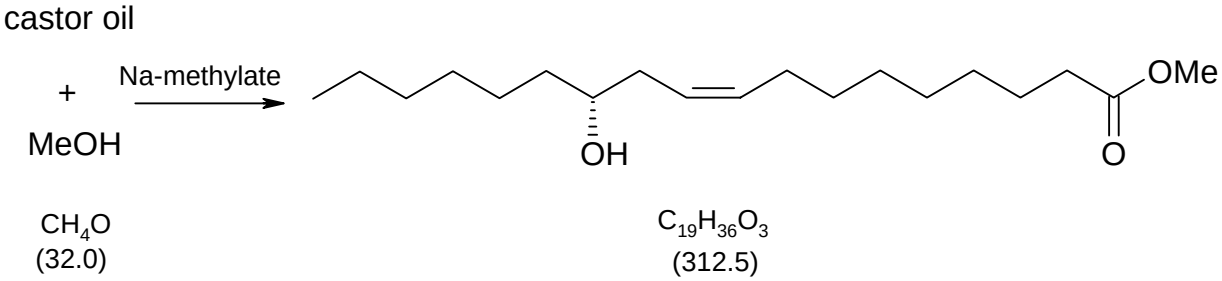
4001 Transesterification of castor oil to ricinoleic acid methyl ester



Classification

Reaction types and substance classes
reaction of the carbonyl group in carboxylic acid derivatives, transesterification
carboxylic acid ester, triglyceride, renewable resources,

Work methods
stirring with magnetic stir bar, shaking out, extracting, filtering, evaporating with rotary evaporator

Instruction (batch scale 10 mmol)

Equipment
100 mL two-neck flask, magnetic stirrer, magnetic stir bar, round bottom flask, separating funnel, rotary evaporator

Substances

castor oil	10 g (about 10 mmol)
methanol	32 g (40 mL, 1.0 mol)
sodium methylate solution (16 %) in methanol	0.3 mL
petroleum ether (bp 60–80°C)	40 mL
sodium sulfate for drying	about 1 g

Reaction
In a 100 mL two-neck flask with magnetic stirrer 10.0 g (about 10 mmol) castor oil are stirred with 32 g (40 mL, 1.0 mol) methanol. After addition of 0.3 mL sodium methylate solution the mixture is stirred about 45 minutes further until the transesterification is completed. The reaction course is followed via thin layer chromatography (see analytics).

Work up
The excessive methanol is evaporated at the rotary evaporator. The remaining crude product is transferred with 40 mL petroleum ether (60-80°C) into a separating funnel and shaken with

20 mL of water. The organic phase is separated and repeatedly shaken with 20 mL of water until the pH-value is neutral. Afterwards the organic phase is dried over sodium sulfate, then filtered and the solvent evaporated at the rotary evaporator.

Yield 9.0 g (28 mmol, 93%); GC purity 89%.

Waste management

Recycling
Petroleum ether and methanol are collected and redistilled.

Waste disposal

Waste	Disposal
sodium sulfate	solid waste, free from mercury
aquaeous phase from shaking out	solvent water mixtures, halogen free

Time
3 hours

Break
After complete transesterification of the castor oil

Degree of Difficulty
Easy

Instruction (batch scale 100 mmol)

Equipment:
1 L two-neck flask, magnetic stirrer, magnetic stir bar, round bottom flask, separating funnel, rotatory evaporator

Substances

castor oil	100 g (about 100 mmol)
methanol	320 g (400 mL, 10 mol)
sodium methylate solution (16 %) in methanol	3 mL
petroleum ether (bp 60-80°C)	300 mL
sodium sulfate for drying	about 5 g

Reaction
In a 1 L two-neck flask with magnetic stir bar 100 g (about 100 mmol) castor oil are stirred with 300 mL methanol. After addition of 3 mL sodium methylate solution the mixture is stirred about 45 minutes further until the transesterification is completed. The reaction course is followed via thin layer chromatography (see analytics).

Work up

The excessive methanol is evaporated at the rotary evaporator and the remaining crude product is transferred with 300 mL petroleum ether (60-80°C) into a separating funnel. After removing of the separated glycerine the organic phase is repeatedly washed with 100 mL water each time until the pH-value is neutral. Afterwards the organic phase is dried over sodium sulfate, then filtered and the solvent evaporated at the rotary evaporator.

