3. Experiment name: Preparation of Benzocaine

Aim: To synthesize Ethyl p-amino benzoate (benzocaine) from p-amino benzoic acid.

References: https://labmonk.com/synthesis-of-benzocaine

Requirements:

Chemicals: p-amino benzoic acid, conc.H2SO4, Sodium carbonate, rectified spirit, charcoal.

Apparatus: Thermometer, conical flask, round bottom flask, refluxes condenser, Buchner funnel, funnel, stirrer, and beaker.

Procedure:

Place 4gm of p-Amino benzoic acid, 27ml of absolute alcohol and 5ml of conc.H₂SO₄ in a dry 100ml round bottom flask fitted with a reflux condenser. Reflux for 2 hours on a water bath till it becomes a clear solution. Pour the hot solution in 80ml of water. If no solid separates, then add little decolourizing carbon, stir and filter. To the filtrate in 250ml beaker add slowely solid sodium carbonate till it is neutral to litmus. Cool and filter the product under suction. Recrystallize the product from 2:1 ethanol-water mixture. Dry the product in air and determine yield and melting point.

Principle:

Benzocaine is synthesized by Fisher Esterification reaction, which converts carboxylic acid and alcohol directly to ester by an acid catalyzed nucleophilic acyl substitution reaction. The interaction between a carboxylic acid and an alcohol is a reversible process and proceeds very slowely. Equilibrium is only attained after refluxing for several days. If about 3% of either conc. sulphuric acid or a dry hydrogen chloride is added to a mixture, the same point of equilibrium can be reached after few hours. When equilibrium quantities of acid and alcohol are employed, only two-third theoretically possible yield of ester is obtained. According to law of mass action, the equilibrium may be displaced in ester by the use of excess of the components. It is frequently convenient to use an excess of the acid, but if the acid is expensive a large excess of alcohol is generally employed. Since sulfuric acid is used in esterification, benzocaine exists as sulphate salt. To release free ethyl-4-amino benzoate, sodium carbonate is added. Excess of sodium carbonate if added can cause a base catalyzed hydrolysis of ester. So it should be added just to make the pH neutral.

Reaction:

Mechanism:

Conclusion: Benzocaine was synthesized and the percentage yield was found to be %.