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EXPERIMENT -1

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

Apparatus/Chemicals used: Burette, Pipette, Conical flask, Measuring cylinder, NaOH Solution, Phenolphthalein indicator (Internal Indicator), Oxalic acid (N/5)

Theory:

Standard solution is one in which exact amount of a substance is present in a definite volume of the solution. or a solution whose concentration (strength) is known to us is also called as standard solution.

Volumetric solutions are classified into following two types:

- (i) Primary Standard Solution
- (ii) Secondary Standard solution

Primary Standard Solution: The substance whose standard solution is prepared by dissolving directly its known amount in a definite volume of solvent or solution is known as a primary standard substances & the solution is called as primary standard solution. Commonly used primary standard substances are anhydrous  $\text{Na}_2\text{CO}_3$ , Oxalic acid etc.

Secondary Standard Solution: The substance whose solution cannot be prepared directly by weighing its definite amount and then dissolving in definite volume of solvent is called secondary standard substance & the solution is called as secondary standard solution. The solution of this type of substance firstly prepared is of approximate strength which is then standardized with a standard solution of a primary standard substance. The common secondary standard substances are alkali hydroxides, inorganic acids and  $\text{KMnO}_4$  etc.

Classification of Methods of Volumetric Analysis: Volumetric analyses are of following types

- (i) Neutralization titrations or Acid Base titrations
- (ii) Oxidation-reduction titrations
- (iii) Precipitation titrations
- (iv) Complexometric titrations

Normality (N): The normality of a solution is the number of gram-equivalents of the solute per litre of the solution.

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



**PROCEDURE:-**

1. Prepare NaOH solution ( More than N/10 concentration solution).
2. Rinse and fill the burette with N/5 oxalic acid solution.
3. Pipette out 10 ml of above prepared NaOH solution in a conical flask. Add 2-3 drops of phenolphthalein indicator solution. The color of the solution becomes pink.
4. Add oxalic acid soln. from burette drop by drop into it till the pink color of solution just disappears.
5. Note down the reading and repeat it until two concordant readings are obtained.

**Observations and Calculations:**

Wt of the empty weighing tube ( $w_1$ ) = gm

Wt of the weighing tube with substance ( $w_2$ ) = gm

Wt of the substance ( $w_2 - w_1$ ) = gm

S. No.	Volume of the NaOH soln. taken for titration (ml) (V <sub>1</sub> )	Burette Reading		Volume of std. soln. of oxalic acid consumed (ml) (V <sub>2</sub> )
		Initial	Final	
1.				
2.				
3.				
4.				

**Calculation of the normality of the solution prepared**

$$N_1 V_1 = N_2 V_2$$



- If the strength of the solution is less than the desired strength then repeat the experiment with the fresh preparation of the sample.
- If the strength of the solution is more than the desired strength follow the method below.

$$N_1 V_1 = N_2 V_2 \\ (\text{For NaOH soln. prepared}) \qquad (\text{For NaOH soln. of Required Strength})$$

$$\text{Or} \qquad N_1 V_1 = N / 10 \cdot 100$$

Volume of distilled water required for dilution =  $(100 - V_1)$  ml

**Result:** Standard solution of N/10 NaOH solution is prepared.

**Precautions:**

1. Solution should be making up to desired volume after complete dissolving the solute.
2. During titration the solution should be stirred thoroughly
3. Do not take mean of burette readings.

**Viva – Voce Questions:-**

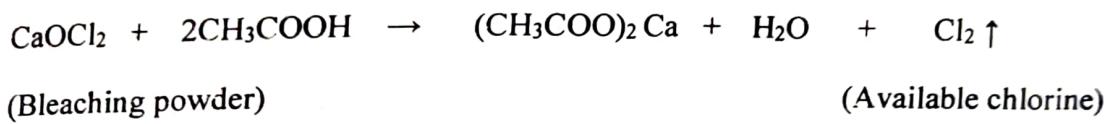
1. What is standard solution?
2. What is Normality?
3. What is molality?
4. What is Molarity?
5. Which indicator is used in this titration? What is its pH range?
6. What is acid-base titration?
7. What are primary standard and secondary standard solutions?
8. What is the color of the phenolphthalein indicator in acidic medium?
9. What is the color of the phenolphthalein indicator in alkaline medium?
10. What are the structures of phenolphthalein in acidic & alkaline medium?

EXPERIMENT- 2

Object:- To determine the percentage of available chlorine in the given sample of Bleaching Powder by iodometric titration using starch as an internal indicator.

Chemicals/Apparatus required:- Bleaching Powder, Potassium Iodide, Acetic Acid, Sodium thiosulphate (hypo), Distilled Water and freshly prepared solution of Starch indicator. Burette, Pipette, Conical Flask, Beaker, Measuring flask (Volumetric flask), funnel, Pastel and Mortar etc.

Theory:- The amount of chlorine liberated by the action of dilute acids on hypochlorite is termed as available chlorine "It is generally expressed as the percentage by weight of bleaching powder." In practice the available chlorine is determined by treating it with sodium thiosulphate solution, in presence of acetic acid, the liberated chlorine is immediately treated with potassium iodide to give free iodine, which actually reacts with standard sodium thiosulphate, hence termed as iodometric titration.

Procedure:-

1. Rinse and fill the burette with N/20 hypo solution.
2. Pipette out 10 ml of bleaching powder solution in a conical flask.
3. Add about 0.5 gm solid KI and 1-2 ml glacial acetic acid. The solution becomes brown in color.
4. Now add hypo solution from burette to it till the brown color changes to light yellow.
5. Now add 1-2 drops of starch solution into it. The color of the solution becomes blue.
6. Now again add hypo solution drop by drop to it till the blue color disappears.
7. Repeat the titration until two concordant readings are obtained.

Observations:-

1. Weight of empty weighing tube = ..... (a) gm
2. Weight of weighing tube + bleaching powder = ..... (b) gm
3. Weight of bleaching powder = ..... (b-a) gm
4. Normality of sodium thiosulphate (hypo) solution = N/20

S. No.	Vol. of bleaching powder Solution(ml) (V <sub>1</sub> )	Burette reading		Vol. of Hypo solution (ml) (V <sub>2</sub> )
		Initial	Final	
1.				
2.				
3.				
4.				

Calculations:-



$$\begin{aligned}
 \text{Normality of available chlorine (N}_1\text{)} &= \frac{N_2 \times V_2}{V_1} \\
 \\ 
 &= N_1 \times 35.5 \text{ gm/l} \\
 \\ 
 &= \frac{N_1 \times 35.5 \times 100}{1000} \text{ gm/100 ml soln} \\
 \\ 
 \% \text{ of available chlorine} &= \frac{N_1 \times 35.5 \times 100 \times 100}{1000 \times (b-a)}
 \end{aligned}$$

Result:- The percentage of available chlorine present in given sample of bleaching powder is  
-----%

**Precautions:-**

1. All the apparatus were cleaned thoroughly.
2. A uniform paste of bleaching powder was prepared.
3. The amount of indicator should be same in all titrations.
4. Do not take mean of burette readings.

