Experiment 6: Natural Product Isolation Jeremy Wolf
08/04/2008 TA: Stefanie Lenz

Introduction:

The purpose of this experiment was extract, isolate, and purify the natural product, trimyristin, from the spice, nutmeg. The trimyristin was extracted by using the solvent, diethyl ether and heat to draw the organic oil out of the ground nutmeg. Once the oil was extracted, the trimyristin was crystallized from the oil by using acetone. Finally, the crystals of trimyristin were analyzed for purity by measuring their melting point.

Experimental Procedure:

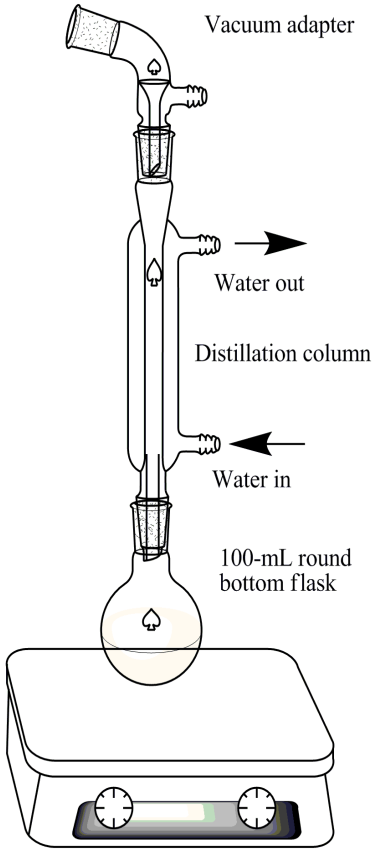
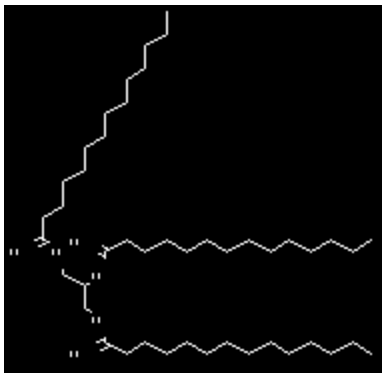
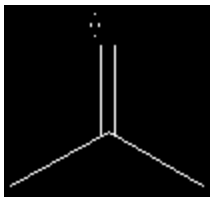


Figure 1: Experimental Set Up

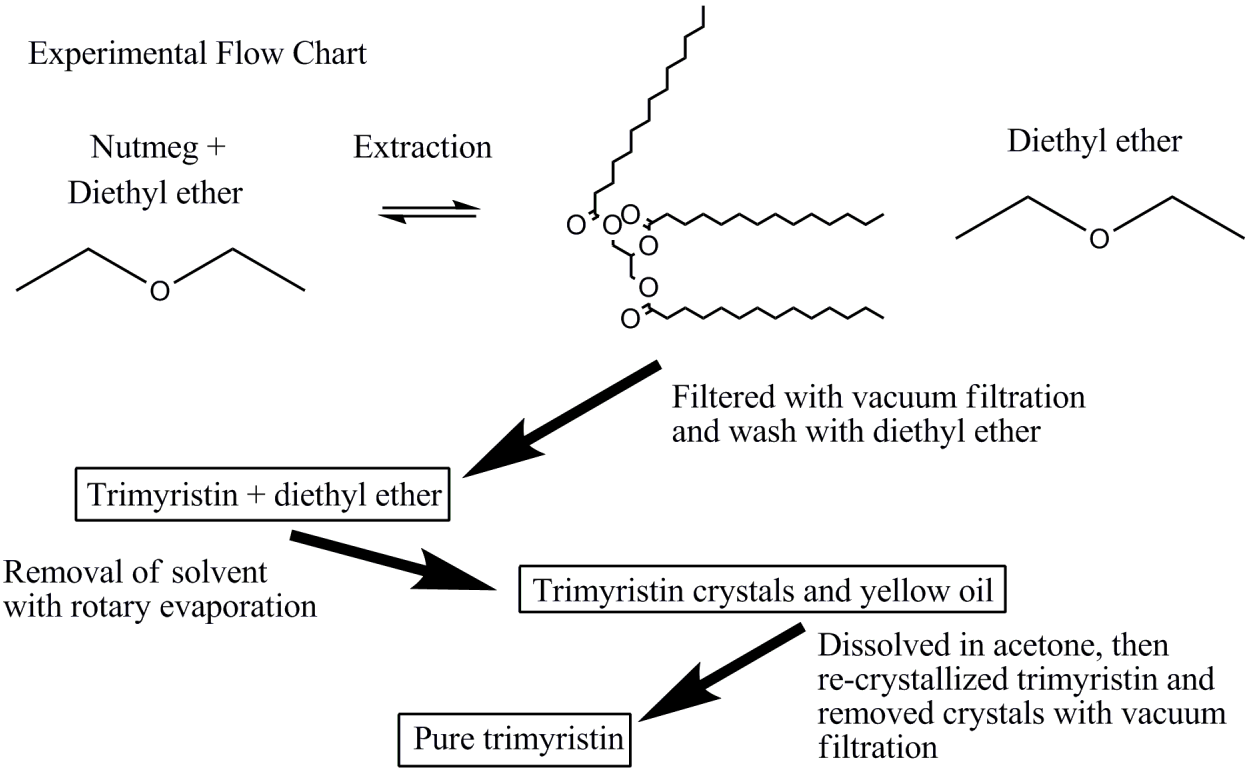
A mixture of 4 grams of ground nutmeg and 20 mL of diethyl ether was combined in a 100-mL round bottom flask and heated with magnetic stirring to reflux with the set up described in Figure 1. The mixture was heated at reflux for 30 minutes. After heating, the mixture was filtered with gravity

filtration and the diethyl ether solvent was removed by rotary evaporation. The resulting oil was dissolved in 4 mL of acetone in a 25-mL Erlenmeyer flask and the flask was heated to completely dissolve the crystals. The flask was then allowed to stand for 30 minutes, then it was placed in an ice bath to allow complete crystallization. The trimyristin crystals were then filtered from the acetone with vacuum filtration, allowed to dry, then melting point and yield data was taken.

