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Supporting Information

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Asymmetric Syntheses of 8-Oxabicyclo[3,2,1]octanes: A Cationic Cascade Cyclization**

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Contents

1 General Information

Reagents and solvents for Mukaiyama-aldol/[1,5] hydride shift cascade reaction were purified prior to use by the following procedures:

$TiCl_4$ solution (1.0 M in CH_2Cl_2) was purchased from Sigma-Aldrich and used as obtained.

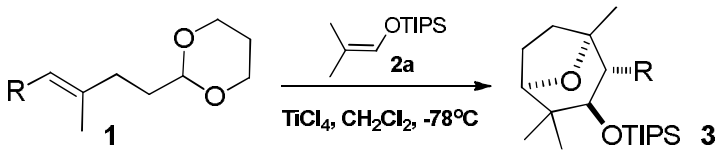
CH_2Cl_2 (Reagent grade, Merck) was distilled from CaH_2.

4Å molecular sieves were purchased from Alfa Aesar and activated at 250 ^oC for 10 hours prior to use.

Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 precoated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm. Further visualization was possible by staining with basic solution of potassium permanganate or acidic solution of ceric molybdate, followed by heating on a hot plate. Flash chromatography was performed using Merck silica gel 60 with distilled solvents. Columns were typically packed as slurry and equilibrated with hexane prior to use.

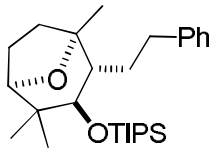
Proton nuclear magnetic resonance (^1H NMR) and carbon nuclear magnetic resonance (^{13}C NMR) spectroscopy were performed on Bruker Advance 300, 400 and 500 NMR spectrometers. Chemical shifts of ^1H NMR spectra are reported as d in units of parts per million (ppm) downfield from $SiMe_4$ (d = 0.0) and relative to the signal of chloroform-*d* (d = 7.264, singlet). Multiplicities were given as: s (singlet); d (doublet); t (triplet); q (quartet); dd (doublet of doublets); ddd (doublet of doublets of doublets); m (multiplets) and etc. The number of protons for a given resonance is indicated by nH. Coupling constants are reported as *J* values in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as d in units of parts per million (ppm) downfield from $SiMe_4$ (d 0.0) and relative to the signal of chloroform-*d* (d = 77.23, triplet). Infrared spectra were recorded on a Shimadzu IR Prestige-21 FT-IR Spectrometer. Liquid samples were examined as film between NaCl salt plates. High-resolution mass spectral analysis (HRMS) was performed on Q-Tof Premier mass spectrometer (Waters Corporation).

2 General Procedure for Cationic Cascade Reaction to Construct 8-Oxabicyclo[3,2,1]octanes



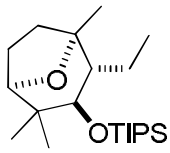
An oven-dried round bottom flask (10 mL) equipped with a magnetic stir bar was charged with 4Å molecular sieves (300 mg), and sealed with a rubber septum. Then acetal (0.20 mmol) and silyl enol ether (0.3 mmol, 1.5 equiv) were dissolved in dry CH_2Cl_2 (2 mL) and added via syringe. After cooling the solution to -78 ^oC, $TiCl_4$ (0.24 mL of a 1.0 M solution in CH_2Cl_2, 0.24 mmol, 1.2 equiv) was added dropwisely. The solution was allowed to stir at -78 ^oC for 10 h and then quenched with sat. $NaHCO_3$ (5 mL), and extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine (30 mL), dried over Na_2SO_4, and concentrated *in vacuo*. The crude product was purified by flash column chromatography on silica gel using appropriate solvents (hexane/diethyl ether mixture) to provide the title compound.

3 Experimental Data for 8-Oxabicyclo[3,2,1]octanes



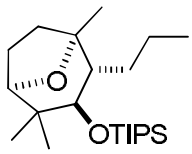
Triisopropyl((1,4,4-trimethyl-2-phenethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)silane (3a for Table 1)

The title compound prepared following the General Procedure described above. Yield: 91% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.93 (s, 3H), 1.04 (s, 3H), 1.07-1.10 (m, 21H), 1.38 (s, 3H), 1.51-1.64 (m, 3H), 1.81 (ddt, J_1 = 5.2 Hz, J_2 = 7.6 Hz, J_3 = 12.5 Hz, 1H), 2.11-2.14 (m, 1H), 2.22-2.28 (m, 1H), 2.45 (dt, J_1 = 5.1 Hz, J_2 = 10.0 Hz, 1H), 2.64-2.73 (m, 2H), 3.67 (d, J = 7.9 Hz, 1H), 3.69 (s, 1H), 7.16-7.20 (m, 3H), 7.25-7.30 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.96, 18.56, 23.88, 25.61, 26.27, 26.81, 35.29, 36.69, 37.66, 37.94, 51.23, 80.49, 80.96, 84.47, 125.90, 128.30, 128.41, 142.50. HRMS (ESI^+) exact mass calcd for $C_{27}H_{46}O_2SiNa$ $[M+Na]^+$ requires *m/z* 453.3165, found *m/z* 453.3164.



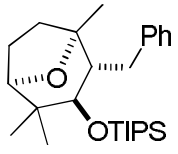
2-Ethyl-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)triisopropylsilane (3b for Table 1)

The title compound prepared following the General Procedure described above. Yield: 85% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.93 (s, 3H), 1.00 (t, J = 7.2 Hz, 3H), 1.03 (s, 3H), 1.08-1.14 (m, 21H), 1.30 (s, 3H), 1.32-1.37 (m, 1H), 1.43 (t, J = 6.1 Hz, 1H), 1.51 (dt, J_1 = 3.8 Hz, J_2 = 12.2 Hz, 1H), 1.75-1.85 (m, 2H), 2.24 (dt, J_1 = 3.6 Hz, J_2 = 11.2 Hz, 1H), 2.42 (dt, J_1 = 5.2 Hz, J_2 = 10.4 Hz, 1H), 3.66 (d, J = 7.8 Hz, 1H), 3.69 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.92, 15.47, 18.50, 23.92, 25.28, 26.22, 26.70, 36.73, 37.87, 53.13, 79.31, 80.94, 84.43. HRMS (ESI^+) exact mass calcd for $C_{21}H_{43}O_2Si$ $[M+H]^+$ requires *m/z* 355.3032, found *m/z* 355.3041.



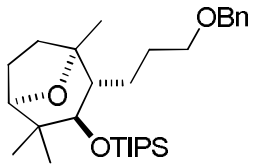
Triisopropyl((1,4,4-trimethyl-2-propyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)silane (3c for Table 1)

The title compound prepared following the General Procedure described above. Yield: 90% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.91 (t, J = 7.2 Hz, 3H), 0.93 (s, 3H), 1.04 (s, 3H), 1.05-1.14 (m, 21H), 1.18-1.26 (m, 1H), 1.28 (s, 3H), 1.32-1.42 (m, 2H), 1.46-1.54 (m, 2H), 1.64-1.73 (m, 1H), 1.79 (ddt, J_1 = 4.9 Hz, J_2 = 7.7 Hz, J_3 = 12.2 Hz, 1H), 2.24 (dt, J_1 = 3.8 Hz, J_2 = 12.0 Hz, 1H), 2.41 (dt, J_1 = 4.2 Hz, J_2 = 8.8 Hz, 1H), 3.65 (d, J = 6.0 Hz, 1H), 3.66 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.93, 14.86, 18.51, 23.91, 24.52, 25.34, 26.23, 26.79, 35.53, 36.65, 37.89, 51.62, 80.17, 80.92, 84.47. HRMS (ESI^+) exact mass calcd for $C_{22}H_{45}O_2Si$ $[M+H]^+$ requires *m/z* 369.3189, found *m/z* 369.3202.



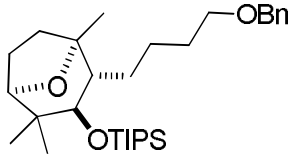
((2-Benzyl-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)triisopropylsilane (3d for Table 1)

The title compound prepared following the General Procedure described above. Yield: 92% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.68 (qd, J_1 = 7.5 Hz, J_2 = 15.1 Hz, 3H), 0.89-0.93 (m, 21H), 1.25 (s, 3H), 1.38 (s, 3H), 1.58 (dt, J_1 = 3.7 Hz, J_2 = 12.2 Hz, 1H), 1.79-1.85 (m, 1H), 1.89 (dd, J_1 = 5.2 Hz, J_2 = 10.8 Hz, 1H), 2.24 (dt, J_1 = 4.0 Hz, J_2 = 12.8 Hz, 1H), 2.46 (dt, J_1 = 5.2 Hz, J_2 = 10.4 Hz, 1H), 2.64 (dd, J_1 = 10.9 Hz, J_2 = 14.0 Hz, 1H), 3.08 (dd, J_1 = 5.1 Hz, J_2 = 14.1 Hz, 1H), 3.59 (s, 1H), 3.73 (d, J = 7.7 Hz, 1H), 7.15-7.19 (m, 3H), 7.24-7.28 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.44, 18.38, 18.41, 24.25, 25.83, 26.14, 28.03, 36.77, 37.03, 38.15, 52.90, 75.52, 81.01, 84.43, 125.81, 128.18, 129.62, 141.51. HRMS (ESI^+) exact mass calcd for $C_{26}H_{45}O_2Si$ $[M+H]^+$ requires *m/z* 417.3189, found *m/z* 417.3204.



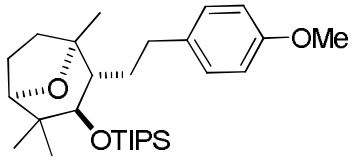
((2-(3-(Benzyloxy)propyl)-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)triisopropylsilane (3e for Table 1)

The title compound prepared following the General Procedure described above. Yield: 84% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.92 (s, 3H), 1.03 (s, 3H), 1.06-1.10 (m, 21H), 1.30 (s, 3H), 1.26-1.33 (m, 1H), 1.48-1.55 (m, 2H), 1.66-1.74 (m, 2H), 1.75-1.85 (m, 2H), 2.24 (dt, J_1 = 3.6 Hz, J_2 = 12.4 Hz, 1H), 2.42 (dt, J_1 = 5.1 Hz, J_2 = 10.7 Hz, 1H), 3.44 (t, J = 6.6 Hz, 2H), 3.66 (s, 2H), 4.50 (s, 2H), 7.27-7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.92, 17.72, 18.53, 23.90, 25.41, 26.24, 26.79, 29.33, 31.46, 36.64, 37.88, 51.41, 70.73, 72.91, 80.12, 80.92, 84.48, 127.51, 127.60, 128.36, 138.58. HRMS (ESI^+) exact mass calcd for $C_{29}H_{51}O_3Si$ $[M+H]^+$ requires *m/z* 475.3607, found *m/z* 475.3595.



((2-(4-(Benzyloxy)butyl)-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)triisopropylsilane (3f for Table 1)

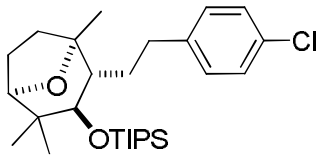
The title compound prepared following the General Procedure described above. Yield: 89% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.92 (s, 3H), 1.03 (s, 3H), 1.07-1.10 (m, 21H), 1.28 (s, 3H), 1.25-1.32 (m, 1H), 1.39-1.54 (m, 4H), 1.59-1.64 (m, 2H), 1.70-1.84 (m, 2H), 2.23 (dt, J_1 = 3.2 Hz, J_2 = 12.8 Hz, 1H), 2.41 (dt, J_1 = 5.1 Hz, J_2 = 10.7 Hz, 1H), 3.47 (t, J = 6.4 Hz, 2H), 3.66 (s, 2H), 4.49 (s, 2H), 7.27-7.36 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.30, 12.94, 17.72, 18.53, 23.91, 25.38, 26.23, 26.82, 27.89, 30.42, 32.84, 36.64, 37.89, 51.73, 70.12, 72.88, 80.13, 80.91, 84.48, 127.47, 127.58, 128.34, 138.67. HRMS (ESI^+) exact mass calcd for $C_{30}H_{53}O_3Si$ $[M+H]^+$ requires *m/z* 489.3764, found *m/z* 489.3752.



Triisopropyl((2-(4-methoxyphenethyl)-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)silane (3g for Table 1)

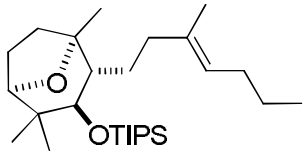
The title compound prepared following the General Procedure described above. Yield: 84% as colorless oil. ^{1}H

NMR (400 MHz, $CDCl_3$): d 0.93 (s, 3H), 1.02-1.10 (m, 24H), 1.38 (s, 3H), 1.50-1.59 (m, 3H), 1.78-1.84 (m, 1H), 2.05-2.11 (m, 1H), 2.25 (td, J_1 = 3.6 Hz, J_2 = 10.0 Hz, 1H), 2.45 (td, J_1 = 4.8 Hz, J_2 = 10.0 Hz, 1H), 2.55-2.62 (m, 1H), 2.64-2.72 (m, 1H), 3.67 (d, *J* = 7.2 Hz, 1H), 3.68 (s, 1H), 3.78 (s, 3H), 6.83 (d, *J* = 8.5 Hz, 2H), 7.09 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.96, 18.55, 23.88, 25.62, 26.27, 26.80, 35.57, 36.68, 36.72, 37.93, 51.09, 55.24, 80.57, 80.96, 84.47, 113.80, 129.19, 134.60, 157.83. HRMS (ESI^+) exact mass calcd for $C_{28}H_{49}O_3Si$ $[M+H]^+$ requires *m/z* 461.3451, found *m/z* 461.3455.



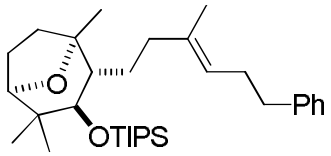
((2-(4-Chlorophenethyl)-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)triisopropylsilane (3h for Table 1)

The title compound prepared following the General Procedure described above. Yield: 85% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.93 (s, 3H), 0.99 (s, 3H), 1.10-1.10 (m, 21H), 1.37 (s, 3H), 1.50-1.59 (m, 3H), 1.77-1.86 (m, 1H), 2.04-2.14 (m, 1H), 2.21-2.28 (m, 1H), 2.41-2.48 (m, 1H), 2.58-2.74 (m, 2H), 3.66 (s, 1H), 3.67 (d, *J* = 8.2 Hz, 1H), 7.10 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.96, 18.54, 23.85, 25.62, 26.26, 26.79, 35.25, 36.63, 36.93, 37.92, 51.00, 80.59, 80.88, 84.47, 128.49, 129.66, 131.63, 140.87. HRMS (ESI^+) exact mass calcd for $C_{27}H_{46}ClO_2Si$ $[M+H]^+$ requires *m/z* 465.2956, found *m/z* 465.2953.



Triisopropyl((1,4,4-trimethyl-2-((*E*)-3-methylhept-3-en-1-yl)-8-oxabicyclo[3.2.1]octan-3-yl)oxy)silane (**3i for Table 1**)

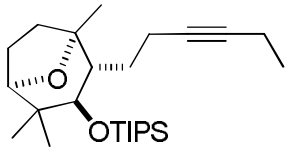
The title compound prepared following the General Procedure described above. Yield: 66% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.89 (t, *J* = 7.4 Hz, 3H), 0.93 (s, 3H), 1.05 (s, 3H), 1.09-1.11 (m, 22H), 1.28-1.38 (m, 3H), 1.31 (s, 3H), 1.46-1.55 (m, 2H), 1.60 (s, 2H), 1.75-1.89 (m, 2H), 1.95 (q, *J* = 7.2 Hz, 2H), 2.02-2.06 (m, 2H), 2.21-2.27 (m, 1H), 2.42 (dt, J_1 = 5.1 Hz, J_2 = 10.6 Hz, 1H), 3.66-3.69 (m, 2H), 5.13 (t, *J* = 7.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.95, 13.85, 16.03, 18.53, 22.96, 23.90, 25.43, 26.23, 26.80, 30.02, 31.97, 36.66, 37.90, 41.57, 51.35, 80.31, 80.98, 84.48, 124.57, 135.41. HRMS (ESI^+) exact mass calcd for $C_{27}H_{53}O_2Si$ $[M+H]^+$ requires *m/z* 437.3815, found *m/z* 437.3812.