Yield: 100 g (0.300 mol, 100 %); GC purity 88%.

Waste management

Recycling

Petroleum ether and methanol are collected and redistilled.

Waste disposal

Waste	Disposal
sodium sulfate	solid residue, free from mercury
aquaeous phase from shaking out	solvent water mixtures, halogen free
glycerine from the separating funnel	organic solvents, halogen free

Time

4 hours

Break

After complete transesterification of the castor oil

Degree of difficulty

Easy

Analytics:

Reaction monitoring with TLC

Sample preparation:

With a pipette one drop of the upper phase of the reaction mixture is diluted with 1 mL dichlormethane.

TLC conditions:

adsorbant:	TLC-aluminium foil (silica gel 60)
eluent:	petroleum ether (60-80 °C)/acetic acid ethyl ester 7 : 3
visualisation:	The TLC-aluminium foil is dipped in 2N H_2SO_4 and afterwards dried with a hot-air dryer.
R_f (educt)	0.47
R_f (product)	0.64

GC:

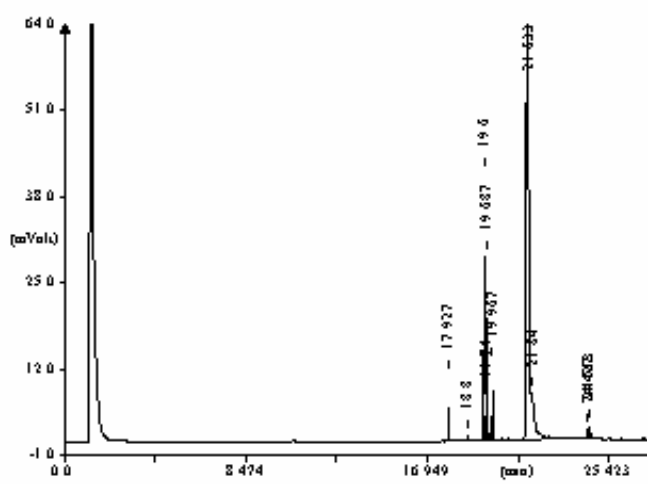
Sample preparation:

One drop of the product is diluted with 10 mL dichlormethane. 0.2 µl from this solution are injected.

GC-conditions:

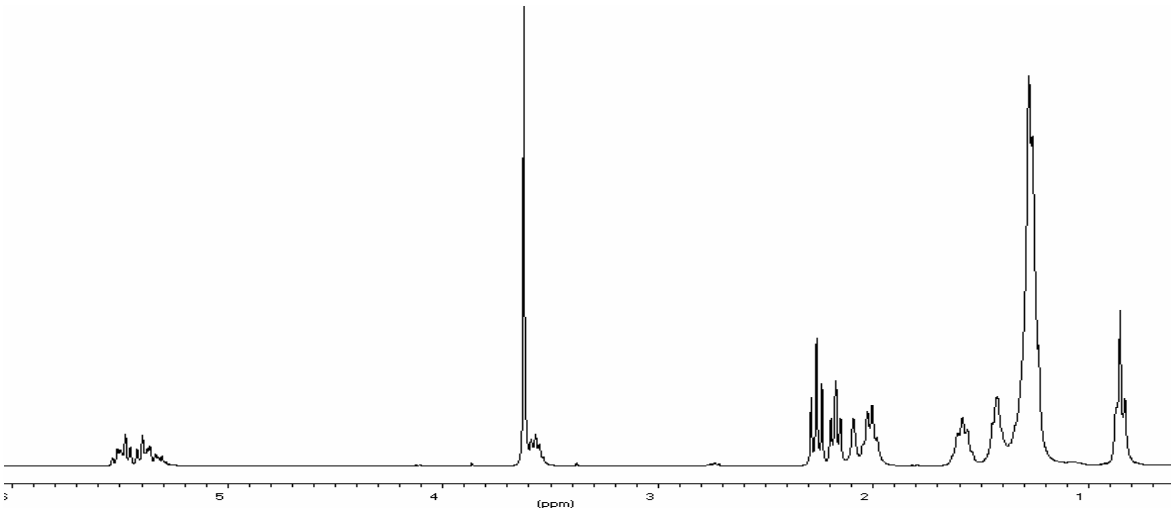
column:	DB-1, 28 m, internal diameter 0.32 mm, film 0.25 µm
inlet:	on-column injection
carrier gas:	hydrogen (40 cm/s)
oven:	90 °C (5 min), 10 °C/min to 240 °C (20 min)
detector:	FID, 270 °C

