

Introduction: Aspirin, or acetylsalicylic acid, is an over the counter medication known today to be useful in relieving muscle pain, headaches, and joint aches. It also is a fever reducer. It works by inhibiting blood from clotting that make the nerve endings receptive to pain. The earliest records of aspirin date all the way back to Hippocrates when he used an extract from the bark of a willow tree to relieve pain and fever symptoms. Johann Buchner, a pharmaceutical professor at the University of Munich, was the first to isolate salicin. In 1829, one year later, Henri Leroux improved Buchner's process of isolation and received 30g of salicin from 1.5kg of bark. At last, in 1838, an Italian chemist by name of Raffaele Piria split the salicin into salicylaldehyde, then converted that into salicylic acid through the processes of hydrolysis and oxidation. Aspirin was patented in 1899 by a chemist named Felix Hofmann who worked for Bayer in Germany. Now, 50 million 5 grain tablets are consumed daily.

Organic chemistry covers a wide range of molecules that all contain hydrogen and carbon. Hydrocarbons are molecules that contain only hydrogen and carbon. Aspirin is composed of oxygen, hydrogen, and carbon atoms, with a chemical formula of $C_9H_8O_4$. The organic synthesis of aspirin involves combining salicylic acid ($C_6H_6O_3$) with acetic anhydride ($C_4H_6O_3$) to make both the acetylsalicylic acid ($C_9H_8O_4$) and byproduct acetic acid ($C_2H_4O_2$)

Aspirin Synthesis Lab

Purpose: To experiment the process of making a pharmaceutical drug.

Materials:

3 Trays

125ml erlenmeyer flask

10ml and 50ml graduated cylinders

Distilled water

Hot bath

Ice bath

2.00g salicylic acid

0.40g sodium acetate

Bunker funnel and filter flask apparatus

600ml beaker

Squirt bottle

Microspatula

Procedure:

- 1). Using the laboratory balance, weight out both 2.00g of salicylic acid and 0.40g of sodium acetate in separate trays. Put measured amount of salicylic acid into the 125ml erlinmyer flask.
- 2). Bring flask and measured out tray of sodium acetate underneath the chemical fume hood. Measure out 2.5 ml of acetic anhydride into the 10ml graduated cylinder. Transfer over to the erlinmyer flask with the salicylic acid. Add sodium acetate. Shake around, mixing well, then place in the hot bath for 20 minutes.
- 3). Take flask out of the hot bath then add two drops of distiller water. Leave for 5 minutes outside of hot bath but still under fume hood. Meanwhile, measure out 30ml of distiller water in the 50ml graduated cylinder.
- 4). Add the 30ml of distiller water to the combined acids in the erlinmyer flask, mix around by gently shaking, then place into the ice bath for 30 minutes. (Someone keep track of the time, please! :))
- 5). The crystallization should of occurred where the acetic acid should have separated from the acetylsalicylic acid. Go over to the sink with the hooked up bunker funnel and filter flask apparatus, and make sure all connections are secure and there are no leaks. Pour contents of erlinmyer flask into the filter, then take the squirt bottle and squeeze a tiny amount of water into the erlinmyer flask to flush it out and add the remaining contents to the filter. Use the Microspatula to get out any excess chunks of crystalized acid. Turn on the and run for 5 minutes while occasionally stirring crystals in filter to separate out all of the acetic acid.
- 6). What remains in the filter is the acetylcyclic acid, or aspirin, in its purest form. Get a mass on this final product using the laboratory balance and record it.
- 7). DO NOT throw away the final product even though Mrs.M has said so. Use the Microspatula to transfer it into the 15ml test tube, seal, and store.

Data Analysis

Data Table:

Flask number: 3

Mass of salicylic acid: 2.00 g

Mass of sodium acetate: .4 g

Volume of acetic anhydride: 2.5 ml

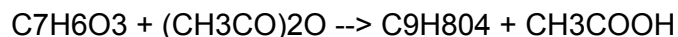
Time in hot water bath: 20 minutes

Time out of hot water bath: 5 minutes

Time in ice bath: 30 minutes

Time out of ice bath: 5 minutes

Mass of product (final): 2.18g



molar mass of salicylic acid ($\text{C}_7\text{H}_6\text{O}_3$)= 138g

molar mass of Aspirin ($\text{C}_9\text{H}_8\text{O}_4$)=180g

Actual amount of salicylic acid ($\text{C}_7\text{H}_6\text{O}_3$) used in lab=2.61g

Actual Yield: 2.18g Aspirin ($\text{C}_9\text{H}_8\text{O}_4$)

