CHEMISTRY-1 LAB

(BS-CH – 191/BS-CH-291)

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Particulars of the Experiments Performed

Experiment No. 1

Title: Conductometric titration for determination of the strength of a given HCl solution by titration against a standard NaOH solution

A] Chemicals Required

1. Oxalic Acid
2. NaOH solution
3. HCl solution
4. Phenolphthalein indicator

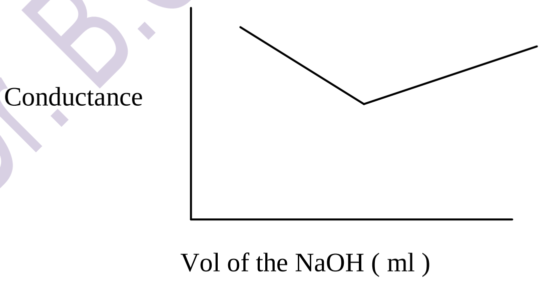
B] Apparatus Required

1. Conductivity meter
2. Burette
3. Pipette
4. Glass Rod

C] Theory

The conductometric method of titration can be used to determine the end point of ionic titrations since the specific conductance of a solution is proportional to the concentrations of the ions present in it. This method can be applied to acidimetric titrations.

When HCl is titrated with NaOH solution, the highly mobile H^+ ions [λ_0 (H^+)≈ 350 $ohm^{-1}cm^{-1}$] are progressively replaced by slower moving Na^+ ions [λ_0 (Na^+) ≈ 50 $ohm^{-1}cm^{-1}$] and the conductance of the solution decreases. After the end point, the conductance rises sharply as there is now an excess of OH^- ions, which are also highly mobile [λ_0 ≈ 198 $ohm^{-1}cm^{-1}$].
Hence the neutralisation of a strong acid by the addition of a strong base leads to a minimum conductance at the end-point. When, the conductance of the solution is plotted against the volume of titrant, a curve of the form shown in the adjoining figure is obtained.



D] Procedure

Part-I: Standardisation of NaOH solution

Approximately 0.1(N) NaOH solution is taken in to a burette. This being a secondary standard solution, must be standardised with the help of a primary standard. Here a known strength exactly 0.1 (N) oxalic acid solution should be used (supplied).

10 ml of the oxalic acid solution is taken into a 250-ml conical flask with the help of a pipette and 2-3 drops of phenolphthalein indicator is added. NaOH solution is run in from burette till the pink colour appears. The process is repeated thrice to get concordant readings. The readings are to be reported in table I.

Part-II: Conductance measurement using conductivity meter

10 ml of unknown HCl solution is pipetted out in the 150mL beaker and 50-60mL water is added. The conductivity cell is immersed in the beaker, then the instrument is switched on and the value is noted.. Standardized ~0.1(N) NaOH solution is taken into the burette and 0.5mL is added to the beaker at a time. The solution is stirred by stirrer and allowed to attain equilibrium after each addition from the burette. The conductance after each addition is noted. The conductance first decreases, reaches minimum and after the neutralisation point it increases. Some readings are to be taken after the minimum conductance value. The readings are to be reported in table II .

E] Results & Discussions

Room Temperature: …………

Table-I: Standardisation of NaOH solution

Sl No.	Vol. of oxalic acid (V_1 ml)	Burette Reading		Vol. of NaOH consumed (ml)	Concordant / Mean volume (V_2 ml)	St. of Oxalic acid (S_1)	St. of NaOH (S_2)
		Initial	Final				
1 2 3	10 10 10					0.1 N	…… N

Calculation: $V_1S_1 = V_2S_2$

Find S_2 here

Table-II: Conductance measurement using conductivity meter

Serial No.	Volume of unknown HCl in which the cell is dipped (V_3 ml)	Volume of NaOH added to the cell from burette (ml)	Observed Conductance [mho cm^{-1}]	Volume of NaOH consumed at end point [From graph] (V_4 ml)	Strength Of NaOH Solution (S_2)	Strength of HCl solution (S_1) $S_1 = (V_4S_2)/V_3$
1.		0.0				

2.	10ml	0.5			.... N	.... N
3.		1.0				
4.		1.5				
5.		2.0				
6.		2.5				
7.		3.0				
8.		3.5				
9.		4.0				
10.		4.5				
11.		5.0				
12.		5.5				
13.		6.0				
14.		6.5				
15.		7.0				
16.		7.5				
17.		8.0				
18.		8.5				
19.		9.0				
20.		9.5				
21.		10.0				
22.		10.5				
23.		11.0				
24.		11.5				
25.		12.0				
26.		12.5				
27.		13.0				
28.		13.5				
29.		14.0				
30.		14.5				
31.		15.0				

When conductance of solution in cell is plotted against volume of NaOH added a ‘V’ shaped curve is obtained and from the point of intersection we get the volume of NaOH required to neutralise the unknown acid solution.

Calculation:

$V_3S_3 = V_4S_2$

F] Conclusions

1. Instrumental methods of titration is more accurate than titrations using indicators which involve colour change during end point.
2. The end point of the titration of coloured solutions can be determined by sharp change in conductance value .

Experiment No 2

Title: pH-metric titration for determination of the strength of a given HCl solution against a standard NaOH solution

A] Chemicals Required:

1. Oxalic Acid
2. NaOH solution
3. HCl solution
4. Phenolphthalein indicator

B] Apparatus Required:

1. pH- meter
2. Magnetic stirrer
3. Burette
4. Pipette

C] Theory:

The pH-metric titration is basically a potentiometric titration. The acid-base titration goes through a variation in pH over a range. The instrument used to measure such pH variations generally consists of a glass electrode. Glass electrode is based on the principle that a potential difference is set up at the interface between glass and a solution in contact and it is dependent upon concentration of the latter.

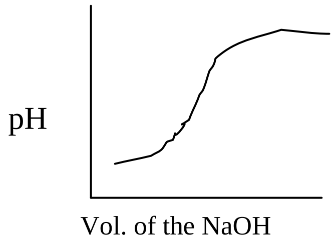
The common type of glass electrode consists of a tube of special soft glass of low melting point and high electrical conductivity terminating in a thin walled bulb. The inside of the electrode often contains Ag-AgCl electrode dipped in a solution of 0.1 (N) HCl or Pt wire dipped in a buffer solution-usually phthalate buffer of pH ~ 4.0. For measurement of pH, both the glass electrode and the reference electrode-usually saturated calomel electrode-are dipped in experimental solution and the potential difference between them is measured.