**Viva-Voce questions**

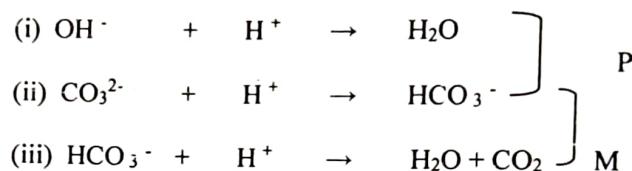
1. What is available chlorine?
2. What is the formula of bleaching powder?
3. What is the chemical name of bleaching powder?
4. What are the two industrial uses of bleaching powder?
5. What is the approx % of available chlorine in bleaching powder?
6. How do you estimate available chlorine in Bleaching powder?
7. What are iodometric & iodimetric titrations?
8. What is the formula of sodium thiosulphate (Hypo)?
9. What are the oxidation numbers of chlorine in bleaching powder?

### EXPERIMENT-3

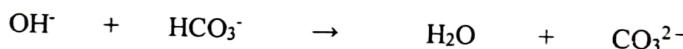
**Object:** To determine the constituent and amount of alkalinity in the given water sample by titrating it against standard HCl solution (N/20) using phenolphthalein and methyl orange as an internal indicators.

**Materials/Chemicals/Apparatus required:** N/20 HCl solution, phenolphthalein and methyl orange indicators, sample solution, burette, pipette, conical flask, beaker, funnel, etc.

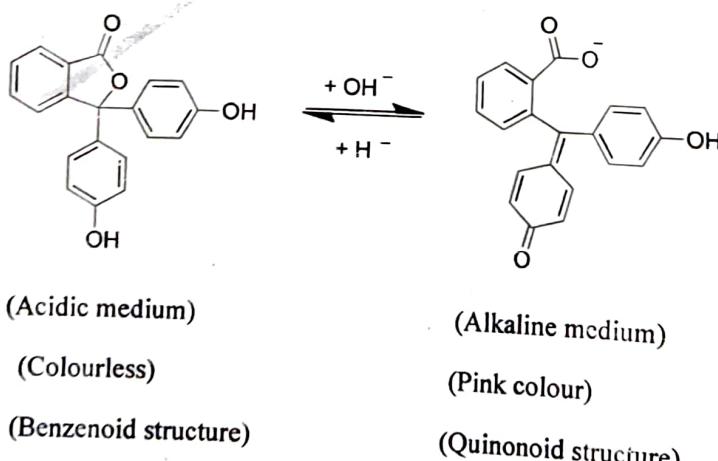
**Theory:** The alkalinity in water is due to the presence of hydroxyl ion ( $\text{OH}^-$ ), carbonate ion ( $\text{CO}_3^{2-}$ ) and bicarbonate ion ( $\text{HCO}_3^-$ ) present in the given sample of water. These can be estimated separately by titrating against standard acid (N/20 HCl) using phenolphthalein and methyl orange as indicators. The chemical reaction involved can be shown by the equations given below:



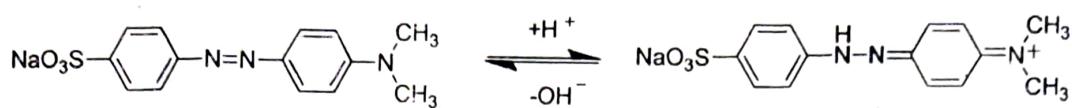
[P]-Stands for phenolphthalein indicator and [M]-stands for methyl orange indicator.  
In solution  $\text{OH}^-$  and  $\text{HCO}_3^-$  ions can't co-exist due to following reaction-



#### Phenolphthalein Structure:-



**Methyl orange Structure :-**



(Benzoid structure)

(Yellow colour)

(Alkaline medium)

(Quinonoid structure)

(Red colour)

(Acidic medium)

**Procedure :-**

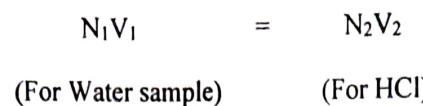
1. Rinse and fill the burette with N/20 HCl.
2. Pipette out 10 ml of water sample into a conical flask. Add 1-2 drops of phenolphthalein indicator. A pink color will appear.
3. Add HCl from burette drop by drop till the pink color just disappears. Note this reading.
4. Now add 1-2 drops of methyl orange indicator to above solution. A yellow color will appear.
5. Add N/20 HCl drop by drop to it till the yellow color just changes to red. Note this reading.
6. Repeat this titration until two concordant readings are obtained.

**Observations:**

S.No.	Volume of sample solution in (ml)	Burette reading				Volume of HCl with phenolphthalein (P) in (ml)	Volume of HCl with methyl orange (M) in (ml)		
		Using phenolphthalein (P)		Using methyl orange (M)					
		Initial	Final	Initial	Final				
1.		0.0	A	A					
2.									
3.									

Calculations :-

(1) For  $\text{CO}_3^{2-}$

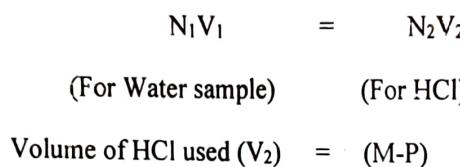


$$\text{Volume of HCl used } (V_2) = 2P$$

Strength =  $N_1 \times \text{equivalent wt. of } \text{CO}_3^{2-} \times 1000 \text{ ppm}$

Alkalinity =  $N_1 \times \text{equivalent wt. of } \text{CaCO}_3 \times 1000 \text{ ppm}$

(2) For  $\text{HCO}_3^-$



Strength =  $N_1 \times \text{equivalent wt. of } \text{HCO}_3^- \times 1000 \text{ ppm}$

Alkalinity =  $N_1 \times \text{equivalent wt. of } \text{CaCO}_3 \times 1000 \text{ ppm}$

Results:

1. The alkalinity due to presence of  $\text{CO}_3^{2-}$  is----- ppm
2. The alkalinity due to presence of  $\text{HCO}_3^-$  is----- ppm
3. The alkalinity due to presence of  $\text{OH}^-$  is----- ppm

Precautions:

1. Phenolphthalein indicator should be added first and then methyl orange.
2. The volume of indicator should same in all the titrations.
3. The reaction mixture should be shaken properly.
4. Do not take mean of burette readings

Viva-Voce questions

1. What is alkalinity of water?
2. What are the constituents responsible for alkalinity of water?

3. Why alkalinity of water cannot be due to simultaneous presence of  $\text{OH}^-$ ,  $\text{CO}_3^{2-}$  and  $\text{HCO}_3^-$ ?
4. What is the use of using two indicators in this titration?
5. Which reaction takes place during phenolphthalein end point?
6. Which reaction takes place during methyl orange end point?
7. What is alkalimetry?
8. What is acidimetry?
9. What are the formula of baking powder & washing soda?
10. What are the equivalent weights of  $\text{Na}_2\text{CO}_3$  &  $\text{NaHCO}_3$  ?
11. What is the pH range of Methyl Orange?
12. Do not take mean of burette readings

