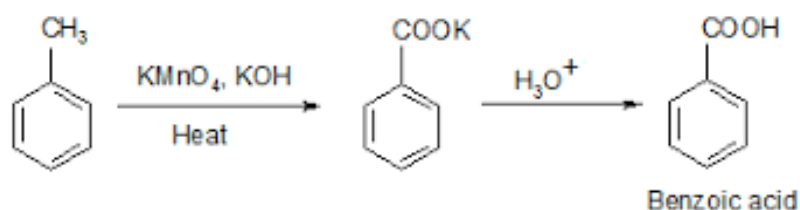


## Experiment No: 5

# Synthesis of benzoic acid from toluene

### Chemicals required:

1. Toluene (2 mL),
2. Potassium permanganate (2.5 g),
3. Sodium carbonate (2 g),
4. Hydrochloric acid (conc.)
5. Sodium sulphite (saturated solution)

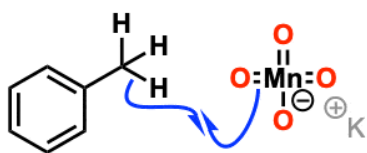


## **Procedure of synthesis**

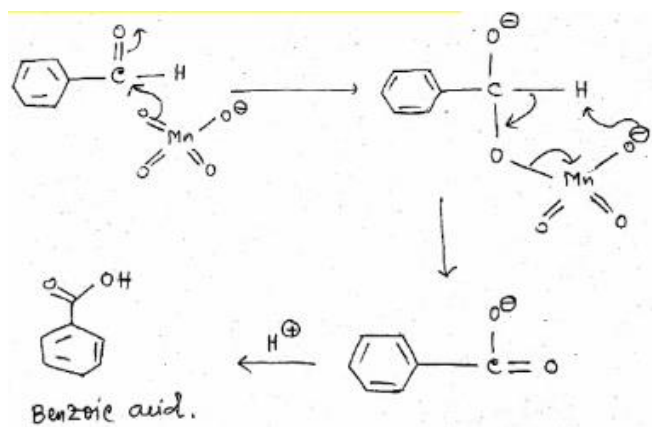
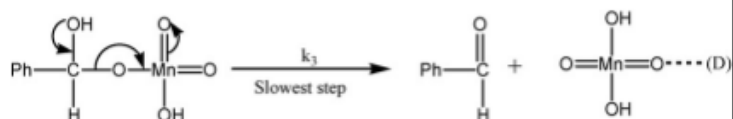
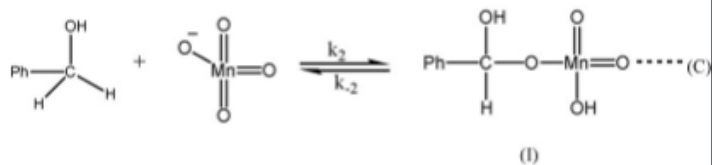
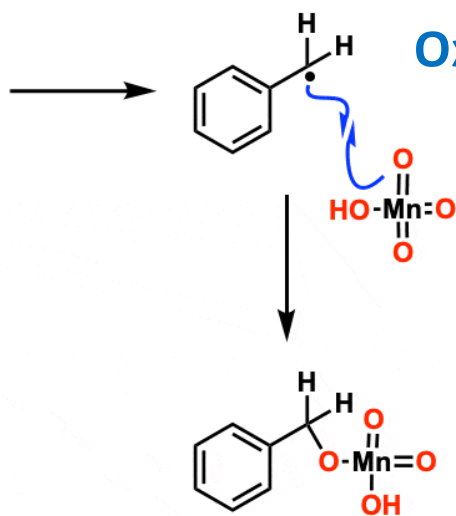
- 1. In a clean 250 mL conical flask take 2 mL of toluene and 2 g of sodium carbonate, dissolved in 30 mL of distilled water.*
- 2. In a 100 mL beaker prepare a saturated solution of potassium permanganate (2.5 g).*
- 3. Add the aqueous solution of permanganate to the mixture of toluene and sodium carbonate slowly till the purple colour of permanganate persists.*
- 4. Add some pieces of pumice stones in the conical flask and cover the mouth of the flask using a funnel.*  
*Place a piece of marble inside the funnel.*
- 5. Gently boil the solution for about 40 minutes.*

- 6. Cool the solution and pour it in a 500 mL beaker.*
- 7. Add a saturated solution of sodium bisulphate till brown precipitate of manganese dioxide dissolves.*
- 8. Acidify the contents of the beaker using concentrated hydrochloric acid (check with a piece of pH paper)*
- 9. White ppt of benzoic acid separate out. Filter the crude product at the suction pump, wash with cold water and dry it.*
- 10. Record the yield.*
- 11. Recrystallise a small amount of crude sample with hot water.*
- 12. Determine the melting point of recrystallized product.*

*Students are advised to follow the following YouTube link:*  
<https://youtu.be/b9cEfagdhSU>



## Oxidation mechanism by $\text{KMnO}_4$



## **Experiment No 6:**

### **Separation of amino acids from their mixture by Paper Chromatographic Technique**

#### **1. Principle:**

In Paper chromatography the mixture of compounds are distributed or partitioned between liquid phases. One phase is the stationary, which is held in the pores of the filter paper used and other is the mobile phase which moves over the paper.

**Capillary action:** the movement of liquid within the spaces of a porous material due to the forces of adhesion, cohesion, and surface tension. The liquid is able to move up the filter paper because its attraction to itself is stronger than the force of gravity.

**Solubility:** the degree in which a material (solute) dissolves into a solvent. Solute dissolves into solvents that have similar properties. This allows different solutes to be separated by different combinations of solvents.

Separation of components depends on both their solubility in the mobile phase and their differential affinity to the mobile phase and the stationary phase.

