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# Laboratory Manual

ENGINEERING CHEMISTRY LAB MANUAL  
CODE NO : CYUBLB3  
(B.Tech 1<sup>st</sup> Semester)

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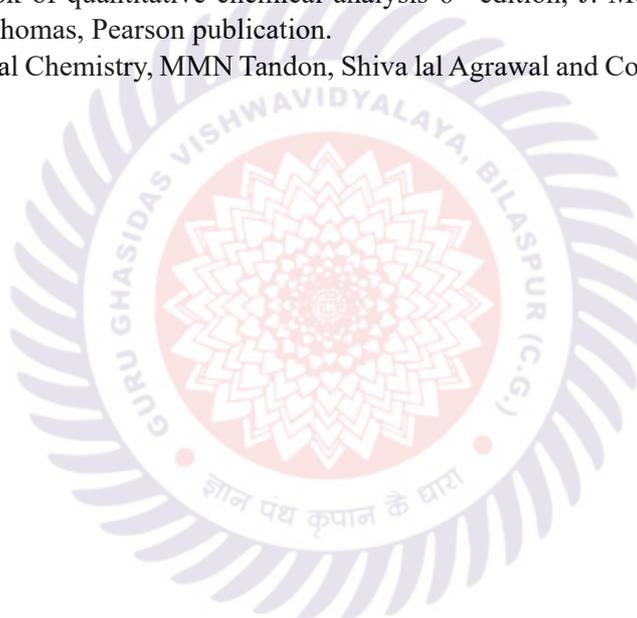
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#### REFERENCE :

1. College Practical Chemistry, VK Ahluwalia, S Dhingra, A Gulati, Universities Press.
2. Advanced Practical Inorganic Chemistry, OP Agrawal, Krishna Publication.
3. Vogel's textbook of quantitative chemical analysis 6<sup>th</sup> edition, J. Mendham, RC Denney, JD Barnes, MJK Thomas, Pearson publication.
4. Unified Practical Chemistry, MMN Tandon, Shiva lal Agrawal and Co., Educational publishers.





## Chemistry Laboratory

# SAFETY RULES

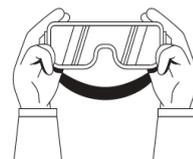


### CLOTHING

Wear appropriate clothing to class and always wear personal protective gear such as safety goggles, lab aprons or coats, and protective gloves.

### READ FIRST

Read through the instructions first and prepare the materials needed before starting any activity.



### FOLLOW INSTRUCTIONS

Follow verbal and written instructions carefully. If you are unsure about any part of the activity, ask your teacher before taking any action.

### MONITOR

Personally monitor all experiments and do not leave them unattended.

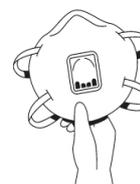


### DON'T EAT OR DRINK

Do not eat, drink, or chew gum in the lab. Do not bring in food or drinks and do not store or place them inside any lab glassware or equipment.

### BE CAREFUL

Handle chemicals, glassware, and equipment with care.



### CHEMISTRY LAB

Do not sniff or taste any chemicals or substances.

### TIDY UP

Keep your work area neat and tidy at all times. Store bags, purses and books in appropriate areas and keep the aisles clear.

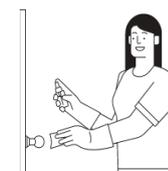


### SAFETY EQUIPEMENT

Know where the safety equipment are located and know what to do in the event of an incident. Immediately report spills or accidents to the teacher.

### BE RESPONSIBLE

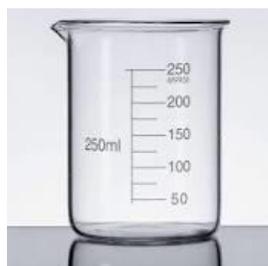
Use the lab responsibly. Only conduct authorized experiments under the supervision of a teacher.



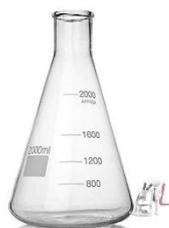
## GENERAL CHEMISTRY LABORATORY RULES

- Never eat or drink in the lab. Food may pick up toxic chemicals.
- Keep the apparatus scrupulously clean.
- Keep the reagents in their proper places after use. Do not alter their position.
- Do not contaminate the reagents.
- Never taste any chemical. Some chemicals are very corrosive and poisonous in very small quantities.
- Keep gas taps and water taps closed when not in use.
- Use either spirit lamp or candle to light the burner.
- Never put anything back into a reagent bottle. Once a reagent has passed the mouth of its container, it has passed the point of no return.
- Always take as little of a chemical as possible. Use only clean, dry spatulas for removing chemicals from bottles. Properly dispose of excess chemicals.
- Do not throw any waste paper /litmus paper etc. into the sink. Throw them into the dust bin.
- Rinse spills off skin immediately. Rinse off any chemicals spilled on the skin immediately with large amounts of water.
- Do not pour concentrated acids into the sink. If they are to be poured, flush them with water.
- To be a better analyst, understand the theory of the experiments you conduct.
- Record your observations as and when you proceed (and not after completion) in a note book and keep it away from reagents and sink.
- Properly dispose of waste chemicals. Certain liquids can be poured into the sink and flushed with water while others are poured into designated waste containers. Most solid wastes are placed in designated crocks. Your instructor will provide disposal instructions each lab.
- Wear a Lab Coat and Safety Goggles and also have a Lab Manual and Calculator while performing experiments in the laboratory. Without them, you are not allowed to enter the lab section.
- Eating, Drinking, Smoking, and Cellphones are Forbidden in the laboratory at all times. Avoid unnecessary movement and talk in the laboratory.
- Any accident involving even the most minor injury must be reported to the lab assistants.
- Do not attempt any unauthorized experiment. Perform only lab operations and activities

## COMMON LABORATORY EQUIPMENTS



**BEAKER :** A beaker is a cylindrical glass or plastic vessel used for holding liquids. It is a multi- purpose piece of equipment used for containing a chemical reaction, measuring liquids, heating them over a Bunsen burners flame or collecting them in a titration experiment.



**ERLENMAYER FLASK :** Erlenmayer flasks are conical shaped flasks, which are made of Pyrex, are safe for chemical storage, heating solutions and recrystallizations. Because of its conical shape they are best for using with equipment's with stopcocks such as separatory funnels and burettes. They are also, like beakers, for containing and transferring liquids not for making measurements



**GRADUATED CYLINDER :** Graduated cylinders are used for measuring the volumes of liquids from a few millilitersto many liters. It is important to choose the graduated cylinder according to the amount of liquid to be measured for more accurate measurements. Always read meniscus point for graduated cylinders, pipettes, volumetric flasks and burettes.



**VOLUMETRIC FLASK :** Volumetric flasks are used for measuring very precise and accurate amounts of aliquid and is used for such when the amount is too much for pipette or burette. They are also used for solution preparation.



### **Pipettes**

**PIPETTE :** Pipette is a glass tube used for the delivery of a measured quantity of liquids.



**BURETTE :** Burette is a vertical cylindrical piece of laboratory glassware with a volumetric graduation on its full length and a stopcock on the bottom. It is used to dispense known amounts of a liquid reagent in a titration experiment.



**STAND :** A metal rod attached to a heavy metal base. The heavy base keeps the stand stable, and the vertical metal rod allows for easy height adjustment of the iron ring/clamp.



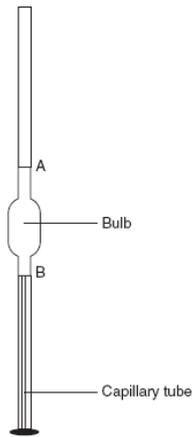
**FUNNEL :** Funnels are used for pouring liquids from one container to another. In addition, with the aid of filter paper, they can be used as separation devices to separate liquids from solids. It is fixed by a ring support on a stand.



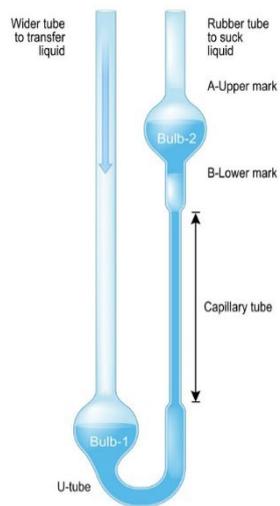
**Filter paper :** paper is used to separate solid particles from liquids. They can have different size with different pore size. Solids that remain on filter paper can later be dried on a watch glass or in an oven.



**WATCH GLASS :** Watch glass is used to allow crystal to dry after they have been filtered. They can be used as an evaporating surface or to cover a beaker that can be heated to very high temperatures.



**STALAGNOMETER** : A stalagmometer is a glass instrument that measures the surface tension of a liquid.



**VISCOMETER** : Instrument for measuring the viscosity (resistance to internal flow) of a fluid.



## SECTION-A

### Iodometric titration of potassium dichromate and sodium thiosulfate

#### Theory:

In this reaction, the strength of sodium thiosulfate is determined by titration with standardized potassium dichromate. This is oxidation reduction as well as iodometric titration.

Titration involving iodine or dealing with iodine liberated in chemical reaction is called iodometric and iodimetric titration respectively. This reaction is iodometric because iodine comes from KI.

