VIJAYAM INSTITUTE OF TECHNOLOGY

(Approved by AICTE, New Delhi & Affiliated to JNTUA, Anantapuramu)
Sundararajapuram (V), G.D.Nellore, Opp. Heritage Dairy, Puttur Road,
Chittoor Dist-517125, Andhra Pradesh
Website: www.vijayamit.com Contact: 08572-236328

CHEMISTRY LAB

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Experiment No: **Date:**

Determination of Strength of Sulphuric acid in Pb-Acid battery

Aim: To determine strength of Sulphuric acid present in the lead-acid battery, by titrating it against sodium hydroxide solution.

Apparatus:

Lead-acid battery, Burette, Pipette, Beakers, Volumetric flask, dropper, Measuring cylinder

Chemicals: Sulphuric acid (from battery), Sodium hydroxide solution, Phenolphthalein indicator, CO_2 free distilled water, Oxalic acid

Principle:

Lead- battery is frequently used in automobiles. A commercial Lead-Acid battery consists of 6 or 12 Lead-Acid cells. Anode is made up of a grid of lead plates coated with finely divided spongy Lead (Pb). Cathode is a grid of lead plates coated with red-brown lead oxide (PbO_2). These electrodes are kept in alternate positions, and are separated by insulating material. These are suspended in dil.H_2SO_4 which acts as an electrolyte. The basic electrochemical reaction (redox reaction) occurring in a single lead acid cell can be written as;

$$Pb + 2H_2SO_4 + PbO_2 \xrightarrow{\text{discharging}} 2\,PbSO_4 + 2\,H_2O$$

During the discharge, lead oxide (PbO_2) is converted to lead sulfate ($PbSO_4$) at the cathode. At the anode lead (Pb) is converted to lead sulfate ($PbSO_4$). This causes the sulfuric acid (H_2SO_4) in the electrolyte to be consumed.

Thus, during the discharge process, the strength of acid decreases due to consumption of H_2SO_4 followed by the formation of water.

To determine exact strength of acid at particular stage of charge, the sample of acid withdrawn from the cell can be titrated with CO_2-free sodium hydroxide solution.

$$H_2SO_4 + 2\,NaOH \rightarrow Na_2SO_4 + 2\,H_2O$$

Sulfuric acid reacts with sodium hydroxide on the 1:2 basis. That means number of moles of sulfuric acid is half that of number of moles of sodium hydroxide consumed. This reaction is monitored with phenolphthalein indicator to get the end point as pink.

Procedure:

Step 1: Standardization of NaOH solution

Rinse the burette with given NaOH solution, then fills it with the NaOH solution. Pipette out 20 mL of given oxalic acid solution into a conical flask. Add 2 drops of phenolphthalein indicator. Titrate the colourless solution with NaOH solution till pale pink colour is obtained as end point. Repeat the titration to get concordant titre values. Record the values in table (1). Then calculatethe exact normality of NaOH solution.

Table 1: Standardization of NaOH solution

S. No.	Volume of Oxalic acid (V_1 mL)	Burette readings (mL)		Volume of NaOH (V_2 mL)
		Initial	Final	
1.				
2.				
3.				
Concordant titre Value =				

$$N_1V_1 = N_2V_2$$

N_1 = Normality of Oxalic acid = 0.1 N

N_2 = Normality of NaOH solution = ?

V_1 = Vol. of Oxalic acid = 20.0 mL

V_2 = Vol. of NaOH solution =______mL

$$N_2 = (N_1V_1)/V_2$$

Normality of NaOH solution (N_2) =_____________N

Step 2: Sampling of H_2SO_4 from Lead acid battery

Put on eye protection and rubber gloves. It is recommended to disconnect the battery especially if on a high rate of charge/discharge. Remove vent cap. Carefully insert the dropper into cell, draw about 2 mL of acid into the dropper and avoid "bumping". Be careful the float is not flooded (too much acid) or sticking to the rubber bulb. Transfer it to a measuring jar. Using micropipette draw exactly 1.0 mL of Sulphuric acid from the measuring jar and then transfer it to a 100 mL clean volumetric flask. Then make up the volumetric flask with distilled water to get 100 mL of acid solution.

