CHEMISTRY
A EUROPEAN JOURNAL

Supporting Information

Rational design of sugar-based “surfactant combined catalyst” for promoting glycerol as solvent for catalysis

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1) Chemicals: Hydroxyethylcellulose (HEC, MS=2.5) was purchased from Dow Chemicals. Silica gel (60 ACC 40-63 µm, surface area: 550 m^2/g) was purchased from CARLO ERBA REACTIFS-SDS. Lauric, oleic, juniperic, 12-hydroxy stearic acids, 1,2-epoxydodecan, cyclohexaneoxide, 2,3-epoxypropylphenylether, diethanolamine, octylamine, 10-undecenoic acid, benzaldehyde, and malononitrile were provided by Sigma. 3,4-dimethoxyphenylacetic acid, cyclohexanecarboxylic acid, and 1,2-epoxy-9-decene were purchased from Alfa Aesar. 2,4-pentanedione, laurylaldehyde, cinnamaldehyde, p-anisaldehyde, nitroethane, chitosan, dodecylamine, butylamine, celite® 545 and sodium periodate were purchased from Acros. 2-cyclohexen-1-one was purchased from Fluka.

2) Apparatus : ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance 300 DPX 300. Chemical shift are expressed in ppm relative to Me_4Si. IR spectra were recorded on a FT-IR Perkin Elmer (spectrum one) using ATR technology. Elemental analyses were measured on a NA 2100 Instrument. Melting point were determined on a BUCHI B-540.

3) Chromatographic analyses

The reaction progress was monitored on a Varian 3300 GPC equipped with a BPX5 column (12m x 0.22 mm; phase thickness: 0.25 µm) supplied by SGE, a Flame Detector Ionization and an injector on-column. Prior analysis, hydroxylated products were silylated according to the Sahasrabuhde method (*J. Am. Oil. Chem. Soc*., 1957, **44**, 376). Beside NMR experiments, purity of products was also confirmed by GC using an internal calibration with dodecane. GC response coefficients are provided below.

4) Phase contrast microscopy

Observations were made at room temperature at 20x magnification using an optical microscope in the phase contrast mode (Nikon Eclipse E-400) equipped with a 3-CCD JVC camera allowing digital images (768 x 512 pixels) to be collected. A drop of the emulsion (about 20mL) was deposited on the glass slide surface (76 mm x 26 mm x 1.1 mm, RS France) and covered with a cover slide (22 mm x 22 mm, Menzel-Glaser). The glass slides were previously cleaned with ethanol and acetone.

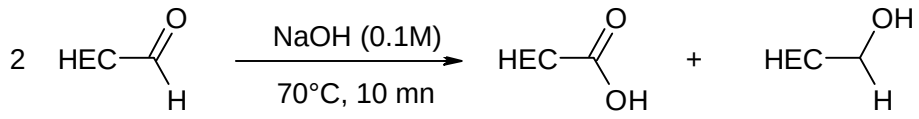
5) Synthesis of APs

<u>Step 1: oxidation of HEC</u>

Two different routes were employed for oxidation of HEC:

Route A: The hydroxyethyl cellulose (100,4 g) was dissolved in 2,3 L of water. Sodium periodate (125.6 g-587.2 mmol) was then directly added to the solution. The mixture was stirred in the dark at room temperature for 24 hours. After this period, the oxidized HEC was precipitated by addition of a mixture acetone/ethanol (2:1 v/v, 6L). The resulting white solid was filtered off and extensively washed with cold acetone/ethanol (2:1 v/v). After freeze-drying, the oxidized HEC was kept under vacuum (15 mmHg).

The number of aldehyde formed on the polysaccharide structure was determined by titration using known method based on the Cannizzaro reaction.[1]



Cannizarro titration

Typically, 100 mg of oxidized HEC were dissolved in a basic solution of NaOH (10mL, 0.1M) and stirred for 10 minutes at 70°C. The resulting solution was then cooled down to room temperature and 10mL of HCl (0.1M) were added. Excess of HCl was titrated with a solution of NaOH (0.02M). The number of aldehyde corresponds to twice of the amount of NaOH used. In the present case, titration indicated a degree of substitution (DS) of 0.3.

Route B: The sodium periodate (15.5 g-72.8 mmol) and hydroxyethyl cellulose (12.2 g) were separately dissolved in 210 mL and 75 mL of water respectively. Both solutions were then mixed and stirred in the dark at room temperature. After 24 h of stirring, 45mL of ethylene glycol was added to the solution and the mixture was subject to dialysis (Spectra/Por membranes; diameter 25.5 mm, flat width: 40 mm, 6000-8000 dalton) during 48 h. The recovered solution in the membranes was then evaporated under reduced pressure. The recovered gel was then freeze-dried affording after 48hours 10g of oxidized polysaccharide.

As previously mentioned, titration of aldehyde with the Cannizarro reaction indicated a DS=0.5.

Typical Infra Red spectrum of oxidized Hydroxyethylcellulose: IR (in KBr) ν cm^{-1} 3419 (OH), 2880 (CH, CH_2), 1732 (C=O), 1634, 1456, 1360, 1063, 886 cm-1.

[1] K. Pommerening, H. Rein, D. Bertram, R. Müller, *Carbohydr. Res.*, (1992), **233**, 219-223

Step 2: general procedure for the reductive amination of oxidized polysaccharides

The fatty amine (butyl-, octyl- or dodecylamine, 32.4 mmol) was first dissolved in 196 mL of methanol before addition of 48mL of water. The oxidized polysaccharide (5 g, DS=0.5 or 0.3) and 600 mg of Pd/C (5% wt) were then added to the solution. The resulting mixture was stirred overnight in an autoclave at room temperature under pressure of hydrogen (40 bars). At the end of the reaction, the Pd/C was then removed by filtration over Celite® 545 and the filtrate was evaporated under reduced pressure affording a yellow gel. The recovered gel was washed five times with hot heptane in order to remove the excess of fatty amine. After this washing, the obtained gel was subject to a freeze-drying affording about 4g of aminopolysaccharide (**AP1a, 1b, 2, 3**).

It has to be noted that, during the synthesis of APs, the hydroxycellulose was partially depolymerized. Indeed, HPLC (gel permeation chromatography, calibration with dextrans) investigations clearly revealed the depolymerization of hydroxyethylcellullose step after step. Initially, measured Mw for the starting hydroxyethylcellullose was 1 267 000 Da. After oxidation with NaIO4, the Mw of HEC dropped to 495 000 Da (dialyzed sample). After, catalytic reductive amination, only oligomers were detected (<26500Da for dialyzed sample). Consequently, all APs used in this study are actually a mixture of different oligomers. The amount of fatty chain was determined by NMR and potentiometric titration (see below).

Determination of the amino content:

The amount of fatty chain grafted over the oxidized polysaccharide was determined by potentiometric titration. In a typical procedure, 0.1 g of APs was dissolved in 10mL of an aqueous solution of HCl (0.01M). Titration of the resulting solution was then carried out with a solution of KOH 0.01M and the pH evolution was monitored over a pH meter 510 from EUTECH instruments.

Determination of the degree of substitution (DS):

The degree of substitution on fatty amine was determined by ^{1}H NMR based on the ratio of integration between the protons held by the fatty chain and the protons held by the “ex-glucose” units. For dodecylamine, the following general equation was used:

$$DS = \frac{16 \times I_{CH_2CH_3}}{25 \times I_{gluc} - 3 \times I_{CH_2CH_3}}$$

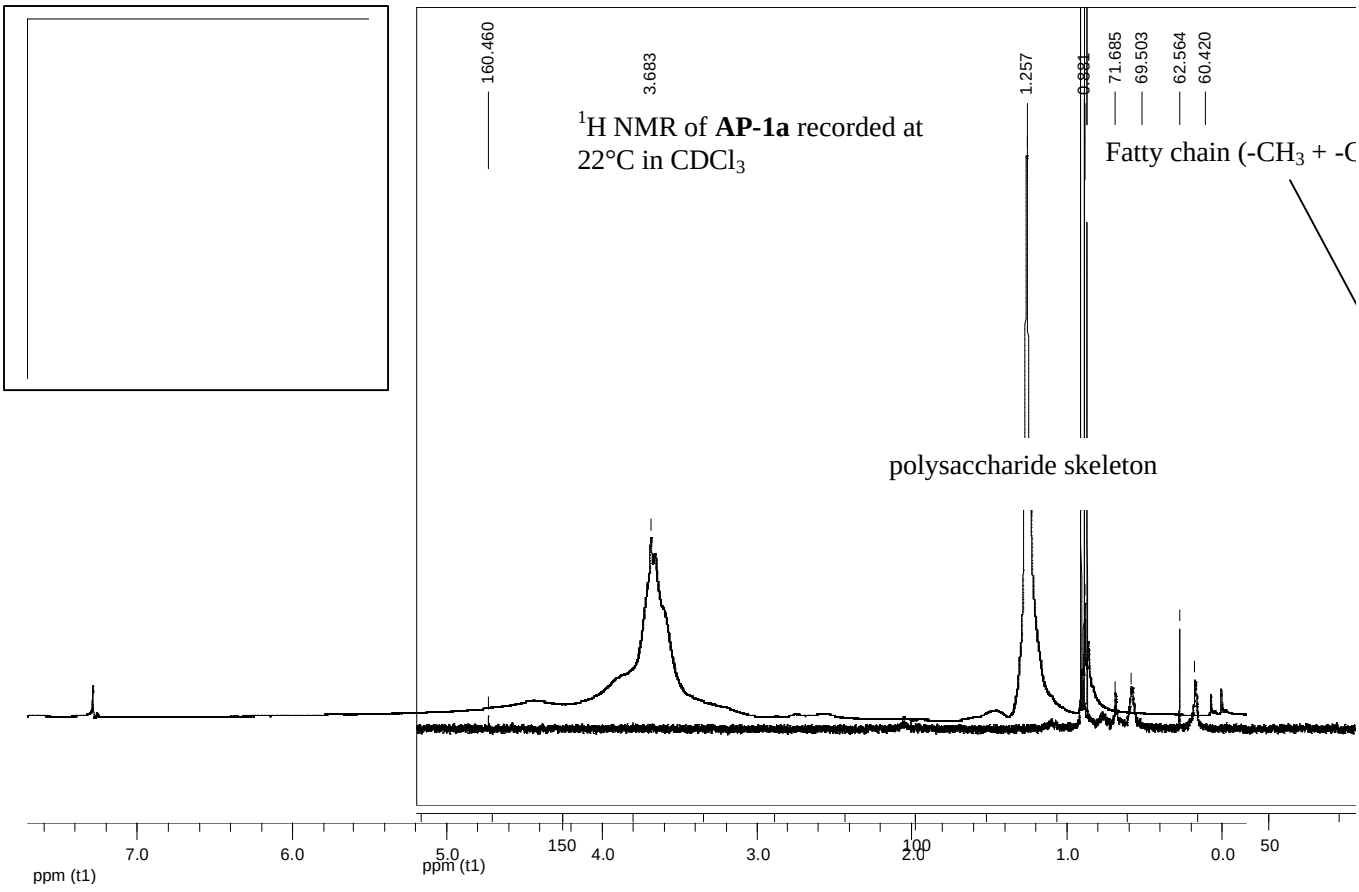
I_{CH2CH3} represents the integration of the proton held by the fatty chain (0.8 and 1.2 ppm)

I_{gluc} represents the integration of the “ex-glucose units” (3.0-4.0 ppm).

Aminopolysaccharide	Amine	DS_{NH}	Amino content (mmol/g)
AP-1a	Dodecylamine	0.5	1.6
AP-1b	Dodecylamine	0.3	1.0
AP-2	Octylamine	0.5	0.8
AP-3	Butylamine	0.5	0.7

All recorded ^{1}H and ^{13}C NMR were similar for **AP1a**, **1b**, **AP-2** and **AP-3**. ^{1}H NMR only differs by the integration of protons held by the fatty chain.

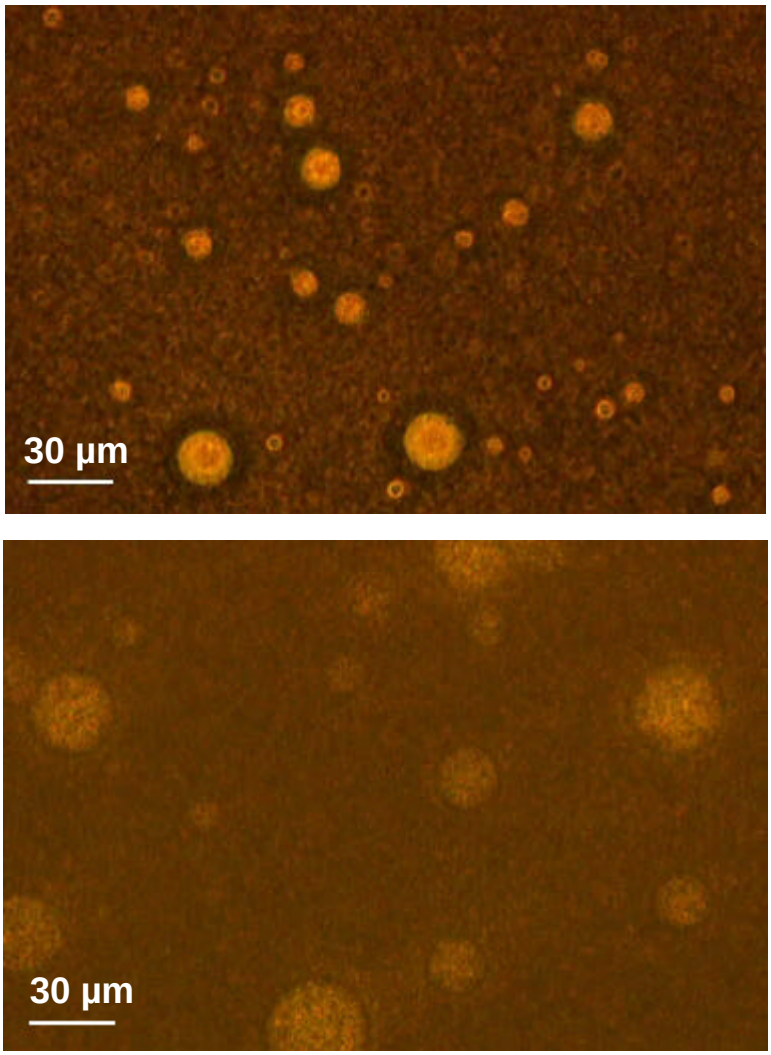
AP-1a: ^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 0.88 (t, 3H, $^{3}J_{H-H}$ = 6.6 Hz, $-CH_3$), 1.25 (m, 20H, $-CH_2$), 3.51 (broad peak, carbohydrate unit, -CHO-, $-CH_2O$), 3.65 (broad peak, carbohydrate unit, -CHO-, $-CH_2O$), 3.69 (broad peak, carbohydrate unit, -CHO-, $-CH_2O$); ^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 14.5 ($-CH_3$), 23.1 ($-CH_2$), 29.7-30.1 ($-CH_2$), 32.3 ($–CH_2$), 61.7 (broad peak, carbohydrate unit –CH-O-, $-CH_2O$), 70.8 (broad peak, carbohydrate unit, -CH-O-, $-CH_2O$), 72.9 (broad peak, carbohydrate unit, -CHO-, $-CH_2O$); IR (in KBr) ν cm^{-1} 3417 (OH), 2924 (CH, CH_2), 1601, 1456, 1354, 1246 cm^{-1}



^{13}C NMR of **AP-1a** recorded

AP-1b: ^{1}H NMR (300 MHz, 22°C, CD_3OD) δ (ppm) 0.76 (br, 3H, -CH_3), 1.15 (m, 20H, -CH_2), 3.45 (broad peak, carbohydrate unit, -CHO-, -CH_2O), 3.53 (broad peak, carbohydrate unit, -CHO-, -CH_2O), 3.72 (broad peak, carbohydrate unit, -CHO-, -CH_2O); ^{13}C (75 MHz, 22°C, MeOD) δ (ppm) 15.3 (-CH_3), 24.5-25.0 (-CH_2), 31.2-33.8 (-CH_2), 62.7 (broad peak, carbohydrate unit –CH-O-, -CH_2O), 71.45 (broad peak, carbohydrate unit, -CH-O-, -CH_2O), 74.03 (broad peak, carbohydrate unit, -CH-O-, -CH_2O); IR ν cm^{-1} 3424 (OH), 2924, 2854 (CH, CH_2), 1607, 1451, 1356, 1052 cm^{-1}

Additional digital images observed by phase contrast microscopy: **AP-1a** in glycerol (25g/L) with 2mL of 1,2-epoxydodecane in ethyl acetate (0.1M)



6) General procedure for the ring opening of epoxides with carboxylic acids (Table 1 and 2 in the article)

Typically, epoxide (1mmol) and carboxylic acid (1mmol) were mixed in 4mL of glycerol in the presence of 20mol% of supported amino groups (APs). The resulting mixture was then stirred

at the desired temperature (90°C or 110°C). For a better determination of the reaction yield, at the end of the reaction, water was added and reaction products were directly extracted with ethyl acetate (this technique does not concern the recycling experiment, see below). After evaporation under reduced pressure, the recovered residue was purified over flash silica gel chromatography using a mixture heptane/ethyl acetate (70/30) as eluent. Analytical data of products are provided below.

7) Recycling experiments (scheme 2 and 3 in the article)

In a typical procedure, 1 mmol of 1,2-epoxidodecane and 1 mmol of dodecanoic acid were mixed in 2mL of glycerol and stirred at 110°C in the presence of 20 mol% of **AP-1b**. At the end of the reaction, the crude mixture was centifugated for 5 minutes at 4000 rpm. The floated organic phase was directly and carefully removed with pipettes without assistance of any organic solvent and 1mmol of epoxidodecane and 1mmol of dodecanoic acid were reloaded. The recycling experiment was successfully repeated 10 times.

As mentioned in the manuscript, starting from **AP-1a,** we observed a decrease of the catalytic activity cycle after cycle mainly because of the partial solubilization of **AP-1a** in the product phase.

Table 1: recycling experiments starting from **AP-1a**

Run 1	Run 2	Run 3
85% yield	60 % yield	40% yield

8) General procedure for the base-catalyzed Knoevenagel reaction.

Typically, aldehyde (1mmol) and malononirile (1mmol) were mixed in 4mL of glycerol in the presence of 20mol% of supported amino groups (**AP-1b**). The resulting mixture was then stirred at 70°C. The reaction progress was monitored by GC. At the end of the reaction, water was added and reaction products were directly extracted with ethyl acetate. After evaporation under reduced pressure, the recovered residue was purified over flash silica gel chromatography using a mixture heptane/ethyl acetate (70/30) as eluent.

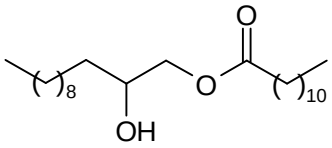
9) General procedure for the base-catalyzed Henry reaction.

Similar procedure than that described above for the Knoevenagel reaction was used. Reaction was performed from benzaldehyde (1mmol) and nitroethane (2mmol).

10) General procedure for the base-catalyzed Michael addition.

Similar procedure than that described above for the Knoevenagel reaction was used. Reaction was performed from 2-cyclohexen-1-one (1mmol) and nitroethane (1mmol).

11) Analytical part (whenever it was possible, analytical data of only one regioisomer was provided)



^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 0.88 (t, 6H, $^{3}J_{H-H}$ = 6.8 Hz, -CH_3), 1.26 (m, 32H, -CH_2), 1.47 (m, 2H, -CH_2), 1.63 (m, 2H, -CH_2), 2.34 (t, 2H, $^{3}J_{H-H}$ = 7.4 Hz, -CH_2-C=O), 3.83 (m, 1H, -CHOH), 3.96 (dd, 1H, $^{2}J_{H-H}$ = 7.5 Hz, $^{3}J_{H-H}$ = 10.9 Hz, -CHO-), 4.15 (dd, 1H, $^{2}J_{H-H}$ = 2.4 Hz, $^{3}J_{H-H}$ = 11.2 Hz, -CHO-).

^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 14.1 (-CH_3), 22.7 (-CH_2), 23.2 (-CH_2), 24.9 (-CH_2), 25,4 (-CH_2), 29.1-29.6 (-CH_2), 31.9 (-CH_2), 33.4 (-CH_2), 34.2 (-CH_2), 68.5 (-CH_2OH), 70.1 (-CHO-), 174.1 (-C=O).

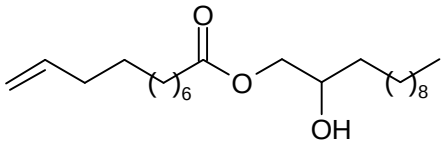
IR (neat) ν 1084, 1734 (C=O), 2849, 2915, 2954 (CH_2, CH_3), 3363 (O-H) cm-^{1}

Elemental analysis: calculated for $C_{24}H_{48}O_3$: %C 74.94%, %H 12.58. Measured: %C 75.39; %H 12.08

HRMS (ESI+): *m/z*: calculated for $C_{24}H_{48}O_3$: 385.3676 [*M*+H]; found: 385.3674 [*M*+H].

GC response factor: 0.6285 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).

Melting Point: 62.5 ± 2 °C



^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 0.88 (t, 3H, $^{3}J_{H-H}$ = 6.4 Hz, -CH_3), 1.26 (m, 26H, -CH_2), 1.47 (m, 2H, -CH_2), 1.81 (m, 2H, -CH_2), 2.04 (m, 3H, -CH_2, -OH), 2.34 (t, 2H, $^{3}J_{H-H}$ = 7.5 Hz, -CH_2-C=O), 3.83 (m, 1H, -CHOH), 3.96 (dd, 1H, $^{2}J_{H-H}$ = 7.2 Hz, $^{3}J_{H-H}$ = 11.1 Hz, -CHO-), 4.15 (dd, 1H, $^{2}J_{H-H}$ = 2.7 Hz, $^{3}J_{H-H}$ = 11.3 Hz, -CHO-), 4.94 (dd, 2H, $^{2}J_{H-H}$ = 10.5 Hz, $^{3}J_{H-H}$ = 11.3 Hz, -CH_2=); 5.77 (m, 1H, -CH=).

^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 14.1 (-CH_3), 22.7 (-CH_2), 23.2 (-CH_2), 24.9 (-CH_2), 25,4 (-CH_2), 28.9-29.6 (-CH_2), 31.2 (-CH_2), 33.4 (-CH_2), 33.8 (-CH_2), 34.2 (-CH_2), 68.5 (-CH_2OH), 70.0 (-CHO-), 114.2 (=CH_2), 139.1 (=CH-), 174.0 (-C=O).

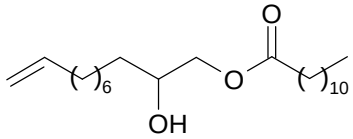
IR (neat) ν 1079, 1643 (C=C), 1735 (C=O), 2848, 2916, 2954 (CH_2, CH_3), 3320 (OH) cm-^{1}

Elemental analysis: calculated for $C_{23}H_{44}O_3$: %C 74.95, %H 12.03. Measured: %C 74.54; %H 11.96

HRMS (ESI+): *m/z*: calculated for $C_{23}H_{44}O_3$: 369.3363 [*M*+H]; found: 369.3370 [*M*+H].

GC response factor: 0.6759 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).

Melting Point: 55.6 ± 2 °C



^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 0.88 (t, 3H, $^3J_{H-H}$ = 6.7 Hz, -CH_3), 1.26 (m, 26H, -CH_2), 1.47 (m, 2H, -CH_2), 1.63 (m, 2H, -CH_2), 2.02 (m, 2H, $^3J_{H-H}$ = 6.9 Hz,-CH_2), 2.22 (br s, 1H, -OH), 2.32 (t, 2H, $^3J_{H-H}$ = 7.5 Hz, -CH_2-C=O), 3.82 (m, 1H, -CHOH), 3.95 (dd, 1H, $^2J_{H-H}$ = 7.2 Hz, $^3J_{H-H}$ = 11.2 Hz, -CHO-), 4.13 (dd, 1H, $^2J_{H-H}$ = 3.0 Hz, $^3J_{H-H}$ = 11.2 Hz, -CHO-), 4.94 (dd, 2H, J_{H-H} = 11.0 Hz, -CH_2=); 5.82 (m, 1H, -CH=).

^{13}C (75 MHz, 22°C, $CDCl_3$,) δ (ppm) 14.1 (-CH_3), 22.7 (-CH_2), 24.9 (-CH_2), 25.4 (-CH_2), 28.9-29.7 (-CH_2), 31.5 (-CH_2), 33.8 (-CH_2), 34.2 (-CH_2), 34.6 (-CH_2), 68.5 (-CH_2O-), 70.0 (-CHOH), 114.2 (=CH_2), 139.1 (=CH-), 174.1 (-C=O).

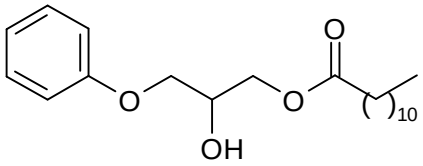
IR (neat) ν 1085, 1643 (C=C), 1731 (C=O), 2848, 2916, 2957 (CH_2, CH_3), 3320 (O-H) cm-^{1}

Elemental analysis: calculated for $C_{23}H_{44}O_3$: %C 74.95, %H 12.03. Measured: %C 75.21; %H 12.15

HRMS (ESI+): *m/z*: calculated for $C_{23}H_{44}O_3$: 369.3363 [*M*+H]; found: 369.3357 [*M*+H].

GC response factor: 0.6635 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).

Melting Point: 55.9 ± 2 °C



^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 0.88 (t, 3H, $^3J_{H-H}$ = 6.5 Hz, -CH_3), 1.25 (m, 16H, -CH_2), 1.62 (m, 2H, $^3J_{H-H}$ = 7.1 Hz, -CH_2), 2.32 (t, 2H, $^3J_{H-H}$ = 7.5 Hz, -CH_2-C=O), 4.01 (m, 1H, -CHOH), 4.25 (m, 4H, -CH_2O), 6.94 (m, 3H, Ph), 7.28 (m, 2H, Ph)

^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 14.1 (-CH_3), 22.7 (-CH_2), 24.7 (-CH_2), 24.9 (-CH_2), 28.1-29.9 (-CH_2), 31.9 (-CH_2), 34.3 (-CH_2), 65.2 (-CH_2OH), 68.6 (-CHO-), 114.5 (=CH-, Ph), 121.4 (=CH-, Ph), 129.5 (=CH-, Ph), 129.9 (=CH-, Ph),158.3 (=CH-, Ph),173.8 (-C=O)

IR (neat) ν 1083, 1175, 1241, 1500, 1600, 1723 (C=O), 2849, 2916, 2942 (CH_2, CH_3), 3485 (OH) cm-^{1}

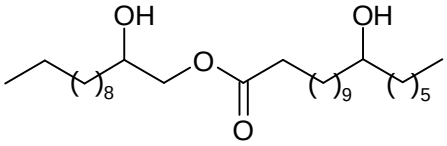
Elemental analysis: calculated for $C_{21}H_{34}O_4$: %C 71.96, %H 9.78. Measured: %C 71.66; %H

9.45

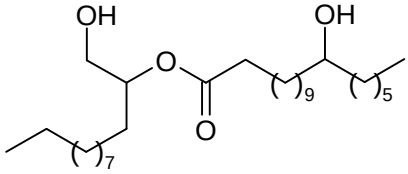
HRMS (ESI+): *m/z*: calculated for $C_{21}H_{34}O_4$: 368.2795 [*M*+NH_4]; found: 368.2791 [*M*+NH_4].

GC response factor: 0.7645 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).

Melting Point: 43.7 ± 2 °C



regioisomer 1



regioisomer 2

^{1}H NMR (300 MHz, 22°C, $CDCl_3$, ***mixture of regioisomers***) δ (ppm) 0.88 (t, 6H, $^3J_{H-H}$ = 6.5 Hz, -CH_3), 1.26 (m, 36H, -CH_2), 1.46 (m, 4H, -CH_2), 1.62 (m, 4H, -CH_2), 2.35 (t, 2H, $^3J_{H-H}$ = 7.5 Hz, -CH_2-C=O), 3.61 (m, -CH_2O-regioisomer 2), 3.82 (m, -CHOH, regioisomer 1), 3.95 (dd, $^2J_{H-H}$ = 7.3 Hz, $^3J_{H-H}$ = 11.2 Hz, -CHO-, regioisomer 1), 4.14 (dd, $^2J_{H-H}$ = 2.9 Hz, $^3J_{H-H}$ = 11.2 Hz, -CHO-, regioisomer 1), 4.90 (m, $^3J_{H-H}$ = 3.2 Hz, -CHOH, regioisomer 2)

^{13}C (75 MHz, 22°C, $CDCl_3$, ***mixture of regioisomers***) δ (ppm) 14.1 (-CH_3), 22.6 (-CH_2), 25.1 (-CH_2), 25.4 (-CH_2), 25.6 (-CH_2), 29.1-29.7 (-CH_2), 31.8 (-CH_2), 33.4 (-CH_2), 34.2 (-CH_2), 37.5 (-CH_2), 64.9 (-CH_2OH, regioisomer 2), 68.6 (-CH_2O, regiosomer 1), 70.0 (-CHOH, regiosomer 1) 72.0 (-CHOH, regiosomer 1) 75.5 (-CHO-, regiosomer 2), 174.1 (-C=O, regiosomer 1), 174.4 (-C=O, regiosomer 2).

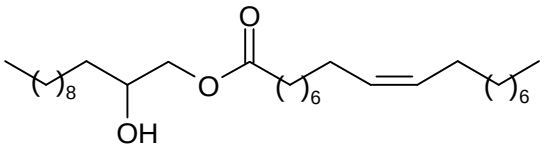
IR (neat) ν 1076, 1180, 1471, 1715 (C=O), 2849, 2915, 2953 (CH_2, CH_3), 3351, 3505 (OH) cm-^{1}

Elemental analysis: calculated for $C_{30}H_{60}O_4$: %C 74.32, %H 12.47. Measured: %C 74.16; %H 12.86

HRMS (ESI+): *m/z*: calculated for $C_{30}H_{60}O_4$: 485.4564 [*M*+H]; found: 485.4559 [*M*+H].

GC response factor: 0.4922 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).

Melting Point: 74.5 ± 2 °C



^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 0.88 (t, 6H, $^3J_{H-H}$ = 6.5 Hz, -CH_3), 1.26 (m, 38H, -CH_2), 1.47 (m, 2H, -CH_2), 1.63 (m, 2H, -CH_2), 2.02 (m, 3H, -CH_2, -OH), 2.29 (t, 2H, $^3J_{H-H}$ = 7.5 Hz, -CH_2-C=O), 3.82 (m, 1H, -CHOH), 3.92 (dd, 1H, $^2J_{H-H}$ = 7.2 Hz, -CHO-), 4.13 (dd, 1H,

$^{3}J_{H\text{-}H}$ = 11.2 Hz, -CHO-), 5.34 (m, 2H, -CH=).

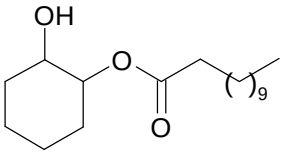
^{13}C (75 MHz, 22°C, $CDCl_3$,) δ (ppm) 14.5 ($-CH_3$), 22.9 ($-CH_2$), 25.8 ($-CH_2$), 26.0 ($-CH_2$), 27.6 ($-CH_2$), 29.4-30.2 ($-CH_2$), 32.3 ($-CH_2$), 33.7 ($-CH_2$), 34.2 ($-CH_2$), 34.6 ($-CH_2$), 68.9 ($-CH_2O-$), 70.4 (-CHOH), 130.2 (=CH-), 130.4 (=CH-), 174.5 (-C=O).

IR (neat) ν 1084, 1177, 1203, 1467, 1723 (C=O), 1655 (C=C), 2853, 2921 (CH_2, CH_3), 3461 (O-H) cm-^{1}

Elemental analysis: calculated for $C_{30}H_{58}O_3$: %C 77.19, %H 12.52. Measured: %C 77.57; %H 12.96

GC response factor: 0.7707 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).

Melting Point: Gum like compound at 25 °C



^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 0.90 (t, 3H, $^{3}J_{H\text{-}H}$ = 6.6 Hz, $-CH_3$), 1.26 (m, 20H, $-CH_2$), 1.65 (m, 5H, $-CH_2$ and -OH), 2.03 (m, 2H, $-CH_2$), 2.30 (t, 2H, $^{3}J_{H\text{-}H}$ = 7.5 Hz, $-CH_2$-C=O), 3.56 (m, 1H, -CHOH-), 4.58 (m, 1H, -CHO-).

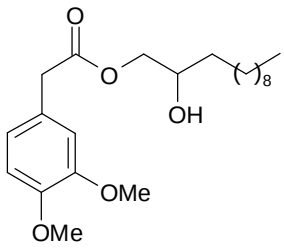
^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 14.1 ($-CH_3$), 22.6 ($-CH_2$), 23.7-25.0 ($-CH_2$), 25.7 ($-CH_2$), 29.1-31.9 ($-CH_2$), 32.8 ($-CH_2$), 33.0 ($-CH_2$), 34.6 ($-CH_2$), 72.8 (-CHO-), 78.0 (-CHOH), 174.2 (-C=O).

IR (neat) ν 1075, 1179, 1455, 1734 (C=O), 2854, 2923 (CH_2, CH_3), 3461 (OH) cm-^{1}

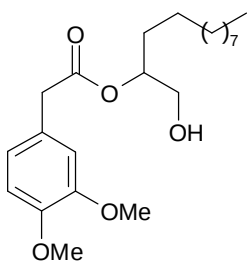
Elemental analysis: calculated for $C_{18}H_{34}O_3$: %C 72.44, %H 11.48. Measured: %C 72.03; %H 11.26

HRMS (ESI+): *m/z*: calculated for $C_{18}H_{34}O_3$: 299.2581 [*M*+H]; found: 299.2582 [*M*+H].

GC response factor: 1.3564 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).



regioisomer 1



regioisomer 2

^{1}H NMR (300 MHz, 22°C, $CDCl_3$, ***mixture of regioisomers***) δ (ppm) 0.88 (t, 3H, $^{3}J_{H\text{-}H}$ = 6.4

Hz, -CH_3), 1.25 (m, 15H, -CH_2), 1.43 (m, 4H, -CH_2), 2.02 (s, 2H, -CH_2-C=O), 3.59 (m, -CHO-, regioisomer 2), 3.60 (s, 3H, -OCH_3), 3.70 (dd, -CHO-, $^2J_{H-H}$ = 3.2 Hz, $^3J_{H-H}$ = 11.9 Hz, regiosiomer 2), 3.87 (m, -OCH_3, -CHOH regioisomer 1), 3.94 (dd, 1H, -CHO-, $^2J_{H-H}$ = 7.2 Hz, $^3J_{H-H}$ = 11.3 Hz, regiosiomer 1), 4.11 (dd, 1H, -CHO-, $^2J_{H-H}$ = 2.9 Hz, $^3J_{H-H}$ = 11.3 Hz, regiosiomer 1), 4.94 (m, 1H, -CHOH, regioisomer 2), 6.81 (m, 3H, Ph).

^{13}C (75 MHz, 22°C, $CDCl_3$, ***mixture of regioisomers***) δ (ppm) 13.9 (-CH_3), 22.5 (-CH_2), 25.0 (-CH_2), 29.1-29.4 (-CH_2), 30.2 (-CH_2, regioisomer 2), 31.7 (-CH_2), 33.1 (-CH_2), 40.9 (-CH_2), 41.0 (-CH_2, regioisomer 2), 55.6 (-OCH_3), 64.5 (CHOH, regioisomer 2), 68.8 (CHOH, regioisomer 1), 69.7 (-CHO-, regioisomer 1), 75.8 (-CHO-, regioisomer 2), 111.0 (-CH, Ph), 112.1 (-CH, Ph), 121.1 (-CH, Ph), 126.3 (-CH, Ph), 148.0 (-CH, Ph), 148.8 (-CH, Ph), 171.8 (-C=O, regiosomer 1), 172.1 (-C=O, regiosomer 2).

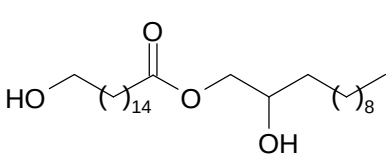
IR (neat) ν 1081, 1027, 1139, 1156, 1230, 1258, 1511, 1709 (C=O), 2850, 2915 (CH_2, CH_3), 3007 (Aromatic =C-H), 3488 (OH) cm^{-1}

Elemental analysis: calculated for $C_{22}H_{36}O_5$: %C 69.44, %H 9.54. Measured: %C 69.22; %H 9.51

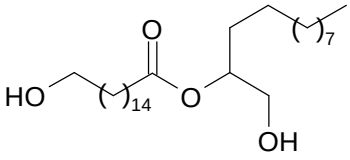
HRMS (ESI+): *m/z*: calculated for $C_{22}H_{36}O_5$: 403.2455 [*M*+Na]; found: 403.2459 [*M*+Na].

GC response factor: 0.9458 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).

Melting Point: 54.5 ± 2 °C



regioisomer 1



regioisomer 2

^{1}H NMR (300 MHz, 22°C, $CDCl_3$, ***mixture of regioisomers***) δ (ppm) 0.88 (t, 3H, $^3J_{H-H}$ = 6.5 Hz, -CH_3), 1.25 (m, 34H, -CH_2), 1.46 (m, 2H, -CH_2), 1.60 (m, 4H, -CH_2), 2.04 (m, 2H, -CH_2), 2.31 (t, 2H, $^3J_{H-H}$ = 7.5 Hz, -CH_2-C=O), 3.63 (t, 2H, $^3J_{H-H}$ = 6.6 Hz, -CH_2OH), 3.69 (m, CH_2O-, regioisomer 2), 3.83 (m, -CHOH, regioisomer 1), 3.96 (dd, -CHO-, $^2J_{H-H}$ = 7.3 Hz, $^3J_{H-H}$ = 11.3 Hz, regiosiomer 1), 4.13 (dd, -CHO-, $^2J_{H-H}$ = 2.9 Hz, $^3J_{H-H}$ = 11.3 Hz, regiosiomer 1), 4.91 (m, -CHOH, regioisomer 2).

^{13}C (75 MHz, 22°C, $CDCl_3$, ***mixture of regioisomers***) δ (ppm) 14.1 (-CH_3), 22.7 (-CH_2), 24.9 (-CH_2), 25.3 (-CH_2), 25.7 (-CH_2), 29.1-29.6 (-CH_2), 31.9 (-CH_2), 32.8 (-CH_2), 33.4 (-CH_2), 33.8 (-CH_2), 34.6 (-CH_2), 63.1 (-CH_2OH), 64.9 (-CH_2O, regiosomer 2), 68.6 (-CH_2O-, regiosomer 1) 70.0 (-CHOH, regiosomer 1) 75.5 (-CHOH, regiosomer 2), 174.1 (-C=O, regiosomer 1), 172.4 (-C=O, regiosomer 2).

IR (neat) ν 1045, 1059, 1200, 1471, 1699 (C=O), 2848, 2913, 2952 (CH_2, CH_3), 3278, 3503

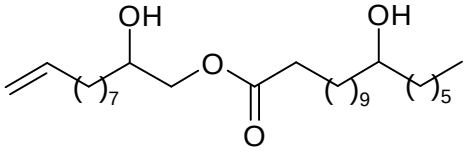
(OH) cm^{-1}

Elemental analysis: calculated for $C_{28}H_{56}O_4$: %C 73.63, %H 12.36. Measured: %C 73.37; %H 12.23

HRMS (ESI+): *m/z*: calculated for $C_{28}H_{56}O_4$: 457.4251 [*M*+H]; found: 457.4246 [*M*+H].

GC response factor: 0.8417 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).

Melting Point: 69.5 ± 2 °C



^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 0.88 (t, 3H, $^3J_{H-H}$ = 6.8 Hz, -CH_3), 1.27 (m, 38H, -CH_2), 1.65 (m, 2H, $^3J_{H-H}$ = 6.8 Hz, -CH_2), 2.02 (m, 2H, -CH_2), 2.34 (t, 2H, $^3J_{H-H}$ = 7.5 Hz, -CH_2-C=O), 3.57 (m, 1H, -CHOH), 3.81 (m, 1H, -CHOH), 3.95 (dd, 1H, $^2J_{H-H}$ = 7.2 Hz, $^3J_{H-H}$ = 11.2 Hz, -CHO-), 4.13 (dd, 1H, $^2J_{H-H}$ = 3.0 Hz, $^3J_{H-H}$ = 11.3 Hz, -CHO-), 4.94 (dd, 2H, J_{H-H} = 11.2 Hz, -CH_2=); 5.81 (m, 1H, -CH=).

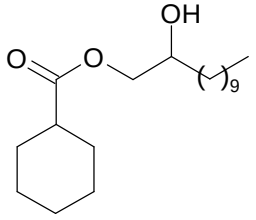
^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 14.5 (-CH_3), 23.0 (-CH_2), 25.3 (-CH_2), 25.7-26.0 (-CH_2), 29.2-30.1 (-CH_2), 30.9 (-CH_2), 32.2-37.8 (-CH_2), 37.9 (-CH_2), 68.9 (-CH_2O-), 70.3 (-CHOH), 72.3 (-CHOH), 114.6 (=CH_2), 139.4 (-CH=), 174.4 (-C=O).

IR (neat) ν 1079, 1464, 1643 (C=C), 1735 (C=O), 2849, 2915 (CH_2, CH_3), 3365 (OH) cm^{-1}

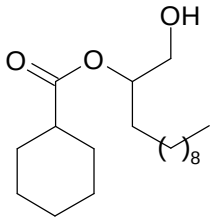
Elemental analysis: calculated for $C_{29}H_{56}O_4$: %C 74.31, %H 12.04. Measured: %C 74.14; %H 11.87

GC response factor: 0.5876 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).

Melting Point: 65.3 ± 2 °C



regioisomer 1



regioisomer 2

^{1}H NMR (300 MHz, 22°C, $CDCl_3$, ***mixture of regioisomers***) δ (ppm) 0.88 (t, 3H, $^3J_{H-H}$ = 6.4 Hz, -CH_3), 1.25 (m, 22H, -CH_2), 1.47 (m, 2H, -CH_2), 1.77 (m, 2H, -CH_2), 2.04 (m, 2H, -CH_2), 2.38 (m, -CH_2-C=O, regioisomer 1 +2), 3.61 (dd, -CHO-, $^2J_{H-H}$ = 6.2 Hz, $^3J_{H-H}$ = 11.9 Hz, regiosiomer 2), 3.69 (dd, -CHO-, $^2J_{H-H}$ = 3.2 Hz, $^3J_{H-H}$ = 11.9 Hz, regiosiomer 2), 3.82 (m, -

CHOH, regioisomer 1), 3.95 (dd, -CHO-, $^{2}J_{H-H}$ = 7.2 Hz, $^{3}J_{H-H}$ = 11.2 Hz, regiosiomer 1), 4.13 (dd, -CHO-, $^{2}J_{H-H}$ = 2.9 Hz, $^{3}J_{H-H}$ = 11.3 Hz, regiosiomer 1), 4.98 (m, -CHOH, regioisomer 2)

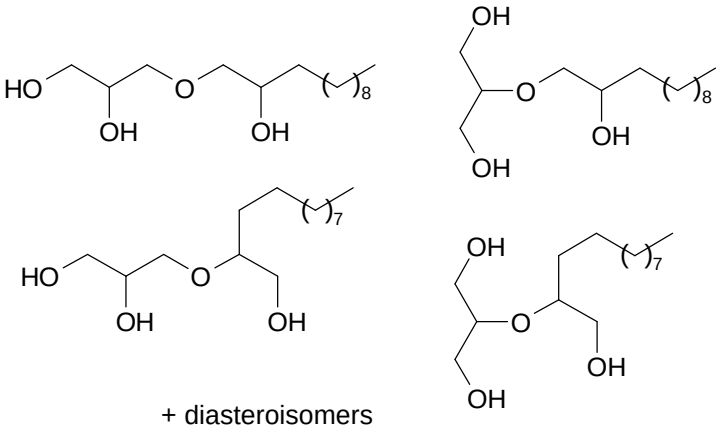
^{13}C (75 MHz, 22°C, $CDCl_3$, ***mixture of regioisomers***) δ (ppm) 14.1 ($-CH_3$), 22.7 ($-CH_2$), 25.3 ($-CH_2$), 29.0-29.6 ($-CH_2$), 31.9 ($-CH_2$), 33.4 ($-CH_2$), 43.2 ($-CH_2$), 65.1 ($-CH_2OH$, regioisomer 2), 68.4 ($-CH_2O$-, regioisomer 1), 70.1 (-CHOH, regioisomer 1), 75.2 (-CHO-, regiosomer 2), 176.3 (-C=O, regiosomer 1), 176.6 (-C=O, regiosomer 2).

IR (neat) ν 1170, 1452, 1464 (CH_2), 1733 (C=O), 2854, 2924 (CH_2, CH_3), 3460 (OH) cm^{-1}

Elemental analysis: calculated for $C_{19}H_{36}O_3$: %C 73.03, %H 11.61. Measured: %C 72.75; %H 11.51

HRMS (ESI+): *m/z*: calculated for $C_{19}H_{36}O_3$: 313.2737 [*M*+H]; found: 313.2734 [*M*+H].

GC response factor: 0.7479 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).



^{1}H NMR (300 MHz, 22°C, $CDCl_3$, ***mixture of regioisomers and diastereosisomers***) δ (ppm) 0.87 (t, 3H, CH_3, $^{3}J_{HH}$=6.3), 1.26 (m, CH_2), 1.39 (m, CH_2), 3.33 (m, CH_2), 3.56 (m, $2CH_2O$), 3.78 (m, CHOH), 3.89 (m, CHOH), 4.28 (m, OH).

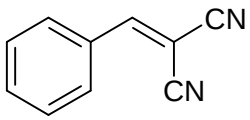
^{13}C (75 MHz, 22°C, $CDCl_3$, ***mixture of regioisomers and diastereosisomers***) δ (ppm) 14.5 (CH_3), 23.1 (CH_2), 26.2 (CH_2), 29.5-30.1 (CH_2), 32.3 (CH_2), 33.5 (CH_2), 61.4, (CH-O), 61.9 (CH-O), 63.7 (CH_2-O), 63.8 (CH_2-O), 70.3 (CH_2-O), 70.4 (CH_2-O), 70.6 (CH-O), 70.9 (CH_2-O), 71.2 (CH_2-O), 72.5 (CH_2-O), 72.7 (CH_2-O), 74.3 (CH_2-O), 75.9 (CH-O), 76.1 (CH-O), 80.9 (CH-O).

IR (neat) ν , 1037, 1051, 1063, 1081, 1118, 1130 (C-O), 2852, 2921, 1480 (CH_2), 2880, 2951, 1480 (CH_3), 3310, 3395 (O-H) cm^{-1}

Elemental analysis: Calculated for $C_{15}H_{32}O_4$: %C 65.18, %H 11.67. Measured: %C 65.66; %H 12.06.

GC response factor: 0.7097 with dodecane as internal standard (BPX5 column 12m x 0.22 mm; phase thickness: 0.25 µm).

Melting point: Gum-like compound at 25 °C



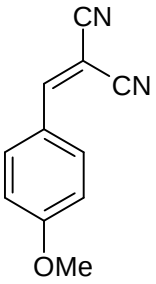
^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 7.54 (m, 2H, $^{3}J_{H\text{-}H}$ = 7.5 Hz, =CH-), 7.63 (m, 1H, $^{3}J_{H\text{-}H}$ = 7.3 Hz, =CH-), 7.78 (s, 1H, -CH=C(CN)$_2$), 7.91 (d, 2H, $^{3}J_{H\text{-}H}$ = 7.5 Hz, -CH=).

^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 82.8 (-C(CN)$_2$), 112.3 (-CN), 113.7 (-CN), 128.7 (C, Ph), 129.1 (=CH, Ph), 130.9 (=CH, Ph), 135.0 (=CH, Ph), 159.6 (-CH=C(CN)$_2$).

IR (neat) ν 958, 1450, 1567, 1589, 2223 (C=C-CN), 2854, 2925 (-CH-), 3033 (Vinyl -CH=CH-) cm^{-1}

Elemental analysis: calculated for $C_{10}H_6N_2$: %C 77.91, %H 3.92, %N 18.17. Measured: %C 77.95; %H 3.85, %N 18.20.

Melting Point: 79.8 ± 2 °C



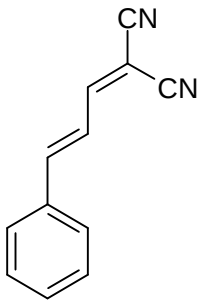
^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 3.85 (s, 3H, -OCH_3), 6.95 (d, 2H, $^{3}J_{H\text{-}H}$ = 7.3 Hz, =CH-), 7.58 (s, 1H, -CH=C(CN)$_2$), 7.86 (d, 2H, $^{3}J_{H\text{-}H}$ = 7.5 Hz, -CH=).

^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 54.8 (-OCH_3), 77.5 (=C(CN)$_2$), 112.3 (-CN), 113.1 (-CN), 123.0 (C, Ph), 132.4 (=CH, Ph), 157.9 (=CH-C(CN)$_2$), 163.8 (CH_3O-C=, Ph).

IR (neat) ν 831, 1019, 1179, 1276, 1511, 1557, 1568, 1604 (Aromatic ring C=C), 2220 (C=N) 2853, 2919 (CH), 3030 (vinyl C=C) cm^{-1} ,

Elemental analysis: calculated for $C_{11}H_8N_2O$: %C 71.73, %H 4.38, %N 15.21. Measured: %C 71.95; %H 4.47, %N 15.04.

Melting Point: 105.4 ± 2 °C



^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 7.27 (d, 2H, *J*= 8.6 Hz), 7.48-4.42 (m, 3H), 7.61-

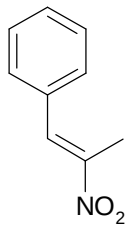
7.59 (m, 3H).

^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 83.1 (=$C(CN)_2$), 111.7 (-CN), 113.5 (-CN), 122.3 (-CH=), 128.9 (=CH, Ph), 129.3 (=CH, Ph), 132.1 (=CH, Ph), 133.9 (C, Ph), 150.5 (-CH=), 160.0 (=CH-$C(CN)_2$).

IR (neat) ν 977, 1178, 1560, 1576, 1608 (Aromatic C=C), 2224 (C=C-CN), 2853, 2925 (-CH-), 3032, 3054 (Vinyl -CH=CH-) cm^{-1}

Elemental analysis: calculated for $C_{12}H_8N_2$: %C 79.98, %H 4.47, %N 15.55. Measured: %C 79.44; %H 4.80, %N 15.60.

Melting Point: 125.8 ± 2 °C

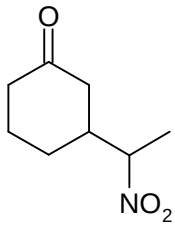


^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 2.45 (s, 3H, CH_3), 7.44 (m, 5H, =CH-), 8.02 (s, 1H, -CH=C-)

^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 14.0 (CH_3), 127.9 (=CH, Ph), 129.9 (=CH, Ph), 132.4 (C, Ph), 133.5 (=CH), 147.8 (=$C(NO_2)$).

IR (neat) ν 979, 1214, 1313, 1506, 1651, 2871, 2947 (CH), 3058 (=CH)cm^{-1}

Elemental analysis: calculated for $C_9H_9NO_2$: %C 66.25, %H 5.56, %N 8.58. Measured: %C 66.03; %H 5.82, %N 8.22.



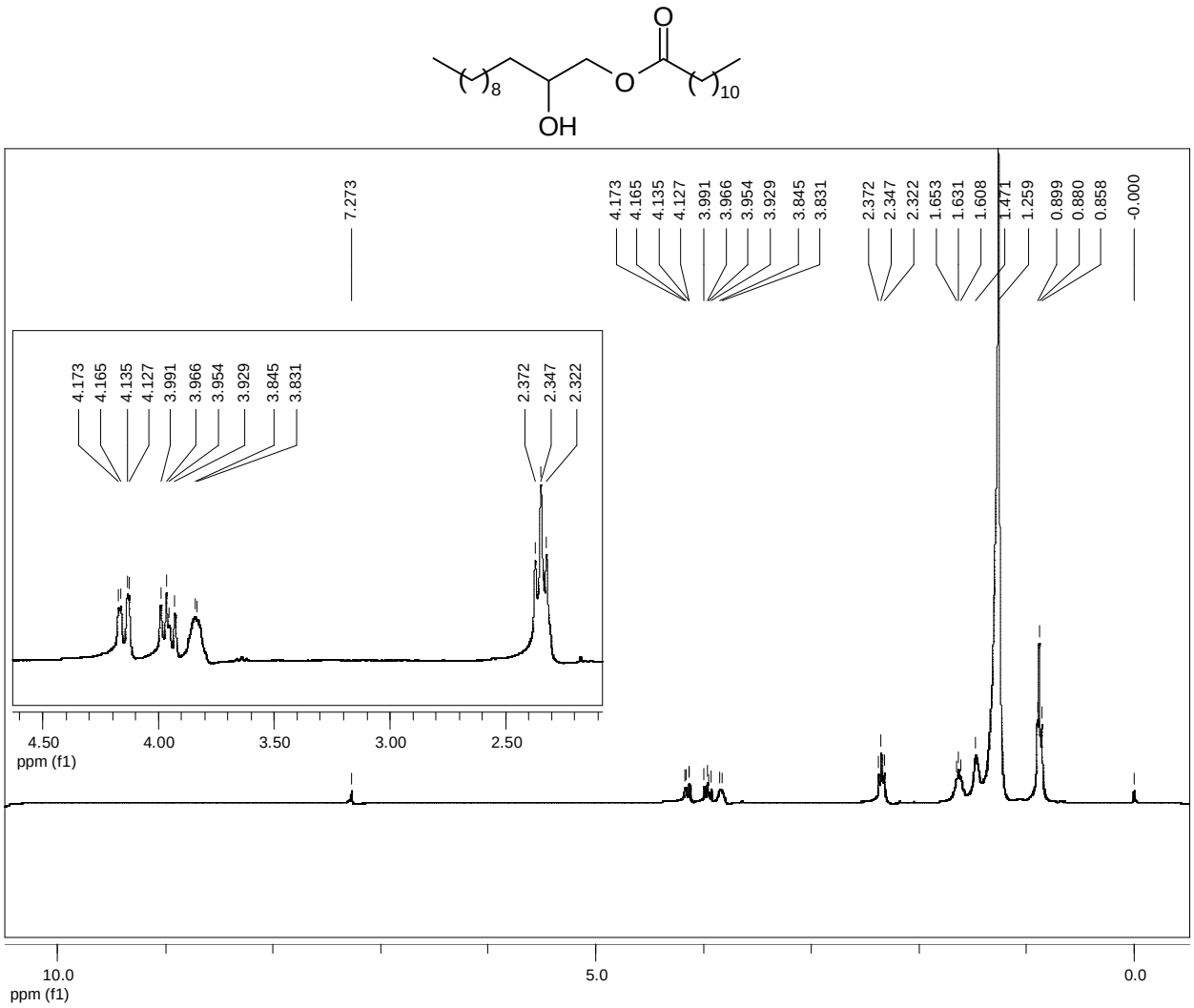
mixture of diastereoisomers

^{1}H NMR (300 MHz, 22°C, $CDCl_3$) δ (ppm) 1.55 (dd, 3H, -CH_3, $^2J_{H-H}$ = 6.8 Hz, $^3J_{H-H}$ = 7.6 Hz), 1.40-2.44 (m, 9H, -CH_2, -CH), 4.5 (m, 1H, -CH).

^{13}C (75 MHz, 22°C, $CDCl_3$) δ (ppm) 16.5 (-CH3), 24.6 (-CH2), 27.3 (-CH_2, diastereoisomer 1), 27.9 (-CH_2, diastereoisomer 2), 41.3 (-CH_2), 42.7 (-CH_2, diastereoisomer 1), 42.9 (-CH_2, diastereoisomer 2), 43.7 (-CH_2, diastereoisomer 1), 44.0 (-CH_2, diastereoisomer 2), 87.4 (-CH-NO_2), 208.9 (C=O, diastereoisomer 1), 209.1 (C=O, diastereoisomer 2).

IR (neat) ν 1391, 1542 (N-O), 1711 (C=O), 2871, 2947 (CH, CH_2, CH_3) cm^{-1} ,

Elemental analysis: calculated for $C_8H_{13}NO_3$: %C 56.13, %H 7.65, %N 8.18. Measured: %C 56.03; %H 7.82, %N 8.32.


O
8
O
10
OH
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4.173
4.165
4.135
4.127
3.991
3.966
3.954
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3.831
2.372
2.347
2.322
1.653
1.631
1.608
1.471
1.259
0.899
0.880
0.858
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3.50
3.00
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ppm (f1)
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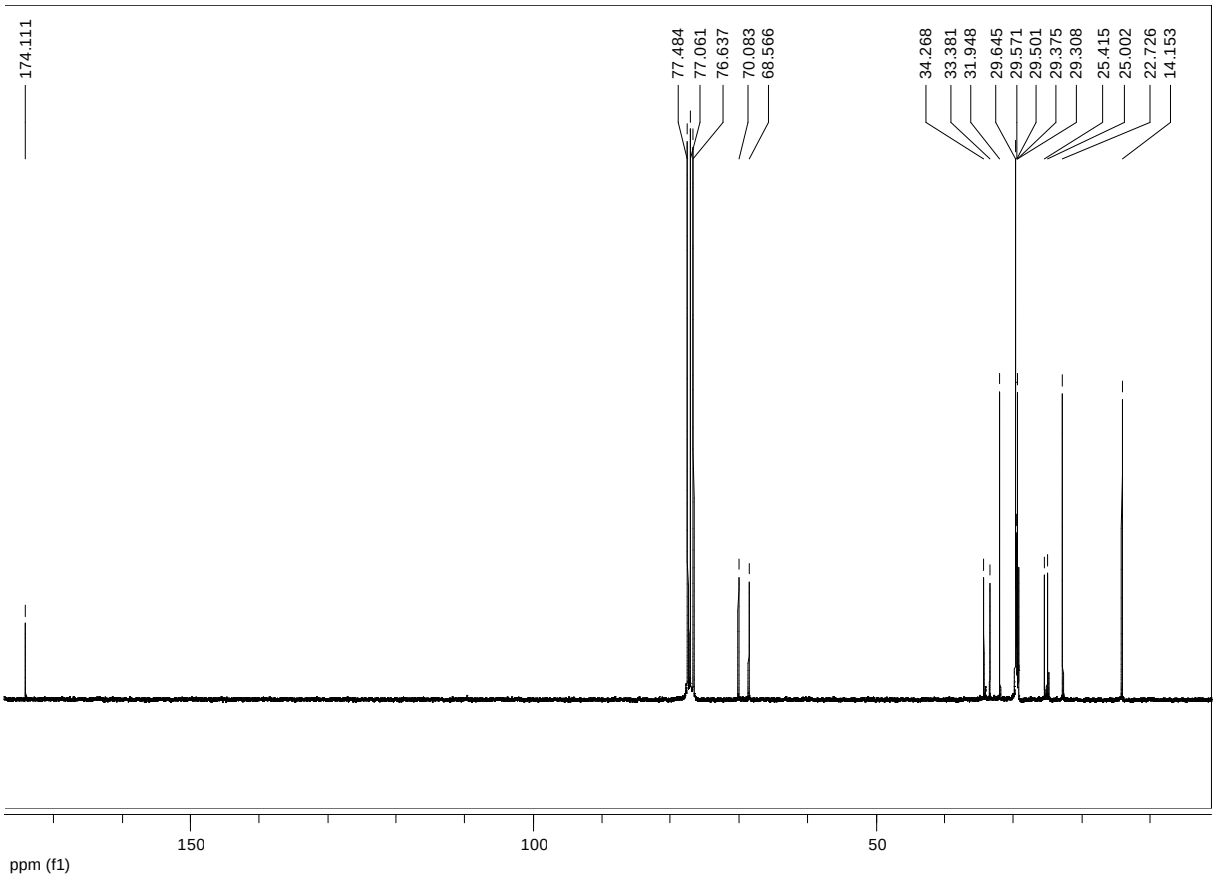
5.0
0.0
ppm (f1)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68.566
34.268
33.381
31.948
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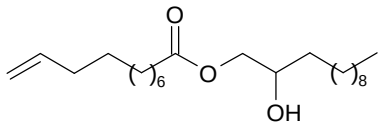
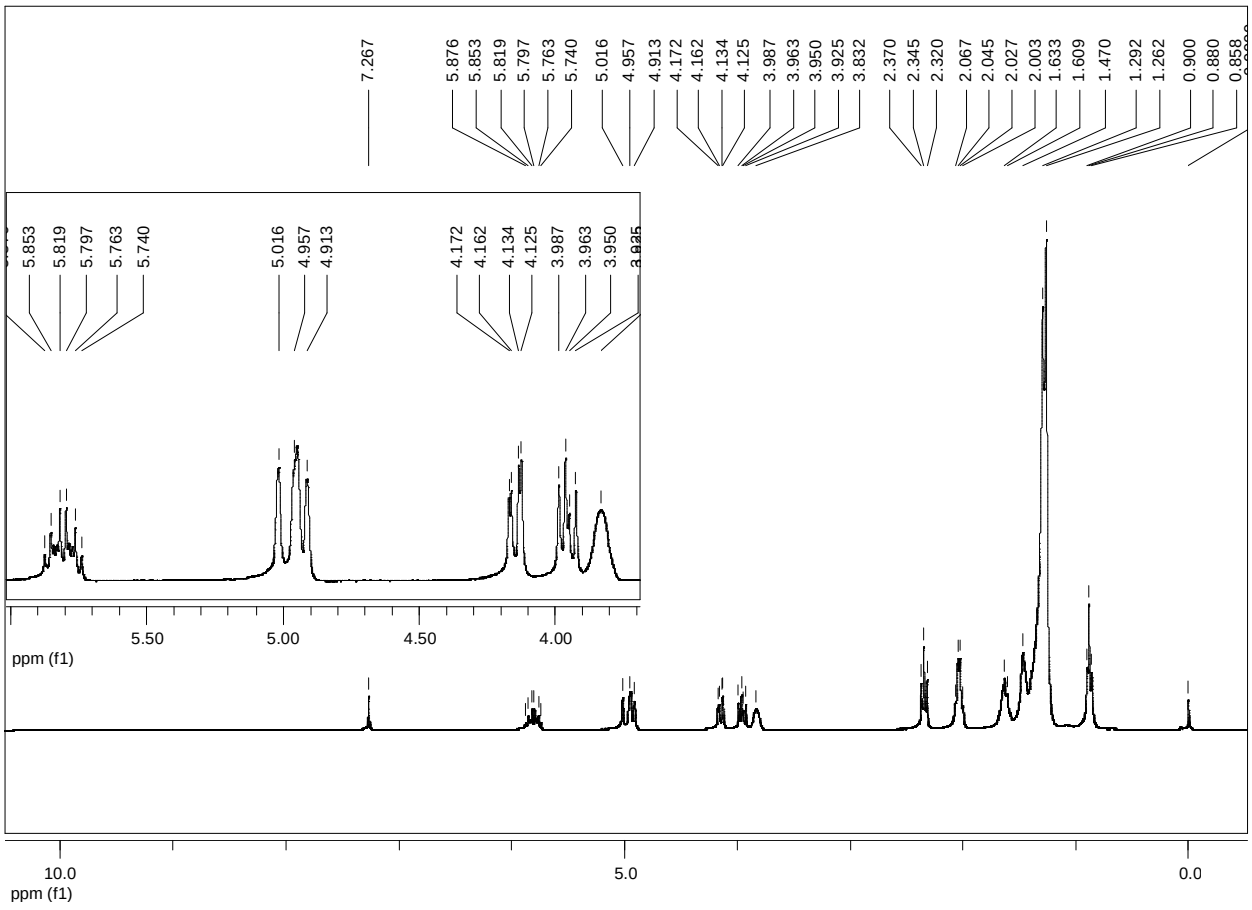
29.375
29.308
25.415
25.002
22.726
14.153
150
100
50
ppm (f1)