Triisopropyl((1,4,4-trimethyl-2-((*E*)-3-methyl-6-phenylhex-3-en-1-yl)-8-oxabicyclo[3.2.1]octan-3-yl)oxy)silane (3j for Table 1)

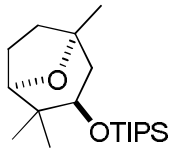
The title compound prepared following the General Procedure described above. Yield: 52% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.93 (s, 3H), 1.02-1.11 (m, 21H), 1.05 (s, 3H), 1.30 (s, 3H), 1.45-1.58 (m 2H), 1.54 (s, 3H), 1.75-1.88 (m, 3H), 2.05 (t, *J* = 8.4 Hz, 2H), 2.21-2.31 (m, 3H), 2.38-2.45 (m, 1H), 2.63 (t, *J* = 7.8 Hz,

2H), 3.67 (s, 1H), 3.68 (d, *J* = 7.9 Hz, 1H), 5.18 (t, *J* = 6.8 Hz, 1H), 7.15-7.29 (m, 3H), 7.26-7.29 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.97, 16.01, 18.55, 23.90, 25.45, 26.24, 26.82, 30.00, 31.97, 36.11, 36.67, 37.91, 41.53, 51.38, 80.35, 80.98, 84.48, 123.57, 125.69, 128.21, 128.50, 136.27, 142.33. HRMS (ESI^+) exact mass calcd for $C_{32}H_{55}O_2Si$ $[M+H]^+$ requires *m/z* 499.3971, found *m/z* 499.3975.



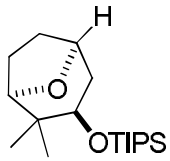
((2-(Hex-3-yn-1-yl)-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)triisopropylsilane (3k for Table 1)

The title compound prepared following the General Procedure described above. Yield: 56% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.93 (s, 3H), 1.05 (s, 3H), 1.10-1.13 (m, 24H), 1.30 (s, 3H), 1.47-1.56 (m, 3H), 1.80 (ddt, J_1 = 5.2 Hz, J_2 = 7.7 Hz, J_3 = 12.6 Hz, 1H), 1.93-2.03 (m, 1H), 2.12-2.27 (m, 5H), 2.43 (ddd, J_1 = 5.2 Hz, J_2 = 9.9 Hz, J_3 = 11.6 Hz, 1H), 3.63 (s, 1H), 3.66 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.43, 12.92, 14.26, 18.52, 19.94, 23.91, 25.38, 26.23, 26.87, 32.36, 36.61, 37.89, 50.63, 79.26, 79.83, 80.77, 81.86, 84.41. HRMS (ESI^+) exact mass calcd for $C_{25}H_{47}O_2Si$ $[M+H]^+$ requires *m/z* 407.3345, found *m/z* 407.3347.



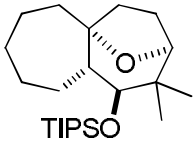
Triisopropyl((1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)silane (3l for Table 1)

The title compound prepared following the General Procedure described above. Yield: 74% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.93 (s, 3H), 1.02 (s, 3H), 1.09-1.12 (m, 21H), 1.29 (s, 3H), 1.47 (ddt, J_1 = 1.8 Hz, J_2 = 4.4 Hz, J_3 = 12.8 Hz, 1H), 1.60 (dd, J_1 = 1.2 Hz, J_2 = 14.3 Hz, 1H), 1.82 (ddt, J_1 = 4.2 Hz, J_2 = 7.9 Hz, J_3 = 12.1 Hz, 1H), 1.98 (ddd, J_1 = 1.5 Hz, J_2 = 4.5 Hz, J_3 = 14.3 Hz, 1H), 2.22 (ddd, J_1 = 4.7 Hz, J_2 = 9.9 Hz, J_3 = 12.1 Hz, 1H), 2.37 (ddd, J_1 = 4.5 Hz, J_2 = 10.1 Hz, J_3 = 12.1 Hz, 1H), 3.68 (d, *J* = 8.1 Hz, 1H), 3.73 (td, J_1 = 1.3 Hz, J_2 = 4.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.79, 18.36, 22.71, 26.26, 26.88, 26.92, 34.56, 38.16, 42.27, 74.05, 78.59, 83.74. HRMS (ESI^+) exact mass calcd for $C_{19}H_{39}O_2Si$ $[M+H]^+$ requires *m/z* 327.2719, found *m/z* 327.2722.



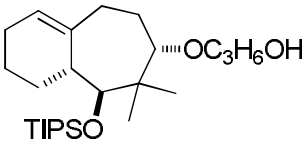
((2,2-Dimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)triisopropylsilane (3m for Table 1)

The title compound prepared following the General Procedure described above. Yield: 46% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.96 (s, 3H), 1.05 (s, 3H), 1.05-1.09 (m, 21H), 1.55 (d, *J* = 14.5 Hz, 1H), 1.72 (ddt, J_1 = 4.0 Hz, J_2 = 7.3 Hz, J_3 = 11.2 Hz, 1H), 1.81 (ddt, J_1 = 4.0 Hz, J_2 = 7.3 Hz, J_3 = 11.2 Hz, 1H), 2.11-2.22 (m, 2H), 2.34-2.40 (m, 1H), 3.69 (s, 1H), 3.69 (d, *J* = 11.9 Hz, 1H), 4.30-4.33 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.78, 18.36, 23.11, 24.85, 26.92, 28.34, 36.31, 39.02, 73.44, 73.79, 82.60. HRMS (ESI^+) exact mass calcd for $C_{18}H_{37}O_2Si$ $[M+H]^+$ requires *m/z* 313.2563, found *m/z* 313.2566.



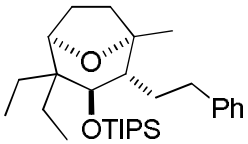
((2,2-Dimethyldecahydro-1*H*-3,5a-epoxyheptalen-1-yl)oxy)triisopropylsilane (3n for Table 1)

The title compound prepared following the General Procedure described above. Yield: 54% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.92 (s, 3H), 1.00 (s, 3H), 1.06-1.11 (m, 21H), 1.25-1.34 (m, 3H), 1.37-1.38 (m, 1H), 1.46 (dt, J_1 = 4.0 Hz, J_2 = 12.0 Hz, 1H), 1.65-1.73 (m, 2H), 1.75-1.90 (m, 6H), 2.28 (dt, J_1 = 4.1 Hz, J_2 = 11.2 Hz, 1H), 2.41 (dt, J_1 = 4.8 Hz, J_2 = 11.2 Hz, 1H), 3.54 (s, 1H), 3.63 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.94, 18.46, 22.38, 23.48, 26.26, 26.42, 30.59, 30.78, 32.21, 37.39, 37.70, 41.08, 52.91, 82.76, 83.06, 83.39. HRMS (ESI$^+$) exact mass calcd for $C_{23}H_{45}O_2Si$ $[M+H]^+$ requires *m/z* 381.3189, found *m/z* 381.3194.



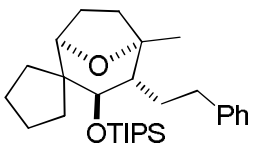
3-((8,8-Dimethyl-9-((triisopropylsilyl)oxy)-2,3,5,6,7,8,9,9a-octahydro-1*H*-benzo[7]annulen-7-yl)oxy)propan-1-ol (3o for Table 1)

The title compound prepared following the General Procedure described above. Yield: 57% as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.84 (s, 3H), 1.11-1.16 (m, 21H), 1.16 (s, 3H), 1.36-1.57 (m, 3H), 1.70-1.91 (m, 4H), 1.97 (s, 2H), 2.05-2.15 (m, 2H), 2.25 (t, J = 12.0 Hz, 1H), 2.49 (d, J = 11.9 Hz, 1H), 2.92 (brs, 1H), 3.53-3.60 (m, 2H), 3.68 (s, 1H), 3.72-3.78 (m, 3H), 5.37 (d, J = 3.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 13.02, 18.19, 18.52, 23.75, 25.51, 29.10, 30.90, 31.47, 32.23, 32.79, 44.79, 48.60, 63.07, 70.95, 82.79, 84.68, 122.52, 140.90. HRMS (ESI$^+$) exact mass calcd for $C_{25}H_{49}O_3Si$ $[M+H]^+$ requires *m/z* 425.3451, found *m/z* 425.3452.



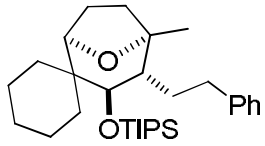
((4,4-Diethyl-1-methyl-2-phenethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)triisopropylsilane (3ab for Table 2)

The title compound prepared following the General Procedure described above. Yield: 85% as colorless oil. Dr: 90:10. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.73 (t, J = 7.6 Hz, 3H), 0.80 (t, J = 7.6 Hz, 3H), 1.03-1.14 (m, 21H), 1.38 (s, 3H), 1.30-1.47 (m, 2H), 1.50-1.69 (m, 5H), 1.76-1.86 (m, 1H), 2.06-2.15 (m, 1H), 2.21-2.27 (m, 1H), 2.41-2.48 (m, 1H), 2.60-2.77 (m, 2H), 3.70 (s, 0.10H for minor isomer), 3.80 (s, 0.90H for major isomer), 3.88 (d, J = 7.77 Hz, 1H), 7.16-7.20 (m, 3H), 7.25-7.30 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 6.73, 8.37, 13.09, 18.60, 22.30, 24.20, 25.64, 25.79, 35.20, 37.94, 41.90, 51.65, 76.82, 80.87, 81.84, 125.91, 128.31, 128.42, 142.44. HRMS (ESI$^+$) exact mass calcd for $C_{29}H_{51}O_2Si$ $[M+H]^+$ requires *m/z* 459.3658, found *m/z* 459.3673.



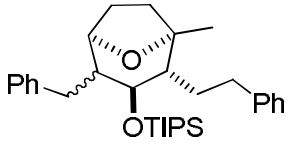
Triisopropyl((5-methyl-4-phenethyl-8-oxaspiro[bicyclo[3.2.1]octane-2,1'-cyclopentan]-3-yl)oxy)silane (3ac for Table 2)

The title compound prepared following the General Procedure described above. Yield: 87% as colorless oil. Dr: 96:4. ^{1}H NMR (400 MHz, $CDCl_3$): d 1.02-1.13 (m, 21H), 1.36 (s, 3H), 1.26-1.41 (m, 1H), 1.48-1.66 (m, 8H), 1.77-1.91 (m, 2H), 1.97-2.11 (m, 2H), 2.22-2.29 (m, 1H), 2.52 (ddd, J_1 = 4.9 Hz, J_2 = 10.0 Hz, J_3 = 11.5 Hz, 1H), 2.63-2.73 (m, 2H), 3.77 (s, 1H), 3.80 (d, J = 7.8 Hz, 1H), 7.16-7.20 (m, 3H), 7.25-7.30 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.95, 18.56, 23.90, 25.36, 25.72, 26.98, 33.03, 33.10, 36.50, 36.94, 37.15, 50.47, 50.99, 80.40, 81.24, 81.70, 125.88, 128.31, 128.40, 142.53. HRMS (ESI$^+$) exact mass calcd for $C_{29}H_{49}O_2Si$ $[M+H]^+$ requires *m/z* 457.3502, found *m/z* 457.3514.



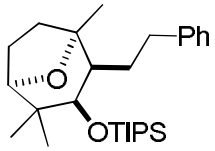
Triisopropyl((5-methyl-4-phenethyl-8-oxaspiro[bicyclo[3.2.1]octane-2,1'-cyclohexan]-3-yl)oxy)silane (3ad for Table 2)

The title compound prepared following the General Procedure described above. Yield: 91% as colorless oil. Dr: 93:7. ^{1}H NMR (400 MHz, $CDCl_3$): d 1.03-1.15 (m, 21H), 1.23-1.34 (m, 4H), 1.39 (s, 3H), 1.41-1.64 (m, 8H), 1.79-1.88 (m, 1H), 1.93 (d, J = 12.8 Hz, 1H), 2.03-2.13 (m, 1H), 2.24-2.30 (m, 1H), 2.42-2.49 (m, 1H), 2.68 (dtd, J_1 = 5.4 Hz, J_2 = 13.2 Hz, J_3 = 19.2 Hz, 2H), 3.68 (s, 1H), 4.26 (d, J = 7.8 Hz, 1H), 7.16-7.20 (m, 3H), 7.26-7.30 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 13.13, 18.62, 21.46, 21.60. 25.46, 25.59, 26.44, 31.68, 33.71, 35.50, 36.90, 37.96, 39.80, 51.63, 79.25, 81.29, 81.42, 125.89, 128.30, 128.40, 142.52. HRMS (ESI$^+$) exact mass calcd for $C_{30}H_{51}O_2Si$ $[M+H]^+$ requires *m/z* 471.3658, found *m/z* 471.3656.



4-Benzyl-1-methyl-2-phenethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)triisopropylsilane (3ae for Table 2)

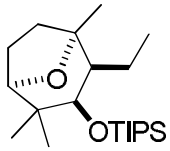
The title compound prepared following the General Procedure described above. Yield: 96% as colorless oil. Dr: 59:41. ^{1}H NMR (400 MHz, $CDCl_3$): d 1.00-1.17 (m, 21H), 1.32 (s, 1.8H) for major isomer, 1.40 (s, 1.2H) for minor isomer, 1.44-1.67 (m, 3H), 1.71-1.83 (m, 1H), 1.88-1.99 (m, 0.6H), 2.05-2.24 (m, 2H), 2.32-2.38 (m, 0.6H), 2.52-2.61 (m, 1H), 2.65-2.79 (m, 3.6H), 2.85 (dd, J_1 = 10.9 Hz, J_2 = 13.0 Hz, 0.4H), 3.87 (dd, J_1 = 2.9 Hz, J_2 = 7.4 Hz, 0.6H), 3.92 (s, 0.4H), 4.07 (d, J = 3.5 Hz, 0.6H), 4.11 (d, J = 7.6 Hz, 0.4H), 7.14-7.32 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.18, 12.89, 18.27, 18.57, 25.38, 25.58, 25.82, 30.04, 32.28, 33.43, 35.48, 36.14, 36.31, 36.46, 37.01, 38.43, 42.04, 49.27, 50.32, 51.20, 75.64, 75.80, 76.87, 80.94, 81.43, 125.93, 125.98, 126.02, 128.33, 128.38, 128.48, 128.94, 129.40, 140.19, 140.90, 142.25, 142.47. HRMS (ESI$^+$) exact mass calcd for $C_{31}H_{47}O_2Si$ $[M+H]^+$ requires *m/z* 479.3345, found *m/z* 479.3353.



Triisopropyl((1,4,4-trimethyl-2-phenethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)silane (5a for Table 4)

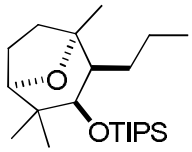
The title compound prepared following the General Procedure described above. Yield: 51% as colorless oil. Dr: 97:3. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.96 (s, 3H), 1.05 (s, 3H), 1.07-1.17 (m, 21H), 1.27 (d, J = 12.3 Hz, 1H), 1.29 (s, 3H), 1.49-1.51 (m, 1H), 1.80-1.87 (m, 3H), 2.28-2.35 (m, 2H), 2.68-2.73 (m, 2H), 3.64 (d, J = 8.0 Hz,

1H), 3.69 (d, *J* = 2.5 Hz, 1H), 7.14-7.19 (m, 3H), 7.25-7.29 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 14.07, 18.81, 18.82, 23.51, 25.90, 26.97, 27.45, 29.35, 31.48, 36.33, 39.35, 46.51, 78.89, 82.74, 83.26, 125.78, 128.29, 128.31, 142.34. HRMS (ESI$^+$) exact mass calcd for $C_{27}H_{47}O_2Si$ $[M+H]^+$ requires *m/z* 431.3345, found *m/z* 431.3347.



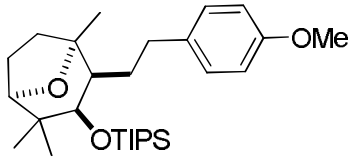
((2-Ethyl-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)triisopropylsilane (5b for Table 4)

The title compound prepared following the General Procedure described above. Yield: 53% as colorless oil. Dr: 93:7. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.96 (s, 3H), 0.99 (t, *J* = 7.6 Hz, 3H), 1.06 (s, 3H), 1.12-1.16 (m, 21H), 1.17-1.26 (m, 2H), 1.30 (s, 3H), 1.40-1.55 (m, 1H), 1.63-1.66 (m, 1H), 1.76-1.87 (m, 1H), 2.21-2.45 (m, 2H), 3.63 (d, *J* = 8.0 Hz, 1H), 3.70 (d, *J* = 3.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.91, 14.01, 15.07, 18.51, 18.78, 18.83, 20.49, 23.50, 25.94, 26.94, 27.50, 31.34, 39.29, 49.46, 78.63, 82.81, 83.23. HRMS (ESI$^+$) exact mass calcd for $C_{21}H_{43}O_2Si$ $[M+H]^+$ requires *m/z* 355.3032, found *m/z* 355.3038.