Here  $K_2Cr_2O_7$  is an oxidizing agent while iodine is a reducing agent. In last step of reaction, iodine is oxidizing agent while thiosulfate ion is reducing agent. In the 2<sup>nd</sup> step of reaction, specific indicator is used that is **Starch** which has significant relation with iodine.

If to a solution containing a little iodine, some starch is added, the colour of the solution turns blue and if to this solution,  $Na_2S_2O_3$  is run in from burette, the blue color of starch iodine complex will disappear from the solution as soon as all the iodine has been reduced to iodine ion.

#### Chemical reaction:



#### Chemicals required:

1.  $Na_2S_2O_3/NaHCO_3$
2.  $K_2Cr_2O_7$
3. KI
4.  $H_2SO_4$
5. Distilled water
6. Starch

#### Procedure:

1. Prepare 10% solution of  $K_2Cr_2O_7$  by dissolving 10g of potassium dichromate in 100ml of water.
2. Add 10ml of potassium dichromate in a flask and add half test tube of  $H_2SO_4$  in it.
3. Add 10-20ml of 5% KI solution in above flask and dilute the mixture of flask with 15-20ml of distilled water.
4. Titrate that solution against  $Na_2S_2O_3$ .
5. When colour of solution changes to yellow, use 1ml of starch as an indicator.
6. Appearance of blue colour indicate end point. Note the volume of sodium thiosulfate used till end point.
7. Repeat the process thrice for better results.

#### Blank Experiment (procedure):

1. Take 10ml of distilled water in place of potassium dichromate, and add half test tube of sulphuric acid.
2. Add 10-20ml of 5%KI solution in the above flask and dilute the solution mixture of flask with 15-20ml of distilled water.
3. Titrate the solution against  $Na_2S_2O_3$ .
4. When colour of the solution changes to transparent, use 1ml of starch as an indicator.

5. Appearance of milky colour indicate end point. Note the volume of sodium thiosulfate used till end point.
6. Repeat the process thrice for better results.
7. Take difference of volume of sodium thiosulfate used in procedure of potassium dichromate and blank experiment and use that volume in calculations.

### Calculations:

Volume of  $K_2Cr_2O_7$  used = 10ml

Sr no.	Burette reading (Initial)(ml)	Burette reading (Final) (ml)	Final Volume (ml)
1.			
2.			
3.			

Volume of  $Na_2S_2O_3$  used for burette according to experimental values =  $V_s$  = ml

### Blank Experiment:

Sr no.	Burette reading (Initial)(ml)	Burette reading (Final) (ml)	Final Volume (ml)
1.			
2.			
3.			

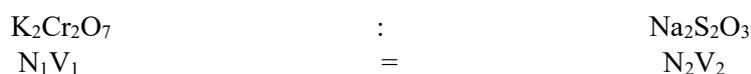
Average volume of  $Na_2S_2O_3$  used during blank experiment =  $V_y$  = ml

Volume of  $Na_2S_2O_3$  used =  $V_s - V_y$  = ml

Volume of  $K_2Cr_2O_7$  =  $V_1$  = 10 ml

Normality of  $K_2Cr_2O_7$  =  $N_1$  =

Normality of  $Na_2S_2O_3$  =  $N_2$  = ?



### Results:

The strength of  $Na_2S_2O_3$  solution is = g/L

### Precautions :

1. Keep your eye in level with the liquid surface while taking the burette reading or while reading the pipette or measuring flask etc.
2. Always read lower meniscus in case of colourless solution and upper meniscus in case of coloured solutions.
3. Do not blow through the pipette to expel the last drop of solution from it, simply touch the inner surface of the titration flask with the nozzle of the pipette for this purpose.

**To determine the Normality and Strength (g/L) of given Ferrous Ammonium Sulfate solution 'A' using standard Ferrous Ammonium Sulfate (N/30) solution 'B' taking KMnO<sub>4</sub> solution as an intermediate.**

**Theory:** Potassium permanganate is a strong oxidant in the presence of sulfuric acid. Mohr salt is a double salt forming a single crystalline structure having the formula FeSO<sub>4</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·6H<sub>2</sub>O. The chemical name for Mohr's salt is ferrous ammonium sulfate.

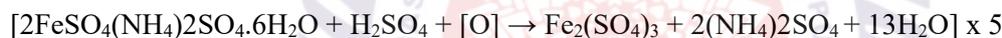
In this titration Mohr salt acts as a reducing agent and potassium permanganate acts as an oxidizing agent. So, the reaction between Mohr's salt and potassium permanganate is a redox reaction. In this redox reaction, ferrous ion from Mohr's salt gets oxidized and pink colored of manganese present in potassium permanganate, which is in the +7 oxidation state gets reduced to colorless Mn<sup>2+</sup> state.

**The chemical reaction and the molecular chemical equation is given below.**

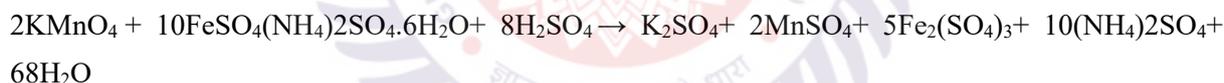
**Reduction half reaction –**



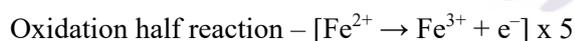
**Oxidation half reaction –**



**Overall reaction –**



**The ionic equation involved in the process is given below.**



This titration is based upon oxidation-reduction titrations. When ferrous ammonium sulfate solution is titrated against potassium permanganate in the presence of acidic medium by sulfuric acid. Acidic medium is necessary in order to prevent precipitation of manganese oxide. Here KMnO<sub>4</sub> acts as a self indicator and this titration is called permanganate titration.

**Materials Required:**

1. Mohr's salt (ferrous ammonium sulfate)
2. Potassium permanganate solution

3. dilute sulfuric acid
4. Chemical balance

### Apparatus Setup:

1. In burette –  $\text{KMnO}_4$  solution
2. In Conical flask – 10ml of Ferrous Ammonium Sulfate (Mohr's salt) + Sulfuric acid
3. Indicator – Self indicator ( $\text{KMnO}_4$ )
4. End Point – Colourless to permanent pale pink color.

### Procedure:

#### A. Preparation of N/30 standard solution of ferrous ammonium sulfate:

The molar mass of Mohr's salt = 392 g/mol

$$\begin{array}{l} \text{Strength} = \text{Normality} \times \text{Equivalent weight} \\ = (N/30) \times 392 = 11.76 \end{array}$$

For preparing 250ml of N Mohr's salt solution, Mohr salt required is  
 $= 11.76/1000 \times 250 = 2.94 \text{ gm}$

1. Weigh an empty watch glass using a chemical balance.
2. Weigh accurately 2.94 gm of Mohr's salt in a chemical balance.
3. With the help of a funnel, transfer the Mohr's salt into the measuring flask.
4. Now wash the funnel with distilled water without removing the funnel from the flask.
5. Make the solution up to the marked point with distilled water and make sure the Mohr's salt is fully dissolved.
6. This solution is N/30 standard solution of Mohr's salt.

#### B. Titration of potassium permanganate solution against N/30 standard ferrous ammonium sulfate (Mohr's salt) solution:

1. Wash and rinse the burette and pipette with distilled water and then rinse with the corresponding solution to be filled in them.
2. Rinse the burette with the potassium permanganate solution and fill the burette with potassium permanganate solution.
3. Fix the burette in the burette stand and place the white tile below the burette in order to find the endpoint correctly.
4. Rinse the pipette and conical flask with standard ferrous sulfate solution.
5. Pipette out 10ml of N/30 standard Mohr's salt solution into the conical flask.
6. Add a test tube full of sulfuric acid in order to prevent oxidation of manganese to form manganese dioxide.
7. Note down the initial reading in the burette before starting the titration.
8. Now start the titration, titrate against potassium permanganate solution and simultaneously swirl the solution in the flask gently.
9. Initially, the purple colour of  $\text{KMnO}_4$  is discharged with ferrous ammonium sulfate. The appearance of a permanent pink colour reveals the end point.
10. Repeat the titration until concordant values are obtained.
11. Note down the upper meniscus on the burette readings.

- Record the reading in the observation table given below in order to calculate the molarity of  $\text{KMnO}_4$  given.

**C. Titration of potassium permanganate solution against given ferrous ammonium sulfate (Mohr's salt) solution:**

- Wash and rinse the burette and pipette with distilled water and then rinse with the corresponding solution to be filled in them.
- Rinse the burette with the potassium permanganate solution and fill the burette with potassium permanganate solution.
- Fix the burette in the burette stand and place the white tile below the burette in order to find the endpoint correctly.
- Rinse the pipette and conical flask with given ferrous sulfate solution.
- Pipette out 10ml of given standard Mohr's salt solution into the conical flask.
- Add a test tube full of sulfuric acid in order to prevent oxidation of manganese to form manganese dioxide.
- Note down the initial reading in the burette before starting the titration.
- Now start the titration, titrate against potassium permanganate solution and simultaneously swirl the solution in the flask gently.
- Initially, the purple colour of  $\text{KMnO}_4$  is discharged with ferrous ammonium sulfate. The appearance of a permanent pink colour reveals the end point.
- Repeat the titration until concordant values are obtained.
- Note down the upper meniscus on the burette readings.
- Record the reading in the observation table given below in order to calculate the normality of given .