The commercial pH meter is calibrated in pH units (digital). Because of the existence of asymmetry potential in the glass electrode, the true value of E^0_G of an electrode is not directly measurable. For this reason the electrode is standardised by means of a series of buffers of known pH. The electrode may be represented as:

$\mathrm{Ag} \mid \mathrm{AgCl(s), HCl} \mid \mathrm{Glass} \mid \text{Test solution} \parallel \text{KCl (saturated)} \mid \mathrm{Hg_2Cl_2} \mid \mathrm{Hg}$

If the pH is plotted against volume of NaOH

A curve of the form shown in the adjoining figure is obtained.



D] Procedure

Part-I: Standardisation of NaOH solution

Approximately 0.1N NaOH solution is taken in a burette. This being a secondary standard solution, it must be standardised with the help of a primary standard. Here a known exactly 0.1N oxalic acid solution should be used (supplied).10 ml of the oxalic acid solution is taken into a 250-ml conical flask using a measuring cylinder. About 100 ml-distilled water is added to the acid solution and then 2-3 drops of phenolphthalein indicator is added. NaOH solution is added from burette till the pink colour appears. The process is repeated thrice to get concordant readings. The readings are given in Table-I.

Part-II: Measurement of pH using pH-meter

1. The instrument is already kept calibrated with buffer solutions of known pH values.
2. 10 ml of an unknown HCl solution is pipetted out in a beaker. 50mL water is added to ensure that the tip of the glass electrode gets dipped in it and the pH of the solution is noted from the digital display unit.
3. The pH of the solution is noted regularly after additions of 0.5 ml of NaOH at a time and stirring the solution.
4. After the end point of the titration is passed, as noted from the marked variation of pH, 5 ml of the titre is added and the end point of titration is located from the graph.
5. Knowing the volume of the standard N/10 NaOH solution added, the strength of the unknown acid solution is calculated from the general formula $V_{NaOH}S_{NaOH} = V_{HCl}S_{HCl}$. The readings are given in Table - II.

E] Results and discussions

Room Temperature: …….

Table-I: Standardisation of NaOH solution

Sl No.	**Vol. of oxalic acid (V_1 ml)**	**Burette Reading**		**Vol. of NaOH consumed (ml)**	**Concordant / Mean volume (V_2 ml)**	**St. of Oxalic acid (S_1)**	**St. of NaOH (S_2)**
		Initial	**Final**				
1	10					0.1 N	……. N
2	10						
3	10						

Calculation: $V_1S_1 = V_2S_2$

Table-II: Measurement of pH using pH-meter

Serial No.	**Volume of unknown HCl in which the cell is dipped (V_3 ml)**	**Volume of NaOH added to the cell from burette (ml)**	**Observed pH**	**Volume of NaOH consumed at end point [From graph] (V_4 ml)**	**Strength of NaOH solution (S_2)**	**Strength of HCl solution (S_1)** $S_3 = (V_4S_2)/V_3$
1.		0.0				
2.		0.5				
3.		1.0				
4.		1.5				
5.		2.0				

6.	10ml	2.5			.... N	.... N
7.		3.0				
8.		3.5				
9.		4.0				
10.		4.5				
11.		5.0				
12.		5.5				
13.		6.0				
14.		6.5				
15.		7.0				
16.		7.5				
17.		8.0				
18.		8.5				
19.		9.0				
20.		9.5				
21.		10.0				
22.		10.5				
23.		11.0				
24.		11.5				
25.		12.0				
26.		12.5				
27.		13.0				
28.		13.5				
29.		14.0				
30.		14.5				
31.		15.0				

When pH of solution in cell is plotted against volume of NaOH added a ‘S’ shaped curve is obtained . At pH = 7 on the y-axis, a horizontal line is drawn parallel to the x-axis so that it intersects the S-shaped curve . From the point of intersection we get the volume of NaOH required to neutralise the unknown acid solution.

Calculation:

$$V_3S_3 = V_4S_2$$

F] Conclusions:

1. The pH of titrant is abruptly changed from 5-6 to 8-9 and a sigmoid curve is obtained.
2. For a strong acid – strong base reaction the neutralization pH is considered as 7 and corresponding alkali volume is calculated. For other types of acid – base titration, this method fails.

Experiment No-3

Title : Determination of Cl^- ion in a given water sample by Argentometric method (Mohr's method)

A] Chemicals required :

1. N / 50 $AgNO_3$ solution
2. K_2CrO_4 indicator

B] Apparatus required :

1. Burette (50 ml)
2. Conical flask (250 ml)

C] Theory

Chlorides ions can be determined by titration of the water sample against a standard solution of $AgNO_3$ using potassium chromate as indicator in a neutral or faintly alkaline medium . When $AgNO_3$ solution is added to the water sample in presence of K_2CrO_4, the chlorides present in it are precipitated as AgCl. As soon as all the chlorides are precipitated out, the extra drop of $AgNO_3$ added in excess gives a red precipitate of silver chromate (Ag_2CrO_4) . This indicates the end point.

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

$$AgNO_3 + K_2CrO_4 = Ag_2CrO_4 + 2KNO_3$$

The pH of the solution should be between 7 to 8 because at higher pH Ag^+ ion is precipitated as AgOH. If the pH is lower than 7 to 8 then the chromate ion is converted to dichromate ion. The required pH range can easily be achieved by adding a pinch of pure $CaCO_3$. Excess $CaCO_3$ being insoluble, does not interfere.

D] Procedure

Part I : Blank correction

100 ml distilled water is taken in a conical flask and 3-4 drops of K_2CrO_4 indicator is added to it. The water is titrated with $AgNO_3$ (N/50) solution till the brick red ppt appears in the solution. The titration is repeated twice to obtain concordant values. The readings are given in table I. Let the concordant / mean reading be X ml. This volume of $AgNO_3$ should be deducted from the volume of $AgNO_3$ used in titrating the sample water as below.

Part II : Determination of Cl^- ion in sample water

100 ml sample water is taken in a conical flask, 1ml K_2CrO_4 indicator solution is added to it and the water is titrated with $AgNO_3$ (N/50) solution till the brick red color appears(ppt) in the solution. The titration is repeated twice to obtain concordant values. The readings are given in table II.

Let the concordant / mean reading be Yml.

