Aim: To prepare and submit cinnamic acid from benzaldehyde.

Reference:

1. Kar Ashutosh, Advanced Practical Medicinal Chemistry, New Age International (P) Limited Publication, New Delhi, Page No. – 182.
2. Advanced Organic Chemistry: Reaction Mechanisms by Reinhard Bruckner, Published by Harcourt Academic Press: 2002; Page No. 419.
3. Systematic Lab Experiments in Organic Chemistry by Arun Sethi: New Age International Publishers; First Edition- 2003, Reprint- 2006, Page No. 706.

Requirement:

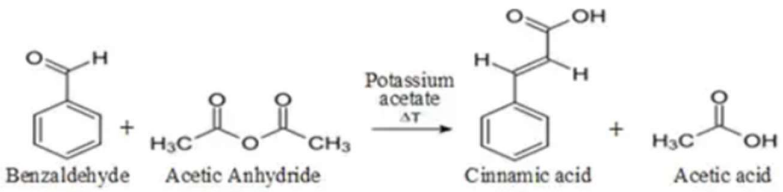
Apparatus: Double naked round-bottomed flask, Reflux condenser set, single naked 500 ml RBF, Beaker, Measuring cylinder, Buchner funnel, etc.

Chemicals: Benzaldehyde (10.5 g), Potassium acetate (6 g), Acetic Anhydride (15 g), Sodium carbonate (20 g), Conc. Hydrochloric Acid – q.s., Rectified Spirit (50 ml).

Principle:

The reaction between an aromatic aldehyde and an aliphatic anhydride is capable of providing an active methylene moiety in the presence of a primary catalyst, such as an acetate ion and a hydronium ion, which yields an α, β-unsaturated carboxylic acid and a mole of acetic acid, i.e., the interaction between benzaldehyde and acetic anhydride in the presence of acetate ion and a hydronium ion yields, cinnamic acid and acetic acid

Reaction:



Use: Cinnamic acid is used in flavors, synthetic indigo, and certain pharmaceuticals. It is used to manufacture methyl, ethyl, and benzyl esters for the perfume industry. It is also used as a precursor of the sweetener aspartame.

Procedure:

About 10.5 g (10 ml) of benzaldehyde, 15 g (14 ml) of acetic anhydride, and 6 g of finely powdered potassium acetate are transferred into a dry 250 ml round-bottomed flask provided with a $CaCl_2$-guard tube at its top-end. Contents of the RB-flask are mixed thoroughly, and the reaction mixture is heated in an oil bath at 160°C for 60 minutes and further at an elevated temperature of 170-180°C for about 3 hours. While still hot (90°-100°C), the flask's contents are poured into a 500 ml round-bottomed flask containing about 50 ml of water, which is fitted for steam-distillation operation. The contents of the flask-1 are rinsed with a little hot water and poured into the flask-2. The resulting solution in the 500 ml RB-flask is made alkaline by gradually adding a saturated solution of Na_2CO_3 with vigorous shaking. The solution is subjected to steam distillation until all the 'unreacted benzaldehyde is removed and the distillate is clear. The contents of the distillation flask are cooled and filtered by suction to get rid of most resinous unwanted by-products. Carefully, the filtrate is rendered to acidic pH by adding concentrated HCl gradually in small lots at intervals and with continuous shaking until the evolution of CO_2 ceases completely. The resulting solution is chilled; cinnamic acid gets separated as colorless crystals, filtered in the Buchner funnel, washed with a little cold water, drained well, and dried at 100 °C.

Calculation:

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Result:

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