

## Experiment #6, Natural Product Isolation

Experimental Organic Chemistry: A Miniscale and Microscale  
approach by Gilbert and Martin, Section 5.4

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### INTRODUCTION:

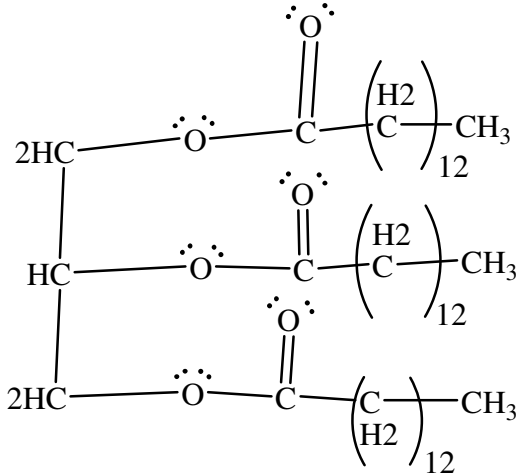
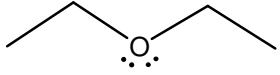
In this experiment, we will extract a natural product, trimyristin, from the spice nutmeg. The process that follows will be a solid-liquid extraction. Although the extraction is a simple version of an extraction and does not yield other structurally related esters, it will serve as a lab to demonstrate how extractions work. Nutmeg, which is insoluble to water, will be dissolved in an organic solvent, such as diethyl ether, dichloromethane, ethanol, and acetone.

Trimyristin, which occurs in many fats and oils, will be isolated without using a series of complex operations. It will be dissolved in diethyl ether and refluxed for 30 minutes. The trimer will be collected using filtration and rotary evaporation.

### PROCEDURE

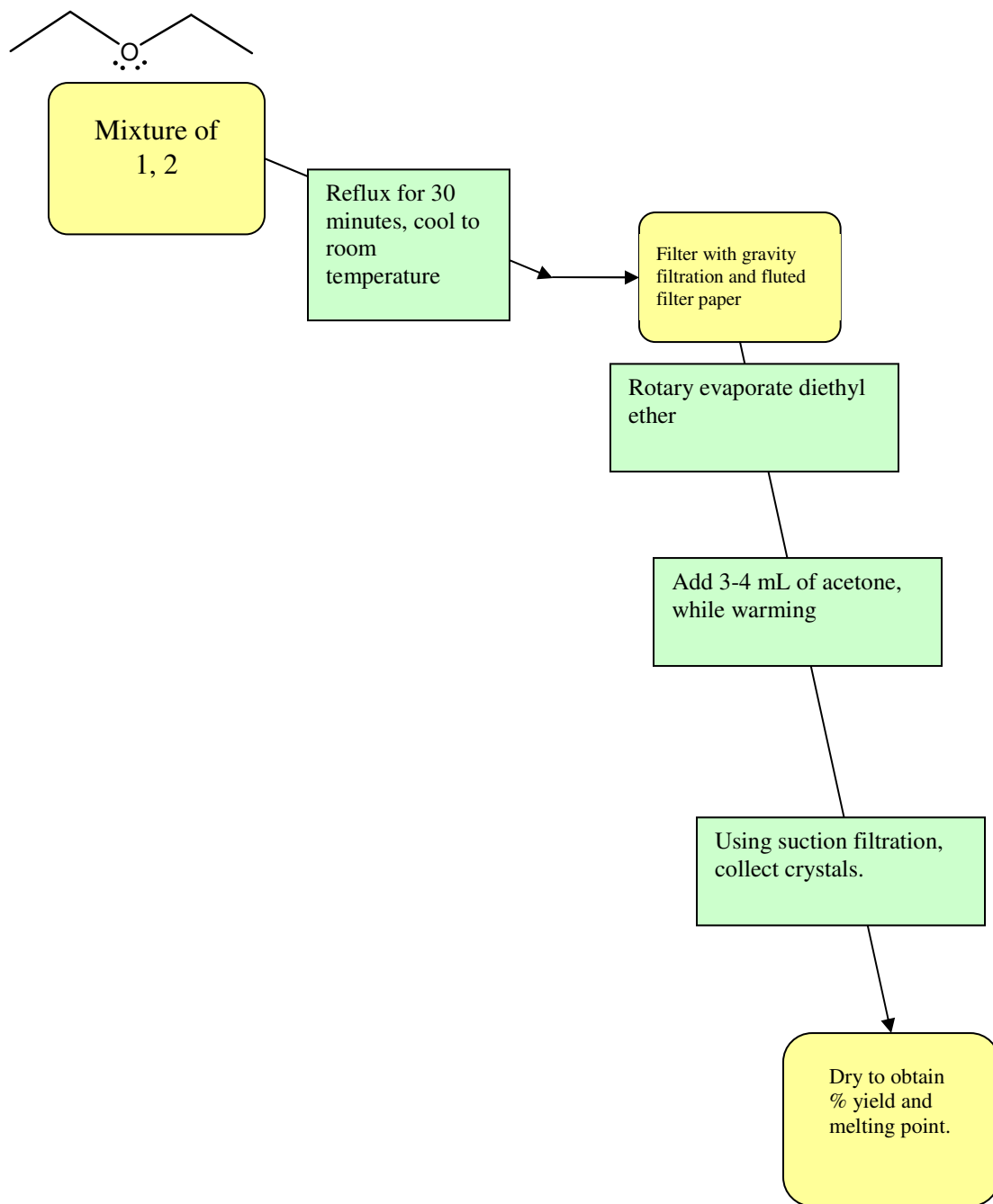
- Place about 4 g of ground nutmeg in a 100-mL round bottom flask containing a stirbar and 20-mL of diethyl ether.
- Set up the lab for simple reflux.
- Be careful with the thermowell under reflux as diethyl ether is flammable. Turn the thermowell to <50 volts.
- Stir the mixture with magnetic stir bar and reflux for 30 minutes.
- Allow the solution to cool to room temperature. Tare the 100-mL round bottom flask.
- Use the stemless glass funnel with a 11 cm fluted filter paper, collect the filtrate in a 50-mL round bottom flask. Wet the filter paper slightly before filtering. Rinse any remaining residue in the 100-mL flask with an additional 5-mL of diethyl ether and pour the solution onto the filter paper.
- The diethyl ether should be removed from the yellow solution after gravity filtration using the rotary evaporator.
- Dissolve the yellow oil in 3-4 mL of acetone while warming the mixture. Pour hot solution to 25-mL flask. Cool to room temperature for 30 minutes.
- Scratch the liquid-air surface to induce crystallization. Cool in ice bath for 15 minutes.
- Using a buchner funnel, collect the crystals with suction filtration.
- Allow the sample to air dry, obtain a weight and melting point, and calculate the % recovery.
- Submit the sample in a carefully labeled vial to the instructor with your completed lab #6.
- Used filter papers may be discarded in the trash can.

