

# RV COLLEGE OF ENGINEERING <sup>®</sup>

*(Autonomous Institution affiliated to VTU, Belagavi)*

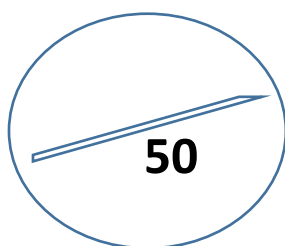
BENGALURU – 560 059



## Department of Chemistry

### Certificate

*This is to certify that \_\_\_\_\_,  
USN \_\_\_\_\_ has satisfactorily completed the experiments in Engineering  
Chemistry for \_\_\_\_\_ Engineering Under Graduate programme  
during the academic year 20 - 20*



*Signature of Head of the Department*

*Signature of Faculty in-charge*

**LAB SEAT No:**

**PERSONAL DETAILS**

<b>Name:</b>	
<b>Branch:</b>	
<b>Section:</b>	
<b>USN No:</b>	
<b>Roll No:</b>	
<b>Semester:</b>	
<b>Phone No:</b>	
<b>College E-mail ID:</b>	
<b>Signature of the Student:</b>	

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*"The meeting of two personalities is like the contact of two chemical substances: if there is any reaction, both are transformed."*

*-Carl Jung*

## INDEX PAGE - ENGINEERING CHEMISTRY PRACTICE

<b>Name :</b>		<b>USN:</b>				
Expt . No.	Title of Experiments	Components				Total marks
		Date of Conduction	MP & Cal	Conduction	I & SR	
1	Volumetric Analysis					
2	Estimation of percentage copper from PCB.					
3	Determination of total acidity of the soft drinks using pH sensors.					
4	Potentiometric estimation of Iron.					
5	Conductometric estimation of strong acid vs strong base					
6	Conductometric estimation of mixture of acids.					
7	Flame photometric estimation of sodium.					
8	Colorimetric estimation of copper from E-waste.					
9	Determination of viscosity coefficient of a given liquid using Ostwald's viscometer.					
<b>Signature of Lab Teachers</b>		<b>Total Marks:</b>  <b>Lab Internal Marks:</b>				

**MP & Cal** – Model Procedure, Graph & Calculation,

**I & SR** – Inference & Societal Relevance

## LABORATORY SAFETY RULES AND GENERAL INSTRUCTIONS

- **Follow Dress Code**

Follow the dress code of the college. Wear white lab coat and shoes while in the laboratory. Take Proper care while performing the experiments.

- **Don't Taste or Sniff Chemicals**

Don't smell or taste chemicals as they may harm you.  
Use fuming chemicals in the fuming hood.

- **Don't Dispose the Chemicals Down the Drain**

Don't dispose all chemicals into the sink.  
Follow the directions of the instructor for chemicals which cannot be put into the sink.  
Flush the sink with water after completing the experiment.

- **Don't Eat or Drink in Laboratory**

Don't eat or drink inside the laboratory.

- **Collect the Data During Lab experiments**

Directly record the readings in the work book using pen and get them attested by the teacher

- **Report all accidents and near-miss incidents to the instructor, immediately.**

To avoid accidents, follow the guidelines below

1. Note the location of safety equipment in the lab (e.g. first aid kits, fire extinguishers).
2. Always read the upcoming experiments carefully and thoroughly. Understand the procedure, toxicity of the chemicals before entering the lab and follow the instructions.
3. Perform authorized experiments. Know what you are supposed to be doing and ask if you are not sure.
4. Keep your work area clean and notify the instructor if there is a spill or breakage. Clean up any spills as per the advice of the instructor.
5. Before you leave the lab, check that your work area is clean and that all utilities are shutoff.
6. Wash your hands with soap before you leave the laboratory.

## General instructions to be followed while carrying out chemical methods of analysis

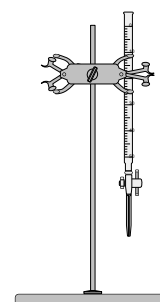
### Pipette:

1. Wash the Pipette minimum three times with tap water.
2. Rinse it once with small quantity of distilled water.
3. Now rinse the pipette with about  $10\text{ cm}^3$  of the solution to be pipetted out and reject the solution to the sink.
4. Draw the solution into the pipette a little above the mark by holding it almost vertically. Close the upper end of it using INDEX FINGER only. Adjust the lower meniscus of the liquid to the mark by rotating the pipette using other fingers and holding the pipette vertical.
5. While transferring the solution from the pipette to the conical flask, hold the pipette vertically till the solution drains out completely. Now touch the tip of the pipette 2-3 times gently to the empty bottom of the conical flask.
6. While transferring the solution do not blow air through the pipette.
7. Now rinse the pipette with about  $10\text{ cm}^3$  of the solution to be pipetted out and reject the solution to the sink.
8. Draw the solution into the pipette a little above the mark by holding it almost vertically. Close the upper end of it using **INDEX FINGER** only. Adjust the lower meniscus of the liquid to the mark by rotating the pipette using other fingers and holding the pipette vertical.
9. While transferring the solution from the pipette to the conical flask, hold the pipette vertically till the solution drains out completely. Now touch the tip of the pipette 2-3 times gently to the empty bottom of the conical flask.
10. While transferring the solution do not blow air through the pipette.



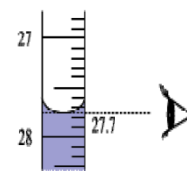
### Burette:

1. Wash the burette minimum three times with tap water.
2. Rinse it once with small quantity of distilled water.
3. Now rinse it once with about  $10\text{ cm}^3$  of the solution to be filled and reject the solution.
4. Fill the burette beyond zero mark using a funnel and adjust the meniscus of the solution to zero mark by running down the solution



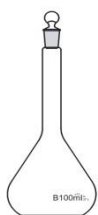
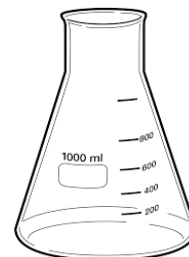
through the nozzle (see that the nozzle part of the burette is completely filled with the solution)

5. Hold the burette vertically and the meniscus to your eye level, while reading the burette.



#### Conical Flask:

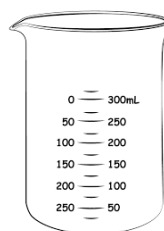
1. Wash the conical flask minimum three times with tap water.
2. Rinse it once with small quantity of distilled water.
3. Do not rinse it with any of the solutions.
4. Reject the contents of the conical flask to the sink after every trial and wash it as mentioned in 1 & 2 (under burette).



**Standard Flask or  
Volumetric Flask**



**Wash Bottle**



**Beaker**



**Measuring Cylinder**

#### Cleaning of apparatus:

- Clean the electrodes and other glassware after the experiment
- Keep the electrodes in distilled water after use.
- Do not spill any chemical or water on the instruments. If happens, immediately dry it using tissue paper.

## EXPERIMENT NO – 1

**Aim:** Volumetric analysis

**Some terms used in volumetric analysis**

**Titrant:** The standard solution of accurately known strength.

**Titrate:** The solution containing the substance whose concentration/strength is to be determined.

**Equivalence point:** The exact or theoretical point at which the reaction is complete.

**End-point:** The point, at which a physical change is observed during titration.

**Indicator:** The substance used for visual detection (by colour change) of completion of reaction at the end-point.

**Types of Indicators:** The following types of indicators are used in volumetric analysis:

- **Acid-base Indicators:** Indicators which are sensitive to pH.
- **Redox Indicators:** Indicators whose oxidised form has a colour different from that of the reduced form.
- **Precipitation Indicators:** Indicators which indicates the endpoint by forming coloured precipitates.
- **Complexometric Indicators:** Indicators which undergo a change in colour on forming complex with metal ions.

**Strength of solution:** The concentration of solution or the amount of substance dissolved in  $1 \text{ dm}^3$  of solution. It is usually expressed in terms of normality, molarity etc.

**Normality (N):** The number of gram equivalent weight of a substance dissolved in  $1 \text{ dm}^3$  of solution.

**Mass of the substance (in  $1 \text{ dm}^3$ ) = Normality \* Gram equivalent weight of the substance**

**Molarity (M):** The number of moles of solute present in  $1 \text{ dm}^3$  of solution.

**Mass of the substance (in  $1 \text{ dm}^3$ ) = Molarity \* Gram equivalent weight of the substance**

**Molality** = Number of moles of solute present in 1 kg of a solvent.

**Parts per million (ppm):** It denotes amount of a substance per 1,000,000 or  $10^6$  parts of solution regardless of the units of measure used as long as they are same.

**Equivalent weight:** The number of parts by weight of the substance which combines with or displaces 1 part by weight of Hydrogen, 8 parts by weight of Oxygen or 35.5 parts by weight of Chlorine.

**Equivalent weight of an acid:** The ratio of molecular weight of acid to basicity of acid or the ratio of molecular weight of acid to the number of Hydrogen atoms replaced in one molecule of acid during the reaction.

**Equivalent weight of a base:** The ratio of molecular weight of base to acidity of base or the ratio of molecular weight of base to the number of  $\text{OH}^-$  groups replaced in one molecule of base during the reaction.



**Equivalent weight of an oxidizing agent or reducing agent:** The ratio of molecular weight of the substance to the number of electrons gained or lost by one molecule during a redox reaction.

**Standard solution:** The solution whose concentration is known accurately.

#### Primary standard solution

A substance which is available in highly pure state, neither hygroscopic nor efflorescent and is stable under the conditions of storage is called primary standard substance. A solution prepared using a primary standard is called primary standard solution.

Example:  $K_2Cr_2O_7$ , Oxalic acid,  $AgNO_3$ , KCl, anhydrous Sodium Carbonate, PHP, etc.

#### Secondary standard solution

A substance which is not available in highly pure state, is hygroscopic or efflorescent and is not stable under the conditions of storage is called secondary standard substance. A solution prepared using a secondary standard is called secondary standard solution.

During the preparation of secondary standard solution an approximate weight of such a substance is dissolved in water and then the strength of this solution is determined by titrating against a primary standard solution this is known as standardization.

Example: NaOH,  $KMnO_4$ , HCl etc.

Chemistry is the scientific study of the properties and behavior of matter. It is a natural science that covers the elements that make up matter to the compounds composed of atoms, molecules, and ions: their composition, structure, properties, behavior, and the changes they undergo during a reaction with other substances. The Nobel Prize in Chemistry has been awarded 114 times to 191 Nobel Prize laureates between 1901 and 2022. This means that a total of 189 individuals have received the Nobel Prize in Chemistry, below is list of Nobel laureates in chemistry 2020, 2021 & 2022.

#### **The Nobel Prize in Chemistry**

**2022-**Carolyn R. Bertozzi, Morten Meldal and K. Barry Sharpless “*for the development of click chemistry and bioorthogonal chemistry*”

**2021-**Benjamin List and David MacMillan “*for the development of asymmetric organocatalysis*”

**2020-** Emmanuelle Charpentier and Jennifer A. Doudna “*for the development of a method for genome editing*”

## EXPERIMENT NO – 1

### Aim: Estimation of percentage of Copper from Printed Circuit Board (PCB)

The consumption of electronic products has grown considerably in the last decades. These products become obsolete in a short period of time, generating electronic waste, which presents loads of materials harmful to health and metals of great value to industries. In this work, an innovative metal concentration technique for PCBs was applied aiming at the valuable metals recovery from ground printed circuit boards (PCBs) of computers that would be discarded.

### Theory:

The substrate most commonly used in printed circuit boards is a glass fiber reinforced (fiberglass) epoxy resin with a copper foil bonded on to one or both sides. PCBs made from paper reinforced phenolic resin with a bonded copper foil are less expensive and are often used in household electrical devices. There are three main different types of PCB materials. Metals like copper, aluminum, and iron are generally used as the conductive layer in PCBs. Printed circuit boards (PCBs) are essential parts of the great majority of electric and electronic devices, which can contain more than 18% of Copper. Printed circuit board recycling has become an important area within the PCB industry in the last few years. The reason for this is simple – printed circuit boards are considered as hazardous waste, as they are manufactured with harsh chemicals and various materials that can harm the environment. Rather than adding to electronic waste, many organizations and manufacturers find it a better option to recycle PCBs.

A solution of copper is made by dissolution of the sample in Nitric acid. Oxides of Nitrogen are destroyed by boiling with Urea. Excess of acid is neutralized by adding Ammonia. The solution is changed to weak acidic medium by adding Acetic acid. Potassium Iodide is added. Iodine is liberated by the cupric ions. Then the solution is titrated against Sodium thiosulphate solution using starch as an indicator. The amount of Sodium-thiosulphate consumed is a measure of the amount of Copper present.



### Description:

#### Part-A: Preparation of copper solution

Weigh exactly the given sample of copper into a clean 250 cm<sup>3</sup> conical flask. Add Conc. Nitric acid drop by drop until the copper dissolves completely. Add 2 tt of distilled water and about 1 g of Urea. Boil for about 1 minute to destroy oxides of Nitrogen. Cool the mixture.

#### Part-B: Estimation of Copper .

Add 1 tt of distilled water to the solution obtained in part-A. Add Ammonium Hydroxide drop by drop until a pale blue precipitate is obtained. Dissolve the precipitate by adding dil. Acetic acid drop by drop, followed by ¼ tt of Acetic acid and 1 tt of 10% KI solution. Add a test tube of distilled water. Swirl the conical flask thoroughly for about 30 seconds. Titrate the liberated iodine against standard Sodiumthiosulphate solution taken in the burette until the solution becomes pale yellow or cream colored. Add about 2 cm<sup>3</sup> of freshly prepared starch solution as an indicator. Continue the titration by adding Sodiumthiosulphate solution **strictly drop by drop** until the dark blue coloration disappears, leaving behind a white ppt.