O
O
6
8
OH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5.819
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5.763
5.740
5.016
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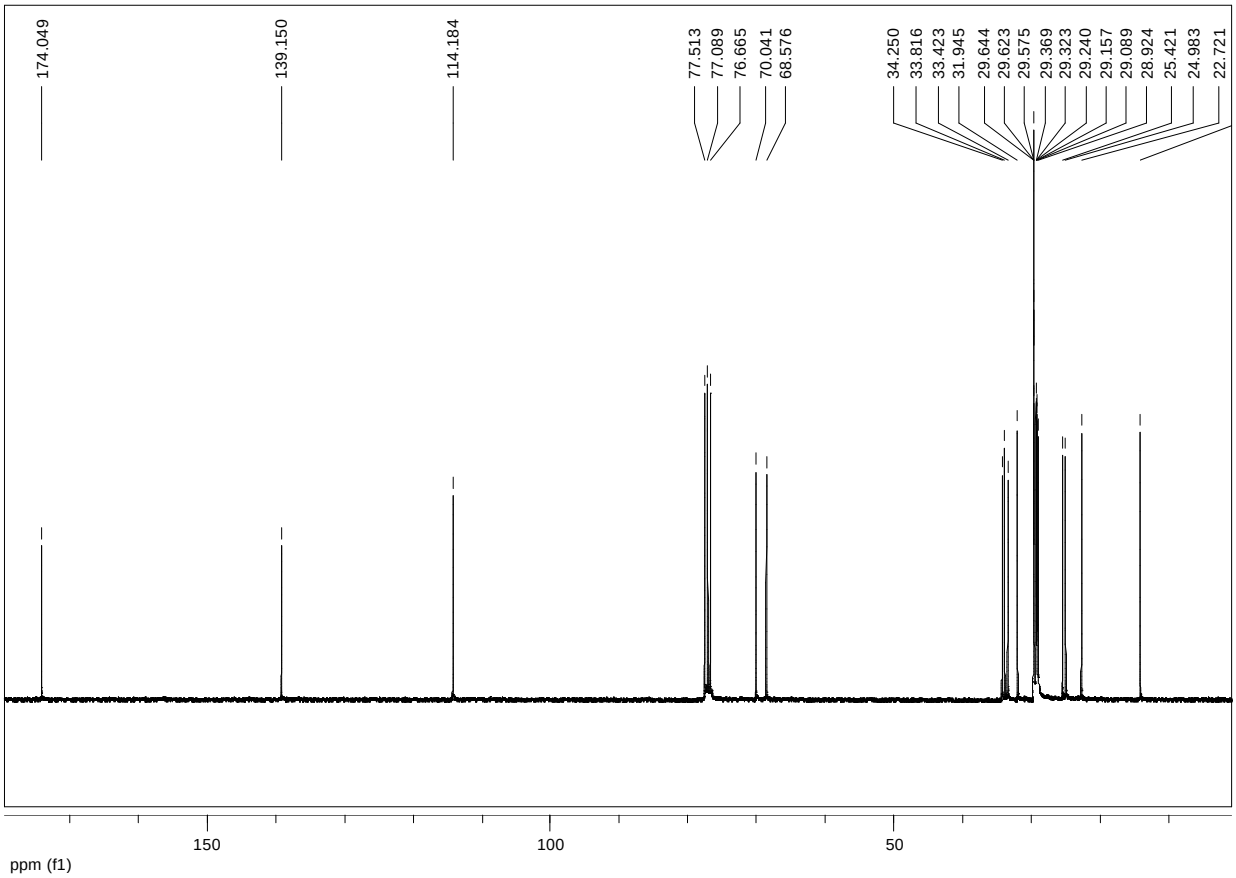
4.172
4.162
4.134
4.125
3.987
3.963
3.950
3.925
3.832
2.370
2.345
2.320
2.067
2.045
2.027
2.003
1.633
1.609
1.470
1.292
1.262
0.900
0.880
0.858
5.50
5.00
4.50
4.00
ppm (f1)
10.0
5.0
0.0
ppm (f1)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68.576
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33.816
33.423
31.945
29.644
29.623
29.575
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28.924
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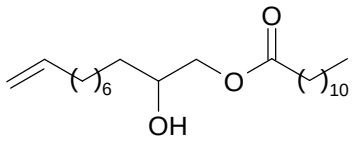
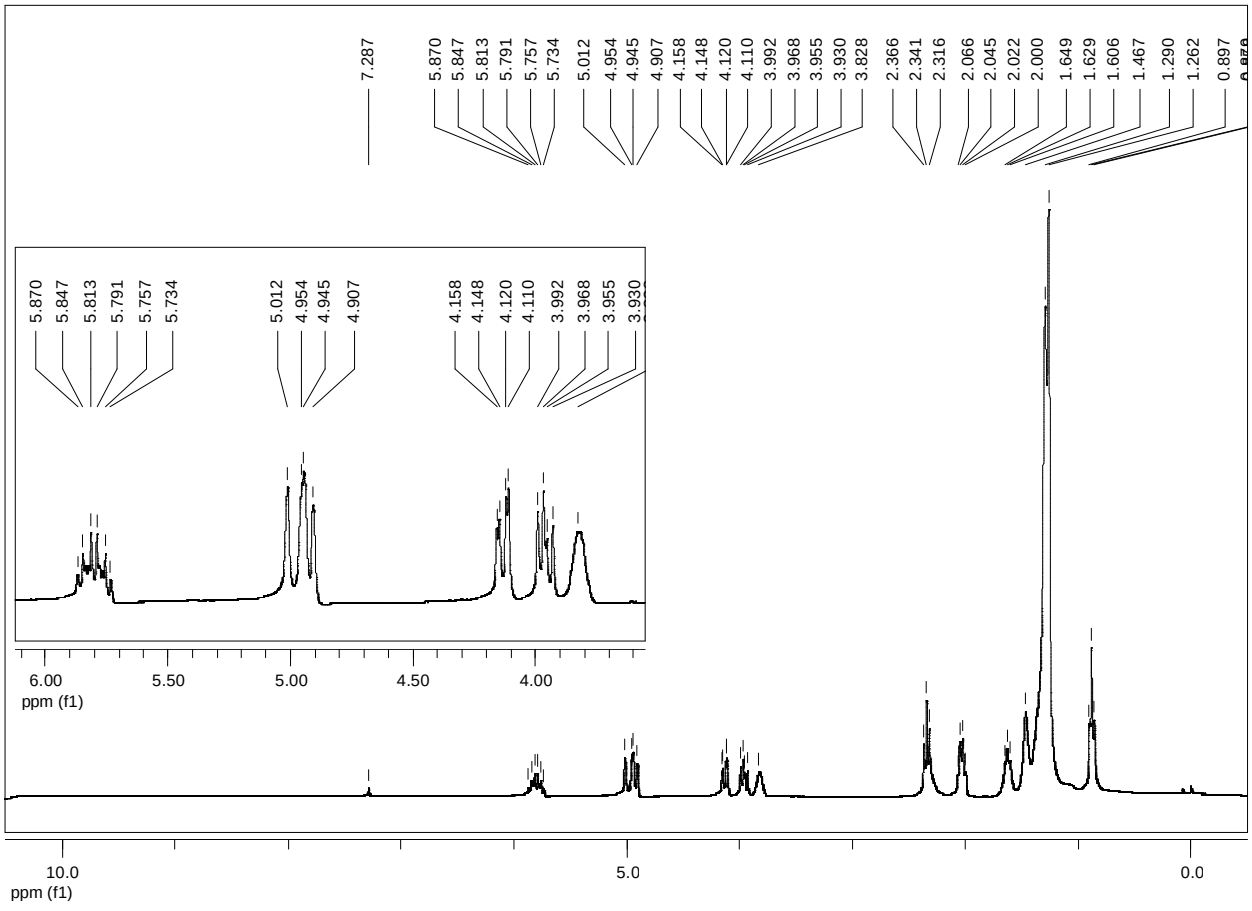
24.983
22.721
150
100
50
ppm (f1)


O
6
O
10
OH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5.734
5.012
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4.945
4.907
4.158
4.148
4.120
4.110
3.992
3.968
3.955
3.930
3.828
2.366
2.341
2.316
2.066
2.045
2.022
2.000
1.649
1.629
1.606
1.467
1.290
1.262
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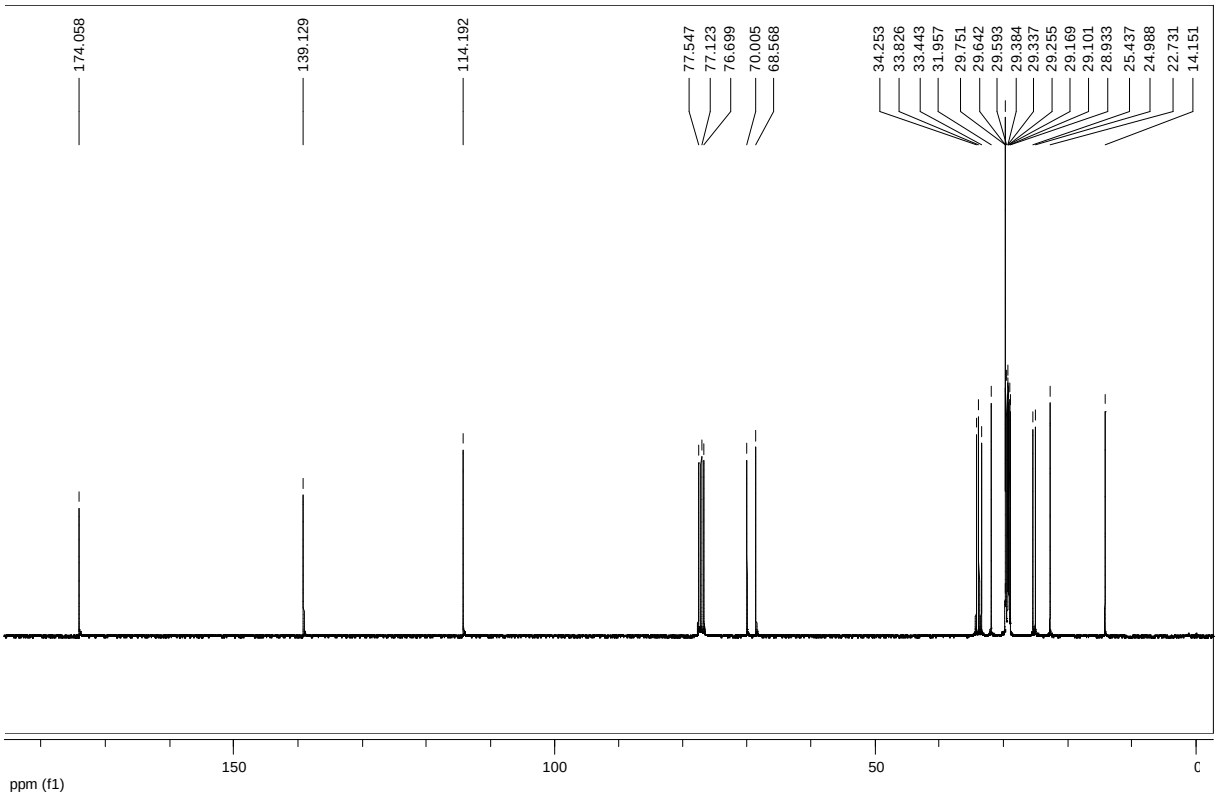
5.50
5.00
4.50
4.00
ppm (f1)
10.0
5.0
0.0
ppm (f1)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29.751
29.642
29.593
29.384
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29.101
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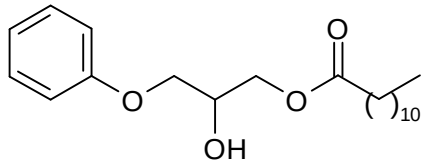
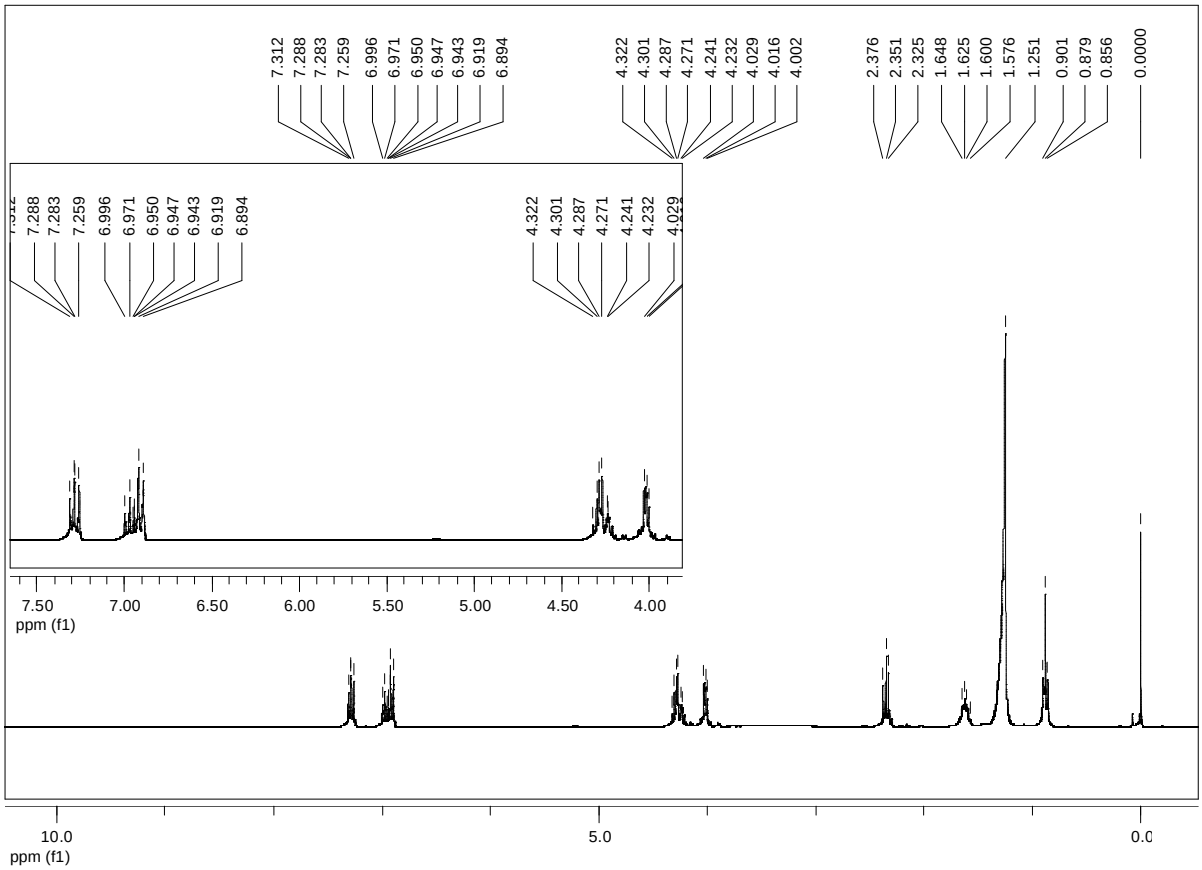
25.437
24.988
22.731
14.151
150
100
50
ppm (f1)


O
O
O
OH
10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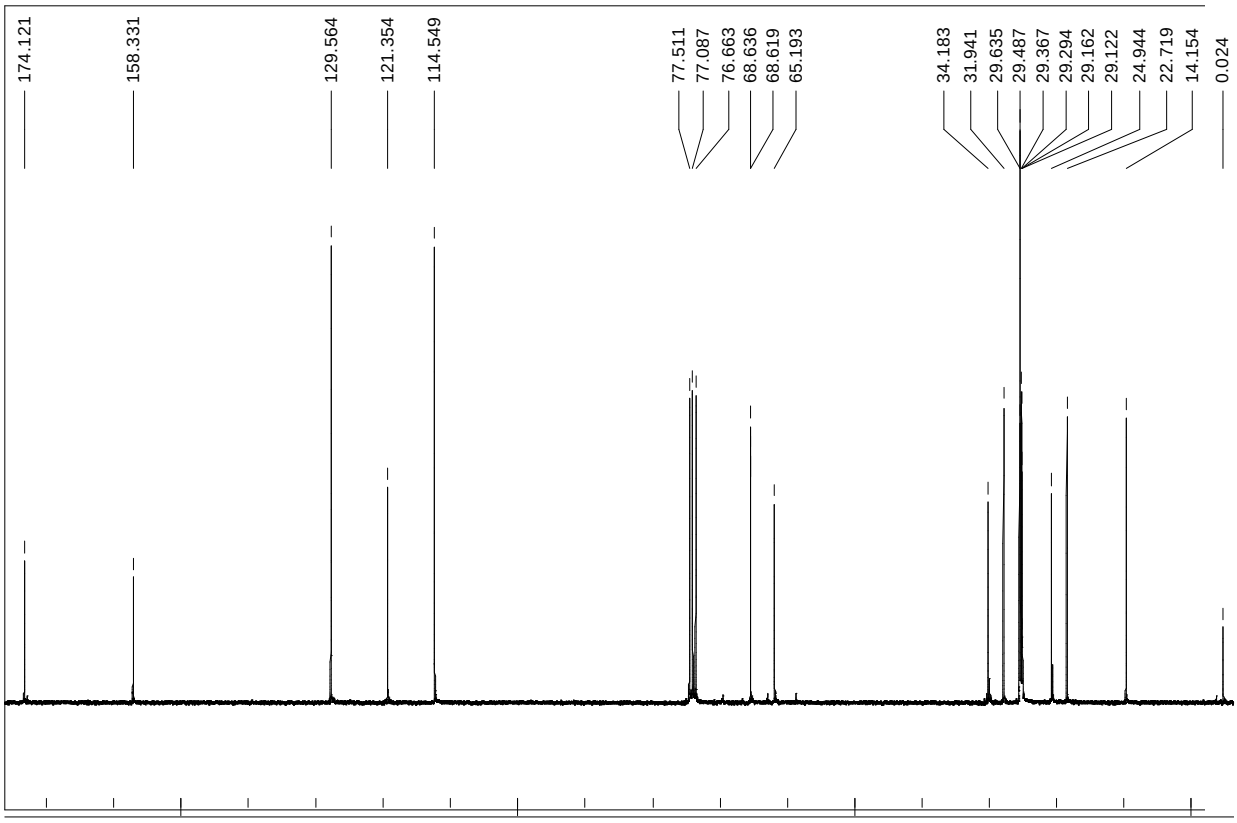
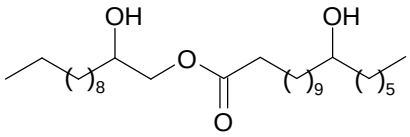
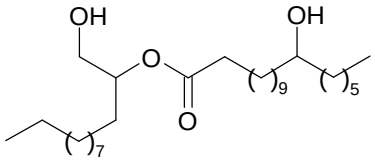
7.283
7.259
6.996
6.971
6.950
6.947
6.943
6.919
6.894
4.322
4.301
4.287
4.271
4.241
4.232
4.029
4.016
4.002
2.376
2.351
2.325
1.648
1.625
1.600
1.576
1.251
0.901
0.879
0.856
0.0000
7.50
7.00
6.50
6.00
5.50
5.00
4.50
4.00
ppm (f1)
10.0
5.0
0.0
ppm (f1)

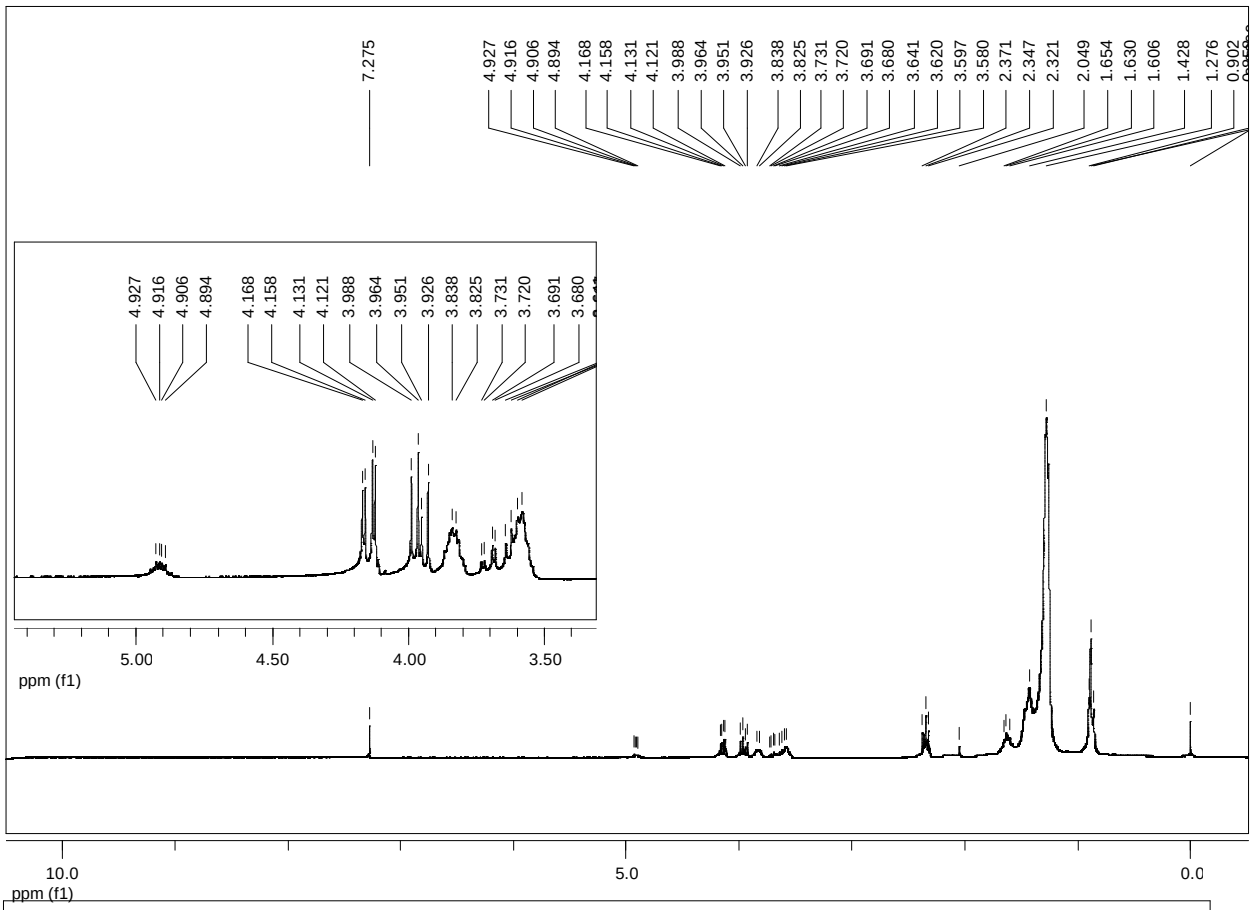




regioisomer 1



regioisomer 2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4.131
4.121
3.988
3.964
3.951
3.926
3.838
3.825
3.731
3.720
3.691
3.680
3.641
3.620
3.597
3.580
2.371
2.347
2.321
2.049
1.654
1.630
1.606
1.428
1.276
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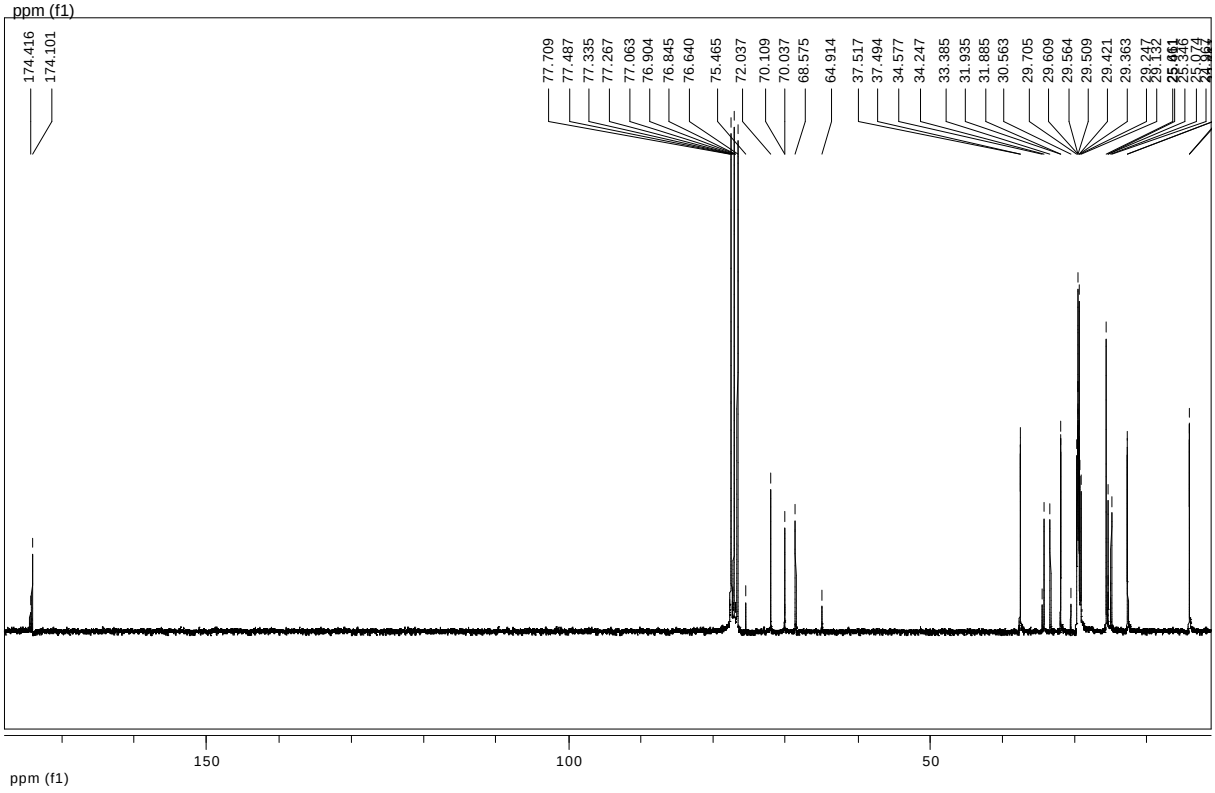
5.00
4.50
4.00
3.50
ppm (f1)
10.0
5.0
0.0
ppm (f1)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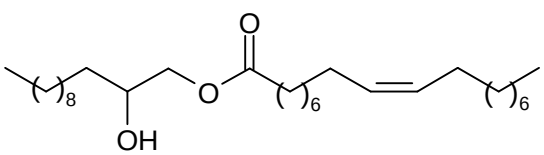
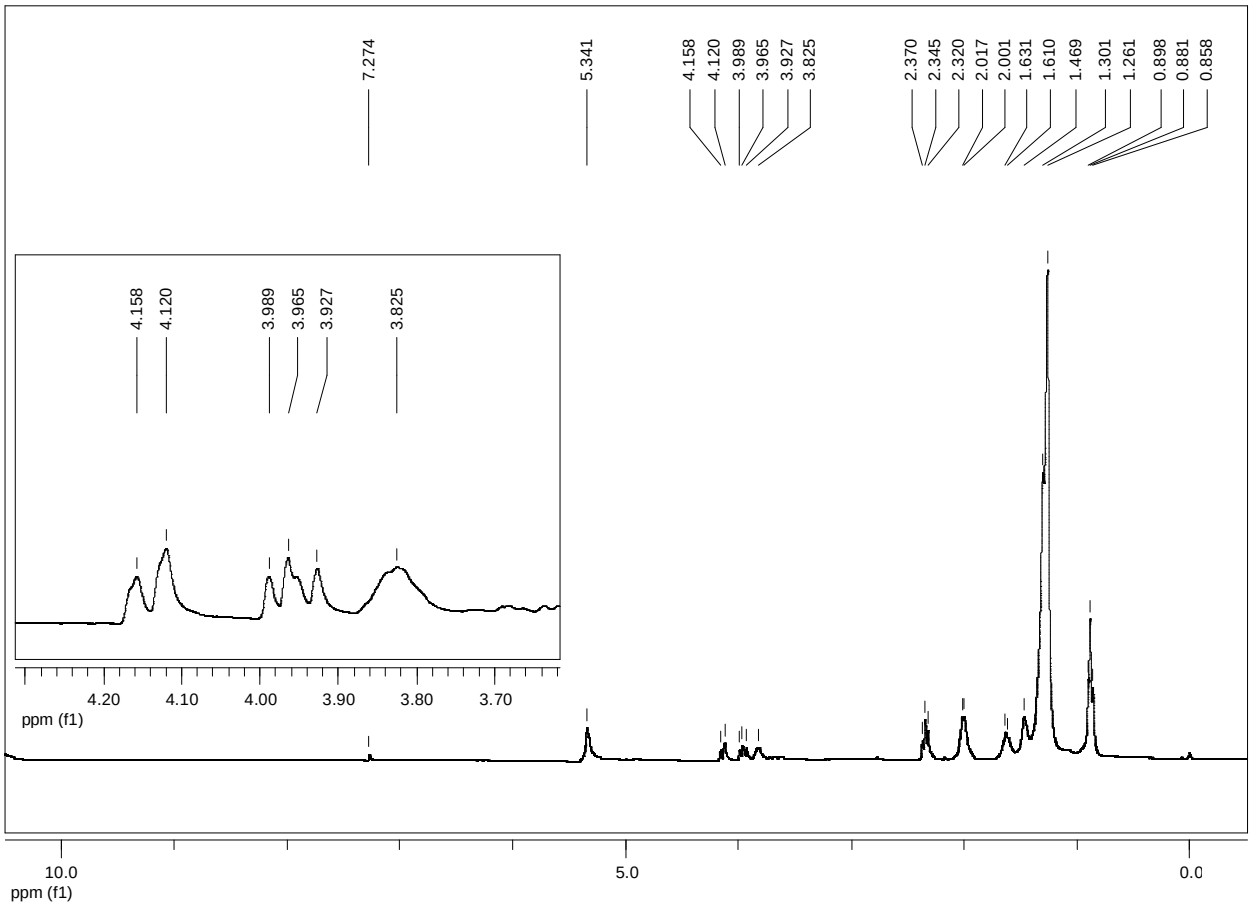
77.267
77.063
76.904
76.845
76.640
75.465
72.037
70.109
70.037
68.575
64.914
37.517
37.494
34.577
34.247
33.385
31.935
31.885
30.563
29.705
29.609
29.564
29.509
29.421
29.363
150
100
50
ppm (f1)


O
8
O
6
6
OH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4.120
3.989
3.965
3.927
3.825
2.370
2.345
2.320
2.017
2.001
1.631
1.610
1.469
1.301
1.261
0.898
0.881
0.858
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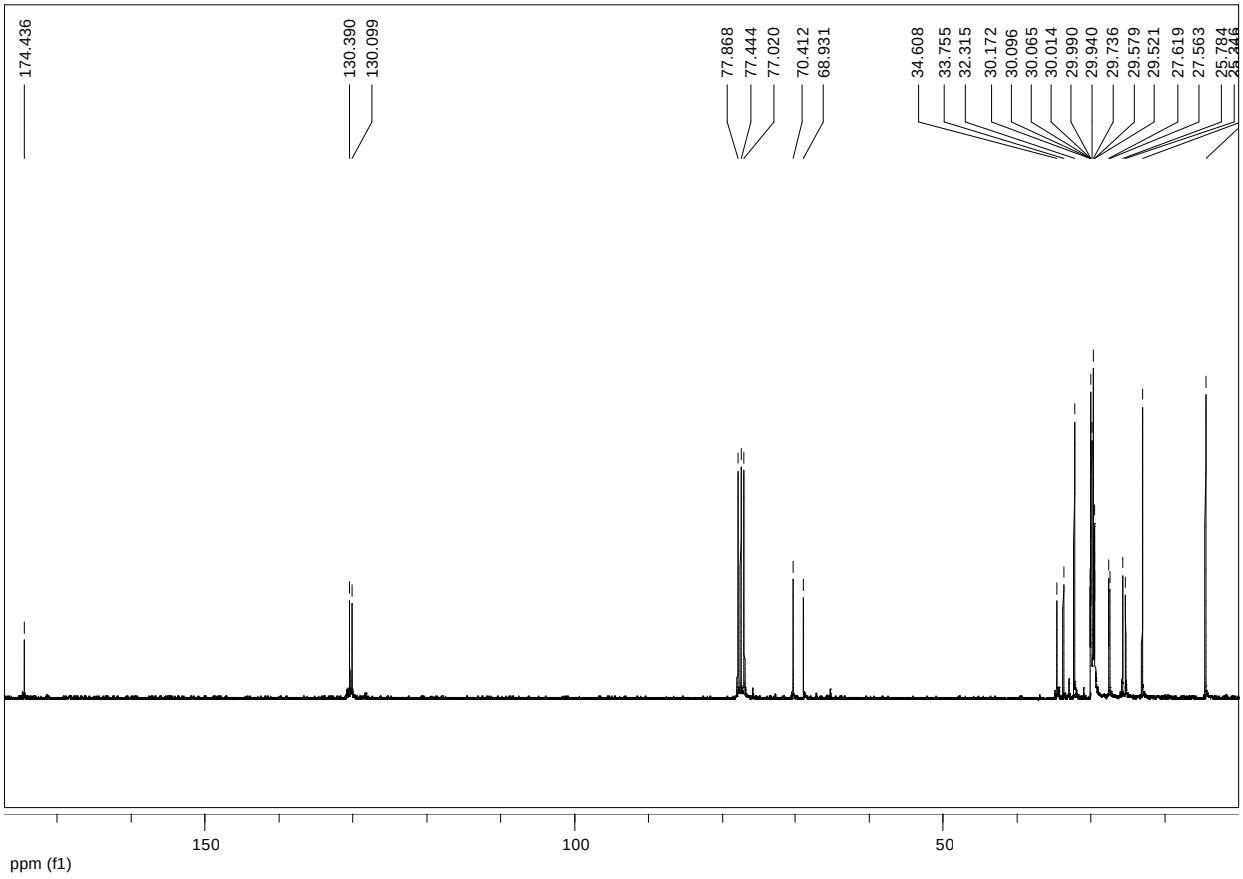
4.00
3.90
3.80
3.70
ppm (f1)
10.0
5.0
0.0
ppm (f1)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70.412
68.931
34.608
33.755
32.315
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30.065
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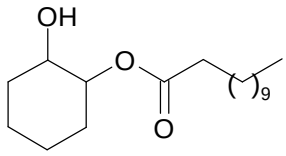
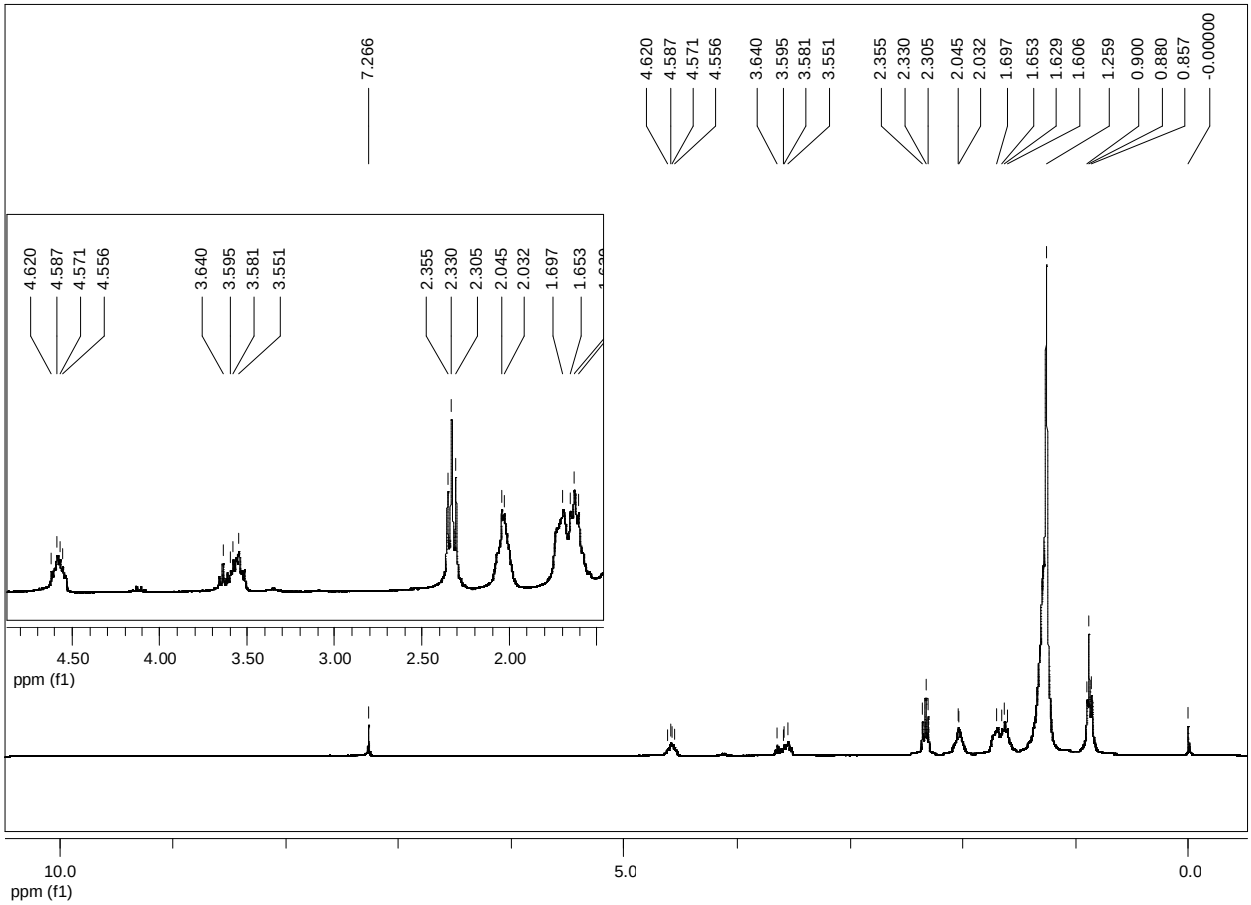
27.619
27.563
150
100
50
ppm (f1)


OH
O
O
9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4.620
4.587
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3.595
3.581
3.551
2.355
2.330
2.305
2.045
2.032
1.697
1.653
1.629
1.606
1.259
0.900
0.880
0.857
-0.00000
4.50
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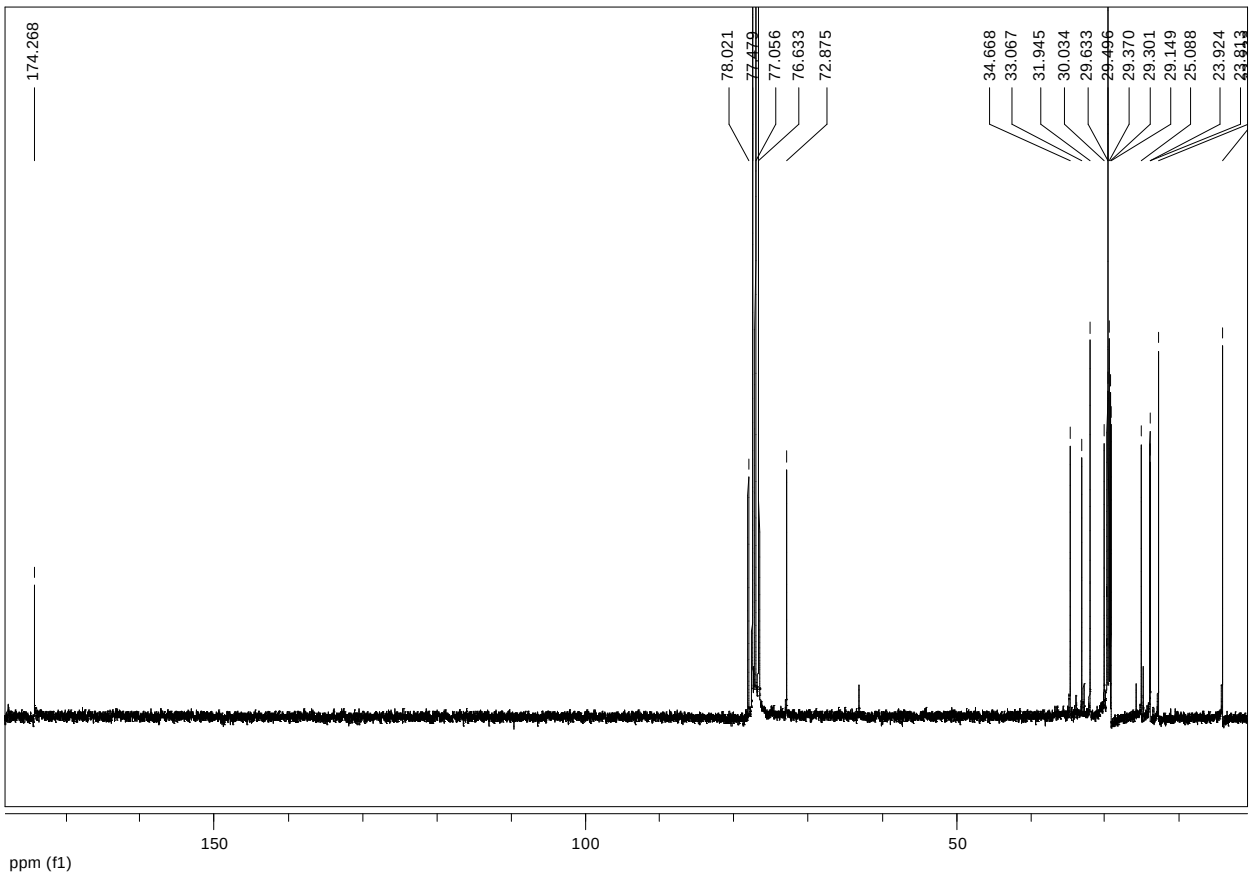
3.50
3.00
2.50
2.00
ppm (f1)
10.0
5.0
0.0
ppm (f1)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78.021
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72.875
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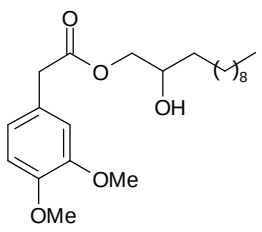
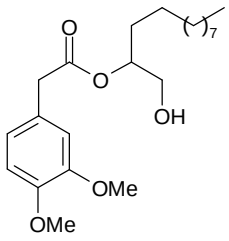
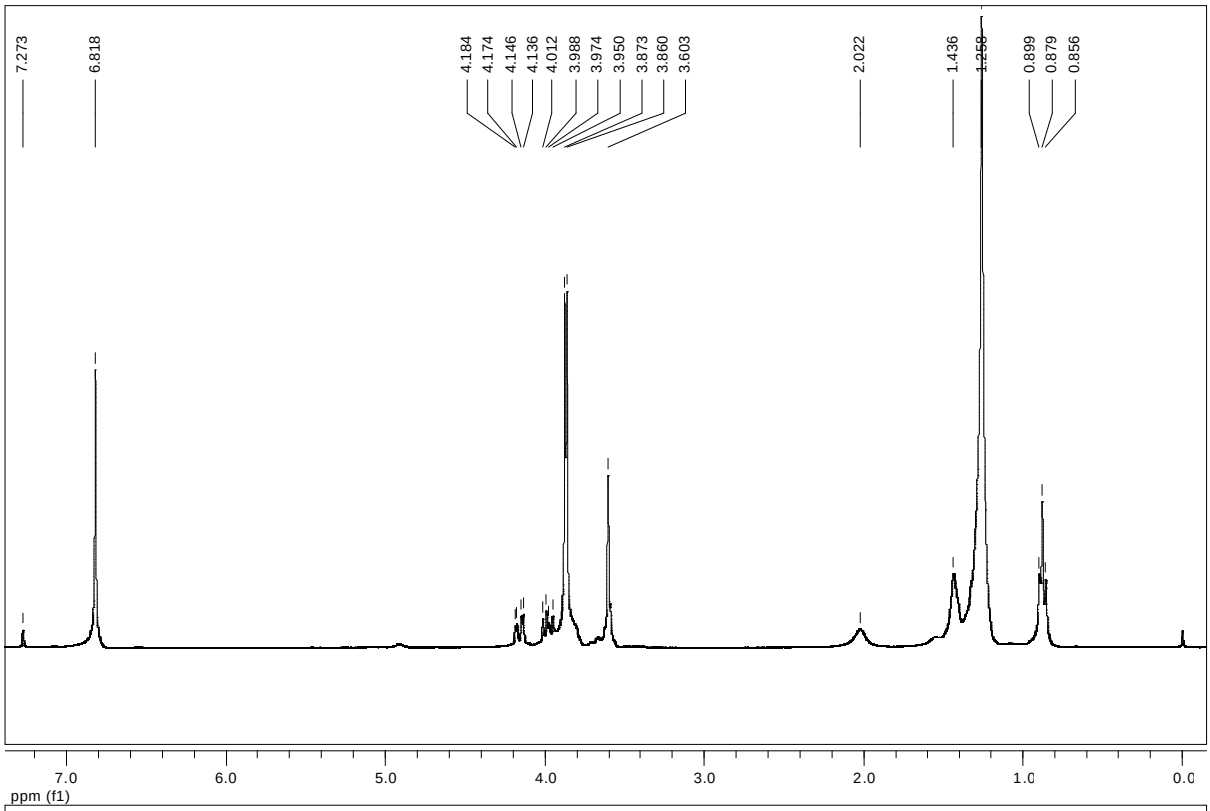
29.370
29.301
29.149
25.088
23.924
150
100
50
ppm (f1)

