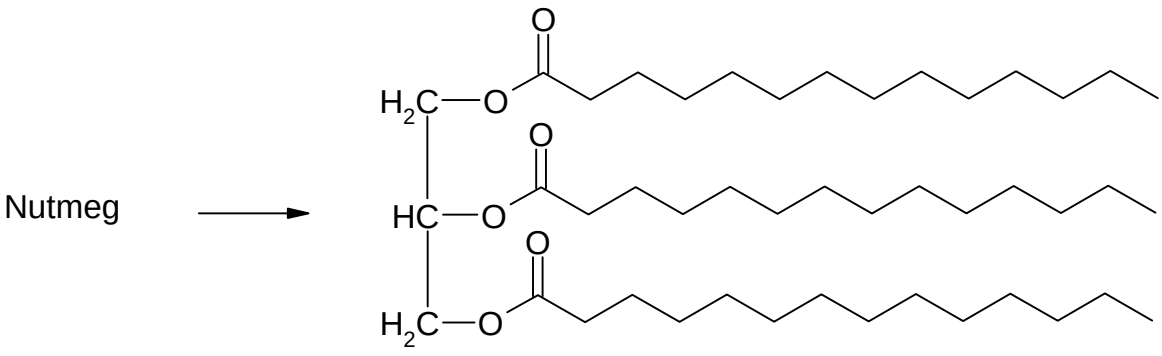
1021 Isolation of trimyristin from nutmeg



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

Classification

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

Work methods
extracting with Soxhlet extractor, evaporating with rotary evaporator, recrystallizing, filtering, heating under reflux, stirring with magnetic stir bar, heating with oil bath

Instruction (batch scale 25 g)

Equipment
250 mL round-bottom flask, 100 mL Soxhlet extractor with extraction sleeve, reflux condenser, heatable magnetic stirrer with magnetic stir bar, rotary evaporator, suction flask, suction filter, desiccator, oil bath

Substances

nutmeg, finely ground	25 g
tert-butyl methyl ether (bp 55 °C)	150 mL
ethanol (bp 78 °C)	etwa 150 mL

Reaction
The reaction apparatus consists of a 250 mL round-bottom flask with a magnetic stir bar and a 100 mL soxhlet extraction unit with a reflux condenser. 25 g of finely ground nutmeg are placed into the extraction sleeve and covered with a little glass wool. 150 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 with a final pressure of 20 hPa. The flask containing the residue is cooled in an ice bath or the refrigerator until the contents has crystallized to a thick slurry.
Yield crude product: 12 g

The crude product is recrystallized from the minimum 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 6.5 g (26% according to the amount of used nutmeg); mp 54-55 °C.

Waste management

Recycling

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

Waste disposal

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

Time

Without recrystallization 6 hours

Break

Before and after the evaporation of the solvent

Degree of difficulty

Easy

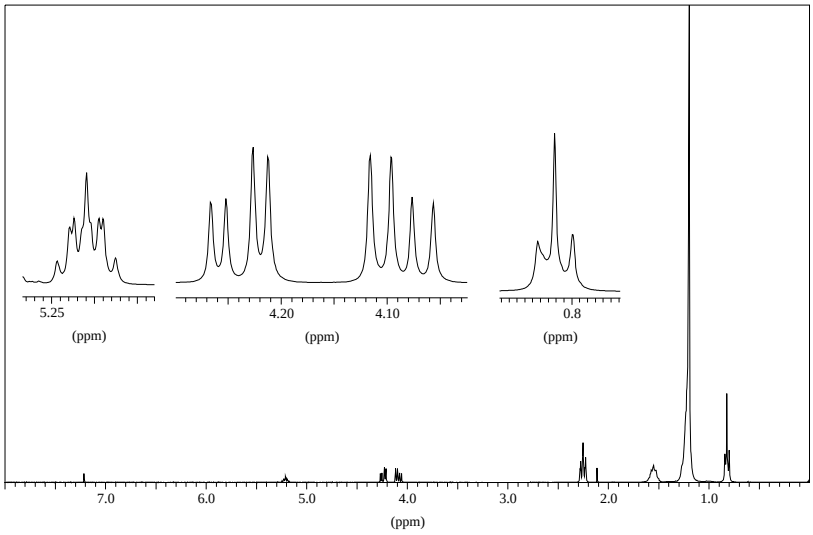
Analytics

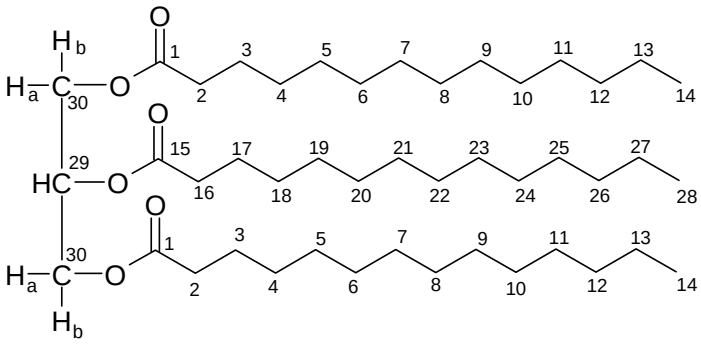
TLC

TLC conditions:

adsorbent:	Macherey and Nagel Polygram SilG/UV plates, 0.2 mm
elution solvent:	cyclohexane/ethyl acetate 9.5: 0.5
visualizing agent:	Vaughn`s Reagent or iodine vapour (Vaughn's Reagent consists of 45 mL water, 5 mL conc. sulphuric acid, 2.4 g ammoniumheptamolybdate tetrahydrate($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) and 0.1 g $Ce(SO_4)_2$.) (Iodine vapour: The dried TLC foil is placed into a vessel containing a few iodine crystals. The vessel is closed and the iodine crystals are heated with the heat gun until an iodine vapour forms and the substance spots become visible).
R_f (trimyristin):	0.51

