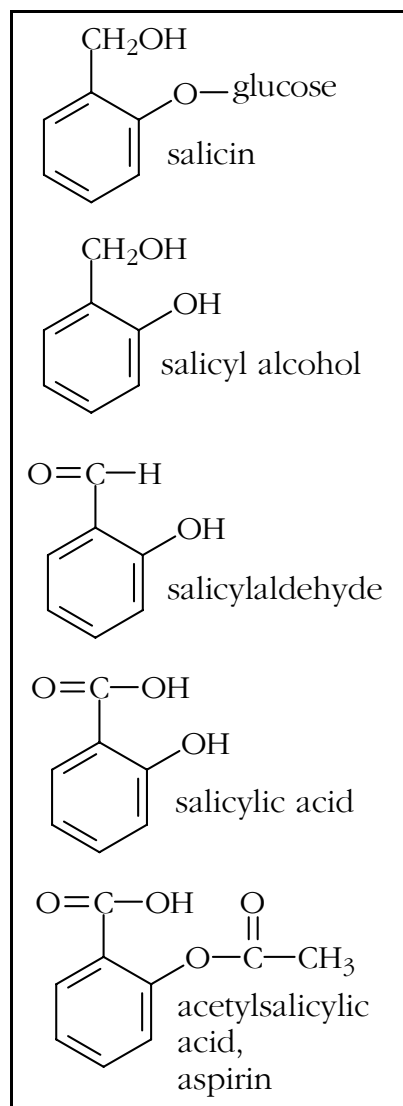


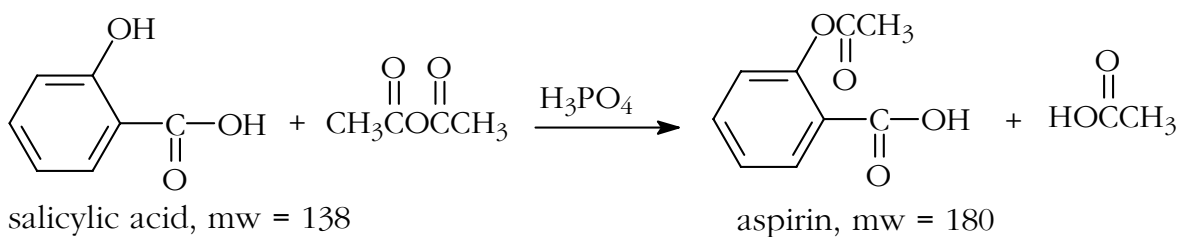
EXPERIMENT #7 – PREPARATION OF ASPIRIN

Introduction

In early-1800, German Egyptologist Georg Ebers bought papyruses from an Egyptian street vendor. The Ebers Papyrus, as they are known, contained a collection of 877 medicinal recipes from Egypt dating to about 2500 BC. Among these recipes was a recommendation for an infusion of dried myrtle leaves for rheumatic and back pain. Hippocrates of Kos (*ca.* 400 BC), often considered the father of modern medicine, recommended a tea extract from the bark of the willow tree for fever and pain. The *antipyretic* (fever-reducer) *analgesic* (pain-reliever) found in these plants is *salicin* (named for *Salix*, Latin for willow), which was isolated by Johann Buchner in 1828 at the University of Munich. Salicin is in a class of compounds known as *glycosides*. Glycosides are compounds that have a sugar portion (*glycose*) bonded to a non-glycose part (the *aglycone*). The aglycone in salicin is salicyl alcohol which is the completely reduced form of salicylic acid. In 1838, Raffaele Piria, working at the Sorbonne in Paris, split salicin into glucose and salicylaldehyde by oxidation and hydrolysis. He then converted the salicylaldehyde, by oxidation, to an acid of crystallized colorless needles, which he named salicylic acid. Salicylic acid had antipyretic and analgesic qualities; unfortunately it was too harsh to the linings of the mouth, esophagus and stomach. On August 10, 1897, Felix Hoffmann, a chemist in the Bayer chemical factory in Germany, prepared the first pure sample of acetylsalicylic acid, which Bayer eventually called aspirin. It, too, had antipyretic and analgesic properties. Although aspirin is more gentle to the stomach than salicylic acid, it can cause ulcers. Since then aspirin has been found to be helpful in preventing strokes and heart attacks. It does this by inhibiting the production of prostaglandins, which are involved in blood clot formation and pain production.



Objective



You will synthesize aspirin and test its purity using the ferric chloride test.

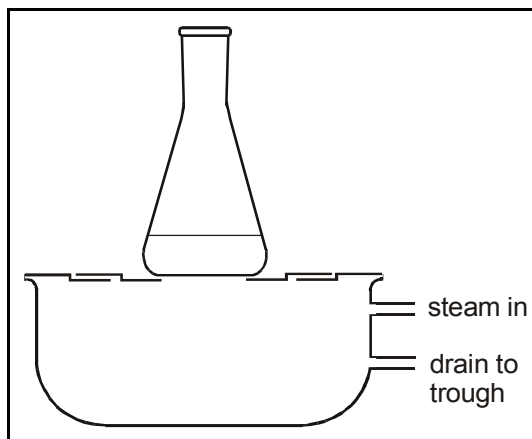
Procedure

Caution!

Acetic anhydride and phosphoric acid are caustic. If any of either gets on you, wash it off with lots of water. Wear goggles, as usual.

1. Set up a steam bath. The top tubing connection is connected by rubber tubing to the steam spigot and rubber tubing is run from the bottom connection to the trough to allow steam and water to drain.

2. Weigh 2.0 g of salicylic acid into a 50 ml Erlenmeyer flask. Using your 10 ml graduated cylinder, add 3 ml of acetic anhydride to the flask and, while swirling, add 3 drops of concentrated phosphoric acid. Place a cork *loosely* into the neck of the flask. **Do not press it into the neck.** We are trying to reduce the evaporation of acetic anhydride, but do not want a pressure build-up in the flask.



Steam Bath with Erlenmeyer Flask

3. Set up the steam bath, using the appropriate number of rings to support the flask, allowing an opening in the center so the steam can heat the flask. Turn on the steam to generate a moderate flow rate. Some water may need to be expelled from the steam line before the steam starts to flow. When steam is coming out of the steam bath, set the flask on the bath. Heat for 15 minutes, swirling the flask occasionally. The solid should dissolve.

4. Remove the Erlenmeyer flask from the bath and let it cool to room temperature. Then slowly pour the solution into a 150 ml beaker containing 20 ml of ice water, mix thoroughly, and place the beaker in an ice bath. Acetic anhydride will be hydrolyzed to acetic acid and aspirin should precipitate in the cold water.

5. Collect the crystals in a Buchner funnel. [Your instructor will demonstrate how to do this.]

6. Wash the crystals with two 5 ml portions of ice water, followed by one 10 ml portion of ice cold ethanol. Allow air to be drawn through the crystals for 5-10 minutes.

7. Store the crystals in a beaker until next time when you will weigh them, test them for purity, and calculate the percent yield.

8. Next Time – Weigh the aspirin. Label 3 10x75mm test tubes: salicylic acid, my aspirin, commercial aspirin. Place a small sample of the corresponding material into each tube. Add 20 drops of deionized water to each tube and agitate to dissolve the solids. Add 10 drops of 1% aqueous iron(III) chloride to each tube; agitate. Record your results; a purple color indicates the presence of salicylic acid.