E] Results and Discussions

TableI : Blank correction

Sl No.	Vol. of hard water sample (ml)	Burette Reading		Vol. of $AgNO_3$ consumed	Concordant / Mean volume of $AgNO_3$ (X ml)
		Initial	Final		
1	100				
2	100				
3	100				

TableII: Determination of Cl^- ion in sample water

Sl No.	Vol. of water sample (ml)	Burette Reading		Vol. of $AgNO_3$ consumed	Concordant / Mean volume of $AgNO_3$ (Yml)
		Initial	Final		
1	100				
2	100				
3	100				

Thus the concentration of Cl^- in given water sample = ………. mg / l .

Calculations:

Volume of (N/50) $AgNO_3$ solution used for titration of water sample = (Y – X) ml

= V_1 ml

Let N_1& N_2 be the normality of $AgNO_3$ and Cl^- solution respectively

Let V_2 be the volume of Cl^- solution = 100 ml

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

$$\text{or} \quad N_2 = \frac{V_1}{50 \times 100} = \ldots\ldots\text{ (N)}$$

Concentration of Cl^- ions = N_2 x Eq. wt of Cl^- = N_2 x 35.5 g/l

= N_2 x 35.5 x 1000 mg /l = ……. mg / l

F] Conclusion :

1**.** Since the concentration of Cl^- ion in sample water is less than 250 mg / l , this water can be safely used for drinking purpose.

2. Mohr's method of argentometric titration is an efficient method of detecting trace qantities of dissolved chlorides (Cl^-) in water.

Experiment No - 4

Title: Removal and estimation of hardness of water by Complexometric titration

A] Chemicals Required:

1. 100 ml tap water
2. pH = 10 buffer solution
3. 1 N NaOH solution
4. EBT indicator solution
5. Murexide indicator.
6. 0.01M di-sodium salt of EDTA

B] Apparatus Required:

1. Conical flask(250 ml)
2. Burette (50 ml)

C] Theory

The hardness of water is the measure of the capacity of the water to react with soap. Calcium and magnesium are the principal cations that impart hardness. The degree of hardness of drinking water has been classified in terms of equivalent $CaCO_3$ concentration as follows:

Soft	Medium	Hard	Very hard
0 – 60 mg/l	60 – 120 mg/l	120 – 180 mg/l	> 180 mg/l

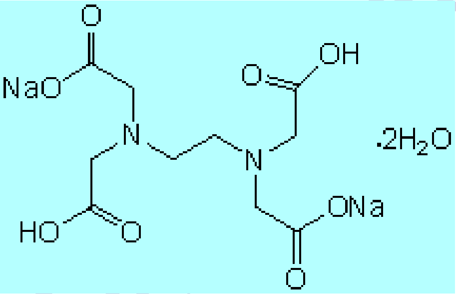


Fig. 1

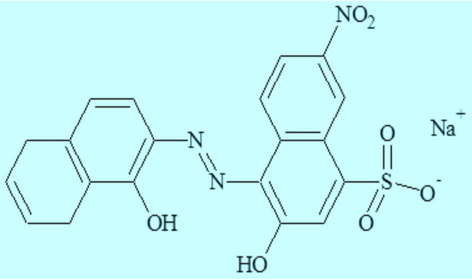


Fig. 2

The total hardness of water is equal to the sum total of alkaline metal cations present in it. The total hardness of a water sample can be determined by titration of the alkaline earth cations with ethylenediaminetetracetic acid (EDTA). The acid itself is practically insoluble in water so it is typical to use the disodium salt of EDTA ("Na_2H_2Y"). The structure of the dehydrate sodium salt is shown in Fig.1.

Eriochrome black T (EBT), is used as an indicator in this titration. Eriochrome Black-T (EBT) is Sodium-1- (1-hydroxy-2-naphthylazo)-6-nitro-2-napthol-4-sulphonate. The structure of theEBT indicator is shown in Fig. 2.

EBT forms 1:1 complexes with calcium and magnesium ions. But EBT-Mg complexes are more stable than EDTA-Mg complexes, whereas EBT-Ca complexes are less stable than EDTA-Ca complexes. Moreover, the EBT-Mg complex is wine-red color while everything else is either colorless or blue (uncomplexed EBT is blue as well).

The structure of Ca-EDTA complex and Mg-EBT complex are shown in Fig. 3 & 4.

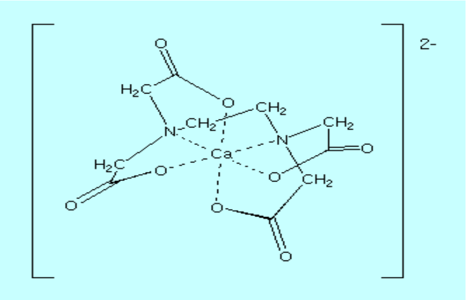


Fig. 3

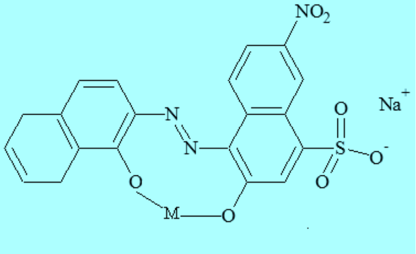


Fig. 4

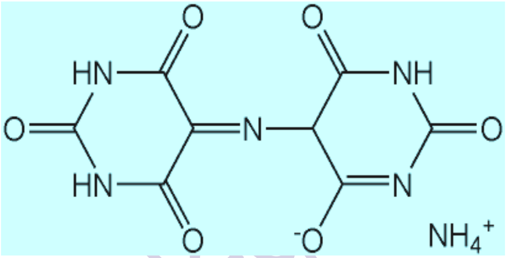


Fig. 5

When EBT is added to the pH 10 buffered water sample the wine-red color of the EBT-Mg complex is evident. During the titration EDTA complexes any calcium ions in the sample and continues to do so in preference to magnesium ions (which are bound to the EBT) until all the calcium ions are complexed. At that point excess EDTA added will remove magnesium ions from the EBT-Mg complex. As the complex comes apart the wine-red color changes to the blue color of uncomplexed EBT. This is the end-point of the titration.

At higher pH, about 12.0, Mg^{2+} ions precipitate and only Ca^{2+} ions remain in the solution. At this pH, murexide indicator forms a pink colour with Ca^{2+} .The structure of murexide indicator is shown in Fig. 5.

When EDTA is added Ca^{2+} gets complexed resulting in the change from pink to purple, which indicates end point of the reaction.