Note:-

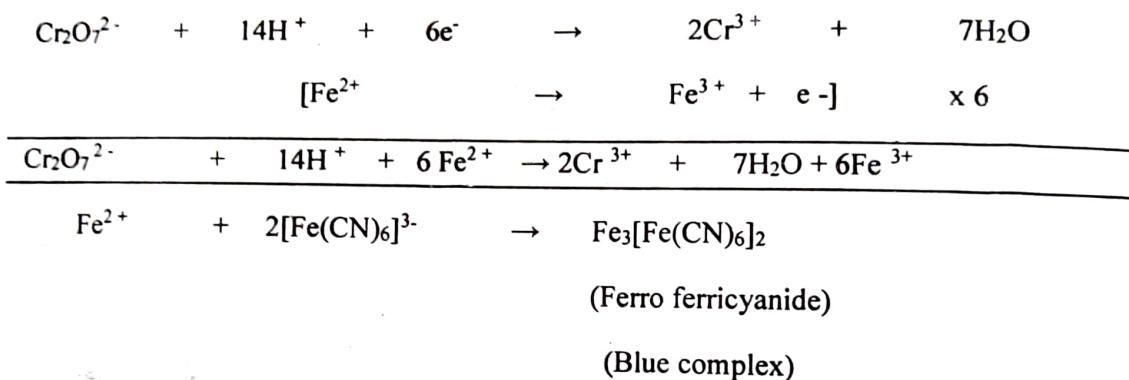
1. If  $P=0$  then only  $\text{HCO}_3^-$  is present.
2. If  $M=0$  then only  $\text{OH}^-$  is present.
3. If  $P=M$  then only  $\text{CO}_3^{2-}$  is present.
4. If  $P > M$  then only  $\text{OH}^-$  &  $\text{CO}_3^{2-}$  are present.
5. If  $P < M$  then only  $\text{CO}_3^{2-}$  &  $\text{HCO}_3^-$  are present.
6. Working pH range of Methyl Orange indicator is from pH 3.1 to 4.4.
7. Working pH range of Phenolphthalein indicator is from pH 8.3 to 10.0.

EXPERIMENT- 4

**Object:** To determine the ferrous content in the supplied sample of iron ore by titrimetric analysis against standard potassium dichromate(N/20) solution using potassium ferricyanide  $K_3[Fe(CN)_6]$  as an external indicator.

**Materials/chemicals/apparatus:-** Standard  $K_2Cr_2O_7$  solution, Potassium Ferricyanide indicator, burette, Pipette, conical flask, glass rod, white glazed tile, iron sample (Mohr's salt) etc.

**Principle:** Potassium dichromate oxidizes ferrous sulphate present in Mohr's salt into ferric sulphate in presence of dilute sulphuric acid. In this titration potassium ferricyanide is used as an external indicator, which gives a greenish blue color due to the formation of ferro ferricyanide complex .

Procedure :-

1. Rinse and fill the burette with N/20  $K_2Cr_2O_7$ .
2. Place small drops of potassium ferricyanide  $K_3[Fe(CN)_6]$  solution indicator on a dry white tile with the help of glass rod.
3. Pipette out 10 ml of iron ore solution in a conical flask and add  $K_2Cr_2O_7$  solution from the burette. After adding one ml solution of  $K_2Cr_2O_7$ , withdraw a drop of this solution from the conical flask with the help of glass rod and mix it with one drop of potassium ferricyanide indicator on the white tile. Appearance of greenish-blue color indicates that the end point has not yet reached.
4. Continue adding  $K_2Cr_2O_7$  and repeat the process of withdrawing a drop of solution and mixing it with one drop of potassium ferricyanide indicator on the white tile. When the indicator drop does not give blue color (or remains yellow), the end point is reached. Note this reading.
5. Repeat the above titration by reducing the volume interval of  $K_2Cr_2O_7$  to get the exact end point.
6. Repeat the process until two concordant readings are obtained.

Observations:-

S.No.	Vol. of sample solution taken (ml)(V <sub>1</sub> )	Burette reading		Color with the indicator	End point range	Vol. of K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> used (ml)(V <sub>2</sub> )
		Initial	Final			
1.				Blue		
				Yellow		
2.				Blue		
				Yellow		
3.				Blue		
				Yellow		

Calculations:

$$\frac{N_1 V_1}{(Iron \text{ sample solution})} = \frac{N_2 V_2}{(Potassium dichromate solution)}$$

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

$$\begin{aligned} \text{Concentration of ferrous content} &= N_1 \times \text{Eq. Wt of Fe}^{2+} \\ &= N_1 \times 56 \text{ gm/litre.} \end{aligned}$$

Result:

The ferrous ion content in the supplied sample of iron ore is .....gm /litre.

Precautions:

1. Burette and pipette should be rinsed properly before starting the titration.
2. The volume of solution taken should maintain properly.
3. Do not take mean of burette readings

Viva-Voce questions

1. What is other name of ferrous ammonium sulphate?
2. Which indicator is used in this titration?
3. What is external indicator?
4. What is the action of indicator?
5. What will happen if indicator is added into titration flask?
6. What is the formula of ferrous ammonium sulphate?
7. Why dil  $H_2SO_4$  is added during titration?
8. Why  $FeSO_4$  is not used in place of ferrous ammonium sulphate?
9. Why  $K_2Cr_2O_7$  is used as an oxidizing agent only in presence of an acid and not in presence of a base?
10. What internal indicators can be used in this titration?
11. Why dil  $H_2SO_4$  is added during prep. of standard solution of Mohr's salt?
12. What are the main ores of Iron?

List of Internal Indicators :-

1. N-Phenylanthranilic acid	-----	Green	to	violet-red
2. Diphenylamine in Conc. $H_2SO_4$	-----	Colorless	to	violet
3. Diphenyl benzidine in Conc. $H_2SO_4$	-----	Colorless	to	violet
4. Ferroin in Conc. $H_2SO_4$	-----	Red	to	faint blue

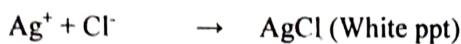
## EXPERIMENT-5

**Object:** To determine chloride ion content in a given water sample by Argentometric method (Mohr's method) using  $K_2CrO_4$  as an internal indicator.

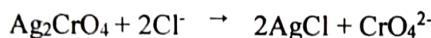
**Material/Chemicals/Apparatus required:** Burette, pipette, conical flask, measuring flask, standard silver nitrate solution (N/20), potassium chromate ( $K_2CrO_4$ ), etc.

**Theory:** Chloride ions are present in water usually as  $NaCl$ ,  $MgCl_2$  and  $CaCl_2$ . Although chloride ions are not harmful as such but their concentration over 250 ppm imparts a peculiar taste to the water thus rendering the water unacceptable for drinking purposes.

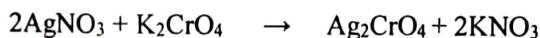
By Argentometric method chloride ions in a water sample, which is neutral or slightly alkaline, can be determined by titrating it against standard silver nitrate solution using potassium chromate as an internal indicator.