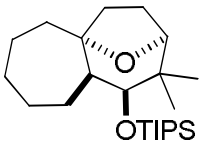
Triisopropyl((1,4,4-trimethyl-2-propyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)silane (5c for Table 4)

The title compound prepared following the General Procedure described above. Yield: 58% as colorless oil. Dr: 93:7. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.91 (t, *J* = 7.11 Hz, 3H), 0.95 (s, 3H), 1.06 (s, 3H), 1.13-1.26 (m, 22H), 1.28 (s, 3H), 1.33-1.51 (m, 4H), 1.69-1.73 (m, 1H), 1.78-1.88 (m, 1H), 2.20-2.44 (m, 2H), 3.63 (d, *J* = 7.1 Hz, 1H), 3.67 (d, *J* = 4.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 14.05, 14.46, 18.51, 18.75, 18.83, 23.54, 23.60, 25.84, 26.96, 27.50, 30.02, 31.37, 39.30, 47.12, 79.10, 82.78, 83.22. HRMS (ESI$^+$) exact mass calcd for $C_{22}H_{45}O_2Si$ $[M+H]^+$ requires *m/z* 369.3189, found *m/z* 369.3192.



Triisopropyl((2-(4-methoxyphenethyl)-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-yl)oxy)silane (5d for Table 4)

The title compound prepared following the General Procedure described above. Yield: 56% as colorless oil. Dr: 95:5. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.95 (s, 3H), 1.05 (s, 3H), 1.07-1.17 (m, 21H), 1.26-1.28 (m, 1H), 1.30 (s, 3H), 1.43-1.51 (m, 1H), 1.76-1.87 (m, 3H), 2.25-2.34 (m, 2H), 2.62-2.66 (m, 2H), 3.63 (d, *J* = 7.9 Hz, 1H), 3.68 (d, *J* = 2.6 Hz, 1H), 3.79 (s, 3H), 6.82 (d, *J* = 8.6 Hz, 2H), 7.07 (d, *J* = 8.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 14.07, 18.82, 23.51, 25.89, 26.69, 27.45, 29.51, 31.48, 35.39, 39.34, 46.43, 55.26, 78.87, 82.75, 83.26, 113.71, 129.17, 134.46, 157.70. HRMS (ESI$^+$) exact mass calcd for $C_{28}H_{49}O_3Si$ $[M+H]^+$ requires *m/z* 461.3451, found *m/z* 461.3448.

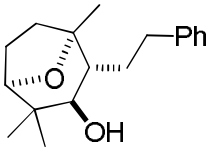


((2,2-Dimethyldecahydro-1*H*-3,5a-epoxyheptalen-1-yl)oxy)triisopropylsilane (5f for Table 1)

The title compound prepared following the General Procedure described above. Yield: 55% for two isomers, 26% yield for the title compound. (another isomer data see **3n**) ^{1}H NMR (400 MHz, $CDCl_3$): d 0.94 (s, 3H), 1.07 (s, 3H), 1.11-1.15 (m, 22H), 1.25-1.38 (m, 5H), 1.49-1.58 (m, 1H), 1.61-1.64 (m, 1H), 1.75-1.87 (m, 4H), 1.92-1.93 (m, 1H), 2.27-2.32 (m, 1H), 2.36-2.42 (m, 1H), 3.67 (s, 1H), 3.67 (d, J = 10.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 13.97, 18.77, 21.98, 23.07, 26.11, 27.69, 27.88, 28.04, 28.66, 31.48, 39.21, 39.61, 47.88, 80.69, 83.74, 85.02. HRMS (ESI$^+$) exact mass calcd for $C_{23}H_{45}O_2Si$ $[M+H]^+$ requires *m/z* 381.3189, found *m/z* 381.3194.

Procedure for removing TIPS group

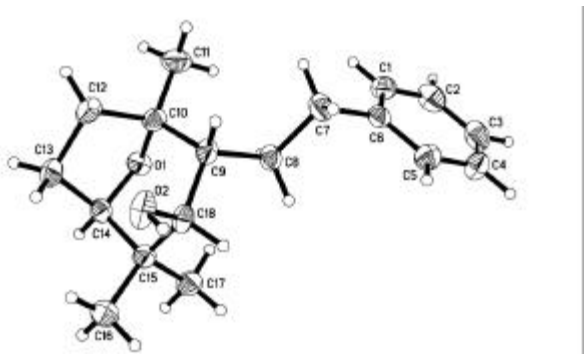
To a solution of corresponding ether (0.1 mmol) in 5 mL THF was added TBAF (1.0 M in THF, 0.5 mL, 3.0 equiv) at 0 °C. The reaction mixture was stirred at room temperature for 12 h. the reaction was quenched with half saturated NH_4Cl (5 mL) and extracted with EA (3 x 10 mL). The combined organic layers were washed with brine (25 mL) and dried over Na_2SO_4, then concentrated in vacuo and purified by chromatography with hexane/EA as eluent gave desired alcohols in 88-94% yields



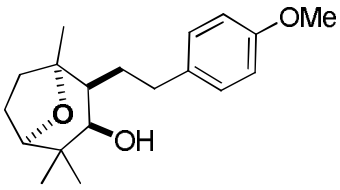
1,4,4-Trimethyl-2-phenethyl-8-oxabicyclo[3.2.1]octan-3-ol (3a' for Scheme 1)

The title compound prepared following the General Procedure described above. Yield: 94% as white solid, bp: 108-110 °C. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.96 (s, 3H), 1.10 (s, 3H), 1.34 (s, 3H), 1.48-1.51 (m, 2H), 1.55-1.71 (m, 2H), 1.84 (ddt, J_1 = 4.9 Hz, J_2 = 7.6 Hz, J_3 = 12.4 Hz, 1H), 2.05 (ddd, J_1 = 5.8 Hz, J_2 = 11.0 Hz, J_3 = 15.7 Hz, 1H), 2.17-2.32 (m, 2H), 2.62-2.77 (m, 2H), 3.59 (s, 1H), 3.73 (d, J = 7.7Hz, 1H), 7.17-7.19 (m, 3H), 7.26-7.31 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 22.73, 25.11, 26.09, 26.82, 33.25, 35.99, 37.26, 37.39, 76.72, 80.80, 83.83, 125.88, 128.34, 128.44, 142.33. HRMS (ESI$^+$) exact mass calcd for $C_{18}H_{27}O_2$ $[M+H]^+$ requires *m/z* 275.2011, found *m/z* 275.2003.

X-ray crystal structure analysis of 3a'



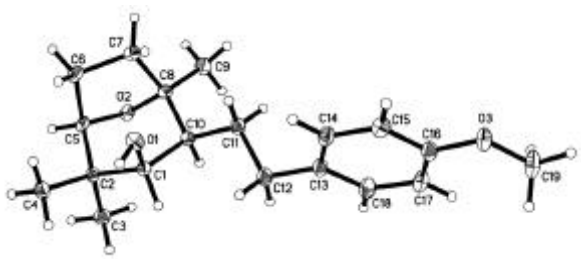
CCDC-873237 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



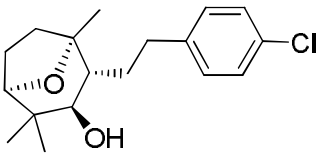
2-(4-Methoxyphenethyl)-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-ol (5d' for Figure 2)

The title compound prepared following the General Procedure described above. Yield: 89% as white solid. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.98 (s, 3H), 1.05 (s, 3H), 1.26 (s, 3H), 1.29 (t, J = 11.6 Hz, 1H), 1.40 (d, J = 3.6 Hz, 1H), 1.58-1.66 (m, 1H), 1.71-1.89 (m, 3H), 2.21 (dtd, J_1 = 4.1 Hz, J_2 = 9.8 Hz, J_3 = 21.4 Hz, 2H), 2.45 (ddd, J_1 = 6.5 Hz, J_2 = 9.7 Hz, J_3 = 13.6 Hz, 1H), 2.72 (ddd, J_1 = 5.1 Hz, J_2 = 10.5 Hz, J_3 = 13.6 Hz, 1H), 3.51 (s, 1H), 3.71 (d, J = 7.9 Hz, 1H), 3.79 (s, 3H), 6.83 (d, J = 8.5 Hz, 2H), 7.12 (d, J = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 22.08, 25.54, 26.57, 26.70, 28.89, 31.21, 32.06, 38.43, 45.18, 55.28, 73.45, 82.18, 82.71, 113.82, 129.17, 134.77, 157.76. HRMS (ESI^+) exact mass calcd for $C_{19}H_{29}O_3$ $[M+H]^+$ requires *m/z* 305.2117, found *m/z* 305.2130.

X-ray crystal structure analysis of 5d'



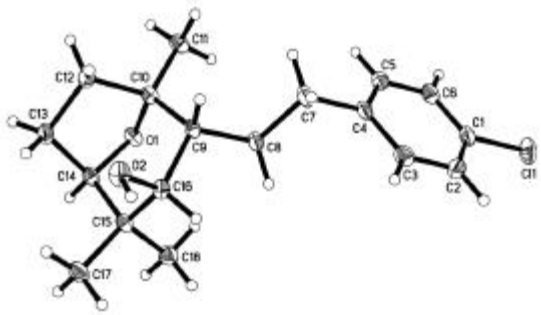
CCDC-873238 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



(1*S*,2*R*,3*R*,5*R*)-2-(4-chlorophenethyl)-1,4,4-trimethyl-8-oxabicyclo[3.2.1]octan-3-ol (3h')

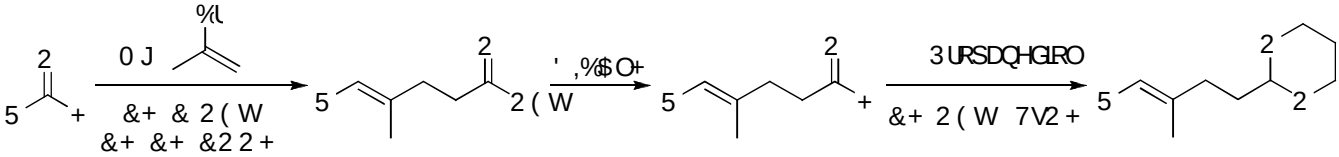
The title compound prepared following the General Procedure described above. Yield: 88% as white solid, bp: 112-114 °C. ^{1}H NMR (400 MHz, $CDCl_3$): d 0.95 (s, 3H), 1.09 (s, 3H), 1.33 (s, 3H), 1.46-1.50 (m, 2H), 1.55-1.69 (m, 2H), 1.84 (ddt, J_1 = 4.8 Hz, J_2 = 7.7 Hz, J_3 = 12.6 Hz, 1H), 2.01 (ddd, J_1 = 5.8 Hz, J_2 = 10.9 Hz, J_3 = 15.8 Hz, 1H), 2.24 (dtd, J_1 = 4.3 Hz, J_2 = 9.9 Hz, J_3 = 13.2 Hz, 2H), 2.58-2.73 (m, 2H), 3.55 (s, 1H), 3.73 (d, J = 7.7 Hz, 1H), 7.11 (d, J = 8.3 Hz, 2H), 7.25 (d, J = 8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 22.70, 25.11, 26.08, 26.81, 33.23, 35.31, 37.21, 37.39, 50.49, 77.17, 80.73, 83.82, 128.53, 129.67, 131.57, 140.73. HRMS (ESI^+) exact mass calcd for $C_{18}H_{26}ClO_2$ $[M+H]^+$ requires *m/z* 309.1621, found *m/z* 309.1611.

X-ray crystal structure analysis of 3h'



CCDC-873239 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

4 Experimental Data for Reaction Precursors

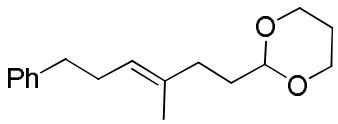


To a solution of corresponding aldehyde (30 mmol) in THF (30 mL) was added 2-propenylmagnesium bromide (1.0 M in THF, 45 mL) dropwise at 0 °C. The mixture was stirred at room temperature for 30 mins, followed by quenching with saturated NH_4Cl solution (50 mL). The ultimate reaction mixture was extracted with Et_2O (2 × 100 mL), washed with brine (100 mL), dried over anhydrous $MgSO_4$, concentrated in *vacuo* provided the allylic alcohols as colorless oil.

A solution of the allylic alcohol (30 mmol) and propanoic acid (1 mL) in triethyl orthoacetate (50 mL) was heated at 145 °C for 2 hours and enthanol was then distilled out. The reaction was cooled to room temperature and washed with saturated $NaHCO_3$ solution (100 mL) and brine (100 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo* to yield the ester as colourless oil in 85-96% yields.

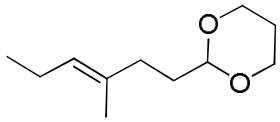
To a solution of the above prepared esters (10 mmol) in 25 mL dry dichloromethane was added DIBAL-H (1 M in heptane, 12 mL, 1.2 equiv) dropwise at -78 °C. The mixture was stirred at that temperature for 2.5 h, then quenched by adding MeOH (5 mL) and saturated aqueous solution of Rochelle's salt (15 mL). The resulting mixture was stirred vigorously until two clear layers were obtained (about 2 h) at room temperature. The two layers were separated and the aqueous layer was extracted with Et_2O (3 × 20 mL). The combined organic layers were washed with brine (40 mL), dried over $MgSO_4$, filtered, and concentrated *in vacuo* to obtain crude aldehyde**s**. Flash column chromatography with hexane/EA as the eluent afforded the desired products as colorless oil in 75-86% yields.

To a solution of aldehyde (2 mmol), TsOH (0.10 equiv) and 1,3-propanediol in 15 mL toluene was added triethylorthoformate (6 mmol, 3.0 equiv) at 0 °C. The reaction mixture was stirred for 12 h at room temperature. The reaction was then quenched with triethyl amine (0.5 mL) and concentrated *in vacuo*. The crude product was purified by flash column chromatography with hexane/EA as the eluent provided the desired products as colorless oil in 78-93% yields.



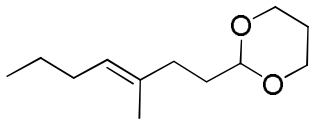
(*E*)-2-(3-methyl-6-phenylhex-3-en-1-yl)-1,3-dioxane (1a for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.32 (d, *J* = 13.4 Hz, 1H), 1.54 (s, 3H), 1.64-1.69 (m, 2H), 2.03-2.13 (m, 3H), 2.30 (q, *J* = 7.4 Hz, 2H), 2.63 (t, *J* = 7.7 Hz, 2H), 3.73 (t, *J* = 12.2 Hz, 2H), 4.09 (dd, J_1 = 5.5 Hz, J_2 = 11.2 Hz, 2H), 4.45 (t, *J* = 5.2 Hz, 1H), 5.20 (t, *J* = 7.1 Hz, 1H), 7.15-7.19 (m, 3H), 7.25-7.28 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 15.95, 25.87, 29.84, 33.60, 33.83, 36.04, 66.89, 102.05, 123.85, 125.67, 128.22, 128.49, 135.07, 142.31. HRMS (ESI$^+$) exact mass calcd for $C_{17}H_{25}O_2$ $[M+H]^+$ requires *m/z* 261.1855, found *m/z* 261.1852.



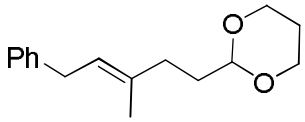
(*E*)-2-(3-methylhex-3-en-1-yl)-1,3-dioxane (1b for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 0.93 (t, *J* = 7.5 Hz, 3H), 1.34 (d, *J* = 13.4 Hz, 1H), 1.59 (s, 3H), 1.65-1.71 (m, 2H), 1.95-2.14 (m, 5H), 3.75 (t, *J* = 12.2 Hz, 2H), 4.10 (dd, J_1 = 4.9 Hz, J_2 = 11.7 Hz, 2H), 4.49 (t, *J* = 5.2 Hz, 1H), 5.14 (t, *J* = 7.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 14.34, 15.78, 21.15, 25.87, 33.66, 33.78, 66.90, 102.13, 126.66, 133.52. HRMS (ESI$^+$) exact mass calcd for $C_{11}H_{21}O_2$ $[M+H]^+$ requires *m/z* 185.1542, found *m/z* 185.1543.



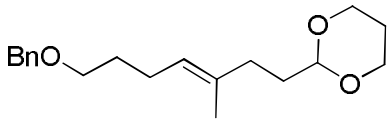
(*E*)-2-(3-methylhept-3-en-1-yl)-1,3-dioxane (1c for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 0.88 (t, *J* = 7.3 Hz, 3H), 1.30-1.39 (m, 3H), 1.59 (s, 3H), 1.67-1.72 (m, 2H), 1.95 (q, *J* = 7.2 Hz, 2H), 2.02-2.14 (m, 3H), 3.75 (t, *J* = 12.3 Hz, 2H), 4.10 (dd, J_1 = 4.9 Hz, J_2 = 11.0 Hz, 2H), 4.49 (t, *J* = 5.2 Hz, 1H), 5.15 (t, *J* = 6.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 13.78, 15.95, 22.95, 25.88, 29.97, 33.69, 33.89, 66.92, 102.14, 124.86, 134.21. HRMS (ESI$^+$) exact mass calcd for $C_{12}H_{23}O_2$ $[M+H]^+$ requires *m/z* 199.1698, found *m/z* 199.1696.