Repeat Part-A and B to conduct a duplicate. Calculate the percentage of copper present in the Brass samples

**Part-C: Calculation**

Normality of sodium thiosulphate = 'Y'
Volume of sodium thiosulphate = 'X' cm <sup>3</sup>
1000 cm <sup>3</sup> 1N sodium thiosulphate = 63.54g of copper
Therefore 'X' cm <sup>3</sup> of 'Y' N sodium thiosulphate = $\frac{63.54 \times X \times Y}{1000}$ g of copper
= 'A' g of Cu

**Model Procedure:****Model Calculation:****Observation and Calculation:**

**Part-A: Preparation of Copper solution**

Sample Details	Sample- 1	Sample –2
Weight of Copper + Weighing Bottle	g	g
Weight of empty Weighing Bottle	g	g
Weight of the copper taken	g	g

**Part-B: Estimation of Copper in Sample solution**

Burette readings (in cm <sup>3</sup> )	Trial-I	Expected Value by teacher	Trial-II	Expected Value by teacher
Final				
Initial				
Volume of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> run down				

**Part-C: Calculation****Sample – 1****Sample-2**

<b>Inference:</b>
<b>Relevance to Society &amp; Environment:</b>

Report:

Sample No.	Weight of Copper sample	Percentage of Copper in the given sample
1		
2		

Evaluation of Experiment - 1		
Components	Marks	
	Max	Obtained
Model Procedure & Calculation		
Burette Reading & Execution		
Inference & Societal Relevance		
Total		
Signature of Teacher		

**Suggestion Needed: after one expt , about chemistry Nobel laureates:**

The **Nobel Prize in Chemistry 2022** was awarded to **Carolyn R. Bertozzi, Morten Meldal** and **K. Barry Sharpless** “for the development of click chemistry and bioorthogonal chemistry”. Sharpless and Meldal have laid the foundation for a functional form of chemistry – *click chemistry* – in which molecular building blocks snap together quickly and efficiently. Bertozzi has taken click chemistry to a new dimension and started utilizing it in living organisms.

## EXPERIMENT NO – 2

**Aim: Determination of Total acidity of the soft drinks using pH sensors.**

**Theory:**

The dissociation constant of a weak acid is denoted as  $K_a$  and it is given by Ostwald's dilution law.



$$K_a = \frac{[\text{H}^+][\text{CH}_3\text{COO}^-]}{[\text{CH}_3\text{COOH}]}$$

The strength of the acid is indicated by its dissociation constant. Dissociation constant is also a measure of the tendency of an acid to donate proton. Generally,  $K_a$  value of weak acids are very small. In order to avoid negative power appearing as  $K_a$  value, a new term called  $\text{p}K_a$  value is coined. If  $K_a$  is the dissociation constant of a weak acid, then

$$\text{p}K_a = -\log_{10} K_a$$

The pH of a weak acid solution and  $\text{p}K_a$  are related as per Henderson - Hasselbalch equation:

$$\text{pH} = \text{p}K_a + \log_{10} \frac{[\text{salt}]}{[\text{acid}]} \dots\dots\dots (1)$$

Addition of a strong base (Sodium Hydroxide) to a weak acid (Acetic acid) produces salt (Sodium Acetate). At equivalence point (end point) all the acid is neutralized. At half equivalence point, half of the acid is neutralized, i.e.,  $[\text{salt formed}] = [\text{acid unreacted}]$ . Therefore, equation (1) reduces to  $\text{pH} = \text{p}K_a$ . This indicates that at half equivalence point the  $\text{p}K_a$  of the weak acid is equal to pH. Therefore, **the  $\text{p}K_a$  of a weak acid can also be defined as the pH at half equivalence point.**

During an acid-base titration, initially the pH value of the solution increases gradually and increases more rapidly thereafter till the equivalence point is reached. Once the equivalence point is crossed, pH increases by small amounts. The variation of pH is measured using combined glass electrode

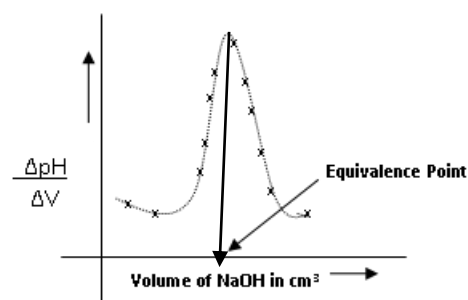
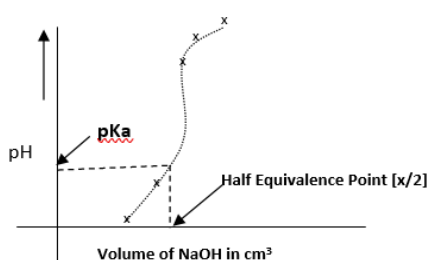
**Description:**

Pipette out  $50 \text{ cm}^3$  of the given soft drinks (weak acid) into a clean  $250 \text{ cm}^3$  beaker. Insert glass electrode - calomel electrode assembly into it and connect it to pH meter. Measure the pH of the acid. Fill the burette with the given sodium hydroxide. In the beginning add  $0.2 \text{ cm}^3$  of NaOH at a time and stir the mixture. Measure the pH. Continue adding  $0.2 \text{ cm}^3$  of NaOH & measure the pH regularly after each addition until the increase in pH is comparatively large. Take a few more reading by adding  $0.2 \text{ cm}^3$  of NaOH.

Determine the  $\text{p}K_a$  of the given weak acid from the two graphs:

(i)  $\frac{\Delta \text{pH}}{\Delta V}$  against volume of NaOH added.

(ii) pH against volume of NaOH added



**Calculation:**

The experiment is divided into two parts.

1. Determination of end point or equivalent point
2. Determination of pKa. (Using table-1 corresponding to pKa find out which weak acid is present)

Table-1

Acid	pKa	Gram equivalent weight
CH <sub>3</sub> COOH (acetic)	4.7	60/1 = 60
HCOOH (formic)	3.7	46/1 = 46
H <sub>2</sub> CO <sub>3</sub> (carbonic)	6.3	62/2 = 31
H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> (oxalic)	1.2	126/2 = 63

From the equivalence point find out the normality of weak acid using the formula

$$(NV)_{\text{acid}} = (NV)_{\text{NaOH}}$$

$$N_{\text{acid}} = \frac{(NV)_{\text{NaOH}}}{V_{\text{acid}}}$$

Amount of acid present in 1000 cm<sup>3</sup> of its solution =  $N_{\text{acid}} \times \text{gram equivalent weight of acid}$

**Model Procedure /Flow Chart:**



<b>Model graph-1:</b>	<b>Model graph-2:</b>
<b>Model Calculation:</b>	

Tabulation:

Volume of NaOH added in cm <sup>3</sup>	pH	$\Delta\text{pH}$	$\Delta V$	$\frac{\Delta\text{pH}}{\Delta V}$
0.0				
0.2				
0.4				
0.6				
0.8				
1.0				
1.2				
1.4				
1.6				
1.8				
2.0				
2.2				
2.4				
2.6				
2.8				
3.0				
3.2				
3.4				
3.6				

**Calculation:**

**Inference:**

**Relevance to Society & Environment:**

**Report: Total acidity of the given soft drink =**

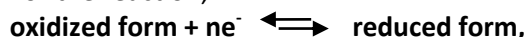
Evaluation of Experiment - 3		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation		
Equivalence Point & Execution		
Inference & Societal Relevance		
Total		
Signature of Teacher		

## EXPERIMENT NO – 4

### Aim: Potentiometric estimation of Iron

Nernst equation gives the relation between the potential at an electrode and the concentration of the active species in the solution.

For the reaction,

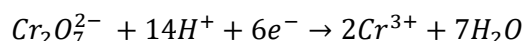
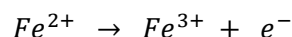


Nernst equation can be written as

$$E = E^\circ + \frac{0.0591}{n} \log \frac{[\text{oxidized form}]}{[\text{reduced form}]}$$

Where  $E^\circ$  is the standard electrode potential and the terms within the brackets represent their concentrations.

It is evident from the equation that the potential depends upon the concentrations of the oxidised and the reduced species in the solution. When a reducing agent in solution is oxidized using an oxidizing agent, the concentrations and hence the potential changes. Potentiometry essentially involves the measurement of change in potential as and when a species in solution is oxidized or reduced. When a titration of a reducing agent is carried out against an oxidising agent, the potential gradually changes in the beginning but changes rapidly near the end point. A plot of change in potential against volume reveals a sudden change in potential at the equivalence point. This sudden change in the present experiment can be explained as follows. The reactions that take place when dichromate is added to ferrous solution are:



The potential of the system before the equivalence point is given by

$$E = E^\circ + \frac{0.0591}{1} \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$$

i.e., the potential is dependent on the concentration of  $Fe^{2+}$  and  $Fe^{3+}$  ions. The potential of the solution will be around 0.75V. At the equivalence point the potential is decided by the concentrations of  $Fe^{2+}$ ,  $Fe^{3+}$ ,  $Cr_2O_7^{2-}$  and  $Cr^{3+}$  ions. The potential at equivalence point is given by

$$\begin{aligned} E &= \frac{E_{Fe^{3+}/Fe^{2+}}^\circ + E_{Cr^{6+}/Cr^{3+}}^\circ}{2} \\ &= \frac{0.75V + 1.33V}{2} = 1.04V \end{aligned}$$

Beyond the equivalence point as no ferrous ions exist, the potentials is determined by the concentration of  $Cr_2O_7^{2-}$  and  $Cr^{3+}$  ions and it is given by

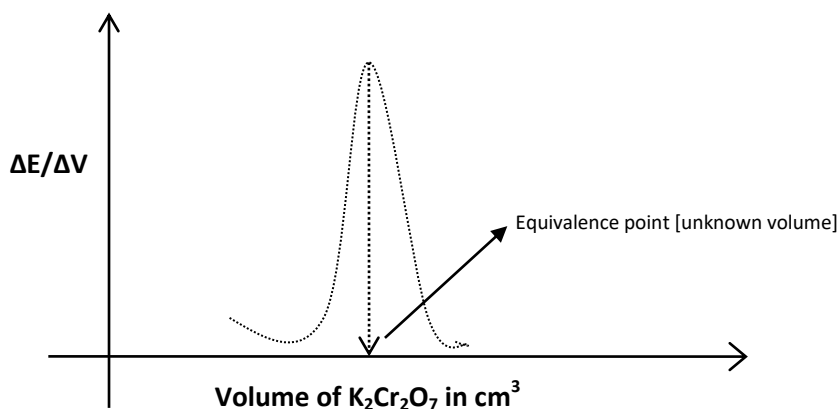
$$\begin{aligned} E &= E^\circ + \frac{0.0591}{6} \log [Cr^{6+}] / [Cr^{3+}] \\ &= 1.33V + \frac{0.0591}{6} \log [Cr^{6+}] / [Cr^{3+}] \end{aligned}$$

therefore, an abrupt increase in the potential is observed at the equivalence point. This increase marks the end point of the titration. Potential change at an electrode cannot be directly measured. The electrode at which the potential changes with concentration (indicator electrode) is connected to another electrode whose potential remains constant [ex:-calomel electrode (Standard reference electrode)], to form a cell.

**EMF =  $E_{\text{cathode}} - E_{\text{anode}}$ .** As the potential of the indicator electrode changes, the EMF of the cell also changes. It is the change in EMF that is measured during a potentiometric titration.

#### Description:

Pipette out 25 cm<sup>3</sup> of Ferrous ammonium sulphate solution in to a clean beaker. Add 1 test tube of dil. Sulphuric acid. Dip the electrode assembly into the solution and connect to a potentiometer. Measure the potential. Add 0.2 cm<sup>3</sup> of Potassium dichromate from a burette. Stir the solution well and measure the potential. Continue the process till the potential shows a tendency to increase rapidly. Now add dichromate in increments of 0.2 cm<sup>3</sup> and measure the potential after each addition. Plot a graph of  $\Delta E/\Delta V$  against volume of dichromate added as shown in the figure and find out the end point. Calculate the normality of the ferrous solution and determine the amount of iron in the given volume.



