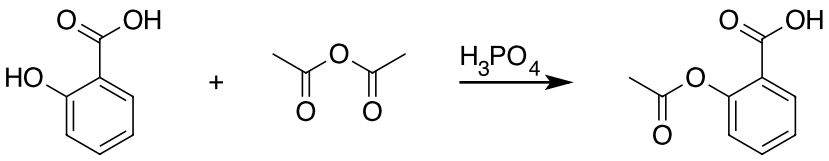
Synthesis of Aspirin

TUD Department of Chemistry

Aspirin is synthesized via the esterification of salicylic acid with acetic anhydride:



Salicylic Acid Acetic Anhydride Aspirin

Place 1.0 g of salicylic acid in a 50 mL Erlenmeyer flask and add 2 mL of acetic anhydride[1,2]. Add a few drops of 85% H_3PO_4, swirl to mix, and place in an 80° C water bath for 20 minutes. Remove the flask from the bath and carefully (the reaction will be vigorous) add 20 drops of water. Cool the flask in an ice bath and add 10 mL of water. You should see crystals begin to form. If no solid forms, scratch the inside of the flask with a glass rod. After the solution is thoroughly cooled, collect the white crystalline product by vacuum filtration. Wash the crystals with no more than 3 mL of ice-cold water[3]. Continue to apply suction until the crystals appear dry. Obtain an approximate crude yield[4] and transfer the crystals to a 50 mL flask for recrystallization.

Recrystallize from hot ethanol and water by dissolving the product in the minimum volume of ethanol (ca. 2-4 mL) and then adding hot water until the solution becomes just cloudy (add no more than 8-10 mL of water). Cool the solution slowly and when it reaches room temperature collect the

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[1] Acetic anhydride reacts with moisture in the air. be sure to tightly cap the bottle quickly after use.

[2] The density of acetic anhydride is about 1.1 g/mL.

[3] The product has appreciable water solubility; excessive washing will decrease your yield.

[4] The crystals will still be wet, so this is only a rough approximation of the crude yield.

recrystallized product by vacuum filtration. Place the crystals in a small beaker and store in the desiccator to dry until the next lab period. At that time weigh the crystals and calculate the %yield. We will also characterize the synthesized aspirin by melting point and IR spectroscopy and will asses its purity using TLC.