**Observations:** Titration of potassium permanganate solution against N/30 standard ferrous ammonium sulfate (Mohr's salt) solution:

S.No	Volume of ferrous ammonium sulfate (Mohr's salt) used	Burette Reading		Volume(V) of $\text{KMnO}_4$ used $V = (y-x)\text{ml}$
		Initial(x)	Final(y)	

**Observations:** Titration of potassium permanganate solution against given ferrous ammonium sulfate

(Mohr's salt) solution:

S.No	Volume of ferrous ammonium sulfate (Mohr's salt) used (given)	Burette Reading		Volume(V) of KMnO <sub>4</sub> used V = (y-x)ml
		Initial(x)	Final(y)	

**Calculations:**

(a) Normality of KMnO<sub>4</sub> solution:

Consider y ml of given KMnO<sub>4</sub> solution is equivalent to 20ml of N/10 Mohr's salt solution.

According to law of equivalents,

$$N_1V_1 = N_2V_2$$

- N<sub>1</sub>, N<sub>2</sub> are normality of Mohr's salt and KMnO<sub>4</sub> solution respectively.
- V<sub>1</sub>, V<sub>2</sub> are volumes of Mohr's salt and KMnO<sub>4</sub> respectively.

$$1/10 \times 20 = N_2 \times y$$

$$N_2 = 2/y$$

$$N = \text{Normality of given KMnO}_4 \text{ solution} = 2/y$$

(b) Normality of given Ferrous ammonium sulfate solution:

$$NV = N_3V_3$$

- N<sub>3</sub>, N are normality of given Mohr's salt and KMnO<sub>4</sub> solution respectively.
- V<sub>3</sub>, V are volumes of given Mohr's salt and KMnO<sub>4</sub> respectively.

$$N_3 = V_3/NV$$

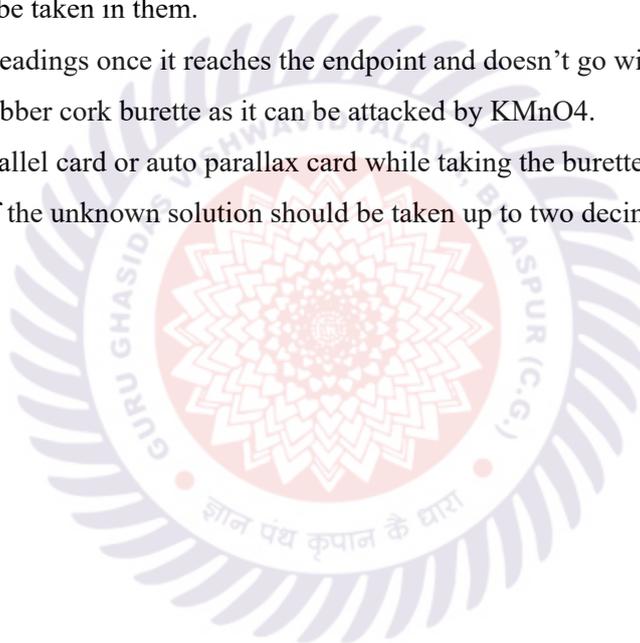
(c) Strength of given Ferrous ammonium sulfate solution:

Strength = Normality x Equivalent mass

**Results and Discussion:** The Normality and Strength (g/L) of given Ferrous Ammonium Sulfate solution is

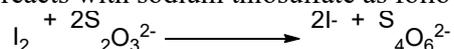
**Precautions:**

1. Potassium permanganate is dark, so always read the upper meniscus.
2. Rinse the pipette and burette before use.
3. Use dilute sulfuric acid for acidifying the potassium permanganate.
4. Clean all the apparatus with distilled water before starting the experiment and then rinse with the solution to be taken in them.
5. Take accurate readings once it reaches the endpoint and doesn't go with average readings.
6. Do not use a rubber cork burette as it can be attacked by  $\text{KMnO}_4$ .
7. Use the antiparallel card or auto parallax card while taking the burette readings.
8. The strength of the unknown solution should be taken up to two decimal places only.



**To determine the concentration of hypo solution ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) iodometrically with given Iodine (N/50) solution.**

**Theory :** Iodine reacts with sodium thiosulfate as follows :



**Chemical required :**

1. Given sodium thiosulfate solution
2. Iodine solution
3. Distilled water
4. Starch solution (10%)

**Procedure :**

1. Rinse and fill the burette with the given sodium thiosulfate solution.
2. Pipette out 20ml of the provided Iodine solution into a conical flask.
3. To it add 20ml of water.
4. Keep the flask below the nozzle of the burette.
5. Now run down sodium thiosulfate solution from the burette into the conical flask till a light yellow colour is obtained.
6. Now add 2-3ml of starch solution.
7. A deep blue colour is obtained.
8. Again, add sodium thiosulfate solution drop by drop till the blue colour just disappears.
9. Repeat the titration till concordant values are obtained.

**Calculation :**

Suppose 20ml of Iodine solution = V ml of Hypo solution

$$20 \times \text{N}/50 = V \times \text{N}_{\text{Na}_2\text{S}_2\text{O}_3}$$

$$\text{Or } \text{N}_{\text{Na}_2\text{S}_2\text{O}_3} = 2\text{N}/5\text{V}$$

**Result :** The Normality and strength of given hypo solution is .....

**Precautions:**

1. Keep your eye in level with the liquid surface while taking the burette reading or while reading the pipette or measuring flask etc.
2. Always read lower meniscus in case of colourless solution and upper meniscus in case of coloured solutions.
3. Do not blow through the pipette to expel the last drop of solution from it, simply touch the inner surface of the titration flask with the nozzle of the pipette for this purpose.

**Find out the Temporary hardness of given water sample using 0.01M EDTA solution, buffer solution (pH-10) and EBT as an indicator.**

### **Principle:**

Hardness in water is due to the presence of dissolved salts of calcium and magnesium. It is unfit for drinking, bathing, washing and it also forms scales in boilers. Hence it is necessary to estimate the amount of hardness producing substances present in the water sample. Once it is estimated, the amount of chemicals required for the treatment of water can be calculated.

The estimation of hardness is based on complexometric titration. Hardness of water is determined by titrating with a standard solution of ethylene diamine tetra acetic acid (EDTA) which is a complexing agent. Since EDTA is insoluble in water, the disodium salt of EDTA is taken for this experiment. EDTA can form four or six coordination bonds with a metal ion.

### **1. Total hardness**

Total hardness is due to the presence of bicarbonates, chlorides and sulphates of calcium and magnesium ions. The total hardness of water is estimated by titrating the water sample against EDTA using Eriochrome Black-T (EBT) indicator. Initially EBT forms a weak EBT-Ca<sup>2+</sup>/Mg<sup>2+</sup> wine red coloured complex with Ca<sup>2+</sup>/Mg<sup>2+</sup> ions present in the hard water. On addition of EDTA solution, Ca<sup>2+</sup>/Mg<sup>2+</sup> ions preferably forms a stable EDTA-Ca<sup>2+</sup>/Mg<sup>2+</sup> complex with EDTA leaving the free EBT indicator in solution which is steel blue in colour in the presence of ammonia buffer (mixture of ammonium chloride and ammonium hydroxide, pH 10).

### **2. Temporary hardness**

Temporary hardness is due to the presence of bicarbonates of calcium and magnesium ions. It can be easily removed by boiling. When water is boiled, temporary hardness producing substances (bicarbonates) are precipitated as insoluble carbonates or hydroxides. This precipitate can be removed by filtration. (The filtrate is used in the next step).

### **3. Permanent hardness**

Permanent hardness is due to the presence of chlorides and sulphates of calcium and magnesium ions. This type of hardness cannot be removed by boiling. The filtrate obtained from the above step contains permanent hardness producing substances and is estimated against EDTA using EBT indicator.

### **Chemicals Required :**

1. Distilled water
2. EDTA solution
3. EBT
4. Buffer solution

### **Procedure:**

The burette is filled with standard EDTA solution to the zero level, following usual precautions.

#### **1. Estimation of Total Hardness**

20 ml of the given water sample is pipetted out into a clean conical flask. 5 ml ammonia buffer and 2 drops of EBT indicator are added and titrated against EDTA from the burette. The end point is the change of colour from wine red to steel blue.

The titration is repeated to get concordant titre value.

## 2. Estimation of Permanent Hardness

100 ml of the given sample of water is pipetted out into a clean beaker and boiled for 20 minutes. It is then filtered to remove the precipitate formed due to the decomposition of temporary hardness producing salts. The filtrate is made up to 100 ml in standard measuring flask (SMF) using distilled water. 20 ml of the made up solution is pipetted out into a conical flask, 5 ml ammonia buffer and 2 drops of EBT indicator are added and titrated against the EDTA. The end point is the change of colour from wine red to steel blue. The titration is repeated to get concordant titre value.

## 3. Temporary Hardness

The temporary hardness is calculated from the total and permanent hardness.