Brick red colour formed due to formation of silver chromate disappears as the solution contains high concentration of  $Cl^-$



When the concentration of chloride ions has decreased, the red colour starts disappearing slowly on shaking and a stage is reached when all the chloride ions have formed  $AgCl$  ppt., then one extra drop of  $AgNO_3$  at this point reacts with potassium chromate and forms a reddish coloured ppt of silver chromate .



**Procedure:-**

**(A) Blank Correction :-**

1. Pipette out 10 ml distilled water in a conical flask and add 2-3 drops of  $K_2CrO_4$  indicator.
2. Titrate it against N/20  $AgNO_3$  soln. till the brick red color appears in the soln.
3. Note the reading and repeat it until two concordant readings are obtained.

**(B) Sample titration :-**

1. Pipette out 10 ml sample water in a conical flask and add 2-3 drops of  $K_2CrO_4$  indicator.
2. Titrate it against N/20  $AgNO_3$  soln. till the brick red color appears in the soln.
3. Note the reading and repeat it until two concordant readings are obtained.

Observations:-

[A] Table for Blank Correction:-

S. No.	Vol. of distilled water (ml) (V <sub>1</sub> )	Burette reading		Vol. of used AgNO <sub>3</sub> soln. (ml) (V)
		Initial	Final	
1.				
2.				
3.				

[B] Table for Sample water:-

S. No.	Vol. of sample water (ml) (V <sub>1</sub> )	Burette reading		Vol. of used AgNO <sub>3</sub> soln. (ml) (V')
		Initial	Final	
1.				
2.				
3.				

Calculations:-

$$\text{Volume of water sample taken for titration} = 10 \text{ ml}$$

$$\text{Volume of AgNO}_3 (\text{N}/20) \text{ used} = (V' - V) = V_2 \text{ ml}$$

$$N_1 \times V_1 = N_2 \times V_2$$

$$N_1 \times 10 = N/20 \times (V' - V)$$



Strength of Cl<sup>-</sup> ions in water sample = Normality × Equivalent weight of Cl<sup>-</sup> gm/litre

= Normality × Equivalent weight of Cl<sup>-</sup> × 1000 ppm

Result:

Chloride content present in the given water sample is ----- gm/litre or ----- ppm

**Precautions:**

1. The whole apparatus should be washed with distilled water before the start of the experiment.
2. The reaction mixture should be briskly shaken during the titration.
3. The end point of the reaction should be carefully observed.
4. The volume of the indicator should be same in all the titrations.
5. The pH of the sample solution should be adjusted to 7-8 ranges by adding acidic/basic solution.
6. Do not take mean of burette readings

**Viva – Voce questions:-**

1. What is the formula of Mohr's salt?
2. Why is it called Argentometric method?
3. In which form chlorides are present in water?
4. How would you determine chloride ion in water?
5. Which indicator is used in this titration?
6. Which type of titration it is?
7. White precipitation of which compound is formed in this titration?
8. What is solubility product?
9. Why  $\text{Ag}_2\text{CrO}_4$  is precipitated after  $\text{AgCl}$  in this titration?
10. Name some other indicators used in precipitation titration.
11. Name two methods used in argentometric titration.
12. Why Mohr's method is carried out in neutral medium?
13. What are the sources of  $\text{Cl}^-$  in water?

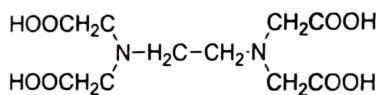
## EXPERIMENT 6

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

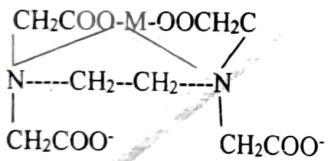
**Materials/chemicals/apparatus required:-** Burette, pipette, conical flask, beaker, measuring cylinder, tripod stand, wire gauze, funnel, filter paper, dropper, standard EDTA Solution (N/20), ammonium buffer solution, Eriochrome Black-T Indicator, Hard water (given water sample).

**Principle:-** When Eriochrome Black-T (indicator) is added to hard water solution at around 10.0 pH . it gives wine red colored unstable complex with  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  ions of the sample water. When this wine red colored complex is titrated against EDTA solution. The color of the complex changes from wine red to original blue color showing the end point.

EDTA (Ethylene Diamine Tetra Acetic acid ) is a well-known complexing agent , which is widely used in analytical work on account of its powerful complexing action and commercial availability .

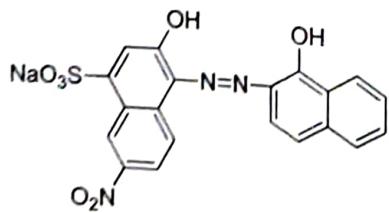


(Structure of EDTA)



EDTA complex with divalent metal cation ( $\text{M} = \text{Ca}^{2+}, \text{Mg}^{2+}$ )

In aqueous solution EDTA ionizes to give two  $\text{Na}^+$  ions and a strong chelating agent. The indicator used is a complex organic compound (sodium-1-(hydroxy-2-naphthylazo)-6 nitro-2-phenolic hydrogen atoms and for simplicity it is represented as  $\text{Na}^+\text{H}_2\text{Ion}^-$ )



(Structure of Eriochrome Black-T)

**Procedure:-**

1. Rinse the burette with N/20 EDTA soln.
2. Pipette out 10 ml of hard water into a conical flask and add 3 ml of basic buffer solution.
3. Add 2-3 drops of indicator Erichrome Black T. The color of the soln. becomes wine red.
4. Add drop by drop EDTA soln. from burette till the wine red color just changes to blue.
5. Repeat this titration until two concordant readings are obtained.
6. Take 50 ml of hard water in a beaker and boil it until the volume is reduced to nearly half.
7. Filter it and make it to 50 ml by adding distilled water.
8. Pipette out 10 ml of this water and titrate it with EDTA as given above.
9. Repeat this titration until two concordant readings are obtained.

**Observations:- (a) For hard water :-**

S. No.	Vol. of hard water (ml)	Burette readings		Vol. of EDTA Solution used (ml)
		Initial	Final	
1.				
2.				
3.				

**(b) For boiled water:-**

S. No.	Vol. of boiled water (ml)	Burette readings		Vol. of EDTA Solution used (ml)
		Initial	Final	
1.				
2.				
3.				

Calculations:-

$$1\text{ml 1M EDTA} = 100 \text{ mg of CaCO}_3$$

$$1\text{ml 1N EDTA} = 50 \text{ mg of CaCO}_3$$

OR

$$N_1 V_1 = N_2 V_2$$

Results:-

The given water sample contains:-

- (i) Total hardness = ----- mg/litre or ppm.
- (ii) Permanent hardness = ----- mg/litre or ppm.
- (iii) Temporary hardness = ----- mg/litre or ppm.

