

Synthesis of acetylsalicylic acid



**ASSIGN
BUSTER**

1. Introduction

The goal of this experiment is to perform a Fischer esterification reaction with salicylic acid and acetic anhydride to synthesize acetylsalicylic acid.¹ The carboxyl group on the acetic acid reacts with the hydroxyl group on the salicylic acid to make an ester product of acetylsalicylic acid.² This esterification reaction is in the presence of a phosphoric acid catalyst which speeds up the rate of the reaction without interfering with the end product.¹ Acetylsalicylic acid also goes by the common name Aspirin which is a drug used for pain relief, fever, and reduction of inflammation.³

Three organic chemistry techniques are used in this experiment including thin layer chromatography (TLC), recrystallization, and vacuum filtration. Thin layer chromatography is used to monitor the reaction to ensure the reaction goes to completion.¹ TLC works by initially spotting a compound on the plate which will have different affinities for the mobile and stationary phases affecting the speed at which it migrates up the plate.⁴ Thus, spots will appear above the initial spot. The distance the substance traveled and the distance of the solvent front will be measured to calculate the retardation/retention value (R_f).¹ The number of spots and retardation/retention factor will be dependent upon the compound and can help to identify it.⁴ For this experiment, TLC is only used to monitor the reaction.¹ Once the initial reactant dot disappears and a new one forms that is the point at which all reactants have been converted to products.¹ Upon completion of the reaction, the next step was to recrystallize the product. Recrystallization utilizes an appropriate solvent to remove impurities from a

<https://assignbuster.com/synthesis-of-acetylsalicylic-acid/>

solution and allows for a solid organic compound to form and grow into a crystal lattice. ¹ In this experiment, water was the solvent added to completely rid the solution of acetic anhydride reactant thus, forming acetylsalicylic solid product within the solution. ¹ The vacuum filtration technique was then used to separate and dry the crystals. ¹ The crystals left in the funnel of the vacuum filter was the acetylsalicylic acid product. ¹

Once the experiment had concluded, different techniques were used to test the purity of the final product. The analytical techniques used were an infrared spectrum and melting point. ¹ Infrared spectrum was used to compare the experimental final product to the standard product. If the peaks representing different functional groups match closely then, the intended product was most likely made correctly. ¹ If it contained additional functional groups, then some of the reactants remained in the product and the reaction did not go to completion to obtain acetylsalicylic acid. ¹ The melting point was also determined to compare the product and its purity to the standard. ¹ A Mel-temp apparatus was used to obtain the melting point range. ¹ The purified product should have a higher melting point than the crude product and the temperature range should be narrow. ¹ If the melting point temperature is low and the range is broad, that could indicate that the product has impurities. ¹

2. Procedure

First, two grams of salicylic acid were placed in a 125 milliliter Erlenmeyer flask. Then, ten drops of 85% phosphoric acid and 5 milliliters of acetic

anhydride was added into the Erlenmeyer flask. The resulting solution was gently mixed. A small amount of this reaction mixture was taken for thin layer chromatography (TLC). Two TLC plates were obtained to monitor the reaction's progress. One plate was used to compare the initial reactants and the reaction after 5 minutes of heating. The second plate was used to compare the initial reactants and the reaction after 10 minutes of heating. The reaction mixture of salicylic acid, acetic anhydride, and phosphoric acid was mixed thoroughly and heated in a water bath at 90 degrees Celsius for 5 minutes. After the solution had been heated for 5 minutes, a small amount of solution was taken from the mixture to dot on the first TLC plate. Then, after 10 minutes of heating, another small amount of solution was taken to dot the second TLC plate. These TLC plates were put into a developing chamber, in this case, a jar, containing a one to one ratio of Ethyl Acetate and Hexane. The solvent front was allowed to move up the plate until it had reached about 2 centimeters from the top and then was taken out of the developing chamber. The TLC plates were examined for reaction completeness. They were examined using UV light to see where the dots had moved up the plate. The solvent front and distance traveled by the substance were measured in centimeters to calculate the retardation/retention factor. Next, the solution was cooled to room temperature. Then, 10 milliliters of water was added to the reaction mixture. While adding in the water, the reaction was continuously mixed with a stirring rod. After a couple of minutes, an additional 10 milliliters of water was added to the solution and crystals started forming inside the flask.