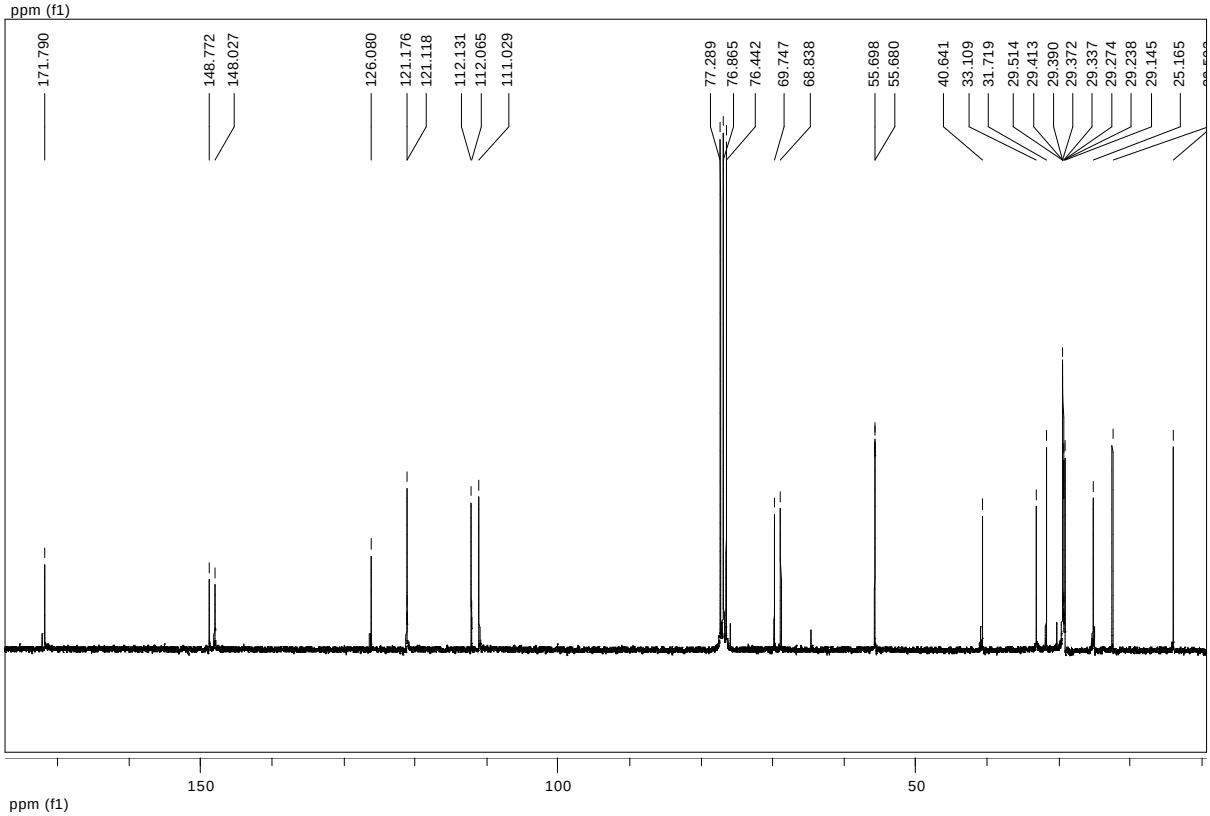


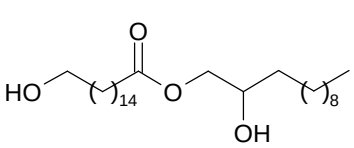
regioisomer 1



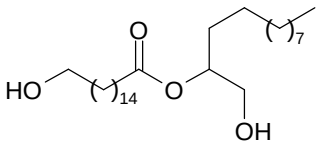
regioisomer 2



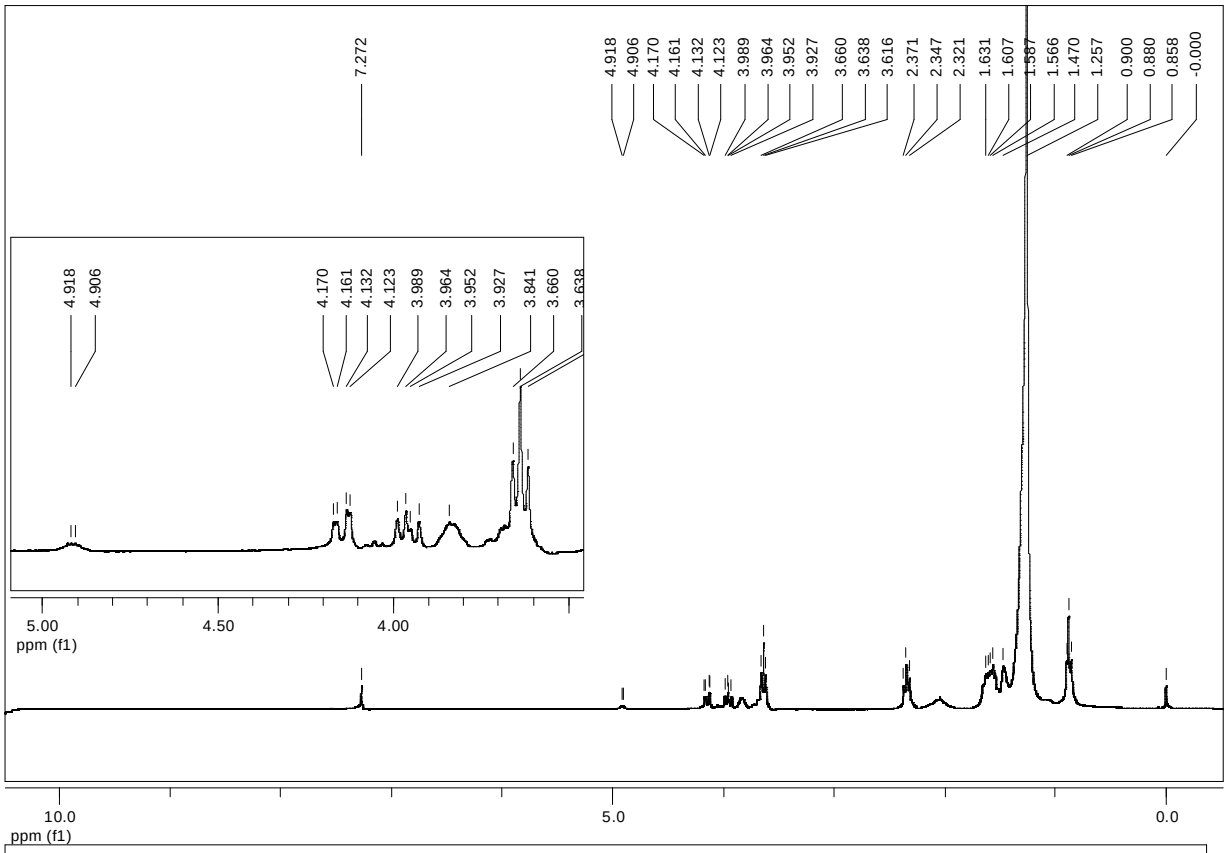


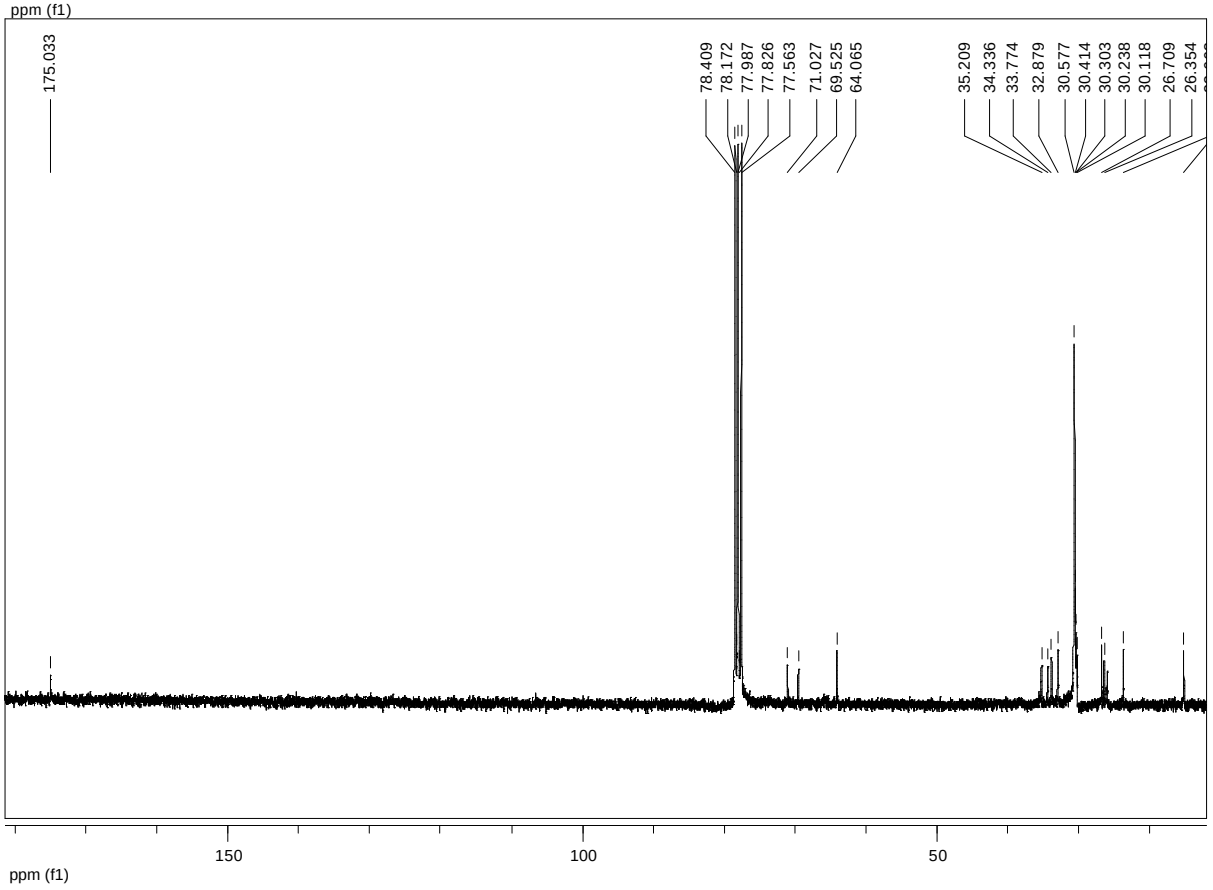


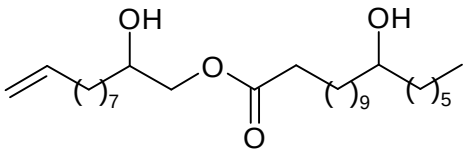
regioisomer 1

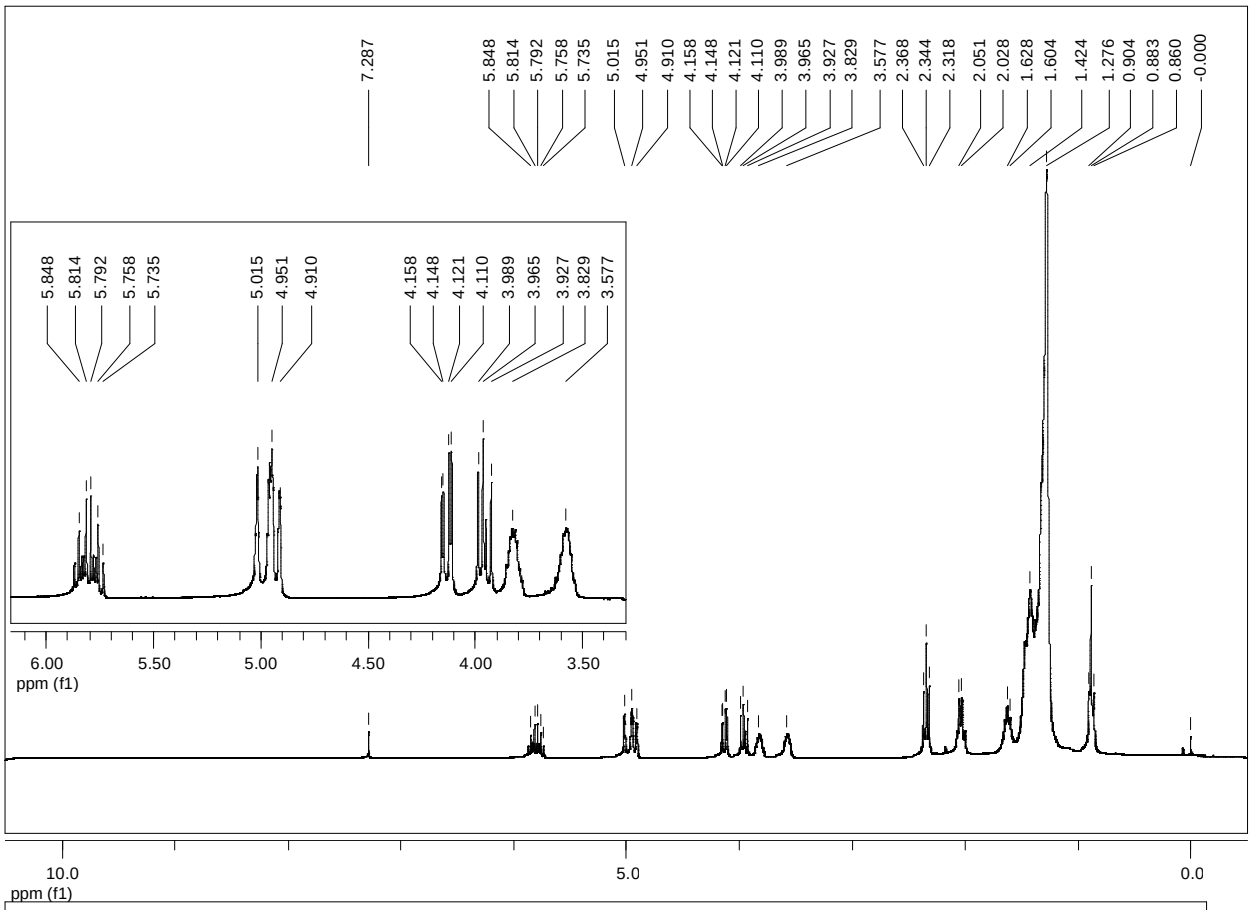


regioisomer 2






OH
OH
O
O
7
9
5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4.110
3.989
3.965
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3.577
2.368
2.344
2.318
2.051
2.028
1.628
1.604
1.424
1.276
0.904
0.883
0.860
-0.000
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5.00
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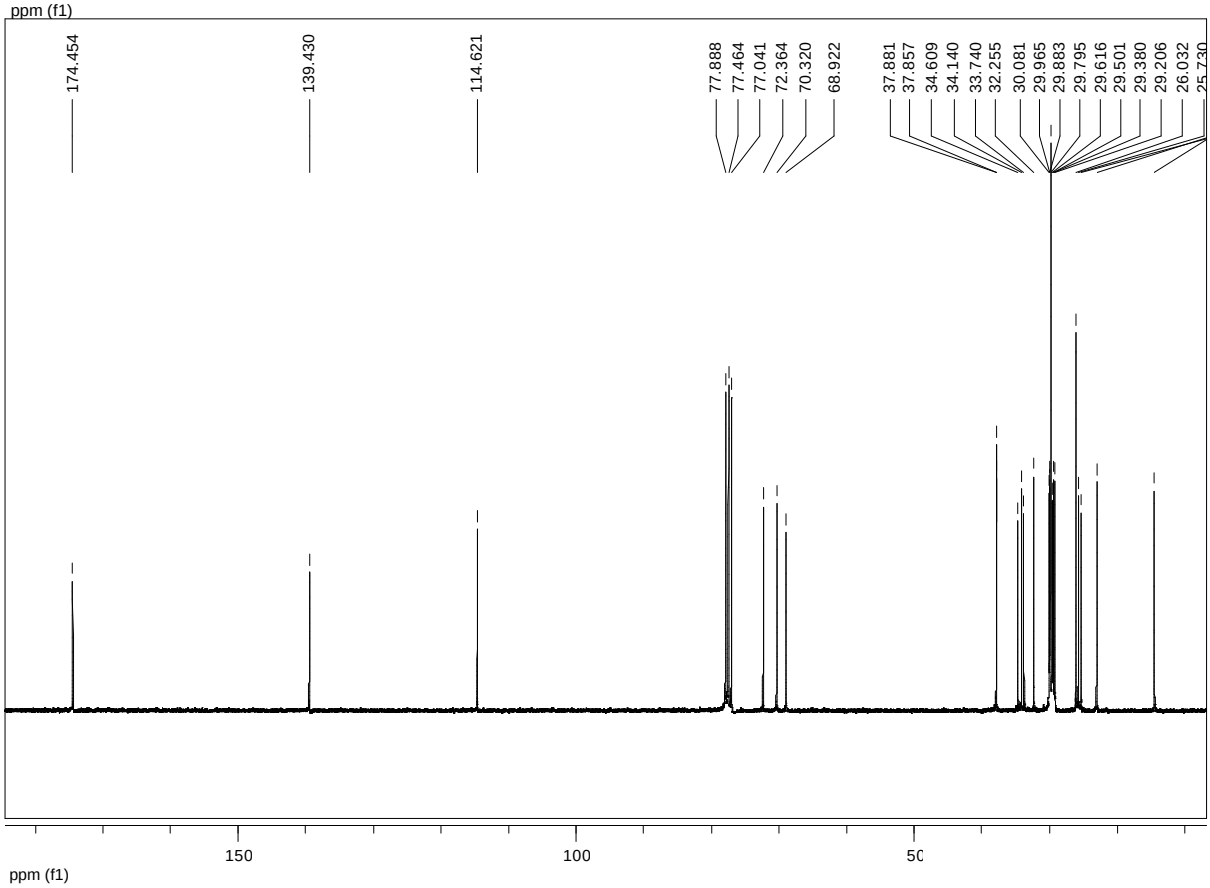
4.00
3.50
ppm (f1)
10.0
5.0
0.0
ppm (f1)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114.621
77.888
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77.041
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70.320
68.922
37.881
37.857
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34.140
33.740
32.255
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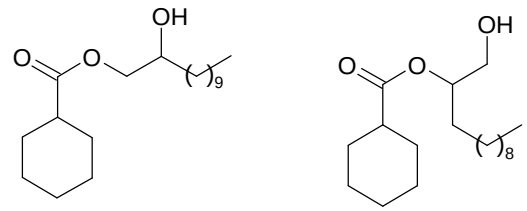
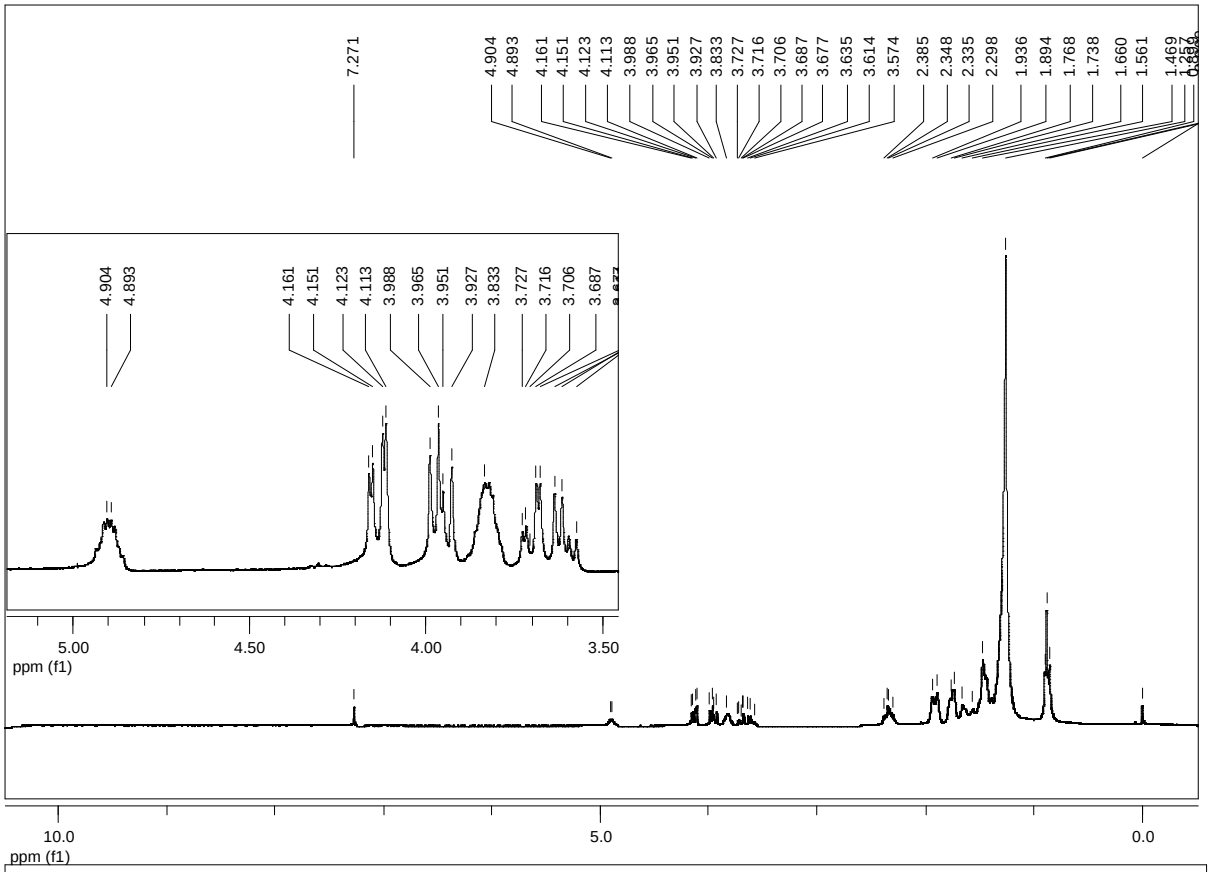
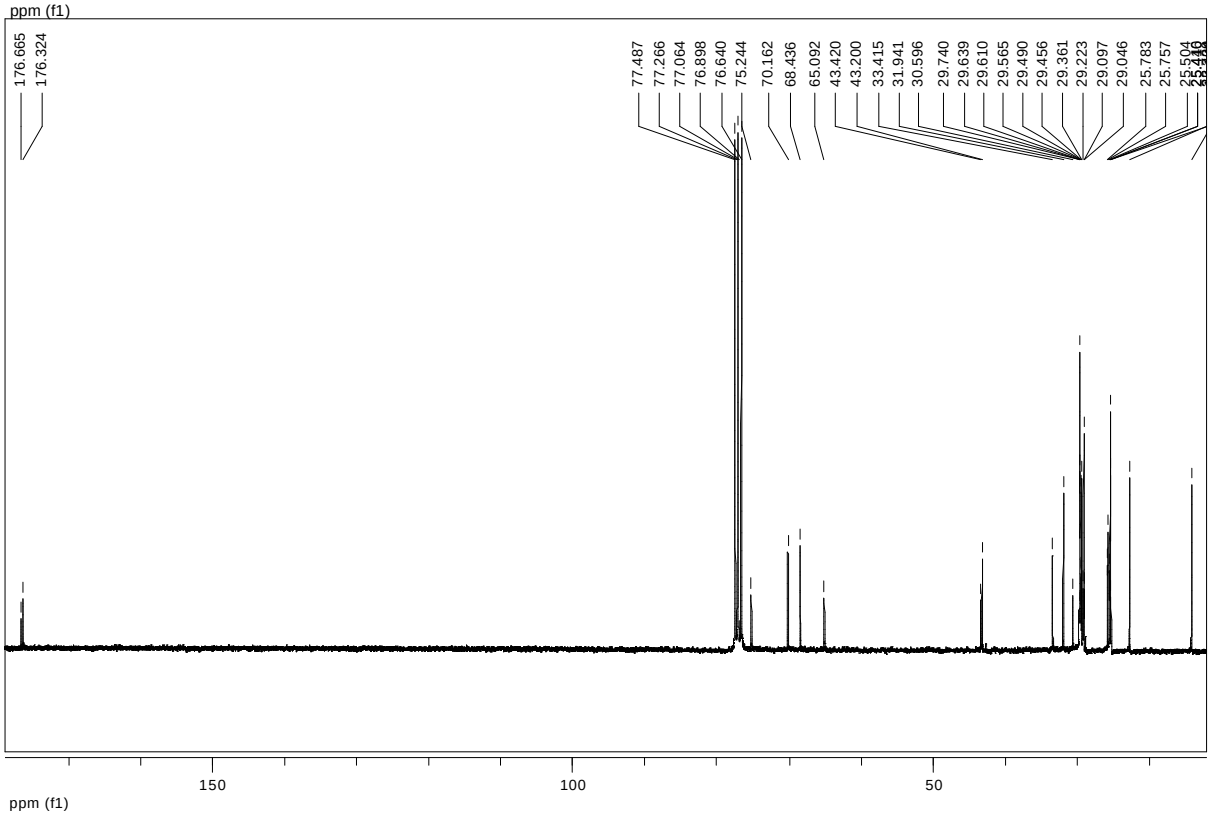
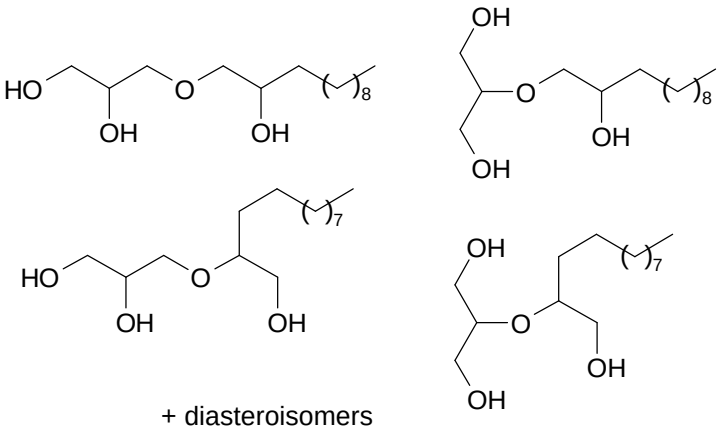
26.032
25.730
150
100
50
ppm (f1)