The result can also be reported in Clark by dividing all results by 14.3

Precautions:-

1. The glassware's should be properly rinsed with distilled water.
2. The reaction mixture should be shaken properly.
3. The end point should be noted correctly.
4. The pH should be maintained during titration.
5. The amount of indicator should be same in all titrations.
6. Do not take mean of burette readings

Viva-Voce questions

1. What is hardness of water?
2. What is temporary hardness of water?
3. Why hardness is expressed in terms of  $\text{CaCO}_3$ ?
4. What is permanent hardness?
5. How would you remove the temporary and permanent hardness of water?
6. What is the unit of hardness?
7. Which indicator is used in this titration?
8. Why pH is adjusted to 10 during titration?
9. How would you adjust the pH?
10. Which buffer is used in this titration?
11. What is the effect on pH by adding 100 ml of water to a buffer solution?
12. What type of titration is this?
13. What are the advantages of EDTA method for hardness determination?

**EXPERIMENT 7**

**Object:-** To determine the viscosity and surface tension of a given liquid by using Ostwald viscometer and stalagmometer respectively. Viscosity of water is 0.0101 poise and Surface tension of water is 72.14 dynes/cm at 25°C

**(A) For viscosity of the given liquid****Materials/chemicals/Apparatus required:-**

Ostwald viscometer, stop watch, R.D. Bottle, weight box, pipette, beaker, given liquid, water sample, etc.

**Principle:-** According to Poiseuill's Equation:-

$$[\eta] = \pi r^4 \rho t / 8 v l \quad \text{eq. (1)}$$

$$\rho = h d g \quad \text{eq. (2)}$$

Where,  $[\eta]$  = Viscosity,  $r$  = Radius,  $\rho$  = hydrostatic pressure,  $t$  = Time of flow of liquid,

$v$  = Volume of liquid,  $l$  = Path length

From above eq. (1) & eq. (2) we get

$$\frac{\eta_1}{\eta_2} = \frac{d_1}{d_2} \times \frac{t_1}{t_2}$$

$d_1$  = density of water

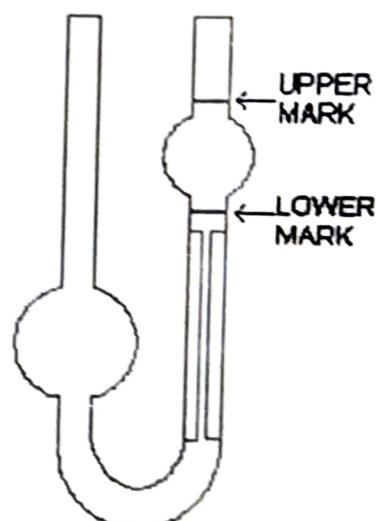
$d_2$  = density of liquid

$t_1$  = time of flow of water

$t_2$  = time of flow of liquid

$\eta_1$  = viscosity of water

$\eta_2$  = viscosity of liquid

**Procedure:-**

1. Introduce a known volume of distilled water in the lower bulb of cleaned & dried viscometer.
2. Hang the viscometer vertically and suck the water through the bulb containing central capillary tube until the water reaches above the upper mark
3. Allow the water to flow down and determine the time of flow between upper mark to lower mark. Take at least 3 readings of time of flow of water and record the time in table ( $t_1$  Sec.).

- The times of flow of given liquid and also record
4. Repeat the above procedure to get the times of flow of given liquid and also record these times in table ( $t_2$  Sec.).
  5. Now weigh empty dry relative density bottle ( $W_1$  gm) and then fill it with distilled water and again weigh it ( $W_2$  gm).
  6. Again make it empty and after drying, fill it with given liquid and weigh it ( $W_3$  gm).

**Observations:-**(a) Table for time of flow :-

S. No.	Time of flow of liquid (sec.)	Average time of flow of liquid (sec.) ( $t_2$ )	Time of flow of water (sec.)	Average time of flow of water (sec.) ( $t_1$ )
1.				
2.				
3.				

(b) For Density:-

$$\begin{aligned}
 \text{Weight of empty R.D. Bottle} &= W_1 \text{ gm} \\
 \text{Weight of R.D. Bottle + water} &= W_2 \text{ gm} \\
 \text{Weight of R.D. Bottle + liquid} &= W_3 \text{ gm} \\
 \text{Weight of liquid } W(l) &= (W_3 - W_1) \text{ gm} \\
 \text{Weight of water } W(w) &= (W_2 - W_1) \text{ gm}
 \end{aligned}$$

**Calculations:-**

$$\frac{\eta_1}{\eta_2} = \frac{d_1}{d_2} \times \frac{t_1}{t_2}$$

$$\text{Where } \frac{d_1}{d_2} = \frac{W(w)}{W(l)}$$

**Result:-**

The viscosity of the given liquid sample = ..... poise.

**Precautions:-**

1. The flow time and density of water/ liquid should be measured carefully.
2. During the preparation of the solution check that there is no turbidity.
3. The viscometer should be hanged vertically.

**Viva-Voce questions**

1. What is viscosity?
2. What is the unit of viscosity?
3. What is the viscosity of water at 25°C?
4. What apparatus is used for determining viscosity of a liquid?
5. What do you determine from pyknometer?
6. What is the least count of chemical balance?
7. What is the weight of rider?
8. What is time of flow?
9. What is the effect of temperature on viscosity?

(B) For surface tension of given liquid

Materials/chemicals/Apparatus required:-

Stalagmometer, R.D. Bottle, weight box, pipette, beaker, given liquid, water sample etc.

Principle:- The surface tension of a liquid by drop number method is calculated by the following equation-

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

$d_1$  = density of given liquid

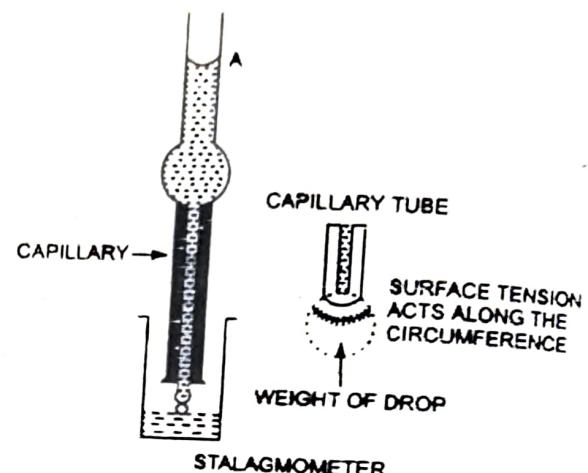
$d_2$  = density of water

$n_1$  = number of drops of liquid

$n_2$  = number of drops of water

$\gamma_1$  = surface tension of liquid

$\gamma_2$  = surface tension of water



Procedure :-

1. Attach small rubber tubing with a small pinch cork to the upper end of the cleaned stalagmometer.
2. Fill stalagmometer with distil water by sucking it above the mark A and close the pinch cork.
3. Clamp the stalagmometer and open the pinch cork such that the liquid flows in the form of drops. Suck the water through the bulb containing central capillary tube until the water reaches above the upper etched mark (A).
4. Close the pinch cock and clamp the stalagmometer.
5. Open the pinch cock gently and adjust it in such a way that the water flows in the form of drops.
6. Count the number of drops obtained when a fixed volume of water between the marks A and B. Take at least 3 readings of number of drops of water and record these number of drops in observation table.
7. Now remove the stalagmometer from the stand, wash it thoroughly and dry it.
8. Fill the stalagmometer with the given liquid and reset it in the stand.