D] Procedure:

Part-I: Estimation of total hardness of water

100-ml tap water is taken in a conical flask using a measuring cylinder. Few drops of ammonia-buffer solution (pH=10) is added to the conical flask till the smell of ammonia becomes persistent. 1 drops of EBT indicator is added to the conical flask. The sample water is then titrated with 0.01(M) EDTA solution till the colour changes from wine-red to blue.The titration is repeated twice to obtain concordant values. The readings are given in table I .

Part-II: Estimation of Calcium hardness of water

100-ml tap water is taken in a conical flask using a measuring cylinder. Approx. 2ml 1N NaOH solution is added to the conical flask to make the pH ~ 12-13 . 0.1 to 0.2 gm of murexide indicator mixture is added to the water sample. The sample water is then titrated with 0.01(M) EDTA solution till the colour changes from pink to purple.The titration is repeated twice to obtain concordant values. The readings are given in table II .

E] Results & Discussions

TableI : Determination of total hardness (Ca and Mg) of water using EDTA solution

Sl No.	Vol. of hard water sample (ml)	Burette Reading		Vol. of EDTA consumed	Concordant / Mean volume of EDTA (V_1 ml)	Hardness of water sample ($10V_1$ ppm)
		Initial	Final			

TableII :

1	100					
2	100					
3	100					

Determination of Calcium hardness of water using EDTA solution

Sl No.	Vol. of hard water sample (ml)	Burette Reading		Vol. of EDTA consumed (ml)	Concordant / Mean volume of EDTA (V_2 ml)	Calcium hardness of water sample ($10V_2$ mg/l)
		Initial	Final			
1	100					
2	100					
3	100					

Calculation:

1000 ml (M) EDTA solution ≡ 100 gm of $CaCO_3$

V ml 0.01M EDTA solution ≡ V mg of $CaCO_3$

100 ml hard water contains V mg of $CaCO_3$

1000 ml hard water contains 10V mg of $CaCO_3$

$\therefore$ Total hardness of given water sample = $10V_1$ = …….. mg / l

$\therefore$ Calcium hardness of given water sample = $10V_2$ = …….. mg / l

$\therefore$ Magnesium hardness of given water sample = $10V_1 - 10V_2$ = …….. mg / l

F] **Conclusion**

1. The total hardness of tap water tested is in the range of 100 mg/l – 120 mg/l in terms of $CaCO_3$ equivalent. Hence the hardness of water is medium
2. Concentration of Ca salts in tap water is more than the Mg

Experiment No. 5(A)

Title: Determination of viscosity of a sugar solution

A] **Chemicals Required:**

Specific Concentration (2% or 3%) sugar solution

B] **Apparatus Required:**

1. Ostwald Viscometer
2. Specific Gravity Bottle
3. Digital stop watch

C] **Theory:**

Let us consider a column of liquid in a capillary glass tube. The liquid column flows downward due to pressure difference. The liquid column in the tube may be considered as being composed of a large number of concentric cylindrical layers, which slide over one another and move downward due to pressure gradient. As the liquid layer in contact with the glass wall tends to move, a shearing effect is produced by the glass wall that destroys the relative motion of the moving layer. In this way each of the moving liquid layer experiences a shearing effect by its preceding liquid layer. Thus the velocity of the different liquid layer is not on the same, but changes uniformly according to their distance from the glass wall. The velocity becomes maximum at the center and minimum at the surface of the wall. The shearing effect or the internal friction which is produced by the glass wall and the different liquid layers, when a liquid flows, and which opposes this flow of the liquid is called viscosity of the liquid. The coefficient of viscosity is a measure of this internal friction or resistance to flow. Newton supposed that the force, which opposes the movement of a particular cylindrical liquid layer, that is the viscous drag, is proportional to the velocity gradient, 'du/dx 'and the area 'A 'of the liquid layer.

The coefficient of viscosity η is numerically equal to the tangential force per unit area required to maintain unit difference of velocity of displacement of two parallel planes at unit distance apart, the space being filled with the viscous liquid.

Coefficient of viscosity = η = Tangential force/(Area × velocity gradient)

The unit of η is thus dyne-second per square-cm. This unit is called a 'POISE' after the name Poiseuille.

According to Poiseuille formula for the flow of a homogeneous liquid through a capillary tube,

$$\eta = \frac{\pi p r^4 t}{8Vl} \text{ .... } \qquad \text{Eq-1}$$

where p = driving force, r = radius of the tube, t = time required for the V volume of the liquid to flow through the tube of length l.

In a simple Ostwald viscometer, the force driving a liquid of viscosity η_1 through the capillary depends on the difference in liquid level 'h', the density 'd_1' and the acceleration due to gravity 'g' and is given by the expression 'hgd_1'. If exactly the same volume of a liquid of viscosity η_2 is introduced into the tube, the driving force becomes equal to 'hgd_2', where 'd_2' is the density of this liquid.

By Eq-1, the viscosity (η) is equal to [$\pi p r^4 t/8Vl$], i.e., for a given apparatus and the same volume V of liquid, η is proportional to the driving force and to the time of overflow. Hence,

$$\text{Relative viscosity} = \eta' = \frac{\eta_2}{\eta_1} = \frac{hgd_2}{hgd_1} \times \frac{t_2}{t_1} = \frac{d_2 t_2}{d_1 t_1} \text{ ....Eq-2}$$

This expression gives the viscosity of the second liquid relative to that of the first.

The viscosity of a solution varies with its concentration. If the viscosities of solutions of various concentrations of a substance are plotted against concentrations, a straight line is obtained. Now by determining the relative viscosity of the unknown solution at the laboratory temperature, its concentration can be found out from the curve.

D] Procedure:

Part-I: Determination of time of flow of liquids

1. The viscometer is washed properly. A certain volume of water is introduced into the larger bulb. By sucking, water is forced up through the capillary until the level of water rises above the upper mark. The liquid is then allowed to flow through the capillary and the stopwatch is started when the meniscus passes the upper mark and stopped when it passes the lower mark. Time is noted in table 1. The procedure is repeated twice.

Part-II: Determination of density of liquid

The weight of empty specific gravity bottle, specific gravity bottle filled with water and specific gravity bottle filled with sugar solutions are taken. The density of sugar solution is calculated and noted in table II.

Part-III: Determination of viscosity of liquid

The viscosity of the sugar solution is determined using Eq. (2) and reported in table III.

E] Result & Discussions

Room temperature : ....

Table-I: Determination of time of flow of liquids

Concentration of sugar solution (%)	No. of observation	Time required (secs)	Mean time (secs) (t)	
			water(t_2)	sugar(t_1)
0 (water)	1.			-
	2.			
	3.			
2 or 3%	1.		-	
	2.			
	3.			