Temporary Hardness = Total Hardness – Permanent Hardness

### Result:

The collected water sample contains

Total hardness = ppm

Permanent hardness = ppm

Temporary hardness = ppm

### Observation table:

#### Titration 1 : Estimation of Total Hardness

S No.	Volume of hard water sample (ml)	Burette Reading		Volume of EDTA solution (ml)	Indicator
		Initial	Final		

#### Table 2 : Estimation of permanent hardness

S No.	Volume of boiled water sample (ml)	Burette Reading		Volume of EDTA solution (ml)	Indicator
		Initial	Final		

### Calculation :

1 ml of 0.01M EDTA = 1mg of CaCO<sub>3</sub>

V<sub>1</sub> ml of EDTA = V<sub>1</sub> mg of CaCO<sub>3</sub>

### Calculation of Total hardness :

Volume of EDTA solution consumed =

Volume of hard water taken =

$$\text{Total hardness} = \frac{\text{Volume of EDTA solution consumed} \times 1000}{\text{Volume of the hard water taken}} \text{ ppm}$$

= .....ppm

Calculation of permanent hardness : .....ml  
 Volume of boiled water taken : .....ml

$$\text{Permanent hardness} = \frac{\text{Volume of EDTA solution consumed} \times 1000}{\text{Volume of the boiled water taken}} \text{ ppm}$$

= .....ppm

**Calculation of temporary hardness :**

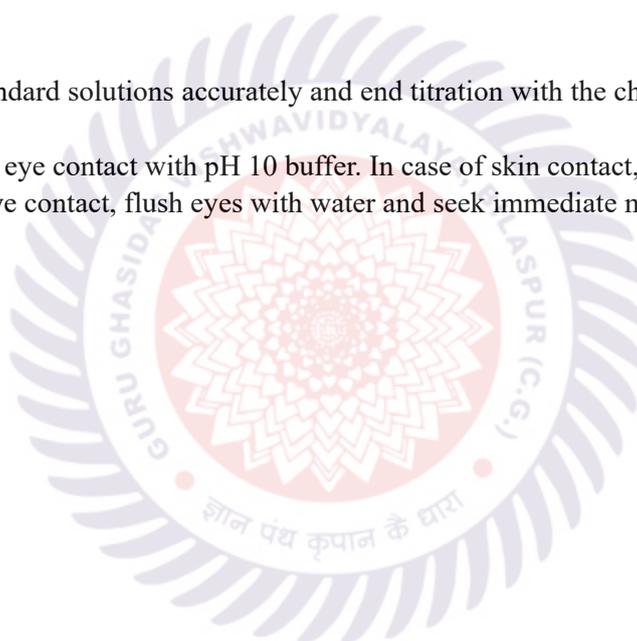
$$\text{Temporary hardness of the given sample of water} = \text{Total hardness} - \text{Permanent hardness}$$

= .....ppm

**Result :** The temporary hardness of the given water sample is .....ppm.

**Precautions :**

1. Prepare the standard solutions accurately and end titration with the change of colour.
2. Avoid skin and eye contact with pH 10 buffer. In case of skin contact, rinse the area for several minutes. For eye contact, flush eyes with water and seek immediate medical advice.

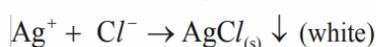


## To determine chloride ion in a given water sample by Argentometric method (Mohr's method)

### Mohr's method:

In Mohr's method, the determination of the end point is based on the formation of a second precipitate which is coloured. The requirement here is that the second precipitate should have solubility slightly greater than the precipitate between the analyte and the titrant. The indicator used is sodium chromate and the second precipitate formed is brick-red coloured silver chromate,  $\text{Ag}_2\text{CrO}_4$ . Volume of 0.001 M  $\text{AgNO}_3$  titrant, On the addition of a solution of silver nitrate to the titrant (containing chloride ions the analyte and chromate ions-the indicator) two precipitates are formed. These are  $\text{AgCl}$  and  $\text{Ag}_2\text{CrO}_4$  respectively.

The reactions can be given as under



The silver chromate having greater solubility stays in solution whereas silver chloride precipitates out. At the end point the excess of silver ions forms the precipitate of silver chromate that imparts distinct colour to the solution thereby marking the end point. The titration is carried out at a pH between 7 to 10. This is so because in acidic solution the chromate ion gets converted into chromic acid and the concentration of chromate ion becomes quite low. It is not sufficient to precipitate the silver ions as silver chromate. On the other hand, at the pH greater than 10 a brown precipitate of hydrated silver oxide is obtained. The pH is maintained in the suitable range by adding sodium bicarbonate to the analyte solution. The Mohr's method suffers with a drawback as it has an inherent titration error. The end point is not observed immediately after the equivalence point but after a little over titration, i.e., after adding a little extra silver nitrate. This necessitates the determination of an indicator blank. This is determined by suspending solid, white, chloride-free calcium carbonate in distilled water and making the volume as that of the titrated solution at the equivalence point. This would have the concentration of  $\text{CrO}_4^{2-}$  equal to that at the equivalence-point. The  $\text{Ag}^+$  reagent is added to this suspension until the end point colour is observed. The volume of  $\text{Ag}^+$  reagent required to match the end point colour is subtracted from the end point volumes measured in the titrations of chloride.

### Material Required :

1.  $\text{NaCl}$  = 10ml (dil solution)
2.  $\text{CaCO}_3$
3.  $\text{NaHCO}_3$  = 10ml (dil solution)
4.  $\text{K}_2\text{CrO}_4$  = 10ml (dil solution)
5.  $\text{AgNO}_3$

### Procedure:

Wash the burette with distilled water and rinse with standardised solution of silver nitrate and then fill the burette with the same.

- Carefully pipette out 10 ml of given solution of chloride ions and transfer into a 100 ml conical flask. Add 1 ml of the potassium chromate indicator solution (it is advisable to use a 1 ml pipette for the purpose).
- Titrate the solution with silver nitrate solution and ensure that the flask is constantly swirled.
  - With the progress of the titration, you would observe that the red colour formed by the addition of each drop of Silver nitrate begins to disappear more slowly; slow down the rate of addition of silver nitrate. The end point is indicated by sudden development of a faint but distinct change in colour to reddish-brown. This does not disappear on swirling the titration flask
- Repeat the procedure for at least three times and record your observation in Observation Table.

**Observation Table :**

SN	Volume of sample solution	Burette initial reading (ml)	Burette final reading (ml)	Volume of AgNO <sub>3</sub>
1	25	0		
2	25	0		
3	25	0		

$$N_1V_1 = N_2V_2$$

$$N_1 = N_2V_2 / V_1$$

N<sub>1</sub> = concentration of chloride ion

V<sub>1</sub> = volume of water sample

N<sub>2</sub> = concentration of AgNO<sub>3</sub>

V<sub>2</sub> = volume of AgNO<sub>3</sub>

$$\text{Strength of chloride ion} = N_1 \times \text{equivalent weight}$$

$$= \dots \times 35.45$$

$$= \dots \text{ g/L}$$

$$\text{Cl}^- \text{ ion concentration in ppm} = \dots \times 1000$$

$$= \dots \text{ ppm}$$

**Result:**

Cl<sup>-</sup> ion concentration in given water sample is .....ppm.

**Precautions :**

1. The pH of the solution must be between 7–8 since chromate ions are converted to something else at lower pH concentrations. Therefore, the desired results are not achieved. So it can be said that pH range is important in determining chloride ions in Mohr's method.
2. A good amount of indicator solution must be used. Otherwise, the endpoint might be achieved too soon, disrupting the chances of getting accurate results.
3. Caution should be taken to notice the colour change of the indicator solution from yellow to brick red, and the tube must be shaken to get accurate results.

## SECTION-B

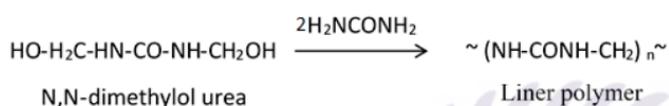
### Preparation of Urea-formaldehyde resins

**Theory :** The two important classes of amino resins are the condensation products of urea and melamine with formaldehyde. Urea formaldehyde resins are formed by condensation of urea and formaldehyde in acidic medium in following steps:

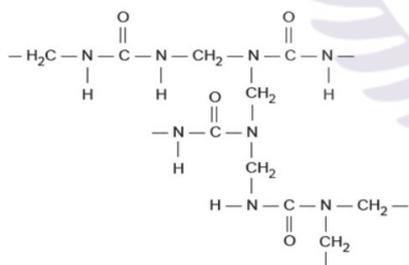
#### Chemistry and Production:



This product condenses with two more urea molecules to form a liner polymer.



Urea is dissolved in aqueous formaldehyde in a throw-away container. Acidification of this solution initiates condensation polymerization, and a hard, white, thermosetting polymer is formed within a few minutes. After washing, the properties of this substance can be investigated. If a mold is used, the experiment could be extended to show the formation of plastic articles made in a mold by condensation polymerization. The reaction is a condensation polymerisation in which water is eliminated as the hydrogen atoms from the ends of one aminogroup from each of two urea molecules combine with the oxygen atom from a formaldehyde molecule. The remaining  $-\text{CH}_2-$  group from the formaldehyde molecule then forms a bridge between two neighboring urea molecules, as shown below. This process, repeated many thousands of times, forms long chains of urea and formaldehyde molecules linked in this way.



