

Siliguri Institute of Technology
Department of Engineering Sciences & Humanities
Chemistry Lab Manual
B. Tech. 1st sem/ 2nd sem

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Experiment 1

ADSORPTION

Determination of adsorption isotherm of acetic acid by activated charcoal.

Principle:

At a given temperature, the mass of a solute (or gas) adsorbed by a solid adsorbent at various concentration (pressure in case of gases) is given by the following empirical relation:

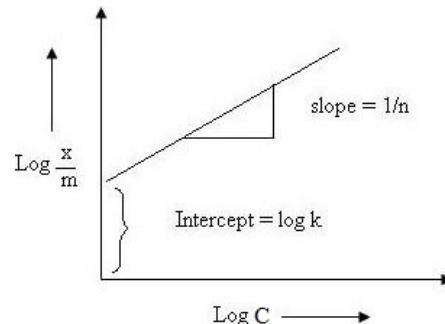
$$\frac{x}{m} = k \times C^{1/n} \quad \dots\dots\dots (1)$$

where x is the mass of the solute (or gas) adsorbed by m gram of the solid at various concentration, k and n constant at a given temperature for the given solid adsorbent and solute.

Equation (1) can also be written in the following manner:

$$\log \frac{x}{m} = \log k + \frac{1}{n} \log C$$

A plot of $\log \frac{x}{m}$ versus $\log C$ gives a straight line with slope equal to $\frac{1}{n}$



Apparatus:

Stoppard bottles (125 ml), burette (50 ml), pipette (10 ml), funnel, funnel holder, beaker (250 ml), conical flask (100 ml).

Reagent:

Acetic acid (0.6 N), NaOH solution (0.2 N), powdered activated charcoal, Phenolphthalein indicator

Procedure:

1. Cleaned and dry stoppard bottles are taken and labeled them from 1 to 4.
2. Burettes are set up and filled one with acetic acid and the other with distilled water.
3. Different volumes of acetic acid and water are mixed in the labeled bottles as given below:

Bottle No.	Vol. of 0.6 N acetic acid	Vol. of water	Total volume in each bottle
1	60	0	60
2	50	10	60
3	40	20	60
4	30	30	60

6. The solutions are mixed well and 3.0 gm of charcoal is added in each bottle.
7. The sample bottles are shaken for an half an hour for homogeneous adsorption of acetic acid by charcoal.
8. Now the contents of bottles are filtered into beaker through filter paper. 10 ml of the filtrate is pipetted out into a 100 ml conical flask and titrated against 0.2 N NaOH solution using phenolphthalein as indicator. Three readings are taken in each case.

Results and Calculation:

Strength of acetic acid solution:

Room Temperature =°C

Weight of activated charcoal in each bottle = 3 gm

Vol. of filtrate for titration in each case = 10 ml

Table 1: Titration of acetic acid in the filtrate.

Bottle No.	Volume of filtrate for titration (ml)	Burette reading: Volume of 0.2 N NaOH solution (ml)	Mean Volume (ml)
1	10		V ₁ =
2	10		V ₂ =
3	10		V ₃ =
4	10		V ₄ =

(i) Bottle No.1

Initial conc. of acetic acid = 0.6 M

10 ml of M₁ acetic acid in filtrate = V₁ ml of 0.2 M NaOH

$$M_1 = \frac{V_1 \times 0.2}{10} = 0.02 V_1 = \text{Concentration after adsorption}$$

Thus change in concentration after adsorption = (0.6 – 0.02 V₁) MAlso if X₁ = Change in con. in grams per 60 ml of the solution in Bottle 1 then

$$x_1 = \frac{\text{Change in conc.in moles} \times \text{Volume taken}}{1000} \times \text{Mol. wt.}$$

$$= \frac{(0.6 - 0.02 V_1) \times 60}{1000} \times 60$$

(ii) Bottle 2:

$$\text{Initial conc. of acetic acid} = \frac{0.6 \times 50}{60} = 0.5 \text{ M}$$

As 10 ml of M_2 acetic acid = V_2 ml of 0.2 N NaOH

$$M_2 = \frac{V_2 \times 0.2}{10} = 0.02 V_2 = \text{Concentration after adsorption}$$

Thus change in concentration after adsorption = $(0.5 - 0.02 V_2)$ M

$$x_2 = \frac{\text{Change in conc.in moles} \times \text{Volume taken}}{1000} \times \text{Mol. wt.}$$

$$= \frac{(0.5 - 0.02 V_2) \times 60}{1000} \times 60$$

Similarly x_3 and x_4 are also calculated.

Table 2: Tabulation of $\log \frac{x}{m}$ and $\log C$.

Bottle No	Initial concentration of acetic acid solution (C)	Amount of acetic acid adsorbed (x_n)	Weight of adsorbent (charcoal) (m)	$\frac{x}{m}$	$ \log \frac{x}{m} $	$ \log C $
1	0.6	x_1				
2	0.5	x_2				
3	0.4	x_3				
4	0.3	x_4				

Now plot the graph by taking $\log C$ along X-axis and $\log \frac{x}{m}$ along Y-axis. A straight line obtained in the plot shows the validity of the Freundlich isotherm.