Table 2: Determination of density of sugar solution

Weight of the empty specific gravity bottle (W_1) = ... g

Weight of the specific gravity bottle filled with water (W_2) = ... g

Weight of water (W_3) = (W_2 - W_1) = ... g

Density of water at room temperature (ρ_2) = ... g / ml

Concentration of sugar solution (%)	Weight of sp. gravity bottle + sugar solution (W_4 g)	Weight of sugar solution (W_5 g) $W_5 = W_4 - W_1$	Density of sugar solution (g/ml) $\rho_1 = \frac{W_5}{W_3} \times \rho_2$

2 or 3%			

Table 3: Determination of viscosity of liquid

Viscosity of water at room temperature (η_2) = ... centipoise

Concentration of sugar solution (%)	**Density of sugar solution (g/ml)** (ρ_1)	**Viscosity (centipoise)** $\eta_1 = \eta_2 \times \frac{\rho_1 t_1}{\rho_2 t_2}$
0	ρ_2 =	η_2 =
2 or 3%		

F] Conclusion

1. Viscosity is directly proportional to concentration of the solution within certain range of concentration.
2. Viscosity is inversely proportional to temperature.

Experiment No. 5(B)

Title: Determination of surface tension of a sugar solution by drop count method

A] Chemicals Required:

Specific Concentration (2% or 3%) sugar solution

B] Apparatus Required:

1. Stalagmometer
2. Specific Gravity Bottle
3. Beaker

C] Theory: When a liquid is allowed to flow through a capillary tube, a drop is formed at its lower end. It increases to a certain size and falls of. The size of the drop depends on the radius of the capillary and the surface tension of the liquid. The surface tension acting along the circumference of the capillary tube supports the drop in the upward direction. The measurement of surface tension of liquid is based on the fact that the drop of the liquid at the lower end of capillary falls down when weight of drop becomes just equal to the surface tension.

1. Now the force of gravity exerted on the drop is given by vdg where v=volume, d= density and g= gravity, the force of gravity will pull down the drop.
2. The force tending to uphold the drop is given by $2\pi r\Upsilon$ where $2\pi r$ is the circumference of the capillary of radius r and Υ is the surface tension.

When the two forces are balanced

$$2\pi r\Upsilon = vdg \quad \ldots\ldots\ldots(1)$$

If n is the number of drop in volume V of the liquid then volume of each drop will be v=V/n

From (1) we have $2\pi r\Upsilon = V/n\ dg$ …………(2)

Consider two liquids of densities d_1 and d_2 having the surface tension Υ_1 and Υ_2. Let the number of drops counted for the same volume v of the two liquids be n_1 and n_2 respectively. Then $2\pi r\Upsilon_1 = v/n_1\, d_1 g$ for 1st liquid ……..(3) and

$$2\pi r\Upsilon_2 = v/n_2\, d_2 g \text{ for 2nd liquid}\ldots\ldots\ldots(4)$$

Dividing equation ¾ we have $\Upsilon_{1/}\ \Upsilon_2 = n_{2/}n_1$ x $d_{1/}d_2$ where Υ_1 and Υ_2 are the surface tensions of two individual liquids and d_1 and d_2 are their densities respectively. Thus for the determination of surface tension of any liquid, the number of drops produced fro equal volume of two liquids and their densities must be known. Knowing the surface tension of the reference liquid (e.g. water) the surface tension of the given liquid can be determined.

D] Procedure: 1. Immerse the lower end of the stalagmometer in a beaker containing water. Suck the watern until it rises above the mark A

2. Now let the water flow out and start counting the number of drops when the water meniscus just reaches the mark A and counting is stopped when the water meniscus passes the lower mark B. Let the number of drops be n_w.

Repeat to get three readings

3. Clean the stalagmometer. Fill it with the sugar solution until it rises above the mark A and count the number of drops when a fixed volume of sugar flows between the mark A and B as before. Let the number of drops be n_s
4. Weigh the empty specific gravity bottle, water filled bottle and sugar filled bottle

Table1: Drop Count for water and sugar solution

Sl. No	water		sugar	
	No. of Drops	Mean	No. of Drops	Mean

1				
2		n_w		n_s

Table 2: Determination of density of sugar solution

Weight of the empty specific gravity bottle (W_1) = ... g

Weight of the specific gravity bottle filled with water (W_2) = ... g

Weight of water (W_3) = (W_2 - W_1) = ... g

Density of water at room temperature (ρ_2) = ... g / ml

Concentration of sugar solution (%)	**Weight of sp. gravity bottle + sugar solution (W_4 g)**	**Weight of sugar solution (W_5 g)** $W_5 = W_4 - W_1$	**Density of sugar solution (g/ml)** $\rho_1 = \frac{W_5}{W_3} \times \rho_2$
2 or 3%			

E] Calculation

$\Upsilon_s/\Upsilon_w = d_s/d_w \times n_w/n_s$

F] Result

The surface tension of the sugar solution= dyne/cm

Date:________ **Experiment No. - 6**

Title: Determination of R_f values of organic dyes by thin layer chromatography (TLC)

Chemicals Required:

1. Silica gel
2 Calcium Sulphate
2. Butanol – 1
3. Propanol – 1
4. Methyl Orange
5. Bromothymol Blue

Apparatus Required:

1. TLC jar with lid
2. Capillaries
3. measuring cylinder
4. Glass plates

Theory: Thin layer chromatography (TLC) is an efficient, fast and simple method of separating the mixture. In this technique a thin layer of silica gel or alumina is spread on a glass or plastic. The basic principle of separation is the selective absorption of the components of the mixture on the stationary phase, when they are carried through it by the flow of mobile phase.

The sample is spotted at one end of glass plate coated with thin layer of adsorbent. The plate is placed in a covered jar containing the solvent for developing. The solvent rises up by capillary action and the solute is distributed between the stationary (absorbent) phase and the mobile phase. A solute which is more strongly adsorbed will migrate slowly up the TLC plate. The sample is subsequently separated.

Procedure: **Preparation of TLC plate:**

1. A clean glass plate is taken. About 100 g of Silica Gel is mixed with 200 ml of water with 10 g of Calcium Sulphate. It is mixed properly to remove lumps and make uniform slurry.
2. The slurry is poured on the glass plate and spread it out with the help of applicator.
3. Let the plate be dried for twenty minutes at 110°C in a hot air oven to dry and activate the adsorbent.