**Chemical required:** Urea (2g), 40% aq formaldehyde solution or formalin (5 mL), conc. H<sub>2</sub>SO<sub>4</sub> (3-4 drops).

#### Procedure :

1. Dissolve 5 g of urea in 10 mL of formaldehyde solution (formalin = 37 –40)% , in the disposable container .
2. Add about 0.5 mL of concentrated sulfuric acid H<sub>2</sub>SO<sub>4</sub> a drop at a time, using a dropping pipette, and stir steadily. Within a minute the solution begins to go milky and eventually a hard, white solid is formed which is difficult to remove from the container. A lot of heat is evolved. Show that the polymer is hard by poking the material with a spatula.
3. Wash the polymer thoroughly before passing around the class, as it is likely to be contaminated with unreacted starting materials.

4. Air dry it and weigh.

**Result :** .....gm of Urea-formaldehyde resin was obtained.

**Precautions :**

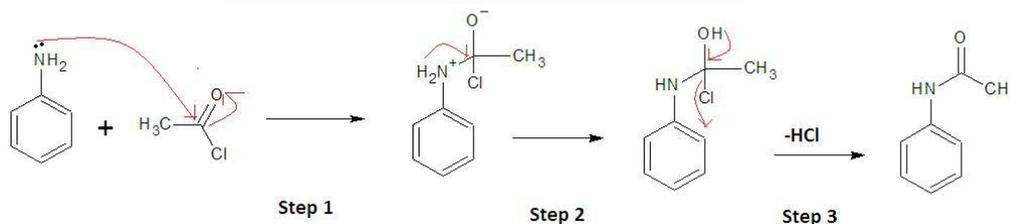
1. While adding concentrated  $H_2SO_4$ , it is better to stay little away from the beaker since the reaction sometimes becomes vigorous.
2. The reaction mixture should be stirred continuously.



## Acetylation of Primary Amine: Preparation of Acetanilide.

**Theory :** It is prepared by acetylation of aniline with acetyl chloride or acetic anhydride and glacial acetic acid.

### Preparation of acetanilide from aniline and acetyl chloride



### Chemicals Required :

1. Aniline
2. Acetyl chloride
3. Glacial acetic acid
4. Ice cold water

### Procedure :

1. Take 1ml of aniline in a beaker and to it add 1ml of glacial acetic acid with constant stirring.
2. Add 1ml of acetyl chloride to the above solution dropwise by keeping the beaker in ice-cold water.
3. Heat the solution on boiling water bath for 5min.
4. Cool the solution. After it has reached room temperature add 10ml of ice-cold water with constant stirring.
5. Filter it and dissolve the obtained brown solid into 10-20ml of distilled water and heat it on a water bath for 5min.
6. Again filter this solution containing unreacted aniline.
7. Leave the obtained filtrate to cool at room temperature.
8. Filter the white coloured crystals of acetanilide and dry in sunlight.

Yield : 1.48 gm

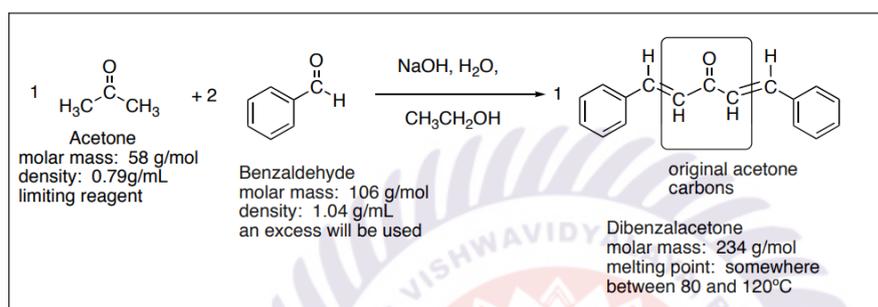
M.P. : 114°C

### Precautions :

1. Aniline is a carcinogenic compound so it must be handled very carefully.
2. Don't inhale the fumes of acetic anhydride.
3. Always dry the crystals before taking their weight and melting point to avoid any error due to moisture.
4. Keep stirring the reaction mixture during the experiment.

## Base Catalyzed Aldol Condensation: Synthesis of dibenzalpropanone.

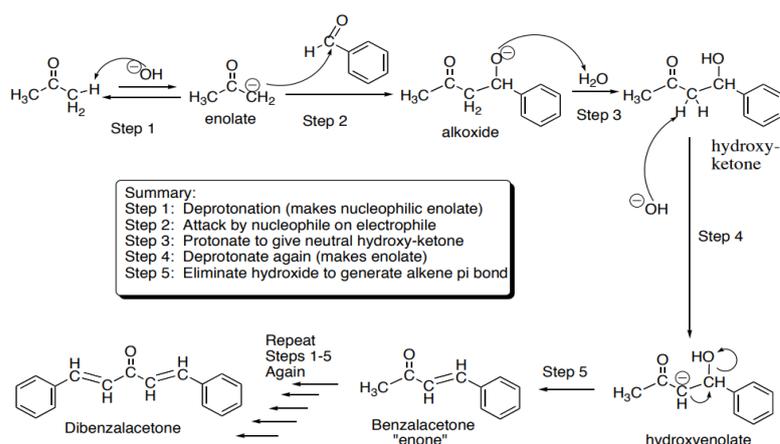
**Principal:** The reaction of an aldehyde with a ketone employing sodium hydroxide as the base is an example of a mixed aldol condensation reaction. You will do a double mixed-aldol condensation reaction between acetone and benzaldehyde. Acetone has  $\alpha$ -hydrogens (on both sides) and thus can be deprotonated to give a nucleophilic enolate anion. The aldehyde carbonyl is much more electrophilic than that of a ketone, and therefore reacts rapidly with the enolate. The alkoxide produced is protonated by solvent, giving  $\alpha,\beta$ -hydroxyketone, which undergoes base-catalysed dehydration. The elimination process is particularly fast in this case because the alkene is stabilized by conjugation to not only the carbonyl but also the benzene.



### Materials Required:

1. Benzaldehyde
2. Acetone
3. Sodium hydroxide solution
4. Methylated spirit
5. Dilute hydrochloric acid
6. Ether

## Mechanism:



## Procedure:

1. Take a conical flask add 10ml freshly distilled benzaldehyde and 20ml of acetone.
2. Place the flask in cold water bath and then add 2.5ml sodium hydroxide drop wise with constant stirring.
3. Maintain the temperature at 30 oC.
4. After the complete addition of sodium hydroxide stir the mixture for 2 hours.
5. Add dilute hydrochloric acid to the reaction mixture and then transfer to a 250ml separating funnel.
6. Add 20ml of chloroform/ether to the mixture and shake thoroughly.
7. Shake the mixture thoroughly, remove the organic layer and repeat the process twice.
8. Cool the mixture in ice-water. Dibenzal acetone separates initially as a fine emulsion and then forms yellow crystals.
9. Distil the residual portion under pressure and collect the fraction boiling at 150 oC.
10. Wash the pale yellow crystals with cold water, dry and crystallize them with ethanol.

Theoretical yield : 4gm

Melting point : 112 oC

**Result:** Dibenzalpropanone was synthesized with .....% yield.

## Precautions:

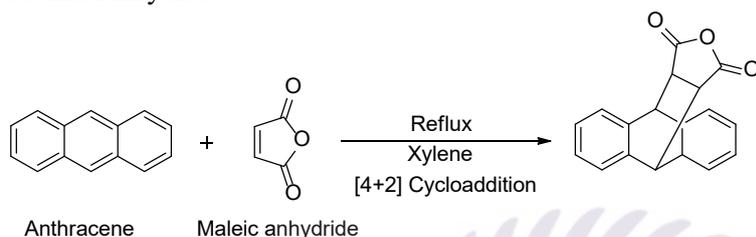
1. Do not come in direct contact with benzalacetone as it acts as an irritant for the skin.
2. Ensure that the temperature does not exceed 30 °C.
3. In case of vigorous reaction inside the flask, remove the cork to release pressure.
4. Remember to keep ethanol and acetone away from the flame

## [4+2] Cycloaddition Reaction: Diels-Alder reaction.

**Theory:** It is an important reaction for the preparation of cyclic compounds. In this reaction a conjugated diene unites with an unsaturated compound (called the dienophile).

The Diels-alder reaction is an example of cycloaddition reaction, i.e. a reaction that leads to the formation of a ring. The reaction is frequently carried out thermally, but in some cases, can be achieved in the presence of a Lewis acid.

Anthracene and maleic anhydride reacts on heating to form the 9,10-Dihydroanthracene-9,10- $\alpha,\beta$ -Succinic Anhydride.



### Material Required :

1. Anthracene : 3gm
2. Dry Xylene : 30ml
3. Maleic anhydride : 1.5gm
4. Ethyl acetate

**Procedure:** Place 3 g of pure anthracene, 30 ml of dry xylene, and 1.5 g of maleic anhydride in a 100 ml dry round-bottomed flask. Attach a water condenser and reflux the contents on a water bath for 25 min with frequent shaking. Cool the mixture and put the solid on a Buchner funnel. Recrystallize the adduct from ethyl acetate.

