

Experiment - 1AIM:

Preparation of benzoic acid from toluene

CHEMICALS REQUIRED

- Toluene (2 mL)
- Potassium permanganate (2.5 g)
- Sodium carbonate (2 g)
- Hydrochloric acid (conc.)
- Sodium bisulphate (saturated solution)

PROCEDURE

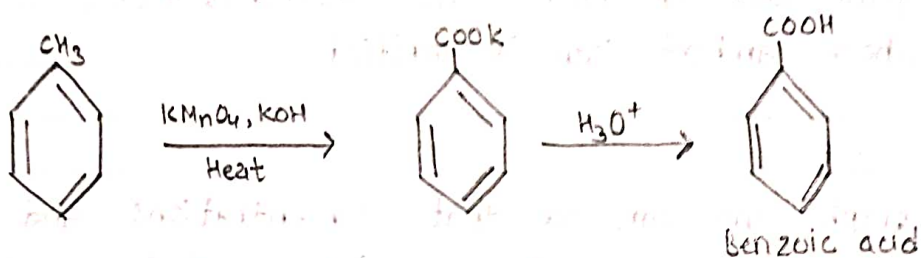
1. In a clean 250 mL conical flask, we take 2 mL of toluene and 2 g of sodium carbonate and dissolve in 30 mL of distilled water.
2. In a 100 mL beaker, we prepare a saturated solution of potassium permanganate (2.5 g)
3. We add the aqueous solution of permanganate to the mixture of toluene and sodium carbonate slowly till the purple colour of permanganate persists.
4. We add some pieces of pumice stones in the conical flask and cover the mouth of flask using a funnel.
5. We place a piece of marble inside the funnel.

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6. We gently boil the solution for about 40 minutes.
7. We cool the solution and pour it in a 500 mL beaker.
8. We add a saturated solution of sodium bisulphate till brown precipitate of manganese dioxide dissolves.
9. We acidify the contents of the beaker using concentrated hydrochloric acid (check with a piece of pH paper.)
10. White ppt. of benzoic acid separates out. We filter the crude product at the suction pump, wash with cold water and dry it.
11. We record the yield.
12. We recrystallize a small amount of crude sample with hot water.
13. We determine the melting point of the recrystallized product.

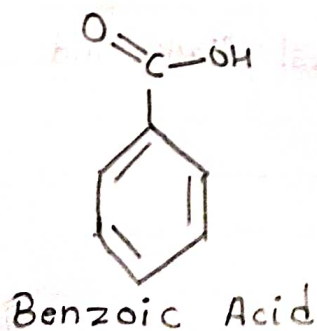
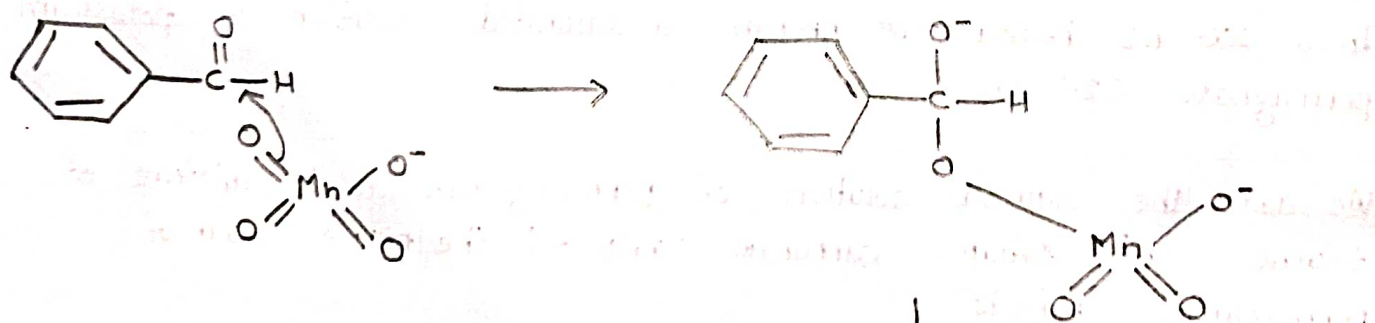
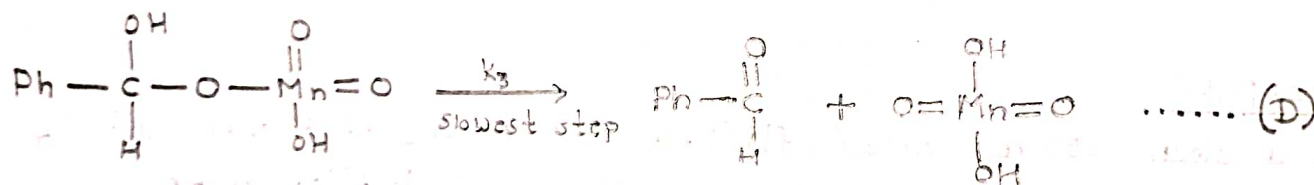
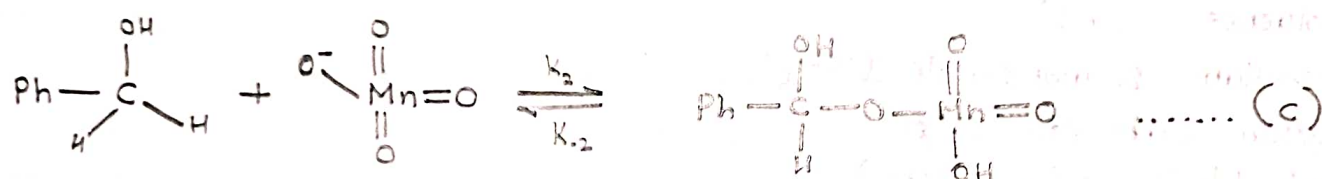
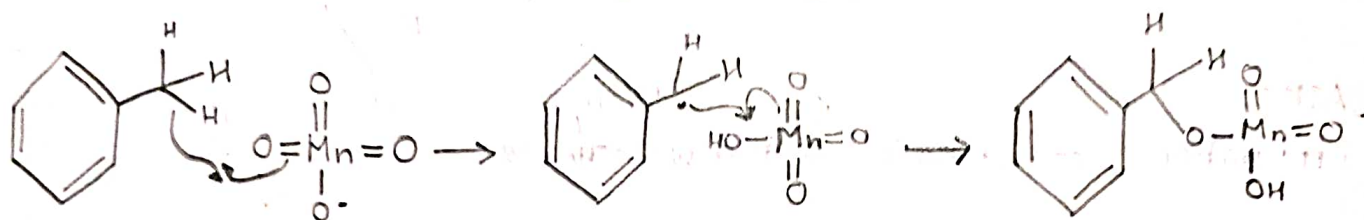
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Reaction involved:





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



## Experiment - 2

### AIM

Separation of amino acids from their mixture by Paper chromatographic technique.

### PRINCIPLE

Paper chromatography is an inexpensive and powerful analytical technique, which requires a piece of paper or strips serving as an adsorbent in the stationary phase across which a particular solution is allowed to pass. 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 to which a material (solute) dissolves into a solvent. Solute dissolves into solvents that have similar properties. This allows different solutes to be prepared 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.

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$$R_f = \frac{\text{distance traveled by the compound}}{\text{distance traveled by the solvent front}}$$

Retention factor is inversely proportional to polarity in this case.

### CHEMICALS REQUIRED

- Amino acids (L-Lysine, L-Alanine, L-Leucine)
- Acetic acid
- 1-Butanol
- Distilled water
- Spraying agent : Ninhydrin (0.3% solution in rectified spirit)

### APPARATUS

- Measuring cylinder
- Test tubes
- Electric air oven
- Solvent chamber (20 cm X 4 cm)
- Sprayer
- Whatman No. 1 chromatography paper (20 cm X 4 cm)

### PROCEDURE

- 1) Preparation of solutions (10-15 mg of each amino acid is mixed and dissolved in 1 mL distilled water) and unknown amino acid in a separate test tube.
- 2) 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.
- 3) 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.

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- 4) Development of the chromatogram.
- 5) Drying in the oven
- 6) Spraying
- 7) Location of the spots with pencil.

### CALCULATIONS :

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.

After completion, we get a figure such as given in fig. 1.

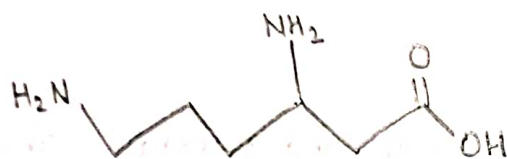
Polarity order :

L-lysine > L-alanine > L-leucine

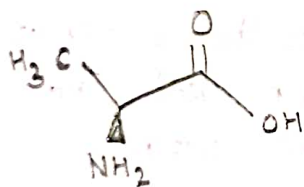
$R_f$  order :

L-leucine > L-alanine > L-lysine

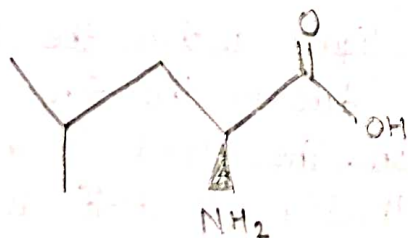
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L-Lysine



L-Alanine



L-leucine



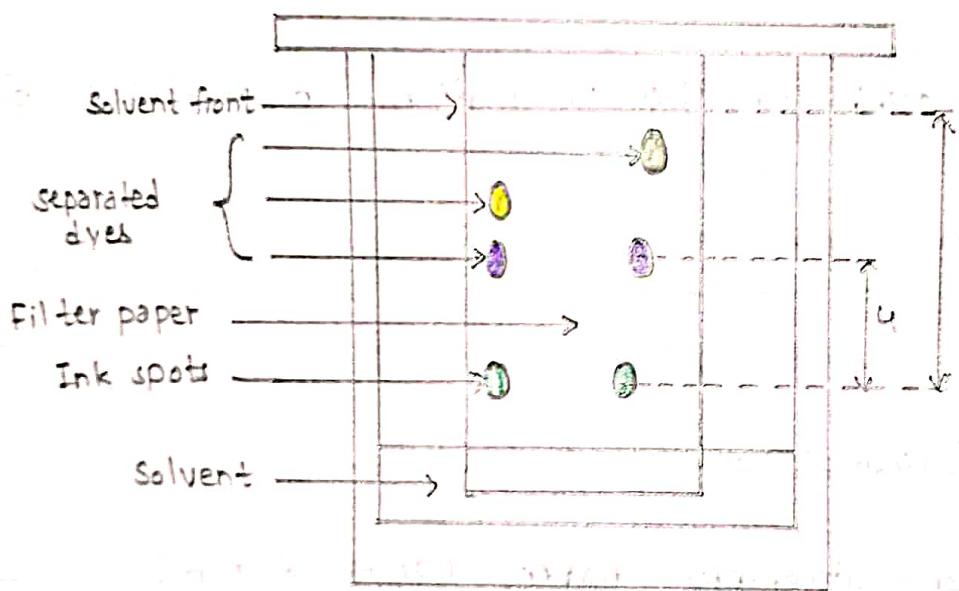


Fig 1

### Chemical Reaction:

