Isolation of Trimyristin from Nutmeg and Preparation of Myristic Acid from Trimyristin by Hydrolysis

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Introduction

Natural product chemistry encompasses the field of isolating organic compounds from living organisms, which are typically plants (Weldegirma, 2018). In general, isolating organic compounds from these living organisms is a difficult task because there are usually numerous compounds in a living organism that makes isolating a particular compound difficult. However, in the case of nutmeg, this isolation is eased when extracting the compound trimyristin because it is found in substantial quantities, often exceeding 25% of the weight of nutmeg but usually below 40% (Weldegirma, 2018). The compound Trimyristin is a triester containing a glycerol backbone with chains of carboxylic acids from the myristic acid. In the case of Trimyristin, the composition is atypical because the three long chains of carboxylic acids are identical. In order to isolate the compound Trimyristin in nutmeg, it is necessary to conduct a procedure referred to as extraction. This extraction procedure consists of removing the solid aspect of nutmeg to a liquid specie, which will be the solvent in the extraction procedure. Then, after the isolation of Trimyristin is completed, it will be crucial to recrystallize to ensure the purity of the compound. This is done by dissolving the adulterated product in a heated solvent which is then allowed to cool and crystallize. Finally, in order to test the purity of the product, the melting point apparatus is employed. Subsequently, it is also crucial to mention the hydrolysis of esters. This hydrolysis of esters, usually referred to as acid and base catalyzed hydrolysis, yields the anion of a carboxylic acid and an alcohol. This is typically caused by the addition of a strong base to a fatty bond, which leads to the production of fatty acid crystals because the triglyceride ester bonds are hydrolyzed (Weldegirma, 2018). In the case of trimyristin, the hydrolysis that takes place is alkaline hydrolysis, which turns into the sodium salt of the myristic acid (Weldegirma, 2018). Moreover, testing for the purity of the compound via the melting point provides information of whether the compound is mixed, and thus impure. To illustrate, the mixed melting point concept states that a pure sample will melt at a higher and broader temperature than an impure sample, which usually melts at a temperature that is lower and broader than the literature melting point value. Due to this, even an equal mixture of two compounds that have the same melting point will result in a lower melting point overall because it is an adulterated mixture.

Subsequently, the reaction that takes place to convert trimyristin to myristic acid is illustrated below (Figure 3), this procedure illustrates the hydrolysis process that takes place when hydroxides are added to trimyristin allowing for the procedure to continue. Then, hydrochloric acid is utilized to yield the desired product, myristic acid, and sodium chloride. However, there is also a potential side reaction that could take place as illustrated below (Figure 4). This side reaction is referred to as the elimination reaction, and could be avoided by keeping the reaction at a low temperature and not overheating the product during the distillation procedure.

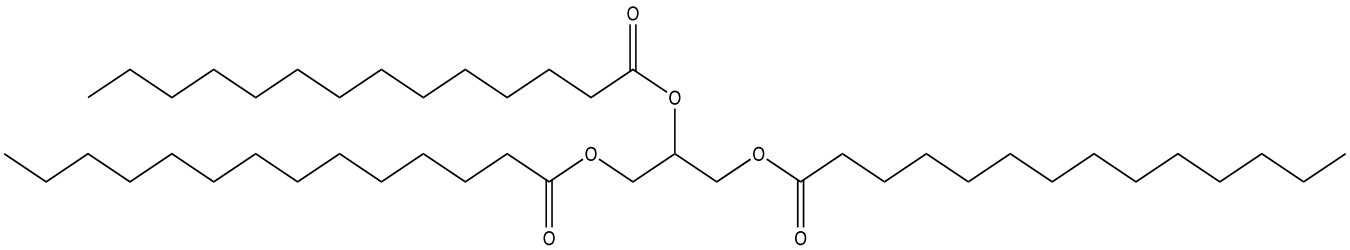


Figure 1. Structure of trimyristin, $C_{45}H_{86}O_6$

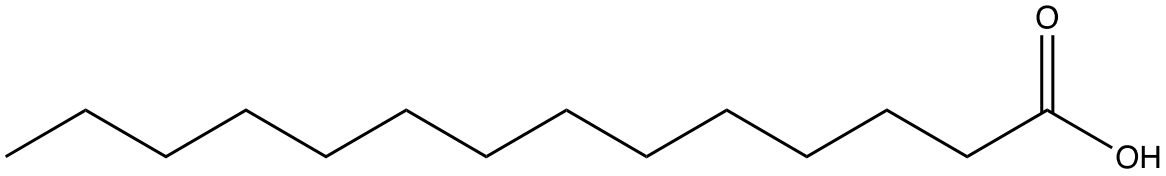


Figure 2. Structure of myristic acid, $C_{14}H_{28}O_2$

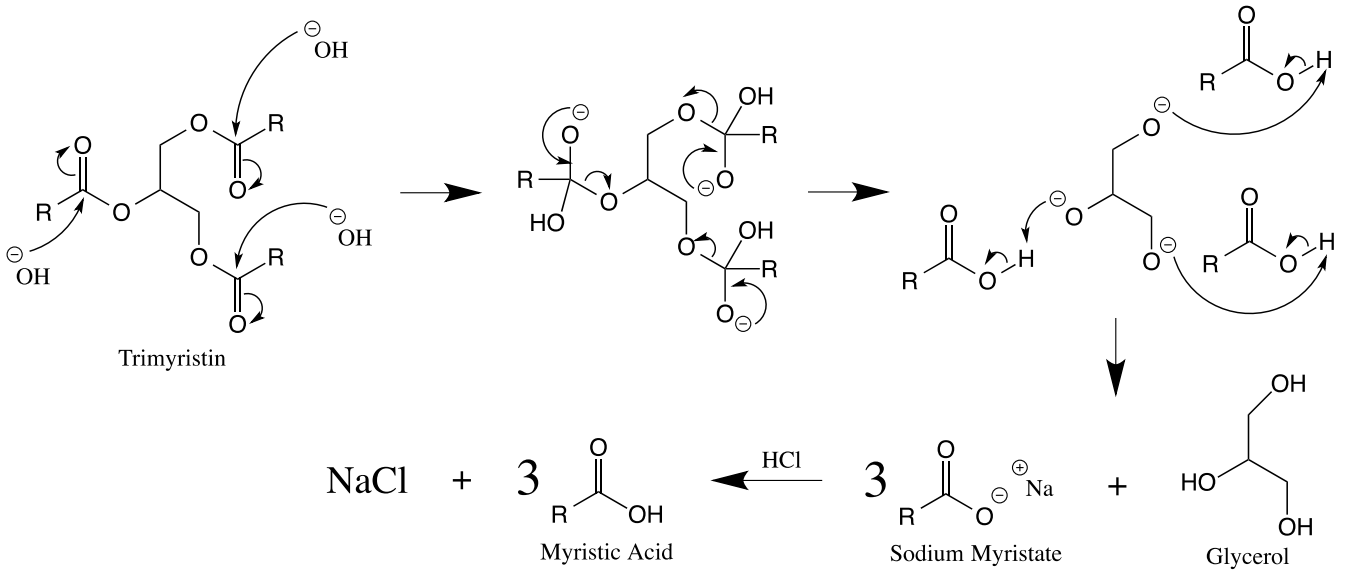


Figure 3. Detailed mechanism for the preparation of myristic acid from trimyristin

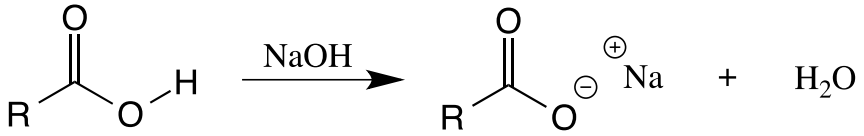
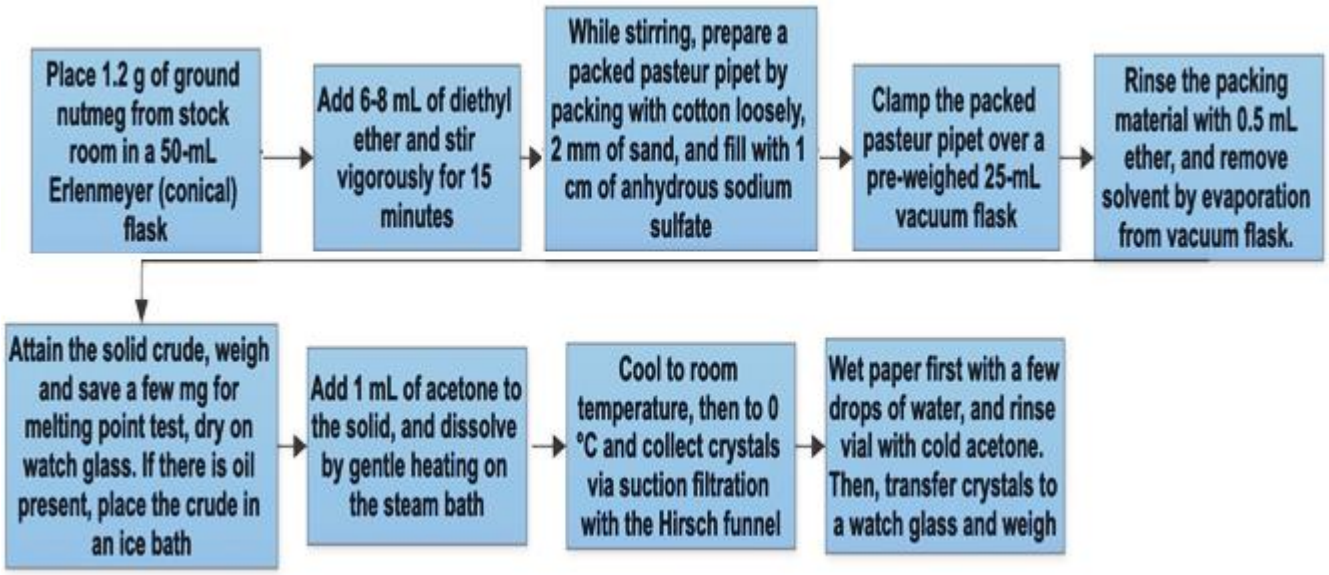