**M.P. :** 262-263 °C.

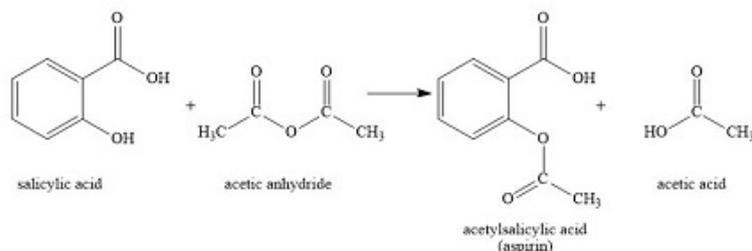
**Result :** The product was synthesized with .....% yield.

### Precautions :

1. The  $\text{CaCl}_2$  drying agent can go in the nonhazardous trash can.
2. From the recrystallization, the ethyl acetate should be put into the non-halogenated hazardous waste, and the filtered solid impurities should go in the solid hazardous waste.
3. Carefully use Anthracene.

## Preparation of aspirin and calculate its yield.

**Theory :** Aspirin is the single most manufactured drug in the world. Aspirin's chemical name is acetylsalicylic acid, and it is synthesized from the reaction of acetic anhydride with salicylic acid in the presence of phosphoric acid as a catalyst. The by-product is acetic acid.



### Chemicals required :

1. Salicylic acid
2. Acetic anhydride
3. H<sub>3</sub>PO<sub>4</sub> (concentrated)
4. 0.1 M Fe(NO<sub>3</sub>)<sub>3</sub>

### Procedure :

1. Obtain approximately 2 g of salicylic acid and determine its mass accurately. Transfer to a 100 mL beaker.
2. Measure ~3 mL of acetic anhydride with your small graduated cylinder, record the exact volume and add this to the 100 mL beaker with salicylic acid.
3. Add 5 drops of concentrated phosphoric acid.
4. Place the 100 mL beaker in the 400 mL beaker of warm water (70–80°C). Use a stirring rod to get the reactants into solution. Allow the mixture to react for about 5 minutes in the warm water.
5. Remove the 100 mL beaker, and while the mixture is still warm, carefully add about 1 mL of cold water, drop by drop to the 100 mL beaker. You will now hydrolyze any excess acetic anhydride. The hydrolysis may cause spattering.
6. Add 15 mL of water and cool the 100 mL beaker in a larger beaker with ice water. Stir with a stirring rod. Allow 5 minutes for the aspirin to crystallize from the solution. In the meantime, put a wash bottle filled with distilled water in ice water to cool.
7. Set up a Büchner filtration system. Place a filter paper in the funnel, and wet the paper thoroughly with distilled water. Turn the water on to allow the paper to be sucked onto the funnel. In order to ensure a good seal, you may want to add a small amount of water into the funnel.
8. Collect the crystals by pouring the solution onto the filter paper. Make sure that the water is fully open for maximum suction filtration. Do not disturb the crystals on the filter paper or you will break the filtration vacuum. Wash any product in the beaker into the funnel with cold distilled water; rinse your stirring rod as well. Remember, any product you lose here will affect your percent yield.
9. Allow the filtration to continue until you see no more water droplets fall from the funnel. Turn the water off. Insert a spatula between the edge of the paper and the side of the funnel and lift the paper from the funnel. Transfer the product to a 100 mL beaker. Scrape all the product from the filter paper with a spatula.

10. Recrystallization of Aspirin : Add about 3 mL of ethanol to the crystals. Warm the solution in a hot water bath. If all the crystals do not dissolve, add another 1–2 mL of ethanol. You want to use as little ethanol as possible in the recrystallization step.
11. Pour 15 mL of warm water into the alcohol solution. Cover the beaker with a watch glass and set aside to cool. (The crystallization process can be sped up by putting the beaker in an ice bath.) Allow 5 minutes for the crystals to crystallize from the solution.
12. Record the mass of your clean, dry filter paper and filter the crystals using suction filtration as before. While the filtration is in progress, determine the mass of a dry, clean watch glass.
13. When the filtration is complete, remove the filter paper and place the paper and crystals on the watch glass. Label your watch glass before putting it in the oven and dry your aspirin in the oven at 110°C for 10–15 minutes.
14. When your product is dry, remove your sample from the oven and allow it to cool. Weigh the watch glass with the aspirin product and record the value in your data table. Record the color and texture of the recrystallized aspirin.
15. Calculate the yield.

**Result :** Aspirin was synthesized with .....% yield.

**Precautions :**

1. Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) is highly corrosive. Avoid contact with your eyes, skin, and clothing. In case of contact, rinse with plenty of water.
2. The aspirin synthesized by unlicensed individuals (such as with lab experiments only) should not be ingested.
3. Irritating vapors: When water is added to the reaction mixture, it produces irritating vapors. Keep safe distance.
4. Bothersome reagents: The salicylic acid reagent can bother the lining of the stomach and one of the reagents may be bothersome to the nose. Avoid ingesting and direct skin contact with them.

## SECTION-C

**To calculate the  $\lambda_{\max}$  of a given compound using UV-visible spectrophotometer.**

**Theory:** Spectrophotometry is a technique that uses the absorbance of light by an analyte (the substance to be analyzed) at a certain wavelength to determine the analyte concentration. UV/VIS (ultra violet/visible) spectrophotometry uses light in the UV and visible parts of the electromagnetic spectrum. Light of this wavelength is able to effect the excitation of electrons in the atomic or molecular ground state to higher energy levels, giving rise to an absorbance at wavelengths specific to each molecule.

When a beam of light is passed through a transparent medium, a portion of it is absorbed and the rest is transmitted. The extent of absorption depends upon the wavelength of radiation, thickness of the medium and the concentration of the solution. If a compound absorbs at a particular wavelength, the intensity of the sample beam (I) will be less in comparison to that of the reference beam ( $I_0$ ). In this way, this instrument is able to compare the absorption of light at each wavelength. The absorption takes place at a particular wavelength can be expressed by the following equation

$$A = \log I/I_0$$

Beer and Lambert proposed two fundamental laws independently, related to the absorption of light. These laws are expressed mathematically in combined form by the following expression.

$$A = \log I/I_0 = \epsilon \times c \times l$$

$$A = \epsilon \times c \times l$$

$$\epsilon = A/c \times l$$

Where c is molar concentration (g mol/litre), l = length in cm of light beam passing the sample and  $\epsilon$  is a constant called molar absorptivity.

The wavelength at which a substance shows maximum absorbance is called absorption maximum or  $\lambda_{\max}$ . The value of is important for several reasons. This wavelength is characteristic of each compound, it provides information on the electronic structure of the analyte, it ensures highest sensitivity and minimize deviations from Beer's Law.

**Apparatus required:** Spectrophotometer, test tube, pipette, conical flask, electronic balance.

### Procedure:

1. Prepare 100 ml, standard solution of the given compound.
2. Switch on the computer and/or the instrument powers; wait for 30 minutes for 'warmup' of the instrument.
3. Take one clean and dry glass (only for visible range scan) or quartz cuvette with a given path length (say, 1 cm). Prepare a blank solution by filling a cuvette 3/4 full with distilled water.
4. By using blank solution (cuvette with distilled water) calibrate the spectrophotometer (absorbance =0, transmittance =100 %) and put out the blank solution.
5. Then fill another cuvette with the stock solution and measure absorbance, note the reading in table.
6. Repeat the step 4 and 5 for all the filters (wave length range)
7. After this make a table of the collected data and plot a graph wavelength vs absorbance on it.

### Observation:

S No.	Wavelength	Absorption

By plotting a graph by taking absorbance at y- axis and wavelength at x-axis and finding out the maximum wavelength.

In the graph we can see that absorbance increases as we increase the wavelength of UV- radiation falling on the solution. There is a point where absorbance is maximum. This point is called  $\lambda_{\max}$ . After this point, suddenly we can see the decreasing pattern of absorbance. Before the  $\lambda_{\max}$  absorbance is increased by increasing wavelength but after the point of  $\lambda_{\max}$  absorbance is decreased by increasing wavelength.

**Result:**  $\lambda_{\max}$  of the given compound is...

**Preautions :**

1. Wipe the outside of each cuvette with a lint-free tissue.
2. Handle cuvettes only by the top edge of the ribbed sides.
3. Dislodge any bubbles by gently tapping the cuvette on a hard surface.
4. Always position the cuvette so the light passes through the clear sides.



## Separation of metallic ions by paper chromatography

### Theory

Chromatography is a modern and sensitive technique used for rapid and efficient analysis and separation of components of mixture and purification of compounds. This process is based on a simple principle of partitioning of various constituents of a mixture between two phases out of which one phase is moving and the other is stationary.