Step 3: Determination of strength of Sulphuric acid

Fill burette with the given NaOH solution. Pipette out 20 mL of Sulphuric acid solution (prepared in step 2) into a conical flask.

(Note: Don't forget to use rubber bulb while pipetting out the acid solution)

Add 2 drops of phenolphthalein indicator. Titrate the colourless solution with NaOH solution till pale pink colour is obtained as end point. Repeat the titration to get concordant titre values. Record the values in table (2). Then calculate the exact normality of Sulphuric acid present in lead acid battery.

Table 2: Determination of strength of Sulphuric acid

S. No.	Volume of Sulphuric acid (V_3 mL)	Burette readings (mL)		Volume of NaOH ($V_{2'}$ mL)
		Initial	Final	
1.				
2.				
3.				
Concordant titre Value =				

$$N_3V_3 = N_2'V_2'$$

N_3 = Normality of Sulphuric acid = ? N_2' = Normality of NaOH solution =___N

V_3 = Vol. of Sulphuric acid = 20.0 mL V_2' = Vol. of NaOH solution =________mL

$$N_3 = (N_2'V_2')/V_3$$

Normality of Sulphuric acid solution (prepared in step 2) =N_3 =________________N

Normality of Sulphuric acid present in lead acid battery = $N_3 \times 100$

Result:

Normality of Sulphuric acid present in lead acid battery = ____________ **N**

Experiment No: **Date:**

Conductometric Titration of Strong Acid vs. Strong Base

Aim: To estimate the strength of acid (HCl) by conductometric method using standard NaOH (1N) solution.

Apparatus: Conductivity meter, Conductivity cell, Micro-burette, Pipette, Beakers, Glass rod, Burette stand

Chemicals: Standard Sodium hydroxide, Hydrochloric acid, Distilled water

Principle:

Conductometric titration is the volumetric analysis, based upon the measurement of the conductance during the course of titration. The number of free ions, charge on the free ions and mobility of the ions affect the conductance of an aqueous solution. When one electrolyte is added to another electrolyte, the change in number of free ions causes a change in the conductance.

When a strong acid, say HCl, is titrated with a strong base NaOH, the following reaction takes place.

$$HCl + NaOH \rightarrow NaCl + H_2O$$

Before the addition of NaOH solution, the acid solution has high conductivity due to the highly mobile H^+ ions. When NaOH is added to the acid, H^+ ions of the acid are neutralized with OH^- ions of the base. Thus, the highly conducting H^+ ions in the solution are replaced by low conducting Na^+ ions, consequently the conductance will be progressively decreased. However, when excess base is added, due to the presence of more labile OH^- ions, the conductance will start increasing. Thus, at the equivalence point the conductance will be minimum.

The equivalence point may be located graphically by plotting the change in conductance as a function of the volume of titrant added.

Procedure:

1. The conductivity cell washed well with conductivity water and dipped in a 100 mL beaker.
2. The burette is rinsed and filled with standard 1N sodium hydroxide solution.
3. Add 20 mL of the given acid and 20 mL of distilled water in the 100 mL beaker.

4. The cell is fixed to the conductivity meter. Note the conductance of HCl solution.

5. For each 0.2 mL addition of sodium hydroxide, the solution is stirred well with glass rod and conductance of the solution is noted in table.

6. Continue the titration, take at least 5-10 readings after the end point where the conductance increases.

7. For each value of observed conductance **C**, corresponding value of corrected conductance **C'** is calculated by applying volume correction which is given by;

$$C' = \frac{(V + U)\,C}{V}$$

Where; V = Volume of HCl solution used in this experiment = 20 mL

U = Volume of NaOH added.

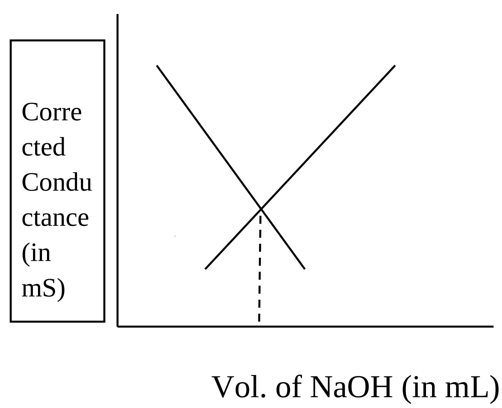
8. The corrected conductance values are plotted against volume of NaOH added.

9. From the curve obtained, the two lines are extrapolated and the end point is the intersection of two straight lines is found.

10. The point of intersection gives the volume of NaOH required for the complete neutralization of the given HCl.

11. Using the volume of NaOH corresponding to the end point, the strength of HCl can be calculated.

Model graph



Observation and Calculations:

S. No.	Volume of NaOH added (U in mL)	Measured Conductance **C** (in mS)	Corrected conductance **C'** = $\frac{C (V+U)}{V}$ (in mS)

$$N_1V_1 = N_2V_2$$

N_1 = Normality of NaOH = 1 N

N_2 = Normality of HCl solution = ?

V_1 = Vol. of NaOH (from graph) = ___mL

V_2 = Vol. of HCl solution = 20.0 mL

$$N_2 = (N_1V_1)/V_2$$

Result:

The strength of given HCl solution (N_2) =__________**N**

Experiment No: **Date:**

Conductometric Titration of Weak Acid vs. Strong Base

Aim: To estimate the strength of acid (CH_3COOH) by conductometric method using standard NaOH (1N) solution.

Apparatus: Conductivity meter, Conductivity cell, Micro-burette, Pipette, Beakers, Glass rod, Burette stand

Chemicals: Standard Sodium hydroxide, Acetic acid, Distilled water

Principle:

Conductometric titration is the volumetric analysis, based upon the measurement of the conductance during the course of titration. The number of free ions, charge on the free ions and mobility of the ions affect the conductance of an aqueous solution. When one electrolyte is added to another electrolyte, the change in number of free ions causes a change in the conductance.

When a weak acid, say CH_3COOH, is titrated with a strong base NaOH, the following reaction takes place.

$$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$$

Initially the conductance is low due to the feeble ionization of acetic acid. On the addition of base, there is decrease in conductance not only due to the replacement of H^+ by Na^+ but also suppresses the dissociation of acetic acid due to common ion acetate. But very soon, the conductance increases on adding NaOH, as NaOH neutralizes the un-dissociated CH_3COOH to CH_3COONa which is the strong electrolyte. This increase in conductance continues raise up to the equivalence point. Beyond the equivalence point, conductance increases more rapidly with the addition of NaOH due to the highly conducting OH^- ions. The equivalence point may be located graphically by plotting the change in conductance as a function of the volume of titrant added.

Procedure:

The conductivity cell washed well with conductivity water and dipped in a 100 mL beaker. The burette is rinsed and filled with standard 1N sodium hydroxide solution. Add 20 mL of the given acid and 20 mL of distilled water in the 100 mL beaker. The cell is fixed to the conductivity meter. Note the conductance of CH_3COOH solution. For each 0.2 mL addition of sodium hydroxide, the solution is stirred well with glass rod and conductance of the solution is noted in table. Continue the titration, take at least 5-10 readings after the end point where the conductance increases.For each value of observed conductance **C**, corresponding value of corrected conductance **C'** is calculated by applying volume correction which is given by;

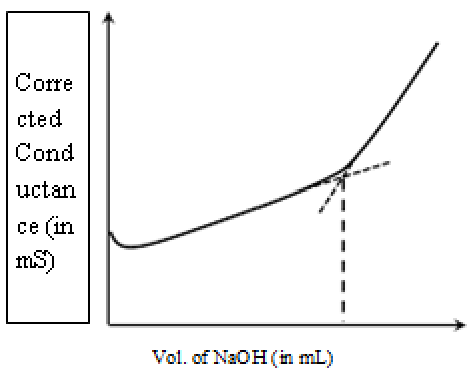
$$\mathbf{C'} = \frac{(V + U)\,C}{V}$$

Where; V = Vol. of CH_3COOH solution used in this experiment = 20 mL

U = Vol. of NaOH added.

The corrected conductance values are plotted against volume of NaOH added. From the curve obtained, the two lines are extrapolated and the end point is the intersection of two straight lines is found. The point of intersection gives the volume of NaOH required for the complete neutralization of the given CH_3COOH. Using the volume of NaOH corresponding to the end point, the strength of CH_3COOH can be calculated.

Model graph



Observation and Calculations:

S. No.	Volume of NaOH added (U in mL)	Measured Conductance **C** (in mS)	Corrected conductance **C'** = $\frac{C\,(V+U)}{V}$ (in mS)

$$N_1V_1 = N_2V_2$$

N_1 = Normality of NaOH = 1 N

V_1 = Vol. of NaOH (from graph) = ___mL

N_2 = Normality of CH_3COOH = ?

V_2 = Vol. of CH_3COOH = 20.0 mL

$$N_2 = (N_1V_1)/V_2$$

Result:

The strength of given CH_3COOH solution (N_2) =__________**N**

Experiment No: **Date:**

Determination of Cell Constant

Aim: To determine the Cell constant of a conductivity cell.

Apparatus: Conductivity cell, beaker, standard flask

Chemicals: Potassium Chloride

Principle:

Cell constant for a cell is defined as the constant factor which stands for the ratio of the specific conductance of a solution and its measured conductance in the cell.

Cell constant = Specific conductance / Measured conductance

The specific conductance of a solution is the conductance of a solution contained between two parallel electrodes of 1 cm^2 cross section and which are kept apart of 1 cm.

Now a days the value of cell constant is read directly from the instrument. Practically it can be calculated from a solution of KCl of known concentration. Table: Determination of cell constant

S. No.	Conc. of KCl solution (N)	Observed Conductance (Ohm-1)	Specific Conductance (Ohm-1 cm-1)	Cell constant (cm-1)
1	0.1		12.86×10^{-3}	
2	0.02		2.61×10^{-3}	
			Average cell constant =	**cm-1**

Procedure: Determination of cell constant

Collect the 0.1 N KCl solution (stock solution). To prepare 0.02 N KCl solution, transfer 20 mL of the provided stock solution into a 100 mL standard flask and dilute it to the mark by adding distilled water. Record the conductance of the 0.1 N and 0.02 N KCl solutions using a conductivity meter and calculate the cell constant.

Result:

The cell constant of the given conductivity cell is = cm^{-1}

Experiment No: **Date:**

Potentiometric estimation of Ferrous using Potassium Dichromate

Aim: To estimate the amount of iron using standard solution of potassium dichromate by potentiometric method.

Apparatus: Pipette, Burette, Standard flask, Potentiometer, Beakers, Measuring jar etc.

Chemicals: Potassium dichromate, Ferrous ammonium sulphate, Dil. H_2SO_4.

Principle:

Potentiometer can be used to follow redox titration. In this, a solution of oxidizing agent namely $K_2Cr_2O_7$ is added to ferrous salt solution. In an acid solution, the orange dichromate ion which is a powerful oxidant is reduced to chromic ion, Cr^{3+} and also it will oxidize Fe^{2+} to Fe^{3+} quantitatively.

$$K_2Cr_2O_7 + 6\ FeSO_4 + 7\ H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3\ Fe_2(SO_4)_3 + 7\ H_2O$$

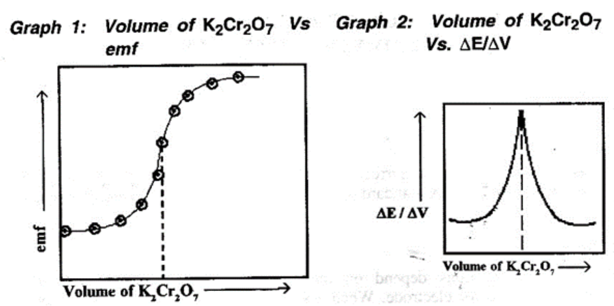
Since dichromate ion is orange in color, Cr^{+3} is pale green, the distinct color change can be used to determine the end point. However, the end point can be more accurately determined by potentiometric measurement. A sharp increase in potential indicates the end point.

Procedure: **Step 1: Pilot titration**

Pipette out 20 mL of ferrous solution from the volumetric flask into a clean 100 mL beaker. Add 20 mL of diluted H_2SO_4 solution to it. Immerse the platinum electrode and calomel electrode in ferrous solution, and connect them to the potentiometer. Fill a burette with standard solution of potassium dichromate. Then, add one mL of the $K_2Cr_2O_7$ solution each time from the burette carefully into the ferrous solution. In each addition of $K_2Cr_2O_7$ solution, stir the contents of the beaker with a glass rod, and note down the corresponding emf. values in table (1). At first, the emf of the cell will be low. By the gradual addition of $K_2Cr_2O_7$ to ferrous solution, emf of cell will increase and leads to sudden rise. Note down the sudden rise and complete the titration by taking at least five values of emf after sudden rise. Draw a graph by taking volume of $K_2Cr_2O_7$ along X-axis and emf along Y-axis. From this graph determine the approximate equivalence point.