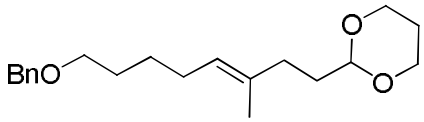
(*E*)-2-(3-methyl-5-phenylpent-3-en-1-yl)-1,3-dioxane (1d for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.32 (d, *J* = 13.4 Hz, 1H), 1.71 (s, 3H), 1.73-1.75 (m, 2H), 2.01-2.11 (m, 1H), 2.13 (t, *J* = 7.2 Hz, 2H), 3.35 (d, *J* = 7.3 Hz, 2H), 3.72 (t, *J* = 12.3 Hz, 2H), 4.08 (dd, J_1 = 4.9 Hz, J_2 = 11.2 Hz, 2H), 4.49 (t, *J* = 5.2 Hz, 1H), 5.37 (t, *J* = 7.3 Hz, 1H), 7.16-7.17 (m, 3H), 7.25-7.28 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 16.13, 25.87, 33.63, 33.88, 34.17, 66.91, 102.02, 123.29, 125.70, 128.31, 128.33, 135.53, 141.69. HRMS (ESI$^+$) exact mass calcd for $C_{16}H_{23}O_2$ $[M+H]^+$ requires *m/z* 247.1698, found *m/z* 247.1692.



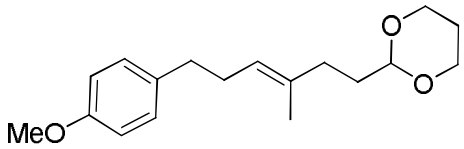
(*E*)-2-(7-(benzyloxy)-3-methylhept-3-en-1-yl)-1,3-dioxane (1e for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.33 (d, *J* = 13.5 Hz, 1H), 1.60 (s, 3H), 1.65-1.71 (m, 4H), 2.03-2.10 (m, 5H), 3.46 (t, *J* = 6.5 Hz, 2H), 3.74 (t, *J* = 12.0 Hz, 2H), 4.10 (dd, J_1 = 4.8 Hz, J_2 = 11.4 Hz, 2H), 4.47-4.49 (m, 3H), 5.14 (t, *J* = 7.1 Hz, 1H), 7.26-7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): d 15.96, 24.45, 25.87, 29.83, 33.67, 33.84, 66.92, 69.91, 72.93, 102.10, 124.11, 127.48, 127.64, 128.35, 134.86, 138.68. HRMS (ESI^+) exact mass calcd for $C_{19}H_{29}O_3$ $[M+H]^+$ requires *m/z* 305.2117, found *m/z* 305.2120.



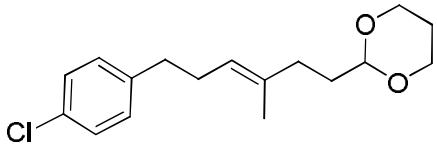
(*E*)-2-(8-(benzyloxy)-3-methyloct-3-en-1-yl)-1,3-dioxane (1f for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.31 (d, *J* = 13.4 Hz, 1H), 1.37-1.44 (m, 2H), 1.58 (s, 3H), 1.59-1.63 (m, 2H), 1.66-1.71 (m, 2H), 1.99 (q, *J* = 7.2 Hz, 2H), 2.02-2.13 (m, 1H), 2.05 (t, *J* = 8.0 Hz, 2H), 3.46 (t, *J* = 6.5 Hz, 2H), 3.74 (t, *J* = 12.0 Hz, 2H), 4.09 (dd, J_1 = 4.8 Hz, J_2 = 11.4 Hz, 2H), 4.48 (t, *J* = 5.2 Hz, 1H), 4.49 (s, 2H), 5.14 (t, *J* = 7.0 Hz, 1H), 7.25-7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): d 15.98, 25.88, 26.36, 27.66, 29.37, 33.67, 33.87, 66.91, 70.41, 72.88, 102.10, 124.66, 127.47, 127.63, 128.35, 134.42, 138.71. HRMS (ESI^+) exact mass calcd for $C_{20}H_{31}O_3$ $[M+H]^+$ requires *m/z* 319.2273, found *m/z* 319.2276.



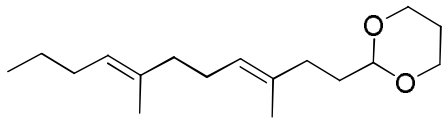
(*E*)-2-(6-(4-methoxyphenyl)-3-methylhex-3-en-1-yl)-1,3-dioxane (1g for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.33 (d, *J* = 13.4 Hz, 1H), 1.54 (s, 3H), 1.64-1.70 (m, 2H), 2.01-2.13 (m, 3H), 2.27 (q, *J* = 7.4 Hz, 2H), 2.57 (t, *J* = 7.7 Hz, 2H), 3.73 (t, *J* = 12.2 Hz, 2H), 3.79 (s, 3H), 4.09 (dd, J_1 = 4.9 Hz, J_2 = 11.6 Hz, 2H), 4.46 (t, *J* = 5.2 Hz, 1H), 5.18 (t, *J* = 7.1 Hz, 1H), 6.81 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 15.97, 25.87, 30.07, 33.60, 33.82, 35.10, 55.25, 66.90, 102.06, 113.64, 123.92, 129.34, 134.43, 134.95, 157.67. HRMS (ESI^+) exact mass calcd for $C_{18}H_{27}O_3$ $[M+H]^+$ requires *m/z* 291.1960, found *m/z* 291.1955.



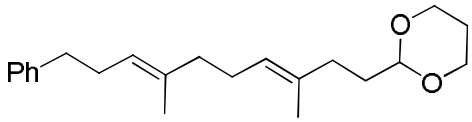
(*E*)-2-(6-(4-chlorophenyl)-3-methylhex-3-en-1-yl)-1,3-dioxane (1h for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.33 (d, *J* = 13.4 Hz, 1H), 1.52 (s, 3H), 1.63-1.69 (m, 2H), 2.04 (t, *J* = 8.0 Hz, 2H), 2.06-2.13 (m, 1H), 2.27 (q, *J* = 7.4 Hz, 2H), 2.60 (t, *J* = 7.6 Hz, 2H), 3.72 (t, *J* = 11.9 Hz, 2H), 4.09 (dd, J_1 = 4.8 Hz, J_2 = 11.3 Hz, 2H), 4.44 (t, *J* = 5.2 Hz, 1H), 5.15 (t, *J* = 7.0 Hz, 1H), 7.10 (d, *J* = 8.2 Hz, 2H), 7.22 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 15.98, 25.86, 29.62, 33.60, 33.78, 35.31, 66.89, 102.00, 123.36, 128.28, 129.85, 131.36, 135.45, 140.68. HRMS (ESI^+) exact mass calcd for $C_{17}H_{24}ClO_2$ $[M+H]^+$ requires *m/z* 295.1465, found *m/z* 295.1468.



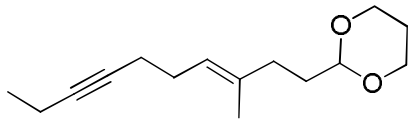
2-((3*E*,7*E*)-3,7-dimethylundeca-3,7-dien-1-yl)-1,3-dioxane (1i for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 0.89 (t, *J* = 7.3 Hz, 3H), 1.30-1.37 (m, 3H), 1.59 (s, 6H), 1.65-1.71 (m, 2H), 1.93-2.11 (m, 9H), 3.75 (t, *J* = 12.4 Hz, 2H), 4.10 (dd, J_1 = 4.9 Hz, J_2 = 11.0 Hz, 2H), 4.48 (t, *J* = 5.2 Hz, 1H), 5.13 (t, *J* = 6.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 13.83, 15.98, 16.00, 23.00, 25.88, 26.63, 30.02, 33.64, 33.84, 33.69, 66.91, 102.13, 124.57, 124.63, 134.12, 134.90. HRMS (ESI^+) exact mass calcd for $C_{17}H_{31}O_2$ $[M+H]^+$ requires *m/z* 267.2324, found *m/z* 267.2329.



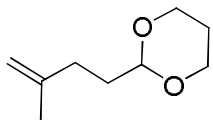
2-((3*E*,7*E*)-3,7-dimethyl-10-phenyldeca-3,7-dien-1-yl)-1,3-dioxane (1j for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.33 (d, *J* = 13.4 Hz, 1H), 1.55 (s, 3H), 1.59 (s, 3H), 1.66-1.71 (m, 2H), 1.96-2.14 (m, 7H), 2.30 (q, *J* = 7.4 Hz, 2H), 2.63 (t, *J* = 7.8 Hz, 2H), 3.74 (t, *J* = 11.4 Hz, 2H), 4.10 (dd, J_1 = 4.8 Hz, J_2 = 11.4 Hz, 2H), 4.49 (t, *J* = 5.2 Hz, 1H), 5.12 (t, *J* = 6.5 Hz, 1H), 5.18 (t, *J* = 6.9 Hz, 1H), 7.15-7.20 (m, 3H), 7.25-7.29 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 15.99, 25.88, 26.60, 29.99, 33.70, 33.84, 36.15, 39.65, 66.92, 102.13, 123.64, 124.48, 125.65, 128.22, 128.48, 134.24, 135.73, 142.43. HRMS (ESI^+) exact mass calcd for $C_{22}H_{33}O_2$ $[M+H]^+$ requires *m/z* 329.2481, found *m/z* 329.2485.



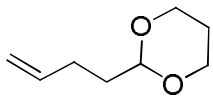
(*E*)-2-(3-methyldec-3-en-7-yn-1-yl)-1,3-dioxane (1k for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.11 (t, *J* = 7.3 Hz, 3H), 1.34 (d, *J* = 13.4 Hz, 1H), 1.62 (s, 3H), 1.67-1.72 (m, 2H), 2.05-2.20 (m, 9H), 3.76 (t, *J* = 12.2 Hz, 2H), 4.10 (dd, J_1 = 5.0 Hz, J_2 = 10.8 Hz, 2H), 4.50 (t, *J* = 5.2 Hz, 1H), 5.19 (t, *J* = 6.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 12.43, 14.33, 16.06, 19.20, 25.87, 27.80, 33.52, 33.81, 66.90, 79.32, 81.63, 102.04, 123.29, 135.50. HRMS (ESI^+) exact mass calcd for $C_{15}H_{25}O_2$ $[M+H]^+$ requires *m/z* 237.1855, found *m/z* 237.1865.



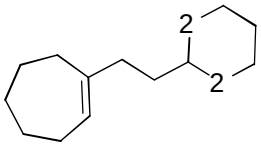
2-(3-Methylbut-3-en-1-yl)-1,3-dioxane (1l for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.34 (d, *J* = 13.5 Hz, 1H), 1.71-1.76 (m, 2H), 1.73 (s, 3H), 2.02-2.14 (m, 3H), 3.76 (dt, J_1 = 2.3 Hz, J_2 = 12.3 Hz, 2H), 4.11 (dd, J_1 = 5.0 Hz, J_2 = 10.8 Hz, 2H), 4.53 (t, *J* = 5.2 Hz, 1H), 4.69 (s, 1H), 4.71 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 22.55, 25.85, 31.92, 33.25, 66.91, 101.96, 109.91 145.20. HRMS (ESI^+) exact mass calcd for $C_9H_{17}O_2$ $[M+H]^+$ requires *m/z* 157.1229, found *m/z* 157.1223.



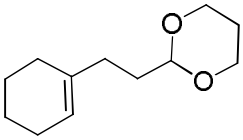
2-(But-3-en-1-yl)-1,3-dioxane (1m for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.34 (d, *J* = 13.4 Hz, 1H), 1.66-1.72 (m, 2H), 2.02-2.18 (m, 3H), 3.76 (dt, J_1 = 2.2 Hz, J_2 = 12.3 Hz, 2H), 4.11 (dd, J_1 = 5.0 Hz, J_2 = 10.9 Hz, 2H), 4.53 (t, *J* = 5.2 Hz, 1H), 4.96 (d, *J* = 10.2 Hz, 1H), 5.03 (d, *J* = 17.1 Hz, 1H), 5.82 (tdd, J_1 = 6.6 Hz, J_2 = 10.2 Hz, J_3 = 16.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 25.84, 28.15, 34.31, 66.90, 101.73, 114.74, 138.03. HRMS (ESI^+) exact mass calcd for $C_8H_{14}O_2Na$ $[M+Na]^+$ requires *m/z* 165.0891, found *m/z* 165.0896.



2-(2-(Cyclohept-1-en-1-yl)ethyl)-1,3-dioxane (1n for Table 1)

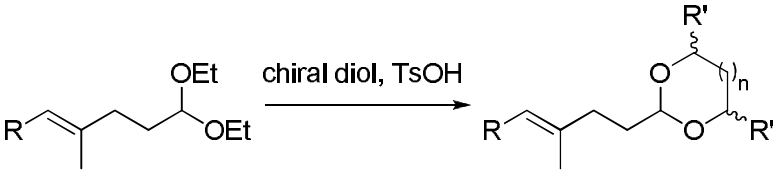
^{1}H NMR (400 MHz, $CDCl_3$): d 1.34 (d, J = 13.4 Hz, 1H), 1.41-1.48 (m, 4H), 1.64-1.73 (m, 4H), 2.03-2.10 (m, 7H), 3.75 (dt, J_1 = 2.4 Hz, J_2 = 12.4 Hz, 2H), 4.10 (dd, J_1 = 4.8 Hz, J_2 = 10.4 Hz, 2H), 4.50 (t, J = 5.6 Hz, 1H), 5.55 (t, J = 10.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): 25.87, 26.83, 27.34, 28.28, 32.71, 32.77, 33.75, 34.38, 66.87, 102.13, 126.02, 143.91. HRMS (ESI^+) exact mass calcd for $C_{13}H_{23}O_2$ $[M+H]^+$ requires *m/z* 211.1698, found *m/z* 211.1701.



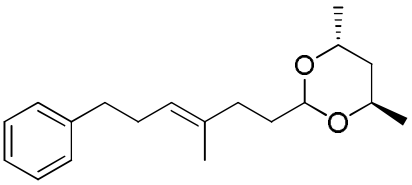
2-(2-(Cyclohex-1-en-1-yl)ethyl)-1,3-dioxane (1o for Table 1)

^{1}H NMR (400 MHz, $CDCl_3$): d 1.33 (d, J = 13.4 Hz, 1H), 1.51-1.71 (m, 6H), 1.89-2.14 (m, 7H), 3.75 (dt, J_1 = 2.4 Hz, J_2 = 12.4 Hz, 2H), 4.10 (dd, J_1 = 5.0 Hz, J_2 = 10.7 Hz, 2H), 4.50 (t, J = 5.2 Hz, 1H), 5.42 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 22.52, 22.98, 25.22, 25.86, 28.34, 32.15, 33.36, 66.89, 102.20, 120.90, 136.98. HRMS (ESI^+) exact mass calcd for $C_{12}H_{21}O_2$ $[M+H]^+$ requires *m/z* 197.1542, found *m/z* 197.1544.

General procedure for chiral acetals synthesis

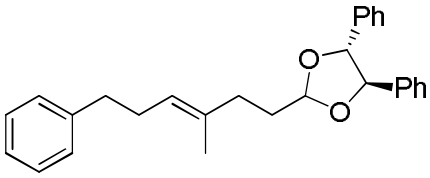


To a solution of corresponding achiral acetal (1 mmol) in DCM (5 mL) was added chiral diol (1.2 mmol, 1.2 equiv) and TsOH (0.1 mmol) at room temperature. The mixture was stirred at room temperature for 5 h, followed by quenching with triethyl amine (0.1 mL). The reaction mixture concentrated in *vacuo* and purified by chromatography with hexane/EA as the eluent provided the desired products as colorless oil in 85-94% yields.



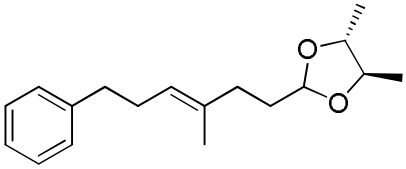
(4*R*,6*R*)-4,6-dimethyl-2-((*E*)-3-methyl-6-phenylhex-3-en-1-yl)-1,3-dioxane (1aa for Table 3)

Yield: 94%. $[a]_D^{20}$ = 16.1° (c = 4.60 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): d 1.20 (d, J = 6.1 Hz, 3H), 1.33 (d, J = 7.0 Hz, 4H), 1.55 (s, 3H), 1.60-1.68 (m, 2H), 1.79-1.87 (m, 1H), 2.05 (t, J = 7.9 Hz, 2H), 2.30 (q, J = 7.4 Hz, 2H), 2.62 (t, J = 7.8 Hz, 2H), 3.91 (dqd, J_1 = 2.4 Hz, J_2 = 6.2 Hz, J_3 = 12.4 Hz, 1H), 4.29 (p, J = 6.8 Hz, 1H), 4.79 (t, J = 5.2 Hz, 1H), 5.20 (t, J = 7.0 Hz, 1H), 7.15-7.19 (m, 3H), 7.25-7.28 (m 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 15.97, 17.24, 21.90, 29.88, 33.56, 34.03, 36.08, 36.86, 67.50, 67.95, 93.98, 123.75, 125.66, 128.22, 128.45, 135.16, 142.34. HRMS (ESI^+) exact mass calcd for $C_{19}H_{29}O_2$ $[M+H]^+$ requires *m/z* 289.2168, found *m/z* 289.2176.



