLECULAR WEIGHT OF A POLYMER

Aim: Determination of molecular weight of a polymer.

Chemicals:

Polyvinyl alcohol

Apparatus:

- Ostwald’s viscometer,
- stopwatch

Theory:

Polymers have molecular weights, which may vary from several thousands to several millions. It is related to the chain length and the extent of cross –linking between different chains. Addition of even a very small amount of a polymer to a solvent of low viscosity causes a sharp increase in its viscosity. The magnitude of increase in viscosity depends on the molecular weight of the polymer.

When a liquid is made to flow through a capillary tube by a pressure gradient and the time for a given volume to flow along the tube is measured, then at a constant temperature, the coefficient of viscosity, η , is given by Poiseuille’s equation .

$$\eta = \frac{\text{Л}\, P r^4\, t}{8\, V\, L}$$

V – Volume of liquid that flows through the tube.

P - Difference in pressure at the two ends of the tube.

r - Radius of the tube.

L - Length of the capillary tube in centimeter.

t – Time of flow in seconds

When viscometer is used for the measurements the liquid flows vertically down the capillary tube and the driving force is given by $P = h p g$

By using an Ostwald's viscometer, relative viscosity (viscosity ratio) $\eta_r = \eta / \eta_o$ can be found out . Here η is the viscosity of polymer solution and η_o is the viscosity of pure solvent.

$$\eta_r = \eta_s / \eta_o = p_s t_s / p_o t_o$$

p_s and p_o are densities of unknown liquid (polymer solution) and pure solvent respectively and t_s and t_o are the corresponding flow times.

For a dilute polymer solution ($C < 0.6\%$), the density of the solution may be taken as that of the solvent, i.e., $p_s = p_o$

$$\eta_r = t_s / t_o$$

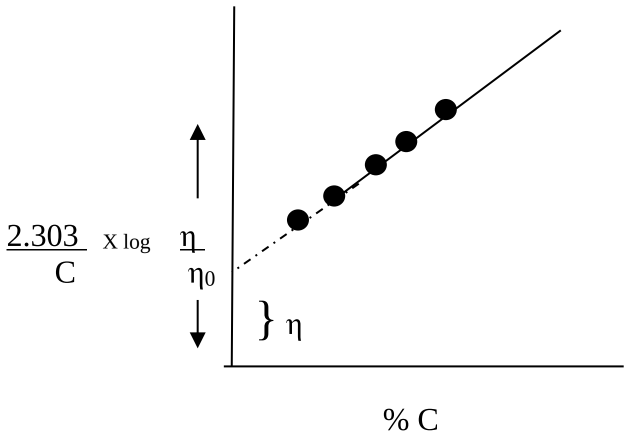
The specific viscosity, η_{sp}, of the polymer solution is obtained by dividing ($\eta_{sp} - \eta_o$) the increase in viscosity of the solvent due to the presence solute by η_o the viscosity of the pure solvent .

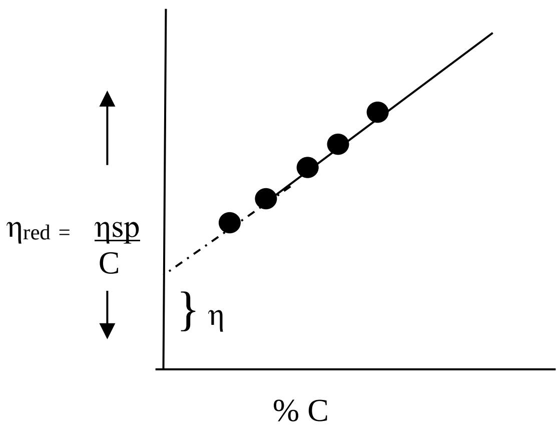
$$\eta_{sp} = (\eta_{sp} - \eta_o) / \eta_o = \eta_r - 1$$

The ratio of the specific viscosity of the solution to its concentration C, expressed in g/ 100 ml is called reduced specific viscosity or simply reduced viscosity η_{red}.

$$\therefore \quad \eta_{red} = \eta_{sp} / C$$

Both ηr and η_{sp} vary sharply with concentration, the variations of η_{red} with concentration is somewhat less and regular, and the plot of η_{red} versus concentration is a straight line given by the equation.





