

# Inorganic and organic chemistry assignment



It is commonly used to reduce minor pain and aches (analgesics), lower fever (antipyretic), thin the blood (anticoagulation), reduce the inflammation such as rheumatism and arthritis (anti-inflammatory), enhances the elimination of uric acid (gout) and reduce the risk of stroke, heart attack and other heart problems in long-term low-doses. The related compound of aspirin from willow bark (*Salix alba*) has been used to relieve pain and reduce fever since hundred of years ago.

The Chinese used the willow bark as a remedy in 500 BC while the record that willow bark could ease aches and pains and lower fever was written by Hippocrates, a Greek physician in 400 BC. However, it was only until 1763 when the Reverend Edward Stone discovered salicylic acid. In 1828, a French pharmacist, Henry Leroy isolated salicylic acid, the active extract of the bark into the crystalline form and Rafael Pair, an Italian chemist separated the salicylic acid in its pure state. It was named salicylic acid (SA), as it was acidic. In 1939, German researchers also isolated the compound from meadowsweet flowers.

Despite being effective, it caused digestive problems and even death. In 1897, Felix Hoffmann, an employee of Bayer & Co in Germany synthesized a stable form of salicylic acid namely aspirin which relieved his father's rheumatism. Bayer patented aspirin on 6 March 1899. One of the reactions used to synthesize aspirin is esterification whereby an acid reacts with an alcohol to produce an ester with the presence of an acid catalyst commonly conc. Sulfuric acid. Esterification Some esters are solids which mostly are insoluble in water as they have a high molecular weight or other properties.

Crystallization is one of the methods used to separate esters from the mixture. The chemical equation The symbolic equation In this reaction, the -OH group from salicylic acid is substituted with -COHO group from acetic anhydride to form ester while the carboxylic acid group of salicylic acid remains unchanged. This reaction is slow because of the pure acetic anhydride involved; therefore, a catalyst, namely concentrated Sulfuric acid is added to speed up the reaction. Synthesis of aspirin involves two steps, which are isolation and reclassification.

Having filtered, cold water was used to wash the crude aspirin to isolate the most of the impurities leaving the crude product and pure solvent on the filter paper. Impotent in reclassification is the solvent used as it has to be able to dissolve the aspirin near its boiling point so that the crystals can be formed during the cooling leaving the impurities dissolved. Sometimes, the best reclassification solvent is a mixture of two miscible solvents, one which dissolves the compound readily, the other which does not (5).

In this experiment a mixed solvent of ethanol and water was used as aspirin is very soluble in ethanol but quite insoluble in water (5). Procedure a) Preparation of Aspirin I. 2. 4 grams of salicylic acid was placed in a dry 100 ml conical flask. T. In the fumed, 6 ml of acetic anhydride was added. Safety Note: Acetic anhydride is a strong irritant and it is corrosive and volatile. Contact with skin should be avoided and the vapor should not be breathed iii. Three or four drops of con. Sulfuric acid was added to the mixture and was swirled to mix ' v.

The mixture was heated in a water bath for 10-15 minutes to complete the reaction. V. The flask was removed from water bath. While it is hot, about 1 ml of distilled water was cautiously added from a dropper to decompose the excess acetic anhydride. V'. An additional of 40 ml of cold water was added to the mixture. The mixture was stirred and rubbed with stirring rod if necessary to induce crystallization. Vial. The product was collected by suction filtration and is washed with a little of cold water. Viii. The crude aspirin was recrystallized with ethanol/water. ) Reclassification of Aspirin The crude product aspirin prepared was relatively impure and might be purified by reclassification. A solvent convenient for this crystallization process was a mixture of ethanol and water. Conical flask. It is warmed on a hot plate if necessary I'. Approximately 30 ml of hot water was added to the solution. Iii. If a solid separates out at this point, the solution is warmed until solid dissolves completely. Iv. The solution was cooled. V. The crystals was filtered using suction filtration and is dried in the oven (100 co) for 15-20 minutes. Vi.

Table 1 shows the amount of ecstastically synthesized compared to the amount of salicylic used. Theoretically, the ratio of the number of moles of the ecstastically acid and the salicylic acid is 1: 1 according to the equation below. Therefore, the weight of aspirin produced should be 3. 1 g. According to Table 1, the ecstastically produced was only 1. Egg instead of 3. 1 g and the percentage yield was only 37. %, which was quite low as the expected percentage yield obtained should be over 60%. This proved that many aspirin crystals formed were lost during the preparation and reclassification.

The possible causes during preparation and reclassification of aspirin could be firstly, after adding 1 ml of distilled water, the excess acetic anhydride was not decomposed completely which would affect the forming of aspirin crystals. Then after adding additional of 40 ml of cool water and the flask was cooled down in an ice bath, the time used to cool the flask might not be long enough as resulting in the incomplete forming of crystals. In addition, the ice bath used might not be cold enough therefore; the reclassification was probably not at the maximum rate.

After that during filtration, some of the crystals formed might rediscover and be filtered as a lot of cold water was poured in to clear the flask and the Boucher funnel. The chance of rediscovering would be greater as the large amount of water was used. Surface of Boucher funnel and in spatula upon transferring the crystals to watch glass. Last but not least, upon transferring the crystals out of the desiccators, the crystals might absorb moisture from the surrounding resulting in the error of the weight of the aspirin produced.

Table 2 shows the range of melting point of the ecstastically acid observed compared to its theoretical melting point. A pure substance has one sharp melting point or liquefies within a narrow range. Whereas an impure substance melts over a range of temperature as the impurities cause the lowering and broadening of the temperature (1). The impure substances usually have lower melting point than the pure substance. The range of melting point ecstastically acid was 133-135°C while the pure ecstastically has melting point of 135°C.