Experiment 2

SURFACE TENSION

Determination of surface tension of the given liquid at room temperature by stalagmometer.

Principle:

The surface tension of the given liquid is determined relative to water at the room-temperature by using stalagmometer. The no. of drops for the same volume of water and the given liquid are counted and let these be as n_1 and n_2 respectively. Now if d_1 and d_2 are densities of water and the given liquid at the room temperature as determined separately by using specific gravity bottle or pycnometer, then the surface-tension γ_2 of the

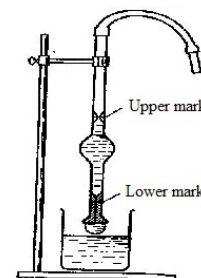
$$\frac{\gamma_1}{\gamma_2} = \frac{n_2}{n_1} \times \frac{d_1}{d_2}$$

Apparatus:

Stalagmometer, Rubber tube, Screwpinch cock, stand, holder, specific gravity bottle, beaker.

Reagent:

Distilled water, unknown liquid



Procedure:

1. The stalagmometer and specific gravity bottle are cleaned thoroughly first with chromic acid solution and washed finally with distilled water and then dried.
2. The density of unknown liquid or solution is measured by using specific gravity bottle.
2. Water is sucked up by immersing lower end in a beaker containing water above the upper mark and tightens the screw of the screw-pinch.
3. The liquid drops are started falling at an interval of about 2-3 seconds in successive drops from stalagmometer by loosing the screw of the screw-pinch carefully. Counting of drops is started when the water meniscus just reaches the upper mark and stopped when the meniscus just passed the lower mark. The experiment is repeated thrice and the mean value was taken.
4. The same experiment is done with unknown liquid or solution.

Results and Calculations:

Room temp. =°C

Density of water at room temperature = d_1 **Table 1:** Density calculation of unknown liquid or solution.

Weight of empty specific gravity bottle (w_1)	Weight of empty specific gravity bottle + water (w_2)	Weight of empty specific gravity bottle + unknown liquid (w_3)	Weight of water = ($w_2 - w_1$) = m_1 gm	Weight of unknown liquid = ($w_3 - w_1$) = m_2 gm

$$\text{Density of unknown liquid} = d_2 = \frac{m_2}{m_1}$$

Table 2: Surface tension calculation

Liquid	No. of drops			Mean Value
	Obs. 1	Obs. 2	Obs. 3	
Water				(n_1)
Unknown liquid or solution				(n_2)

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

$$\gamma_2 = \gamma_1 \times \frac{n_1}{n_2} \times \frac{d_2}{d_1}; \text{ where, } \gamma_1 = \text{surface tension of water at room temperature.}$$

By substituting the values of n_1 , n_2 , d_1 , d_2 , γ_1 and γ_2 , the surface tension of unknown liquid at room temperature is obtained.

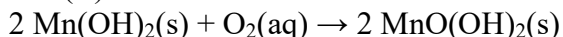
Final Result:

The relative surface tension of unknown liquid with respect to water at room temperature is

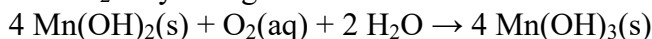
Experiment 3**DISSOLVED OXYGEN ANALYSIS****Estimation of dissolved oxygen in a given sample of water.****Principle:**

The Winkler test is used to determine the level of dissolved oxygen in water samples and to estimate the biological activity in the water sample. An excess of Manganese (II) salt, iodide (I⁻) and hydroxide (OH⁻) ions are added to a water sample causing a white precipitate of Mn(OH)₂ to form. This precipitate is then oxidized by the dissolved oxygen in the water sample into a brown Manganese precipitate. In the next step, a strong acid (either hydrochloric acid or sulphuric acid) is added to acidify the solution. The brown precipitates then convert the iodide ion (I⁻) to Iodine. The amount of dissolved oxygen is directly proportional to the titration of Iodine with a thiosulphate solution.

First Manganese (II) sulfate is added to an environmental water sample. Next, Potassium iodide is added to create a pinkish-brown precipitate. In the alkaline solution, dissolved oxygen will oxidize manganese (II) ions to the tetravalent state.

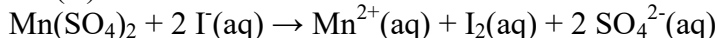


MnO(OH)₂ appears as a brown precipitate. There is some confusion about whether the oxidised manganese is tetravalent or trivalent. Some sources claim that Mn(OH)₃ is the brown precipitate, but hydrated MnO₂ may also give the brown colour.

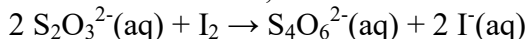


The second part of the Winkler test reduces acidifies the solution. The precipitate will dissolve back into solution. The acid facilitates the conversion by the brown, Manganese-containing precipitate of the Iodide ion into elemental Iodine.

The Mn(SO₄)₂ formed by the acid converts the iodide ions into iodine, itself being reduced back to manganese(II) ions in an acidic medium.



Thiosulfate solution is used, with a starch indicator, to titrate the iodine.

**Apparatus:**

Chemical balance, Weighing bottle, volumetric flask, Burette, Erlenmeyer flask, Pipettes.