The value of the constant is given by the intercept on the ordinate. This constant, which becomes independent of concentration is the limiting value of the reduced viscosity and is termed as limiting viscosity or intrinsic viscosity, [η].

Mathematically,

$$[\eta] = \lim_{C \to O} \frac{\eta_{red}}{C}$$

Or $$\eta_{sp}/C = mC + [\eta]$$

For linear polymer the intrinsic viscosity [η] and the molecular weight M are generally found to obey the Mark – Kuhn – Houwink empirical equation.

$$[\eta] = KM^a$$

Where **K** and **a** are constants for a particular polymer / solvent / temperature system. **K** and **a,** values are known for a polymer / solvent combination, M may be calculated from the determined value of [η] .

The values of **K** and **a** are given below for some polymers (25^0C).

Sr. No.	Polymer	Solvent	K	A
1.	Cellulose acetate	Acetone	1.49×10^{-4}	0.82
2.	Polystyrene	Toluene	3.70×10^{-4}	0.62
3.	Polymethyl Methacrylate	Benzene	0.94×10^{-4}	0.76
4.	Polyvinyl alcohol	Water	2.00×10^{-4}	0.76

Procedure:

Part – A: To determine the time of flow for a given pure solvent

1. Take a clean and dry Ostwald's viscometer.
2. Clamp the wider arm of the viscometer to the retort stand in such a way that viscometer is in perfectly vertical position.
3. Introduce 30 ml of pure solvent into wider arm of viscometer.
4. Attach a rubber tube to a narrow arm of viscometer and suck the pure solvent by rubber tube till it rises above the upper mark of narrow arm above bulb B
5. Allow the pure solvent to flow down, and start the stopwatch as the liquid crosses the upper mark.
6. Stop the stopwatch as the liquid crosses the lower mark.
7. Note the time in seconds required by solvent to flow from upper mark to lower mark on narrow arm of viscometer.
8. Take two such readings and find out the mean time in seconds required to flow for the solvent.

Part – B: To determine the time flow for given polymer solution

1. Drain out the water from viscometer and rinse the viscometer using 4-5 ml of 0.2% polymer solution.
2. Now introduce 30 ml of 0.2 % of polymer solution (polyvinyl alcohol in water) in clean viscometer and repeat the procedure as in part A to take two readings.
3. Find mean time of flow for 0.2 % of polymer solution.
4. Similarly repeat the procedure for 0.3 %, 0.4 %, 0.5 % and 0.6 % polymer solution and record the time

Observation table:

Sr. No	Solvent/ Polymer solution	Time 't' (seconds)			$\eta_r = \eta / \eta_o =$ t / t_o	$\eta_{sp} = \eta_r - 1$	$\eta_{red} = \eta_{sp} / C$	(2.303/C) * (log η / η_o)
		1	2	Mean				
1	0 % (Pure solvent)							
2	0.2 % polymer sol^n							
3	0.3 % polymer sol^n							
4	0.4 % polymer sol^n							
5	0.5 % polymer sol^n							
6	0.6 % polymer sol^n							

From appropriate values of K and value of [η] from graph find out molecular weight of polymer by using equation

1) $\eta = KM^a$

$$M^a = \frac{\eta}{K}$$

$$a \ \log M = \log \frac{\eta}{k}$$

Result Table:

Polymer used	Molecular weight

Signature of Teacher:

SINHGAD ACADEMY OF ENGINEERING
CHEMISTRY ENGINEERING DEPARTMENT

Date:

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8. PROXIMATE ANALYSIS

AIM: To determine moisture, volatile matter and ash content in a given sample of coal.

APPARATUS/INSTURMENTS: Oven, Muffle Furnace, Crucible

CHEMICALS : Coal sample

THEORY :

In order to find suitability of coal for a particular domestic or industrial use proximate analysis is carried out. Proximate analysis is the simplest type of analysis of coal and it gives information about the practical utility of coal. In the proximate analysis, moisture, volatile matter, ash and fixed carbon are determined.