Paper chromatography is a type of partition chromatography in which filter paper is used as an adsorbent. Moisture (water molecules) adsorbed by this acts as a stationary phase and the solvent as a moving phase. One drop is put at one end of the filter paper strip and dried. The paper is placed in container, with suitable solvent, vertically in such a way that the lower end of the paper (where the mixture spot is placed) dips in the solvent and the spot remains slightly above the solvent level. Due to capillary action, the solvent rises up in the filter paper. As it rises, the various components of the mixture rise up at different rates depending upon their distribution coefficients and thus get separated from one another. When solvent has reached its maximum height, it is noted. The filter paper, now called chromatogram, is taken out of chamber and dried. It is then sprayed with some suitable solvent. When different components of the mixture produce coloured spots, the different components of the mixture are identified. The distance travelled by a solute is proportional to the distance travelled by solvent. This ratio is constant called  $R_f$ .

$$R_f = \frac{\text{Distance travelled by metal ion}}{\text{Distance travelled by solvent}}$$

With the help of  $R_f$  values, the various components of a mixture can be identified. The  $R_f$  value of any substance does not exceed one, it does not depend upon the length of the paper.

### Apparatus and Reagents.

- (1) Chromatographic Paper: Whatman filter paper No.1 or 3 having the size 30 cm × 1.5 cm or 30 × 7 cm.
- (2) Chromatographic Jar
- (3) Salt Solutions: Mixture of 1% solution of silver nitrate, mercurous nitrate and lead nitrate.
- (4) Developer or Developing Solvents: n-Butyl alcohol mixed with 5% (V/V) glacial acetic acid and water.
- (5) Visualising agents: Dilute solution of potassium chromate or 0.5% dithiozone solution in chloroform.

### Procedure:

1. Cut Whatman No.1 filter paper in a suitable size (30 cm 70 cm). Draw a line horizontally across the width of filter paper strip using a lead pencil. This line should be about 2 cm from the end of the filter paper. Place a drop of the mixture on the centre of the line and allow the drop to dry. Place the salt solution drop on this spot and allow to dry and set the apparatus.
  2. Place the solvent in the bottom of the apparatus and hung or suspend the paper in such a way that the lower end of the filter paper dips (upto 1 cm) in the solvent.
  3. It should be noted that the paper strip should not touch the bottom and the pencil line at which the mixture drop is placed should be at least 2-3 cm above the solvent.
  4. Leave the apparatus undisturbed for some time (two hours). The solvent rises upwards. When the solvent has travelled a reasonable distance (say 15 cm), the paper is taken out and allowed to dry. Location of the place upto where the solvent has travelled is marked by a pencil.
  5. After this, the filter paper strip is sprayed with a diluted solution of potassium dichromate. Different metal ions produce different coloured spots at different places.
- Calculate  $R_f$  values of different cations on the basis of coloured spots.

$$R_f = \frac{\text{Distance travelled by metal ion}}{\text{Distance travelled by solvent}}$$

**Observation.**

The distance travelled by solvent on the paper =  
Distance of metal ion spot from original point =

**Calculation**

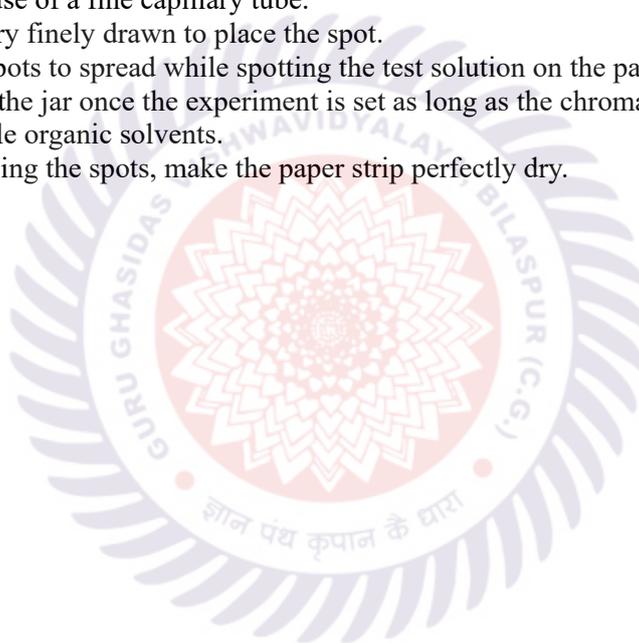
$$R_f = \frac{\text{Distance travelled by metal ion}}{\text{Distance travelled by solvent}}$$

**Result**

The given metallic ions are separated by calculating their  $R_f$  value.

**Precautions :**

1. Always make use of a fine capillary tube.
2. Use the capillary finely drawn to place the spot.
3. Do not allow spots to spread while spotting the test solution on the paper.
4. Do not disturb the jar once the experiment is set as long as the chromatogram is developed.
5. Carefully handle organic solvents.
6. Before developing the spots, make the paper strip perfectly dry.



## Determination of surface tension of a liquid by stalagmometer

**Theory:** The molecules in a liquid are held together by force of attraction, which causes the surface of the liquid to behave like a stretched skin. Surface tension is the result of these forces of attraction. It is defined as the force acting on per unit length of the surfaces of the liquid vertical to the surface and is measured in dynes per cm (force/length).

When a definite volume of a liquid is allowed to drop through a jet the number of drops formed depends on the surface tension of the liquid. This is the principle employed by Traube's Stalagmometer to measure the surface tension of a liquid.

Traube's Stalagmometer consists of pipette-shaped tube with a capillary, the end of which is flattened, ground and polished. Flattening of the end provides a leave dropping surface. There are two defining marks A and B one above and the other below the bulb. The liquid is sucked a little above the upper mark A and is allowed to drop.

A drop which is just to leave the jet is balanced by two forces (1) the forces of gravity making the drop to fall, and (2) the force due to surface tension tending to uphold the drop.

The force due to gravity =  $V.d.g$ . where  $V$  is the volume of the drop,  $d$  is density of the liquid, and  $g$  is acceleration due to gravity.

The force due to the surface tension tending to uphold the drop is  $2\pi r\gamma$  where  $2\pi r$  is the circumference of the drop and  $\gamma$  is the surface tension of the liquid. At the moment when the drop is just to leave the jet, these two forces are exactly balanced.

$$2\pi r\gamma = V.d.g.$$

If  $n$  is the number of drops formed by a liquid of volume  $V$  and density  $d$ , the weight of a single drop.

$$= V.d.g./n$$

Hence, for the same volume  $V$  of two liquids

$$2\pi r\gamma_1 = V.d_1.g./n_1 \quad \dots\dots\dots(1)$$

$$2\pi r\gamma_2 = V.d_2.g./n_2 \quad \dots\dots\dots(2)$$

$\gamma_1$  and  $\gamma_2$  are the surface tensions and  $n_1$  and  $n_2$  are the number of drops formed by two liquids of densities  $d_1$  and  $d_2$  respectively for the same volume.

Dividing (1) by (2)

$$\gamma_1/\gamma_2 = d_1.n_2/d_2.n_1 \quad \dots\dots\dots(3)$$

For a given liquid surface tension decreases with rise to temperature. Macleod (1923) discovered the following relationship governing the variation of surface tension of a given liquid with temperature :

$$\gamma = k(D-d)^4$$

Where  $D$  and  $d$  are the densities of the liquid and its vapour respectively,  $k$  is a constant,  $d$  being very small in comparison with  $D$ , may be neglected.

$$\gamma = kD^4$$

Or  $k = \gamma^{1/4}/D$

For one gm molecule, Sugden (1924) discovered that a function called "parachor" expressed by an additive property of a molecule.

$$[P] = M\gamma^{1/4}/D \quad \dots\dots\dots(4)$$

Parachor value for the liquid can thus be calculated if molecule weight, density and surface tension of the liquid are known.

**Apparatus required:** Stalagmometer, an organic liquid such as benzene, 15-20 ml of distilled water, pycnometer, beaker, clamp and about quarter metre long rubber tubing.

**Procedure:** Wash the Stalagmometer first with chromic acid, then with distilled water. Dry and clamp it accurately vertical. Suck up the distilled water a little beyond the upper mark and release. When the meniscus reaches the upper mark, start counting the drops and when the meniscus reaches the lower

mark, stop the counting. The Stalagmometer should be such that the rate of drop is 15-20 drops per minute. Repeat it four or five times.

Empty and dry the stalagmometer and repeat experiment with the given liquid. Find out the densities of the liquid with the help of pycnometer.

Observation: Temperature ...<sup>0</sup>C

For the measurement of drop number

	Water	Unknown Liquid
S.No.	Number of drops Between Two Marks	Number of drops Between Two Marks
1.		
2.		
3.		
	Mean	Mean

For Density

Wt. of Pycnometer =

Wt. of Pycnometer + water =

(Wt. of Pycnometer + water - Wt. of Pycnometer) = Wt. of water =  $W_1$

Wt. of Pycnometer =

Wt. of Pycnometer + liquid unknown =

(Wt. of Pycnometer + liquid unknown - Wt. of Pycnometer) = Wt. of liquid unknown =  $W_2$

Density of liquid =  $W_2/W_1$

**Calculation.**

$$\gamma_1/\gamma_2 = d_1.n_2/d_2.n_1$$

$$\text{Surface tension of liquid } (\gamma_2) = \frac{\text{Density of liquid} \times \text{no. Of drops of water}}{\text{density of water} \times \text{no. Of drops of liquid} \times \text{surface tension of water.}}$$

**Result:** the surface tension of the given liquid .....dynes per cm at .....<sup>0</sup>C

**Precautions :**

1. The stalagmometer should be cleaned and dried before use.
2. While sucking the liquid into the stalagmometer, no air bubble should be formed.
3. Stalagmometer should be held in a vertical position throughout the drop counting process.
4. Drops should be properly formed.
5. Same stalagmometer should be used for water and liquid.