Once a large number of crystals were visible, a vacuum filtration technique was utilized to separate and dry the crystals from the solution. Once dry, the solid product in the funnel was weighed to determine the percent yield of the product. Next, analysis of the experimental product included obtaining an infrared spectrum and determining the melting point range and then, comparing it to the standard product.

3. Results and Data

A) Limiting Reactant

Salicylic Acid Calculation:

Formula: $(\text{Mass of reactant}) \times (\text{Molecular weight of reactant}) \times (\text{Moles of Product}) / (\text{Moles of Reactant})$

2.0 g Salicylic Acid. $\times 1 \text{ mol S.A.} / 138.121 \text{ g S.A.}$

$\times 1 \text{ mol Acetylsalicylic Acid} / 1 \text{ mol S.A.}$

= 0.0145 moles of Acetylsalicylic Acid

Acetic Anhydride Calculation:

Formula: $(\text{Volume of reactant}) \times (\text{Density of reactant}) \times (\text{Molecular weight of reactant}) \times (\text{Moles of Product}) / (\text{Moles of Reactant})$

5 ml Acetic Anhydride $\times 1.08 \text{ g} / 1 \text{ ml}$

$\times 1 \text{ mol Acetic Anhydride} / 102.09 \text{ g}$

$\times 1 \text{ mol Acetylsalicylic Acid} / 1 \text{ mol Acetic Anhydride}$

= 0.0529 moles of Acetylsalicylic Acid

Limiting Reactant : Salicylic Acid

The limiting reactant was salicylic acid because it had a smaller yield of acetylsalicylic acid product. Therefore, the reaction will stop yielding product once the 2 grams of salicylic acid reactant are gone.

B) Theoretical Yield

Formula: (Product yield from limiting reactant) x (Molar mass of product)

0.0145 moles of Acetylsalicylic Acid x 180.157 g 1 mol Acetylsalicylic Acid

= 2.609 g of Acetylsalicylic Acid

C) Percent Yield

Percent Yield = Actual Yield / Theoretical Yield

x 100

1.205 g / 2.609 g

x 100 = 46.2% yield

D) Thin Layer Chromatography Results (TLC)

R_F Calculation: 0 ≤

Retention/Retardation Factor (R_F) = Distance traveled by substance

Distance traveled by Solvent ≤ 1

TLC Plate 1: Salicylic Acid Reaction Mixture & Mixture after 5 minutes of Heating

Initial Dot Compound and Number	Dot	Distance Substance Traveled (cm)	Solvent Front (cm)	R _f Value
1) Salicylic Acid Mixture	1A	4.7 cm	7.6 cm	0.618
1) Salicylic Acid Mixture	1B	5.7 cm	7.6 cm	0.750
2) 5 Minutes After Reacting	2A	4.6 cm	7.6 cm	0.605
2) 5 Minutes After Reacting	2B	5.6 cm	7.6 cm	0.737

TLC Plate 2: Salicylic Acid Reaction Mixture & Mixture after 10 minutes of Heating

Initial Dot Compound and Number	Dot	Distance Substance Traveled (cm)	Solvent Front (cm)	R _f Value
1) Salicylic Acid Mixture	1A	4.0 cm	7.5 cm	0.533
1) Salicylic Acid Mixture	1B	5.3 cm	7.5 cm	0.707
2) 10 Minutes After Reacting	2A	4.4 cm	7.5 cm	0.587
2) 10 Minutes After	2B	5.3 cm	7.5 cm	0.707

Reacting

E) Infrared Spectroscopy (IR) Analysis

Literature Values

Wavenumber (cm ⁻¹)	Peak Description	Functional Group
2983 cm ⁻¹	Weak, Broad	O-H stretch
2872 cm ⁻¹	Weak, Broad	C-H stretch
1754 cm ⁻¹	Strong, Sharp, Narrow	C= O stretch
1135 cm ⁻¹	Weak, Sharp	C-O stretch
918 cm ⁻¹	Strong, Sharp	= C-H bending