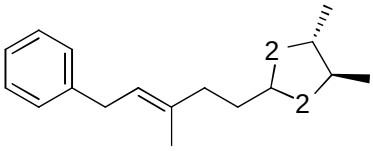
(4*R*,5*R*)-2-((*E*)-3-methyl-6-phenylhex-3-en-1-yl)-4,5-diphenyl-1,3-dioxolane (1ab for Table 3)

Yield: 85%. $[a]_D^{20}$ = 10.0° (*c* = 1.03 in $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): d 1.62 (s, 3H), 1.97-2.02 (m, 2H), 2.26-2.37 (m, 4H), 2.66 (t, *J* = 7.8 Hz, 2H), 4.73 (d, *J* = 7.8 Hz, 1H), 4.76 (d, *J* = 7.6 Hz, 1H), 5.30 (t, *J* = 7.0 Hz, 1H), 5.50 (t, *J* = 4.5 Hz, 1H), 7.18-7.35 (m, 15H); ^{13}C NMR (100 MHz, $CDCl_3$): d 16.07, 29.99, 33.14, 33.66, 36.08, 84.94, 86.81, 124.01, 125.71, 126.35, 126.81, 128.09, 128.25, 128.47, 128.50, 128.54, 128.58, 135.03, 136.95, 138.59, 142.32. HRMS (ESI^+) exact mass calcd for $C_{28}H_{30}O_2Na$ $[M+Na]^+$ requires *m/z* 421.2144, found *m/z* 421.2151.



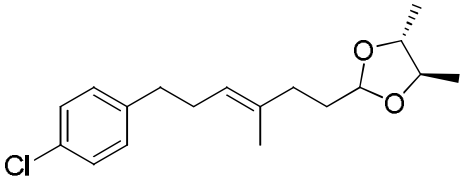
(4*R*,5*R*)-4,5-dimethyl-2-((*E*)-3-methyl-6-phenylhex-3-en-1-yl)-1,3-dioxolane (1a' for Table 3)

Yield: 90%. $[a]_D^{20}$ = 59.8° (*c* = 0.95 in $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): d 1.22 (d, *J* = 5.6 Hz, 3H), 1.29 (d, *J* = 5.6 Hz, 3H), 1.55 (s, 3H), 1.69-1.74 (m, 2H), 2.06-2.10 (m, 2H), 2.30 (q, *J* = 7.6 Hz, 2H), 2.61-2.65 (m, 2H), 3.58-3.64 (m, 2H), 5.02 (t, *J* = 4.7 Hz, 1H), 5.22 (t, *J* = 7.0 Hz, 1H), 7.15-7.19 (m, 3H), 7.25-7.28 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 16.03, 16.98, 17.31, 29.94, 33.16, 33.69, 36.06, 78.10, 79.75, 103.03, 123.66, 125.67, 128.21, 128.48, 135.11, 142.33. HRMS (ESI^+) exact mass calcd for $C_{18}H_{27}O_2$ $[M+H]^+$ requires *m/z* 275.2011, found *m/z* 275.2013.



(4*R*,5*R*)-4,5-dimethyl-2-((*E*)-3-methyl-5-phenylpent-3-en-1-yl)-1,3-dioxolane (1d' for Table 3)

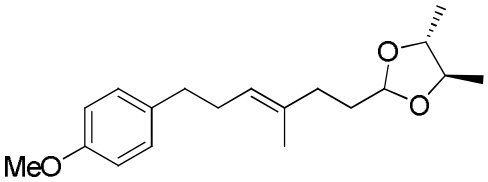
Yield: 87%. $[a]_D^{20}$ = -11.9° (*c* = 1.54 in $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): d 1.22 (d, *J* = 5.6 Hz, 3H), 1.28 (d, *J* = 5.6 Hz, 3H), 1.72 (s, 3H), 1.75-1.80 (m, 2H), 2.14-2.18 (m, 2H), 3.35 (d, *J* = 7.3 Hz, 2H), 3.57-3.61 (m, 2H), 5.04 (t, *J* = 4.7 Hz, 1H), 5.39 (t, *J* = 7.3 Hz, 1H), 7.16-7.18 (m, 3H), 7.25-7.28 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 16.26, 16.98, 17.30, 33.19, 33.71, 34.18, 78.12, 79.76, 103.00, 123.07, 125.70, 128.31, 128.34, 135.59, 141.67. HRMS (ESI^+) exact mass calcd for $C_{17}H_{25}O_2$ $[M+H]^+$ requires *m/z* 261.1855, found *m/z* 261.1866.



(4*R*,5*R*)-2-((*E*)-6-(4-chlorophenyl)-3-methylhex-3-en-1-yl)-4,5-dimethyl-1,3-dioxolane (1h' for Table 3)

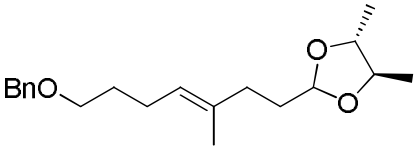
Yield: 88%. $[a]_D^{20}$ = 29.0° (*c* = 0.99 in $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): d 1.22 (d, *J* = 5.8 Hz, 3H), 1.28 (d, *J* = 5.8 Hz, 3H), 1.53 (s, 3H), 1.68-1.73 (m, 2H), 2.06-2.10 (m, 2H), 2.27 (q, *J* = 7.4 Hz, 2H), 2.59 (t, *J* = 7.6 Hz,

2H), 3.57-3.63 (m, 2H), 5.00 (t, *J* = 4.7 Hz, 1H), 5.17 (t, *J* = 7.1 Hz, 1H), 7.09 (d, *J* = 8.4 Hz, 2H), 7.22 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 16.06, 16.97, 17.30, 29.71, 33.13, 33.65, 35.34, 78.11, 79.75, 102.97, 123.20, 128.26, 129.85, 131.36, 135.47, 140.69. HRMS (ESI$^+$) exact mass calcd for $C_{18}H_{26}ClO_2$ $[M+H]^+$ requires *m/z* 309.1621, found *m/z* 309.1630.



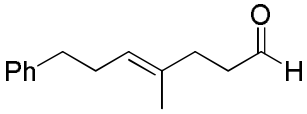
(4*R*,5*R*)-2-((*E*)-6-(4-methoxyphenyl)-3-methylhex-3-en-1-yl)-4,5-dimethyl-1,3-dioxolane (1g' for Table 3)

Yield: 92%. $[a]_D^{20}$ = 8.3° (*c* = 1.81 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): d 1.21 (d, *J* = 5.5 Hz, 3H), 1.28 (d, *J* = 5.5 Hz, 3H), 1.54 (s, 3H), 1.69-1.74 (m, 2H), 2.06-2.10 (m, 2H), 2.26 (q, *J* = 7.4 Hz, 2H), 2.56 (t, *J* = 7.7 Hz, 2H), 3.57-3.61 (m, 2H), 3.76 (s, 3H), 5.01 (t, *J* = 4.7 Hz, 1H), 5.20 (t, *J* = 6.9 Hz, 1H), 6.80 (d, *J* = 8.5 Hz, 2H), 7.08 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 16.06, 16.99, 17.32, 30.17, 33.18, 33.71, 35.13, 55.19, 78.10, 79.74, 103.03, 113.63, 123.75, 129.34, 134.40, 134.99, 157.69. HRMS (ESI$^+$) exact mass calcd for $C_{19}H_{29}O_3$ $[M+H]^+$ requires *m/z* 305.2117, found *m/z* 305.2122.



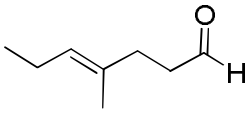
(4*R*,5*R*)-2-((*E*)-7-(benzyloxy)-3-methylhept-3-en-1-yl)-4,5-dimethyl-1,3-dioxolane (1e' for Table 3)

Yield: 89%. $[a]_D^{20}$ = 8.4° (*c* = 1.14 in $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): d 1.22 (d, *J* = 5.5 Hz, 3H), 1.28 (d, *J* = 5.5 Hz, 3H), 1.60 (s, 3H), 1.60-1.75 (m, 4H), 2.05-2.11 (m, 4H), 3.45 (t, *J* = 6.5 Hz, 2H), 3.57-3.60 (m, 2H), 4.48 (s, 2H), 5.03 (t, *J* = 4.7 Hz, 1H), 5.16 (t, *J* = 6.9 Hz, 1H),7.25-7.34 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): d 16.07, 16.99, 17.31, 24.45, 29.80, 33.24, 33.70, 69.86, 72.90, 78.10, 79.74, 103.04, 123.91, 127.47, 127.63, 128.35, 134.90, 138.68. HRMS (ESI$^+$) exact mass calcd for $C_{20}H_{31}O_3$ $[M+H]^+$ requires *m/z* 319.2273, found *m/z* 319.2269.



(*E*)-4-methyl-7-phenylhept-4-enal (4a for Table 4)

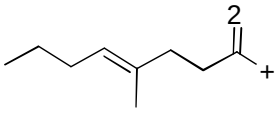
^{1}H NMR (400 MHz, $CDCl_3$): d 1.55 (s, 3H), 2.27-2.32 (m, 4H), 2.49 (t, *J* = 7.4 Hz, 2H), 2.63 (t, *J* = 7.7 Hz, 2H), 5.21 (t, *J* = 7.0 Hz, 1H), 7.16-7.19 (m, 3H), 7.25-7.29 (m, 2H), 9.73 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 16.05, 29.88, 31.83, 35.91, 42.12, 124.69, 125.77, 128.26, 128.48, 133.79, 142.08, 202.65. HRMS (ESI$^+$) exact mass calcd for $C_{14}H_{19}O$ $[M+H]^+$ requires *m/z* 203.1436, found *m/z* 203.1434.



(*E*)-4-methylhept-4-enal (4b for Table 4)

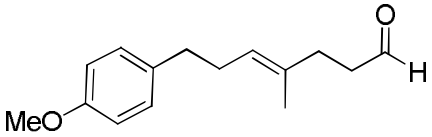
^{1}H NMR (400 MHz, $CDCl_3$): d 0.93 (t, *J* = 7.5 Hz, 3H), 1.61 (s, 3H), 1.99 (p, *J* = 7.2 Hz, 2H), 2.32 (t *J* = 7.5 Hz, 2H), 2.52 (t, *J* = 7.5 Hz, 2H), 5.16 (t, *J* = 6.8 Hz, 1H), 9.76 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 14.21, 15.91,

21.15, 31.78, 42.15, 127.51, 132.25, 202.77. HRMS (ESI^+) exact mass calcd for $C_8H_{15}O$ $[M+H]^+$ requires *m/z* 127.1123, found *m/z* 127.1126.



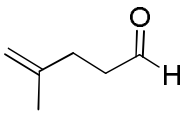
(*E*)-4-methyloct-4-enal (4c for Table 4)

^1H NMR (400 MHz, $CDCl_3$): d 0.88 (t, *J* = 7.4 Hz, 3H), 1.32-1.37 (m, 2H), 1.62 (s, 3H), 1.96 (q, *J* = 7.2 Hz, 2H), 2.33 (t, *J* = 7.5 Hz, 2H), 2.52 (t, *J* = 7.4 Hz, 2H), 5.17 (t, *J* = 7.0 Hz, 1H), 9.76 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 13.76, 16.07, 22.82, 29.96, 31.90, 42.19, 125.73, 132.93, 202.78. HRMS (ESI^+) exact mass calcd for $C_9H_{17}O$ $[M+H]^+$ requires *m/z* 141.1279, found *m/z* 141.1278.



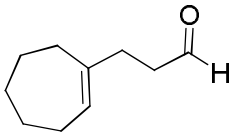
(*E*)-7-(4-methoxyphenyl)-4-methylhept-4-enal (4d for Table 4)

^1H NMR (400 MHz, $CDCl_3$): d 1.55 (s, 3H), 2.26-2.32 (m, 4H), 2.49 (t, *J* = 7.1 Hz, 2H), 2.57 (t, *J* = 7.7 Hz, 2H), 3.78 (s, 3H), 5.19 (t, *J* = 7.0 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 2H), 7.08 (d, *J* = 8.4 Hz, 2H), 9.73 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 16.06, 30.11, 31.83, 34.98, 42.12, 55.26, 113.66, 124.77, 129.34, 133.68, 134.19, 157.75, 202.69. HRMS (ESI^+) exact mass calcd for $C_{15}H_{20}O_2Na$ $[M+Na]^+$ requires *m/z* 255.1361, found *m/z* 255.1357.



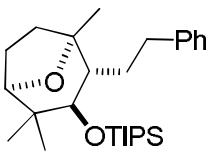
4-Methylpent-4-enal (4e for Table 4)

^1H NMR (400 MHz, $CDCl_3$): d 1.75 (s, 3H), 2.35 (t, *J* = 7.4 Hz, 2H), 2.58 (dt, J_1 = 1.3 Hz, J_2 = 7.3 Hz, 2H), 4.69 (s, 1H), 4.77 (s, 1H), 9.78 (t, *J* = 1.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 22.61, 29.82, 41.76, 110.68, 143.75, 202.23. HRMS (ESI^+) exact mass calcd for $C_6H_{11}O$ $[M+H]^+$ requires *m/z* 99.0810, found *m/z* 99.0811.

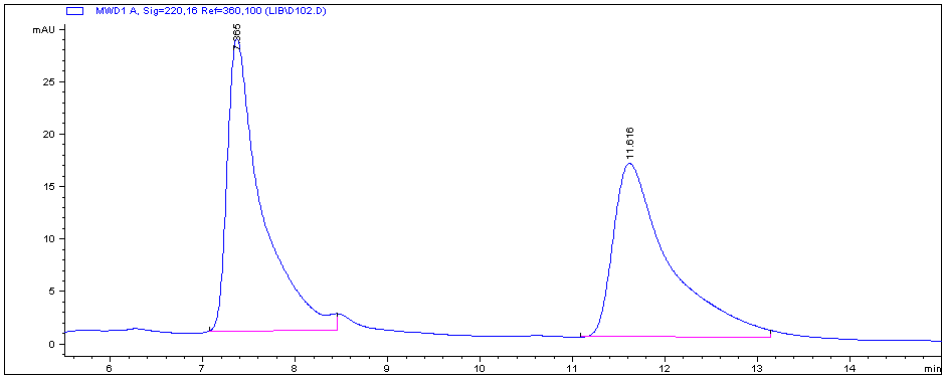


3-(Cyclohept-1-en-1-yl)propanal (4f for Table 4)

^1H NMR (400 MHz, $CDCl_3$): d 1.41-1.50 (m, 4H), 1.70-1.76 (m, 2H), 2.04-2.11 (m, 4H), 2.32 (t, *J* = 7.4 Hz 2H), 2.49 (dt, J_1 = 1.9 Hz, J_2 = 7.4 Hz, 2H), 5.57 (t, *J* = 6.4 Hz, 1H), 9.75 (t, *J* = 1.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): d 26.66, 27.14, 28.20, 32.43, 32.53, 32.83, 42.17, 127.01, 142.49, 202.90. HRMS (ESI^+) exact mass calcd for $C_{10}H_{17}O$ $[M+H]^+$ requires *m/z* 153.1279, found *m/z* 153.1277.

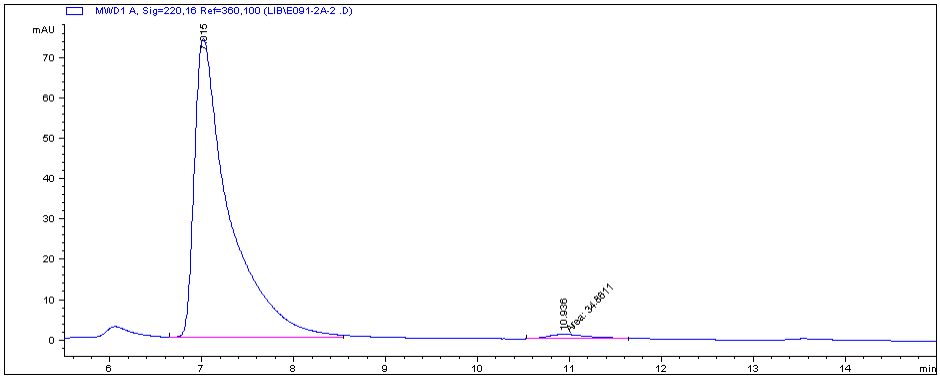


The enantiomeric excess was determined by chiral HPLC (Chiralpak OD-H, 0.1% *i*-PrOH/hexanes, flow rate 1.0 mL/min, ? = 220 nm); t_r = 7.37 min (major) and 11.62 min (minor) min. 97% ee. Yield: 88%. $[a]_D^{20}$ = 27.9° (*c* = 1.52 in $CHCl_3$).