Reagents:

Potassium dichromate A.R., Na₂S₂O₃ solution, MnSO₄ solution (5%), KI solution (20%), freshly prepared starch solution (1%) as indicator.

Procedure:

1. Preparation of 250 ml standard N/50 potassium dichromate (K₂Cr₂O₇) solution:

About 0.245 g potassium dichromate is weigh out accurately in 250 ml volumetric flask and dissolved in distilled water.

2. Standardization of sodium thiosulphate (Na₂S₂O₃) solution with the standard potassium dichromate solution:

25 ml potassium dichromate solution is pipetted out in a 250 ml conical flask. 10 ml 20% KI solution and 5 ml conc. HCl solution is added to above solution stepwise and shaken the solution well. The

mouth of the conical flask is covered with a watch glass and kept it in dark for 10 minutes. The solution is then titrated with sodium thiosulphate solution from burette rapidly until the colour of the solution become light yellow. 2 ml starch solution is added to it and the colour of the solution is changed to deep blue. The titration is continued slowly with continuous shaking until the blue colour just disappeared and colour is changed to light green.

3. Estimation of dissolved oxygen:

100 ml water sample is pipetted out in 500 ml conical flask. 1ml MnSO₄ solution, 1 ml alkaline KI solution and 0.5 ml conc. H₂SO₄ is added to it. The liberated I₂ is titrated against standard sodium thiosulphate (Na₂S₂O₃) solution with continuous shaking. When the colour of the solution is changed to light yellow, 2 ml starch solution is added and the solution is turned deep blue. Again the solution is titrated against standard sodium thiosulphate (Na₂S₂O₃) solution with continuous shaking until the blue colour just disappeared.

Results and Calculation:

Table 1: Preparation of 250 ml standard N/50 potassium dichromate (K₂Cr₂O₇) solution.

Initial weight (W ₁) g	Final weight (W ₂) g	Weight taken (W ₁ -W ₂) g

Strength of potassium dichromate solution (S₁) = (W₁-W₂) / 0.245 * 0.02 (N)

Table 2: Standardization of sodium thiosulphate (Na₂S₂O₃) solution.

Sl. No.	Volume of K ₂ Cr ₂ O ₇ solution pipetted out (V ₁ ml)	Burette reading: Volume of Na ₂ S ₂ O ₃ solution in ml	Mean Volume (V ₂ ml)	Strength of NaOH solution (S ₂)
1.	25			
2.	25			
3.	25			

Strength of Na₂S₂O₃ solution (S₂) = V₁*S₁/ V₂ (N)

Table 3: Estimation of dissolved oxygen in a given water sample.

Sl. No.	Volume water sample pipetted out	Burette reading: Volume of Na ₂ S ₂ O ₃ solution in ml	Mean Volume (V ₃ ml)
1.	100		
2.	100		
3.	100		

Volume of water sample taken = 100 ml

Volume of standard sodium thiosulphate (Na₂S₂O₃) solution required = V₃ ml

Strength of dissolved oxygen (S₃) = V₃ * S₂ / 100 (N)

Amount of oxygen in water = S₃ * 8 g / lit

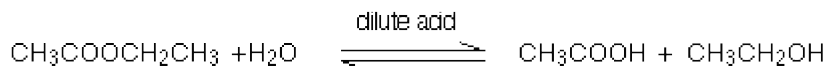
= S₃ * 8 * 1000 mg /lit (ppm)

Experiment 4
CHEMICAL KINETICS

Determination of rate constant of acid catalyzed hydrolysis of ethyl acetate.

Principle:

Because the reaction is reversible, an equilibrium mixture is produced containing all four of the substances in the equation. In order to get as much hydrolysis as possible, a large excess of water can be used. The dilute acid provides both the acid catalyst and the water.



The rate constant is calculated from the integrated equation of first order reaction:

$t = \{2.303 \log (a / a-x) / k\}$, where a = initial concentration, $(a - x)$ = concentration at time t .

Apparatus:

Chemical balance, Weighing bottle, volumetric flask, Burette, Erlenmeyer flask (100 ml and 250 ml), Pipettes, water bath.

Reagents:

Oxalic acid A.R., (N/5) NaOH solution, (N/2) HCl solution, Phenolphthalein indicator.

Procedure:

1. Preparation of 250 ml standard N/10 oxalic acid ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) solution:

About 1.575 g oxalic acid is weighed out accurately in 250 ml volumetric flask and dissolved in distilled water.

2. Standardization of supplied NaOH solution with the standard oxalic acid solution:

25 ml NaOH solution is pipetted out in a 250 ml conical flask. Two drops of Phenolphthalein indicator is added to it and the solution become pink. The solution is titrated against standard oxalic acid solution and at the end point the solution become colourless.

3. Determination of rate constant:

i) 100 ml 0.5 (N) HCl solution is taken in a clean dry 250 ml conical flask, corked and set it in water bath at room temp.

ii) 5 ml pure ethyl acetate is pipetted out and added to the above solution.

iii) 15 g pounced ice or 15 ml chilled water is taken each of the five 100 ml conical flask.

iv) The reaction mixture is shaken and 10 ml of it is pipetted out to a 100 ml conical flask, 15 ml water is added, and finally put it on the water bath at temperature 50-60°C for about an hour. It is titrated against standard NaOH solution to obtain the value of V_α .

v) 10 ml of the reaction mixture is pipetted out and transferred it at once to first conical flask containing chilled water. It is titrated against standard NaOH solution to obtain the value of V_o . The titration is repeated at every ten minutes for four times more to obtain the value of V_t .