Experimental Values

Wavenumber (cm ⁻¹)	Peak Description	Functional Group
2980 cm ⁻¹	Very Weak, Broad	O-H stretch
2870 cm ⁻¹	Very Weak, Broad	C-H stretch
1749 cm ⁻¹	Medium, Sharp,	C= O stretch

	Narrow	
1134 cm ⁻¹	Medium, Sharp	C-O stretch
914 cm ⁻¹	Strong, Sharp, Narrow	= C-H bending

The previous two tables comparing the literature IR spectrum peaks to the experimental IR spectrum peaks indicate that the peaks were very similar. In comparing the wavenumbers of the peaks, they are nearly identical. The only major differences are the intensity and the width of the peaks. However, these slight differences did not impact the identity of the functional groups. All the functional groups identified by the experimental and literature IR spectrum align with the acetylsalicylic molecule.

F) Melting Point Range

Temperature at first observable drop: 88.4 degrees Celsius

Temperature when completely liquid: 96.4 degrees Celsius

Experimental Melting Point Range	Literature Melting Point of Acetylsalicylic Acid
----------------------------------	--

88.4 - 96.4 degrees Celsius	135 degrees Celsius
-----------------------------	---------------------

The melting point of the product obtained in the experiment varies by a significant amount to the standard melting point. It is much lower than what was expected. This indicates that there may have been impurities in the product.

<https://assignbuster.com/synthesis-of-acetylsalicylic-acid/>

1. Discussion

It is important to note that according to the experimental procedure the initial reactants from the prepared Erlenmeyer flask were supposed to be taken and initially spotted on the TLC plates before heating. However, due to miscommunication, the reactants used to spot the TLC plates were taken after heating had started. By the time the mistake was realized, the reaction had already been heated for about 2 or 3 minutes, in which, the solution was then taken to dot the plates from the Erlenmeyer flask. So, the reaction could have already started resulting in faulty data.

In analyzing the results, there was an inconsistency in the TLC reading. In both the 5-minute test plate and the 10-minute test plate the reactant had two dots and the product had two dots in the same position as seen by the retardation/retention factor values. This indicates that the initial reactants were the same as the product in both 5 minutes and 10 minutes after reacting. This result could have been due to errors when carrying out the procedure in the lab as mentioned before. However, this error indicates that this reaction underwent rapidly to form the product in only a couple of minutes.

The melting point of the experimental acetylsalicylic acid was very different from the standard acetylsalicylic acid. The experimental melting point was much lower than the standard and also had a broad temperature range. This could be due to impurities in the product or human error when monitoring and recording the melting point range. The infrared spectrum reading of the experimental product was similar to the standard infrared spectrum for

acetylsalicylic acid. There were differences in peak intensity and width, but overall, the functional groups remained the same.

Due to inconsistencies in the data and errors occurring while conducting the experiment, the final product was not successful. Even though the experimental IR spectrum was similar to the literature IR spectrum, the melting point range differed by over 30 degrees. It is most likely that impurities remained in the product during the recrystallization process resulting in the unsatisfactory data. These impurities could also explain the differences in peak intensity and width in the infrared spectrum. In conclusion, the acetylsalicylic acid product was made in the experiment but, it was not pure.

References

1. Weldegirma, S.; *Experimental Organic Chemistry: Laboratory manual*, 1st Edition;

Department of Chemistry at University of Central Florida: Florida, 2018

2. [Http://journal.ru/wp-content/uploads/2017/03/a-2017-023.pdf](http://journal.ru/wp-content/uploads/2017/03/a-2017-023.pdf). (2017). *Aspirin*.

doi: 10.18411/a-2017-023

3. Uniprix. (n. d.). Aspirin (acetylsalicylic acid (ASA)) – Information about this drug. Retrieved

from <https://www.uniprix.com/en/drug-lexicon/1325/aspirin>

4. Libretexts. (2018, August 06). Thin Layer Chromatography. Retrieved from

<https://assignbuster.com/synthesis-of-acetylsalicylic-acid/>

https://chem.libretexts.org/Demos,_Techniques,_and_Experiments/General_Lab_Techniques/Thin_Layer_Chromatography