NATURAL PRODUCT ISOLATION: TRIMYRISTIN EXTRACTION FROM NUTMEG

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**Introduction:** The purpose of this experiment is to isolate trimyristin from nutmeg. This is an atypical natural product extraction. Most extractions are more complex, since a variety of products are extracted in the solvent. In this experiment, relatively pure trimyristin will be isolated.

Nutmeg spice comes from the seeds of the *Myristica fragrans* tree in the East Indies. Trimyristin is found in the fixed oil of nutmeg. The fixed oil comprises approximately 24-40% of the nutmeg seed. Trimyristin comprises 73% of the fixed oil. Overall, trimyristin should have a percent recovery of 18-29%.\(^1\) Figure 1 shows how trimyristin is triester formed from the dehydration reaction between glycerol and myristic acid.

![Figure 1](image_url)

**FIGURE 1** The formation of trimyristin from glycerol and myristic acid.

**Experimental Procedure:** A reflux apparatus was assembled. A 3.99g sample of nutmeg was placed in the 100mL round-bottom flask with a stirbar. Diethyl ether (20.52mL) was added to the round-bottom flask. The Thermowell heater was set to 30V and the flask was refluxed at a rate of 1drop/2seconds for 30 minutes. The mixture was gravity filtered with fluted, 11cm filter

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paper into a 50mL round-bottom flask. Additional ether was used to rinse the flask and the filter paper.

The ether solution was rotary evaporated. The resulting oil was dissolved in 3-4mL of acetone. The mixture was warmed, and the solution was poured into a 25mL Erlenmeyer flask. The solution was cooled to room temperature. The flask was scratched at the air-liquid interface, and the flask was cooled in an ice bath for 15 minutes. The crystals were isolated using a Büchner filter. The flask and crystals were washed with a small amount of room-temperature acetone. The purified sample of trimyristin was air-dried and weighed. The melting point of the trimyristin was measured on Mel-Temp #13.

Table of Reactants:

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<tr>
<td>diethyl ether</td>
<td><img src="image" alt="Structure" /></td>
<td>74.1224</td>
<td>20mL</td>
<td>Solvent</td>
<td>ρ = 0.7134g/mL m.p. = -116.3°C b.p. = 34.6˚C ρ_vap = 2.6 P_vap = 442mmHg Sol = 6.9g/100mL H_2O</td>
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Table of Products:

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<tr>
<td>Trimyristin</td>
<td><img src="image" alt="Structure" /></td>
<td>723.1708</td>
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<td>m.p. = 56-58˚C</td>
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Results and Observations: After 30 minutes of refluxing, only a marginal amount of the solvent had evaporated. While gravity filtering, some nutmeg got into the 50mL round-bottom flask, so the ether solution had to be filtered twice. After extraction and purification, 0.07g of trimyristin was obtained. The percent recovery for this experiment was 2%. The resulting product was a dark, yellow-brown, viscous oil. A couple drops of clear liquid, presumably water, were also present. A large amount of cream-colored, fluffy crystals were left after Büchner filtering. It appeared that the crystals were slightly contaminated. Half of the crystals were a darker tan color, and half of the crystals were a lighter cream color. The melting point of the lighter cream-colored crystals was 52.5-54.0°C.

Conclusions: The melting point of the crystals supports the isolation of trimyristin. The melting point range of 52.5-54.0°C was only 2-3°C lower than the literature value of 56-58°C. The small range of 1.5°C suggests relatively pure crystals. The fact that half of the crystals were a darker shade indicates some impurities though. The 2% recovery was a very low compared to the predicted 18-29% recovery. One possible cause of the low percent recovery was the fact that the ether solution had to be filtered twice. More of the trimyristin was probably absorbed into the filter paper. A second possible cause was the fact that room temperature acetone was used to wash the crystals in the Büchner filter. This could have caused some of the crystals to redissolve and pass through the filter paper. The acetone should have been chilled in an ice bath.
1) Why is diethyl ether rather than acetone chosen as the extraction solvent?

   Trimyristin is not very soluble in acetone. That is why acetone was used as the crystallization solvent. The trimyristin has to be soluble in the solvent to get extracted from the nutmeg. Trimyristin is more soluble in ether than in acetone.

5) A certain plant material is known to contain mainly trimyristin and tripalmitin, in approximately equal amounts. Tripalmitin, the structure of which you should look up in a reference source, has mp 66-67°C. Extraction of these two compounds from the plant material with diethyl ether gave an oil after removal of the solvent, and this oil was difficult to crystallize. Explain this result.

   Figure 2 shows that tripalmitin is almost identical to trimyristin. The only difference is that tripalmitin has fatty acid chains of sixteen carbons rather than fourteen carbons. Trimyristin has a melting point of 56-58°C while tripalmitin has a melting point of 66-67°C. Tripalmitin has a higher melting point because it has longer fatty acid chains, and therefore it has more London dispersion forces. As the mixture of oils is cooled, tripalmitin will start to crystallize first. This means the concentration of tripalmitin will decrease in the solution. This lowers the melting point of the mixture closer to the eutectic temperature, the temperature at which tripalmitin and trimyristin both crystallize. The eutectic temperature will be lower than the individual melting points. The oil is hard to crystallize because the eutectic temperature happens to be closer to room temperature than the individual melting points. The oil might have to be chilled in an ice bath even to get crystals to start forming. Even if the crystals do form, they will not be pure.
They will still be a mixture of tripalmitin and trimyristin. Further purification methods like chromatography would need to be employed to separate the two triesters.

**FIGURE 2** Structural diagrams for trimyristin and tripalmitin.