INSTRUCTION MANUAL FOR CHEMISTRY LABORATORY

(B. Tech. 1st/2nd Semester)

Course Name : Engineering Chemistry − I

Course Code : CYN11501/CYN12501

[Branch: Computer Science and Engineering (CSE), Electronics and Communication Engineering (ECE), Electrical Engineering (EE)]

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Practical: List of Experiments

Part A:

1. To determine the percentage of available chlorine in the supplied sample of bleaching powder.

2. To determine the total, permanent hardness, Ca^{2+} and Mg^{2+} hardness in supplied water sample by titrating with standard EDTA solution.

3. To determine the alkalinity of the supplied water sample.

4. To determine the strength of supplied $K_2Cr_2O_7$ solution using Ferrous Ammonium Sulphate solution as intermediate and Potassium Ferricyanide solution as an external indicator.

5. Preparation of Methyl Orange from sulfanilic acid using a diazonium coupling reaction.

Part B:

6. Verification of Kohlrausch's 1st law for strong electrolytes using conductometric measurement.

7. Verification of Ostwald's dilution law for weak electrolytes using conductometric measurement.

8. Determination of the strength of a strong acid by strong base using conductometric titration.

9. Preparation of Daniell Cell and its related study.

10. Determination of viscosity average molecular weight of a polymer sample by Ostwald Viscometer.
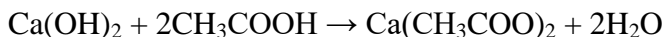
Part C:

11. Synthesis of TiO_2 nanoparticle via sol-gel method and determining the phase with X-ray Diffraction experiment.

Experiment: 1

To determine the percentage of available chlorine in the supplied sample of bleaching powder.
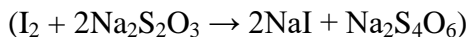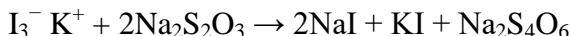
Chemicals and glassware: Sodium thiosulphate, potassium iodide, glacial acetic acid, starch solution, porcelain dish, test tube, 250 mL volumetric flask, funnel, burette, pipette, beaker, and conical flask.

Reactions: i) $CaOCl_2 + H_2O \rightarrow Ca(OH)_2 + Cl_2$ ($Cl_2 + H_2O \rightleftharpoons HOCl + HCl$)

                   $Ca(OH)_2 + 2CH_3COOH \rightarrow Ca(CH_3COO)_2 + 2H_2O$

                  ii) $Cl_2 + 2KI \rightarrow 2KCl + I_2$

                  iii) $I_2 + KI \rightarrow I_3{}^- K^+$

                    $I_3{}^- K^+ + 2Na_2S_2O_3 \rightarrow 2NaI + KI + Na_2S_4O_6$

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

Procedure: Weigh accurately about 1.5 g of bleaching powder in the porcelain dish, and rub it with the flat end of the test tube after adding a little amount of deionized water until a thick paste is formed. Transfer the contents of the dish completely into a 250 mL volumetric flask by adding more deionized water and dilute it upto the half of volumetric flask. Add 3 mL of glacial acetic acid and a little amount of deionized water to the dish, wash, and transfer it into the volumetric flask. Shake the volumetric flask after washing the funnel by adding around 20−30 mL of deionized water to get a clear solution and then, dilute it with deionized water to the mark. Pipette out 25 mL of the clear solution and transfer it into a conical flask. Add 2 mL of glacial acetic acid and 1 mL of KI solution resulting in a deep brown solution. Immediately, titrate the liberated iodine with standard Hypo solution (Sodium thiosulphate, $Na_2S_2O_3$) taken in the burette till the faint yellow colour of the remaining iodine. Now, add about 10 drops of starch solution which gives a deep blue colour to the contents of the conical flask. Continue the titration with Hypo solution till the blue colour just disappears and becomes a colourless solution.

Repeat the process by taking 25 mL of beaching powder solution until get two concordant readings with Hypo solution.

<div align="center">(Eq. Wt. of Hypo = 248.19 g)</div>

Observations:

1. Weight of bleaching powder taken $= W$ g   = ……………. g
2. Strength of Hypo solution (supplied)   = ………. g/L

Table: Volume of Hypo solution used for 25 mL of Bleaching Powder solution

S. No.	Burette reading		Volume used, $(V_2 - V_1)$ mL	Concordant Value, V mL
	Starting Point, V_1 mL	End Point, V_2 mL		
1				
2				
3				

Calculations:

Consider, X is the strength of available chlorine in normality (N), then

$$N_1 V_1 = N_2 V_2$$
$$(Cl_2) \quad (Na_2S_2O_3)$$
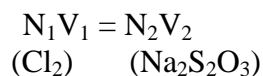
$$X \times 25 = \frac{V \times \text{Strength of Hypo solution} \left(\frac{g}{L}\right)}{\text{Eq. Wt. of Hypo}}$$

$$\therefore \quad X = \frac{V \times \text{Strength of Hypo solution} \left(\frac{g}{L}\right)}{25 \times \text{Eq. Wt. of Hypo}} \text{ N}$$

i.e., X mole of chlorine is available in 1 L or 1000 mL of the bleaching powder dissolved solution.

∴ The strength of available chlorine (g) in 1000 mL of the bleaching powder dissolved solution,

$$Y = \text{Normality} \times \text{Eq. Wt. of chlorine } (Cl_2) = X \times 35.45 \text{ g}$$

i.e., Y g of chlorine is available in 1000 mL of the bleaching powder dissolved solution.

In 250 mL,

$$Z = \frac{Y \times 250}{1000}$$

∴ Z g of chlorine is available in 250 mL of the bleaching powder dissolved solution.

i.e., W g of bleaching powder contains Z g of available chlorine.

∴ The percentage of available chlorine in the supplied sample of bleaching powder is

$$= \frac{Z \times 100}{W} \text{ \%}$$

Result: The % of available chlorine in the supplied sample of bleaching powder is ………….

Experiment: 2

To determine the total hardness, Ca^{2+} hardness, and Mg^{2+} hardness in the supplied water sample by titrating with standard EDTA solution.

Chemicals and glassware: Calcium carbonate, hydrochloric acid, EDTA solution, buffer solution (pH 10), Eriochrome Black T, Murexide, sodium hydroxide, weighing tube, 250 mL volumetric flask, funnel, glass rod, conical flask, pipette, burette, and beaker.

Theory: For determining Calcium and Magnesium hardness separately, the total hardness is being determined first by titration against Ethylenediaminetetraacetic acid (EDTA) using the buffer of pH 10 and Eriochrome Black T (EBT) indicator. In the next step, Magnesium ions are precipitated in the form of $Mg(OH)_2$ by adding NaOH which rises pH of the solution to 12.5. Calcium hardness left in the water can be determined by titration using Murexide indicator.

Reaction:

$(Ca^{2+}$ or $Mg^{2+})$ **EDTA** $[M(EDTA)]^{2-}$
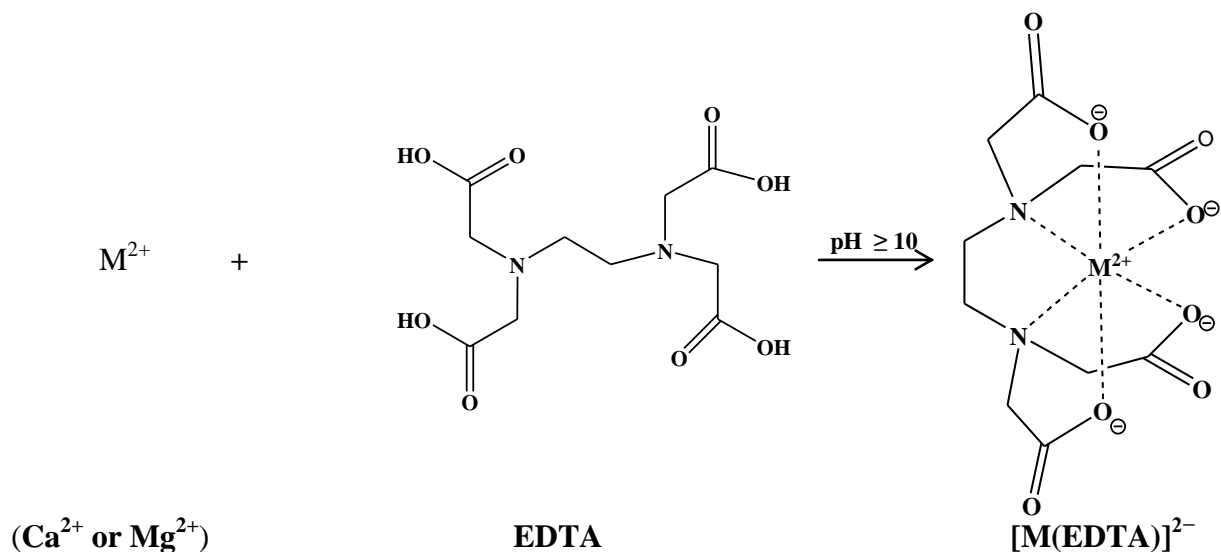
Procedure:

Standardization of EDTA solution
Prepare a standard solution of $CaCO_3$ by dissolving 0.125 g of $CaCO_3$ in minimum amount of dil. HCl by boiling. Cool and make the solution in a 250 ml volumetric flask using deionized water. Take 25 mL of standard solution in a conical flask and add 2 mL buffer solution of pH 10 (supplied). Now, add 2−3 drops of the EBT indicator to the solution- a wine red colour appears. Titrate against EDTA solution till a blue colour appears. Note the observation for two concordant readings (V_s mL) in Table 1.

To determine the total hardness of the water sample, take 25 mL of water sample in a conical flask, add 2 mL of buffer solution of pH 10, and 2−3 drops of the EBT indicator. Titrate against standard EDTA solution and record the readings as V_t in Table 2.

To determine the calcium hardness, Transfer 25 mL of water sample into a conical flask with the help of a pipette. Add 5 mL of NaOH and 3−4 drops of Murexide indicator. Shake the flask and titrate it against standard EDTA solution until the colour changes from slight reddish to slight pink. Repeat the titration until two concordant readings are obtained. Record the observations as V_c in Table 3.

Observations:

1. Weight of empty weighing tube $= W_1$ g = ………… g
2. Weight of tube + $CaCO_3$ powder $= W_2$ g = ………… g
3. Weight of tube after transferring the substance to volumetric flask $= W_3$ g =………g
4. Weight of $CaCO_3$ powder actually taken $W = (W_2\text{-}W_3)$ g = …….g
5. Observation for standardisation of EDTA (V_s) (Table 1) = ……….. mL
6. Observation for Titration with EBT (V_t) for total hardness (Table 2) = ……… mL
7. Observation for Titration with Murexide (V_c) (Table 3) = ……… mL

Table 1: Titration value for standardization of EDTA solution against standard $CaCO_3$ solution.

S. No.	Burette reading		Volume used, (V_2-V_1) mL	Concordant Value, V_s mL
	Starting Point, V_1 mL	End Point, V_2 mL		
1.				
2.				
3.				

Table 2: Titration value for total hardness of unknown water sample

S. No.	Burette reading		Volume used, (V_4-V_3) mL	Concordant Value, V_t mL
	Starting Point, V_3 mL	End Point, V_4 mL		
1.				
2.				
3.				

Table 3: Titration value for Calcium hardness of unknown water sample

S. No.	Burette reading		Volume used, $(V_6 - V_5)$ mL	Concordant Value, V_c mL
	Starting Point, V_5 mL	End Point, V_6 mL		
1.				
2.				
3.				

Calculations:

1. **Standardization of EDTA solution**

    Weight of $CaCO_3$ in 250 ml = W g

    ∴ Weight of $CaCO_3$ per litre = 4W g

    Normality of $CaCO_3$ solution = $\dfrac{4W}{50}$ = N_2 (say)

    Then,

    $N_1 \times V_s = N_2 \times V_2$ where N_1 is the normality of EDTA solution.
    (EDTA) ($CaCO_3$)

    $$N_1 = \frac{4W}{50} \times \frac{25}{V_s} = \dots\dots N$$

2. **Total hardness of a given water sample**

    $$N_1 \times V_t = N_2 \times 25$$
    (EDTA)   (Water sample)

    $$N_2 = \frac{N_1 \times V_t}{25}$$

    Total hardness of the water sample in ppm = $N_2 \times$ gram eq. wt. of $CaCO_3$ g/1000 mL

    $$= \frac{N_1 \times V_t \times 50 \times 1000}{25} \text{ mg/1000 mL or ppm}$$

3. **Calcium hardness of a given water sample**

    Calcium hardness of the water sample in ppm = $\dfrac{N_1 \times V_c \times 50 \times 1000}{25}$ ppm

Results:

(I)   Total hardness of a given water sample = ... ppm

(II) Ca-hardness of a given water sample = ... ppm

(III) Mg-hardness of a given water sample = (Total hardness – Ca-hardness)
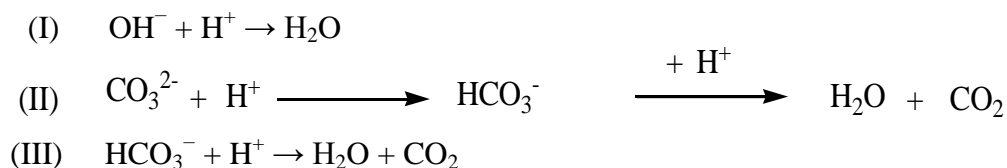
    = ……………. ppm

Experiment: 3

To determine the alkalinity of the supplied water sample.

Chemicals and glassware: Sodium carbonate, acid solution, phenolphthalein, methyl orange, 250 mL volumetric flask, funnel, glass rod, weighing tube, conical flask, pipette, and burette.

Theory: In a water sample, the alkalinity is generally due to the presence of hydroxide, carbonate and / or bicarbonate ions. Alkalinity can be expressed as follows:

$$\text{Alkalinity (mol/L)} = [HCO_3^-] + 2\,[CO_3^{2-}] + [OH^-] - [H^+]$$

It is determined by the titration with the standard acid solution. Titration is performed in the presence of two indicators i.e. phenolphthalein and methyl orange to characterize the type of alkalinity present and the composition of alkalinity.

(I) $OH^- + H^+ \rightarrow H_2O$

(II) $CO_3^{2-} + H^+ \xrightarrow{\hspace{2cm}} HCO_3^- \xrightarrow{\;+\;H^+\;} H_2O + CO_2$

(III) $HCO_3^- + H^+ \rightarrow H_2O + CO_2$

Procedure:

Standardization of acid solution:
Weigh accurately 0.265 g of Na_2CO_3 (dried) A. R. grade, transfer it in a 250 mL volumetric flask. Dissolve in about 100 mL deionized water and make it up to the mark by diluting with more deionized water. Pipette out 25 mL of this solution in a conical flask add 2−4 drops of methyl orange indicator and titrate with acid solution. Note down the end point when light yellow colour changes to red. Repeat the titration for two concordant readings. Note down the readings as V_s as given below in the Table 1.

Titration of unknown water sample:
Pipette out 25 mL of water sample in a clean conical flask, add 2−4 drops of phenolphthalein indicator and titrate with standard acid solution till pink colour disappears. Record the volume of acid used as V_p mL. To the same solution, add 2−3 drops of methyl orange indicator, and continue the titration with same acid solution. At the end point, light yellow colour sharply changes to red. Record the reading as V_m mL. Repeat the titration by taking 25 mL of water sample until get two concordant observations as given below in the Tables 2 and 3.

Observations:

1. Weight of empty weighing tube $\qquad = W_1$ g = ………… g
2. Weight of tube + Na_2CO_3 powder $\qquad = W_2$ g = ………… g
3. Weight of tube after transferring the substance to volumetric flask = W_3 g =………g
4. Weight of Na_2CO_3 powder actually taken $\qquad W = (W_2-W_3)$ g = ……g
5. Volume of the acid used for standardisation with Na_2CO_3 $\quad = V_s$ mL = ………. mL

Table 1: Titration value for standardization of acid solution

S. No.	Burette reading		Volume used, (V_2-V_1) mL	Concordant Value, V_s mL
	Starting Point, V_1 mL	End Point, V_2 mL		
1.				
2.				
3.				

Titration values for unknown water sample

Table 2: Using phenolphthalein indicator

S. No.	Burette reading		Volume used, (V_4-V_3) mL	Concordant Value, V_p mL
	Starting Point, V_3 mL	End Point, V_4 mL		
1.				
2.				
3.				

Table 3: Using methyl orange indicator

S. No.	Burette reading		Volume used, (V_5-V_3) mL	Concordant Value, V_m mL
	Starting Point, V_3 mL	End Point, V_5 mL		
1.				
2.				
3.				

Calculations:

(a) Standardization of acid by Na$_2$CO$_3$

N$_1$ is the normality of acid solution.

$$N_1V_1 = N_2V_2$$
$$\text{(Acid)} \quad \text{(Na}_2\text{CO}_3)$$

$$N_1 \times Vs = \frac{W}{53} \times \frac{1000}{250} \times 25$$

$$N_1 \times V_s = \frac{4W}{53} \times 25$$

Therefore, $$N_1 = \frac{4W}{53} \times \frac{25}{V_s} = \ldots\ldots\ldots N$$

(b) Determination of the alkalinity of the supplied water sample

$$N_1 \times V = N_2 \times 25 \quad \text{where N}_2 \text{ is the normality of alkaline water (CaCO}_3).$$
$$\text{(Acid)} \quad \text{(Water sample)}$$

$$N_2 = \frac{N_1 \times V}{25}$$

Alkalinity of the water sample in ppm $= N_2 \times$ gram eq. wt. of CaCO$_3$ $\qquad$ g/1000 mL

$$= \frac{N_1 \times V}{25} \times 50 \times 1000 \qquad \text{mg/1000 mL or ppm}$$

Therefore,

Alkalinity up to phenolphthalein end point (in ppm),
$$\mathbf{P} = \frac{N_1 \times V_p \times 50 \times 1000}{25} \quad \text{ppm}$$

Similarly, alkalinity up to methyl orange end point (in ppm),
$$\mathbf{M} = \frac{N_1 \times V_m \times 50 \times 1000}{25} \quad \text{ppm}$$

The type of alkalinity can be obtained from the following table (interpreting by P and M)

S. No.	Condition	OH^- in ppm	CO_3^{2-} in ppm	HCO_3^- in ppm
(I)	$P = 0$	Nil	Nil	M
(II)	$P = M$	P	Nil	Nil
(III)	$P = \frac{1}{2} M$	Nil	2P	Nil
(IV)	$P > \frac{1}{2} M$	2P – M	2(M - P)	Nil
(V)	$P < \frac{1}{2} M$	Nil	2P	M – 2P

Results:

(I) OH^- alkalinity = ... ppm

(II) CO_3^{2-} alkalinity = ... ppm

(III) HCO_3^- alkalinity = ... ppm

Experiment: 4

To determine the strength of supplied $K_2Cr_2O_7$ solution using Ferrous Ammonium Sulphate (FAS) solution as an intermediate and Potassium Ferricyanide solution as an external indicator.

Chemicals and glassware: Potassium dichromate, ferrous ammonium sulphate, potassium ferricyanide, dil. H_2SO_4, 250 mL volumetric flask, conical flask, pipette, burette, funnel, glass rod, weighing tube, and white tile.

Reactions:

$$K_2Cr_2O_7 + 6(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O + 7H_2SO_4 \longrightarrow 3Fe_2(SO_4)_3 + Cr_2(SO_4)_3 + K_2SO_4 +$$

$$6(NH_4)_2SO_4 + 43H_2O$$

In general, $\quad Cr_2O_7^{2-} + 6Fe^{2+} + 14H^+ \longrightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$
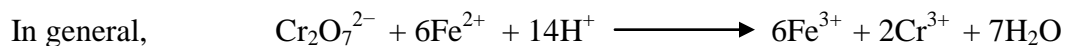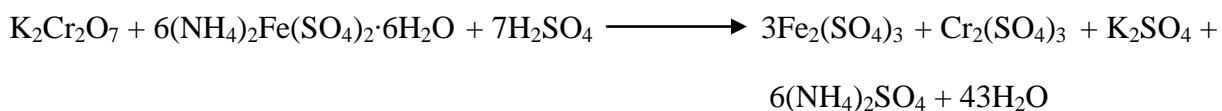
Procedure:

Standardization of FAS solution:
Weigh out exactly about 0.306 g of $K_2Cr_2O_7$ crystals in weighing tube. Transfer it into a 250 mL volumetric flask, dissolve it with deionized water, and make the solution up to the mark. Fill the burette with prepared $K_2Cr_2O_7$ solution. Pipette out 25 mL of Ferrous Ammonium Sulphate solution (FAS, supplied) in the conical flask and add 5 mL of dil. H_2SO_4 (supplied). Put a number of drops of freshly prepared Potassium Ferricyanide solution (supplied) on a clean dry white tile with the help of a glass rod. Now, add $K_2Cr_2O_7$ solution gradually 1 mL at a time from the burette. After adding of each mL, shake the contents of the conical flask and put a drop of the solution over one drop of the indicator on the tile with the help of a glass rod. After touching the drop, wash the rod thoroughly in running (tap) water follow by deionized water and dry it. The end point is the disappearance of the blue color when a drop of the solution is in contact with the indicator placed on a tile. Suppose, the drop gets coloured at 15 mL of $K_2Cr_2O_7$ solution and remains unchanged at 16 mL. This indicates that the end point lies between 15 mL and 16 mL of $K_2Cr_2O_7$ solution. Again, take 25 mL of FAS solution in the conical flask, add 5 mL of dil. H_2SO_4 and titrate upto 15 mL of $K_2Cr_2O_7$ solution directly from the burette. Later, add $K_2Cr_2O_7$ solution gradually 0.1 mL at a time and test the solution as before. Find the end point at which color unchanged. Repeat the procedure until to get atleast two concordant readings.

Titration of supplied unknown $K_2Cr_2O_7$ solution:
Fill the burette with unknown $K_2Cr_2O_7$ solution, pipette out 25 mL of FAS solution in a conical flask, and follow the same procedure until get two concordant readings.

(Eq. Wt. of $K_2Cr_2O_7$ = 49.03)

Observations:

1. Weight of empty weighing tube $= W_1$ g = ………… g
2. Weight of tube + $K_2Cr_2O_7$ crystal $= W_2$ g = ………… g
3. Weight of tube after transferring the substance to volumetric flask $= W_3$ g = ………….. g
4. Weight of $K_2Cr_2O_7$ crystal actually taken, $W = (W_2 - W_3)$ g = ………… g

Table 1: Titration values to determine the concentration of supplied FAS solution

Volume of FAS solution for standardization, V = 25 mL

S. No.	Burette reading		Volume used, (V_2-V_1) mL	Concordant Value, V_s mL
	Starting Point, V_1 mL	End Point, V_2 mL		
1.				
2.				
3.				

Table 2: Titration value for unknown $K_2Cr_2O_7$ solution

Volume of FAS solution for unknown $K_2Cr_2O_7$ solution, V = 25 mL

S. No.	Burette reading		Volume used, (V_4-V_3) mL	Concordant Value, V_u mL
	Starting Point, V_3 mL	End Point, V_4 mL		
1.				
2.				
3.				

Calculations:

(a) Standardization of FAS by known $K_2Cr_2O_7$ solution

$$N_1V_1 = N_2V_2$$
$$(FAS) \quad (K_2Cr_2O_7)$$
$$N_1 \times V = N_2 \times V_s$$

$$N_1 \times 25 = \frac{W}{49.03} \times \frac{1000}{250} \times V_s$$

$$\text{Normality of FAS, } N_1 = \frac{4W}{49.03} \times \frac{V_s}{25} = ……….. N$$

(b) Determination of the strength of a supplied unknown $K_2Cr_2O_7$ solution

$$N_1V_1 = N_2V_2$$
$$\text{(FAS)} \quad \text{(}K_2Cr_2O_7\text{)}$$
$$N_1 \times V = N_2 \times V_u \qquad \text{where } N_2 \text{ is the normality of unknown } K_2Cr_2O_7 \text{ solution.}$$

Therefore, $\qquad N_2 = \dfrac{N_1 \times 25}{V_u} = \text{...........} \ N$

The strength of supplied $K_2Cr_2O_7$ solution $= N_2 \times 49.03 = \text{............} \ g/L$

Result:

The strength of supplied $K_2Cr_2O_7$ solution is …………. g/L.

Experiment: 5

Preparation of Methyl Orange from sulfanilic acid using a diazonium coupling reaction.

Chemicals and glassware: Sulfanilic acid, anhydrous sodium carbonate, sodium nitrite, Conc. HCl, *N,N*-dimethylaniline, glacial acetic acid, sodium hydroxide, sodium chloride, 250 mL beaker,
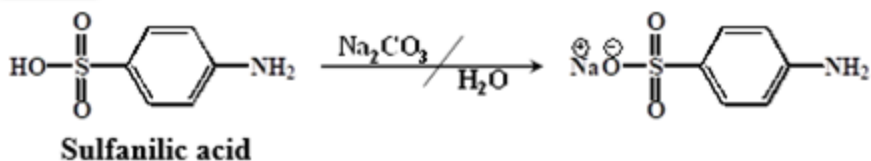
Theory:

Methyl orange is an azo dye that forms beautiful orange crystals and is used as an acid-base indicator. The anion form is yellow and the acid form is red.
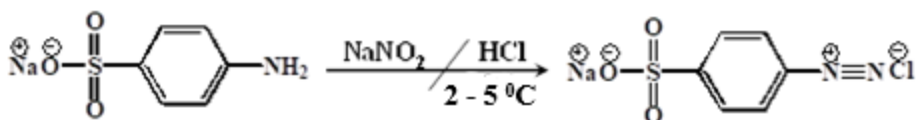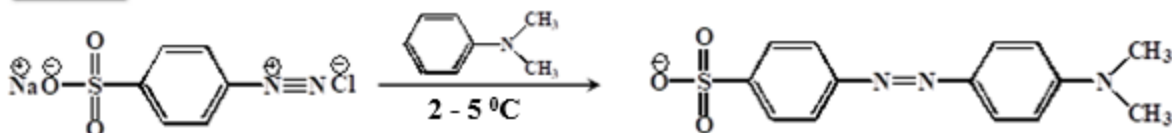
Structure of Methyl Orange

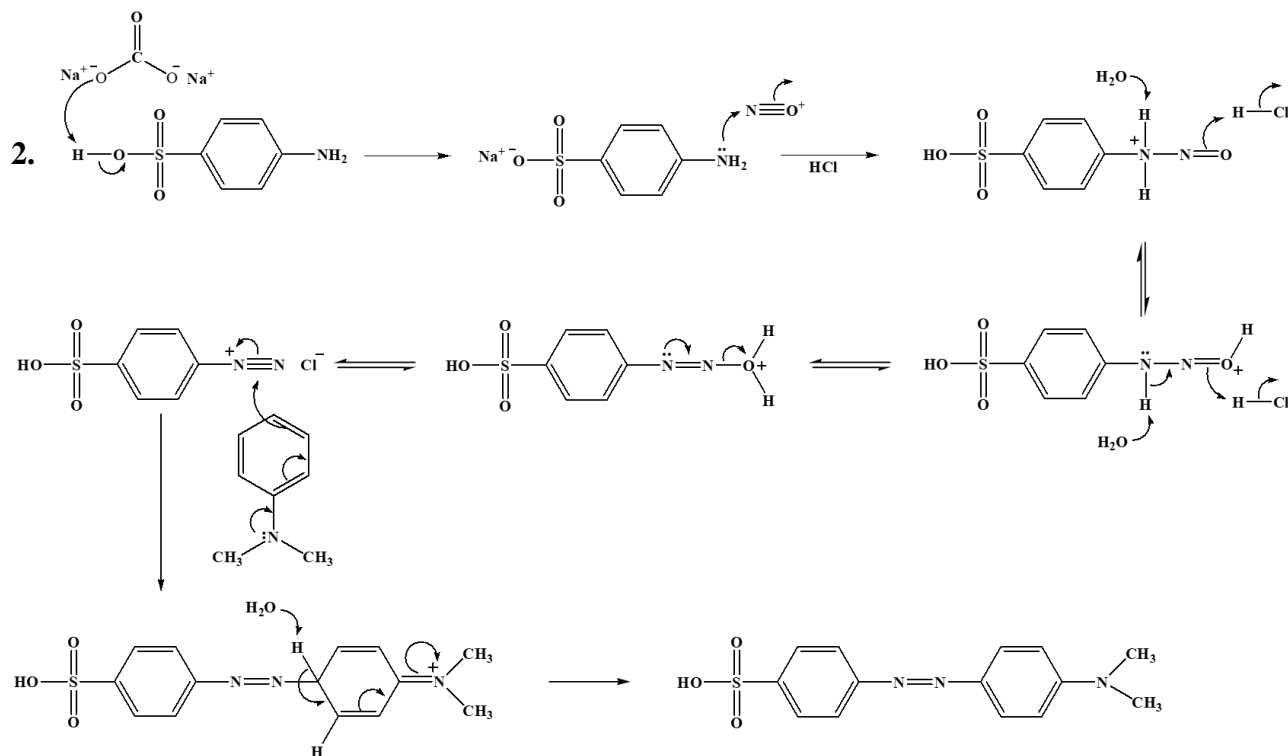|	
Yellow, pH > 4.4	**Red, pH < 3.1**
	(Inner salt form)
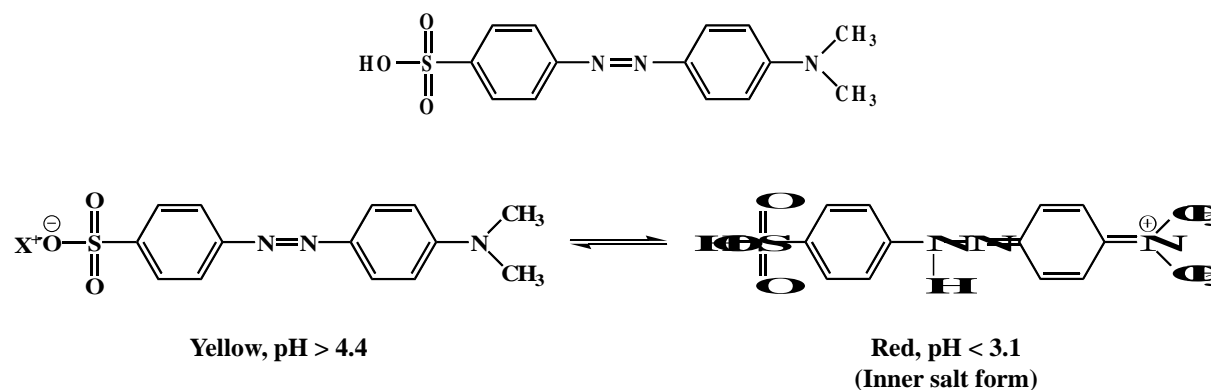
Reactions:

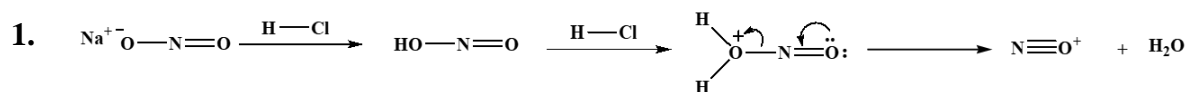Step-I

Sulfanilic acid

Step-II

Step-III

Reaction mechanism:

1.

2.

Procedure:

Take 5 g of sulfanilic acid, 1.5 g of anhydrous sodium carbonate, and 50 mL of deionized water in a 250 mL beaker and stir the mixture to obtain a clear solution (warm if necessary). Cool the resulting solution in ice-salt freezing mixture. To this cold solution, first add sodium nitrite solution (2 g of Sodium Nitrite dissolved in 15 mL of ice-cold water) and then, add ice-cold hydrochloric acid solution (5 mL of Conc. HCl in 25 mL of ice-cold water) slowly with continuous stirring. During this addition, the temperature of the overall solution should be maintained between 2−5 °C. Test for free nitrous acid with potassium iodide starch paper. Fine crystals of Diazobenzene sulphonate will separate after 15 min. **Do not filter as this will be dissolved during next stage of preparation**.

In a 100 mL beaker, mix 3.1 mL of *N,N*-dimethylaniline and 1.5 mL of glacial acetic acid in 8 mL of deionized water. Cool the mixture in an ice bath and then, add this solution to the diazotized solution slowly with vigorous stirring and maintain the temperature between 2−5 °C.

Allow the mixture to stand for 15 minutes at the temperature of 2−5 °C and separate the red orange form of the methyl orange. Then, make the resulting solution alkaline by dropwise adding 40 mL of 20% NaOH solution with continuous stirring. Heat the reaction mixture nearly to boil, add 15 g of sodium chloride, and stir it in hot condition until a clear solution is obtained. Cool the solution in an ice bath to separate the solid product, filter, and wash with ice-cold water. Recrystallize the solid product from hot water, filter, and then, dry in air.

Results:

Yield of the product, methyl orange = ……… g

Colour = …………

Experiment: 6

To verify Kohlrausch's 1st law for strong electrolytes using conductometric measurements and determine the limiting molar conductivity (Λ_m^0) of NaCl.

Chemicals and glassware: NaCl, 100 mL volumetric flask, 100 mL beaker, and pipette.

Theory:

Conductance of a solution containing an electrolyte can be determined by dipping a conductivity cell into the solution.

Ohm's law may be rearranged into: $\quad I = \dfrac{V}{R} = GV$

where **G** is the **conductance** and is reported in siemens, S (1 S = 1 Ω^{-1} = 1 A V^{-1}). In older literature, conductance is reported in 'reciprocal ohms', Ω^{-1} (mho).

The conductance of a sample depends on its dimensions (as well as the temperature and pressure). For a sample of length L (distance between two electrodes) and cross-section A (area of cross section of electrode), the conductance is:

$$G = \kappa\left(\dfrac{A}{L}\right) \qquad \text{i.e.} \qquad \kappa = G\left(\dfrac{L}{A}\right) = \dfrac{1}{R}\left(\dfrac{L}{A}\right)$$

where κ (kappa) is the **conductivity**, with unit: siemens per meter (S m^{-1}) or siemens per centimetre (S cm^{-1}). (L/A) is called cell constant, unit: m^{-1} or cm^{-1}.

The conductance of the solution is measured by dipping platinum electrode into the solution. This measurement is done by conductivity cell. The cell is connected with the conductivity-meter. Alternating Current (AC) should be used.

Molar conductivity (Λ_m) of an electrolyte [in S m^2 mol^{-1} or S cm^2 mol^{-1}] is given by,

$$\Lambda_m = \kappa\,/c$$

Where, κ (kappa) is conductivity (in S m^{-1} or S cm^{-1}) and c is molar concentration of the electrolyte (in mol dm^{-3} or mol L^{-1}).

Strong electrolytes are substances that are virtually ionised in solution and includes ionic solids and strong acids. It has been observed that, at low concentrations, the molar conductivities (Λ_m) of strong electrolytes vary linearly with the square root of the concentration (c):

$$\Lambda_m = \Lambda_m^0 - K\sqrt{c}$$

This variation is called **Kohlrausch's law.** The constant Λ_m^0 is the **limiting molar conductivity**, the conductivity in the limit of zero concentration. The constant K is found to depend more on

the stoichiometry of the electrolyte (i.e. whether it is of the form MA or M_2A, etc.) than on its specific identity.

Procedure:

1. Prepare 1M NaCl solution in a 100 mL volumetric flask with deionized water.
2. Pipette out 10 mL of 1M NaCl solution and prepare 0.1M NaCl solution in a 100 mL volumetric flask (1st solution).
3. Pipette out 25 mL of 0.1 M NaCl solution, transfer it into another (2nd solution) empty 100 mL volumetric flask and make it up to 100 mL by deionized water to obtain 0.025 M solution. Remaining volume (75 mL) of 0.1 M solution will be used for conductivity measurements.
4. Use the procedure mentioned in step-2 to prepare 6.25×10^{-3} M (3rd solution) from 0.025 M solution.
5. Similarly, prepare 1.5625×10^{-3} M (4th solution) from 6.25×10^{-3} M solution and 3.9062×10^{-4} M (5th solution) from 1.5625×10^{-3} M solution in 100 mL volumetric flasks.
6. Measure κ for five solutions at room temperature (…… °C).
7. Report κ and Λ_m in tabular form (table 1).
8. Plot Λ_m (Y-axis) against $\sqrt{c}$ (X-axis) on a graph paper and determine the value of Λ_m^0 for NaCl from the graph from Y-intercept.

Table 1:

S. No.	Concentration of NaCl solution (c) (unit: M)	$\sqrt{c}$	Conductivity (κ, kappa) (unit: S/cm)	Molar conductivity ($\Lambda_m = 1000\ \kappa\ /c$) (unit: S cm^2 mol^{-1})
1.	0.1			
2.	0.025			
3.	0.00625			
4.	0.0015625			
5.	0.00039			

Conductivity of deionized water = …….. S/cm

Result:

Limiting molar conductivity (Λ_m^0) of NaCl at …… °C is …… S cm^2 mol^{-1} or ……. S m^2 mol^{-1}.
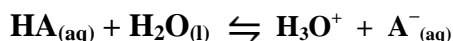
Experiment: 7

To verify Ostwald's dilution law for weak electrolytes using conductometric measurements and to determine the limiting molar conductivity (Λ_m^0) of acetic acid.

Chemicals and glassware: Acetic acid, 100 mL volumetric flask, 100 mL beaker, and pipette.

Theory:

For a weak acid,

$$HA_{(aq)} + H_2O_{(l)} \leftrightharpoons H_3O^+ + A^-_{(aq)}$$

$$K_a = \frac{[H_3O^+][A^-]}{[HA]}$$

where, K_a is the acidity constant of an acid.

If the degree of deprotonation of an acid is α at a molar concentration of c, at equilibrium,

$[H_3O^+] = \alpha c$, $[A^-] = \alpha c$, $[HA] = (1 - \alpha) c$

Ignoring activity coefficients, the acidity constant K_a, is:

$$K_a = \frac{\alpha^2 c}{(1 - \alpha)} \quad \text{........................ (1)}$$

The acid is fully deprotonated at infinite dilution; its limiting molar conductivity is Λ_m^0. The measured molar conductivity Λ_m is given by,

$$\Lambda_m = \alpha \, \Lambda^\circ_m \quad \text{........................ (2)}$$

From eq. (1), we get

$$\frac{1}{\alpha} = 1 + \frac{\alpha c}{K_a}$$

By using eq. (2), we get **Ostwald's dilution law:**

$$\frac{1}{\Lambda_m} = \frac{1}{\Lambda^\circ_m} + \frac{(\Lambda_m c)}{K_a (\Lambda^\circ_m)^2}$$

Procedure:

1. Prepare 1M CH_3COOH solution in a 100 mL volumetric flask with deionized water.

2. Pipette out 10 mL of 1M CH_3COOH solution and prepare 0.1M CH_3COOH solution in a 100 mL volumetric flask (1st solution).

3. Pipette out 25 mL of 0.1 M CH_3COOH solution, transfer it into another (2nd solution) empty 100 mL volumetric flask and make it up to 100 mL by deionized water to obtain 0.025 M solution. Remaining volume (75 mL) of 0.1 M solution will be used for conductivity measurements.

4. Use the procedure mentioned in step-2 to prepare 6.25×10^{-3} M (3rd solution), from 0.025 M solution.

5. Similarly, prepare 1.5625×10^{-3} M (4th solution) from 6.25×10^{-3} M solution and 3.9062×10^{-4} M (5th solution) from 1.5625×10^{-3} M solution in 100 mL volumetric flasks.

6. Measure κ for five solutions at room temperature (……. °C).

7. Report κ and Λ_m in tabular form (table 1).

Table 1:

Sl. No.	Concentration of CH_3COOH solution (c) (unit: M)	Conductivity (κ, kappa) (unit: S/cm)	Molar conductivity (Λ_m) (unit: S cm^2 mol^{-1})	$\dfrac{1}{\Lambda_m}$	$\Lambda_m c$
1.	0.1				
2.	0.025				
3.	0.00625				
4.	0.0015625				
5.	0.00039				

Conductivity of deionized water = …….. S/cm

A graph plot of $\dfrac{1}{\Lambda_m}$ (y-axis) against ($\Lambda_m c$) (x-axis) should give a straight line, where y-intercept is $\dfrac{1}{\Lambda°_m}$ and Λ_m^0 can be calculated from the value of y-intercept.

Result:

Limiting molar conductivity of CH_3COOH at ……. °C is …… S cm^2 mol^{-1} or ……. S m^2 mol^{-1}.

Experiment: 8

Determination of the strength of a strong acid by strong base using conductometric titration.

Chemicals and glassware: NaOH, HCl, oxalic acid, phenolphthalein indicator, 100 mL volumetric flask, 100 mL beaker, pipette, and burette.

Theory:

When a strong acid HCl is taken in a beaker as titrant and NaOH solution is added gradually to it as titre from a burette, the reaction occurring during neutralization is given by,

$$HCl \ + \ NaOH \ \rightarrow \ NaCl \ + \ H_2O$$
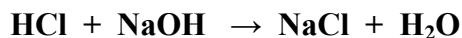
As NaOH solution is gradually added, the H^+ ions having high ionic conductivity are replaced by Na^+ ions having lower ionic conductivity and hence, conductivity of the solution in the beaker gradually decreases. At equivalence point, the conductivity would be minimum. After equivalence point, the conductivity of the solution increases due to accumulation of Na^+ and OH^- ions. If the conductivity corresponding to the volume of NaOH solution be plotted, two lines having opposite slopes would be obtained. The point of intersection of the two straight lines would give the equivalent point.

Procedure:

1. Prepare 0.5N NaOH solution in a 100 mL of volumetric flask.
2. Prepare 0.1N oxalic acid solution in a 100 mL of volumetric flask.
3. Standardize the prepared NaOH solution by oxalic acid solution (25 mL in a conical flask) using phenolphthalein indicator.
4. Pipette out 25 mL of a given unknown HCl solution and transfer it into a 100 mL beaker. Dip the electrode in the solution (if electrode is above the solution, add some volume of deionized water to dip it). Note down its conductivity. Then, add 0.5 mL of NaOH solution each time from the burette, mix the solution and note the conductivity (never lift the electrode from the beaker solution once the NaOH addition is started).
5. Plot conductivity (in S/cm) (y-axis) vs total volume of NaOH added (x-axis) on a graph paper (two lines with opposite slopes would be obtained). The point of intersection of the two straight lines would give the equivalence point. Find out the equivalence point from the graph.
6. Calculate the strength of a given HCl solution in normality.

Table 1: Standardization of NaOH solution

Volume of 0.1 N oxalic acid in a conical flask = 25 mL

S. No.	Burette reading		Volume of NaOH used, (V_2-V_1) mL	Concordant Value, V mL
	Starting Point, V_1 mL	End Point, V_2 mL		
1				
2				
3				

$$N_1V_1 = N_2V_2$$
$$\text{(NaOH)} \quad \text{(Oxalic acid)}$$

Strength of NaOH solution, $N_1 = \dfrac{N_2V_2}{V_1} = \dfrac{0.1 \times 25}{\text{Volume of NaOH}} = \text{..........} \text{ N}$

Table 2: Conductometric titration

Strength of NaOH solution = ………… N

Volume of HCl solution used = 25 mL

Temperature = …….. °C

S. No.	Total volume of NaOH added (mL)	Conductivity (κ, kappa) (S/cm)
1.	0.5	…
2.	1.0	…
3.	1.5	…
…	…	…

$$N_1V_1 = N_2V_2$$
$$\text{(HCl)} \quad \text{(NaOH)}$$

Strength of a given HCl solution, $N_1 = \dfrac{N_2V_2}{V_1}$

$$= \dfrac{\text{Strength of NaOH (N)} \times \text{Total volume of NaOH added (mL)}}{25}$$

$$= \text{..........} \text{ N}$$

Result:

The strength of a given HCl solution = ………. N.

Experiment: 9

**Preparation of Daniell Cell and its related study.**

Objective:
 • To build a complete galvanic cell (Daniell cell).
 • To find the EMF of this system under standard conditions.

Chemicals and glassware: Copper sulphate ($CuSO_4 \cdot 5H_2O$), zinc sulphate ($ZnSO_4 \cdot 7H_2O$), potassium nitrate, Zn plate, Cu plate, 50 mL beaker, 100 mL volumetric flask, and burette.

Theory:

The Daniell Cell in 1836 was the starting point of modern electrochemistry because, for the first time, a reliable source of current at a precise voltage was available. It also was the first practical cell that does not generate a gas when operating. This cell was used as the first standard to define the unit "Volt".

$$Zn_{(s)} + Cu^{2+}_{(aq)} \longrightarrow Zn^{2+}_{(aq)} + Cu_{(s)} \text{ with E}^{\circ} = 1.10 \text{ V}$$
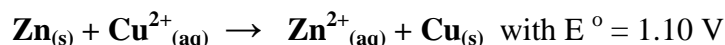
However, when the concentration of either $Cu^{2+}_{(aq)}$ or $Zn^{2+}_{(aq)}$ changes in the solution, the EMF of the cell (or potential) also changes according to the Nernst equation.

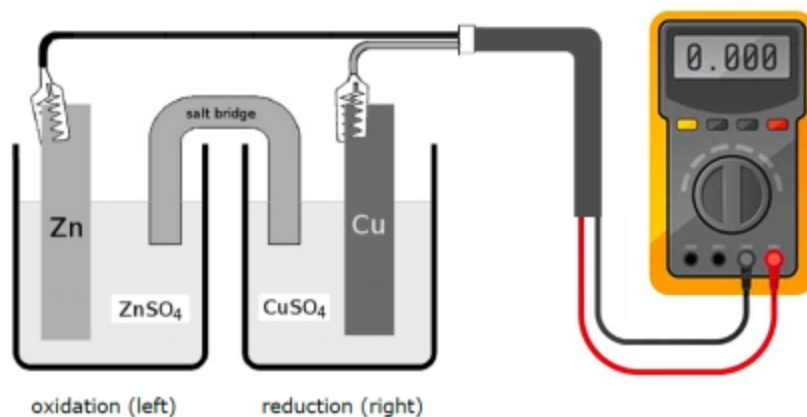$$E = E^{\circ} - \frac{RT}{nF} \ln Q$$

where $Q = \dfrac{[products]}{[reactants]}$

$$E = E^0 - 0.0296 \log \frac{[Zn^{2+}]}{[Cu^{2+}]} \quad \text{at 25 °C} \ldots\ldots\ldots\ldots (1)$$

[$R = 8.314$ J.K^{-1}.mol^{-1}, $T = 298$ K, $n = 2$ (electron exchanged), $F = 96485$ C.mol^{-1}]

The purpose of this lab is to use the Daniell cell to corroborate the Nernst law (potential vs. concentration). It will also be used to find the concentrations (activity) of copper in a Cu^{2+} (sat.) reference electrode as well as in an unknown sample of Cu^{2+}.

Figure: Laboratory set up of Daniell Cell.

Two 50 mL beakers are used as half-cell. The salt bridge is a rolled filter paper having both extremities in contact with each solution together. A few drops of 1.5M KNO_3 are added to wet the paper at the center to ensure ionic conductivity. A digital voltmeter is used to measure the EMF of the cell. The device set to "mV" position. It is essential to use a voltmeter to read the voltage since this instrument does not require any current from the circuit to perform its measurement (high impedance). A coaxial cable ending with two alligator clips is used. The black cable (low potential) is connected to the electrode performing an oxidation here the zinc. The connections are made according to cell representation (electrons from the left to the right).

(left = black cable) Zn | Zn^{2+} (0.10 M) || Cu $^{2+}$ (0.10 M) | Cu **(red cable = right)**

Procedure:

Prepare 0.10 M $CuSO_4 \cdot 5H_2O$ and 0.10 M $ZnSO_4 \cdot 7H_2O$ solution in 100 mL volumetric flasks. Pour about 30 mL of both solutions in two separate 50 mL beakers. Dip Zn and Cu electrodes in $ZnSO_4$ and $CuSO_4$ solutions, respectively. Take a rolled filter paper and wet with deionized water follow by a few drops of KNO_3 solution. Place this wet filter paper in between these two beakers as a salt bridge. Connect both electrodes with the alligator clips in such a way that to get a positive voltage reading. Record the reading as $E°$.

Fill the supplied unknown $CuSO_4$ solution in a burette. Add 30 mL and 50 mL in two separate 100 mL volumetric flasks and dilute with deionized water to the mark. Repeat the procedure by taking 30 mL of diluted unknown $CuSO_4$ solution and measure the emf.

Observation:

The standard emf of constructed daniell cell, $E° = $ …….. V

Table 1:

S. No.	Concentration of ZnSO$_4$ solution (in M)	Volume of unknown CuSO$_4$ solution (in mL)	Emf (in V)		Concentration of diluted unknown CuSO$_4$ solution (in M)	Concentration of supplied CuSO$_4$ solution (in M)
1.	0.10	30	E_1		x_1	
2.	0.10	50	E_2		x_2	

Using equation (1), calculate the concentration of supplied CuSO$_4$ solution.

Result:

The concentration of supplied CuSO$_4$ solution is ………… M.

Experiment: 10

Determination of viscosity average molecular weight of a polymer sample by Viscometer.

Chemicals and glassware: Starch, 100 mL volumetric flask, 100 mL beaker, pipette, Ostwald viscometer, and stopwatch.

Theory:

Addition of even a very small amount of a polymer to a solvent of low viscosity causes a sharp increase in its viscosity. The magnitude of increase in viscosity depends in addition, to other factors such as concentration, shape and size of the solute molecules, on the molecular weight of the polymers.

The ratio of the coefficient of viscosity of the solution (η_s) to the coefficient of viscosity of the pure solvent (η_o) at the same temperature is known as viscosity ratio or relative viscosity (η_r) which can be written as:

$$\eta_r = \frac{\eta_s}{\eta_o} = \frac{\rho_s t_s}{\rho_0 t_o}$$

where, ρ_s and ρ_o are respective densities of polymer solution and pure solvent; t_s and t_0 are the corresponding efflux time. For a dilute polymer solution ($\approx$ 0.5 g/ 100 mL) the two densities can be taken as equal. Then the equation becomes:

$$\eta_r = \frac{t_s}{t_o}$$

$$\eta_{sp}(specific) = \frac{\eta_s - \eta_0}{\eta_o} = \frac{\eta_s}{\eta_o} - 1 = \eta_r - 1 = \frac{t_s}{t_o} - 1$$

The ratio of the specific viscosity of the solution to its concentration C, is called viscosity number or reduced specific viscosity.

$$\eta_{red} = \frac{\eta_{sp}}{C}$$

C = Concentration in g/ 100 mL

The plot of η_{red} *versus* C is a straight line is extrapolated to zero conc. (i.e., cut into the Y-axis, η_{red}). The intercept on the ordinate is the viscosity at zero conc. or the limiting viscosity and termed as limiting viscosity number or intrinsic viscosity, [η], which is related to molecular weight $\overline{Mv}$, by an empirical equation.

$$[\eta] = K \, \overline{Mv}^{\alpha}$$

where 'K' and 'α' are constants for a particular polymer/ solvent/ temperature system and can be obtained from the books.

Procedure:

Preparation of polymer solution: Weigh accurately 500 to 100 mg of the well dried powdered polymer (whatever concentration you need to prepare) and transfer to a 100 mL volumetric flask. Add 80−90 mL of the solvent (in which polymer is soluble) stopper the flask and suspend in a thermostatic water bath at 25 °C, shake occasionally to dissolve the sample and then fill the solvent up to the mark.