#### Calculation:

Volume of  $K_2Cr_2O_7$  required for the reaction =  $V \text{ cm}^3$  (From graph),  $N_{\text{FAS}} = \frac{(NV) K_2Cr_2O_7}{V_{\text{FAS}}}$

( $N_{\text{FAS}} = N_{\text{Iron}}$ )

Amount of iron presented in 1000 cm<sup>3</sup> of its solution =  $N_{\text{FAS}} \times \text{gram equivalent weight of Iron} = \dots\dots\dots \text{B g}$

#### Model Procedure /Flow Chart:

**Model graph:**

**Model Calculation:**

Tabulation:

Volume of $K_2Cr_2O_7$ added in $cm^3$	Potential (E) In mV	$\Delta E$	$\Delta V$	$\Delta E/\Delta V$
0.0				
0.2				
0.4				
0.6				
0.8				
1.0				
1.2				
1.4				
1.6				
1.8				
2.0				
2.2				
2.4				
2.6				
2.8				
3.0				
3.2				
3.4				
3.6				



**Calculation:**

**Inference:**

**Relevance to Society & Environment:**

**Report: Amount of Iron present in 1000 cm<sup>3</sup> of FAS solution =                      g**

Evaluation of experiment - 5		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation		
Equivalence Point & Execution		
Inference & Societal Relevance		
Total		
Signature of Teacher		

## EXPERIMENT NO – 5

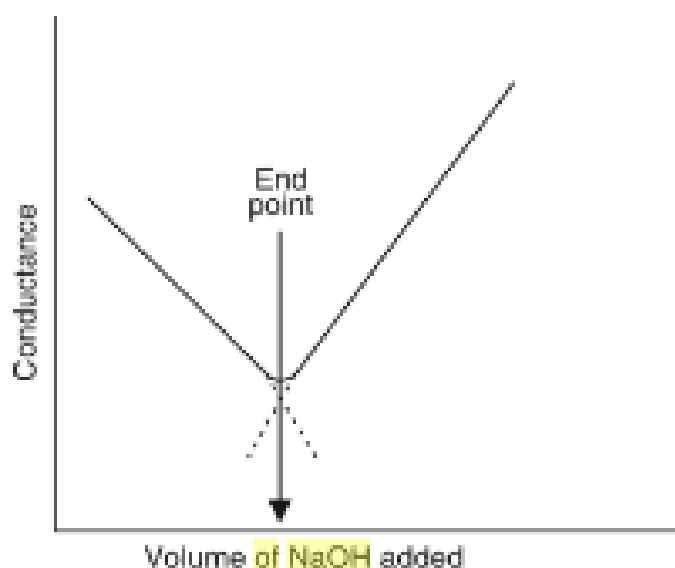
**Aim:** Conductometric estimation of strong acid vs strong base

### Theory:

Conductance of any solution (C) is the ease with which current flows through it. It is the reciprocal of resistance (R) offered by the solution.

$$C = 1/R$$

Conductance of a solution depends on number of ions, charge on the ions and also on the mobility of the ions. During the course of a titration, the conductance of the solution may change due to any of the above reasons. Such titrations in which conductance measurements are made use of in determining the equivalence point are called Conductometric titrations. Among all Conductometric titrations, reaction involving strong acid (HCl) and strong base (NaOH) is the most common activity. Hydrochloric acid is a strong electrolyte and would be completely in dissociated state. The conductance of this solution would be high. Upon addition of NaOH, highly mobile  $H^+$  ions of the acid are replaced by less mobile  $Na^+$  ions. Hence the conductance would decrease steeply. The same trend would continue till all the  $H^+$  ions available in the acid are completely reacted with the added NaOH. Now, the addition of NaOH would add more and more of  $Na^+$  and  $OH^-$  ions. Due to relatively higher mobility of  $OH^-$  ions, the conductance of the solution increases again. A typical graph of conductance v/s volume of NaOH (added) is given below.



**Model Procedure /Flow Chart:**

**Model graph:**

**Tabulation:**

<b>Volume of NaOH in cm<sup>3</sup></b>	<b>Conductance in m S</b>
0.0	
0.2	
0.4	
0.6	
0.8	
1.0	
1.2	
1.4	
1.6	
1.8	
2.0	
2.2	
2.4	
2.6	
2.8	
3.0	
3.2	
3.4	
3.6	
3.8	

**Calculation:**

**Inference:**

**Relevance to society & environment:**

**Report:**

1. Normality of the HCl = ..... N
2. Amount of HCl present in the given solution = ..... g

Evaluation of Experiment -5		
	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation		
Equivalence Point & Execution		
Inference & Societal Relevance		
Total		
Signature of Teacher		

## EXPERIMENT NO – 6

### Aim: Conductometric estimation of mixture of acids

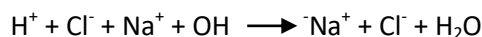
Conductance of any solution (C) is the ease with which current flows through it. It is the reciprocal of resistance (R) offered by the solution.

$$C = 1/R$$

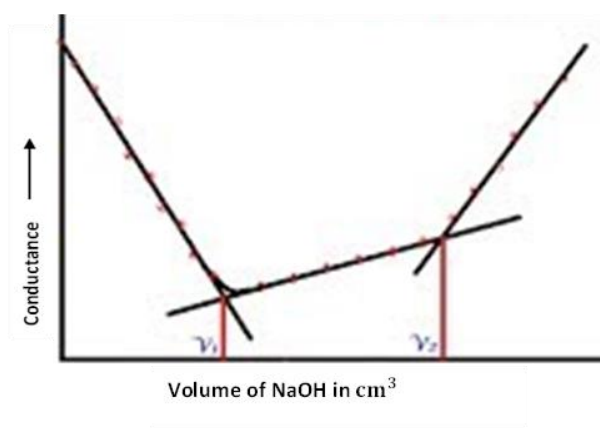
Conductance of a solution depends on number of ions, charge on the ions and also on the mobility of the ions. During the course of a titration, the conductance of the solution may change due to any of the above reasons. Such titrations in which conductance measurements are made use of in determining the equivalence point are called Conductometric titrations.

Among all Conductometric titrations, reaction involving strong acid (HCl)/ weak acid (CH<sub>3</sub>COOH) and strong base (NaOH) is the most common activity.

Hydrochloric acid is a strong electrolyte and would be completely in dissociated state. The conductance of this solution is the maximum, before the addition of NaOH. Upon addition of NaOH, highly mobile H<sup>+</sup> ions of the acid are replaced by less mobile Na<sup>+</sup> ions.



Hence the conductance would decrease steeply. The same trend would continue till all the H<sup>+</sup> ions available in the acid are completely reacted with the added NaOH. After the neutralization of all hydrogen ions of strong acid, sodium hydroxide solution will neutralize hydrogen ions of acetic acid. Now, the addition of NaOH would add more and more of Na<sup>+</sup> and OH<sup>-</sup> ions. Due to relatively higher mobility of OH<sup>-</sup> ions, the conductance of the solution increases again. A typical graph of conductance vs. volume of NaOH (added) is given below.



The break in the curve corresponds to the two equivalence points corresponding to two acids. From the volume of NaOH required to neutralize HCl or CH<sub>3</sub>COOH, the amount of HCl or CH<sub>3</sub>COOH is calculated.

### Description:

Pipette out 50cm<sup>3</sup> of acid into a clean 200 cm<sup>3</sup> beaker. Dip the conductivity cell in the acid, such that the two platinum electrodes are immersed completely. The cell is connected to the conductivity bridge. Measure the conductance. Take the given NaOH solution in a burette rinsed with the same solution. Rundown 0.2 cm<sup>3</sup> of NaOH solution into the beaker and shake well. Note down the conductance. Note down the conductance by adding 0.2 cm<sup>3</sup> of NaOH each time to the beaker. Conductance decreases in the beginning, increases slightly and finally starts increasing by large value. Take around 8-10 readings on the increasing side. Draw a graph of



conductance against volume of NaOH added. 1<sup>st</sup> equivalence point in the curve corresponds to the neutralization of HCl and the difference of two equivalence point corresponds to the neutralization of CH<sub>3</sub>COOH acid. Calculate the normality and amount of HCl and CH<sub>3</sub>COOH present in 1000 cm<sup>3</sup> of its solution.

**Calculation:**

$$(NV)_{\text{acid}} = (NV)_{\text{NaOH}}$$

$$N_{\text{acid}} = \frac{(NV)_{\text{NaOH}}}{V_{\text{acid}}}$$

Amount of HCl or CH<sub>3</sub>COOH present in 1000 cm<sup>3</sup> of its solution =  $N_{\text{acid}} \times \text{gram equivalent weight of acid}$

**Model Procedure /Flow Chart:**

**Model graph:**

**Model Calculation:**

**Tabulation:**

Volume of NaOH in cm <sup>3</sup>	Conductance in mS
0.0	
0.2	
0.4	
0.6	
0.8	
1.0	
1.2	
1.4	
1.6	
1.8	
2.0	
2.2	
2.4	
2.6	
2.8	
3.0	
3.2	
3.4	

Volume of NaOH in cm <sup>3</sup>	Conductance in mS
3.6	
3.8	
4.0	
4.2	
4.4	
4.6	
4.8	
5.0	
5.2	
5.4	
5.6	
5.8	
6.0	

**Calculation:**

**Inference:**

**Relevance to Society & Environment:**

**Report:**

1. Normality of HCl = ..... N, Normality of CH<sub>3</sub>COOH = .....N
2. Amount of HCl present in 1000 cm<sup>3</sup> of its solution = ..... g
3. Amount of CH<sub>3</sub>COOH present in 1000 cm<sup>3</sup> of its solution = ..... g

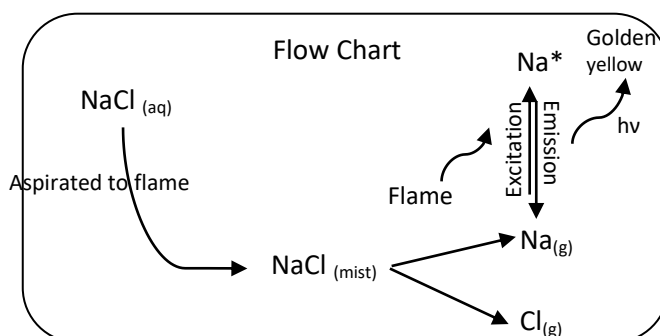
Evaluation of experiment - 6		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation		
Equivalence Point & Execution		
Inference & Societal Relevance		
Total		
Signature of Teacher		

## EXPERIMENT NO – 07

**Aim:** Flame photometric estimation of sodium.

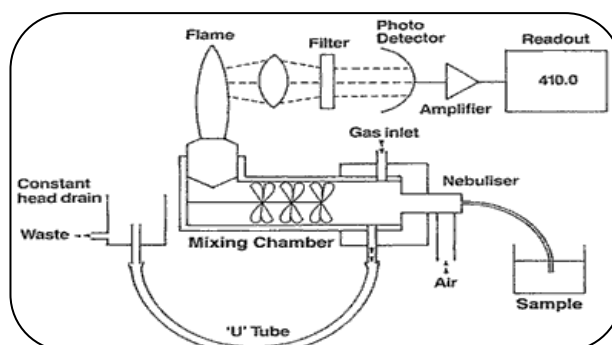
**Theory:**

Flame Photometry is an atomic emission technique used for detection of metals. If a solution containing metallic salts is aspirated into a flame, a vapour will be formed which contains metallic atoms. The electrons from the metallic atoms are then excited from ground state ( $E_1$ ) to higher energy state ( $E_n$ ) where  $n = 2, 3, 4$ , etc., by making use of thermal energy of flame. From higher energy states, these electrons will return to the ground state by emitting radiations ( $E_n - E_1 = h\nu$  where  $n = 2, 3, 4$ , etc., which are the characteristic of each element.



Flame photometer correlates the emitted radiations with the concentration of these elements. It is simple and rapid method for the elements that can be easily excited (Sodium and other alkali metals).

A flame photometer is composed of the pressure regulator, flow meter, an atomizer, burner, optical system, photosensitive detector and output recorder. A filter corresponding to the wavelength of light emitted by the element whose concentration is to be determined, is inserted between the flame and the detector. Propane gas is used as fuel and air or Oxygen is used as oxidant. Combination of these two will give a temperature of  $1900^\circ\text{C}$ . The whole analysis depends on the flow rate of the fuel, oxidant, the rate of introduction of the sample and droplet size.



The sample containing the analyte is aspirated into the flame through nebulizer. Radiation from resulting flame is collected by the lens and allowed to pass through an optical filter, which permits only the radiation characteristic of the element under investigation into the photocell. The output from the photocell represents the concentration and nature of the element.

**Description:**

Transfer 2,4,6,8 and 10 cm<sup>3</sup> of standard sodium solution into different 25 cm<sup>3</sup> volumetric flasks from the burette. Make up all the solutions using double distilled water. Stopper the flasks and shake well to get uniform concentration. To the given unknown solution, add double distilled water and shake well. Switch on the instrument; turn the gas supply on and light the gas at the burner. Adjust the air supply from the compressor to 10 lbs/sq inch using pressure regulator knob. Place the sodium filter (589nm) in position. Now dip the capillary tube in a cell containing double distilled water. The stream of air atomized as a fine mist draws up the liquid. Regulate the gas supply so that the colour of the flame completely turns to blue. Adjust the flame photometer to zero by means of zero control knob. Feed the various sodium solutions prepared, through the flame one by one including the unknown solution. Note down the flame photometer readings. Plot a graph of flame photometer readings against the volume of the solution get the calibration curve. Using the curve obtained find out the volume of the unknown solution containing sodium and calculate the amount of sodium in it.

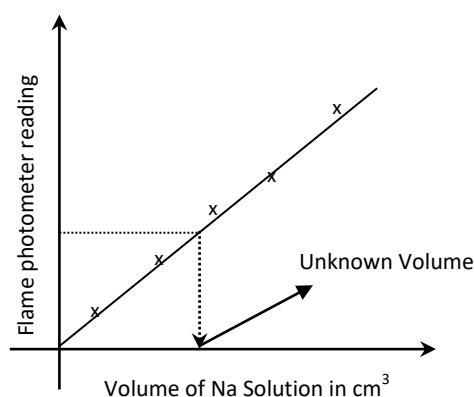
#### Calculation:

Amount of NaCl in the given 100cm<sup>3</sup> solution= 2.5 g

58.5 g of NaCl contains 23 g of Na.