Table 1: Pilot titration

S. No.	Volume of $K_2Cr_2O_7$ (mL)	EMF (mV)
1	0	
2	1	
3	2	
4	3	
5	4	
6	5	
7	6	
8	7	
9	8	
10	9	
11	10	
12	11	
13	12	
14	13	
15	14	



Step 2: Fair titration

To find exact equivalence point, do the same titration once again, by repeating above procedure, in the vicinity of the volume where the sudden rise occurs. In this fair titration add 0.2 mL of the $K_2Cr_2O_7$ solution each time from the burette into the ferrous solution. Note down the corresponding e.m.f. values in table (2).

Draw a graph between average volume of $K_2Cr_2O_7$ solution along X-axis and $\Delta E/\Delta V$ along Y-axis. In this curve the equivalence point is indicated by the maximum. From this end point volume of $K_2Cr_2O_7$ solution, calculate the amount of iron (II) present in the given solution.

Table 2: Fair titration

S. No.	Vol. of $K_2Cr_2O_7$ (mL)	EMF (mV)	ΔE	ΔV	$\frac{\Delta E}{\Delta V}$

$$N_1V_1 = N_2V_2$$

N_1=Normality of $K_2Cr_2O_7$ = 0.2N N_2= Normality of iron (II) solution = ?

V_1=Vol. of $K_2Cr_2O_7$ solution =_______mL V_2=Vol. of iron (II) solution = 20.0 mL

$$N_2 = (N_1V_1) / V_2$$

Normality of iron (II) solution (N_2) =_________N

Amount of iron (II) present in the 100 mL solution

$$= \frac{N_2 \times \text{Eq. wt. of Fe} \times 100}{1000} \text{ g}$$

=____________ g

Result:

The amount of iron (II) present in given solution =____________________**g**

Experiment No: **Date:**

Estimation of Ferrous Iron by Dichrometry

Aim: To estimate the amount of iron (II) in the given solution using standard solution of potassium dichromate.

Apparatus: Pipette, Burette, Standard flask, Conical flask, Funnel, Beakers, Measuring jar etc.

Chemicals: Potassium dichromate, Diphenylamine indicator, Ferrous ammonium sulphate, Ortho-phosphoric acid and Concentrated Sulphuric acid.

Principle:

Ferrous ions are oxidized to ferric ions by dichromate ions in acidic solution. The completion of the oxidation reaction is marked by the appearance of blue violet colour of the diphenylamine, which is used as an internal indicator.

$$K_2Cr_2O_7 + 6\ FeSO_4 + 7\ H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3\ Fe_2(SO_4)_3 + 7\ H_2O$$

Once all the ferrous ions are oxidized by dichromate ions in the solution, then diphenylamine (indicator) undergoes oxidation to form diphenyl benzidine which is blue violet in colour. The equivalent weight of iron is its atomic weight i.e. 55.86 since one equivalent of potassium dichromate oxidizes one equivalent of iron.

Procedure:

Estimation of Iron (II)

Rinse the burette with given $K_2Cr_2O_7$ solution and then fill it with the same $K_2Cr_2O_7$ solution up to zero mark. Make the given ferrous solution up to the mark in a 100 mL standard flask with distilled water and shake the standard flask well for uniform concentration. Pipette out 20 mL of the above ferrous solution into a clean conical flask, add 5 mL of acid mixture (1:3 ratio of phosphoric acid and sulphuric acid) and 2 drops of diphenylamine indicator. Titrate the resulting solution with $K_2Cr_2O_7$ solution taken in the burette until blue violet colour is obtained as end point. Repeat the titration to get concordant titre values. Record all the titre values in table. Then calculate the normality of iron solution. Use it to calculate amount of iron.