Moisture is absorbed by porous coal. It decrease with increasing rank and is a rough indicator of the age or degree of coalification. It reduces the calorific value of coal. A lot of heat is wasted in evaporating the moisture during combustion.

Coal with high volatile matter burns with long smoky flame. Volatile matter consists of combustible gases like CO, H_2, CH_4 & HC and incombustible gases like CO_2 and N_2. The calorific value of such coals is less as the heat produced is distributed over large space.

Ash is non-combustible matter left over after burning of coal. It reduces the calorific value of coal. Ash carried with coal increases its weight without adding to the calories available. Ash usually consists of silica, alumina, iron oxide, lime, magnesia etc.

PROCEDURE:

PART A: MOISTURE CONTENT IN COAL

1) Clean a porcelain crucible. Dry the crucible by heating in a oven at 100 ^{oC} for 5 minutes. Cool the crucible in desiccator and weigh it accurately.
2) Weigh exactly about 1 gram of powdered coal sample in the porcelain crucible.
3) Place the crucible without lid in hot air oven. Heat it to 105 - 110 ~ for about one hour. Remove the crucible from oven and cool it in desiccator. Weigh it accurately.
4) Repeat the procedure by heating and cooling the crucible till constant weight is obtained.

5) Hence calculate percentage of moisture in coal.

OBSERVATIONS:

Sr. No.	Observation	Symbol	Value
1	Wt of dry porcelain crucible	W	
2	Wt of dry porcelain crucible + coal	W_1	
3.	Wt of coal taken	X	
4	Wt of crucible + coal after heating	W_2	
5	Wt of moisture content	$W_1 - W_2$	

CALCULATIONS:

$$\text{Percentage of moisture} = \frac{\text{loss in weight of coal}}{\text{weight of coal taken}} * 100$$

PART B : VOLATILE MATTER IN COAL

1. Clean a crucible. Dry it in oven. Dry the crucible by heating in a oven at 110 ^{oC} for 1 hour. Cool the crucible in desiccator and weigh it accurately.
2. Weigh exactly about 1 gram of powdered coal sample in the porcelain crucible.
3. Place the crucible without lid in a muffle furnace maintained at 950 +-20 ^{oc} for about 7 minutes.
4. Take out the crucible .First bring down its temperature rapidly (to avoid oxidation of the contents) by placing it on a cold iron plate.
5. Then transfer the warm crucible to a desiccator to bring it to room temperature.
6. Take the weight of crucible and its contents and find the percentage of volatile matter in coal.

OBSERVATIONS:

Sr. No.	Observation	Symbol	Value
1	Wt of dry porcelain crucible	W	
2	Wt of dry porcelain crucible + coal	W_1	
3	Wt of coal taken	X	
4	Wt of crucible + coal after heating	W_2	
5	Wt of volatile matter content	$W_1 - W_2$	

CALCULATIONS:

$$\text{Percentage of volatile matter} = \frac{\text{Loss in weight of moisture free coal}}{\text{Weight of coal taken}} * 100$$

PART C: ASH CONTENT IN COAL

1. Weigh exactly about 0.5 gm of powdered coal sample in a previously dried and weighed silica crucible.
2. Heat the crucible without lid in the muffle furnace at 750 +- 20^o C for half an hour.
3. Remove the crucible from the furnace. Cool it in air initially and then in desiccator.
4. Weigh it accurately and find the percentage of ash in coal.

OBSERVATIONS:

Sr. No.	Observation	Symbol	Value
1	Wt of dry porcelain crucible	W	
2	Wt of dry porcelain crucible + coal	W1	
3	Wt of coal taken	X	
4	Wt of crucible + coal after heating	W2	
5	Wt of ash content	$W_2 - W$	

CALCULATIONS:

$$\text{Percentage of ash} = \frac{\text{Weight of ash / residue}}{\text{Weight of coal taken}} * 100$$

Percentage of carbon = 100 - % of (moisture + volatile matter + ash)

RESULT TABLE:

Sr. No.	**Observations**	**Values**
1	Percentage of moisture	
2	Percentage of volatile matter	
3	Percentage of ash	
4	Percentage of carbon	

SIGNATURE OF TEACHER: