# Esterification

To synthesis ecstatically acid by stratification. Introduction: A pleasant, often fruity, odor is characteristic of some of the simpler esters. Ethyl butyrate smells similar to pineapples, the odor of n-propel acetate is reminiscent of pears and isopleths acetate has a strong banana fragrance. Esters are derivatives of the carboxylic acids and contain the following functional group: The synthesis of an ester can be accomplished in one of several ways. An stratification occurs when an alcohol and a carboxylic acid are reacted in the reserve of a mineral acid catalyst, such as sulfuric acid.

Because these reactions result in an equilibrium mixture of both products and reactants, the reaction conditions must be manipulated in order to produce a reasonable yield. A large excess of one of the reactants can be used in the starting mixture or alternatively, one of the products can be removed as the reaction proceeds (as by a distillation), to shift the equilibrium to the right. Other synthetic pathways to esters also exist. Acid chlorides react with alcohols to yield an ester and hydrochloric acid.

A small amount of pyridine is usually deed to the reaction mixture to neutralize the resulting acid. Acid anhydride can also react with alcohols to produce esters. Unlike the reactions between carboxylic acids and alcohols, neither of these two methods for preparing esters results in an equilibrium mixture. Ecstatically acid, or aspirin, is one of the most widely used and versatile drugs known today. It was first synthesized by Charles von Gerhardt in 1 853 and was later patented by a German dye chemist named Frederica Brayer in 1893 who recognized its potential as an analgesic (pain reliever).

Salicylic acid, a impotent of willow and poplar bark, had been used as a pain killer for centuries, but its highly acidic property caused irritation of the mucous membranes of the mouth and throat and also resulted in uncomfortable gastric pain. By transforming the acidic phenol functionality into an ester group, the compound retained its analgesic properties but lost some of its irritating side- effects. In addition to relieving pain, aspirin is also an antipathetic (fever reducer) and an anti-inflammatory agent (used for arthritis). Like all drugs, aspirin also has some undesirable side effects which should be tote.

When taken in large quantities (several grams per day), gastric problems can result. Its use has been implicated in Rexes syndrome, a brain disorder that can affect people under the age of 18. Some people are highly allergic to aspirin. Finally, aspirin interferes with platelets and affects normal blood clotting which can lead to hemorrhaging in extreme cases. However, its anticoagulant properties can also be used to advantage in preventing blood clots from forming in the arteries. Recent studies have shown that the consumption of one half of an aspirin tablet per day can help to prevent heart attacks and strokes.

Aspirin can be synthesized by the stratification of salicylic acid via reaction with acetic anhydride. A few drops of phosphoric acid are added to serve as a catalyst for the reaction. The use of acetic anhydride as a reactant, instead of acetic acid, results in a rapid and irreversible conversion of salicylic acid to ecstatically acid. Experimental Procedure: Synthesis of ecstatically acid 1 . A 400 ml beaker containing about 150 ml of water was heated to the boiling point on a hot plate. 2. 2 g of salicylic acid was weighed out and it was

placed in a 100 ml conical flask. . 5 ml of acetic anhydride was added into the conical flask. . 5 drops of 85% phosphoric acid, HAPPY was added. The flask was swirled to mix the reactants and then it was clamped in the boiling water bath. It was heated for about 8 minutes. 5. The flask was removed carefully from the hot water bath and the hot plate was turned off. 2 ml of distilled water was added cautiously to the flask. The water will react with any unrelated acetic anhydride which remains. 6. Once the reaction between acetic anhydride and water had subsided, 40 ml Of water Was added to the flask. 7. The contents was allowed to cool at room temperature for minutes then the flask was placed in ice. . The contents was stirred with a glass rod periodically during this cooling period. The solid product was collected by vacuum filtration and it was washed with cooled water. 9. A small portion (spatula-tip full, 20-30 MGM) of this crude ecstatically acid was set aside. 10. To recitalist the ecstatically acid, it was transferred to a 100 ml conical flask and 10 ml of 95% ethanol was added. 1 1. The flask was heated slowly on a hot plate just until the aspirin completely dissolved. As soon as all of the solid has dissolved, the flask was removed from the hot plate. 12.

Water was added to the solution in 5 ml portions, it was swirled after each addition, until a total of 40 ml of water has been added. 13. The flask was placed in an ice bath for 10 minutes to complete the crystallization. 14. The purified aspirin was collected by vacuum filtration. The crystals was washed in the funnel with ice-cold water. The vacuum was left on for a few minutes to air dry the purified aspirin, the crystals was then pressed between two pieces of filter paper to thoroughly dry them. 5. The mass, yield and melting point of dried aspirin were obtained. Results: Weight of salicylic acid used: 2.