Figure 4. Potential undesired (side) reaction that can take place during the hydrolysis of trimyristin

Experimental Section

Part 1 – Isolation of Trimyristin



Part 2 – Preparation of Myristic Acid (Hydrolysis)

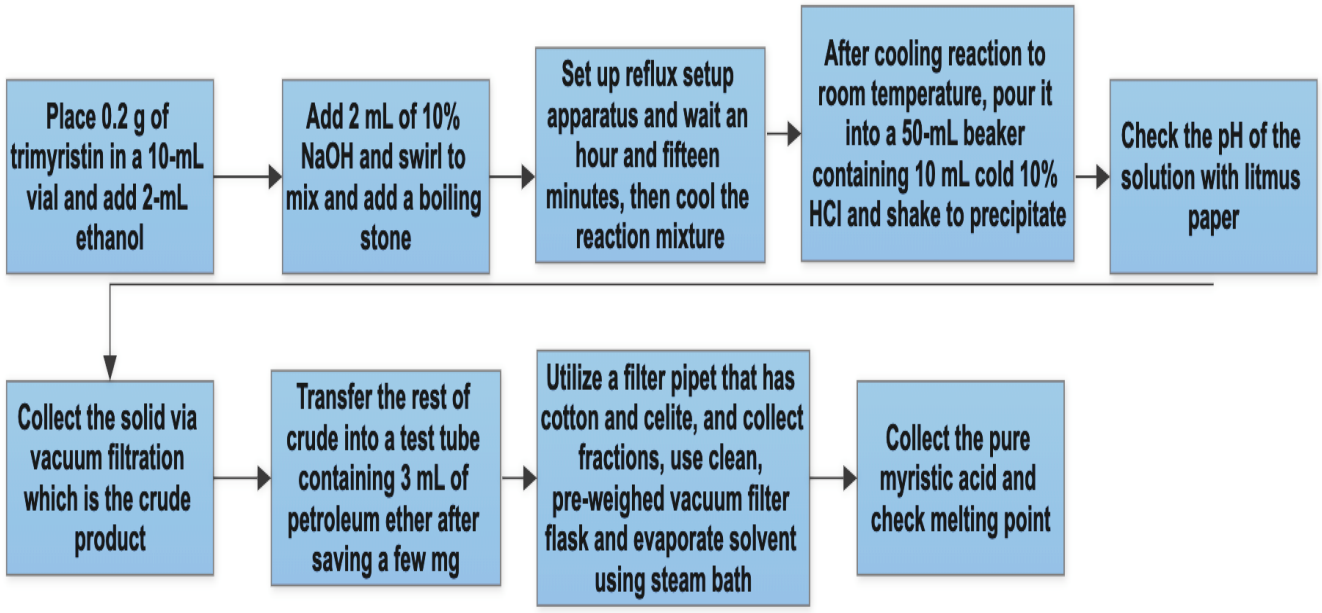


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Table 1. Chemicals extracted from nutmeg and yielded from hydrolysis