Results and Calculation:

Table 1: Preparation of standard 0.1 (N) oxalic acid solution.

Initial weight (W_1) g	Final weight (W_2) g	Weight taken ($W_1 - W_2$) g

Strength of oxalic acid solution (S_1) = $(W_1 - W_2) / 1.575 * 0.1$ (N)

Table 2: Standardization of NaOH solution.

Sl. No.	Volume of NaOH solution pipetted out (V_2 ml)	Burette reading: Volume of oxalic acid solution in ml	Mean Volume (V_1 ml)	Strength of NaOH solution (S_2)
1.	25			
2.	25			
3.	25			

Strength of NaOH solution (S_2) = $V_1 * S_1 / V_2$ (N)

Table 3: Titration of ethyl acetate solution by standard NaOH solution.

Temperature: °C

V_o = ml

V_α = ml

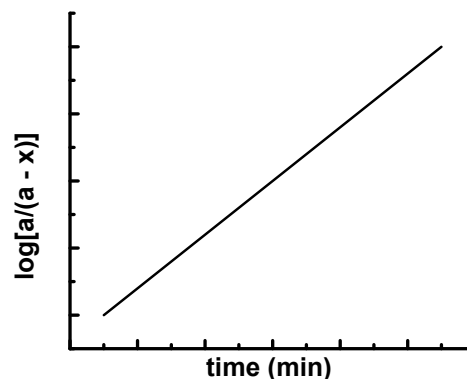
$a = (V_\alpha - V_o) =$ ml

Time in minutes (t)	Vol. of NaOH (V_t)	$(V_\alpha - V_o) / (V_\alpha - V_t)$	$\log_{10} [(V_\alpha - V_o) / (V_\alpha - V_t)]$
10	$V_{10} =$		
20	$V_{20} =$		
30	$V_{30} =$		
40	$V_{40} =$		

Plot of ' $\log_{10} [(V_\alpha - V_o) / (V_\alpha - V_t)]$ ' against ' t ' gives a straight line passing through the origin.

Slope of line = $k / 2.303$;

$k = \text{slope} * 2.303 =$ min^{-1}



Experiment 5**HETEROGENEOUS EQUILIBRIUM****Determination of partition coefficient of acetic acid between n-butanol and water.****Principle:**

When two immiscible solvents are in contact with each other, a dissolved substance (solute) will distribute itself between two according to a definite equilibrium. The ratio of the concentrations of the substance (solute) in the aqueous phase, C_{aq} , and organic phase, C_{org} at equilibrium at a given temperature gives the partition coefficient K .

$$K = C_{aq} / C_{org}$$

Apparatus:

Chemical balance, Weighing bottle, volumetric flask, Bottle, Shaker, Burette, Erlenmeyer flask, Pipettes.

Reagents:

Acetic acid, n-butanol, Oxalic acid A.R., 0.5 (N) NaOH solution, Phenolphthalein indicator.

Procedure:

1. Preparation of 250 ml standard N/10 oxalic acid ($H_2C_2O_4 \cdot 2H_2O$) solution:

About 1.575 g oxalic acid is weighed out accurately in 250 ml volumetric flask and dissolved in distilled water.

2. Standardization of supplied NaOH solution with the standard oxalic acid solution:

10 ml NaOH solution is pipetted out in a 100 ml conical flask. Two drops of Phenolphthalein indicator is added to it and the solution become pink. The solution is titrated against standard oxalic acid solution and at the end point the solution become colourless.

3. Preparation of water & n-Butanol immiscible solution:

50 ml of supplied acetic acid solution is pipetted out in 250 ml glass stopper bottles. 50 ml of n-Butanol is added to each of the bottle and shaken vigorously for one hour and allowed to stand for half an hour so as to allow the layers to separate clearly.

4. Determination of strength of aqueous and organic layers:

a) 10 ml of aqueous layer is pipetted out in a 100 ml conical flask and titrated with 0.5 (N) standard NaOH solution using Phenolphthalein indicator. Concentration of acetic acid in aqueous layer (C_{aq}) can be calculated from the end point.

b) 10 ml of organic layer is pipetted out in a 100 ml conical flask and titrated with 0.5 (N) standard NaOH solution using Phenolphthalein indicator. Concentration of acetic acid in aqueous layer (C_{org}) can be calculated from the end point.

Results and Calculation:**Table 1:** Preparation of standard 0.1 (N) oxalic acid solution.

Initial weight (W_1) g	Final weight (W_2) g	Weight taken ($W_1 - W_2$) g

Strength of oxalic acid solution (S_1) = $(W_1 - W_2) / 1.575 * 0.1$ (N)

Table 2: Standardization of NaOH solution.

Sl. No.	Volume of NaOH solution pipetted out (V_2 ml)	Burette reading: Volume of oxalic acid solution in ml	Mean Volume (V_1 ml)	Strength of NaOH solution (S_2)
1.	10			
2.	10			
3.	10			

Strength of NaOH solution (S_2) = $V_1 * S_1 / V_2$ (N)

Table 3: (a) Determination of concentration of acetic acid in aqueous layer (C_{aq}).