**TABLE 1: REAGENTS**

| <u>Flow Chart Number</u> | <u>Compound</u>  | <u>Melting/Boiling Point (°C)</u> | <u>Molecular weight (g/mol)</u> | <u>Solubility</u>   |
|--------------------------|--|-----------------------------------|---------------------------------|---|
| <b>1</b>                 |  <p>Trimyristin</p>   | (melting)<br>56-57                | 723.14                          | Insoluble in water, density = 0.885 g/mL, yellowish-grey solid, 20-25% of dried nutmeg. |
| <b>2</b>                 |  <p>Diethyl ether</p> | (boiling)<br>35                   | 74.12                           | Sweet pungent odor, clear colorless liquid, density = 0.7135                            |

## RESULTS AND OBSERVATIONS

*Flow Chart of Reaction* (The numbers in the following flow chart are derived from Table 1: Reagents)



## RESULTS AND OBSERVATIONS (cont.)

3.90 g ground nutmeg

**TABLE 2: RESULTS**  
(using mel temp #16)

| <b>Compound</b> | <b>Empty watchglass and filter paper (g)</b> | <b>Watchglass, filterpaper, &amp; Trimyristin (g)</b> | <b>Trimyristin weight (g)</b> | <b>Standard Melting Point (°C)</b> | <b>Experimental Melting Point (°C)</b> |
|-----------------|--|---|-------------------------------|------------------------------------|--|
| Trimyristin     | 54.90  | 55.24   | 0.34                          | 56-57                              | 45.4-47.6                              |

**TABLE 3: RECOVERY AMOUNTS**

| <b>Compound</b> | <b>Amount Recovered (g)</b> | <b>Theoretical Amt to be Recovered (g)</b> | <b>% Recovery (%)</b> |
|-----------------|-----------------------------|--|-----------------------|
| Trimyristin     | 0.34                        | $20\% * 3.90 = 0.78$                       | 43.6%                 |

## INTERPRETATION OF INSTRUMENTAL DATA

During the lab, 3.90 g of nutmeg was mixed with 20 mL of diethyl ether and refluxed for 30 minutes. Gravity filtration produced a yellow liquid, which was rotary evaporated. A more viscous yellow oil was produced and was washed with 3-4mL of acetone. Crystals formed after cooling the oil. Very small white crystals with a slight yellow hue were formed. 43.6% of product was recovered. This was a decent % yield considering product was left in the flask despite multiple washings of extra acetone. The broad melting point was not surprising because yellow impurities were visibly seen. The experimental melting point was approximately 10°C below the accepted value. Impurities could have been other substances that were extracted in the nutmeg.

## CONCLUSION

In this experiment, two solutes were separated using a solid-liquid extraction. Based on the solubility of the nutmeg, diethyl ether was used to dissolve the reactant. Trimyristin was isolated after 30 minutes of reflux. The resulting product had a 43.6% yield and was not really pure based on the melting point. There were no expected problems besides a lack of suction on the rotary evaporator. The only improvement worth mentioning would be to be careful when using the buchner funnel, because crystals can escape under the filter if not properly wetted using solvent (acetone).

## ANSWERS TO ASSIGNED QUESTIONS

- 1) Diethyl ether was used as a solvent instead of acetone because of its limited polarity. Diethyl ether was able to dissolve the trimyristin. In addition, the boiling point of diethyl ether was approximately 35°C. This low boiling point was not close to the trimyristin and was able to be refluxed successfully. However, if acetone was used, which has a b.p. of 56.3°C, the reflux would have had to occur at a higher temperature, thus increasing the risk of losing product. Trimyristin had a b.p. of 56-57, which was too close to acetone. Therefore the solvent and solute's b.p. should not be close to one another.
  
- 5) Oiling out is a significant indication of impurities. The trimyristin and tripalmitan have similar melting points and thus were probably dissolved in solution together when crystallization was attempted. The large molecule of tripalmitan would be a preventative force in forming crystals as well. (Gilbert and Martin 93-94)