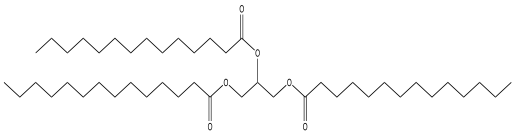
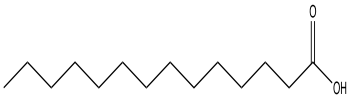
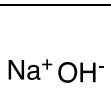
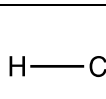
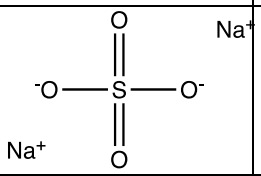
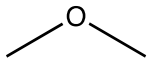
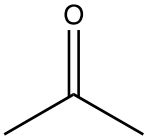
Chemical	Trimyristin	Myristic acid
Formula	$C_{45}H_{86}O_6$	$C_{14}H_{28}O_2$
Structure		
Molar Mass	723.16 g/mol	228.3709 g/mol
Melting Point	56-58 °C	54.4 °C
Boiling Point	311 °C	250.5 °C
Color	White to yellowish-gray solid	White crystalline solid
Toxicity	Avoid release into environment, causes target toxicity	Irritates mucous membranes
Hazards	Avoid breathing dust	May cause eye and skin irritation
Systematic IUPAC name	1,2,3-propanetriyl tri(tetradecanoate)	1-tetradecanoic acid

Table 2. Chemicals utilized during the extraction and hydrolysis process

Chemical	Sodium hydroxide	Hydrochloric acid	Anhydrous sodium sulfate	Ether (Dimethyl ether)	Acetone
Formula	NaOH	HCl	Na_2SO_4	C_2H_6O	C_3H_6O
Structure					
Molar Mass	39.997 g/mol	36.46 g/mol	142.04 g/mol	46.07 g/mol	58.08 g/mol
Melting Point	318 °C	-114.2 °C	884 °C	-141 °C	-95 °C
Boiling Point	1,388 °C	-85.05 °C	1,429 °C	-24 °C	56 °C
Color	White crystalline solid	Colorless	Colorless	Colorless	Colorless
Toxicity	Acute toxicity if swallowed	Specific target organ toxicity	Acute oral toxicity	Acute oral toxicity	May cause respiratory irritation
Hazards	Causes severe skin burns	Causes severe skin burns and eye damage	Combustible dust	Causes skin burns and eye irritation	Causes skin burns and eye irritation
Systematic IUPAC name	Sodium hydroxide	Hydrochloric acid	Sodium sulfate	Dimethyl ether	Propan-2-one

Results

Table 3. Mass, percentage yield, and melting point of trimyristin and myristic acid

Chemical	Mass (g)	Percentage Yield (%)	Experimental Melting Point (°C)	Theoretical Melting Point (°C)
Pure Trimyristin	0.16	13.33	54-56	56-58
Pure Myristic Acid	0.65	57.26	46-48	54-55

Calculations:

Percentage Yield = $\frac{\text{Actual Yield}}{\text{Theoretical Yield}}$ x 100

Percentage yield of trimyristin = $\frac{0.16\text{ g}}{1.2\text{ g}}$ x 100 $= 13.33\%$

Percentage yield of myristic acid = $\frac{\text{actual moles}}{\text{theoretical moles}} = \frac{0.00285}{0.004977}$ x 100 = 57.26%

Number of moles of trimyristin = $\frac{\text{Weight of trimyristin utilized}}{\text{Molar mass of trymiristin}} = \frac{1.2\text{ g}}{723.16\text{ g/mol}}$ = 0.001659 moles

Number of moles of myristic acid = $\frac{\text{Weight of myristic acid}}{\text{Molar mass of myristic acid}} = \frac{0.65\text{ g}}{228\text{ g/mol}} = 0.002850$ moles

1 mole of trimyristin = 3 moles of myristic acid x moles of trimyristin = 3x moles of myristic acid

Experimental number of moles of myristic acid = 0.002850 moles

Theoretical number of moles of myristic acid = 3(0.001659 moles) = 0.004977 moles

Limiting Reactant Calculations

$$10\text{ NaOH} \times 2\text{ mL NaOH} \times \frac{1\ mol\ NaOH}{39.997\ g\ NaOH} = 0.005\text{ mol NaOH}$$

$$2\text{ mL ethanol} \times \frac{0.991\ g\ ethanol}{1\ mL\ ethanol} \times \frac{1\ mol\ ethanol}{46.07\ g\ ethanol} = 0.04302\ moles$$

$$\frac{0.005\ mol\ NaOH}{0.001659\ mol\ trimyristin} = \frac{30.14\ mol\ NaOH}{1\ mol\ trimyristin} > \frac{3\ mol\ NaOH}{1\ mol\ trimyristin}$$

Trimyristin was found to be the **limiting reactant.**

Melting point of the mixture of equal amounts of trimyristin and myristic acid: 45-47 °C