Table: Estimation of Iron (II)

S. No.	Volume of Iron (II) solution (V_2 mL)	Burette readings (in mL)		Volume of $K_2Cr_2O_7$ solution (V_1 mL)
		Initial	Final	
Concordant titre Value =				

$$N_1V_1 = N_2V_2$$

N_1=Normality of $K_2Cr_2O_7$ = 0.02N N_2= Normality of iron (II) solution = ?

V_1=Vol. of $K_2Cr_2O_7$ solution =______mL V_2=Vol. of iron (II) solution = 20.0 mL

$$N_2 = (N_1V_1) / V_2$$

Normality of iron (II) solution (N_2) =________N

Amount of iron (II) present in the 100 mL solution

$$= \frac{N_2 \times \text{Eq. wt. of Fe} \times 100}{1000} \text{ g}$$

=_____ g

Result:

The amount of iron (II) present in given solution =___________________**g**

EXPERIMENT NO: **DATE:**

PREPARATION OF BAKELITE

Aim: Synthesis of Phenol-Formaldehyde Resin/Bakelite through Condensation Polymerization.

Apparatus: Conical flask, Beaker, Wash bottle, and a Burette.

Chemicals: Formaldehyde, glacial acetic acid, Phenol, Conc. H_2SO_4

Principle: Bakelite (also known as phenol-formaldehyde resin) is a thermosetting polymer formed due to condensation polymerization between phenol and formaldehyde. It hardens upon heating due to crosslinking. The condensation reaction occurs in a controlled acidic or basic environment produces ortho- and para-hydroxymethyl phenol and their derivatives.

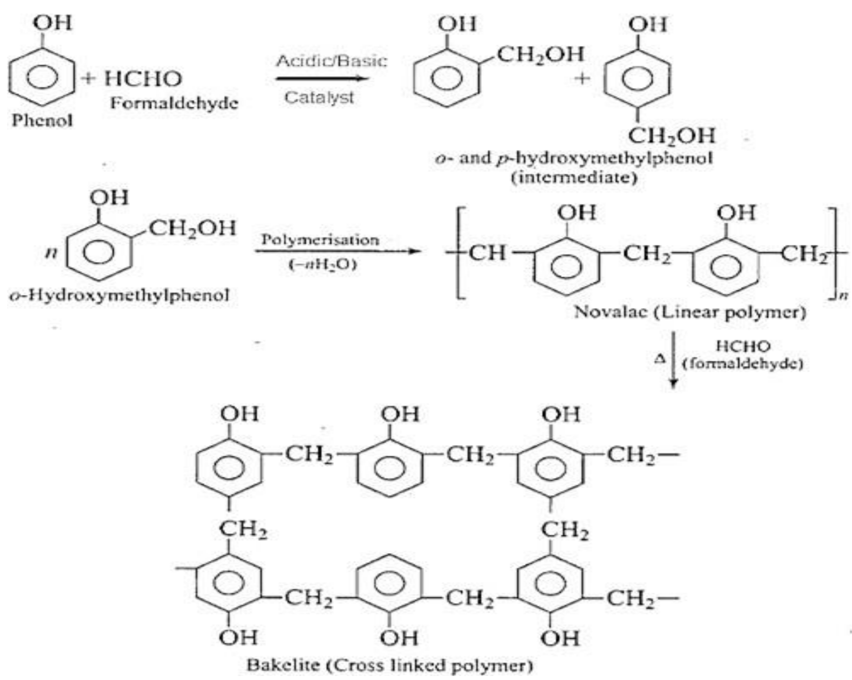
The preparation of phenol-formaldehyde resin involves two steps as follows:

1. Formation of ortho/para-(hydroxymethyl)phenol derivative

Initially the monomers combine to form ortho/para-(hydroxymethyl)phenol derivative depending upon phenol to formaldehyde ratio.

2. Formation of Novolac & Bakelite

The phenol formaldehyde derivatives react among themselves or with phenol to give a linear polymer called Novolac which upon heating to form a higher cross-linked polymer known as Bakelite.



Procedure:

2.5 mL of formaldehyde solution is added using a measuring cylinder to a 250 mL beaker containing 5 mL of glacial acetic acid. Carefully add 2 mL of Phenol filled in the burette to the reaction vessel and finally 1 mL of Conc. H_2SO_4 is added to the reaction mixture. After stirring, the solution is heated until pink colour appears. Cool the mixture and shake vigorously until a white resin is seen. The residue obtained should be subjected to filtration, followed by multiple rinses with de-ionized water. Finally, dry and weigh the resulting product.