9. Count the number of drops fallen for the same volume of the given liquid between the marks A and B. Take at least 3 readings of number of drops of liquid and record these number of drops in observation table.
10. Now weigh empty dry relative density bottle ( $W_1$  gm) and then fill it with distilled water and again weigh it ( $W_2$  gm).
11. Again make it empty and after drying, fill it with given liquid solution and weigh it ( $W_3$  gm).

Observations:-

(a) Table for time of flow:-

S. No.	Number of drops of liquid	Average ( $n_1$ )	Number of drops of water	Average ( $n_2$ )
1.				
2.				
3.				

(b) For Density:-

$$\begin{aligned}
 \text{Weight of empty R.D. Bottle} &= W_1 \text{ gm} \\
 \text{Weight of R.D. Bottle + water} &= W_2 \text{ gm} \\
 \text{Weight of R.D. Bottle + liquid} &= W_3 \text{ gm} \\
 \text{Weight of liquid } W(l) &= (W_3 - W_1) \text{ gm} \\
 \text{Weight of water } W(w) &= (W_2 - W_1) \text{ gm}
 \end{aligned}$$

Calculations:-

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

$$\text{Where } \frac{d_1}{d_2} = \frac{W_3 - W_1}{W_2 - W_1}$$

Result:-

The surface tension of the given liquid = ----- dynes/cm.

**Precautions:-**

- (1) The number of drops and density of water/liquid should be measured carefully.
- (2) The stalagmometer should be hanged vertically.
- (3) No air bubble should be formed while sucking the water/liquid into the stalagmometer.

**Viva-Voce questions**

1. What is surface tension of a liquid?
2. What is the unit of surface tension?
3. What is the surface tension of water at 25°C?
4. What Apparatus is used for determining surface tension of a liquid?
5. What do you determine from pyknometer?
9. What is the effect of temperature on surface tension?

## EXPERIMENT 8

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

**Material required:-** Coal, Analytical balance, Electric oven, Desiccator, silica crucible, long legged tong etc.

**Theory:-** Proximate analysis is the simplest type of analysis and it informs about the practical utilization of coal . The analysis is an assay rather than true analysis since the results have no absolute significance. However when the analysis is carried out in accordance with standard specifications , the reproducible results can be obtained which can be utilized for accessing the application of a fuel for a particular domestic or industrial use .

**Moisture :-** Coal contains two types of moisture :-

(a) **Free or surface moisture** :- The moisture which is lost in air drying is called as free moisture or surface moisture .

(b) **Internal moisture** :- The Internal moisture is that type of moisture which is retained by air dried coal. It is rough indicator of the coal maturity ( age or degree of coalification ) .

**Observations and calculations :-**

$$1. \text{ Weight of empty crucible} = X_1 \text{ gm.}$$

$$2. \text{ Weight of crucible + coal sample} = X_2 \text{ gm.}$$

$$3. \text{ Weight of crucible + coal sample after heating} = X_3 \text{ gm.}$$

$$\therefore \text{Weight of coal sample} = (X_2 - X_1) \text{ gm.}$$

$$\therefore \text{Weight of moisture} = (X_2 - X_3) \text{ gm.}$$

$$(X_2 - X_3) \times 100$$

$$\% \text{ of moisture} = \frac{(X_2 - X_3)}{(X_2 - X_1)} \times 100$$

**Result:-** Moisture content in given coal sample is ----- %

**Precautions:-**

1. Avoid mechanical loss due to rapid expulsion of volatile matter and temperature should be raised at slow rate.
2. After or before weighing the crucible should not be hold with fingers. The long legged tong should be used for putting the crucible inside the oven & in desiccators.
3. The weighing should be made up to fourth pace of decimal.

Viva-Voce questions

1. How many types of moistures are present in coal?
2. What is proximate analysis and what we analyze in it?
3. What chemical is placed inside the desiccator (or what is desiccant) and what is its utility?
4. What is calorific value?
5. Which form of coal has highest calorific value?

### EXPERIMENT 9

Object:- To determine strength of given HCl solution by titrating it against N/5 NaOH solution pH metrically.

Materials/chemicals/apparatus required:- pH meter, glass electrode, reference electrode, beaker, burette, Glass rod, NaOH solution (N/5) etc.

Principle:- When an alkali is added to an acid solution, the pH of the solution increase slowly, but at the vicinity of the equivalence point , the rate of change of pH of the solution is very rapid. From the sharp break in the curve, we can find the equivalence point, from which the strength of an acid can be calculated by normality equation.

Observations:-

Volume of acid (HCl) taken = 50 ml

Volume of alkali taken (ml)	0	1	2	3	4	5	6	7	8	9	10	11	12
pH of solution													

Calculations: -

$$N_1 V_1 = N_2 V_2$$

(For HCl)                  (For NaOH)

Strength of HCl (s) = Normality × Equivalent weight (in gm./litre)  
= Normality × 36.5 (in gm./litre)

Result: - The strength of the given acid (HCl) solution is ..... gm./litre

Precautions:-

- (1) The temperature control knob of the pH meter should be adjusted to the room temperature.
- (2) After the addition of the alkali, the solution should be thoroughly stirred.
- (3) First standardize the pH meter by taking a buffer solution of known pH.
- (4) Electrodes must be immersed properly in the solution.

Viva – Voce Questions

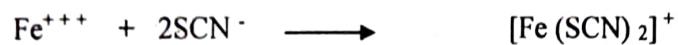
1. What is pH?
2. Which electrode do you use in this titration?
3. Do you use any indicator in this titration?
4. What is the construction of electrode?
5. What is the desirable pH range of drinking water?
6. How do you standardize pH-meter?
7. What is the effect of temperature on pH?
8. What is the pH of N/100 HCl solution?
9. What is the pH of  $10^{-8}$  N HCl solution?
10. Which will have more pH 0.1 M HCl or 0.1 M  $\text{CH}_3\text{COOH}$ ?
11. What is the pH of a solution containing 0.4 gm NaOH per litre?
12. Why standard hydrogen electrode is generally not used in pH measurement?
13. How the equivalence point in acid-base titrations is determined pH metrically?
14. Do you use any indicator in this titration?
15. How the equivalence point in acid-base titrations is determined pH metrically?

## EXPERIMENT 10

**Object:-** To determine iron concentration in the given sample solution by colorimetric method by using KSCN as a color developing agent and the measurement are carried out at  $\lambda_{\max}$  480nm .

**Materials/chemicals/apparatus required:-** Ferrous ammonium sulphate solution (100 ppm), potassium thiocyanate solution (KSCN 20%), 4N HNO<sub>3</sub>, colorimeter, distilled water, test tubes, test tube stand, graduated pipette (measuring pipette), wash bottle, beaker, Cuvette, etc.