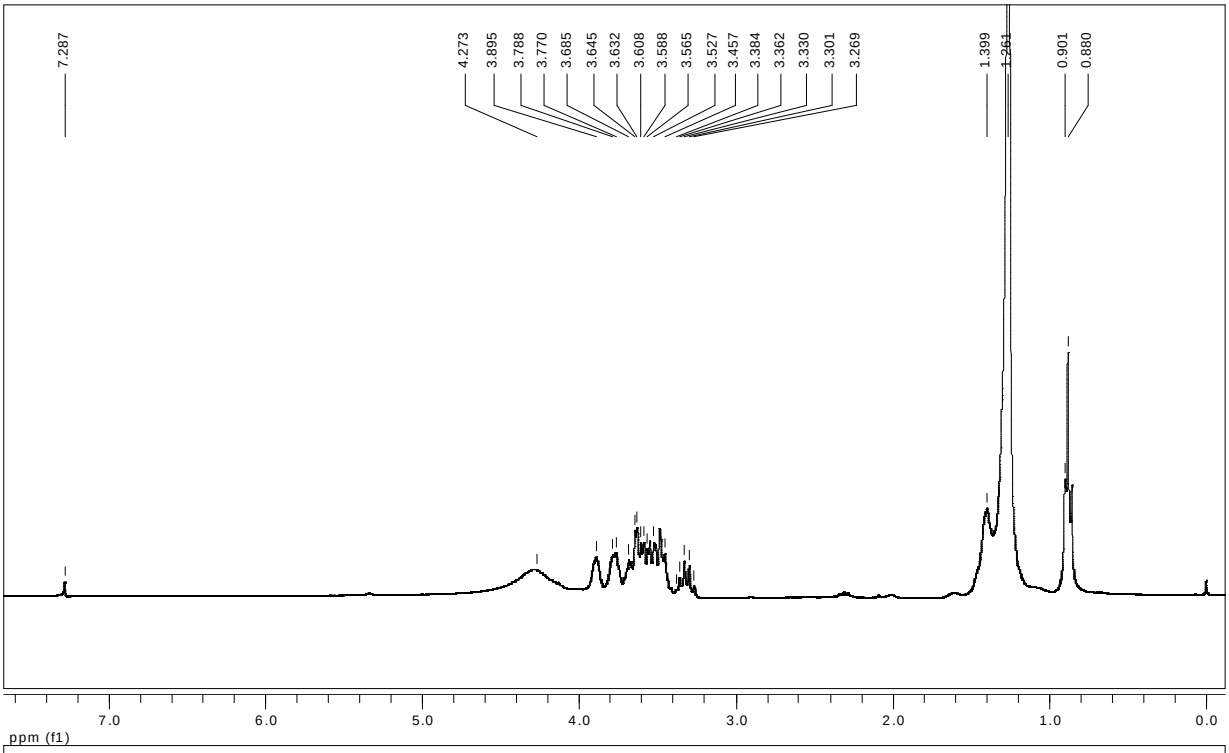
regioisomer 1 regioisomer 2

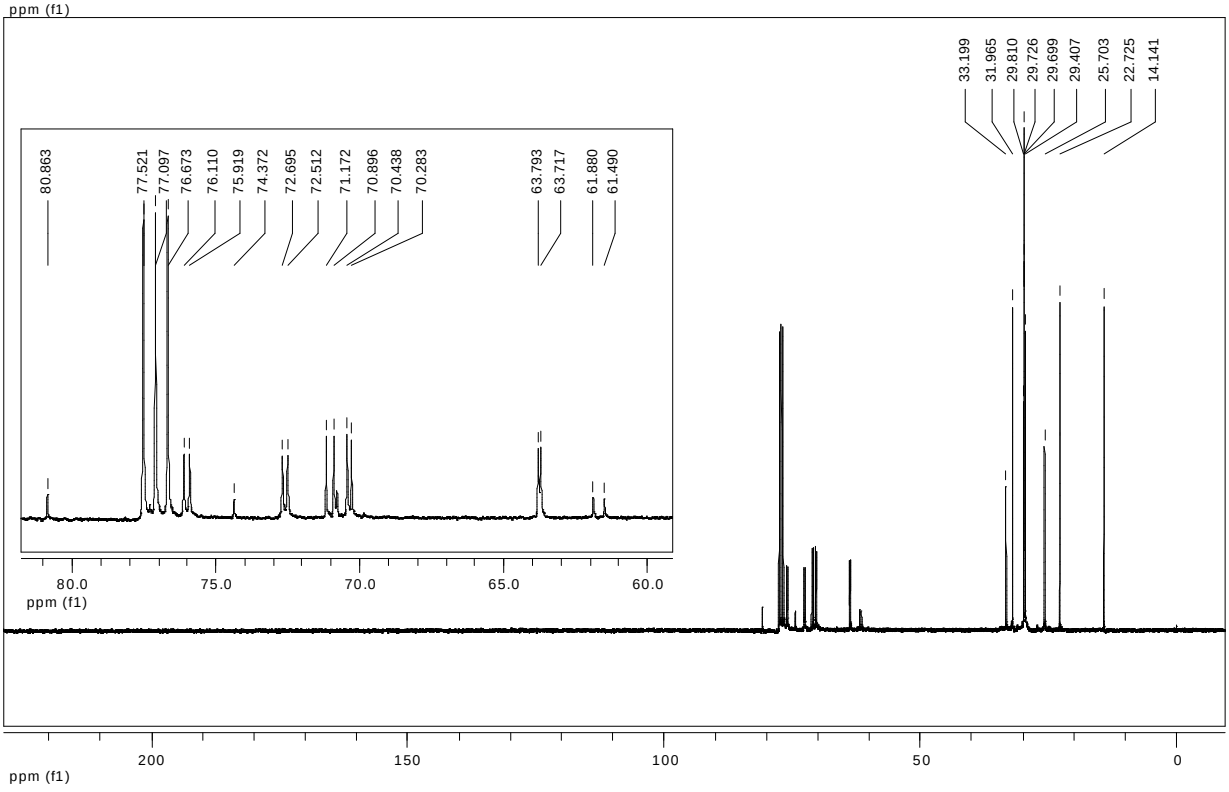


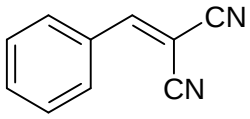


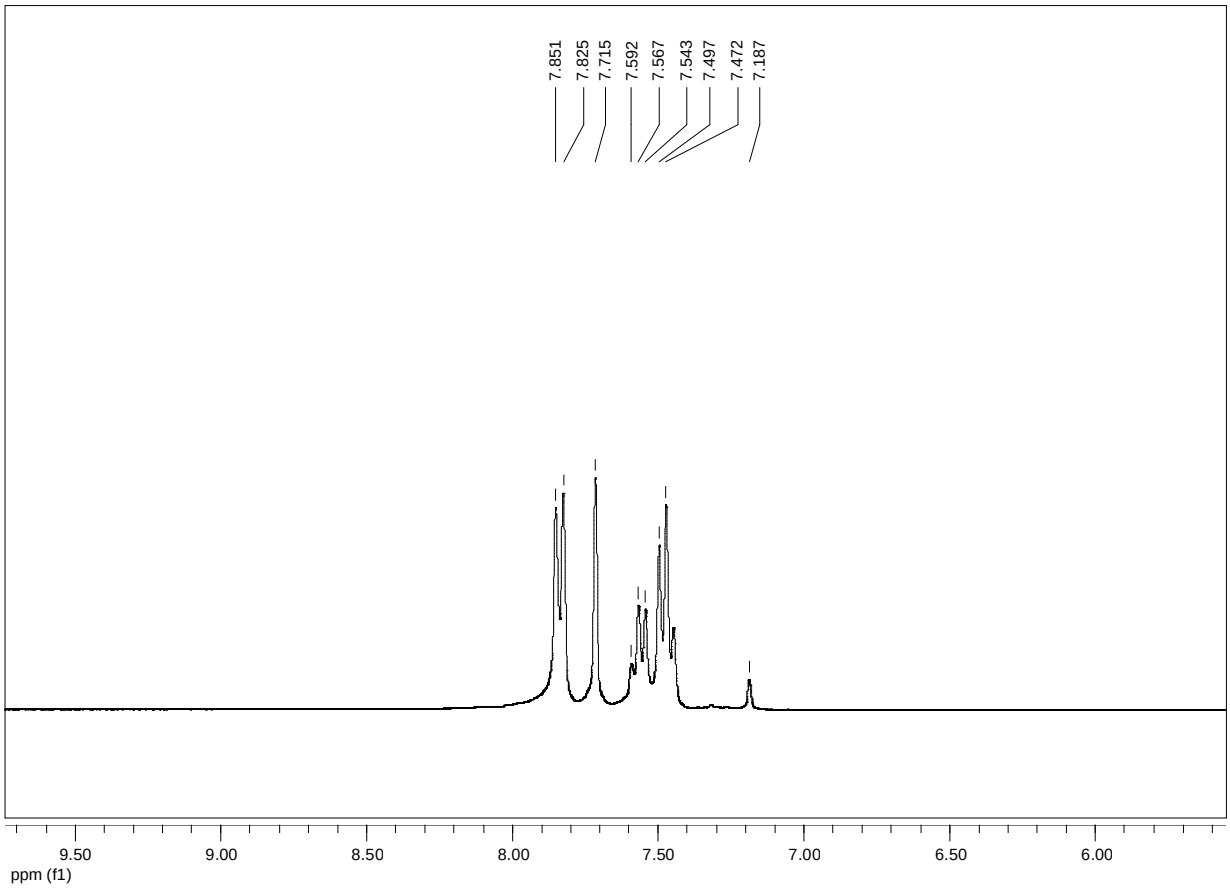


+ diasteroisomers






CN
CN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7.567
7.543
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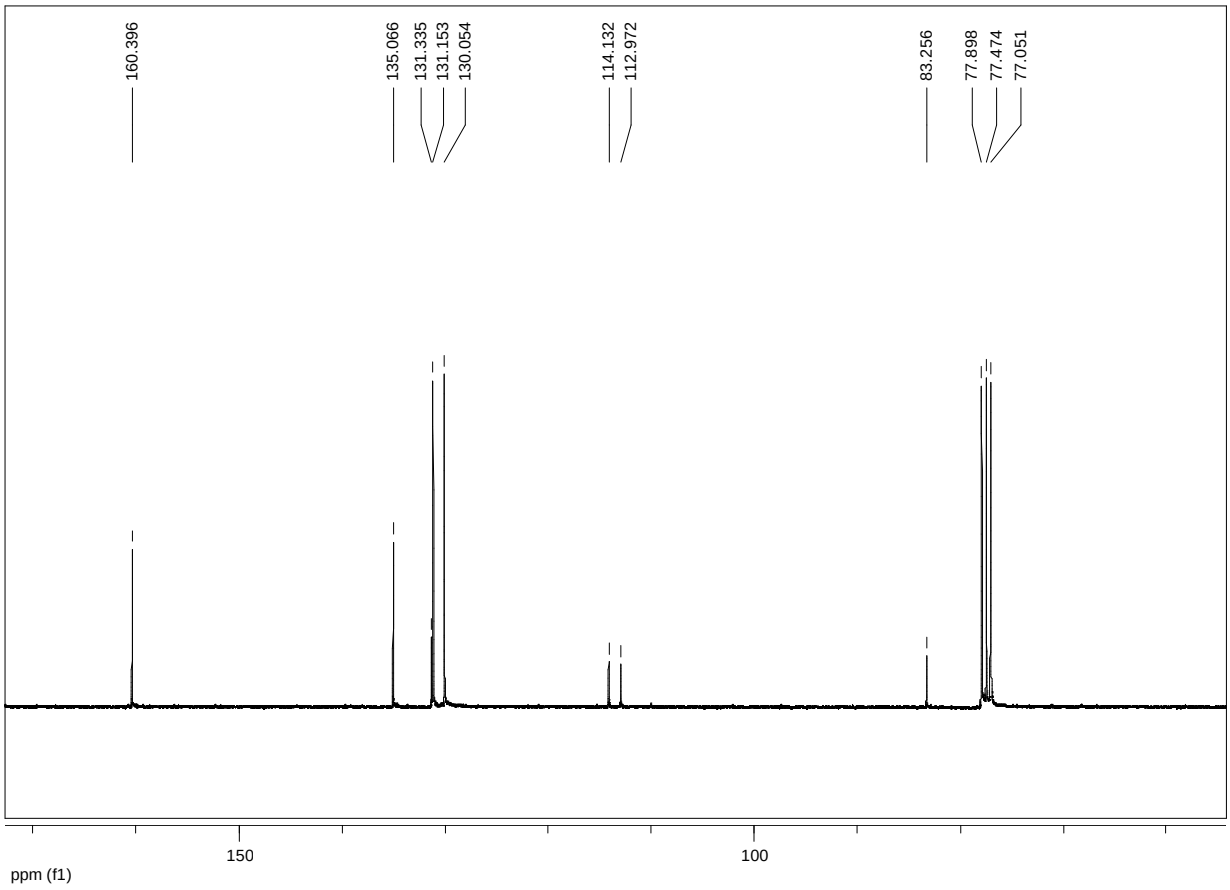
7.00
6.50
6.00
ppm (f1)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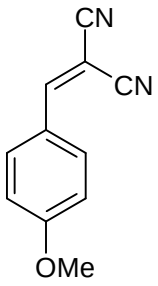
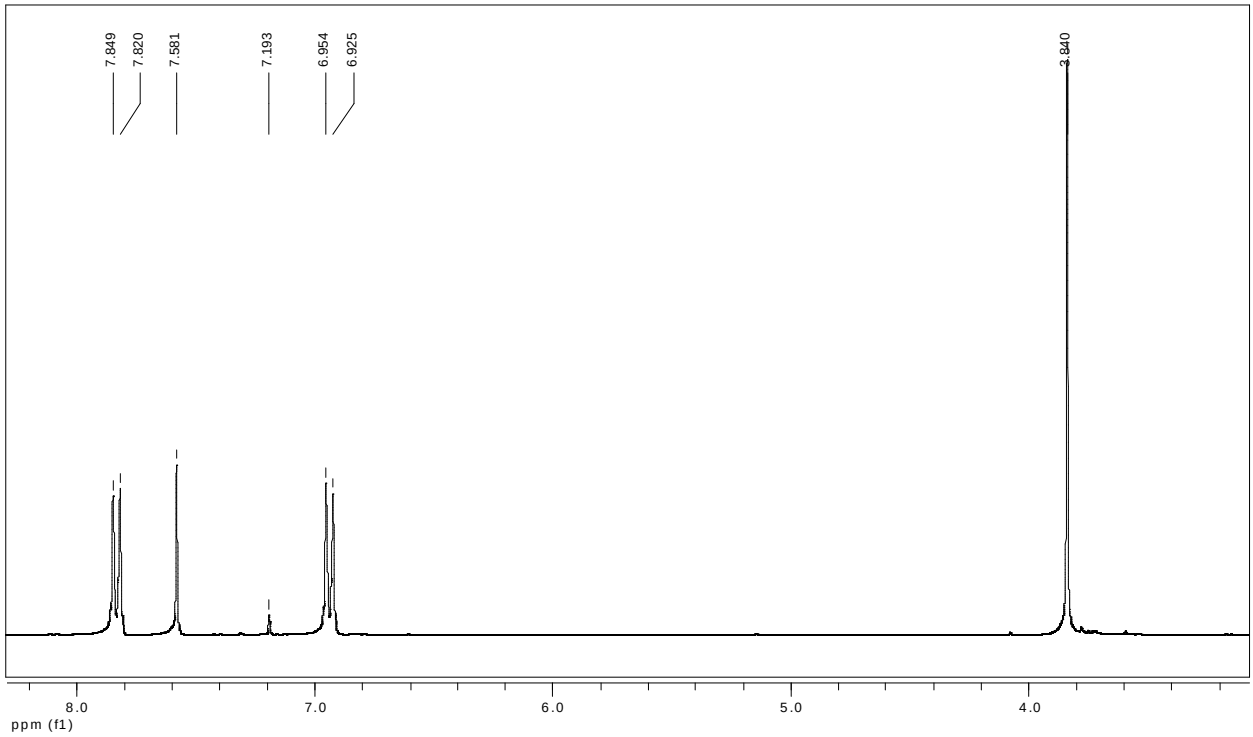
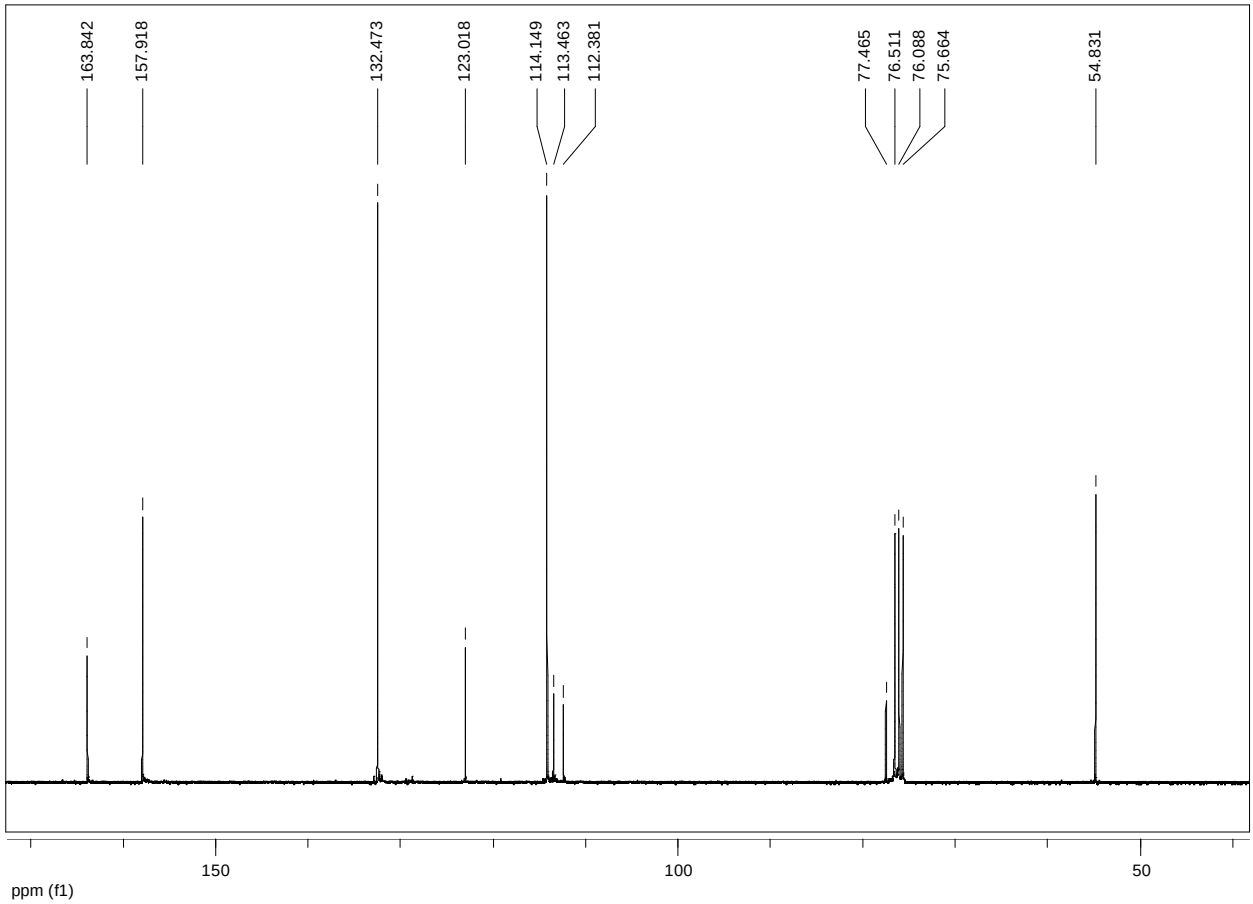
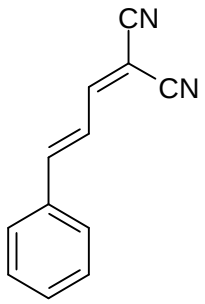
131.153
130.054
114.132
112.972
83.256
77.898
77.474
77.051
150
100
ppm (f1)


CN
CN
OMe


7.849
7.820
7.581
7.193
6.954
6.925
3.840
8.0
7.0
6.0
5.0
4.0
ppm (f1)


163.842
157.918
132.473
123.018
