

Objective

- i) To prepare acetylsalicylic acid (aspirin)
- ii) To synthesize acetylsalicylic acid from salicylic acid
- iii) To test presence of salicylic acid in the product

Introduction / Theory



In this reaction, the hydroxyl group (-OH) on the benzene ring in salicylic acid reacts with acetic anhydride to form an ester functional group. Thus, the formation of acetylsalicylic acid is referred to as an esterification reaction. This reaction requires the presence of an acid catalyst, indicated by the H^+ above the equilibrium arrows.

When the reaction is complete, some unreacted salicylic acid and acetic anhydride will be present along with acetylsalicylic acid, acetic acid, and the catalyst. The technique used to purify the acetylsalicylic acid from the other substances is called crystallization. The basic principle is quite simple. At the end of this reaction, the reaction mixture will be hot, and all substances will be in solution. As the solution is allowed to cool, the solubility of acetylsalicylic acid will decrease, and it will gradually come out of solution or crystallize. Because the other substances are either liquids at room temperature or are present in much smaller amounts, the crystals formed will be composed mainly of acetylsalicylic acid. Thus, a separation of acetylsalicylic acid from the other materials will have largely been accomplished. The purification process is facilitated by the addition of water after the crystals have formed. The water decreases the solubility of acetylsalicylic acid and dissolves some of the impurities.

To purify the product, even more, a recrystallization procedure will also be performed. In order to prevent the decomposition of acetylsalicylic acid, ethyl acetate, rather than water, will be used as the solvent for recrystallization. The most likely impurity in the product after purification is salicylic acid itself, which can arise from an incomplete reaction of the starting materials or from hydrolysis (reaction with water) of the product during the isolation steps. The hydrolysis reaction of acetylsalicylic acid produces salicylic acid. Salicylic acid and other compounds that contain a hydroxyl group on the benzene ring are referred to as phenols. Phenols form a highly colored complex with ferric chloride (Fe^{3+} ion). Aspirin is not a phenol, because it does not possess a hydroxyl group directly attached to the benzene ring. Because aspirin will not give the color reaction with ferric chloride, the presence of salicylic acid in the final product is easily detected. The purity of your product will also be determined by obtaining the melting point.

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ML Yell



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