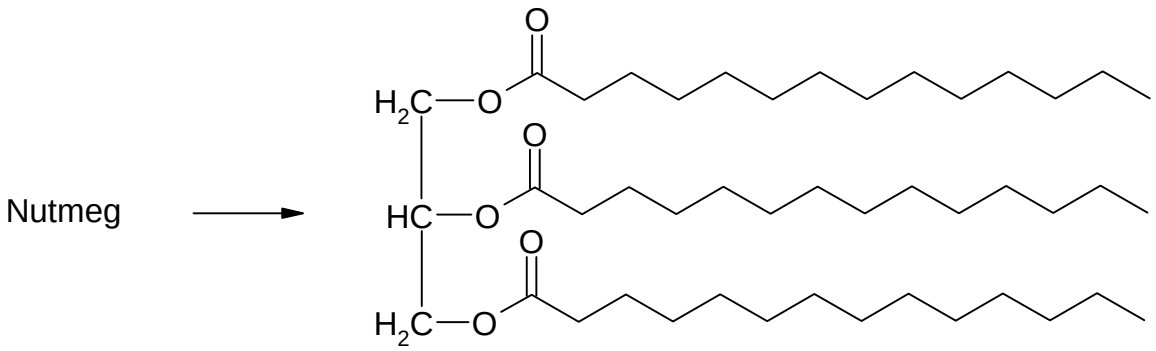
5019 Isolation of trimyristin from nutmeg



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

Classification

Reaction types and substance classes
isolation of natural products
carboxylic acid ester, triglyceride, natural product

Work methods
microwave-assisted extraction, recrystallizing, filtering, evaporating with rotary evaporator

Instruction (batch scale 25 g)

Equipment
microwave heating system ETHOS 1600 and hot extraction filtration apparatus HEF 270, rotary evaporator, suction flask, suction filter

Substances

ethanol (bp 78 °C)	240 mL
nutmeg powder	9-15 g

Reaction
For the set up of the extraction apparatus in the microwave see:
“Technical Instructions. Hot extraction filtration apparatus for microwave systems”.

In each of the inner glass vials of the apparatus, 3-5 g nutmeg powder, a magnetic stir bar and 40 mL ethanol are placed. A further 40 mL ethanol are filled in between the inner vials and the outer wall.

Setting parameters:
The program Easywave® is opened on the PC and the following parameters are set:
t_1 (time to reach the set temperature): 5 min;
t_2 (time at set temperature): 10 min;

T_0 (room temperature);
T_1 (set temperature): 120 °C;
P_1 (power in step 1): 700 W;
P_2 (power in step 2): 500 W.
The magnetic stirrer is activated and the program is started.

Work up
The recovered ethanol extracted is stored overnight in the refrigerator. The precipitated trimyristin is filtered using a funnel or a small frit and then dried. If the crystals are not colourless, the crude product is recrystallized from ethanol.

Yield of pure trimyristin from 10 g Muscat powder: 0.6-0.9 g (4-6% when compared to the amount of nutmeg used); colourless, very fine crystals; mp 54-55 °C (lit. 54-55 °C)

The filtrate is concentrated on the rotary evaporator, leaving a light brown oil. The following fatty acids can be identified in this oil, after transesterification with methanolic potassium hydroxide: Capronic acid, tridecanoic acid, myristin acid, pentadecanoic acid, palmitin acid, *cis*-10-heptadecenoic acid and oleic acid.

Comments
If the nutmeg powder is provided by grinding whole nutmeg immediately before starting the extraction, the yield can be increased up to 20 % trimyristin. The amount of total extract is then increased to about 50 %.

Waste management

Recycling
The evaporated ethanol is collected and redistilled.