Measurement of flow time with Ostwald Viscometer: Wash the viscometer with chromic-sulphuric acid mixture and keep for drying in air oven. Pipette 20 mL of the solvent into the wide arm (reservoir R) and affix in a thermostatic water bath. Allow 5−10 minutes for attainment of temperature. Using a rubber ball, force air into the wide arm so as to raise the level of liquid into the capillary arm above the higher mark M_1. Then release the pressure and with a timer measure the time in which the liquid meniscus moves from the upper mark M_1 to the lower mark M_2 and use the flow time as t_0.
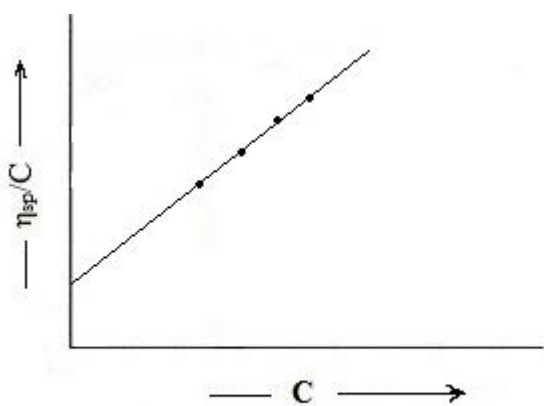
Plot for calculation of $[\eta]$
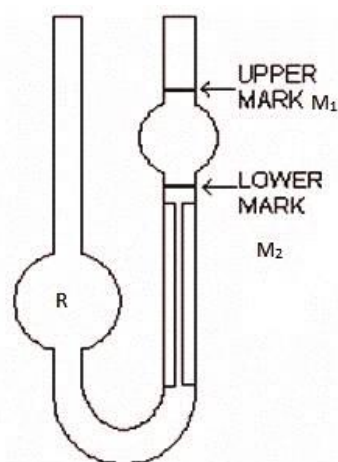
Ostwald Viscometer

Pour out the liquid from the wide arm as completely as possible. Clean the viscometer, dry it and fill it with 20−25 mL of the polymer solution and determine the flow time in the similar condition (t_s). Similarly determine the flow times for solutions of minimum three different concentrations.

Observation and Calculation:

Temperature of the experiment = …….. °C

Solvent used =

Values of constant for

Polymer / solvent / 25 °C   K   =

α   =

Volume of liquid used for the experiment   = …….. mL

Polymer solution Concentration, C (g/100 mL)	Flow time in seconds		Relative viscosity $\eta_r = \dfrac{t_s}{t_o}$	Specific viscosity $\eta_{sp} = \eta_r - 1$	Reduced viscosity $\eta_{red} = \dfrac{\eta_{sp}}{C} \times 100$
Pure solvent	t_0				
0.1	t_1				
0.2	t_2				
0.3	t_3				
0.4	t_4				
0.5	t_5				

$t_s = t_1, t_2, t_3, t_4,$ & t_5

Plot the graph between $\eta_{sp/C}$ *versus* Concentration and get the value of $[\eta]$ limiting viscosity.

Calculation of molecular weight:

(K and α values are given)

$$[\eta] = K \, \overline{Mv}^{\alpha}$$

$$\log [\eta] = \log K + {}^{\alpha}\log \overline{Mv}$$

$$\log \overline{Mv} = \frac{\log[\eta] - \log K}{\alpha}$$

$$\overline{Mv} = \text{antilog} \left[\frac{\log[\eta] - \log K}{\alpha} \right]$$

Result: The viscosity average molecular weight of the polymer is ………….

Experiment: 11

Synthesis of metal oxide nanoparticles by sol-gel process and its characterization by XRD.

Chemicals and glassware: Titanium (IV) isopropoxide, toluene, isopropanol, 100 mL round bottom flask, and silica crucible.

Introduction:

In materials science, the sol–gel process is a method for producing solid materials, mainly metal oxides from small molecules. The process involves the conversion of monomers into a colloidal solution (*sol*) that acts as the precursor for an integrated network (or *gel*) of either discrete particles or network polymers. Sol–gel process is used to produce metal oxide nanoparticles. Titanium dioxide (TiO$_2$) is one of the metal oxides that can be synthesized by the sol-gel method and it has been widely used in the fields including catalysis, photocatalysis, antibacterial agents, and as a pigment to provide whiteness and opacity to products such as paints, toothpastes, foods, plastics, papers, inks, supplements, coatings, medicines, etc. TiO$_2$ has three crystallographic polymorphs: anatase, rutile, and brookite.

Reactions:
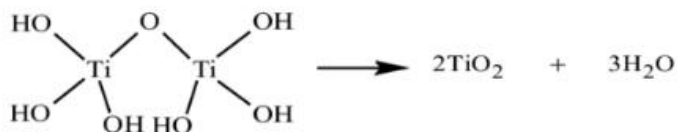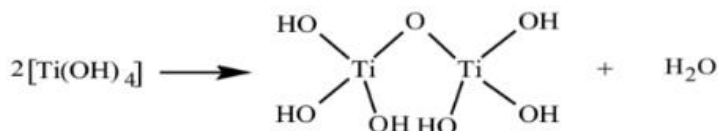
Overall reaction,
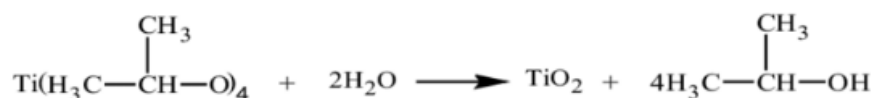
Procedure: Take 10 mL of Titanium (IV) isopropoxide in a 100 mL round bottom (R. B.) flask. Add 20 mL of dry Toluene followed by 4−5 mL of dry isopropanol into it. Stir the mixture for 10 min to give a clear solution. Place the R. B. flask into an ice bath, equipped with a magnetic stirrer. Then, add 1 drop of deionized water into the reaction mixture and stop the stirring as the reaction completes, which will be confirmed by observing the gel formation. Allow the gel to age for 1 h. Thereafter, dry the gel at 80 °C for 12 h in an oven and then, transfer the resulting powder to a silica crucible to be kept in a furnace and anneal at 450 °C for 2 h. Characterize and confirm the product by an X-ray diffraction (XRD) technique. Index the XRD patterns as below figure:
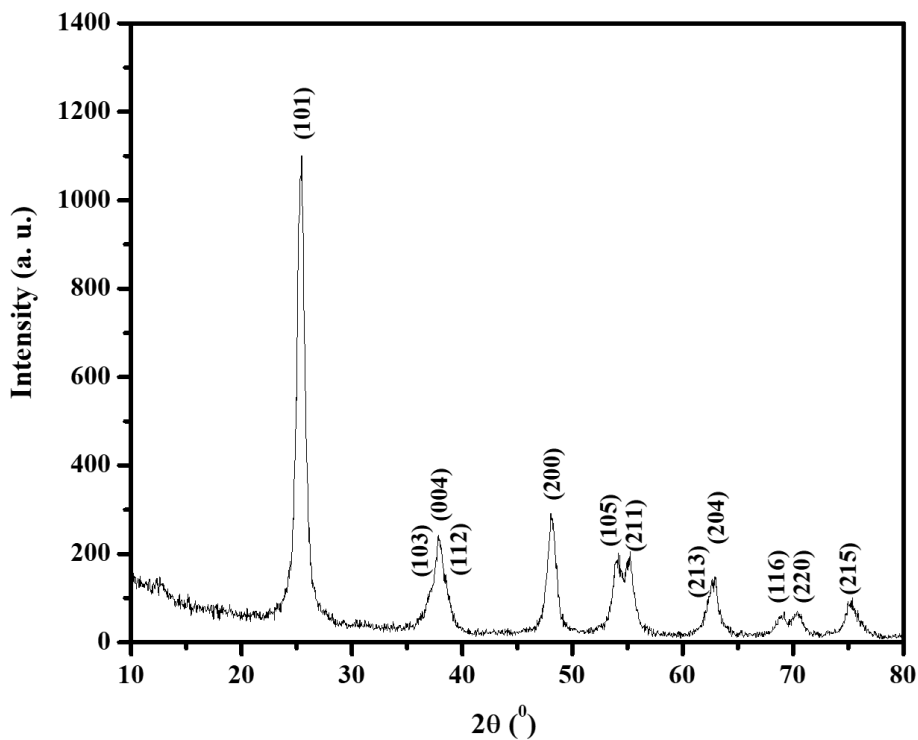
XRD profile of anatase phase, TiO$_2$

Result: ……………………………………