0021 g Weight of watch glass: 20. 536 g Weight of watch glass t product: 21. 6651 g Weight of product: 1. 3115 g no. Of mole of salicylic acid: = 0. 01450 mol Volume Of acetic anhydride used 5 ml Density of acetic anhydride = 1. 082 g/ml Mass of acetic anhydride used = 1. 082 g/ml x 5 ml = 5. 41 g no. Of mole of acetic anhydride: - 0. 0530 mol = 3. 66> 1 Since the mol of salicylic acid is smaller than the mol of acetic anhydride, salicylic acid is a limiting agent. Mol of salicylic acid produced 1 mol of ecstatically acid. 0. 01450 mol of salicylic acid produced 0. 01450 mol of ecstatically acid. Theoretical weight of ecstatically acid: 0. 1450 mol x 180. 16 gnu-mol = 2. 6123 g Percentage yield: x 100% = 50. 20 % 50 % Melting point of aspirin: 138 - 140 co Discussion: Aspirin is an effective analgesic (pain reliever), antipathetic (fever reducer) and anti-inflammatory' agent and is one of the most widely used non-prescription drugs. The active ingredient in aspirin was found to be salicylic acid. The structure of salicylic acid is shown below. In this experiment, aspirin is formed from the reaction between salicylic acid and acetic anhydride through stratification. Aspirin can be made by reacting salicylic acid with acetic acid in the presence of an acid catalyst.

The phenol group on the salicylic acid forms an ester with the carboxylic group on the acetic acid. However, this reaction is slow and has a relatively low yield. If acetic anhydride was used instead of acetic acid, the reaction will be much faster and has a higher yield (since acetic anhydride is much more reactive than acetic acid). Therefore, we used acetic anhydride for shorten the period f stratification. In order for reaction to occur faster, phosphoric acid was added as a catalyst. Then the solution was being placed

in the hot water bath to heat up and thus triggering the reaction between salicylic acid and acetic anhydride.

The reaction was shown as below: In this experiment, the salicylic acid is the limiting reactant and the acetic anhydride is in excess. The addition of distilled water into the mixture after it is being removed from the water bath is to let the water react with the remaining acetic anhydride in the mixture which are still not being reacted. A vigorous reaction will occur as the decomposition of the excess acetic anhydride is exothermic. Once the decomposition of the excess acetic anhydride has completed, more water was added, forming completing the reaction.

Now the solution contains two kinds of products, which are ecstatically acid and acetic acid, according to the reaction shown below. The reason why acetic acid is dissolved in this process but not ecstatically acid was due to the hydrogen bonding of acetic acid can form with water while ecstatically acid cannot. This is due to the large carbon group maintained by ecstatically acid which caused it to have difficulties from forming hydrogen bond with water. The aspirin collected will then be purified by rationalizations. In this purification method, the crude aspirin will be dissolved in a small amount of warm ethanol.

Water was then be added and the solution was cooled slowly in the ice bath and then chilled. The ecstatically acid will recitalist, and the solid impurities (unrelated salicylic acid) should remain dissolved in the solution. The solid aspirin will again be collected using vacuum filtration. Rationalizations was needed to obtain a pure product from the crude product. Therefore, this

aspirin should be more pure than the original aspirin. The final product was left in air dry and weighed. The theoretical and percentage yield was calculated. The weight of ecstatically acid obtained was 1. 115 g while the percentage yield of product was 50%. Besides, the melting point of ecstatically acid we obtained was in the range of 1 38 - 140 By comparing these observed melting points with the literature value of 138 - CHIC for ecstatically acid, it was found that the values were exactly the same, indicating that both rodents were ecstatically acid( previously stated as ecstatically acid and acetic acid). The above reaction was an example of an organic synthesis called stratification. Stratification is the acid catcalled reaction of a carboxylic (-COHO) group and an -OH group of an alcohol or phenol to form a carboxylic ester.

In the synthesis of aspirin, the -OH group is the phenol OH group attached to ring of the salicylic acid. The acetic group, ; COACH comes from acetic anhydride, and the reaction is catcalled by phosphoric acid, HAPPY. Salicylic acid has a higher priority as it has a benzene ring which s more stable than the open chain of acetic anhydride because it has more resonance. Salicylic acid reacts better with acetic anhydride than acetic acid, so acetic acid will provide the acetic group which will react with the alcoholic - OH group on the salicylic acid.

The hydrogen ion from the hydroxyl group in the salicylic acid breaks away first, then combines with the oxygen which has a negative charge after acetic anhydride is broken into two parts, which are an alkyl carbonyl group and -? CHOC- group. This is how acetic acid is formed. Later on, the alkyl carbonyl group of acetic anhydride which now has positive charge due to the

lack Of oxygen atom, connects to the oxygen atom which has a negative charge, lastly produced ecstatically acid as a neutral compound product.

The mechanism of how an ecstatically acid formed from stratification reaction will be shown: Precaution steps: 1 . When handling phosphoric acid, students should be aware of spill it out because it is corrosive. 2. When adding water to the heated solution, students should be careful and added in small amount of water because it may splatter to the surroundings due to the vigorous reaction. 3. The elution was allowed to cool at room temperature before putting it into the ice bath for the process of crystallization in order to get crystals in a nicer form and in correct manner.