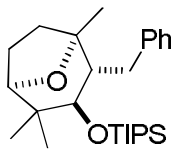
Signal 1: MWD1 A, Sig=220,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.365	BB	0.3637	726.02765	27.89085	50.9160
2	11.616	BB	0.5976	699.90558	16.54170	49.0840

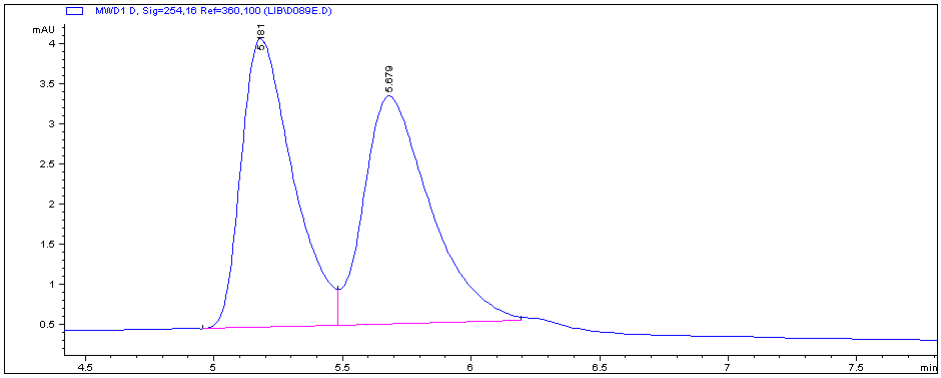


Signal 1: MWD1 A, Sig=220,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.015	VB	0.3672	1951.85181	74.12254	98.2453
2	10.936	MM	0.5328	34.86113	1.09049	1.7547

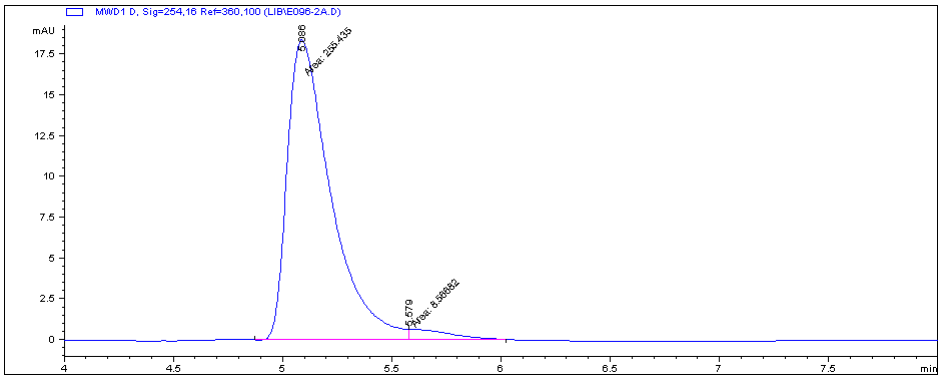


The enantiomeric excess was determined by chiral HPLC (Chiralpak AD-H, 0.1% *i*-PrOH/hexanes, flow rate 1.0 mL/min, ? = 254 nm); t_r = 5.18 min (major) and 5.68 min (minor) min. 94% ee. Yield: 91%. $[a]_D^{20}$ = -16.2° (*c* = 2.60 in $CHCl_3$).

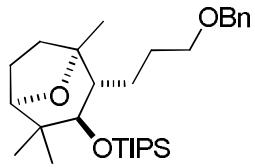


Signal 4: MWD1 D, Sig=254,16 Ref=360,100

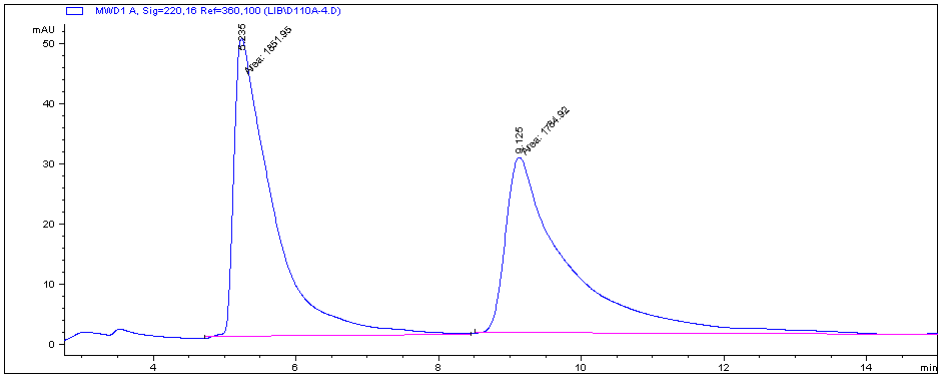
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.181	BV	0.2113	50.25304	3.59111	49.6802
2	5.679	VB	0.2656	50.89997	2.83311	50.3198



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.086	MF	0.2312	255.43469	18.41743	96.7550
2	5.579	FM	0.2199	8.56682	6.49374e-1	3.2450

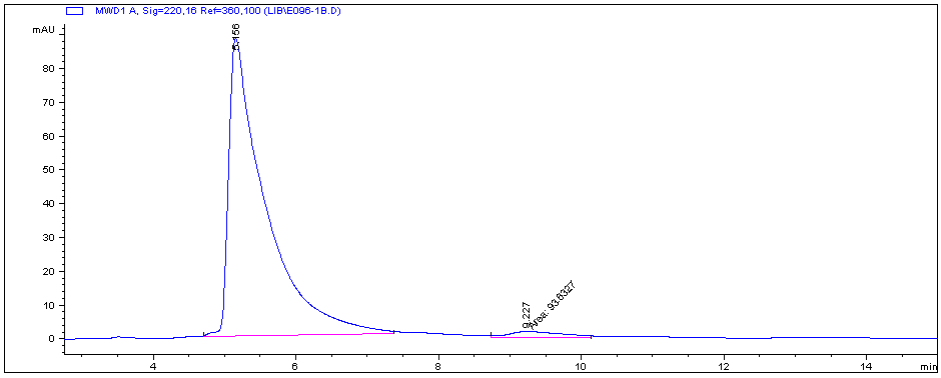


The enantiomeric excess was determined by chiral HPLC (Chiralpak OD-H, 0.5% *i*-PrOH/hexanes, flow rate 1.0 mL/min, ? = 220 nm); t_R = 5.24 min (major) and 9.13 min (minor) min. 94% ee. Yield: 79%. $[a]_D^{20}$ = 4.6° (*c* = 1.68 in $CHCl_3$).



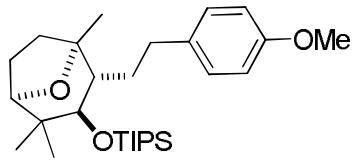
Signal 1: MWD1 A, Sig=220,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.235	MM	0.6240	1851.94946	49.46545	50.9215
2	9.125	MM	1.0193	1784.92236	29.18522	49.0785

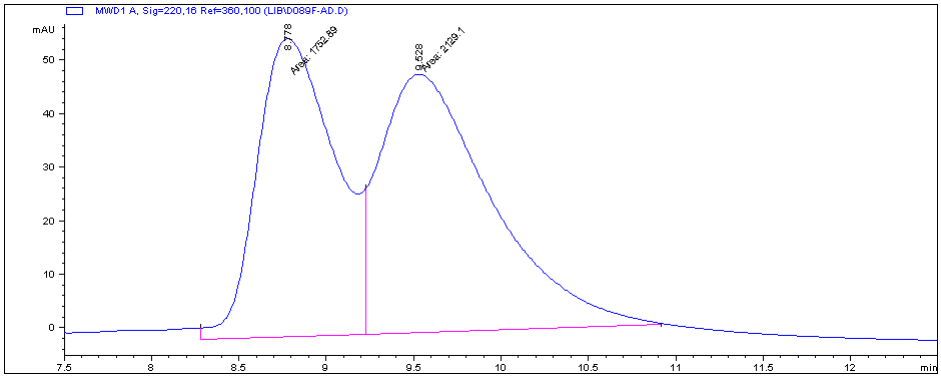


Signal 1: MWD1 A, Sig=220,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.156	BB	0.4800	3172.93408	88.12731	97.1336
2	9.227	MM	0.8742	93.63274	1.78508	2.8664

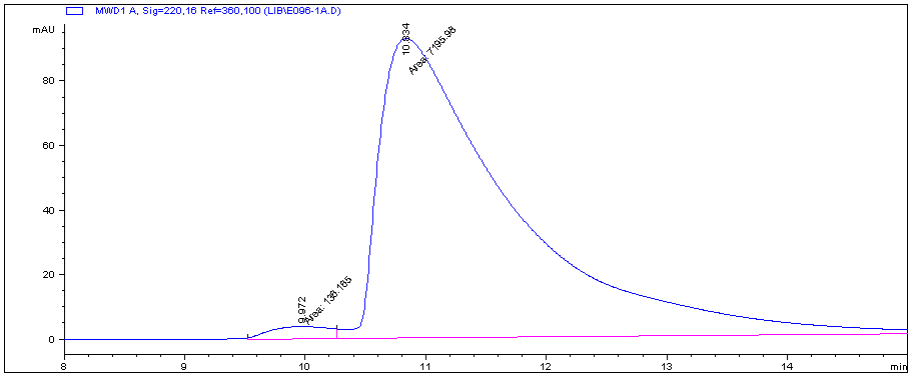


The enantiomeric excess was determined by chiral HPLC (Chiralpak AD-H, 0.5% *i*-PrOH/hexanes, flow rate 1.0 mL/min, ? = 220 nm); t_r = 8.78 min (minor) and 9.53 min (major) min. 95% ee. Yield: 74%. $[a]_D^{20}$ = 29.6° (*c* = 1.98 in $CHCl_3$).



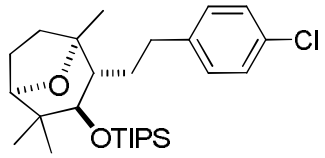
Signal 1: MWD1 A, Sig=220,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.778	MF	0.5228	1752.88940	55.88053	45.1544
2	9.528	FM	0.7330	2129.10498	48.41377	54.8456

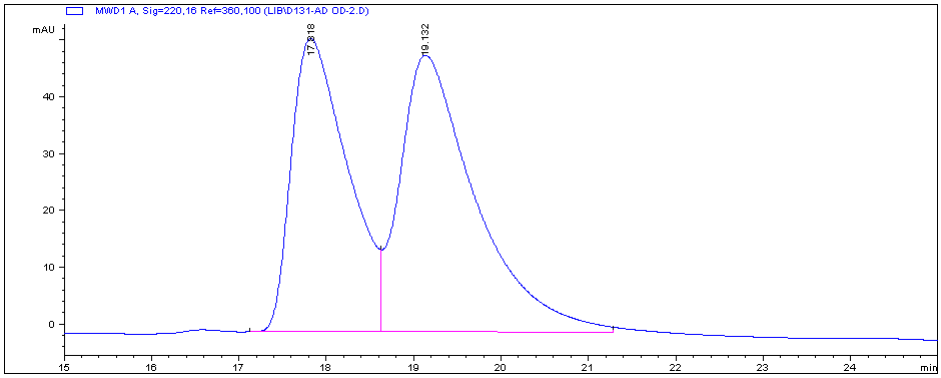


Signal 1: MWD1 A, Sig=220,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.016	BB	0.5228	166.27803	4.72258	2.6696
2	8.507	BB	0.7471	6062.32422	118.84359	97.3304

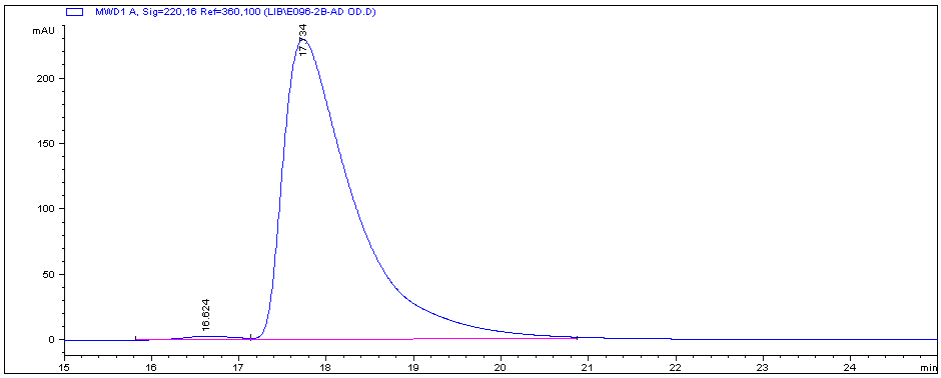


The enantiomeric excess was determined by chiral HPLC (Chiralpak AD-H + OD-H, 0.1% *i*-PrOH/hexanes, flow rate 0.7 mL/min, ? = 220 nm); t_r = 17.82 min (minor) and 19.13 min (major) min. 98% ee. Yield: 80%. $[a]_D^{20}$ = 32.9° (*c* = 2.93 in $CHCl_3$).



Signal 1: MWD1 A, Sig=220,16 Ref=360,100

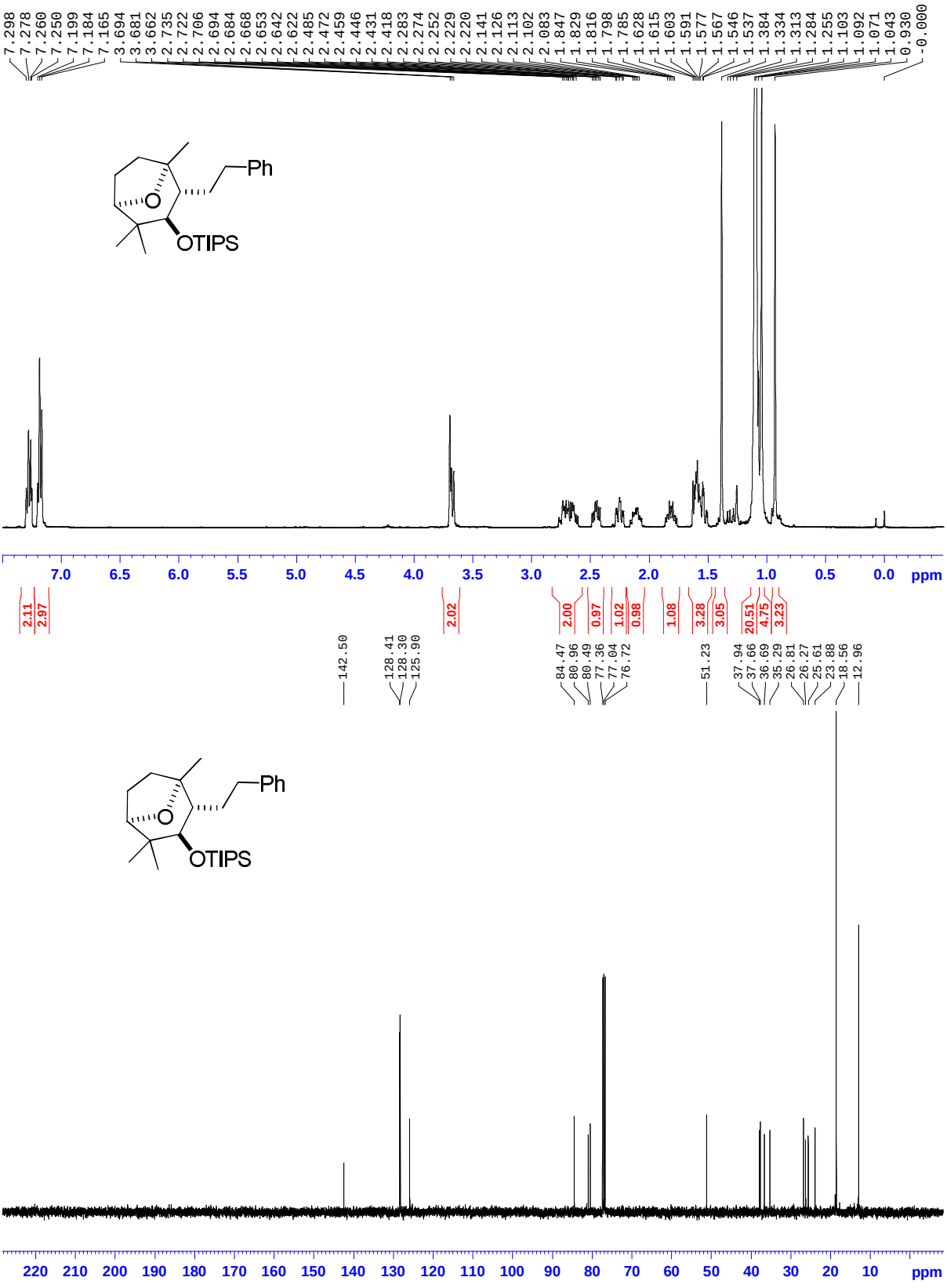
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.818	BV	0.6712	2316.30078	51.64791	44.1935
2	19.132	VB	0.8677	2924.97217	48.76922	55.8065



