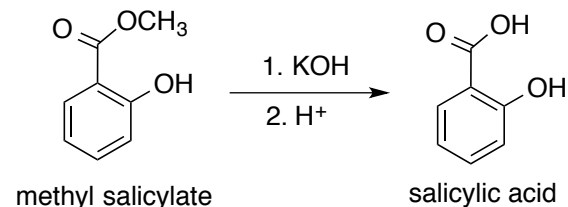


Synthesis of Salicylic Acid

This reaction is simply a hydrolysis of an ester to a carboxylic acid. If you recall the separations lab you did last semester, you will also recognize the purification strategy employed.



Required Chemicals/Amounts and Glassware

5 g sodium hydroxide

2.2ml/2.5g of methyl salicylate

70-75 ml of 1 M sulfuric acid

100ml round-bottom flask (used for reflux)

100ml, 125/150ml, and 250ml Erlenmeyer flask

Buchner funnel (used for vacuum filtration)

Step 1

Weigh out 5 g of sodium hydroxide and dissolve it in 25 ml of water (deionized water) in a 100 ml round-bottom flask. Mix this solution with 2.2 ml of methyl salicylate (2.5g, 0.033 mol). A white solid will quickly form (the white solid is the salt from reaction 1). Attach a reflux condenser to begin the reflux process. Make sure to secure the condenser with clamps on a stand (see figure 1).

Before the reflux begins, add one or two boiling chips to the reaction mixture to prevent violent boiling or bumping of the solution when it is heated. Heat the solution at boiling for 15 minutes using a heating mantle. All of the solid which formed initially will go into solution as it is warmed.

After refluxing the reaction for 15 minutes, turn off the heating mantle and remove the reflux condenser. Let the mixture cool to about 50° (until cool enough to touch). Add a few pieces of ice, bringing the temperature to 30° or so.

Obtain 70-75 ml of 1 M sulfuric acid solution and place in a 100 ml Erlenmeyer flask. Carefully add 5 ml of the H₂SO₄ solution at a time. Swirl the reaction mixture to mix it up, and then add more as needed. Add the H₂SO₄ solution until a heavy white precipitate of the salicylic acid forms and remains in the mixture when it is well mixed. Approximately 60-75 ml will need to be added (a slight solid will begin to form when about 40ml is added). If the mixture becomes hot, cool it a bit before adding more acid.

After you have added enough 1 M sulfuric acid to give a heavy white precipitate, add 5 ml more acid to insure complete precipitation of the salicylic acid. In order to cool the solution, place in an ice bath and allow to cool for 5-10 minutes. While the solution is cooling, set up vacuum filtration using a Buchner funnel (secure with stand). Once the solution has cooled, place in vacuum filter to separate the solid and liquid. Remaining solid can be rinsed with deionized water (see figure 2).

Step 2 (Recrystallization of Salicylic Acid)

Using a hot plate, heat up 55 ml of water in a 125/150 ml Erlenmeyer flask, water should be hot but not boiling. To do the recrystallization, add the crude salicylic acid to the water (small chunks will form, swirl slightly to dissolve). When most of the salicylic acid has dissolved, heat the mixture to boiling (add one or two boiling chips to prevent violent boiling or bumping). If the solid does not completely dissolve, add a few more ml of hot water to complete the dissolution.

If the water of salicylic acid solution has some insoluble materials present other than boiling chips that were used, the solution needs to be filtered. If filtration is not necessary, go directly to cooling step.

Step 3 (Cooling the solution)

Allow solution to cool to room temperature on lab-bench top. Then cool in an ice-water bath to 0-5° for 10-15 minutes. Filter the crystals of salicylic acid using vacuum filtration. Rinse remaining liquid with water. When filtration is complete, pour a few milliliters of ice-cold water through the crystals to dissolve any impurities that may have clung to them.

Pull air through the crystals for a few minutes to help their drying. Water does not evaporate quickly, so crystals should be kept in an open space over night, or until the following week of lab. Clean up materials and place top of Buchner funnel in 250 ml Erlenmeyer flask to dry.

Step 4 (Weighing Product)

Once salicylic acid product is thoroughly dry, weigh product. Calculate percentage yield. A yield of 65% is the norm in the experiment.

Step 5 (Recording Melting Point)

To obtain melting point of final product, take three samples product and place into melting point apparatus. Reference tables give its melting point as 159° or 160°. Will probably observe a melting range of two to three degrees.