Discussion

After completing the experiment, it was crucial to compare the theoretical melting points of trimyristin and myristic acid with their literature values. In the case of the pure trimyristin, which was purified via recrystallization, the experimental melting point was found to be 54-56 °C which is lower than the theoretical value of 56-58 °C. Thus, in this case, the experimental melting point was slightly lower. Moreover, for myristic acid, the experimental melting point was found to be 46-48 °C which was substantially lower from the theoretical value of 54-55 °C. Moreover, the experimental range was also larger. In general, this is due to the mixed melting point concept which states that impure compounds will melt at a lower temperature than the pure compound. This melting point depression was evident in both experimental melting point values. However, the difference in melting point was a lot larger for myristic acid. This could have been due to improper drying of the product, or improper yield of the product, causing it to have some impurities. Moreover, not working in a sterile environment could have also contaminated the sample. Finally, the melting point apparatus was difficult to read, so it could have been possible that the reading was inaccurate. Nonetheless, it seems that it is most probable that the compound was impure. On the other hand, the similar melting point range and temperature values obtained experimentally for trimyristin illustrate that the extracted product was relatively pure.
Subsequently, the percentage yield of trimyristin and myristic acid were calculated to determine how much of the trimyristin was successfully extracted, and how much myristic acid resulted from hydrolysis. In this case, the percentage yield of trimyristin was 13.33%, which is relatively lower than the amount usually present in nutmeg. However, this could have been due to loss of product during the extraction procedure. Then, the percentage yield of myristic acid was calculated by multiplying the moles of trimyristin acid by three to obtain the theoretical amount of moles that the hydrolysis would yield. Then, the actual amount of moles of myristic acid were divided by the theoretical amount. In this case, the percentage yield of myristic acid was 57.26% which was higher than 50%. Nonetheless, this yield could contain impurities causing the melting point to be relatively lower and the percentage yield to be higher. However, it is also possible that some side reactions took place causing the yield to be lower. If the temperature was kept lower and the product was purified successfully, it is predicted that the yield would have been greater.

The mixed melting point concept explains that pure compounds will have melting point ranges that are similar to the literature values with very narrow ranges. On the other hand, impure compounds will have dissimilar melting points when compared to the literature values along with broader ranges. The mixed melting point concept is utile during the preparation of myristic acid. For example, trimyristin and myristic acid almost have identical melting points. Thus, in order to determine if hydrolysis actually yielded myristic acid from trimyristin, the mixed melting point concept has to be used. Thus, taking the melting point of a 50:50 mixture of the supposed trimyristin and myristic acids will result in a lower melting point than when taking both melting points separately, which would be nearly identical. Since trimyristin and myristic acid are different compounds, the mixture would behave as if it was an impure sample (Ibanez et al., 2003).

Conclusion

In general, the theoretical background and the results obtained were connected. Although the percentage yield of trimyristin obtained was lower than expected, it could have been explained by the loss of product during the procedure since the nutmeg needed to be transferred into glassware, leading to the loss of product. Since the sample size is small, the percentage yield would have largely decreased if even a slight amount of product was lost. However, the pure amount of trimyristin obtained seemed to be relatively pure since the melting point range was narrow and nearly identical to the theoretical melting point of the compound. On the other hand, myristic acid was able to be obtained, but did not seem to be as pure. However, this does not mean that the theoretical background is not connected to the results. In fact, this explains why the melting point of myristic acid was lower than expected. Essentially, it is due to the mixed melting point concept, which explains that impure samples melt at a lower temperature than their pure counterparts. The experimental data reveal that trimyristin can be extracted from nutmeg and purified via recrystallization. Even more, trimyristin can be hydrolyzed to yield myristic acid. Consequently, the results also provide insight into the mixed melting point concept because an impure sample of myristic acid seemed to have a lower melting point, as expected. The techniques applied in this experiment can be applied to a broad range of other situations. For example, extraction techniques are popular in medicine. In general, extraction can be utilized to obtain antioxidant products from Rosemary plants and purified in a similar way. These antioxidants can then be consumed as supplements and medicines (McCullough and Guy, 1957). Antioxidants are important because they enhance the ability of the body to combat free radicals. Nonetheless, the experiment seems to have accomplished what it set out to do. The extraction of trimyristin and the hydrolysis of the product to yield myristic acid were carried out. Then, the compounds were tested for their melting points to determine their purity after recrystallization. Finally, the mixed melting point concept was employed to ensure that the compounds obtained were different.

References

Ibanez, E.; Kubátová, A.; Señoráns, F.; Cavero, S.; Reglero, G.; Hawthorne, S. Subcritical Water Extraction of Antioxidant Compounds from Rosemary Plants." Journal of agricultural and food chemistry, 2003, 51(2), 375-382.

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