Result: The weight of the product obtained = g

Experiment No: **Date:**

Verification of Lamberts-Beer's Law

<u>Aim:</u> To verify Lambert-Beer's law for $KMnO_4$ colorimetrically

<u>Apparatus:</u> Colorimeter, cuvette, test tubes, Burettes or graduated cylinders

<u>Chemicals:</u> $KMnO_4$, Distilled water

<u>Principle:</u> The Beer-Lambert law states that the absorbance of a solution is directly proportional to the concentration of the absorbing species in the solution and the path length.

Mathematically, the law can be written in the following form,

$$\mathbf{A} = \varepsilon\, C\, l$$

Where,

A = absorbance

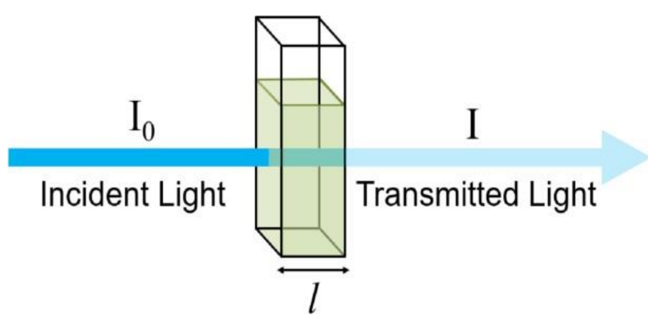
ε = molar absorptivity (constant)

C = Concentration of the sample solution

L = Path length of the cell

The absorbance changes with concentration, A higher concentration of the colored solution absorbs more light (and transmits less) than a solution of lower concentration.

Mathematically absorbance can be given as,



$$\mathbf{A} = \log \frac{I_0}{I}$$

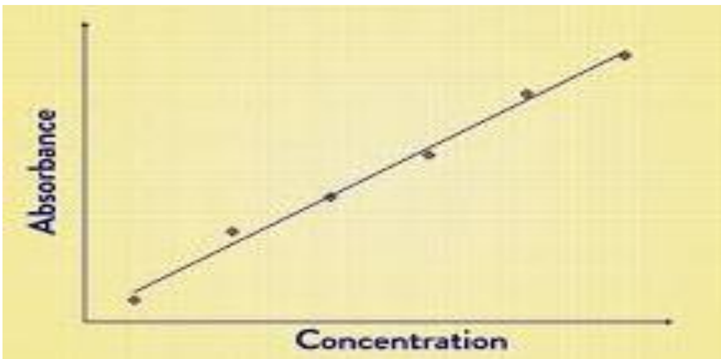
Here, Io and I are the incident and transmitted intensities of light

<u>Procedure:</u>

Take small volumes 0.01M $KMnO_4$ solution and distilled water in separate beakers; fill in the separate graduated burettes. Label five clean, dry, test tubes 1–5. Use Burettes to prepare five standard solutions according to the volumes listed in the table (Concentration can be calculate by $M_1V_1 = M_2V_2$). Thoroughly mix each solution.

- ✓ Switch on the instrument; wait for 15 minutes.
- ✓ In the instrument one can select the absorbance or % transmittance and wavelength range of interest.
- ✓ Take clean cuvette and fill with distil water as blank for calibration.
- ✓ Now record the absorbance value with aqueous $KMnO_4$ solution with lowest concentration.
- ✓ Repeat the procedure for Test Tubes 2 to 5, starting from the lowest concentrations to next higher concentrations of $KMnO_4$. Every time one should rinse the cuvette taking a small portion of the solution to be analyzed next.
- ✓ Plot a curve between Absorbance v/s concentrations. Check whether it is a liner plot or not.

Model graph



Observation and Calculations:

Test Tube No.	0.01 M $KMnO_4$ (mL)	Distill Water (mL)	Concentration (M)	Absorbance
1	2	8	0.002	
2	4	6	0.004	
3	6	4	0.006	
4	8	2	0.008	
5	10	0	0.01	

Result: A linear curve is obtained between Absorbance v/s concentrations that prove the existence of Lamberts-Beer's law.