Therefore, 1 cm<sup>3</sup> of the given stock solution contains =  $\frac{wx23}{58.5x100}$  = 'A'g of Na

From the graph calculate the volume of unknown solution and amount of sodium present in the unknown solution.



#### Model Procedure /Flow Chart:

**Model Calculation:**

**Model graph:**

**Tabulation:**

Sl. No.	Vol. of NaCl in cm <sup>3</sup>	Flame Photometer Reading	Wt. of Sodium in mg
1.	2		
2.	4		
3.	6		
4.	8		
5.	10		
6.	Unknown		

**Calculation:**

**Inference:**

**Relevance to Society & Environment:**



**Report:**

1. Volume of unknown solution = .... cm<sup>3</sup>
2. Amount of Sodium in the given unknown solution = ..... mg.

Evaluation of experiment - 07		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation		
Expected Volume & Execution		
Inference & Societal Relevance		
Total		
Signature of Teacher		

## EXPERIMENT NO – 8

### Aim: Colorimetric estimation of copper from E-waste

#### Theory:

When a monochromatic light of intensity  $I_0$  is incident on a medium, a part of it may be absorbed ( $I_a$ ), a part of it may be transmitted ( $I_t$ ) and the remaining part may be reflected ( $I_r$ ).

$$\text{Thus, } I_0 = I_a + I_t + I_r$$

$I_r$  can conveniently ignore when the medium is transparent like perfect glass.

$$\text{Therefore, } I_0 = I_a + I_t$$

If  $I_t/I_0$  is called transmittance then  $\log(I_0/I_t)$  is called absorbance or optical density ( $A$ ). The relation between absorbance, concentration of the medium and path length or thickness of the medium is referred as Beer-Lambert's law.

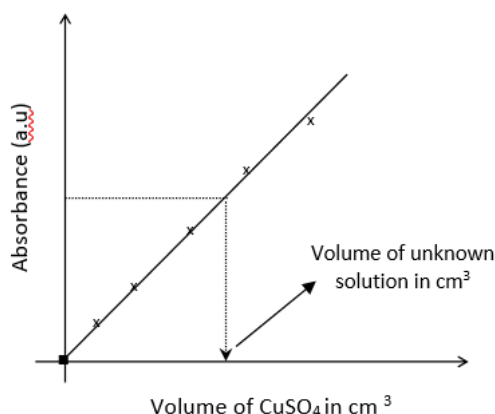
$$A = \epsilon c l$$

Where 'A' is absorbance, 'c' is concentration, 'l' is thickness and 'ε' is a constant called molar extinction coefficient. ε is constant for a given substance.

Chemical analysis through measurement of absorption of light of a particular wavelength is known as colorimetry. The absorbance of light of a particular wavelength by a substance in solution varies directly with its concentration and the thickness of the solution. When the thickness of the medium is kept constant, the absorbance directly depends upon the concentration.

#### Description:

Transfer 2,4,6,8 and 10 cm<sup>3</sup> of given copper sulphate solution into different 25 cm<sup>3</sup> volumetric flask from a burette. Add 2.5 cm<sup>3</sup> of ammonia to all the flasks and makeup all the solutions up to the mark using distilled water. Stopper the flasks shake well to get uniform concentration. Prepare a blank solution by taking 2.5 cm<sup>3</sup> of ammonia in another 25 cm<sup>3</sup> volumetric flask. Make up that solution also up to the mark using distilled water and shake well. To the given unknown solution also add 2.5 cm<sup>3</sup> of ammonia, make it up to the mark and shake well. After 10 min. measure the absorbance of all solutions by using 620 nm filter of a colorimeter. Note down the optical density of all solutions and tabulate the readings. Draw a calibration curve by plotting absorbance against volume of copper sulphate taken. Using the calibration curve, find out the unknown volume of copper sulphate solution given and calculate the amount of copper present in it



**Calculation:**

Weight of copper sulphate pentahydrate present in  $100\text{ cm}^3$  of the given solution = 55 mg

Weight of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in  $1\text{ cm}^3$  of its solution =  $X/100\text{ mg} = \dots\dots\dots = 'Y'\text{ mg}$

Weight of copper present in  $1\text{ cm}^3$  of its solution =  $\frac{'Y' \cdot 63.54}{249.54} = 'W'\text{ mg}$

**Model Procedure /Flow Chart:****Model graph:**

**Model Calculation:**

**Tabulation:**

Sl. No.	Vol. of $\text{CuSO}_4$ in $\text{cm}^3$	Optical Density	Wt. of Copper (mg)
1.	0 [ Blank]	_____	_____
2.	2		
3.	4		
4.	6		
5.	8		
6.	10		
7.	Unknown		

**Calculation:**

**Inference:**

**Relevance to Society & Environment:**

**Report:**

1. Volume of unknown solution = .....cm<sup>3</sup>
2. Amount of Copper in the given unknown solution = ..... mg

Evaluation of experiment - 8		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation		
Expected Volume & Execution		
Inference & Societal Relevance		
Total		
Signature of Teacher		

## EXPERIMENT NO – 9

**Aim:** Determination of viscosity coefficient of a given liquid using Ostwald's viscometer

**Theory:**

Viscosity arises due to friction between moving layers of a liquid. A liquid flowing through a cylindrical tube of uniform diameter is expected to move in the form of molecular layers. Layers close to the surface are almost stationary while that at the axis of the tube moves faster than any other intermediate layer. A slow-moving layer exerts a drag or frictional force on its nearest moving layer, backwards. This property of the liquid which retards or opposes the motion between the layers is called viscosity. **The coefficient of viscosity is defined as the tangential force per unit area required to maintain a unit velocity gradient between the two successive layers of the liquid situated at unit distance apart.** The coefficient of viscosity of a liquid is given by the Poiseuille's formula

$$\eta = \frac{\pi P r^4 t}{8 V L}$$

Where 'V' is the volume of the liquid, 'r' is the radius of the tube and 'P' is the pressure between the two ends of the tube and ' $\eta$ ' is the coefficient of viscosity. If equal volumes of the two different liquids are allowed to flow through the same tube under identical conditions, then

$$\frac{\eta_l}{\eta_w} = \frac{t_l d_l}{t_w d_w}$$

The time ' $t_l$ ' taken by the given liquid to travel through a certain distance in the tube is determined. The time ' $t_w$ ' taken by standard liquid (water) to travel through the same distance is measured. Knowing the densities of the two liquids ( $d_l$  and  $d_w$ ) and also the coefficient of viscosity of the standard liquid, coefficient of viscosity of test liquid is calculated.

**Description:**

Take a clean and dry viscometer. Fix it vertically to a stand. Using graduated pipette, transfer known volume (say 10 cm<sup>3</sup>) of the given liquid into the viscometer. Then it is placed in a water bath such that its big bulb is completely immersed in water. Suck the liquid above the upper mark of the small bulb of the viscometer. Allow it to flow through the capillary. When the liquid crosses the upper mark of the small bulb, start a stop watch and when the liquid crosses the lower mark, stop the watch. Note down the time of flow in seconds.

Repeat the experiment 3-4 times and calculate the average time of flow.

Remove the liquid from the viscometer, clean well with acetone and dry it in oven. Cool the viscometer to room temperature. Similarly, measure the time of flow of water by taking the same volume. Calculate average time of flow of water. Note down the room temperature. Knowing the density, viscosity of water and density of the given liquid, viscosity of the liquid can be calculated using the formula given below.

$$\eta_l = \frac{t_l d_l \eta_w}{t_w d_w}$$

**Model Procedure /Flow Chart:**

**Model Calculation:**



**Observation:**

Liquid	Time of Flow		
	in min: sec	in seconds	Average in seconds
Test liquid			$t_i =$
Water			$t_w =$

**Calculation:**

**Inference:**

**Relevance to Society & Environment:**

**Report: The coefficient of viscosity of the given liquid at .....<sup>0</sup>C =**  **$\times 10^{-4} \text{ Nsm}^{-2}$**

Evaluation of experiment - 9		
Components	Marks	
	Max	Obtained
Model Procedure, Model Graph & Calculation		
Expected Value & Execution		
Inference & Societal Relevance		
Total		
Signature of Teacher		

## EXPERIMENT NO – 10

### Aim: Electroplating of copper on metallic objects

#### Theory:

Electroplating is one kind of surface finishing. There are many other kinds. Everyone has seen and handled electroplated objects, even if they didn't know it. Examples include kitchen and bathroom faucets, inexpensive jewelry and the trim on some automobiles. There are thousands of examples. In fact, it is certain that nearly every piece of metal you have ever seen has been through some kind of surface finishing process. There are three basic reasons for surface finishing: to improve appearance, to slow or prevent corrosion (rust) and to increase strength and resistance to wear (in the case of "engineering" finishes). An object may be processed for any or all of these reasons.

The term electroplating means the coating of an object with a thin layer of metal by use of electricity. The metals most often used are gold, silver, chromium, copper, nickel, tin and zinc, but many others are also used. The object to be plated is usually a different metal, but can be the same metal. Electrodeposition of metals is performed by immersing a conductive surface in a solution containing ions of the metal to be deposited. The surface is electrically connected to an external power supply, and current is passed through the surface into the solution. Copper (II) ions go into solution from anode and plated out at the cathode (metal surface to be coated).

**Cathode:**  $\text{Cu}^{2+}(\text{aq}) + 2 \text{e}^- \rightarrow \text{Cu}(\text{s})$

**Anode:**  $\text{Cu}(\text{s}) \rightarrow \text{Cu}^{2+}(\text{aq}) + 2 \text{e}^-$

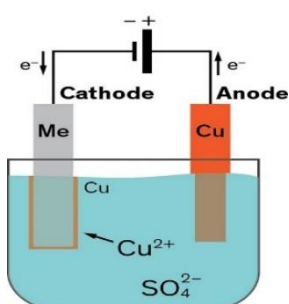
Commercial plating is done very slowly in order to obtain a smooth, even coating of the plated metal. Although this experiment does not produce plating of commercial quality, it gives you the opportunity to study the chemistry of an important commercial process. This general method is also used in purifying copper. A small cathode of pure copper is used with a larger anode of impure copper. As the electrolytic cell operates, pure copper is transferred to the cathode.

#### Chemicals and Equipment Required:

1. Electrolyte solution (200 g  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  + 25.0 ml concentrated  $\text{H}_2\text{SO}_4$  solution in enough distilled or deionized water to make 1 L of solution), Metal coins, vinegar, Sodium Chloride
2. Power supply (6.0-9.0 volts, 0.60-1.0 amps), connecting wires with alligator clips, 16-18-gauge copper wire, 250 ml beaker, cardboard square (approx. 15 cm on a side)

#### Description:

Pour 200 ml of the electrolyte solution into the beaker. Electrolyte solution is a strong solution of copper sulfate plus a few drops of sulfuric acid in order to keep the solution acidic. Attach connecting wires with alligator clips to the terminals of the power supply or a 6 volt battery. Clean the coins with a mixture of 3 g NaCl and 15 ml vinegar; rinse and dry. Tightly wrap one end of a 10-cm length of copper around each coin, leaving 5-6 cm of wire free. Weigh each coin-copper wire assembly and record the weight. Push the free end of each wire through the cardboard square and place the square over the beaker so that the coin "electrodes" are immersed in the electrolyte solution as shown below. Note: the two electrode assemblies must not touch.



Attach the connecting wires to the top of the copper wire assemblies. Allow the electroplating cell to operate for 30-60 minutes. Record the exact time the cell was operating (optional). Record the ammeter reading from the meter or power supply (optional). Remove each “electrode;” dry, being careful not to lose any of the copper plating; weigh each and record.

**Model Procedure:**
**Observation:**

Sl No.	Weight of the coin before electroplating	Weight of the coin after electroplating	Time duration for passing current	Current in ampere

**Inference:**
**Relevance to Society & Environment:**
**Report:**

Amount of Copper deposited =            mg

## EXPERIMENT NO – 11

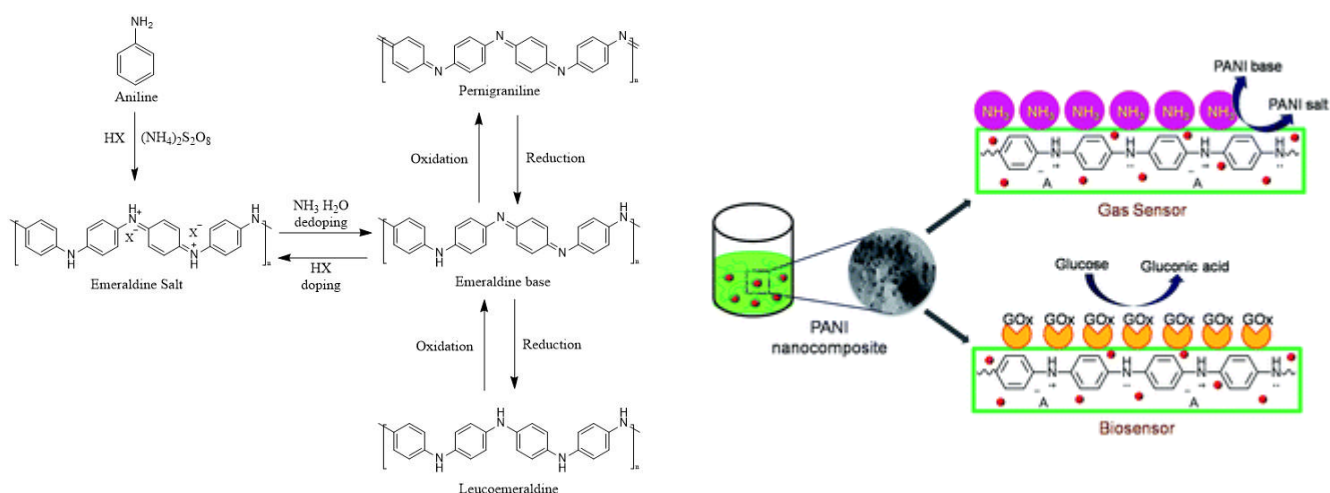
### Aim: Synthesis and fabrication of conducting polyaniline and its application in gas sensing

Conductive polymers or, more precisely, intrinsically conducting polymers (ICPs) are organic polymers that conduct electricity. Polyaniline was first described in the mid-19th century by Henry Letheby, who investigated the electrochemical and chemical oxidation products of aniline in acidic media. He noted that reduced form was colourless but the oxidized forms were deep blue. Conductive polymers are prepared by many methods. Most conductive polymers are prepared by oxidative coupling of monocyclic precursors. The low solubility of most polymers presents challenges. Some researchers add solubilizing functional groups to some or all monomers to increase solubility.