**Principle:-** Ferrous ammonium sulphate solution is oxidized into ferric solution by oxidizing agent. Ferric ion reacts with thiocyanate ion to give a number of intensively red colored complex ions, which remain in true solution. Depending upon the thiocyanate concentration a series of ions can be obtained.



In general, the complexation of Fe<sup>+++</sup> with SCN<sup>-</sup> can be formulated as [Fe(SCN)<sub>n</sub>]<sup>3-n</sup> where n varies from 1 to 6. When the concentration of thiocyanate is low, the predominant colored species is [Fe(SCN)]<sup>2+</sup>, at 0.1 M thiocyanate concentration the predominant color species is [Fe(SCN)<sub>2</sub>]<sup>+</sup>, at very high thiocyanate concentration it is [Fe(SCN)<sub>6</sub>]<sup>3-</sup>.

A large excess of thiocyanate should be used to increase the intensity and also the stability of the complex ion. In order to suppress the hydrolysis of Fe<sup>+++</sup> the solution is to be maintained highly acidic by using HCl or HNO<sub>3</sub>



The iron concentration is measured at  $\lambda_{\max}$  480 nm.

According to **Lamberts law**, when a beam of monochromatic light is passed through homogeneous solution then the decrease in intensity of light with thickness of absorbing medium is directly proportional to the intensity of light, i.e.

$$-\frac{dI}{dx} \propto I \quad \text{or} \quad -\frac{dI}{dx} = k I \quad \text{or} \quad -\frac{dI}{I} = k dx$$

Integrating with in proper limits

$$\int_{I_0}^{I_t} dI/I = -k \int_0^L dx \quad \text{or} \quad \ln I_t / I_0 = -k L \quad \text{or} \quad I_t / I_0 = e^{-kL} \quad \text{or} \quad I_t = I_0 e^{-kL}$$

According to **Beer's law**, when a beam of monochromatic light is passed through homogeneous solution then the decrease in intensity of light with thickness of solution is directly proportional to the intensity of light as well as concentration (C) of solution i.e.,

$$-\frac{dI}{dx} \propto C I \quad \text{or} \quad -\frac{dI}{dx} = k' C I \quad \text{or} \quad -\frac{dI}{I} = k' C dx$$

Integrating with in proper limits

$$\int_{I_0}^{I_t} \frac{dI}{I} = -k' C \int_0^l dx \quad \text{or} \quad \ln \frac{I_t}{I_0} = -k' C l \quad \text{or} \quad 2.303 \log \frac{I_t}{I_0} = -k' C l$$

$$\log \frac{I_0}{I_t} = \frac{k' C l}{2.303} \quad \text{or} \quad A = \epsilon C l$$

where  $\epsilon = k'/2.303$  (Absorption Coefficient), and  $\log I_0/I_t = A$  (Absorbance or Optical density),  $l$  = length of solution

**Procedure:-** First of all prepare standard FAS solution of different concentrations as given below

Solution No.	(1) Blank Solution	(2)	(3)	(4)	(5)
FAS (ml) (100 ppm)	0	1	2	3	4
Distilled water (ml)	5	4	3	2	1
KSCN (20%) ml	5	5	5	5	5
HNO <sub>3</sub> (4N)	5	5	5	5	5

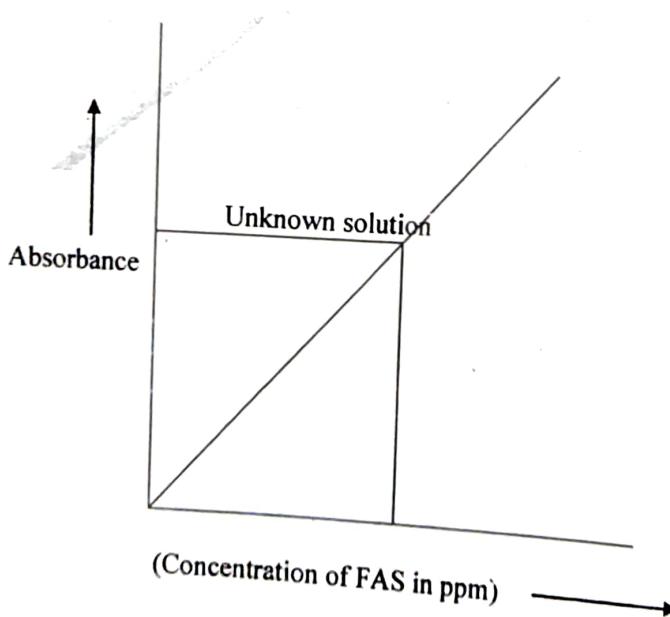
**Before starting the experiment set the instrument as follows:-**

1. Connect the spectrophotometer to the power supply and set the switch to the "ON" position. Spectrophotometer should display zero on transmittance scale. If it does not, then adjust it to zero by using knob labeled as % T adjustment.
2. Select appropriate wave length (480nm) region with the help of wave length knob.
3. Open the lid of the sample housing and insert a cuvette containing soln. no. -1(Blank Solution). Close the lid properly.

4. With the help of control knob make adjustment in such a manner that needle points to 100% transmittance or zero optical density ( i.e. absorbance = 0 )
5. Remove the cuvette after opening the lid and then close the lid tightly again.
6. Check whether the needle points to 100% transmittance or zero optical density. If it does not, then adjust again.
7. Now measure the absorbance of each of above prepared standard solution (Solution. no. 2, 3, 4 & 5) and unknown solution at 480nm against blank solution within 5 minutes of developing the colour. Tabulate the readings and plot a graph between absorbance and concentration. Find out the corresponding concentration of absorbance of unknown sample with the help of graph.

Observations:-

Soln. No.	Concentration of FAS	Absorbance
1.		
2.		
3.		
4.		
5.	Unknown solution	

Calculations:-

Using standard graph find concentration of unknown  $\text{Fe}^{2+}$  solution.

**Result:-**

The concentration of ferrous ion in a given sample solution is .....mg/ 15ml

**Precautions:-**

1. For calibration dilute solution should be used.
2. Cuvette should be used with great care.
3. The cuvette should be washed and cleaned properly.
4. Adjustment of pH should be done by adding sodium acetate properly.

**Viva-Voce Questions**

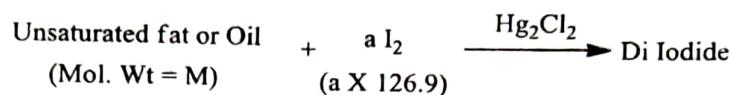
1. What is colorimeter?
2. What is spectroscopy?
3. What is Lambert's law?
4. What is Beer's law?
5. How do you prepare stock solution?
6. How do you calculate absorbance of the solution?
7. What color is developed by the reaction of ferrous ion and KSCN?
8. What are the advantages of colorimetric method over titrimetric & gravimetric method?