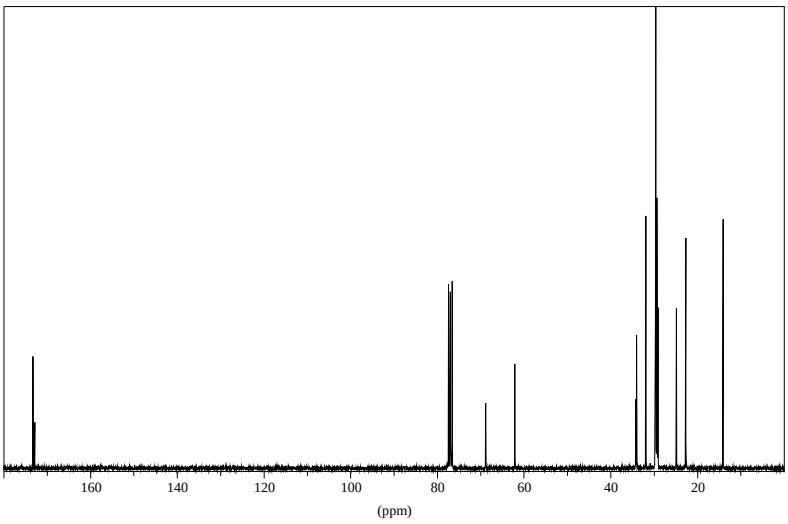
^{1}H NMR spectrum of the pure product (300 MHz, $CDCl_3$)

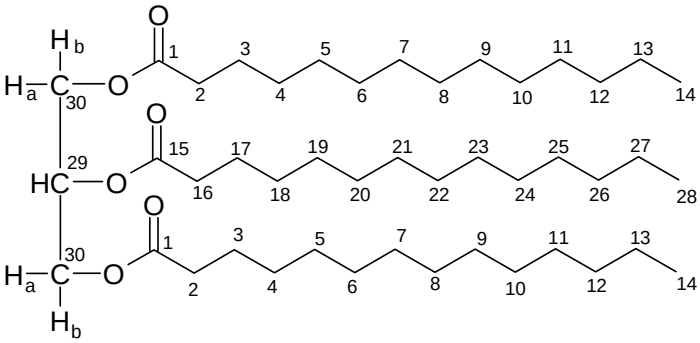




δ (ppm)	Multiplicity	Number of H	Assignment
0.90	m	9	14-H, 28-H
1.2-1.4	m	60	4-H up to 13-H, and 18-H up to 27-H
1.5-1.7	m	6	3-H, 17-H
2.33	m	6	2-H, 16-H
4.16	dd	2	30-H_a
4.31	dd	2	30-H_b
5.28	m	1	29-H
7.26			solvent
2.11			acetone (impurity)

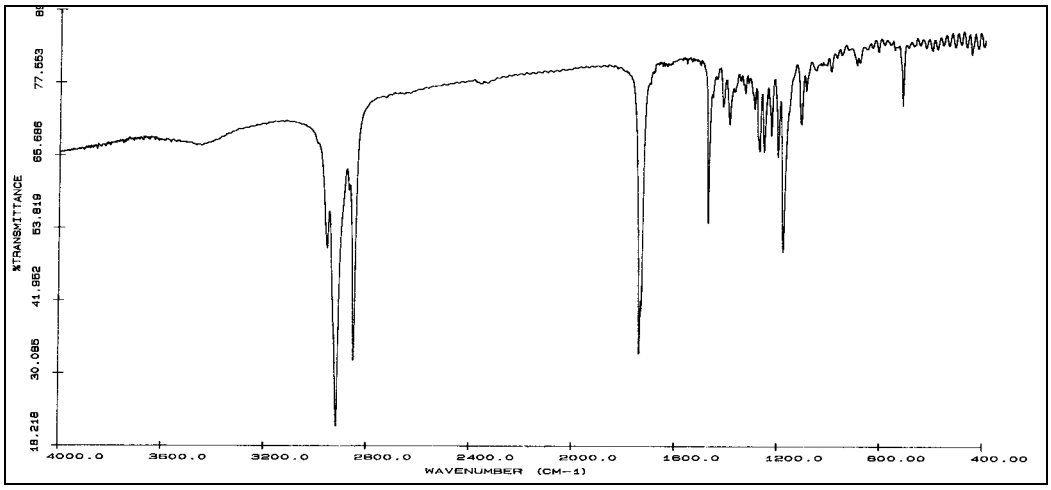
^{13}C NMR spectrum of the pure product (300 MHz, $CDCl_3$)





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