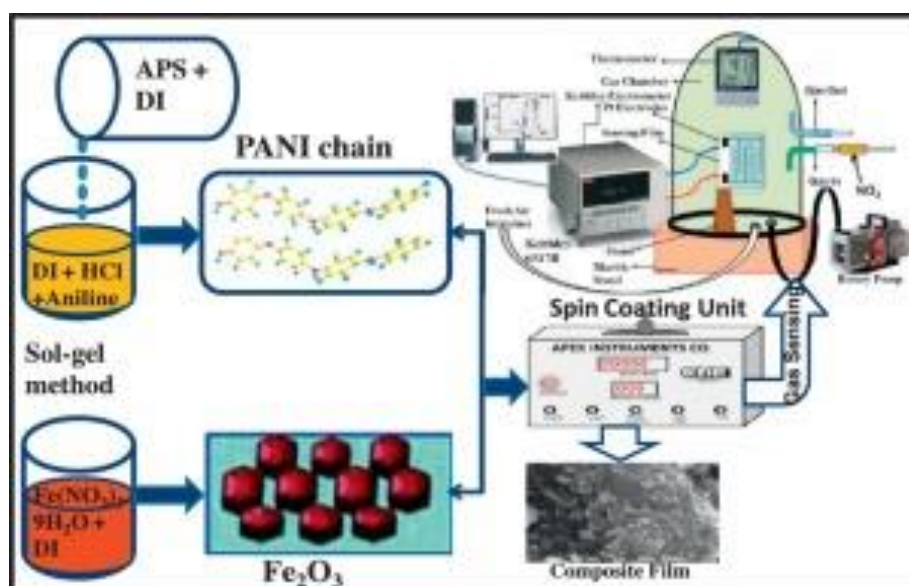
### Polyaniline (PANI) synthesis.

PANI was synthesized by oxidative polymerization using 0.1 M anilinium hydrochloride (ANHC) in 1 M HCl and ammonium peroxydisulfate (APS) as oxidant agent (equimolar to aniline). The polymerization reaction was made in a cooled ( $0\text{ }^{\circ}\text{C}$ – $0.3\text{ }^{\circ}\text{C}$ ) and stirred reactor. PANI can be shaped into various structures with different morphologies and the possibility of obtaining nanofibers, in addition to thin films, has opened a rapid development of chemical sensors, with improved processability and functionality. The synthesis and fabrication model is shown below.

### Synthesis:



### Fabrication: Spin Coating technique:

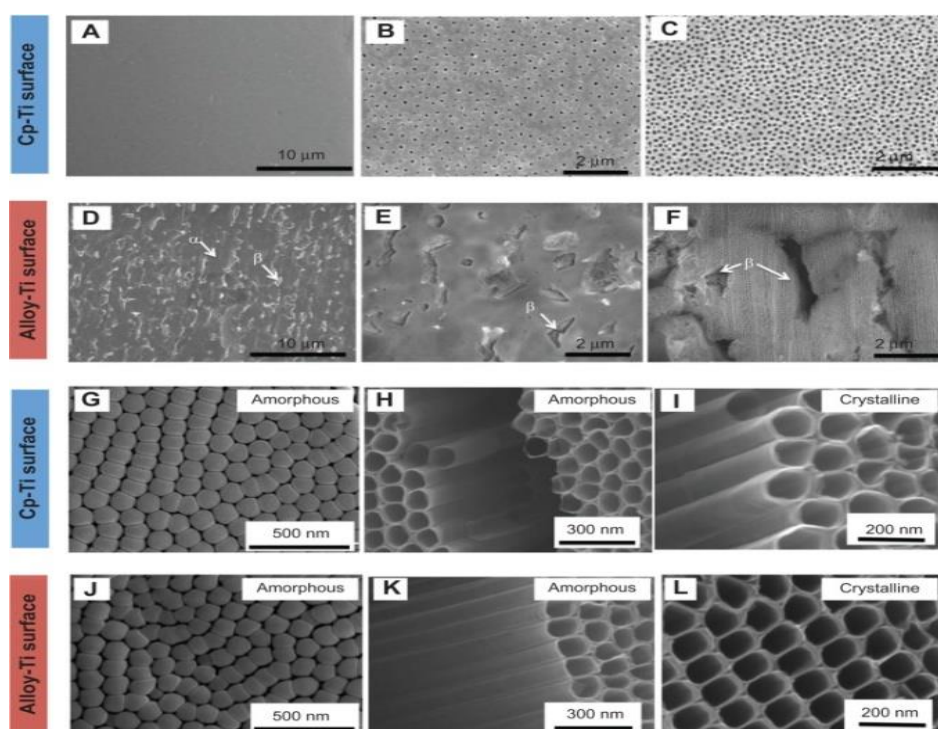


## EXPERIMENT NO – 12

### Aim: Study the surface morphology of nanomaterials using scanning electron microscopy

Nano science and nanotechnology involve the study and working with matter on an ultra-small scale. Nanomaterial is of enormous scientific attention as they are effectively a bridge between bulk materials and atomic or molecular structures. A bulk material must have constant physical properties apart from of its size, but at nano scale this is often not the case. The properties of the materials modify as their size approaches the nano scale and as the percentage of atoms at the surface of a material suits important. For bulk materials bigger than one micrometer the percentage of atoms at the surface is less, relative to the total number of atoms of the material. Therefore these interesting and sometimes unexpected properties are partly due to the surface that leads in nanoparticles, which is not the case in bulk materials. Interest in metal nanoparticles is driven which reveal novel chemical and physical properties due to their small physical dimensions. Owing to their small size and large surface area the metal nanoparticles have unique electronic, mechanical, magnetic and chemical properties that are different from those of bulk materials. For these reasons metallic nanoparticles have found applications in different fields such as electronic, thermal, catalysis, photonics, biosensors and optoelectronics.

**Scanning electron microscope (SEM)** is one of the most widely used techniques used in characterization of nanomaterials and nanostructures. The signals that derive from electron-sample interactions reveal information about the sample including surface morphology (texture), chemical composition of the sample. The surface morphology strongly determines the conditions of reagents adsorption and diffusion, and information on the surface layer composition allows estimating stability of the properties of the support upon its contact with a solution of the active component precursor and under high-temperature treatments. Schematic surface morphology different types of material are shown below.

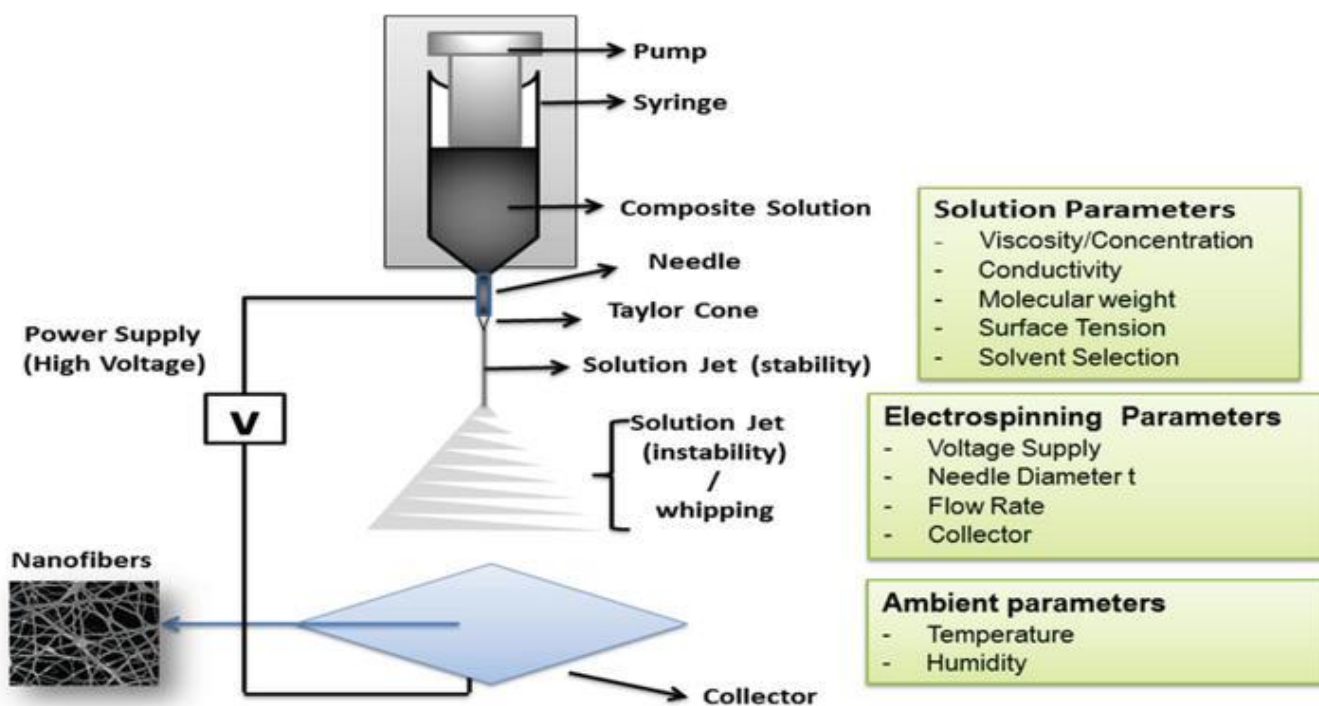


## EXPERIMENT NO – 13

Aim: Fabrication of thin-film gas sensors using spin coating and electro-spinning technique

Electrospinning involves an electrohydrodynamic process, during which a liquid droplet is electrified to generate a jet, followed by stretching and elongation to generate fiber. Electrospinning offers advantages like, control over morphology, porosity and composition using simple equipment. Variety of fibres can be used in the process. Nanofibers with of 40-2000 nm can be produced by selecting suitable combination of polymer and solvent to be used. In a typical electrospinning or electrospray process, an electrostatic force is applied to overcome the surface tension of a charged liquid which normally comes out of a syringe with a metallic needle connected to a high-voltage power supply.

Electrospinning basically depends on the high electrostatic forces. Factors that influence the electrospinning process are polymer concentration, solution viscosity and flowrate, electric field intensity, the work distance, and air humidity. Electrospinning is gaining more and more interest due to its versatility, simplicity and economy as well as the possibility of producing fibers from various types of polymeric, ceramic and metallic materials. Nanofibrous layers produced by this method are characterized by high quality and the desired physicochemical properties. The process is shown below:





## EXPERIMENT NO – 14

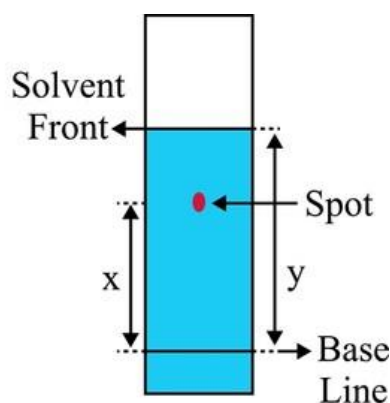
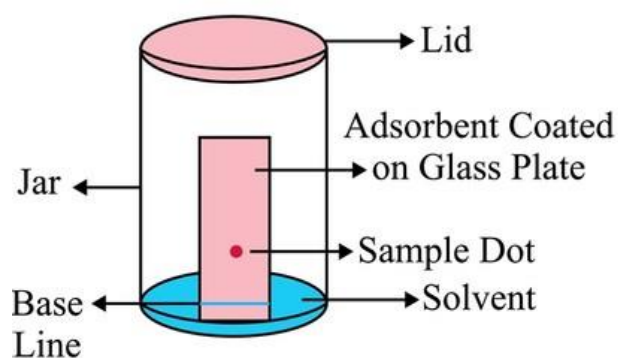
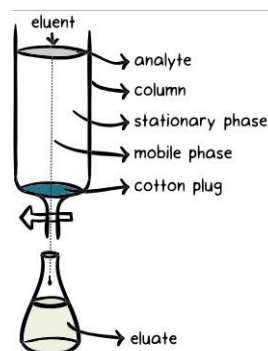
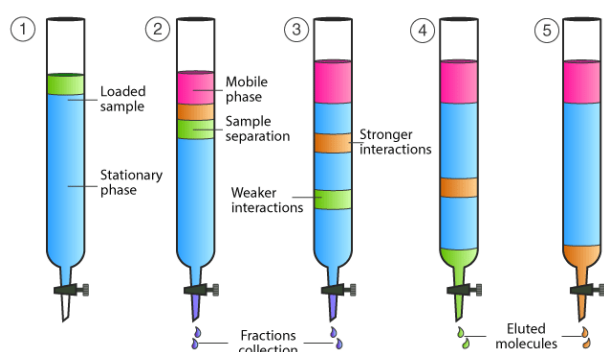
**Aim:** Separation of organic compounds using column chromatographic technique and monitoring by thin layer chromatographic technique

**Column chromatography** in chemistry is a chromatography method used to isolate a single chemical compound from a mixture. Chromatography is able to separate substances based on differential adsorption of compounds to the adsorbent; compounds move through the column at different rates, allowing them to be separated into fractions. The technique is widely applicable, as many different adsorbents (normal phase, reversed phase, or otherwise) can be used with a wide range of solvents. The technique can be used on scales from micrograms up to kilograms. The main advantage of column chromatography is the relatively low cost and disposability of the stationary phase used in the process. The latter prevents cross-contamination and stationary phase degradation due to recycling. Column chromatography can be done using gravity to move the solvent, or using compressed gas to push the solvent through the column.

A thin-layer chromatograph can show how a mixture of compounds will behave when purified by column chromatography. The separation is first optimised using thin-layer chromatography before performing column chromatography. The *mobile phase* or *eluent* is a solvent or a mixture of solvents used to move the compounds through the column. It is chosen so that the retention factor value of the compound of interest is roughly around 0.2 - 0.3 in order to minimize the time and the amount of eluent to run the chromatography. The eluent has also been chosen so that the different compounds can be separated effectively. The eluent is optimized in small scale pretests, often using thin layer chromatography (TLC) with the same stationary phase. Diagrammatic representation as shown below:

### COLUMN CHROMATOGRAPHY

BYJU'S  
The Learning App





### Basics of Computational Method of Analysis.

Expt.	Computational Method of Analysis (CMA)
15	Introduction to computational chemistry software's (Chem sketch, argus lab, Avogadro, discovery studio, VMD, CORINA to design molecular structure, visualization, data analysis (origin, excel, xmgrace).
16	Optimization of geometry and Prediction of HOMO, LUMO, density surface and energy of small molecules using Chem sketch and argus lab software and their significance in materials science.
17	Prediction of absorption spectra and vibrational spectra of small molecules with its interpretation and applications in designing sensors

#### Introduction about software:

**ChemSketch** is a molecular modelling program used to create and modify images of chemical structures. Also, there is a software that allows molecules and molecular models displayed in two and three dimensions, to understand the structure of chemical bonds and the nature of the functional groups. <https://www.acdlabs.com/resources/free-chemistry-software-apps/chemsketch-freeware/>

**ArgusLab** is a program to build graphic representations of molecular models. Using this program, you will be able to show molecular models to pupils, or even design matters by combining different elements. You will be able to include in your model several atoms, residues, groups and calculations. Every component can be edited to meet your needs. You can use hydrogen, carbon, nitrogen, oxygen, chlorine and fluorine atoms. You can join those atoms using any kind of bond possible. This way you will be able to build simple or complex molecules. If you need it, ArgusLab lets you check a built-in periodic table. You can zoom in to make the model bigger, or zoom out to see less details. ArgusLab also allows you to build surfaces, calculate energy, optimize geometry, perform Gaussian calculations, plot molecules and everything you will need to customize your molecular model. The results can be printed or plotted. They can be saved in ArgusLab proprietary format. They can also be exported to BMP, JPG, TIFF or POV Ray formats. <https://arguslab.en.softonic.com/>

**Avogadro** is a molecule editor and visualizer designed for cross-platform use in computational chemistry, molecular modelling, bioinformatics, materials science, and related areas. <https://avogadro.cc/>

**Discovery Studio** is a suite of software for simulating small molecule and macromolecule systems. It is developed and distributed by Dassault Systemes BIOVIA (formerly Accelrys). The product suite has a strong academic collaboration programme, supporting scientific research and makes use of a number of

software algorithms developed originally in the scientific community, including CHARMM, MODELLER, DELPHI, ZDOCK, DMol3 and more. <https://discover.3ds.com/discovery-studio-visualizer-download>

**Visual Molecular Dynamics (VMD)** is a molecular modelling and visualization computer program. VMD is developed mainly as a tool to view and analyze the results of molecular dynamics simulations. It also includes tools for working with volumetric data, sequence data, and arbitrary graphics objects. Molecular scenes can be exported to external rendering tools such as POV-Ray, RenderMan, Tachyon, Virtual Reality Modeling Language (VRML), and many others. Users can run their own Tcl and Python scripts within VMD as it includes embedded Tcl and Python interpreters. VMD runs on Unix, Apple Mac macOS, and Microsoft Windows. VMD is available to non-commercial users under a distribution-specific license which permits both use of the program and modification of its source code, at no charge. <https://www.ks.uiuc.edu/Research/vmd/>

The classic command-line version of **CORINA** is a fast and powerful 3D structure generator for small and medium-sized, drug-like molecules. Its robustness, comprehensiveness, speed and performance and its additional structure clean-up and standardization methods make CORINA Classic a perfect application to convert large chemical datasets and databases.

Fast and powerful 3D structure generator for small and medium sized, typically drug-like molecules. Its robustness, comprehensiveness, speeds and performance makes CORINA a perfect application to convert large chemical datasets or databases. <https://www.iucr.org/resources/other-directories/software/corina>

### **Data Analysis:**

**Data analysis** is vital in many fields for not only knowing how to strategize in business but also in demonstrating the situation to others. Proper graphing of data allows businesses within a scientific or engineering field to present data to shareholders and the public as well as aiding the decision-making process. **Origin** is a great example of a widely used professional data analysis tool which can make presenting data easier than ever. Origin is designed to make analysing and presenting data easier. Graphing has little extra touches that show how familiar the creators are with daily tasks such as skipping weekends and holidays in financial plots. Graphs can be exported to PowerPoint and processed in batches. Further features can be added with modular apps for specific tasks or you can customise your own routines if you know your script. The software is even free to try. <https://origin.en.softonic.com/>

**Microsoft Excel** is a spread sheet developed by Microsoft for Windows, macOS, Android and iOS. It features calculation or computation capabilities, graphing tools, pivot tables, and a macro programming

language called Visual Basic for Applications (VBA). Excel forms part of the Microsoft Office suite of software. <https://www.microsoft.com/en-in/microsoft-365/excel>

**Grace, also known as Xmgrace**, ACE/gr, and Xvgr, is a free WYSIWYG 2D graph plotting tool used by relax **grace** user functions to provide the highest quality 2D graphing capabilities. **Grace** is a free WYSIWYG 2D graph plotting tool, for Unix-like operating systems. The package name stands for "GRaphing, Advanced Computation and Exploration of data." Grace uses the X Window System and Motif for its GUI. It has been ported to VMS, OS/2, and Windows 9\*/NT/2000/XP (on Cygwin). In 1996, Linux Journal described Xmgr (an early name for Grace) as one of the two most prominent graphing packages for Linux. <https://plasma-gate.weizmann.ac.il/Grace/>

For doing the calculations of molecular structure different types of inputs can be given to the computer. After doing the calculations using semiempirical methods / quantum calculations or Abinitio calculations the outputs are interpreted in terms of meaningful physical significance. One the input is 3D cartesian coordinates of the molecular structure. For small molecules or biopolymers 3D coordinates can be obtained through XRD / NMR etc. It is always a challenge for the computational chemist where the cartesian coordinate is not available. In those cases the coordinates are generated using some available softwares. The softwares build the structures using the bond length and bond angles data which is taken from the library. There are many free academic and commercial softwares available to get the 3D coordinates of the chemical structure. In this section we will describe how to generate the 3D structure using BIOVIA Discovery Studio. We will be discussing step by step procedure.

Step 1: About the software

Step 2: Installation of BIOVIA Discovery Studio

Step 3: Drawing the structure using user graphics interface

Step 4: Energy optimization

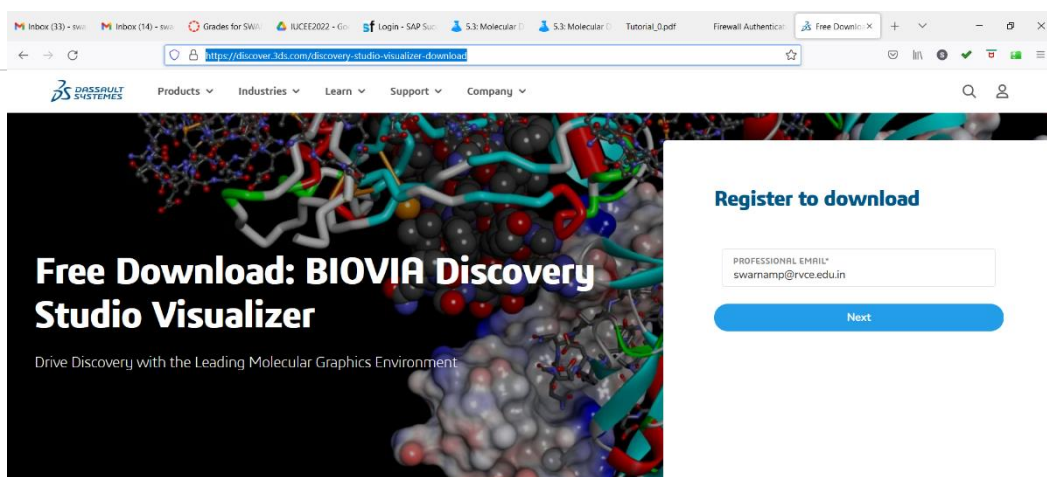
Step 5: Saving the coordinates

### About the software

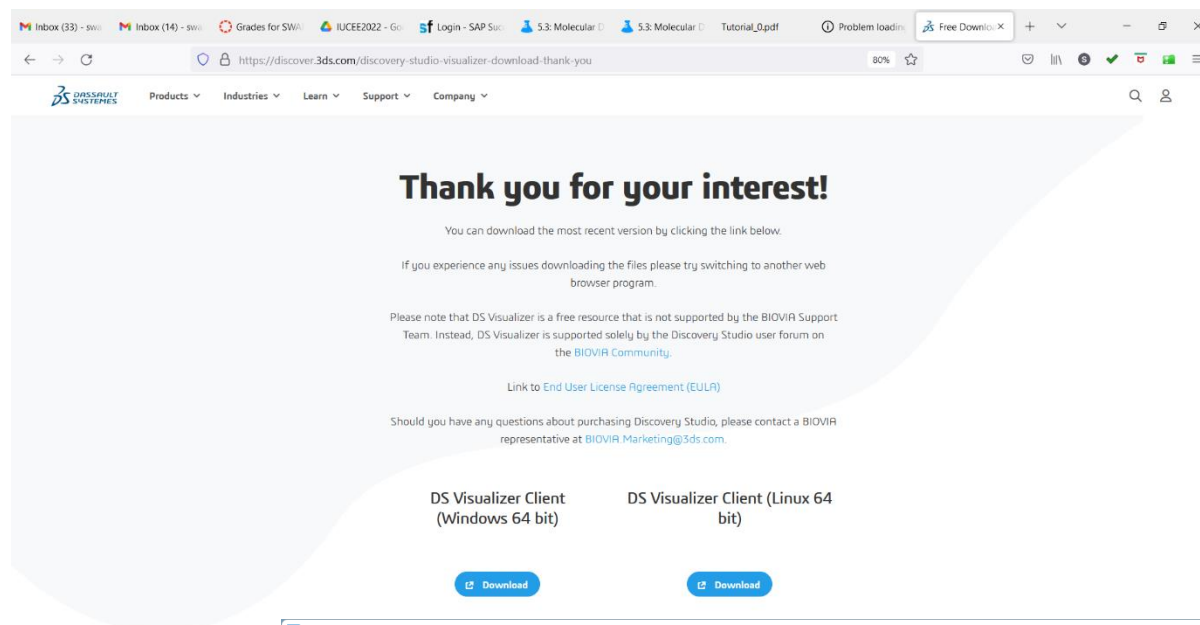
Molecular visualization is a key aspect of the analysis and communication of modeling studies. BIOVIA Discovery Studio is a comprehensive suite of validated science applications built on BIOVIA Pipeline Pilot. The software delivers a unique blend of open, scalable, collaborative research tools designed for today's Life Sciences discovery research needs.