Sl. No.	Volume of aqueous layer pipetted out (X ml)	Burette reading: Volume of NaOH solution in ml	Mean Volume (Y ml)	Concentration of acetic acid in aqueous layer (C_{aq})
1.	10			
2.	10			
3.	10			

Concentration of acetic acid in aqueous layer (C_{aq}) = $Y * S_2 / X$ (N)

Table 3: (b) Determination of concentration of acetic acid in organic layer (C_{org}).

Sl. No.	Volume of organic layer pipetted out (X ml)	Burette reading: Volume of NaOH solution in ml	Mean Volume (Y ml)	Concentration of acetic acid in organic layer (C_{org})
1.	10			
2.	10			
3.	10			

Concentration of acetic acid in organic layer (C_{org}) = $Y * S_2 / X$ (N)

Partition coefficient, $K = C_{aq} / C_{org}$

Experiment 6**VISCOSITY OF LIQUID OR SOLUTION**

Determination of relative viscosity of unknown liquid or solution with respect to water at room temperature by Ostwald viscometer.

Principle:

Viscosity is a measure of the resistance of a fluid which is being deformed by either shear stress or tensile stress. In everyday terms (and for fluids only), viscosity is "thickness" or "internal friction". Thus, water is "thin", having a lower viscosity, while honey is "thick", having a higher viscosity. Put simply, the less viscous the fluid is, the greater its ease of movement (fluidity). Viscosity describes a fluid's internal resistance to flow and may be thought of as a measure of fluid friction.

The ratio of the shearing stress to the velocity gradient is a measure of the viscosity of the fluid and is called the coefficient of viscosity η , or $\eta = F \cdot x / A \cdot v$. The cgs unit for measuring the coefficient of viscosity is the poise.

The experimental determination absolute viscosity coefficient of liquid is a difficult task, but the measurement of viscosity coefficient of a liquid relative to that of some reference liquid (e.g. water) is simple and adequate for our purpose.

Let equal volume of unknown liquid or solution and water having co-efficient of viscosity (η_s) and (η_w) flow down the same Ostwald's Viscometer in time t_s and t_w , then

Co-efficient of viscosity (η_s) of unknown liquid or solution = $[(\rho_s \cdot t_s) / (\rho_w \cdot t_w)] \cdot \eta_w$

where, ρ_s and ρ_w are the density of unknown liquid or solution and water respectively.

Apparatus:

Chemical balance, Ostwald's Viscometer, Stop Watch, Specific Gravity Bottle.

Reagents:

Distilled water, unknown liquid or solution.

Procedure:

1. Determination of specific gravity of solution:

Determinations of specific gravity of unknown liquid or solution is performed by specific gravity bottle method from the known density of water.

2. Measurement of time of flow:

Time of flow is measured by the Ostwald's Viscometer.

Results and Calculation:

Table 1: Determination of specific gravity of unknown liquid or solution.

Temperature of water: °C

Density of water (ρ_w) (given):

Weight of the empty sp. gravity bottle (W_1 g):

Weight of the empty sp. gravity bottle + water (W_2 g):

Weight of the empty sp. gravity bottle + sugar solution (W_3 g)

Sp. gravity of unknown liquid or solution (ρ_s) = $(W_3 - W_1) / (W_2 - W_1)$

Table 2: Determination of co-efficient of viscosity (η_s) of unknown liquid or solution.

Temperature of water: °C

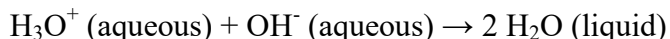
Co-efficient of viscosity (η_w) of water (given):

Sl. No.	Water		Liquid under experiment	
	Time of flow (s)	Mean time (t_w)	Time of flow (s)	Mean time (t_s)
1				
2				
3				

Co-efficient of viscosity (η_s) of unknown liquid or solution = $[(\rho_s * t_s) / (\rho_w * t_w)] * \eta_w$

Experiment 7**CONDUCTOMETRIC TITRATION****Determination of strength of a given solution of HCl by titration against a standard solution of NaOH.****Principle:**

When an acid is added to a base, there is a reaction between the hydronium ions (H_3O^+) and the hydroxyl ions (OH^-). This can be represented by the following ionic reaction:



When the amount of base increases, the conductance will be lowered as the result of the disappearance of hydronium ions (H_3O^+). (The hydronium ions (H_3O^+) have a high molar conductivity while the cations from the base have a much lower molar conductivity). When the hydronium ions (H_3O^+) have all been neutralized, the increase in excess base concentration will result in a sudden increase in the conductivity of the solution. This phenomenon is due to the high molar conductivity of hydroxyl ions (OH^-).

Apparatus:

Chemical balance, Weighing bottle, volumetric flask, Burette, Erlenmeyer flask, Pipettes, Conductivity Bridge, conductivity cell.

Reagents:

Oxalic acid, (N/5) NaOH solution, (N/20) HCl solution, Phenolphthalein indicator.

Procedure:

1. Preparation of 250 ml standard N/10 oxalic acid ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) solution:

About 1.575 g oxalic acid is weighed out accurately in 250 ml volumetric flask and dissolved in distilled water.

