Name: Anna Li Report Submission Date: 4/25/20

Experiment Title: **Isolation of Trimyristin from Nutmeg**

Section Day and Time Friday PM: If Make-Up, lab performed on date:

Teaching Assistant: Sarah Amorin

Lab Technique: Leave this section blank in your report. But, make sure to tell Gradescope it is on page one of your lab report. Your TA will assign the technique points when they grade the post-lab. Do not delete this text.

Purpose: The purpose of the experiment is to extract trimyristin from nutmeg with tert-butyl methyl ether and purify it through recrystallization, as well as to produce myristic acid through hydrolysis of the trimyristin with 6 M sodium hydroxide and 95% ethanol followed by addition of concentrated hydrochloric acid and water. Both the trimyristin and myristic acid will be identified by their melting points.

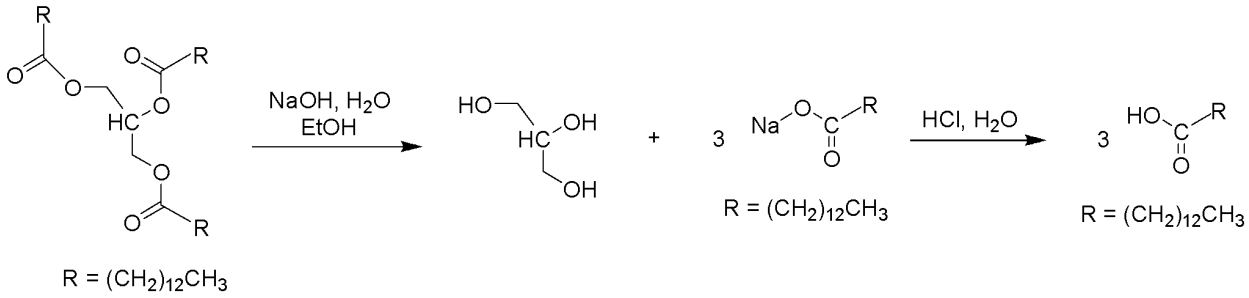
Pre-Lab: Leave this section blank in your post-lab report. But, make sure to tell Gradescope it is on the appropriate page. *Your TA should have graded the prelab before you began the experiment*. They will assign the awarded points for the prelab with the report. Do not delete this text.

In-Lab Observations and Recordings:

- Added 3 mL TBME to 1.013 g ground nutmeg
- Crude trimyristin
    - During filtration, precipitate formed at tip of pipet; rinsed tip with drops of TBME to dissolve blockage
    - As solvent evaporated, flask cooled down and white deposit formed, eventually becoming powder
    - Erlenmeyer flask: 14.461 g
    - Crude weight: 15.025 g – 14.461 g = 0.564 g
    - % recovery: 0.564 g/1.013 g x 100 = 55.68%
- Purified trimyristin
    - Recrystallized in ~2 mL acetone
    - Pale yellow solid
    - Recrystallized weight: 0.990 g – 0.440 g (filter paper) = 0.550 g
    - % recovery: 0.550 g/1.013 g x 100 = 54.29%
    - MP: 56-57 °C
- Did not recrystallize a second time (MP already matches literature value)
- Hydrolysis and acidification
    - Used 0.101 g trimyristin
    - Cloudy precipitate formed upon addition of HCl
    - Filtration: rinsed w/ ice-cold water 4 x 1 mL
    - Myristic acid
        - Off-tan solid

- recovered myristic acid was 0.0907 g
- % yield: 0.0907 g/0.09570 g x 100 = 94.78%
    - 0.101 g/723.16 g = 0.0001397 mol trimyristin = 0.0004190 mol myristic acid
    - Theoretical yield: 0.0004190 mol x 228.4 g/mol = 0.09570 g
- MP: 52-54 °C

Reaction Scheme:



Experimental Procedure:

Ground nutmeg (1.013 g) was put into a round-bottomed flask. Tert-butyl methyl ether (3 mL) was added and the mixture was refluxed for 10 minutes. The flask was then allowed to cool for several minutes, after which the liquid portion was siphoned off, filtered through a micro-scale filtration apparatus, and collected in a 25 mL Erlenmeyer flask. A pipet bub was attached to the filtration apparatus and squeezed to apply pressure to the system in order to complete the filtration. The round-bottomed flask was then rinsed twice with fresh tert-butyl methyl ether (2 mL), each time using 1 mL and heating the flask for about 2-3 minutes while swirling, and filtering as before into the Erlenmeyer flask. The solids remaining in the round-bottomed flask were removed to waste and the Erlenmeyer flask was passed under an air stream to evaporate the solvent. It was then air dried for 5 minutes and massed. Next, the crude trimyristin solid was recrystallized with acetone (2 mL). The crystals were collected by vacuum filtration on a Hirsch funnel and rinsed with ice-cold acetone (1 mL). They were dried under a stream of air for a few minutes. Melting point and mass were taken. Then, some trimyristin (0.101 g, 0.0001397 mol) was transferred to a clean round-bottomed flask and 6 M sodium hydroxide (2 mL) and 95% ethanol (2 mL) was added. The solution was refluxed for 45 minutes. Afterwards, it was cooled for 20 minutes to room temperature and poured into a 50 mL beaker with distilled water (8 mL). Concentrated hydrochloric acid (2 mL) was added dropwise, swirling the flask every few drops. Once the acid was added, the beaker was cooled in an ice bath for 10 minutes. Finally, the precipitate that formed was collected via vacuum filtration on a Hirsch funnel and was rinsed with ice-cold water (4 mL) 4 times, using 1 mL at a time. Finally, the solid myristic acid was dried, weighed, and had its melting point taken.

Results

Reaction Product	Melting Point (°C)	Starting mass (g)	Percent Yield	Percent Recovery	Final Mass (g)	Crude Percent Recovery	Crude Mass (g)	Charac-teriza-tion Method
Trimyr-istin	56-57	1.013 (nutmeg)	N/A	54.29	0.550	55.68	0.564	MP
Myris-tic Acid	52-54	0.101 (trimyristin)	94.78	N/A	0.0907	N/A	N/A	MP

Discussion:

The purpose of this experiment was to extract trimyristin from ground nutmeg using tert-butyl methyl ether and to purify it through recrystallization. Furthermore, myristic acid was produced from the trimyristin through hydrolysis with 6 M sodium hydroxide and 95% ethanol followed by concentrated hydrochloric acid and water.

Crude trimyristin had a mass of 0.564 g, or a percent recovery of 55.68% based on the original mass of the ground nutmeg which was 1.013 g. Since nutmeg is made of many compounds aside from trimyristin, this seemingly low recovery is still reasonable. This percent recovery is even likely higher than it should be, since the crude trimyristin was not completely dry when it was massed. The mass of the recrystallized trimyristin, was 0.550 g, which resulted in a percent recovery of 54.29%. It is lower than that of the crude material, probably because the recrystallization process removed other ether-soluble compounds in nutmeg from the trimyristin solution. Meanwhile, the mass of recovered myristic acid was 0.0907 which amounted to a percent yield of 94.78% based on the 0.101 g of trimyristin starting material. It appears that the reaction proceeded very successfully towards the myristic acid product and minimal material was lost in the few transfers of material.

The experimental melting points of both trimyristin and myristic acid were nearly identical to literature values at 56-57 °C and 52-54 °C, respectively. This suggests that both the extraction of trimyristin and production of myristic acid proceeded as expected. Since the measured melting points were both over narrow ranges and very accurate to literature values, the final products were probably relatively pure.

Both the literature and experimental results show that the melting points of trimyristin and myristic acid are close. This is probably because trimyristin essentially consists of three molecules of myristic acid. Its carboxylic acid portion is so much larger than the glycerol part that it is most likely the biggest contributor to physical properties like melting point. Thus, even without the glycerol, the myristic acid melts at a similar temperature to trimyristin.

Postlab Questions:

1. A saturated fat has no alkene groups in its carboxylic acid portion while monounsaturated fats have one alkene group. Fats with more than one alkene group are polyunsaturated. There are no alkene groups in the fatty acid chains of trimyristin, so it is a saturated fat.
2. In the older procedure, other ether-soluble compounds in nutmeg in the acetone may be caught in the trimyristin crystals, because unlike in a normal recrystallization, placing the warm solution directly into an ice bath makes crystals form too quickly to exclude impurities.
3. 1.8 g/891.48 g = 0.0020 mol tristearin
   0.0020 mol x 3 = 0.0060 mol stearic acid
   0.0060 mol x 284.48 g = 1.7 g stearic acid
4. The purpose of heating a reaction mixture is to speed up the reaction by providing energy and increasing the mixing of reagents.
5. If the mixture were heated at 40°, most of the trimyristin would not separate so hydrolysis would only yield small amounts of myristic acid.