### Installation

Register to download the file for installation (<https://discover.3ds.com/discovery-studio-visualizer-download>)



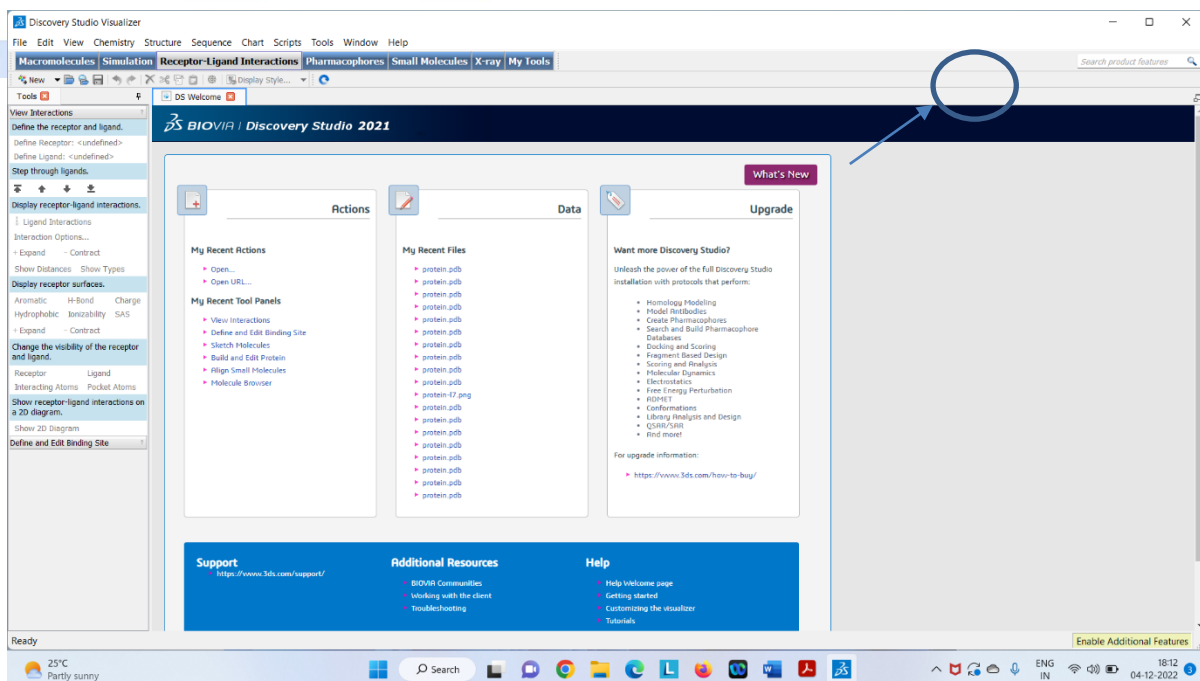
After registering with official email id it will give option to select for Winows/Linux operating system you are interested to download. Click on the required operating system, the file will download. The file will be saved in the download folder.



**Drawing the structure using user graphics interface**

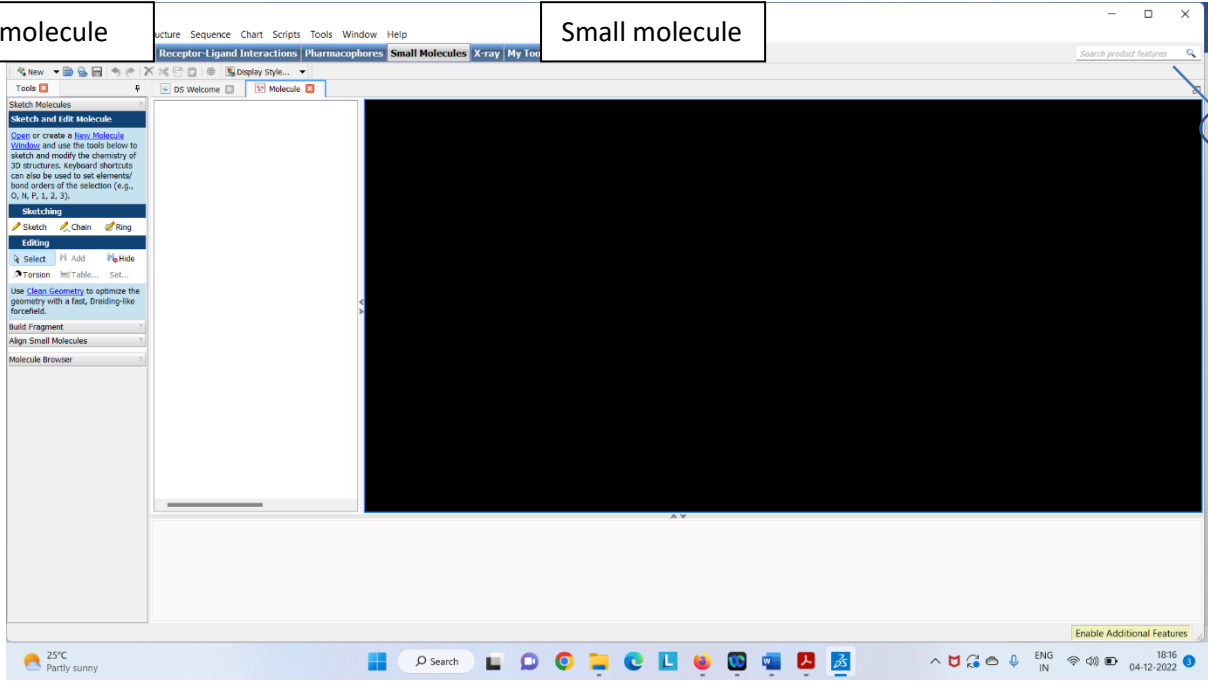
Double click on the Discovery studio icon to open.

**New** Click on new to open new window for drawing the structure.



Sketch molecule

Small molecule



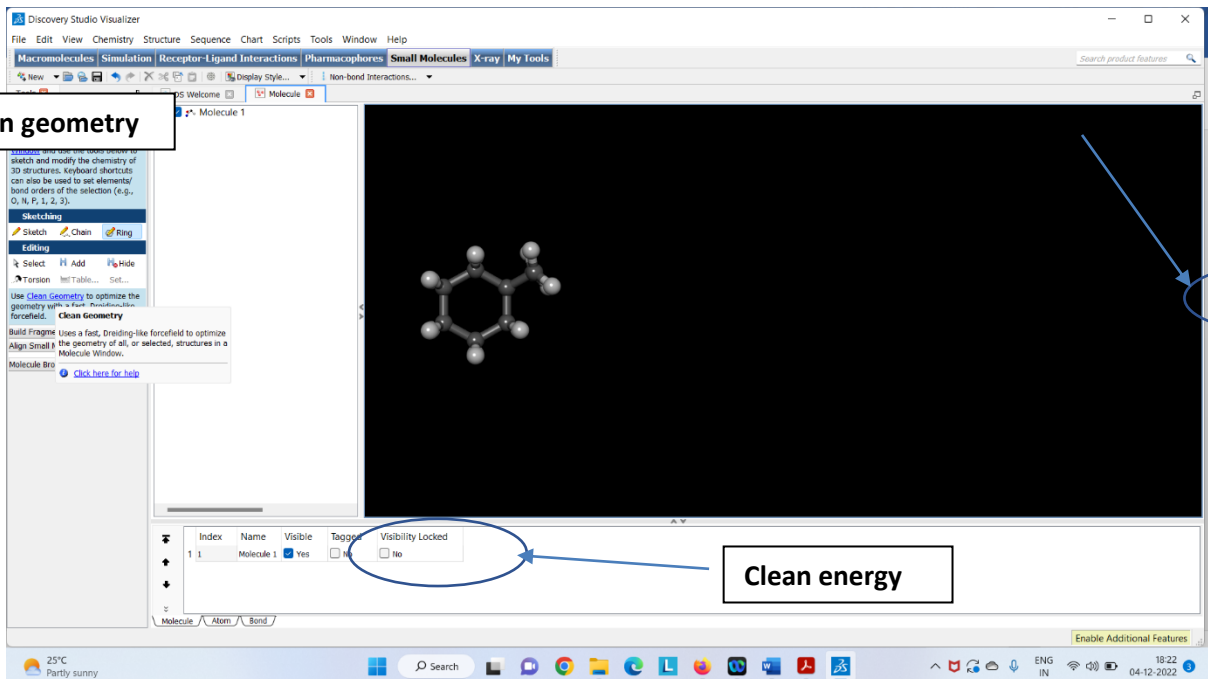
Click on small molecules to draw structures. To edit or sketch the molecule use the options given in left hand side. Change the atoms and bonds as required for

calculation and add Hydrogen.

## Energy optimization

Clean geometry

Clean energy

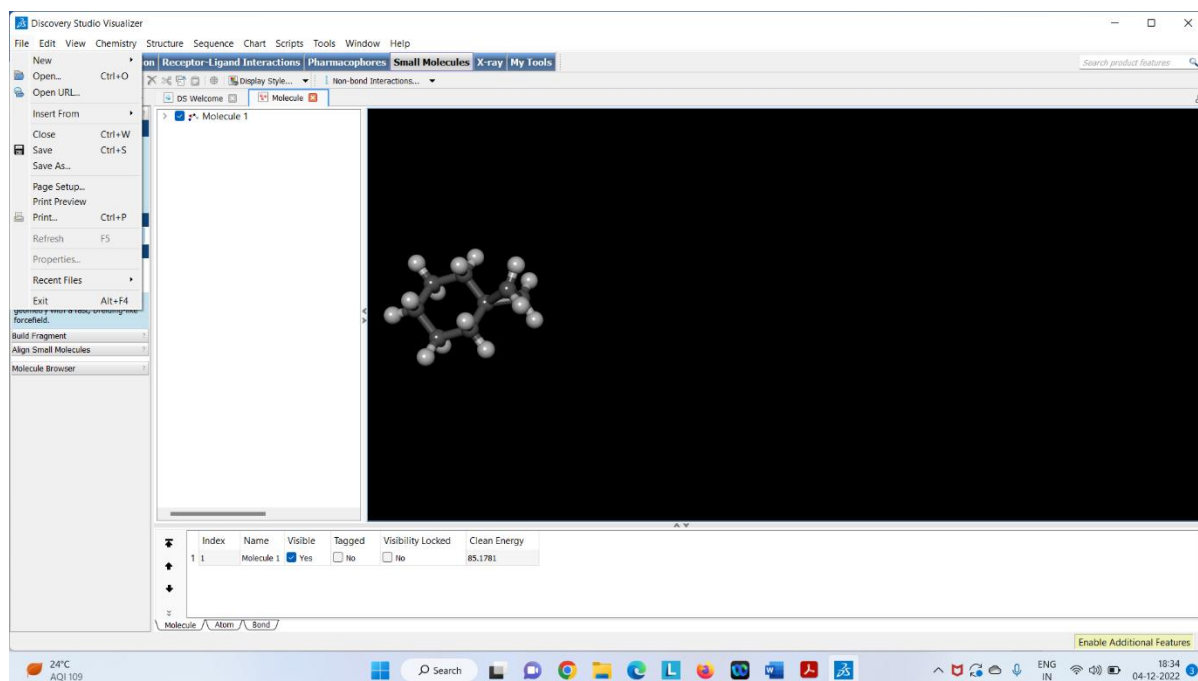


click on clean geometry to optimize the structure. As you click on clean geometry observe the energy change under clean energy. If there is not much

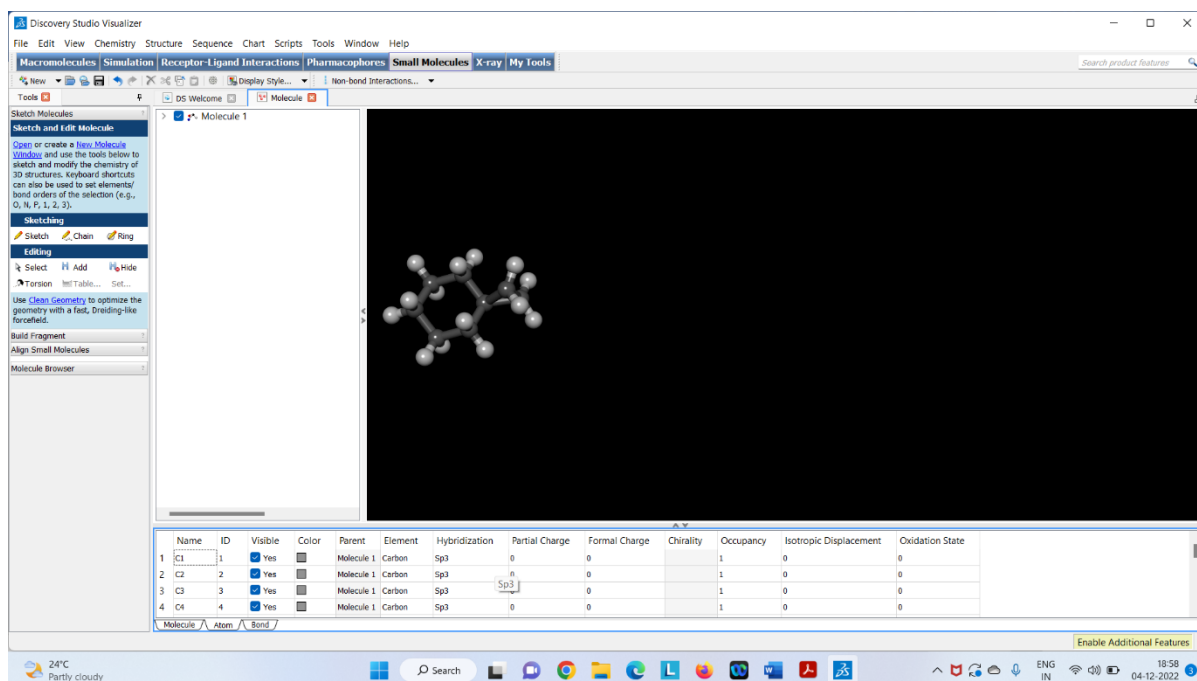
change in energy then understand the energy is optimized.

## Saving the coordinate

Once the energy optimization is done click on file and select save as.