2. Standardization of supplied NaOH solution with the standard oxalic acid solution:

25 ml NaOH solution is pipetted out in a 250 ml conical flask. Two drops of Phenolphthalein indicator is added to it and the solution become pink. The solution is titrated against standard oxalic acid solution and at the end point the solution become colourless.

3. Titration of NaOH solution by standard HCl solution conductometrically:

25 ml given HCl solution is pipetted out in a 250 ml beaker and 125 ml deionised water is added to it. Conductivity cell is placed in a beaker so that the electrodes are completely immersed in the acid solution. Conductance of the solution is measured and noted down. Initially 10 drops and then 5 drops (near the end point) of NaOH solution are added and conductance is measured after each addition.

Plot the '**conductance**' against correspond '**titre values**', draw the straight lines and obtain the point of equivalence at intersection.

Results and Calculation:**Table 1:** Preparation of standard 0.1 (N) oxalic acid solution.

Initial weight (W_1) g	Final weight (W_2) g	Weight taken ($W_1 - W_2$) g

Strength of oxalic acid solution (S_1) = $(W_1 - W_2) / 1.575 * 0.1$ (N)

Table 2: Standardization of NaOH solution.

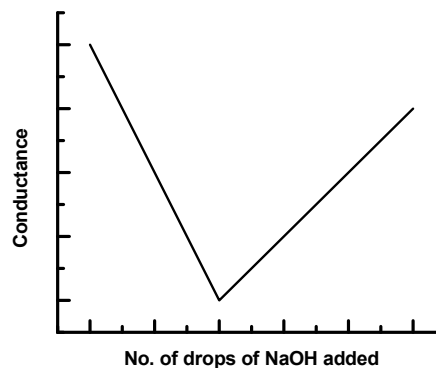
Sl. No.	Volume of NaOH solution pipetted out (V_2 ml)	Burette reading: Volume of oxalic acid solution in ml	Mean Volume (V_1 ml)	Strength of NaOH solution (S_2)
1.	25			
2.	25			
3.	25			

Strength of NaOH solution (S_2) = $V_1 * S_1 / V_2$ (N)

Table 3: Titration of NaOH solution by standard HCl solution conductometrically.

Volume of given acid solution (V_3) = 25 ml

Sl. No.	No. of drops of NaOH solution added	Conductance (Ohm^{-1})



No. of drops of NaOH solution at equivalence point (x) (from plot) = _____

_____ drops = _____ ml

x drops = _____ ml (V_4)

Strength of HCl solution (S_3) = $V_4 * S_2 / V_3$ (N)

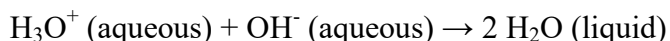
Experiment 8

pH-METRIC TITRATION

Determination of strength of a given solution of HCl by titration against a standard solution of NaOH.

Principle:

When an acid is added to a base, there is a reaction between the hydronium ions (H_3O^+) and the hydroxyl ions (OH^-). This can be represented by the following ionic reaction:



If the pH of an acid solution is plotted against the amount of base added during a titration, the shape of the graph is called a titration curve. All acid titration curves follow the same basic shapes.

At the beginning, the solution has a low pH and climbs as the strong base is added. As the solution nears the point where all of the H^+ is neutralized, the pH rises sharply and then levels out again as the solution becomes more basic as more OH^- ions are added.

$$\text{pH} = -\log [\text{H}^+]$$

Apparatus:

Chemical balance, Weighing bottle, volumetric flask, Burette, Erlenmeyer flask, Pipettes, pH-meter.

Reagents:

Oxalic acid A.R., (N/5) NaOH solution, (N/20) HCl solution, Phenolphthalein indicator.

Procedure:

1. Preparation of 250 ml standard N/10 oxalic acid ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) solution:

About 1.575 g oxalic acid is weighed out accurately in 250 ml volumetric flask and dissolved in distilled water.

2. Standardization of supplied NaOH solution with the standard oxalic acid solution:

25 ml NaOH solution is pipetted out in a 250 ml conical flask. Two drops of Phenolphthalein indicator is added to it and the solution become pink. The solution is titrated against standard oxalic acid solution and at the end point the solution become colourless.

3. Titration of NaOH solution by standard HCl solution with pH-meter:

25 ml given HCl solution is pipetted out in a 250 ml beaker and 125 ml water is added to it. Electrode cell is placed in a beaker so it is completely immersed in the acid solution. pH of the solution is measured and noted down. Initially 10 drops and then 5 drops (near the end point) of NaOH solution are added and pH is measured after each addition.

Plot the 'pH' against correspond 'titre values', connect the points and obtain the point of equivalence at pH 7.

Results and Calculation:

Table 1: Preparation of standard 0.1 (N) oxalic acid solution.

Initial weight (W_1) g	Final weight (W_2) g	Weight taken ($W_1 - W_2$) g

Strength of oxalic acid solution (S_1) = $(W_1 - W_2) / 1.575 * 0.1$ (N)

Table 2: Standardization of NaOH solution.

