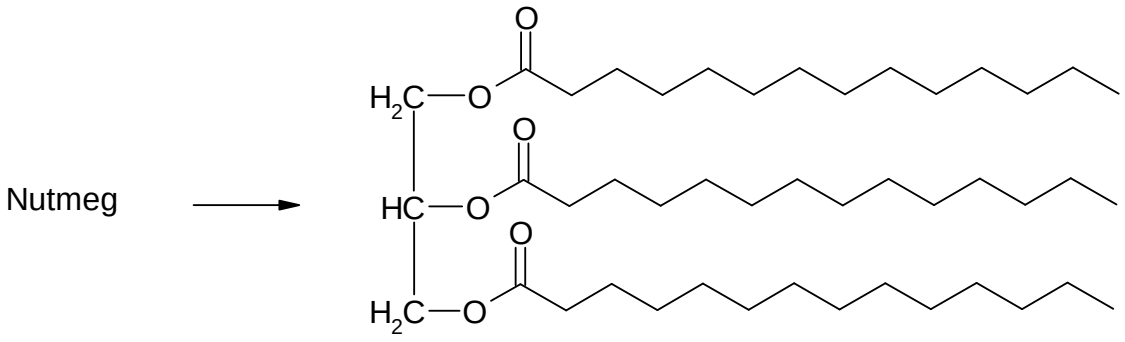
Isolation of trimyristin from nutmeg



$C_{45}H_{86}O_6$
(723.2)

Equipment

Two-necked flask 100 mL, Soxhlet extractor 30 mL, glass wool, extraction cone, heatable magnetic stirrer with magnetic stir bar, oil bath, reflux condenser, rotary evaporator, ice bath, exsiccator with drying agent, suction filter, suction flask

Substances

Nutmeg	8 g
Ethanol	about 50 mL
tert-Butyl methyl ether	50 mL
Sodium hydroxide	2.4 g
Concentrated hydrochloric acid	20 mL

Reaction

The reaction apparatus consists of a 100 mL two-necked flask with a magnetic stir bar and a 30 mL soxhlet extraction unit with a reflux condenser. 8 g of finely ground nutmeg are placed into the extraction sleeve and covered with a little glass wool. 50 mL *tert*-butyl methyl ether are placed into the flask and whilst stirring, the solvent is heated to reflux until the solvent leaving the extraction sleeve is colourless (approximately 5 hours).

Work up

The solvent is evaporated at 60 °C at the rotary evaporator. The flask containing the residue is cooled in an ice bath or the refrigerator until the contents have crystallized to a thick slurry.

Crude product yield: 4 g

The crude product is recrystallized from a small amount of ethanol. Prior to filtering the crystals, the flask is placed into the refrigerator for at least 30 minutes. The crystalline slurry is filtered and the product is dried in an evacuated desiccator over silica gel. Should the crystals not be colourless after the first recrystallization, a second recrystallization is carried out.

Yield: 2 g; melting point 54-55 °C

Hydrolysis of the triglyceride

0.3 g of the product are placed into a 100 mL round-bottom flask. 10 mL of a 6 M sodium hydroxide solution in water are added with the necessary safety precautions. Then, 10 mL ethanol are added and the solution is heated slightly under reflux for one hour. If ethanol is lost, it has to be refilled aproximately to the level at the start of the reaction.

The solution is poured into 100 mL water in a large beaker. After addition of 20 mL concentrated hydrochloric acid, a solid white product is formed, which is filtered, washed with 5 mL of water and dried.

Analysis of the fatty acids

Approximately 10 mg of the product of the hydrolysis are placed in a 100 mL measuring flask. Fill up with 50 mL of methanol and 50 mL of water. Calculate the approximate molarity of the solution, assuming it is pure tetradecanoic acid (synonymous to myristic acid).

Calculate the nominal mass of myristic acid and of similar fatty acids that you might expect in your product. What kind of isotope patterns do you expect?

A mass spectrum of the dissolved fatty acids in solution is generated with the mass spectrometer with electron spray interface (ESI). Include spectra in positive and negative mode in your report and discuss every peak, even if you are not sure what it is.

Duration of the experiment

Two days

Where can I stop the experiment

Before and after the evaporation of the solvent

Recycling

The evaporated *tert*-butyl methyl ether and the evaporated ethanol from the mother liquor are collected and redistilled.

Suggestions for waste disposal

Waste	Disposal
residue from mother liquor	domestic waste
residue from extraction	domestic waste

Questions

1. What are the main sources of hazard for health and environment in the experiment?
2. What do you have to keep in mind when setting up the glass apparatus?
3. What are the advantages of a soxhlet extractor in comparison to a simple extraction e.g. in a round-bottom flask?
4. What active substance(s) are in nutmeg? What kind of effects do they have on humans upon ingestion? Where would you expect to find them after the experiment?
5. Please write down the mechanism of the ester hydrolysis.
6. Where does the glycerin go?
7. Answer the questions regarding the MS analysis above.