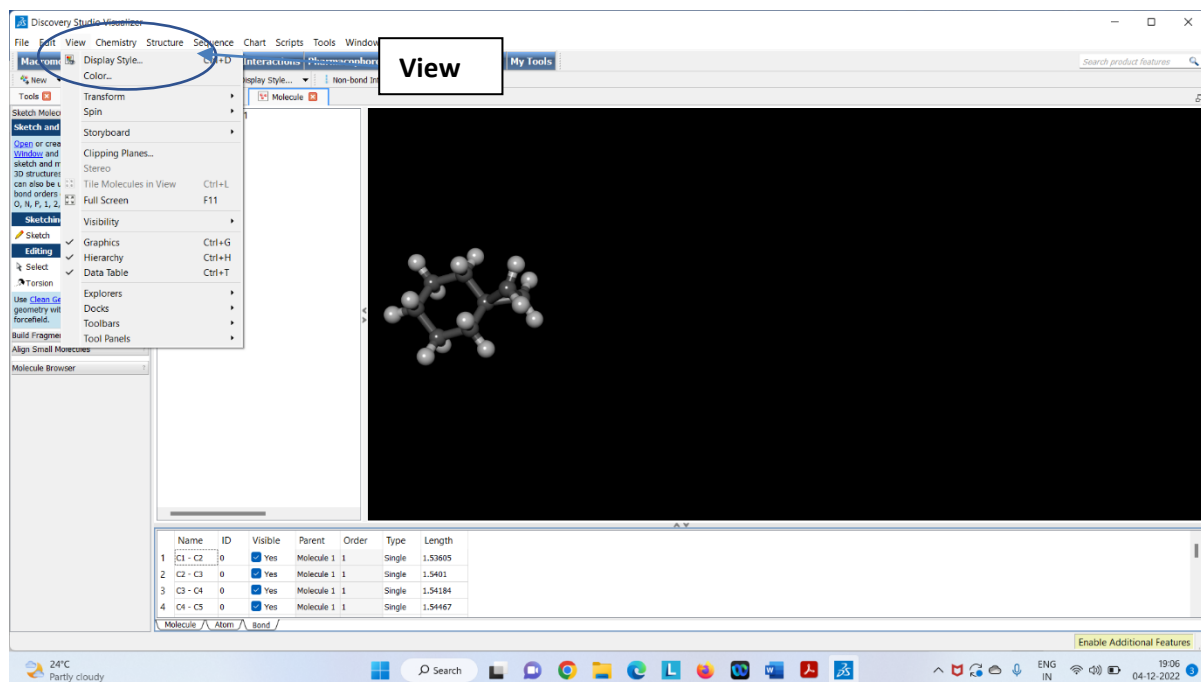


Depending on the software where you want to use the input select the output file type. After saving the output file, you can open the file and see how the cartesian coordinates are written. Further to know about the details of the structure click on atom, so that it will give each atom type, hybridization, oxidation state, partial charge etc. Similarly, click on bond to see the bond order and bond length.



## Visualisation

Now, that you have drawn the structure visualize it using different options. Click on view to change color and display type.



Change the display style and color as per

requirement and save it as picture to use for presentation or publication.

## Data analysis

Excel or any other similar tool like origin or xmgrace (unix) can be used to plot the graph. Take any sets of data arbitrarily and try to plot as line graph, bar graph or different types of graphs. In excel go to insert and click on graph and see the different options available to plot graphs

## Expected to do the following

- Draw any five-chemical structure, optimize the geometry, visualize in different options, view the details of atoms and bonds
- Make a summary report of your all observations with pictures
- Plot 3 different types of graphs using any available data or your own data. Explain scientifically which type of data you can use for each graph.



A Drawing package is useful to have so molecular, shortened structural and full structural formulae can be inserted into Activity sheets, tests and Prelims, and Powerpoint presentations. It is also possible to generate information such as the systematic name of a compound and its molecular weight using these application programs. This Tutorial gives some of the operations possible in ChemSketch®.

This can be obtained from <http://www.acdlabs.com/download/chemsk.html>

While you have to register for the copy, it is free.

This tutorial gives enough information to get started on and to use ChemSketch® to prepare Teaching and Learning materials, but a more detailed tutorial on its use can also be downloaded from the site.

Once ACD/ChemSketch has been installed on your computer, follow these basic steps to start it:

1. Start Microsoft Windows.

2. Double-click the ChemSketch icon.

-OR-

From the Start/Run menu in the Windows 95/98/2000 or NT taskbar, choose ACD/Labs and then choose the ChemSketch icon.

-OR-

Double-click the program file "chemsk.exe" in the folder where you have installed all ACD software. By default this is ACD50.

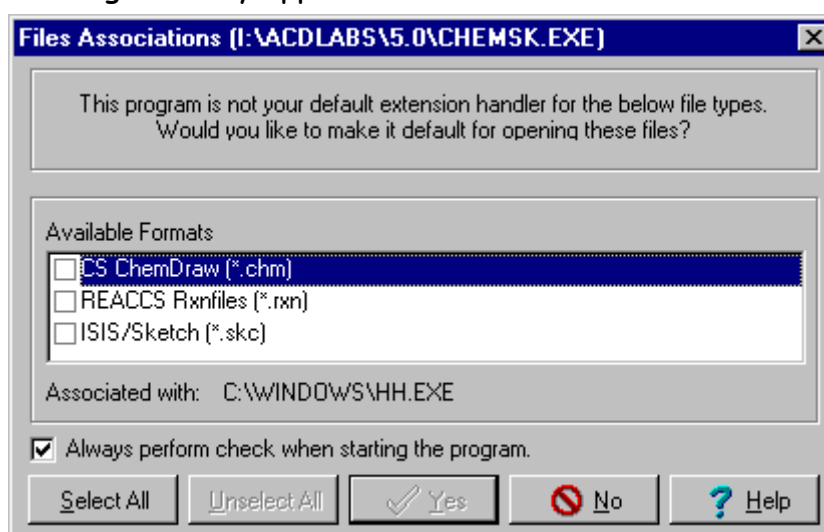
-OR-

If you have other ACD/Labs programs running, from the ACD/Labs menu choose ChemSketch.

You should see an opening splash screen. If this is the freeware version, you will see the **ACD/Labs Products** screen. Click **OK** to close it. If you wish to suppress this dialog box for the subsequent start-ups, choose **Help > ACD/Labs Products...** and clear the **Show this Screen at Start-up** check box.

### **Starting the program for the first time**

The File Association dialog box may appear.



1. This contains a selectable list of file extensions and file types—CS ChemDraw (\*.CHM), REACCS Rxnfiles (\*.RXN), ISIS/Sketch (\*.SKC) and possibly others—which you may



- want to open automatically with ACD software from now on. If so, click the check boxes of the file formats you want to add, and then click the Yes button.
2. If you do not want to have ChemSketch automatically open files with the listed extension, or are not sure, leave the check boxes blank and click the No button.
  3. Then you will see a Tip of the Day box, which you can close after reading.

## **Changing File Associations**

If you have not selected all formats, the default file association can be viewed or changed at any time by going to the File menu and selecting File Association. If you have selected all formats, then you receive a message, "all supported file types are already associated with the current application." In this case, to change the file associations, you can do it through Windows Explorer.

1. Open Windows Explorer, and select a file with the extension for which you want to create the association.
2. Hold down the SHIFT key and right-click on the file. From the pop-up menu, select Open With...
3. Set the application that should be used to open the file and select the Always use this program... check box.
4. Click OK and close Windows Explorer.

## **Structure and Draw Mode icons**

When ACD/ChemSketch starts up, you will find many menu commands and toolbar buttons appear dimmed (inactive). They will be made available as soon as you draw a structure.

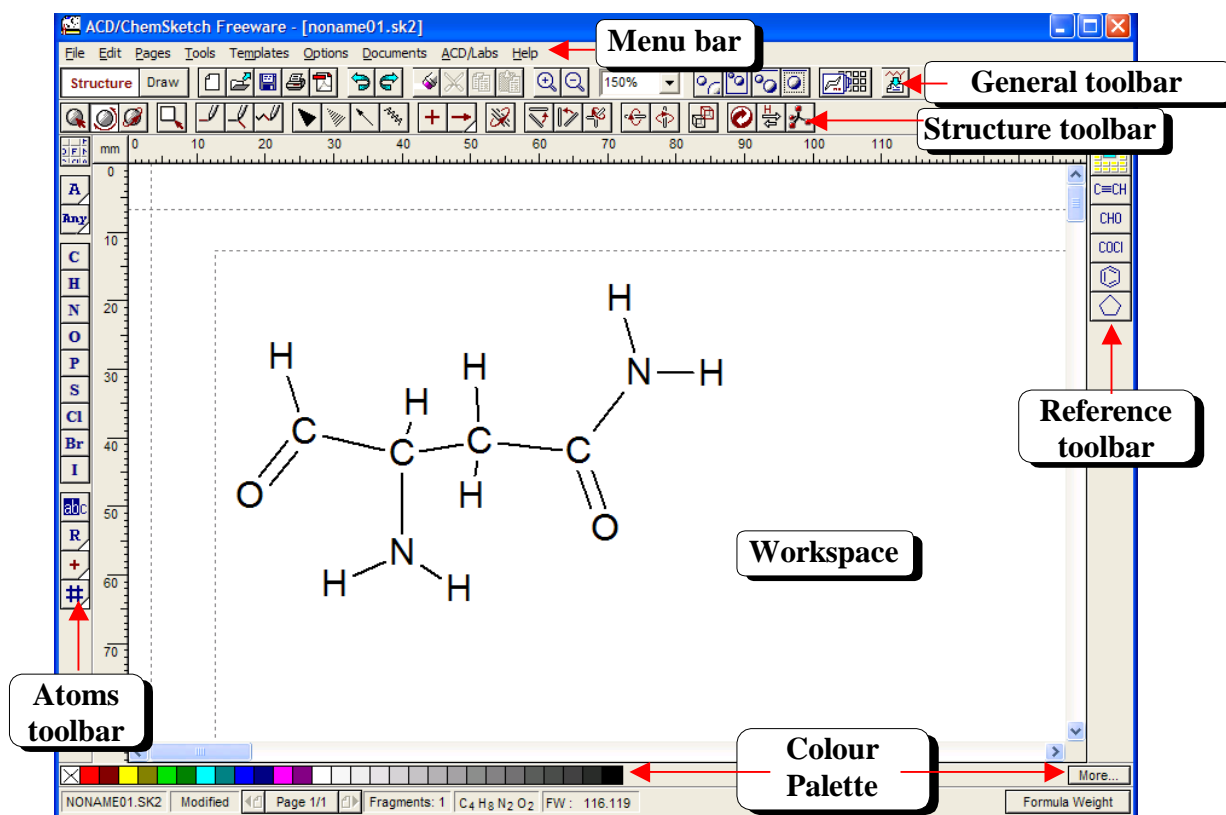
In the ChemSketch window, there are two modes, Structure and Draw. You switch between them using the buttons in the upper left-hand corner:



In the Structure Mode you draw structures and reaction schemes while the Draw Mode presents you with the tools for entering text and drawing various graphical objects.

## Toolbars in Structure Mode Screen

Below you can see the screen with the Structure Mode enabled. The names and positions of some of the toolbars are shown.



The **Structure toolbar** contains tools for drawing and manipulating chemical structures.

The **Atoms toolbar** is displayed vertically to the left of the screen and contains buttons representing atoms, as well as tools for changing atom properties (charge, valence, numbering, etc.). Simply click an atom if you wish to display it in a structure.

**Workspace** is the open area in the middle where structures are drawn.

**Colour Palette** at the bottom of the workspace allows you to quickly colour atoms and bonds in the selected chemical structures.

The **Reference toolbar** is placed to the right of the window and contains the Table of Radicals and various buttons representing ready-made structures and radicals you can take from the table.

## Some useful Buttons in Structure mode

### The 'Undo' Button:



It is not uncommon, especially when first using the Drawing Package to make mistakes when drawing structures.

This button can be a lifesaver! It resets the ChemSketch screen to exactly what it was before you made the mistake.

Note: as soon as the 'Undo' button is clicked, the 'Redo' button next to it becomes active. This button will allow you to move forward again if you undo too many steps.

### The 'Full Page' button:



This allows you to see how the page will look in a printout. If necessary move the objects on the page to arrange them properly.

### The 'Template' button:



This button brings up a list of templates such as Lewis dot diagrams, Lab equipment, Orbitals, Hazard Symbols etc which you can copy and paste into a ChemSketch® page.

### The 'Optimization (or Clean)' button:



This will optimize bond angles etc after you have altered a structure so that it looks more viable.

### The 'Delete' button:



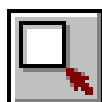
This allows you to 'rub out' parts of molecules or even whole structures as required.

### The 'Selector' button:

Lasso selector



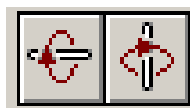
Rectangle selector



Select atoms, bonds and fragments by using either of these selectors.  
fragment(s), click anywhere on an empty space.

To deselect

### The 'Flip' buttons:



These will flip the structure Top to Bottom or Left to Right.

**The 'Move' buttons:**

Once a fragment has been selected, you can move it in these three ways.

**The 'Drawing' buttons:**

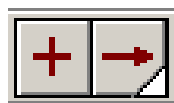
These allow the structures to be drawn in the format chosen.

**The 'Bond' buttons:** These allow **stereo bonds**

or **coordinate bonds**



to be drawn.

**The 'Reaction' buttons:**

Use these to give the plus sign and the arrow in equations. Clicking on the small white rectangle in the bottom right corner of the arrow icon gives various types of reaction arrows to choose from.

**The 'Change Position' button:**

This allows you to change the orientation of groups in a chain.

**The '3-D Optimization' button:**

This allows you to rotate a structure in 3-D.