### Experiment No 11

**Object:-** To determine iodine value of given oil sample.

**Appratus/Chemicals required:-** Burette pipette, conical flask, beaker, sodium thiosulphate, Wij's solution, potassium iodide, carbon tetrachloride/chloroform, starch solution etc.

**Theory:-** Iodine value is defined as the number of grams of iodine needed for the halogenations of 100 grams of oil or fat.



From the above equation,  $a \times 126.9$  gm of iodine will add to  $M$  gm of unsaturated fat or oil.

$$\text{Number of gms of iodine needed for 100gms of oil or fat (iodine number)} = \frac{a \times 126.9}{M} \times 100$$

Thus iodine number or iodine value = Number of double bonds present in the acid component of the glyceride.

#### Significance:-

A high iodine number suggests that more double bonds are present in the glyceride while a low iodine number suggests the presence of few double bonds. Thus, iodine number gives an idea of "drying" character of fat or oil and help in the classification of oils into drying, semi-drying or non-drying categories.

**Procedure:-** Remove all the solid material and moisture present in the oil sample by straining it through a filter paper. Now take 25 ml each of  $\text{CCl}_4$  and Wij's solution in 500 ml titration flask. Depending upon the iodine value (200-50) of the sample, accurately weigh 0.05-0.5 gm. of the sample. Transfer it to one of the two flasks. Close the flasks with glass stoppers moistened with 10% KI solution. Shake the flasks to mix the contents intimately and keep it at below  $30^\circ\text{C}$  temp. in dark for about one hour. Now to both the flasks add 20 ml of KI solution and about 100 ml of distilled water. Now add sodium thiosulphate solution (N/10) so that the color of the solution becomes light yellow. Now add few drops of starch indicator solution so that the color becomes blue. Again add sodium thiosulphate drop by drop from burette until the solution becomes colourless. Record this reading in observation table 2.

Repeat this titration until two concordant readings are obtained. The same procedure and same reagents are used in second flask, only oil is not taken for blank correction and readings are recorded in table 1.

**Observations:-**

- (i) Weight of empty weighing tube =  $x_1$  gm
- (ii) Weight of weighing tube + oil sample =  $x_2$  gm
- ∴ Weight of the oil sample taken =  $(x_2 - x_1)$  gm =  $x$  gm.

**Table-1 Table for blank correction :-**

S. No.		Burette Reading		Volume of sodium thiosulphate consumed (ml) ( $V_b$ )
		Initial	Final	
1.				
2.				
3.				
4.				

**Table-2 Table for oil sample:-**

S. No.	Volume of oil (ml)	Burette Reading		Volume of sodium thiosulphate consumed (ml) ( $V_s$ )
		Initial	Final	
1.				
2.				
3.				
4.				

**Calculations:-**

- (i) Volume of  $\text{Na}_2\text{S}_2\text{O}_3$  (0.1N) equivalent to iodine (or ICl) consumed by  $x$  gm of the oil =  $(V_b - V_s)$  ml
- (iii) Iodine present in  $(V_b - V_s)$  ml of 0.1N iodine solution =  $\frac{(V_b - V_s) \times 0.1 \times 126.9}{1000} = y$  gm (let)
- (iv) Iodine value =  $y \times 100 / x$  gm

**Result:-**

The iodine value of the given oil sample is = .....

**Precautions:-**

1. Always add the Wij's solution with the help of burette. Never suck it into the pipette with mouth.
2. The addition of Wij's solution or KI solution to the two flasks should be done simultaneously as far as possible.
3. Use completely dry sample and glassware.
4. During titration, the flasks should be frequently shaken vigorously. This is to ensure complete titration of the iodine present in carbon tetrachloride layer.
5. Do not take mean of burette readings

**Viva-Voce questions:**

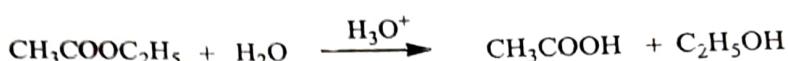
1. Why is the glass stopper moistened with KI solution?
2. Why is the reaction mixture kept in dark?
3. What is the effect of the presence of moisture in the sample on the test?
4. Can you predict contamination in any specific oil by measuring its iodine value?
5. How oils are classified based on their iodine value?
6. What type of oil can be used for the manufacture of paints and varnishes?
7. What type of oil can be used as lubricant?

### Experiment No 12

**Object:** Show that the hydrolysis of an ester in presence of an acid is an example of first order reaction.

**Apparatus and chemicals required:** Burette Pipette, Conical flask HCl (N/100), NaOH (N/50), ethyl acetate, phenolphthalein, ice etc.

**Theory:** When an ester is hydrolyzed in presence of an acid the carboxylic acid and alcohol are formed



It is found that in this reaction  $\text{H}_2\text{O}$  is present in excess, so its concentration is not changing appreciably and the concentration of ester is only changing appreciably so it is first order reaction.

The value of specific rate constant ( $k$ ) is calculated by following equation:

$$k = \frac{2.303}{t} \log \frac{V_\infty - V_t}{V_\infty - V_0}$$

Where  $V_0$  = volume of NaOH used initially

$V_t$  = Volume of NaOH used at time  $t$

$V_\infty$  = Volume of NaOH used at infinite time

**Procedure:** Fill the burette with NaOH(N/50) solution. Take 100ml of given HCl solution in conical flask or in a beaker. Now add 10 ml methyl acetate in to it. Now shake this solution and pipette out 10 ml of this solution immediately and pour it in to ice cold water. Add 2 drops of phenolphthalein indicator in to it. Now add NaOH solution from burette with constant stirring to it until permanent light pink colour is obtained. Note this reading in observation table. After each 10 minute take out 10ml solution and titrate it as above and note the reading in observation table In the end warm the solution and pipette out 10 ml solution and titrate it with NaOH solution directly as above .Note this reading in observation table.

Observation:

S. No.	Volume of the solution (ml)	Time in minute	Burette Reading		Volume of NaOH solution used (ml)
			Initial	Final	
1.	10.0				
2.	10.0				
3.	10.0				
4.	10.0				
5.	10.0				
6.	10.0				
7.	10.0				

Calculation: Calculate the value of specific rate constant (K) at different times by following equation

$$k = \frac{2.303}{t} \log \frac{V_0 - V_t}{V_0 - V_0}$$

Result:- Since the value of specific rate constant is almost constant from first order rate equation, and hence the hydrolysis of ester in presence of an acid is an example of first order reaction.

Precautions:

1. The reaction of the hydrolysis of an ester should be carried out at constant temperature except when hydrolysis at infinite time is done.
2. Ice should be essentially used for freezing the reaction after withdrawal of the reaction mixture from the conical flask.
3. After withdrawal of the reaction mixture, the titration should be carried out rapidly.

Viva voce Questions:-

1. What do you mean by order of reaction?
2. What do you mean by molecularity of reaction?
3. What are pseudo molecular reactions?
4. What is the unit of specific rate constant for first order reaction?
5. What is the effect of temperature on specific rate constant?
6. What are the examples of zero order, first order and second order reactions?