Sl. No.	Volume of NaOH solution pipetted out (V_2 ml)	Burette reading: Volume of oxalic acid solution in ml	Mean Volume (V_1 ml)	Strength of NaOH solution (S_2)
1.	25			
2.	25			
3.	25			

$$\text{Strength of NaOH solution } (S_2) = V_1 * S_1 / V_2 \text{ (N)}$$

Table 3: Titration of NaOH solution by standard HCl solution conductometrically.

Volume of given acid solution (V_3) = 25 ml

Sl. No.	No. of drops of NaOH solution added	pH

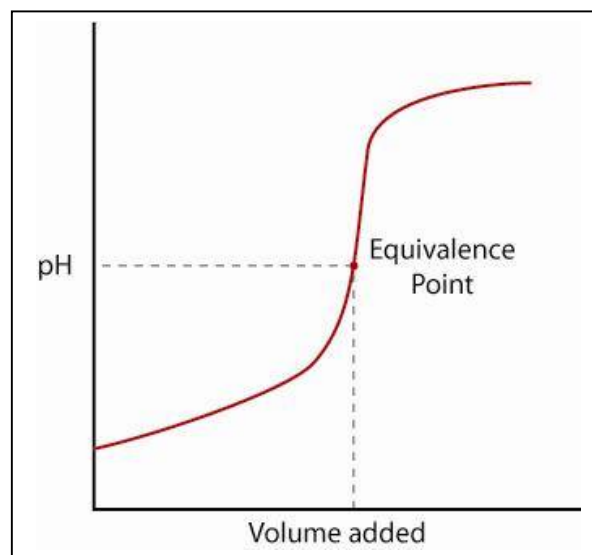
No. of drops of NaOH solution at equivalence

point (x) (from plot) = _____

_____ drops = _____ ml

x drops = _____ ml (V_4)

Strength of HCl solution (S_3) = $V_4 * S_2 / V_3$ (N)



Experiment 9**ACID VALUE****Determination of Acid Value of an Oil.****Principle:**

The acid value of oil is defined as a number of milligrams of KOH required to neutralize the free acid present in 1 g of the oil sample.

For the determination of acid number, first a known weight of the oil sample is dissolved in a suitable solvent. Then it is titrated with a standard alcoholic solution of KOH to a definite end-point.

Apparatus:

Conical flask, burette, pipette (10 ml), beaker, measuring cylinder, dropper, funnel, water bath etc.

Reagent:

Standard (0.01 N) alcoholic KOH solution, titration solvent, Phenolphthalein or Naphtholbenzoin indicator.

Procedure:

About 5 g of the oil under test is weighed into a 250 ml conical flask and 50 ml neutral alcohol is added to it. The flask is heated over a water bath for about 30 minutes (in case of fatty acid oil, solution is shaken vigorously instead of heating). The flask is cooled to room temperature and few drops Phenolphthalein indicator is added. The solution is titrated with standard (0.01 N) KOH solution until a faint permanent pink colour appeared at the end point.

$$\text{Acid value} = \frac{\text{Volume (in ml) KOH solution used}}{\text{Weight of the oil sample taken (in g)}} \times 0.56,$$

where 0.56 represents the amount of KOH in mg per ml of (0.01 N) KOH solution.

Results and Calculations:**Table 1:** Weight of the sample taken.

Initial weight (x_1) gm	Final weight (x_2) gm	Weight taken ($x_1 - x_2$) gm

Table 2: Volume of (0.01 N) alcoholic KOH solution.

Sl. No.	Sample	Volume of (0.01 N) alcoholic KOH solution in ml	Mean Volume (V ml)
1.	Test 1		
2.	Test 2		
3.	Test 3		

$$\text{Acid value} = \frac{\text{Volume KOH solution used (in ml)}}{\text{Weight of the oil sample taken (in g)}} \times 0.56$$

$$= \frac{V}{(x_2 - x_1)} \times 0.56$$

Experiment 10**ARGENTOMETRIC TITRATION****Estimation of Cl⁻ ion in a given sample of water by Argentometric method.****Principle:**

Precipitation titrations are based upon reactions that yield ionic compounds of limited solubility. The most important precipitating reagent is silver nitrate. Titrimetric methods based upon silver nitrate are sometimes termed argentometric methods. Potassium chromate can serve as an end point indicator for the argentometric determination of chloride, bromide and cyanide ions by reacting with silver ions to form a brick-red silver chromate precipitate in the equivalence point region.

The Mohr method uses chromate ions as an indicator in the titration of chloride ions with a silver nitrate standard solution. After all the chloride has been precipitated as white silver chloride, the first excess of titrant results in the formation of a silver chromate precipitate, which signals the end point.

The reactions are: $\text{Ag}^+ + \text{Cl}^- \rightarrow \text{AgCl}(s)$
 $2\text{Ag}^+ + \text{CrO}_4^{2-} \rightarrow \text{Ag}_2\text{CrO}_4(s)$

By knowing the stoichiometry and moles consumed at the end point, the amount of chloride in an unknown sample can be determined. This report describes experiments aimed at determining the concentration of chloride in a solid sample.

Apparatus:

Chemical balance, Weighing bottle, volumetric flask, Burette, Erlenmeyer flask, Pipettes.

Reagents:

Silver nitrate, dry NaCl, 4% K₂CrO₄ solution as indicator.