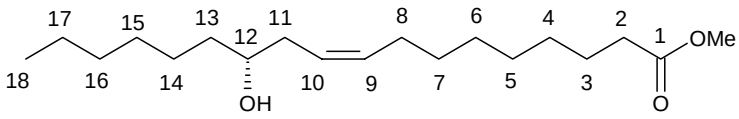
GC of the product



Retention time (min)	Substance	Peak area %
17.93	palmitic acid methyl ester	0.9
19.61	stearic acid methyl ester	4.3
19.69	oleic acid methyl ester	2.7
19.97	linoleic acid methylester	1.0
21.69	ricinoleic acid methyl ester	89.4

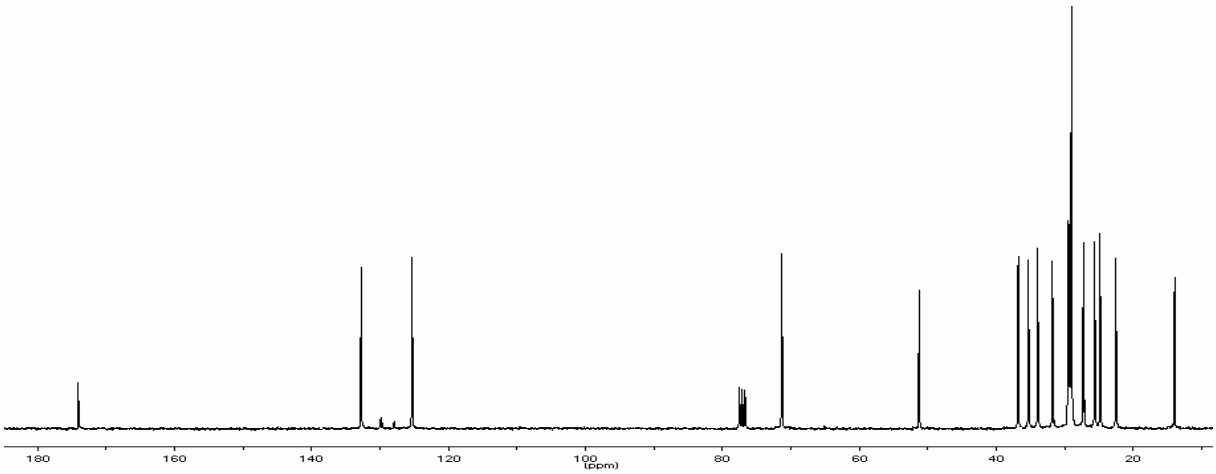
^{1}H NMR spectrum of the product (300 MHz, $CDCl_3$**)**

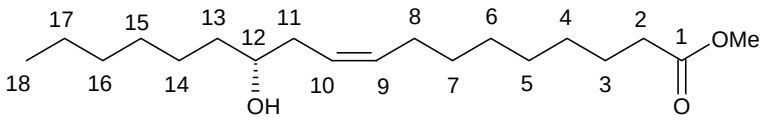




δ (ppm)	Multiplicity	Number of H	Assignment
0,84	T	3	18-H
1,24	M	16	remaining CH_2
1,41	M	2	13-H
1,59	M	2	3-H
2,02	M	2	8-H
2,09	S	1	-OH
2,19	M	2	11-H
2,28	T	2	2-H
3,59	M	1	12-H
3,61	S	3	$-OCH_3$
5,35	M	1	9-H
5,49	M	1	10-H

^{13}C NMR spectrum of the product (75.5 MHz, $CDCl_3$)





δ (ppm)	Assignment
174.0	C=O
132.6	C-10
125.3	C-9
71.2	C-12
51.1	O-CH3
36.7	C-11
35.2	C-13
33.8	C-2
22.4	C-17
13.8	C-18
76.5-77.5	solvent