Application of the organic dyes:

4. The solutions of each of the given organic dyes is taken with capillary applicator and touched at the bottom of the silica gel coated glass plate. The spot of the solution should not be larger than 2mm in diameter. The spot is allowed to dry.

Developing

5. The developing solutions, also called adsorbate, are ethanol and butanol.
6. The ethanol and butanol solutions are placed into two different jars and the plate is placed inside the jar in such a manner that the lower edge of the absorbent layer is immersed in the solvent. The jar is covered and the solvent is allowed to ascend along the plate.
7. When the developer ascends to the required height (3/4 of the plate) the compound front and solvent front are marked and measured with scale.

Visualization

8. The centre of each spot is marked. The distance travelled by each component and the solvent is measured. The R_f value is determined.

Observations and Calculations

Sl. No.	**Organic Dyes**	**Distance of organic dye the line of application (dm)**	**Distance of solvent (ds)**	**R_f = dm / ds**
1.	**Methyl Orange**			
2.	**Bromothymol Blue**			

Precautions:

1. The plate prepared should be of uniform thickness.
2. The spot of the compound should not exceed 2mm in diameter.
3. Different spot should be at least 5mm apart.

Experiment No - 7

Title: Determination of 'Partition Coefficient' of acetic acid between n-butanol & water

A] Chemicals Required

1. Acetic Acid
2. n-butanol
3. Oxalic Acid
4. Phenolphthalein indicator
5. NaOH solution

B] Apparatus Required

1. 250-ml reagent bottles (2)
2. 100 ml measuring cylinder
3. 50 ml burette
4. Electro-mechanical shaker
5. 250 ml conical flask
6. 10 ml pipette

C] Theory

When a solute is shaken with two immiscible solvents, it will distribute itself between the two solvents in accordance with 'Nernst Distribution Law' if it is soluble in each of them. If the solute has normal molecular weight in both the solvents (i.e., neither associated nor dissociated), the partition will take place according to the following equation:

Distribution coefficient (Partition coefficient), $K_d = C_1/C_2$;

where C_1 and C_2 refer to the concentrations of the solute in the solvents 1 and 2 respectively.

The value of K_d is constant and is independent of the amount of solute present, provided that the temperature is constant.

If however, in one solvent, the solute has the normal molecular weight, but in the second solvent, say in solvent 2, is associated as

$$n\,(HA) \rightleftharpoons (HA)_n$$

then, at a given temperature the ratio C_1/C_2 will no longer be constant. According to the law of mass action, the concentration of the single molecules in the second solvent is proportional to the nth root of the total concentration. Therefore, the ratio $C_1/(C_2)^{1/n}$ should be constant.

In case of molecules like benzoic acid, which generally exists as dimer in aprotic solvents, the value of 'n' is expected to be 2.

To obtain a constant value of K_d, various powers of C_1 and C_2 may be tested. This would also give some idea about the nature of association and dissociation of the solute in the solvents concerned.

D] Procedure

Part-I: Standardisation of NaOH solution

Approximately 0.1N NaOH solution is taken in a burette. This being a secondary standard solution, it must be standardised with the help of a primary standard. Here a known exactly 0.1N oxalic acid solution should be used (supplied).10 ml of the oxalic acid solution is taken into a 250-ml conical flask using a measuring cylinder. About 100 ml-distilled water is added to the acid solution and then 2-3 drops of phenolphthalein indicator is added. NaOH solution is added from burette till the pink colour appears. The process is repeated thrice to get concordant readings. The readings are given in table-II.

Part-II: Determination of Partition Coefficient of Acetic Acid

The chemicals and required apparatus are collected and cleaned and then the compositions of two bottles are prepared according to the table – I.

Table I : Composition of each bottle

Bottle no.	Vol. of acetic acid in n-butanol (ml)	Vol. of n-butanol (ml)	Vol. of distil water
1.	30	10	60
2.	20	20	60

After preparing the two bottles the following steps are performed:

a) The bottles are properly stoppered and placed in a mechanical shaker for shaking uniformly. The shaking process is continued for ~ 20 minutes. Then the bottles are allowed to stand undisturbed for about 10 minutes to ensure complete separation of two layers; the upper layer is the 'organic layer (n-butanol)' while the lower layer is the 'aqueous layer'.

b) 10 ml of n-butanol layer from the bottle-1 is pipetted out in a conical flask and this is titrated against (0.1 N) NaOH solution using phenolphthalein indicator. The process is repeated to get the concordant values.

c) Similarly 10 ml of aqueous layer from bottle-1 is pipetted out in a conical flask and this is titrated against (0.1 N) NaOH solution using phenolphthalein indicator. The process is repeated to get the concordant titre values.

d) The above procedure is carried out for bottle-2 and the readings are given in table-III.

E] Results & Discussions

Room Temperature: …………

Table-II: Standardisation of NaOH solution

Sl No.	Vol. of oxalic acid (V_1 ml)	Burette Reading		Vol. of NaOH consumed (ml)	Concordant / Mean volume (V_2 ml)	St. of oxalic acid (S_1)	St. of NaOH (S_2)
		Initial	Final				
1	10					0.1 N	...... N
2	10						
3	10						

Calculation:

$$V_1S_1 = V_2S_2$$

Table III : Standarization of aqueous layer and n-butanol layer by NaOH solution

Bottle no.	**Layer**	**Volume of layer (V_1 ml)**	**Burette reading**		**Volume of NaOH consumed (ml)**	**Mean volume (ml)**	**St. of NaOH (S_2)**	**St. of acetic acid (S_1)**	**K_d** $K_d=S_b/S_w$
			Initial	**Final**					
1.	butanol (upper)	10				V_b =		S_b =	
		10							
	water (lower)	10				V_w =		S_w =	
		10							
2.	butanol (upper)	10				V_b =		S_b =	
		10							
	water (lower)	10				V_w =		S_w =	
		10							

Calculation:

Bottle No. 1:

Concentration of acetic acid in n-butanol layer = $S_b = [V_b S_2/10]$ (N)

Concentration of acetic acid in aqueous layer = $Sw = [V_w S_2/10]$ (N)

Therefore Partition coefficient = $K_d = S_b/S_w$

[One can take the reverse ratio also. However, amendments are to be made in the table for this consideration.]

Similar technique is adopted bottle-2 and the partition coefficient (K_d) is calculated separately.