Signal 1: MWD1 A, Sig=220,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.624	BV	0.6624	130.37111	3.07639	0.9985
2	17.734	VB	0.8217	1.29265e4	229.30748	99.0015

Spectrum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2.722
2.706
2.694
2.684
2.668
2.653
2.642
2.622
2.485
2.472
2.459
2.446
2.431
2.418
2.283
2.274
2.252
2.229
2.220
2.141
2.126
2.113
2.102
2.083
1.847
1.829
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1.798
1.785
1.628
1.615
1.603
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1.567
1.546
1.537
1.384
1.334
1.313
1.284
1.255
1.103
1.092
1.071
1.043
0.930
-0.000
Ph
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OTIPS
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6.0
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4.0
3.5
3.0
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2.0
1.5
1.0
0.5
0.0
ppm
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2.02
2.00
0.97
1.02
0.98
1.08
3.28
3.05
20.51
4.75
3.23
142.50
128.41
128.30
125.90
84.47
80.96
80.49
77.36
77.04
76.72
51.23
37.94
37.66
36.69
35.29
26.81
26.27
25.61
23.88
18.56
12.96
Ph
O
OTIPS
220
210
200
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180
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150
140
130
120
110
100
90
80
70
60
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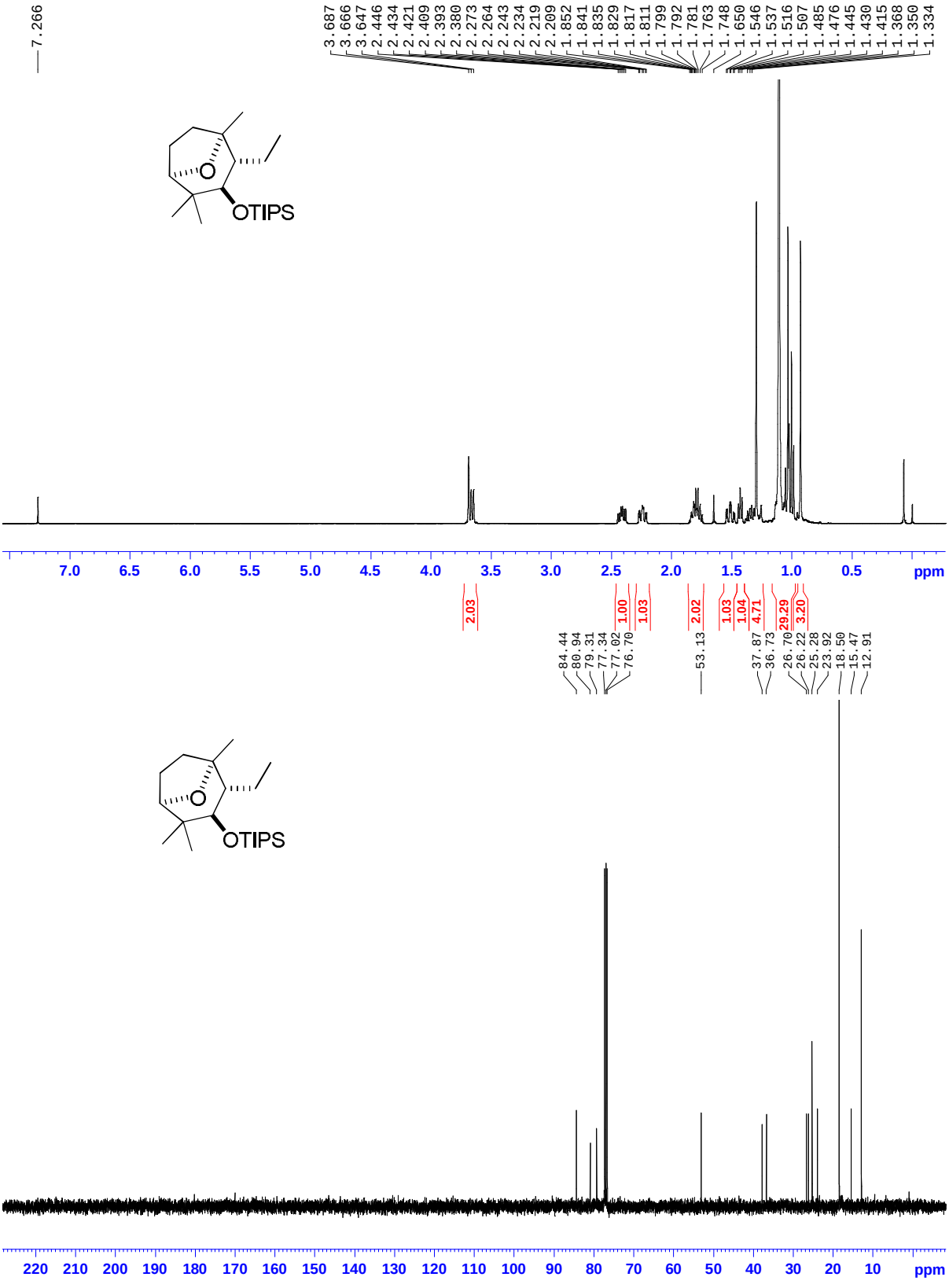
30
20
10
ppm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1.829
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1.811
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1.748
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1.445
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1.368
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1.334
OTIPS
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2.03
1.00
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2.02
1.03
1.04
4.71
29.29
3.20
84.44
80.94
79.31
77.34
77.02
76.70
53.13
37.87
36.73
26.70
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18.50
15.47
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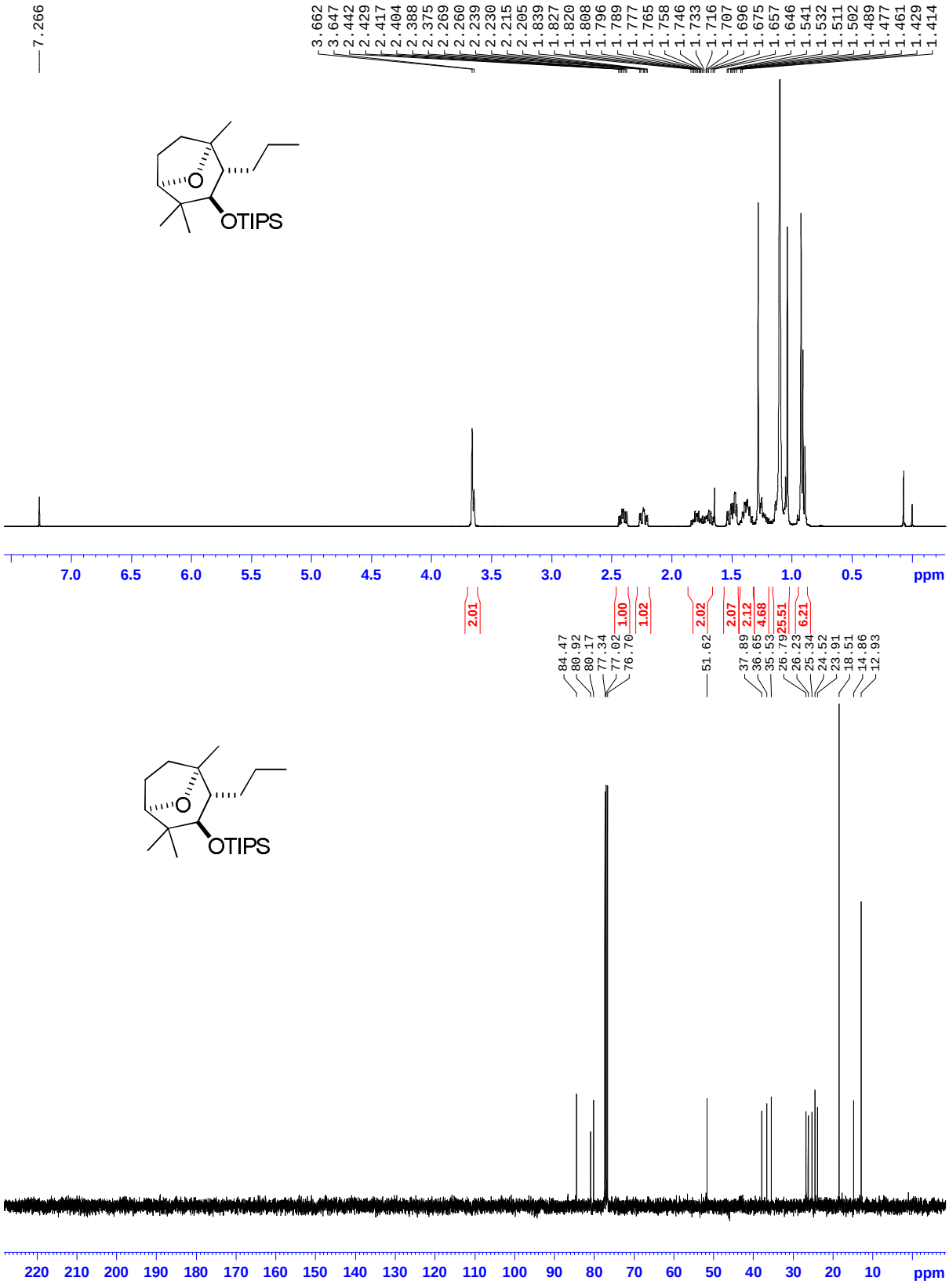
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1.827
1.820
1.808
1.796
1.789
1.777
1.765
1.758
1.746
1.733
1.716
1.707
1.696
1.675
1.657
1.646
1.541
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1.511
1.502
1.489
1.477
1.461
1.429
1.414
OTIPS
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1.02
2.02
2.07
2.12
4.68
25.51
6.21
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80.17
77.34
77.02
76.70
51.62
37.89
36.65
35.53
26.79
26.23
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23.91
18.51
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12.93
OTIPS
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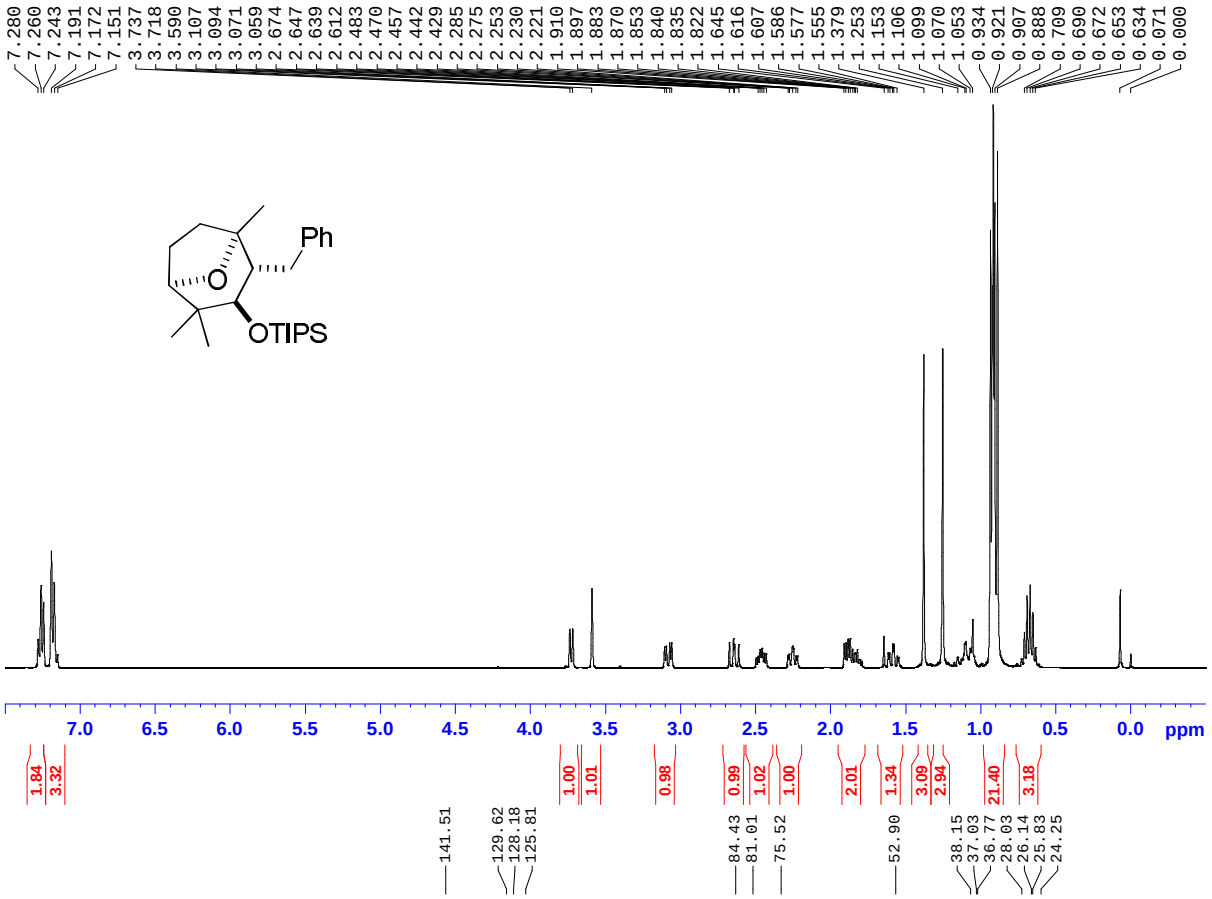
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2.647
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2.612
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2.230
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1.840
1.835
1.822
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1.607
1.586
1.577
1.555
1.379
1.253
1.153
1.106
1.099
1.070
1.053
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0.907
0.888
0.709
0.690
0.672
0.653
0.634
0.071
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Ph
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OTIPS
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6.0
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5.0
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2.5
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0.5
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ppm
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1.00
1.01
0.98
0.99
1.02
1.00
2.01
1.34
3.09
2.94
21.40
3.18
141.51
129.62
128.18
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36.77
28.03
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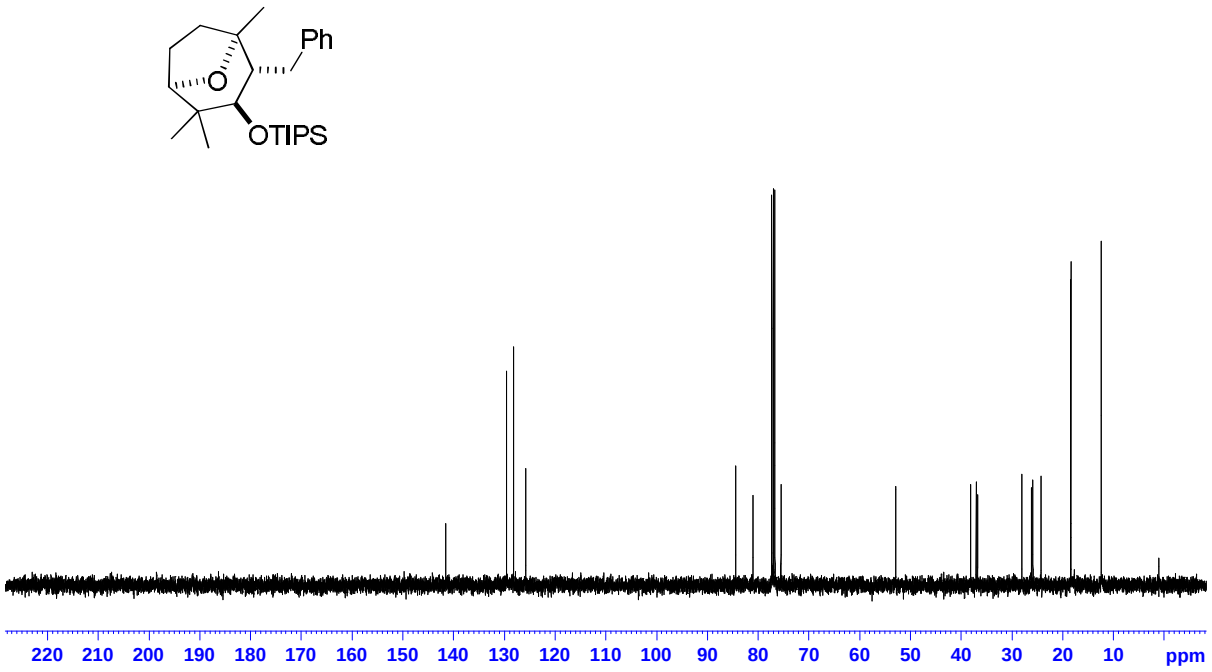
25.83
24.25


