# **Isolation of Aspirin from Tablets**

## CHEM HELP ASAP

### experiment video: https://youtu.be/c 9VfN0n3Vo

#### Purpose

This experiment demonstrates a solid-liquid extraction for the isolation of a pure material from a mixture.

## Background

Organic chemistry experiments often emphasize the synthesis of new materials. Another aspect of organic chemistry is the separation of pure substances from a mixture based on the properties of the different components in the mixture. Experiments of this type abound in organic laboratories. Extraction of caffeine from tea and extraction of trimyristin from ground nutmeg are two examples. Both of these involve solid-liquid extractions in which the desired compounds dissolves into a solvent (the liquid) while the other components do not dissolve (the solid).

Another extraction that relies on a solid-liquid extraction is the isolation of aspirin from aspirin tablets. The extraction begins with grinding of the tablets to increase the surface area of the solid material. The solids are then treated with isopropanol. Aspirin or *O*-acetylsalicylic acid, which is the majority of the mass of each tablet, dissolves into the solvent while the other components (e.g., binders and salts in the tablet) remain as a residue. Filtration of the solids affords the aspirin/isopropanol solution. Addition of water causes the aspirin to precipitate. The aspirin can then be recovered through filtration (Scheme 1).



**Scheme 1.** Solid-liquid extraction of aspirin from tablets.

The precipitation of aspirin from isopropanol through addition of water gives a higher yield of product if the water has a low pH. A 1% solution of  $H_2SO_4$  has a pH of approximately 1. The low pH ensures that essentially all the aspirin is present in its neutral, carboxylic acid form, which is less soluble in water (Scheme 2).



Scheme 2. Acid-base equilibrium of aspirin

#### Procedure

Procedure adapted from ... <u>https://bit.ly/3prNiLe</u> (This is not a proper citation, but the web document has no citation information).

Grind 30 aspirin tablets (325 mg aspirin per tablet) in a mortar and pestle. Transfer the solids into a 125-mL Erlenmeyer flask. Add 50 mL isopropanol and allow the mixture to stir for 30 min. Filter the resulting mixture into a 250-mL Erlenmeyer flask. Use a minimal volume of fresh isopropanol to rinse the original flask and filter paper. Transfer the isopropanol solution into 250 mL of 1% aqueous H<sub>2</sub>SO<sub>4</sub>. Cool the solution in an ice bath for 30 min to precipitate aspirin. Filter the solids with a Büchner funnel. Rinse the product with cold 1% aqueous H<sub>2</sub>SO<sub>4</sub>. Spread the collected solid on a tared watch glass and allow the solid to dry. Determine the mass of the product and percent recovery based on the amount of aspirin in the original tablets. Determine the melting range of the product and compare it to the literature value for aspirin. Interpret the provided NMR spectrum of the product. Record all observations in your notebook.