Waste disposal

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

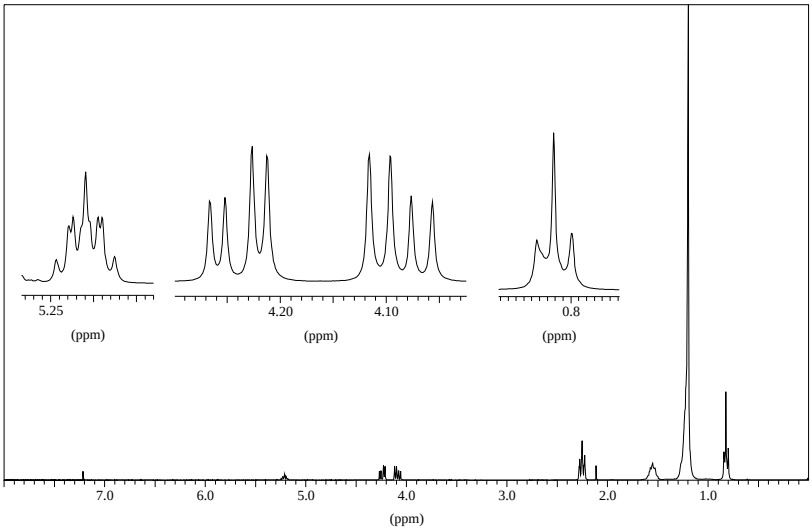
Time
Without recrystallization 1 hour

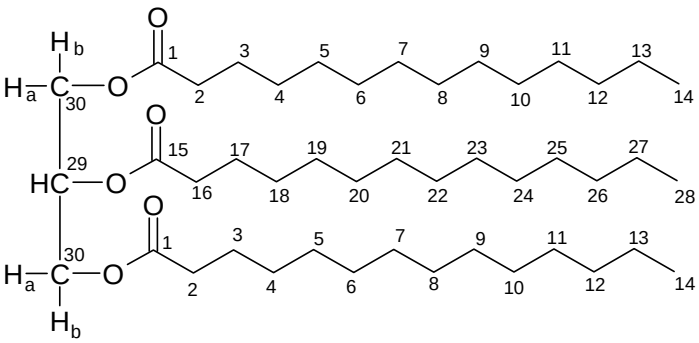
Break
Almost at any time, as long as the solvent is taken out of the reaction vials whilst still hot.

Degree of difficulty
Medium

Analytics

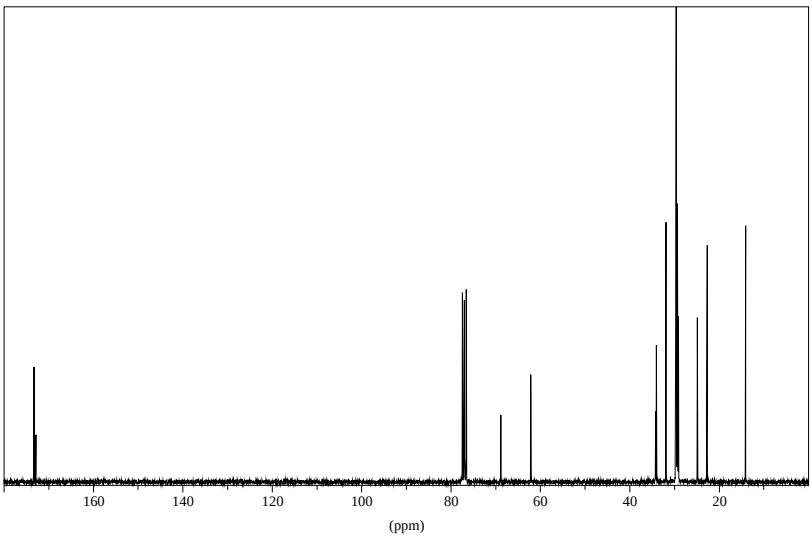
^{1}H NMR spectrum of the pure product (300 MHz, $CDCl_3$)

