Alan Yu Organic Lab 309:03 Santanu Malakar

Experiment 4: Synthesis of Salicylic Acid from Wintergreen Oil February 11, 2019

Purpose:
The purpose of the experiment is to synthesize salicylic acid from methyl salicylate.
Equations
Equations:
Mechanisms:
Wednamonio.
Amounts and Properties:

Chemical	Amount	Mol Weight	MP	BP	Density
Methyl Salicylate	10 mmol	152.1	-8	223	1.174
Salicylic Acid		138.1	159		
6M NaOH	15 mL				
3M Sulfuric Acid	16 mL				

Hazards and Safety:

Table 1: Important Chemical Data

Concentrated NaOH solutions are very corrosive and can cause severe damage to skin and eyes. Wear gloves and goggles at all times. Methyl salicylate can irritate eyes and skins; avoid contact and inhalation. To dispose: Dispose the waste and products in marked containers unless told otherwise.

Procedure:

Reaction:

- 1. Obtain heat source and position correctly. Assemble the system for heating under reflux. Check is system is secured to ring stand.
- 2. Acquire approximately 10 mmol of methyl salicylate into a boiling flask and 15 mL of 6M NaOH with stirring rod. Add both reagents through different funnels or else a solid will form in the funnel.
- 3. Make sure water is flowing through the condenser jacket and have system checked before heating.
- 4. Heat the reaction mixture under reflux for 30 minutes starting from the time the reaction mixture starts to boil.
- 5. If after 30 minutes there is still an oily layer or the mixture is cloudy, continue heating under reflux the oily layer or cloudiness fades away.
- 6. When complete, remove condenser as soon as it is cool enough to handle.
- 7. Remove stirring rod and transfer reaction mixture to a beaker large enough to contain it and the sulfuric acid to be added.
- 8. Slowly add 16 mL of 3M sulfuric acid while stirring and test the pH of the top layer.
- 9. If the pH is above 2, make it so the pH is under 2. Cool the mixture in an ice bath for about 10 minutes.

Separation:

1. Collect the salicylic acid by vacuum filtration and wash on filter with small amounts of cold water.

Purification and Analysis:

- 1. Purify acid by recrystallization from boiling water. No need to filter the hot solution.
- 2. Collect product by vacuum filtration and wash it with a little amount of cold water.
- 3. Dry to constant mass and weigh.
- 4. Measure melting points of dry product and 1:1 mixture of product.

Observations:

While heating under reflux, the liquid produced was a yellowish liquid and seemed like there were two layers formed. After transferring the liquid into a new beaker for the sulfuric acid to be added, adding the sulfuric acid created a cloudy white solid to form.

Measurements:

Table 2: Measurements during experiment

Amount used of Methyl Salicylate	1.428g
Theoretical Amount of Methyl Salicylate volume wise:	21.313 mL
Mass of Dried Solid:	7.184g
Recovered Amount:	1.558g
Start Temp to melt:	145 degrees C
End Temp to melt:	150 degrees C

Data and Calculations:

$$10 \frac{\frac{mmol*1mol}{1000 \, mmol}*152.1 \, g}{1 \, mol} = 1.521 \, g \, needed$$

System before: 91.664g, System after: 93.092g

$$93.092g - 91.664g = 1.428g$$
 used

$$1.428 \frac{g*100 \, mL}{6.7 \, g} = 21.313 \, mL$$

The yield was 1.558g.

% yield:
$$1.558g / 1.297g \times 100 = 120\%$$
 yield

% yield: 1.558g / 1.29/g x 100 = 120% yield

Theoretical yield: 1.428g
$$\frac{\frac{1 \, mol}{152.1 \, g} *1 \, mol}{1 \, mol} *138.1 \, g}{1 \, mol} = 1.297 \, g$$

Discussion:

For the yield of the salicylic acid, it turned out to be a 120% yield. This was most likely due to the drying process and didn't dry the salicylic acid completely. The excess weight came from the water that was still lurking within the crystalized salicylic acid. For the melting point, this error was seen because salicylic acid's melting point is 159, the range that the result melted it was from 145 - 150 proving that there was some sort of liquid, most likely water remaining in the sample.

Conclusions:

Since the sample was not dried, there can be no determination of any significant material losses unless under speculation. To state, transferring the material from beaker to beaker and when it was drying after filtration, there will be some loss, but maybe around .05g which is pretty significant because of how much the crystals like to stick to the sides of the containers.

Exercises:

1. A) 1.428g used \rightarrow 2 mol of NaOH per 1 mol of Methyl Salicylate.

$$\frac{1.428 \, g}{152.1 \, g} = .009389 \, mol \, Methyl \, Salicylate$$

$$\frac{6 \, mol}{1 \, L} \, NaOH \, x \, \frac{1 \, mol \, MS}{2 \, mol \, NaOH} \, x \, \frac{1}{.009389 \, mol \, MS} = 319.5 \, \frac{1}{L}$$

$$\frac{1}{319.5} \, x \, 1000 = 3.130 \, mL$$

- B) 15 mL 3.130 mL = 11.870 mL
- C) The amount needed to react and remove excess NaOH and disodium salicylate would be the same as the amount of excess NaOH plus how much sodium Salicylate was produced. So it would be 15 mL due to 3M sulfuric acid having 2 mol of Hydrogens per compound. D) Since there was 16 mL of sulfuric acid added and the required amount was 15 mL, the excess was 1 mL.
- 2. A) The hydrogen atoms on the phenol group and the carboxylic acid of salicylic acid are the acidic hydrogens. From the methyl salicylate, the phenol one is more acidic than the other group formed.
 - B) Methyl Salicylate + 2 NaOH → Sodium Salicylate + Water + Methanol

- 4. Solubility is .14g / 100mL, water used was 21.50 mL. From that $.14g / 100mL \times 21.50 mL = .0301 g lost$.
- 5. A) Since the pH is only 4, the reaction would not be able to drive towards completion. This yielded less than what the experiment should've yielded. B) Since there was an oily layer, that means the reflux wasn't towards completion and that the wintergreen oil was still there. This leads to a smaller yield at the end. C) Since aspirin has a melting point of 135 degrees C and salicylic acid has a melting point of 159, the result at the end would be that the salicylic acid that was supposed to be made would be concluded that it wasn't made because of a plateau for the melting point range.
- 6. During the heating phase, the methanol is separated due to the nature of it being a low boiling temperature liquid. Filtration can easily separate the sodium sulfate from the salicylic acid.