Procedure:

1. Preparation of 250 ml standard (N/10) silver nitrate (AgNO₃) solution:

About 4.2467 g silver nitrate is weighed out accurately in 250 ml volumetric flask and dissolved in distilled water. The solution is kept in brown-glass bottle to protect it from sunlight as much as possible.

2. Estimation of Cl⁻ in a given water sample:

50 ml water sample is pipetted out in 250 ml conical flask. 1 ml of 4% K₂CrO₄ solution is added to it and colour of the solution became light yellow. The solution is titrated with standard (N/10) silver nitrate (AgNO₃) solution with continuous shaking. The conical flask was kept against a white background to facilitate the easy detection of end point. Initially the red colour produced by addition of each drop of AgNO₃ solution disappeared rapidly on shaking.

The titration is continued until there a permanent colour change from yellow to reddish precipitate of Ag₂CrO₄ (s). The titration is repeated for concordant data.

3. Blank titration:

50 ml distilled water is pipetted out in 250 ml conical flask. 1 ml of 4% K₂CrO₄ solution is added to it and colour of the solution became light yellow. The water is titrated with standard (N/10) silver

nitrate (AgNO_3) solution with continuous shaking. The conical flask was kept against a white a white back ground to facilitate the easy detection of end point. The titration is continued until the colour is matched to that of the chloride sample water that is titrated in the previous stage. The titration is repeated for concordant data.

Results and Calculation:

Table 1: Preparation of 250 ml standard (N/10) silver nitrate (AgNO_3) solution.

Initial weight (W_1) g	Final weight (W_2) g	Weight taken ($W_1 - W_2$) g

Strength of silver nitrate solution (S_1) = $(W_1 - W_2) / 4.2467 * 0.1$ (N)

Table 2: Estimation of Cl^{-1} ion in a given water sample.

Sl. No.	Volume water sample pipetted out	Burette reading: Volume of AgNO_3 solution in ml	Mean Volume (V_1 ml)
1.	50		
2.	50		
3.	50		

Table 3: Blank titration.

Sl. No.	Volume water sample pipetted out	Burette reading: Volume of AgNO_3 solution in ml	Mean Volume (V_2 ml)
1.	50		
2.	50		
3.	50		

Volume of standard silver nitrate (AgNO_3) solution required for Cl^{-1} ion = $(V_1 - V_2)$ ml

Strength of Cl^{-1} ion in water sample = $[(V_1 - V_2) * S_1] / 50$ (N)

The Cl^{-1} ion concentration = $\{(V_1 - V_2) * S_1\} / 50\} * 35.5$ g/lit

Reference:

1. Arthur I. Vogel: Quantitative Inorganic Analysis including Elementary Instrumental Analysis, ELBS, Longmann Group, 5th Edition, 1989.
2. R. Mukhopadhyay and P. Chatterjee: Advanced Practical Chemistry, Books and Allied, Kolkata, 2004.
3. K. S. Mukherjee: Text Book on Practical Chemistry, NCBA, Kolkata, 2008.

Question for Viva-Voce

1. What is titration? Define standard solution.
2. Define primary standard solution and secondary standard solution with examples.
3. What do you mean by alkalinity of water? How alkalinity is measured?
4. Why phenolphthalein cannot be used for titrating a weak base like bicarbonate ions?
5. Give indicator ranges for phenolphthalein and methyl orange indicators, along with colour in acidic and alkaline medium.

Sl. No.	Indicator	pH range	Colour in	
			Alkaline medium	Acidic medium
1.	Methyl orange			
2.	Phenolphthalein			

6. What is hardness of water? What are reasons for the hardness of water?
7. Differentiate between temporary and permanent hardness of water?
8. How permanent hardness of water can be removed?
9. Write the structural formula of EDTA.
10. What is redox titration? Describe with an example.
11. Differentiate between: i) Oxidising and reducing agent, ii) Internal and self indicator.
12. During preparation of Mohr's salt solution, why dil. H_2SO_4 is added?
13. What is the oxidation state of Cr in $K_2Cr_2O_7$ and Mn in $KMnO_4$?
14. Calculate the equivalent weight of $KMnO_4$ in acidic and alkaline medium. (K=39, Mn=55, O=16)
15. Write the integrated rate equation of a first order reaction. What is the SI unit of rate constant of first order reaction?
16. Describe pseudo –unimolecular reaction with an example.
17. How rate of reaction changes with temperature? Write Arrhenius equation and describe it.
18. Define pH. What is the range of pH scale?
19. Write down the relation between pH and pOH at 298 K.
20. What is the chemical composition of buffer having pH i) about 5 and ii) about 10.
21. Draw the nature of graph for the pH-metric titration of i) strong acid vs. strong base, ii) strong acid vs. weak base, iii) weak acid vs. strong base.
22. Define electrochemical cell. What is the difference between electrolytic and galvanic cells?
23. What is the difference between ionic and electronic conductance?
24. Draw the nature of graph for the conductometric titration of i) strong acid vs. strong base, ii) strong acid vs. weak base, iii) weak acid vs. strong base.
25. What is partition co-efficient?
26. What is viscosity? How viscosity changes with temperature for liquids and gases?