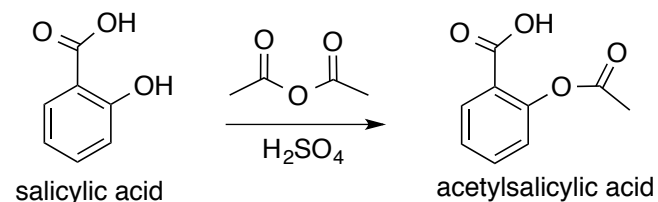


## The Synthesis of Acetylsalicylic Acid (Aspirin)

Here we are taking the salicylic product that we made and making aspirin from it. In this case we are making an ester by reaction the phenol hydroxyl with acetic anhydride (an activated carboxylic acid). The purification is very simple and you should be rewarded with plenty of white solid.



### Required Chemicals/Amounts and Glassware

2.5 ml of acetic anhydride

3-5 drops of concentrated sulfuric acid

4ml of ethanol

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50ml Erlenmeyer flask

Buchner funnel (used for vacuum filtration)

50ml beaker

Watch-glass (to cover beaker)

### Step 1

Before beginning this step, make sure salicylic acid product is thoroughly dry. Weigh about 1.25 grams of salicylic acid. Put the weighed sample into an Erlenmeyer flask. Add 2.5 ml of acetic anhydride to the salicylic acid. Make sure to add the sample under the hood for this step, since acetic anhydride fumes are toxic.

Now add 3-5 drops of concentrated sulfuric acid from a dropper and mix the chemicals well by swirling the flask lightly. The mixture may become warm from the exothermic reaction; allow to cool for about ten minutes. To complete the reaction, heat for five minutes in a 45-55° water bath. Hot tap water can be used to make the bath.

Chill mixture in an ice-water bath and scratch the bottom of the flask with a stirring rod until a semi-crystalline paste has formed. The tiny pieces of glass made by scratching serve as nuclei for aspirin crystals. Remove the flask from the ice-water bath, once the paste has formed. Add 10-15ml of cold water and 15 grams of ice to the flask. Stir the mixture with a stirring rod to break up the pasty solid and to melt the ice.

Once the ice has melted, filter the mixture by vacuum filtration in a Buchner funnel. Rinse the remaining crystals that have collected in the flask out with cold water and onto the filter (see figure 1).

#### Step 2 (Recrystallize the Aspirin)

Recrystallize the aspirin by dissolving it in 4ml of ethanol (not more) in a 50ml beaker. If the crystals do not dissolve at room temperature, warm the mixture with a hot bath.

Probably no filtration is necessary at this stage; however, if the ethanol solution has any insoluble particles in it, filter it through a small fluted filter paper. If you carry out the filtration step, you will have to rinse off the filter paper carefully with another millimeter of ethanol to recover all of the aspirin and also to reheat the filtrate so that all of the aspirin is completely dissolved.

Pour 10ml of warm water into the solution (hot tap water is acceptable). Cover the beaker with a watch-glass to retard evaporation and let the solution cool at room temperature for 10-15 minutes. Set the beaker in an ice bath to accelerate the cooling process. When it looks as though recrystallization is complete, filter the solid using vacuum filtration.