Theoretical Yield of Aspirin calculation:

$$=(\text{Actual amount of salicylic acid } (\text{C}_7\text{H}_6\text{O}_3) \text{ used in lab}) \times \left\{ \frac{\text{molar mass of Aspirin } (\text{C}_9\text{H}_8\text{O}_4)}{\text{molar mass of salicylic acid } (\text{C}_7\text{H}_6\text{O}_3)} \right\} = 2.18\text{g}$$

Actual Yield: 2.18 g

Percent yield: $(\text{actual}/\text{theoretical}) \times 100 = 100.9\%$

Percent Error: $((\text{actual} - \text{theoretical})/\text{theoretical}) \times 100 = -16.47\%$

Conclusion: The purpose of this experiment was to experiment with making a pharmaceutical drug. Our purpose was satisfied with how we succeeded in making aspirin. The actual yield of the aspirin was obtained through a filtration method in which the acetylsalicylic acid separated from the acetic acid, leaving only the product we wanted. We did, however, have a percentage error, and the reason for our error was that some acetylsalicylic acid was lost in the experiment. This could have been because all of the final product was not scraped with the Microspatula into the tray we measured the final mass in. Also, the crystallization process may have not been completely thorough, in that more crystals could have been produced if the ice bath was more accurate towards the ideal temperature for crystallization or there were more time for the process of crystallization altogether.

Aspirin Coating Lab

Introduction: Enteric coatings are used on medications that are irritants to the stomach lining. Acetylsalicylic acid can irritate mucosa in the gastrointestinal tract causing problems such as gastrointestinal bleeding, heart burn, nausea, vomiting, hemorrhage, peptic ulcers, and esophageal ulcerations. In order to prevent these side effects, an enteric coating is needed so that the aspirin does not dissolve in the stomach but rather in the alkaline environment in the small intestine.

Purpose: To make aspirin safer on gastrointestinal tract by adding a coating.

Materials:

20ul micro pipets

100ml graduated cylinder

Heating stir plate

3 envelopes gelatin

75 ml water

Shellac

Tweezers

Paintbrush

125ml erlenmeyer flask

Vegetable oil

Drop tray

- 1). With gloved hand, use fingertip to coat the drop trays with vegetable oil.
- 2). Measure 75 ml of water in erlenmeyer flask, bring over to heating stir plate and bring to a boil.
- 3). Add the three packets of gelatin and boil until fully dissolved.
- 4). Remove from hot plate. Place correct tip on micropipet and adjust to 12.5ul. Extract gelatin using the micropipet and place 3 injections (37.5ul total) in one section of drop plate. Repeat until all the sections have 37.5 ul gelatin.
- 5). Using tweezers, pick up approximately 5 grains of aspirin (tip of the tweezers) and using

paintbrush dipped in shellac, paint over the aspirin grains still held between tweezers. Once coat is applied, place in the center of one gelatin section on drop plate. Repeat until every section on the drop plate has shellac coated aspirin.

6). Using remaining gelatin and micropipet, transfer 37.5 ul of remaining gelatin on top of the shellac coated aspirin and gelatin already on the drop plate.

7). Store overnight

8) Take pills out of drop tray and store in prescription bottle, sealable jar, or test tube

Data Analysis

After performing this experiment, our capsule making was a success. We made 21 pills with a hard, outer coating.

Problems: This was the very first time this experiment has been performed. Being a completely new experiment, Larissa left much room for improvement. Most everything went well, however, the gelatin and water mixture in the erlinmyer flask had boiled over at one point. Also, we had originally planned on putting 125ul of solution into the drop plate using a micropipet but couldn't because our micropipets we had in the lab were not big enough. But 37.5ul (75ul total) in each drop tray section seemed to make a good size capsule. Our pills looked flat as well because of the way the drop tray molded them to look that way. Then method of painting over the acid crystals was a bit tedious in that the process was slow and labor intensive. No weights (in grams) were measured or recorded to find the exact quantities of aspirin and shellac that were used in making our pills.

Solutions:

- bigger micropipet

- bigger erlinmyer flask so the solution doesn't boil over and result in a lengthy cleanup process

- better mold instead of a drop tray to make our pills look more like capsules found on the market.

- record and measure all aspirin crystals that were used for each pill along with the shellac that was applied.

- perhaps pre-measuring and setting aside the acid crystals, then painting them over with shellac prior to making the gelatin would have been a better way to go about the procedure.

Conclusion:

We intended to make a coating to make aspirin safer to consume. Our experiment was successful in that we did make coatings for our acetylcyclic acid crystals, thus, made our aspirin better on the gastrointestinal tract if it is to be consumed. Again, this was a completely

new experiment, hence more pills could be made in the future if the problems we ran into are resolved the next time this experiment is performed.