114.149
113.463
112.381
77.465
76.511
76.088
75.664
54.831
150
100
50
ppm (f1)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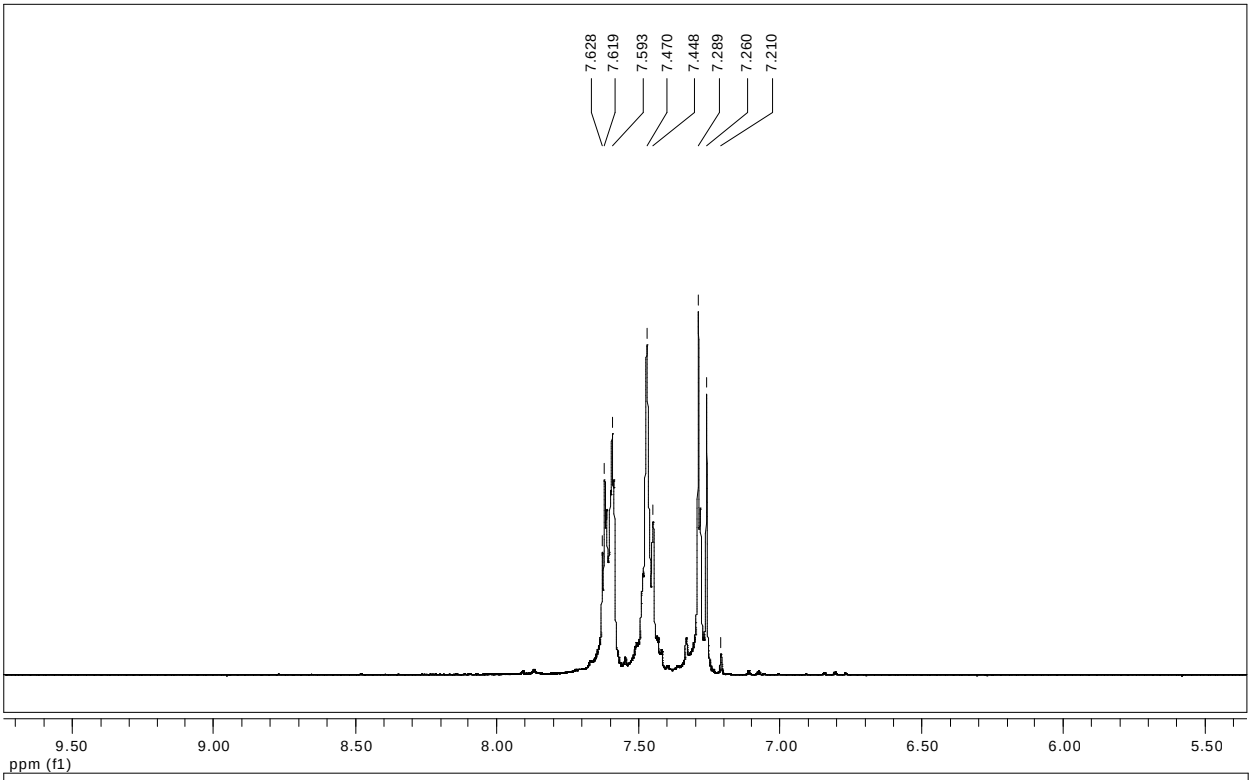
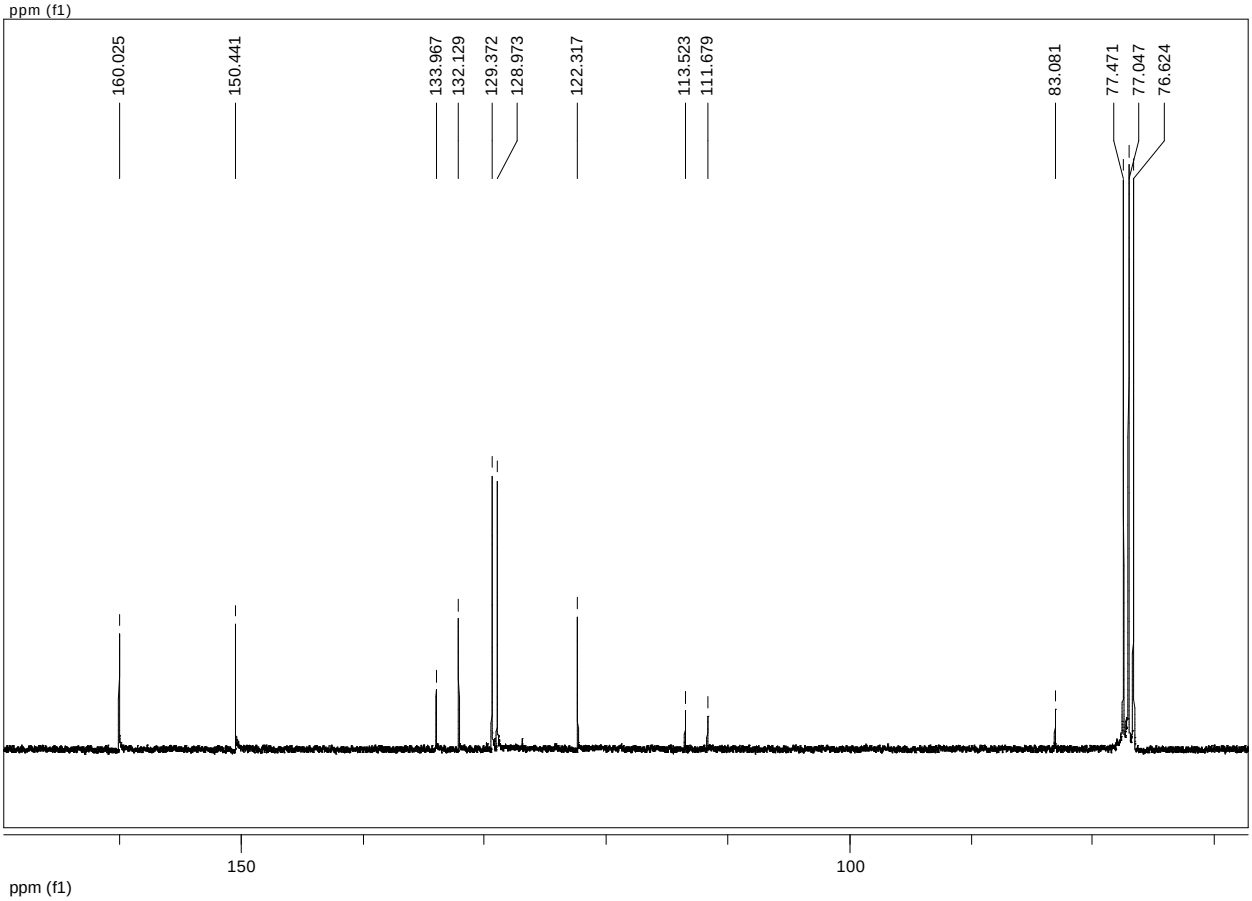
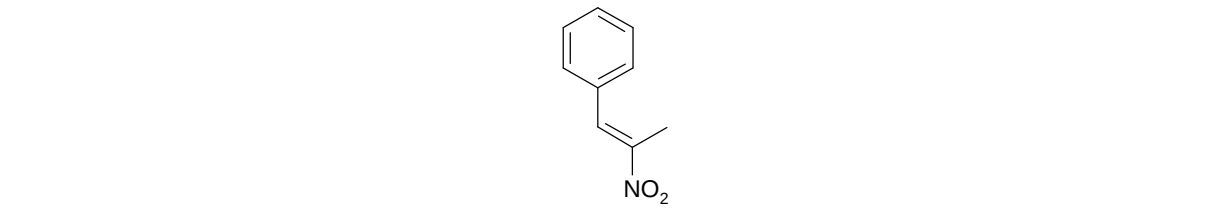
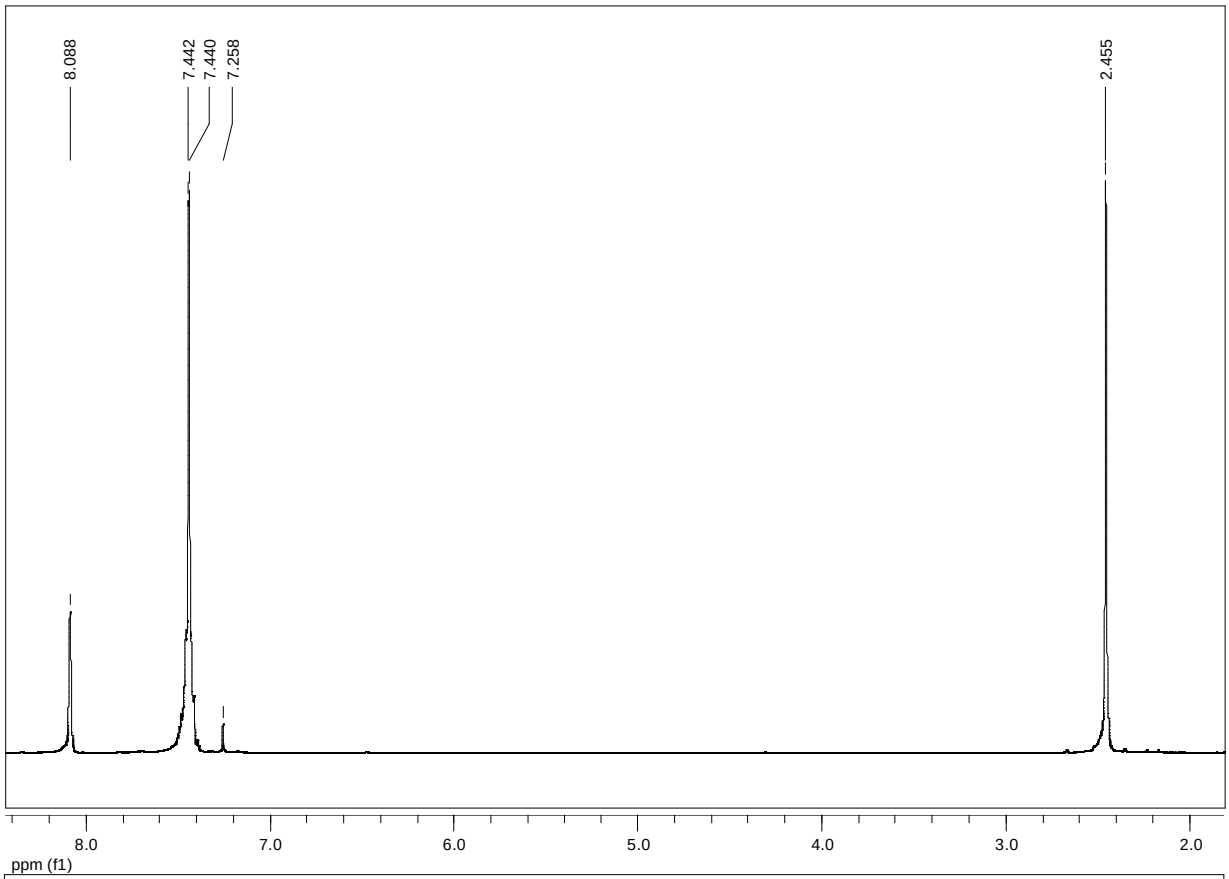
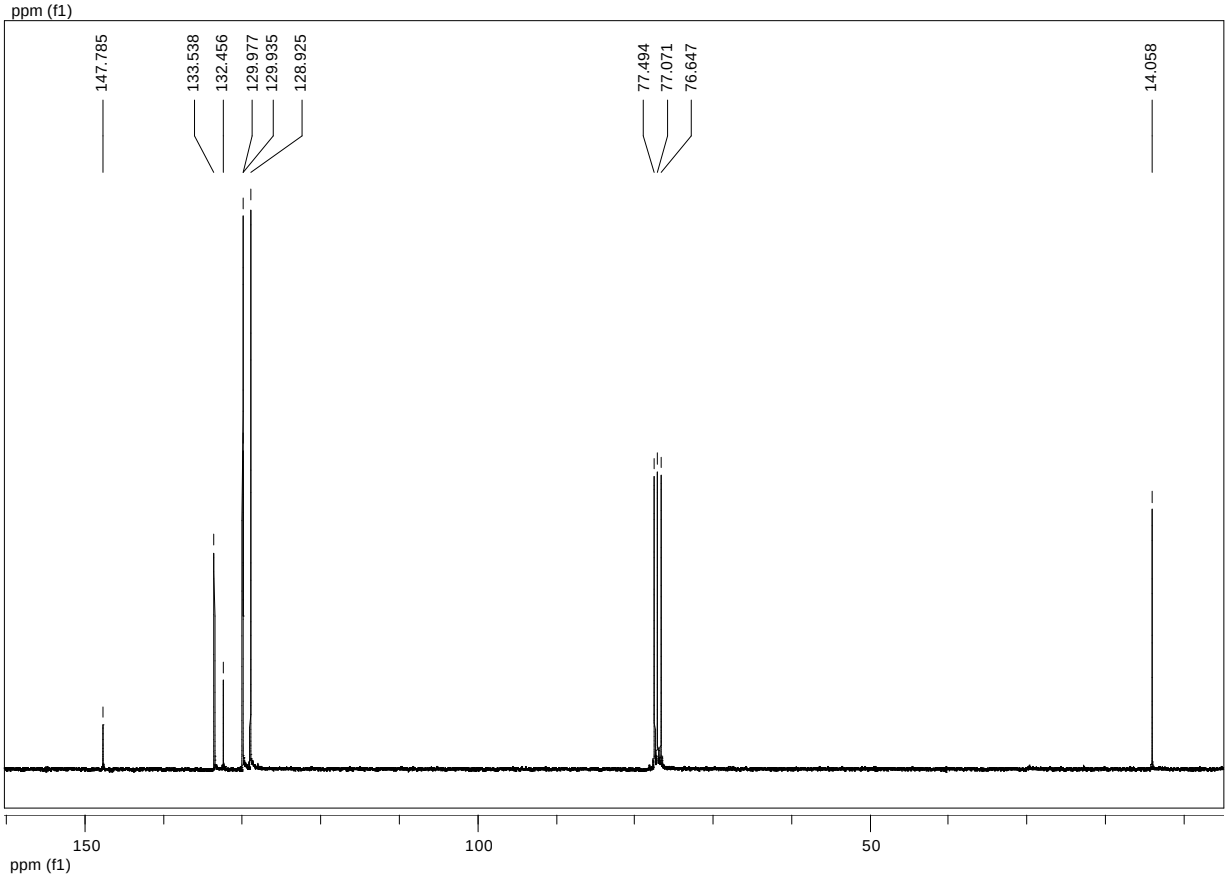
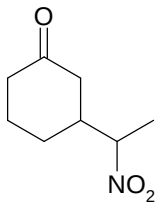

7.628
7.619
7.593
7.470
7.448
7.289
7.260
7.210
9.50
9.00
8.50
8.00
7.50
7.00
6.50
6.00
5.50
ppm (f1)


160.025
150.441
133.967
132.129
129.372
128.973
122.317
113.523
111.679
83.081
77.471
77.047
76.624
150
100
ppm (f1)


NO2


8.088
7.442
7.440
7.258
2.455
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (f1)


147.785
133.538
132.456
129.977
129.935
128.925
77.494
77.071
76.647
14.058
150
100
50
ppm (f1)



mixture of diastereoisomers