*(2) Chemical required:*

- a. Amino acids (L-Lysin, DL-Alanin, L-Leucine)*
- b. Acetic acid*
- c. I-Butanol*
- d. Distilled water*
- e. Spraying reagent: Ninhydrin (0.3% solution in rectified spirit)*

*(3) Apparatus:*

*Measuring cylinder*

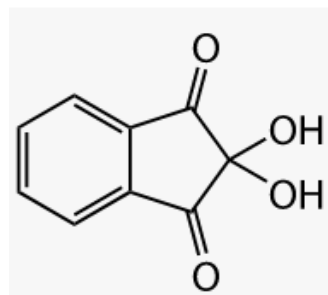
*Test tubes*

*Electric air oven*

*Solvent chamber (20 cm × 4 cm)*

*Sprayer*

*Whatman No. 1 chromatographic paper (20 cm × 4 cm)*



Structure of Ninhydrin

#### *4. Procedure:*

- (i) Preparation of solutions (10-15 mg of each amino acid is mixed and dissolved in 1 ml distilled water) and unknown amino acid in separate test tubes.*
- (ii) The chromatographic paper strip is taken and a base line is drawn above 4 cm from the lower end. Spots are given with the help of the capillary tubes.*
- (iii) Developing solvent- Butanol: AcOH : H<sub>2</sub>O = 12:3:5 and poured into the jar before 1 hr. of developing and lid is placed properly.*
- (iv) Development of the chromatogram.*
- (v) Drying in oven.*
- (vi) Spraying.*
- (vii) Location of the spots with pencil.*

### *5. Calculation:*

*The movement of any substance relative to the solvent front in a chromatographic system is constant.*

*In paper chromatography it is defined as,*

*$R_F$  value- Distance moved by the substance/distance moved by the solvent front.*

*Q: What is Retention factor ( $R_f$  value)?*

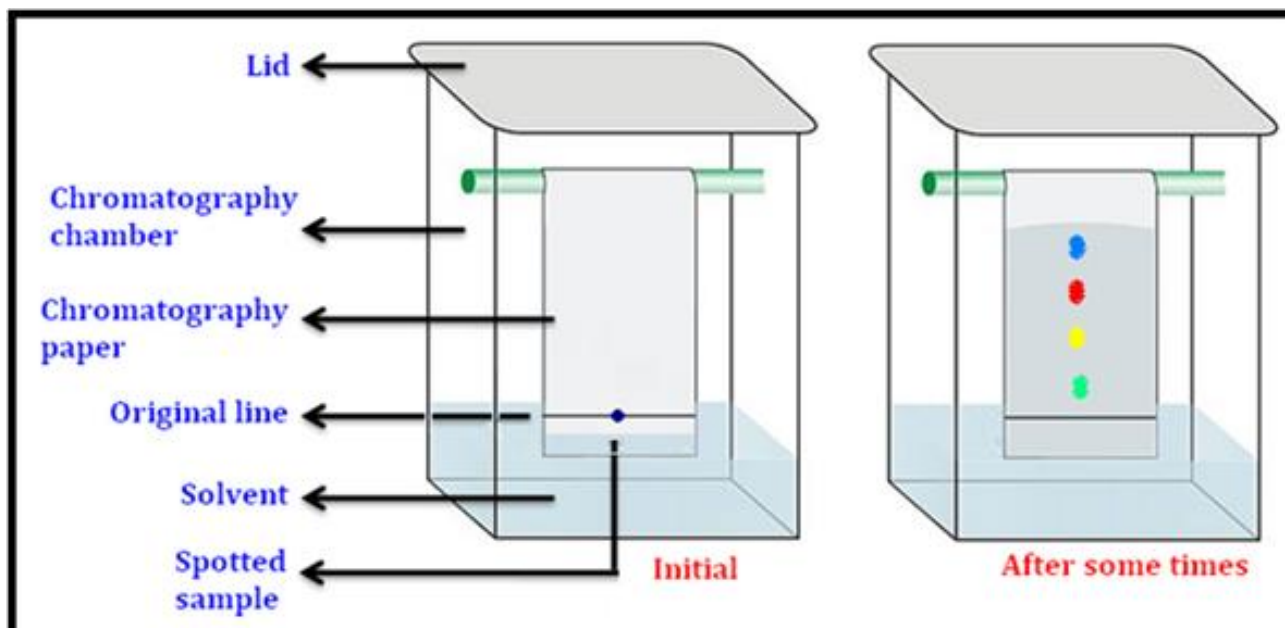
**It is defined as the distance moved up or travelled by the component from the original line to the distance travelled by the solvent from the original line.**

$$R_f = \frac{\text{Distance travelled by the component from the original line}}{\text{Distance travelled by the solvent from the original line}}$$



## Ascending and Descending Paper Chromatography

The type of paper chromatography in which the solvent rises up is called Ascending paper chromatography. Alternatively, the solvent may be taken on the top in a container and be allowed to come down, in which case it is termed as Descending paper chromatography. Below is shown the ascending paper chromatography.



## Home work:

- a. **Define and explain the terms:** chromatography, adsorption chromatography, partition chromatography, column chromatography, TLC, paper chromatography, retardation factor etc.
- b. Explain the principle of adsorption chromatography and partition chromatography.
- c. Students are advised to utilize the knowledge of this experiment in their respective engineering field.
- d. Students can identify the components based on their  $R_f$  value.

***All the students are advised to follow the following YouTube link***

<https://youtu.be/OzQ1dOF6QAY>

**N.B: All the Online experiments have to write in same Laboratory Note Book (LNB) and have to submit in Chemistry Lab, when the Institute will reopened.**

## Colour development of ninhydrin with amino acids