F] Conclusion

1. Acetic acid is found to be more soluble in organic solvent than water.
2. The relative solubility of natural products into aqueous and organic solvents can be determined.

Date: ……… **Experiment No. - 8**

Title: To study the adsorption of acetic acid on active charcoal and to verify the Fruendlich isotherms

A] Chemicals Required:

1. Activated charcoal
2. 0.5M acetic acid
3. 0.1 M NaOH solution

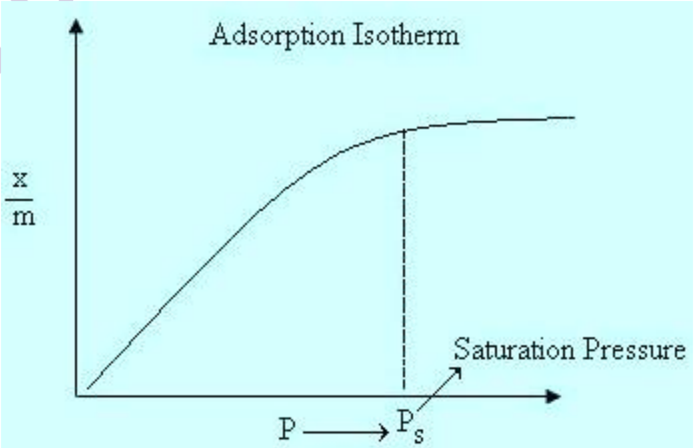
B] Apparatus Required:

1. Conical flask(250 ml)
2. Burette (50 ml)
3. Filterpaper
4. Stirrer

C] Theory

Adsorption is the adhesion of atoms, ions or molecules from a gas, liquid or dissolved solid to a surface. This process creates a film of the adsorbate on the surface of the adsorbent. This process differs from absorption, in Which a fluid (the adsorbate) is dissolved by or permeates a liquid or solid (the absorbent) respectively. Adsorption is a surface phenomenon, while adsorption involves the whole volume of the material.

The term sorption encompasses both processes, while desorption is the reverse of it. The process of adsorption is usually studied through graphs known as adsorption isotherm. It is the graph between the amounts of adsorbate (x) adsorbed on the surface of adsorbent (m) and pressure at constant temperature. Similar to surface tension, adsorption is a consequence of surface energy. In a bulk material, all the bonding requirements (be they ionic, covalent or metallic) of the constituent atoms of the material are filled by other atoms in the material. However, atoms on the surface of the adsorbent are not wholly surrounded by other adsorbent atoms and therefore can attract adsorbates. The exact nature of the bonding depends on the details of the species involved, but the adsorption process is generally classified as physisorption (characteristic of weak van der Waals forces) or chemisorption (characteristic of covalent bonding). It may also occur due to electrostatic attraction.



In the process of adsorption, adsorbate gets adsorbed on adsorbent.

$$\text{Adsorbate} + \text{Adsorbent} \underset{\text{desorption}}{\overset{\text{Adsorption}}{\rightleftharpoons}} \text{Adsorption}$$

$$A + B \rightleftharpoons AB$$

According to Le-Chatelier principle, the direction of equilibrium would shift in that direction where the stress can be relieved. In case of application of excess of pressure to the equilibrium system, the equilibrium will shift in the direction where the number of molecules decreases. Since number of molecules decreases in forward direction, with the increases in pressure, forward direction of equilibrium will be favoured.

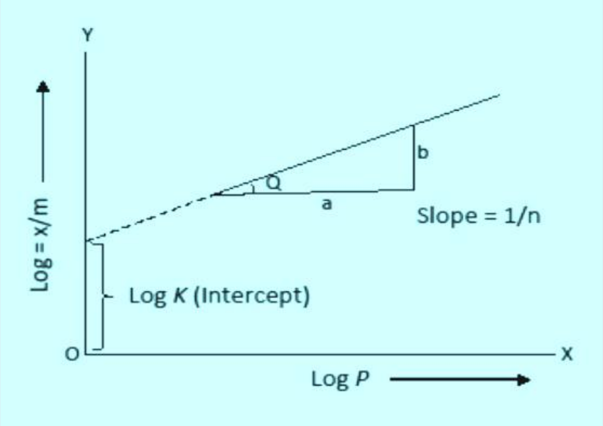
From the graph, we can predict that after saturation pressure Ps, adsorption does not occur anymore. This can be explained by the fact that there are limited numbers of vacancies on the surface of the adsorbent. At high pressure a stage is reached when all the sites are occupied and further increase in pressure does not cause any difference in adsorption process. At high pressure, adsorption is independent of pressure.

Freundlich Adsorption Isotherm

In 1909, Freundlich gave an empirical expression representing the isothermal variation of adsorption of a quantity of gas adsorbed by unit mass of solid adsorbent with pressure. This equation is known as Freundlich Adsorption Isotherm or Freundlich Adsorption equation or simply Freundlich Isotherm.

$$\frac{x}{m} = kP^{\frac{1}{n}}$$

Where x is the mass of the gas adsorbed on mass m of the adsorbent at pressure p and k, n are constants whose values depend upon adsorbent and gas at particular temperature. Though Freundlich Isotherm correctly established the relationship of adsorption with pressure at lower values, it failed to predict value of adsorption at higher pressure.



Limitations of Freundlich Isotherm

Freundlich isotherm only approximately explains the behaviour of adsorption. The value of 1/n can be between 0 and 1, therefore the equation holds good only over a limited range of pressure.

- When 1/n = 0, x/m is constant, the adsorption is independent of pressure.
- When 1/n =1, x/m = kP, i.e. x/m $\propto P$, adsorption is directly proportional to pressure.

Experimental results support both of the abovementioned conditions. At high pressure, the experimental isotherms always seem to approach saturation. Freundlich isotherm does not explain this observation and therefore, fails at high pressure.

D] Procedure

A well cleaned and dried six stoppered reagent bottles are taken and leveled from 1 to 6 as shown in the below mentioned table.

Table 1. Different concentrations of acetic acid in bottles 1 to 6

Bottle No	**1**	**2**	**3**	**4**	**5**	**6**
Acetic acid 0.5 M in mL	50	40	30	20	10	0
Distilled water in mL	0	10	20	30	40	50

Bottle No	**Final reading**	**Volume of 0.1M NaOH used (mL)**	**Mean**
1 (i) (ii)			
2 (i) (ii)			
3 (i) (ii)			
4 (i) (ii)			
5 (i)			

(ii)			
6 (i) (ii)			

Weigh 2 g of activated charcoal and in quick succession add to each of the above 6 bottles separately. Then shake the bottles at room temperature for about 30 minutes. After this the contents inside the bottles are filtered into six different conical flasks using six different filter paper separately and also labelled for proper identification. Filtration is followed by titration of each of the six conical flasks (taking 10 mL of aliquots each time with a pipette) separately by using 0.1M NaOH solution and phenolphthalein as indicator. Titraton of the stock solution of the acetic acid also should be carried out with 0.1M NaOH solution.