**Determination of the percentage composition of the given mixture consisting of two liquids A and B (non- interacting system) by viscosity method.**

**Theory:** Flowing is one of the important properties of liquids. All liquids do not flow at the same ease. Some liquids flow more easily than others. The property of liquids that determines its flow is termed as viscosity or the measure of resistance of flow of a liquid is defined by its viscosity. The tendency of resistance depends upon the intermolecular attractions, the molecular weight and the nature of liquid.

When a liquid flows through a narrow tube, all the parts of the liquid do not move with the same velocity through the tube, The middle layers move more rapidly than the layers next to the sides of the tube. Thus there is a movement of different layers of a liquid past one another. When a liquid layer flows past another layer of the liquid, the flow is retarded due to internal friction or viscosity of the liquid. The retarding force produced by the internal friction of the two layers is proportional to the velocity gradient  $dv/dx$  normal to the direction of the flow and area of contact A between the two layers, i.e.,  $F \propto Adv/dx$

Or  $F = \eta Adv/dx$

Where  $\eta$ , the constant of proportionality is called the coefficient of viscosity.  $\eta$  is measured in C.G.S. units and has the dimensions of  $gm\ cm^{-1}\ sec^{-1}$ . The unit is the ‘poise’.

A number of solutions containing A and B in different proportions are prepared and the time taken by such mixtures to flow to a specific mark is noted by a viscometer. Time taken by the unknown mixture of flow is also noted. Then a graph is plotted between the time of flow and composition of the mixture. With the help of this graph, the composition of the unknown mixture is determined.

**Apparatus Required:** Ostwald viscometer, stop watch, a large beaker, 1 ft long rubber tubing, 10 ml pipette, unknown liquid, clamp.

**Method:** A number of mixture solutions are prepared. Generally, 90%, 80%, 70%, 60%,50%,40%,30%,20% and 10% of solutions containing A are prepared. Then fill each solution in the viscometer separately and find out the time of flow from mark A to B. Find out the time of flow for the solution of unknown concentration.

**Observation:**

Temperature of room =

S No.	Composition of the mixture		Time taken to flow from mark A to B (seconds)
	A	B	
1.	90	10	
2.	80	20	
3.	70	30	
4.	60	40	
5.	50	50	
6.	40	60	
7.	30	70	
8.	20	80	
9.	10	90	
10.	Unknown solution		

**Calculations:**

A graph is plotted between the percentage composition and time of flow. From the graph and the time of flow for the unknown solution, the composition of the unknown solution is determined.

**Result:** The given solution contains....% A and ....%B.

**Precautions :**

1. To watch the motion the liquid used must be transparent.
2. Ensure that your spindle is clean, shaft is not bent and has not any dings or dents.
3. Ensure that you do not introduce air bubbles into your sample during preparation.



## Determination of the relative viscosity of given liquid by Ostwald's viscometer

**Theory:** Flowing is one of the important properties of liquids. All liquids do not flow at the same ease. Some liquids flow more easily than others. The property of liquids that determines its flow is termed as viscosity or the measure of resistance of flow of a liquid is defined by its viscosity. The tendency of resistance depends upon the intermolecular attractions, the molecular weight and the nature of liquid.

When a liquid flows through a narrow tube, all the parts of the liquid do not move with the same velocity through the tube, The middle layers move more rapidly than the layers next to the sides of the tube. Thus there is a movement of different layers of a liquid past one another. When a liquid layer flows past another layer of the liquid, the flow is retarded due to internal friction or viscosity of the liquid. The retarding force produced by the internal friction of the two layers is proportional to the velocity gradient  $dv/dx$  normal to the direction of the flow and area of contact  $A$  between the two layers, *i.e.*,

$$F \propto Adv/dx$$

Or 
$$F = \eta Adv/dx$$

Where  $\eta$ , the constant of proportionality is called the coefficient of viscosity.  $\eta$  is measured in C.G.S. units and has the dimensions of  $\text{gm cm}^{-1} \text{sec}^{-1}$ . The unit is the 'poise'.

Instead of determining the absolute viscosities of liquids, it is simpler to compare a liquid with one of known the same conditions. A simple type of apparatus for comparing viscosities of different liquids is the Ostwald viscometer. Its left hand limb consists of a fine capillary tube with two defining marks A and B. A definite volume, say, 10 ml of the liquid is delivered into the right hand limb and it is sucked up in the left hand limb a little beyond the mark A by means of a rubber tubing attached to the left hand limb. The liquid is released. When the meniscus passes the mark A, stop watch is started and when the meniscus arrives at B, the watch is stopped. The flow is noted. If  $t_1$  is the flow for the liquid unknown and  $t_2$  is the flow time for the known liquid, then

$$\frac{t_1}{t_2} = \frac{\eta_1/d_1}{\eta_2/d_2}$$

$$\frac{\eta_1}{\eta_2} = \frac{d_1 t_1}{d_2 t_2}$$

$$\eta_2 = \frac{\eta_1 \times d_2 \times t_2}{d_1 \times t_1}$$

Where,  $\eta_1$  and  $d_1$  are the viscosity and density respectively of the unknown liquid and  $\eta_2$  and  $d_2$  are the viscosity and the density respectively of the known liquid.

The expression gives the viscosity of one liquid, with reference to the other of known viscosity, usually water.

**Apparatus:** Ostwald viscometer, stop watch, a large beaker, 1 ft long rubber tubing, 10 ml pipette, unknown liquid, clamp.

### Procedure:

- (1) Thoroughly clean the viscometer with chromic acid solution and then with distilled water. Rinse with alcohol and ether. Then dry it by passing hot air.
- (2) Clean a piece of rubber tubing internally to remove dust. Attach it to the left hand limb above the mark A.
- (3) Clamp the viscometer accurately vertical in a large beaker, containing water so that the mark A is below the surface of water. This will give viscosity at room temperature.
- (4) Pipette out 10 ml distilled water, introduce it into the right hand limb of the viscometer and allow the water there for 15-20 minutes to reach the temperature of the bath.
- (5) Suck up the liquid by means of the rubber tubing a little above the mark A and then release it to flow down under the influence of gravity. Start the stop watch when the liquid just arrives at A, and then stop

the watch when the liquid is just at B. The difference between the initial and final readings of the stopwatch is the time of flow for water. Note and repeat the process at least half a dozen times and record the reading of the time of outflow in each case.

(6) Empty the viscometer and dry. Clamp it vertically as before and pipette out 10 ml of benzene or the other unknown liquid which ever is given into the right limb. Suck up the liquid by means of rubber tubing as before and release to flow down under the influence of gravity. Note the time of outflow for the liquid. Repeat the process 6 times. Record the readings.

(7) Determine the relative density of the unknown liquid, by means of a pyknometer. Tabulate your observations as follows:

**Observation:** (1) For measurement of flow time

S.No.	Water		Unknown Liquid	
	Time of flow in seconds	Mean time	Time of flow in seconds	Mean time
1.				
2.				
3.				

(2) Density

A. Water

$$\begin{aligned} \text{Wt. of pyknometer + water} &= M_1 \text{ gm} \\ \text{Wt. of pyknometer} &= M_2 \text{ gm} \\ \text{Wt. of water} &= M_2 - M_1 \text{ gm} = W_1 \text{ gm} \end{aligned}$$

B. Unknown liquid

$$\begin{aligned} \text{Wt. of pyknometer + liquid} &= M_3 \text{ gm} \\ \text{Wt. of pyknometer} &= M_1 \text{ gm} \\ \text{Wt. of liquid} &= M_3 - M_1 \text{ gm} = W_2 \text{ gm} \end{aligned}$$

Calculations:

- Density of water at the room temperature =  $d_1$
- Density of the liquid compared with water =  $d_2$

$$= W_2/W_1$$

$$\frac{\eta_1}{\eta_2} = \frac{d_1 t_1}{d_2 t_2}$$

$$\eta_2 = \frac{\eta_1 \times d_2 \times t_2}{d_1 \times t_1}$$

$$= \frac{(\text{density} \times \text{sec}) \text{ for liquid}}{(\text{density} \times \text{sec}) \text{ for water}} \times \text{viscosity for water in poise}$$

$\eta_2$  is the viscosity of liquid relative to water at room temperature. This is the relative viscosity of liquid assuming the viscosity of water to be unity. Absolute viscosity = Relative viscosity  $\times$  viscosity of water at the temperature.

**Result:** The viscosity of the given unknown liquid is .....poise.

**Precautions:**

- The viscometer must be perfectly clear and dry.
- The viscometer should be held vertically

- The rate of flow should be such that it takes about 2-10 minutes to reach from the upper mark to the lower mark.

