

Dr. BRR. GOVERNMENT COLLEGE, JADCHERLA, MAHABUBNAGAR (Dist.) DEPARTMENT OF CHEMISTRY Student Study Project 2021 -22

Topic PREPARATION OF ASPIRIN

Conducted by students

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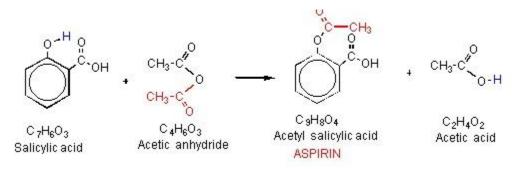
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Introduction:

Aspirin is the common name for the compound acetylsalicylic acid, widely used as a fever reducer and as a pain killer. Salicylic acid, whose name comes from Salix, the willow family of plants, was derived from willow bark extracts. In folk medicine, willow bark teas were used as headache remedies and other tonics. Nowadays, salicylic acid is administered in the form of aspirin which is less irritating to the stomach than salicylic acid.

Objective:

To prepare aspirin, salicylic acid is reacted with an excess of acetic anhydride. A small amount of a strong acid is used as a catalyst which speeds up the reaction. In this experiment, phosphoric acid will be used as the catalyst. The excess acetic acid will be quenched with the addition of water. The aspirin product is not very soluble in water so the aspirin product will precipitate when water is added. The synthesis reaction of aspirin is shown below:



The aspirin isolated as "crude product". If impurities are present in crude sample, the melting point range for product will be lower than the range of pure aspirin. Melting point range of the salicylic acid (starting material) is 158-161 °C. The purity of the product was also analyzed by melting point apparatus. The melting point range of pure aspirin is 138-140°C.

1. Weigh out 3.0 g of salicylic acid and place in a 250 ml Erlenmeyer flask.

2. Measure out 6.0 ml of acetic anhydride and add this to flask. Don't let the acetic anhydride contact skin and don't get the vapors in eyes.

3. Carefully add 5 to 10 drops of 85% phosphoric acid, a catalyst, to the flask and swirl to mix everything thoroughly.

4. Still in the hood, heat the mixture for about 10 min. in a beaker of warm water (70-80 oC).

5. After heating, cautiously add 20 drops of distilled water.

6. Next add 20 ml of distilled water and cool in an ice bath. If crystals do not appear, scratch the walls of the flask with a stirring rod to induce crystallization.

7. Filter the solid aspirin through a piece of pre-weighed filter paper using a Buchner funnel and the aspirator. Wash the crystals with 2-3 ml of chilled water.

8. Measure the melting point range with the Meltemp Apparatus and compare to the value for pure aspirin of 138-140 oC.

9. Calculate the weight of the product by subtracting the weight of the paper from the total. The theoretical (maximum) yield is 3.9 grams.

Safety Considerations:

This experiment uses salicylic acid, acetic anhydride and phosphoric acid. The salicylic acid and aspirin may cause irritation to your skin or eyes, but are basically not hazardous. An excess of these can be disposed of in the sink or if packaged, in the trash. If spill some, wipe it up with a wet paper towel and throw the towel in the trash. The acetic anhydride and phosphoric acid can cause bad burns. Use them in the hood. Be sure to wear gloves and safety goggles when using these chemicals. Excess chemicals must be disposed of in the plastic tub of water. This will convert the acetic anhydride to vinegar and dilute the phosphoric acid.

References:

- $1.\ http://www.teachersfirst.com/lessons/forensics/ink-lab.html$
- $2.\ www.reachout michigan.org/funex periments/quick/csustan/mrsketch.htm$