E] Results & Discussions

Room temperature=……..^{o}C
Weight of the activated charcoal in each bottle= 2g
Volume of acetic acid solution for titration in each case= 10 mL

Table2: Determination of initial concentration of acetic acid in solution

F] Calculation

(i) Initial concentrations of Ci in bottles 1 to 6

Bottle No	Initial Concentartion (Ci)	Value (M)
1	50/50 x 0.5 M	
2	40/50 x 0.5 M	
3	30/50 x 0.5 M	
4	20/50 x 0.5 M	
5	10/50 x 0.5 M	
6	0/50 x 0.5 M	0 M

(ii) Equilibrium concentration of the acid solutions in each of the bottles (1 to 6)after adsorption with charcoal. Molarity of acetic acid after adsorption is calculated as shown,

Bottle No	Initial conc before adsorption (Ci)	Equilibrium conc of acid after adsorption (C)	Amount of acid adsorbed, x=(Ci - C)/20
1			
2			
3			
4			

5			
6			

$M_{\text{acetic acid}}$ = (0.1M NaOH x Volume of NaOH)/ Volume of acetic acid taken for titration
(iii) Amount of acetic acid adsorbed =x
x= decrease in the amount of acetic acid in solution
= decrease in acetic acid x volume of solution in litres
= (Ci – C) x 50/1000 = (Ci – C) /20 moles

The amount of adsorbent (m) = 2g

Bottle No	**log C**	**log(x/m)**
1		
2		
3		
4		
5		
6		

A Graph is plotted log(x/m) (as ordinate) versus log C (as abscicca). The straight line of proves the validity of Freundlich adsorption isotherm.

Experiment No. - 9

Title: Determination of dissolved oxygen in the given sampleof water.

A] Chemicals Required

1. Na_2SO_4 (N/10)
2. $MnSO_4$ solution
3. KI
4. Starch
5. conc. H_2SO_4

B] Apparatus Required

1. Burette
2. Pipette (10 ml)
3. Conical flask
4. Beaker (250 ml)

C] Theory

Oxygen itself is not a pollutant in water but its deficiency is anindicator of several types of pollution in water.

Dissolved oxygen (DO) is determined by Winkler's method or iodometric titration. The dissolved oxygen in water oxidizes KI and an equivalent amount of iodine is liberated. This iodine is titrated against a standard hypo solution. However since dissolved oxygen in water is in molecular state and is not capable of reacting with KI, therefore an oxygen carrier such as manganese hydroxide is used.

The method involves introducing a conc. solution of $MnSO_4$, NaOH and potassium iodide, azide reagent, into the water sample. The white precipitate of $Mn(OH)_2$ which is formed, is oxidized by oxygen in water sample to give a brown precipitate of basic manganic oxide $MnO(OH)_2$. This $MnO(OH)_2$, in acidic medium dissolves and liberates free iodine from the added KI in a equivalent amount of dissolved oxygen in water sample. This liberated I_2 is then titrated against $Na_2S_20_3$ solution using starch as indicator. The reactions in- volved are:

$$MnS0_4 + 2NaOH \rightarrow Mn(OH)_2 + Na_2SO_4$$

$$4\,Mn(OH)_2 + O_2 \rightarrow 2MnO(OH)_2$$

Basic Manganic oxide

$$2MnO(OH)_2 + H_2SO_4 \rightarrow MnSO_4 + 2H_2O + O$$

$$2KI + H_2SO_4 + O \rightarrow K_2S0_4 + H_20 + I_2$$

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

The nitrites present in water, interfere with the titration as these can also liberate I_2 from KI

$$2HNO_2 + H_2SO_4 + 2KI \rightarrow 2NO + K_2SO_4 + 2H_2O + I_2$$

Thus to destroy nitrite, sodium azide is used.

$$2\ NaN_3 + H_2\ SO\ 4 \rightarrow \underset{\text{(Hydrazoic acid)}}{2HN_3} + Na_2SO_4$$

$$HNO_2 + HN_3 \rightarrow NO_2 + N_2 + H_2O$$

D] Procedure

1. A known amount of sample water (say 250 ml) is taken iljt a stoppered bottle avoiding contact with air.
2. Add 0.2 ml of $MnSO_4$ solution to it by means of a pipette, dipping the end well below the surface of water. Also add 2 ml of alkaline iodide-azide solution to it.
3. Stopper the bottle and shake thoroughly. Allow the brown precipitates of $MnO\ (OH)_2$ formed, to settle down.
4. When some portion of the liquid below the stopper is clear, add 2 ml of conc. H_2S04 with the help of pipette. Stopper and mix till the precipitate is completely dissolved. The characteristic brown colour of iodine is produced.
5. Transfer 100ml of the above solution in a 250 ml flask with a pipette. Titrate the liberated I_2 with standardized sodium thiosulphate solution until the sample solution becomes pale yellow.
6. Add 2 ml of starch solution, the solution will turn blue.

7. Continue titration till the blue colour disappears.

8. Repeat to get another reading.

E] Results and Discussion

Sl. No.	Vol of the solution taken in the titration flusk (ml)	Burette Reading		Volume of the titrant used (ml)
		Initial	Final	
1.				
2.				
3.				

Calculation

Hypo O_2 in water

$N_1V_1 = N_2V_2$

$1/100 \text{ x } V_1 = N_2 \text{ x } 100$

$N_2 = V_1 / 10000$

Strength of dissolved oxygen = N_2 x Eq. wt

$= V_1 / 10000 \times 8g / L$

$= V_1 / 10000 \times 8 \times 1000$ mg / L

$= 0.8\ V_1$ ppm

Result: The amount of dissolved oxygen in water = ………. Ppm

F] Conclusion

1. The water should be taken in the stoppered bottle vary carefully without trapping air bubbles which could raise oxygen level by aerating the sample.
2. $MnSO_4$ and alkaline iodine – azide solutions are added to the water sample just below the surface of water.
3. Whole of the precipitate of $MnO(OH)_2$ should be dissolved in H_2SO_4.