Table of Reagents

Compound	Molar Weight	Physical Properties
Trimyristin (found in Nutmeg) $C_{45}H_{86}O_6$ 	723.16 g/mol	MP = 56 – 58^{0}C
Diethyl ether $C_4H_{10}O$ 	74.1224 g/mol	BP = 34.6^{0}C Density = 0.7134 g/mL Flammable Liquid
Acetone C_3H_6O 	58.08 g/mol	

Experimental Flow Chart:



Observations and Results:

- The observed oil was yellow in color
- The melting point of the trimyristin crystals was 49^0C

Total yield = 0.21 grams/4 grams x 100 = 5.25% yield

Conclusion:

The purpose of this laboratory activity was achieved. Diethyl ether was used to dissolve the oil out of the nutmeg powder. The trimyristin was extracted due to the principle that like dissolves like. Trimyristin is mostly a nonpolar molecule, with one end, the triglyceride, having a polar component. Diethyl ether is also a nonpolar molecule overall, but the oxygen – carbon bonds are themselves, polar. This similarity is what causes the trimyristin to have an affinity for the solvent and allows the trimyristin to be extracted from the nutmeg.

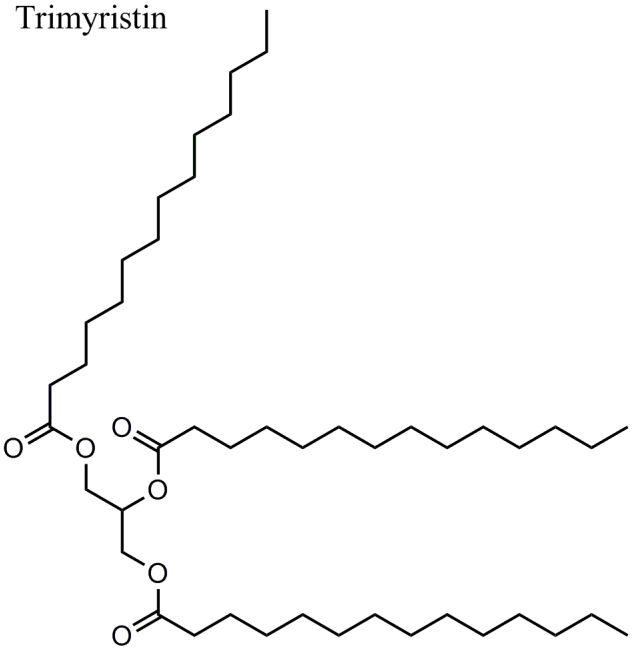
Relatively speaking a small amount of the trimyristin was recovered, only 5.25% of the mass of the nutmeg. The process of recovery may have led to this small amount of trimyristin extracted. There were numerous opportunities to lose product in the process. During the reflux heating, if the reaction was not heated for long enough or if the solvent evaporated during the reaction, trimyristin could have been lost. The oil that was extracted was impure, by nature. During the rotary evaporation, the solvent could have evaporated with some of the desired product. When the acetone was added

to the oil, some of the trimyristin could have remained dissolved in the acetone solution.
The oil that was extracted from the nutmeg was impure because the diethyl ether would pull out more than just the desired product. The organic solvent will pull many nonpolar molecules out of the nutmeg, but the trimyristin was the component that should have re-crystallize with the addition of the acetone. The acetone is a polar solvent, so it dissolved the polar impurities in the oil and the nonpolar component, trimyristin, crystallized out of the solvent.

Answers to Selected Exercises (pp. 149-150, #1 & 5)

#1 – Diethyl ether was chosen as the solvent because it is less polar than acetone. Overall, diethyl ether has no dipole moment because the dipoles of the O-C bonds are equal and opposite, so they cancel each other out. However, the bonds between the oxygen and carbon atoms are polar, giving the molecule some polar character. The trimyristin molecule also has polar components, but overall, there is much more of the molecule that is nonpolar. Acetone, conversely, is a polar molecule, with an overall dipole moment. Since like dissolves like, the diethyl ether will dissolve the trimyristin, while the acetone will not. In the reaction vessel, the diethyl ether serves as a solvent, removing the trimyristin from the nutmeg in the solution.
#5 – Each organic component would be extracted in an oil. An oil by itself is very difficult to crystallize, but a multiple component oil would be even more difficult to crystallize because the oil is an impure mixture. Impure mixtures blend the properties of the components, like melting point and crystallization temperature. A multiple-component mixture is very difficult to isolate any single component. Since the two triglycerides are very similar in their properties, it would be very difficult to separate the components, even if they would crystallize.

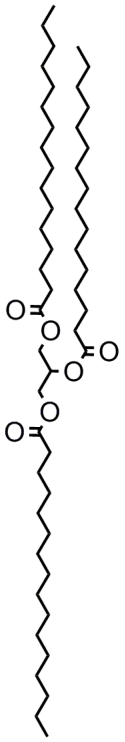
Trimyristin



propane-1,2,3-triyl tritetradecanoate

Chemical Formula: $C_{45}H_{86}O_6$
Molecular Weight: 723.16
MP = 56 - 58

Tripalmitin



Chemical Formula: $C_{51}H_{98}O_6$
Molecular Weight: 807.32
MP = 66 - 67