Fats and Oils Isolation and Hydrolysis of Nutmeg Oil (Trimyristin)

Pre-Lab:

➤ Draw the mechanism for the saponification of the trimyristin by sodium hydroxide to produce glycerol and sodium myristate, C₁₃H₂₇COONa. (You need only show the process for one of the esters, you may use the symbol R for simplification.) Assume that trimyristin is RCOOR' and the products are RCOO⁻ Na⁺ (myristic acid) and R'OH (glycerol). This is a nucleophilic acyl substitution.

Answer the following questions:

- 1) Describe what you will do for the recrystallization of Trimytristin.
- 2) Why should the acid solution be chilled?
- 3) What is the concentration of the acid solution you are making?

Procedure:

A) Isolation of Nutmeg Oil (Trimytrisin)

- 1. Place 0.8 grams of ground nutmeg in a small round–bottom flask and add 4 mL of methylene chloride. (Note: both CH₂Cl₂ and oil are nonpolar, so CH₂Cl₂ removes oil from the nutmeg.)
- 2. Attach a condenser with the water line attached and heat the mixture to a gentle reflux for 20 minutes.
- 3. Allow it to cool and carefully decant the solution from the ground nutmeg into a 25 mL Erlenmeyer flask. Keep the solution in the flask on the side.
- Repeat the extraction on the leftover ground nutmeg with another 4 mL portion of methylene chloride. Start the 20-minute reflux again and do the decanting. Combine the solutions from both extractions
- 5. Do a hot filtration if you get solids in your flask. (Hot filtration: Pre-heat the solution gently in a water bath before filtering it through a filter paper. Solution should be warm so that only insoluble substances will collect on the filter paper and that all the soluble substances will pass through the filter.)
- 6. Evaporate the solution to dryness in a water bath to obtain crude trimyristin. Measure the mass.
- 7. Recrystallize the product using 95% ethanol to obtain pure nutmeg oil.
- 8. Measure the mass of the pure oil.

B) Hydrolysis of Trimyristin and Formation of Myristic Acid

The equation for the hydrolysis of trimyristin is shown below. Glycerol and myristate ion are produced.

$$CH_{2}$$
 CH_{2} C

- 1. In a small round–bottom flask mix the extracted trimyristin with 1 mL of 6 M sodium hydroxide and 2 mL of water and heat to reflux for one hour. At the end of this period, the mixture should have largely cleared, indicating complete hydrolysis.
- 2. While your sample is refluxing, prepare a dilute acid mixture to acidify your sodium-myristate by adding 1.0 mL of 12 M of HCl acid to 10.0 ml of cold water. Keep this acid mixture chilled when your reaction mixture is added to it.
- 3. Immediately after the reflux (otherwise you may get some precipitate in your reflux sample if you let it cool for too long), mix the basic trimyristin mixture with stirring into the dilute hydrochloric acid solution that you prepared. Myristic acid will form as a solid.
- 4. Collect the acid by vacuum filtration using a small Hirsch funnel and allow it to air dry. Determine the mass and the melting point of the acid. Show your product to the instructor.

For Your Report:

- 1. Calculate the amount of nutmeg oil per gram of nutmeg.
- 2. Calculate percent yield.
- 3. Discuss the purity of your product.