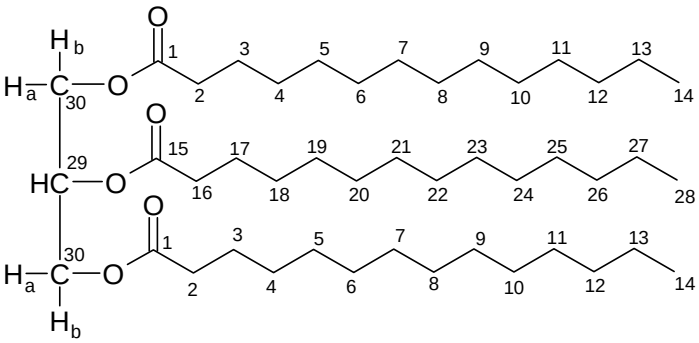




δ (ppm)	Multiplicity	Number of H	Assignment
0.90	m	9	14-H, 28-H
1.2-1.4	m	60	4-H up to 13-H, and 18-H up to 27-H
1.5-1.7	m	6	3-H, 17-H
2.33	m	6	2-H, 16-H
4.16	dd	2	30-H_a
4.31	dd	2	30-H_b
5.28	m	1	29-H
7.26			solvent
2.11			acetone (impurity)

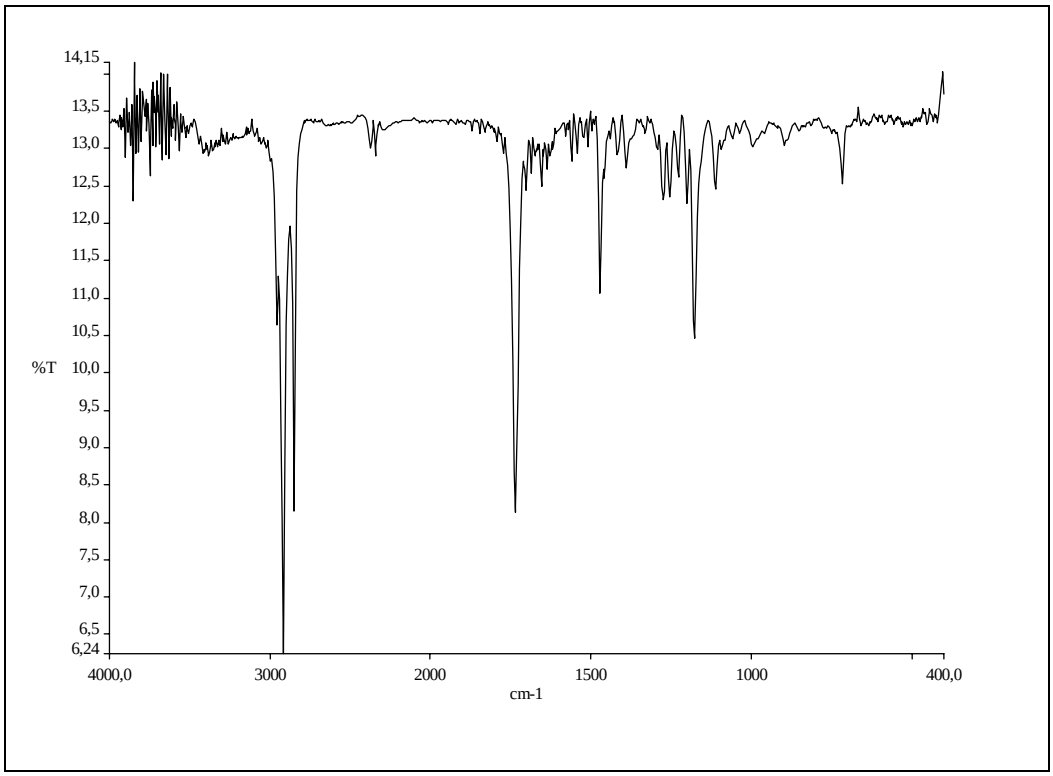
^{13}C NMR spectrum of the pure product (300 MHz, $CDCl_3$)





δ (ppm)	Assignment
14.08	C-14, C-28
22.66	C-13, C-27
24.85, 24.89	C-3, C-17
29.06-31.90	C-4 up to C-12 and C-18 up to C-26
34.04, 34.20	C-2, C-16
62.08	C-30
68.85	C-29
172.85,	C-15
173.26	C-1
76.5-77.5	solvent

IR spectrum of the pure product (KBr)



(cm^{-1})	Assignment
2950 - 2850	C – H – valence, alkane
1730	C = O – valence, ester