Ph
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OTIPS
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110
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50
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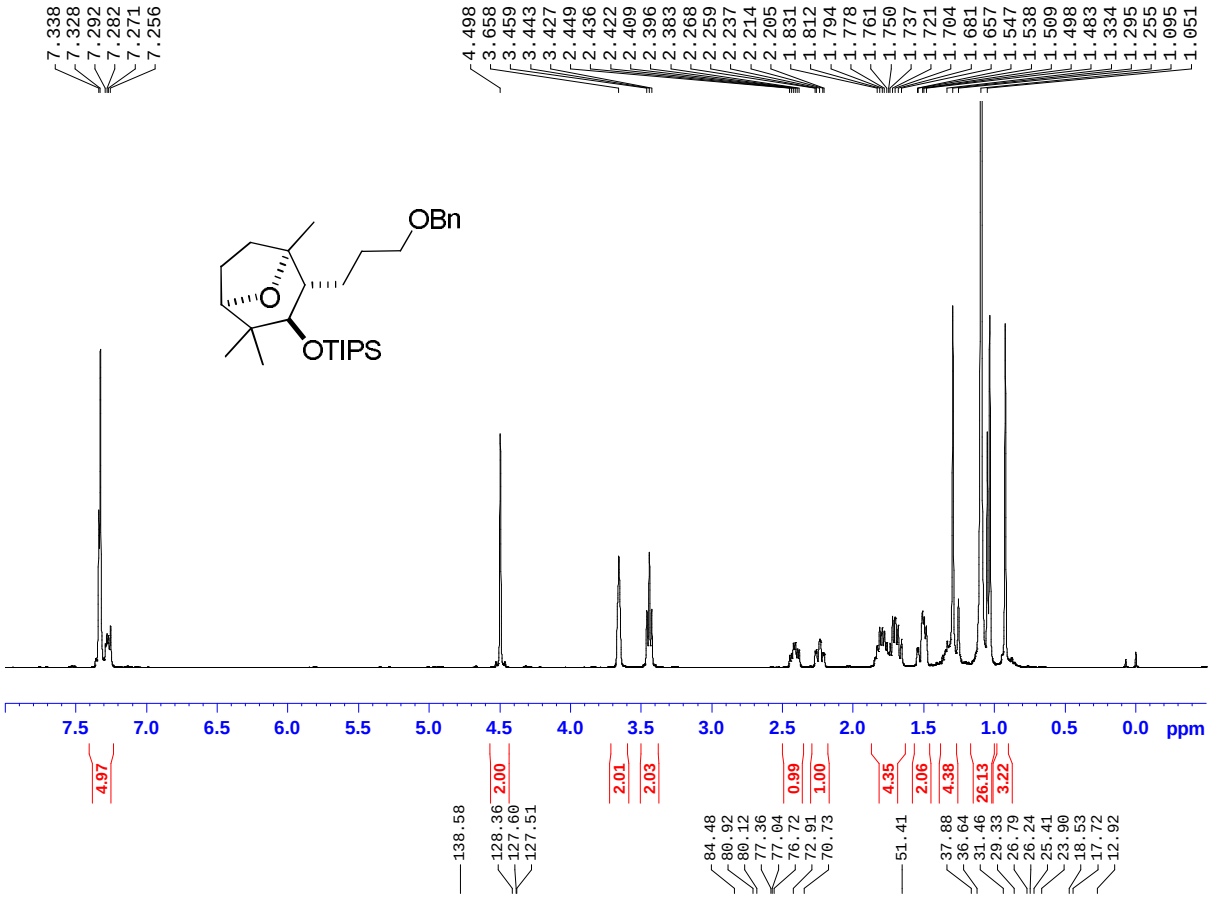
30
20
10
ppm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2.409
2.396
2.383
2.268
2.259
2.237
2.214
2.205
1.831
1.812
1.794
1.778
1.761
1.750
1.737
1.721
1.704
1.681
1.657
1.547
1.538
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1.498
1.483
1.334
1.295
1.255
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OBn
OTIPS
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5.0
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4.0
3.5
3.0
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0.5
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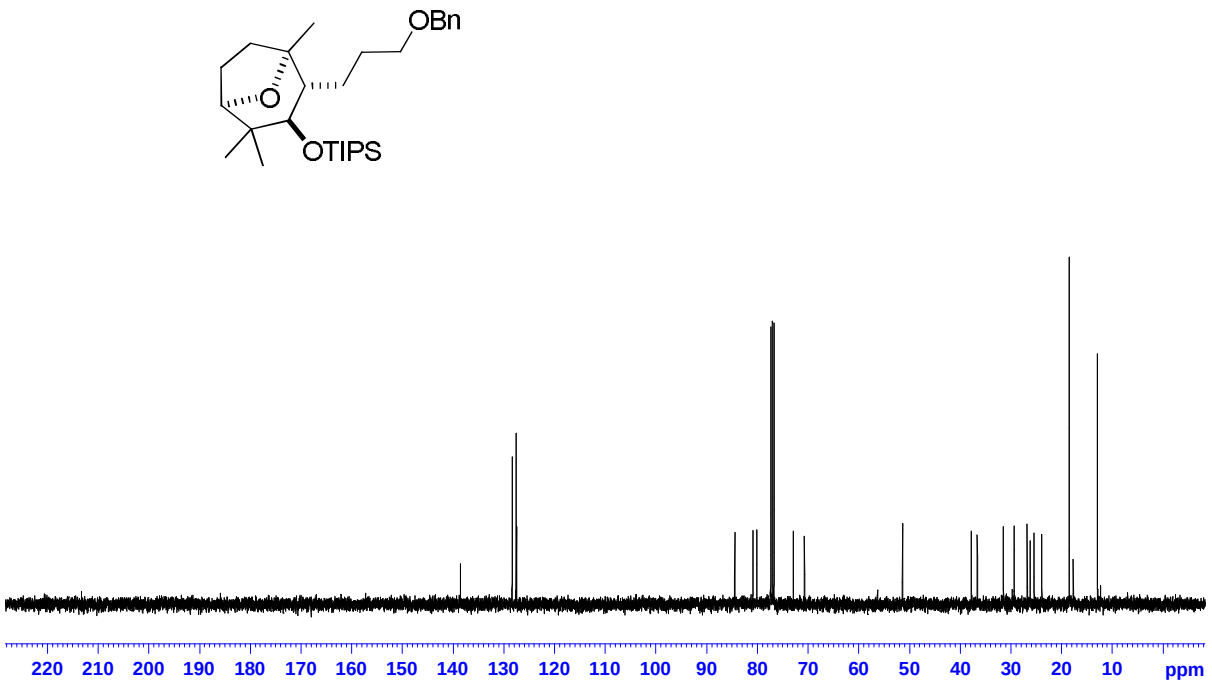
3.22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37.88
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31.46
29.33
26.79
26.24
25.41
23.90
18.53
17.72
12.92
OBn
OTIPS
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210
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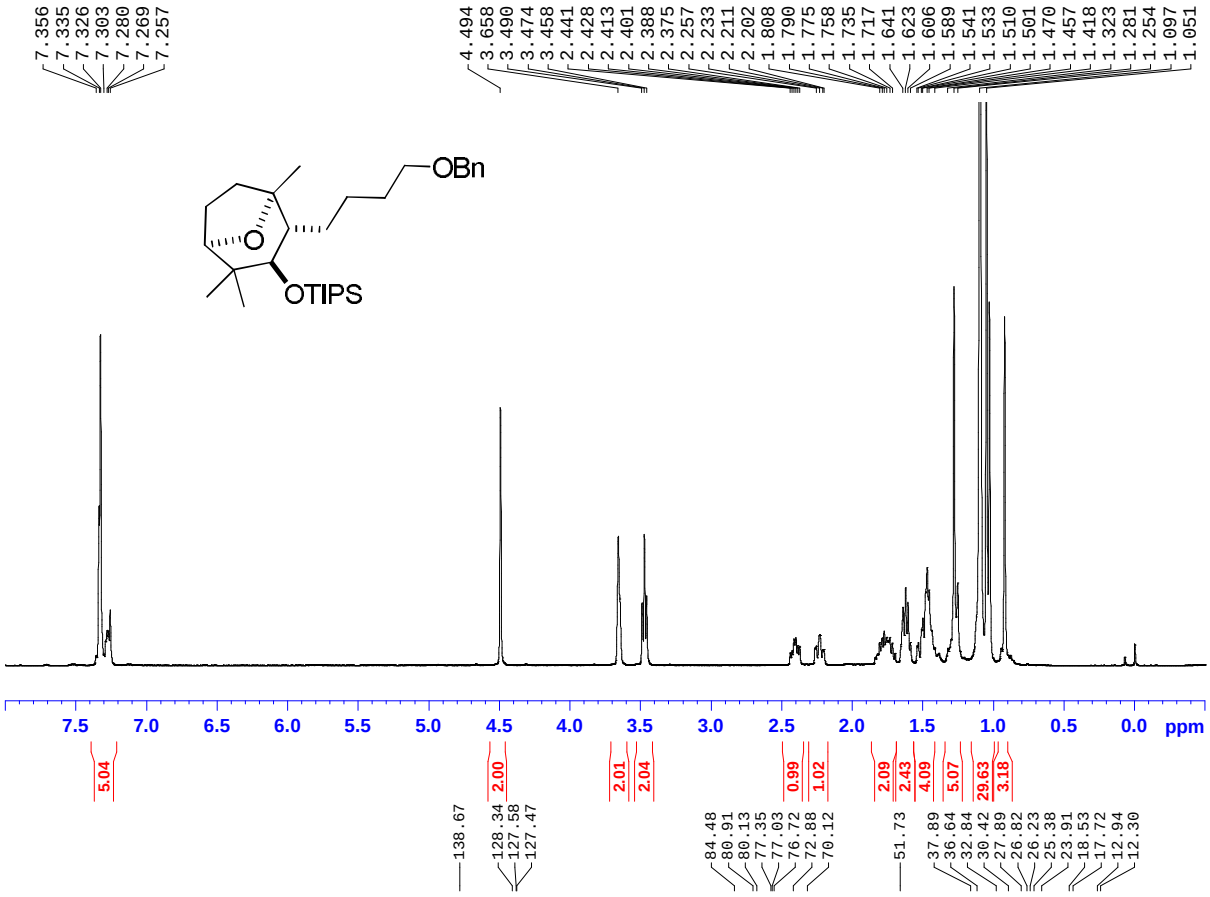
10
ppm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2.428
2.413
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2.257
2.233
2.211
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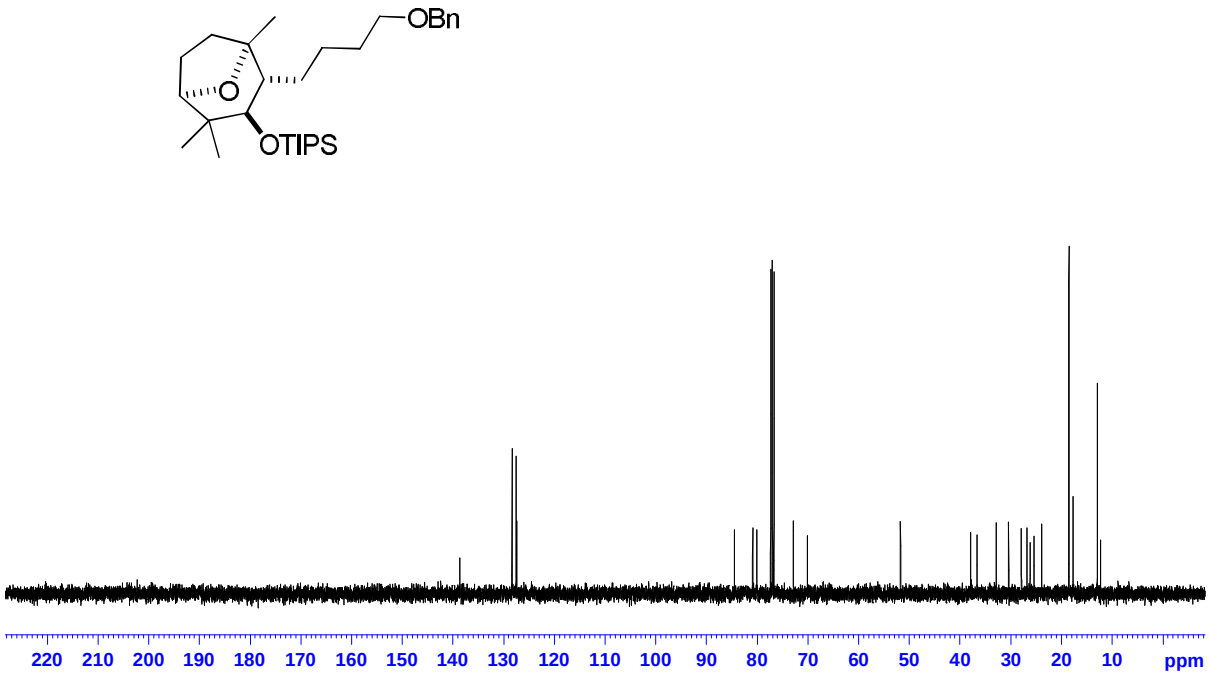
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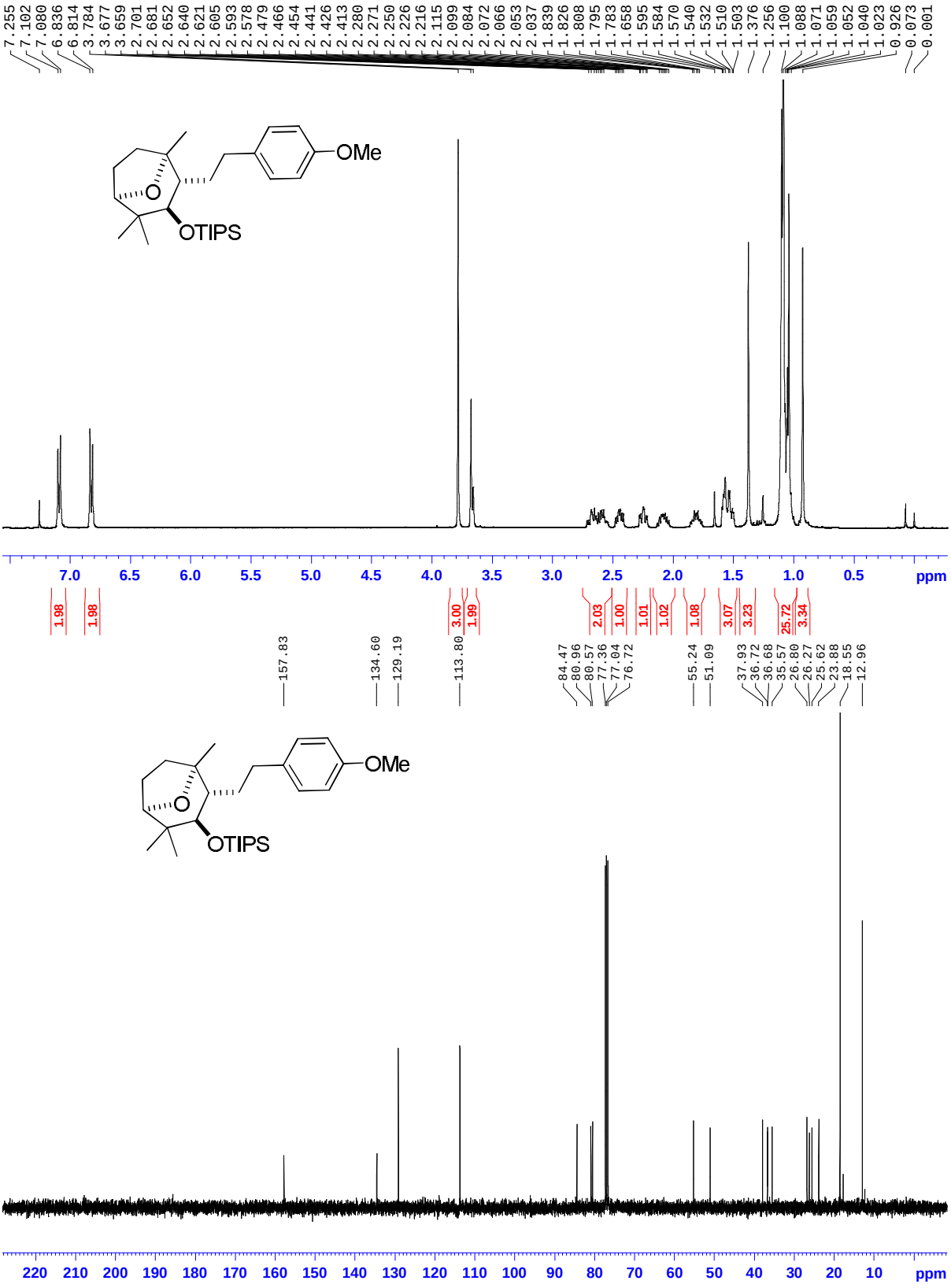
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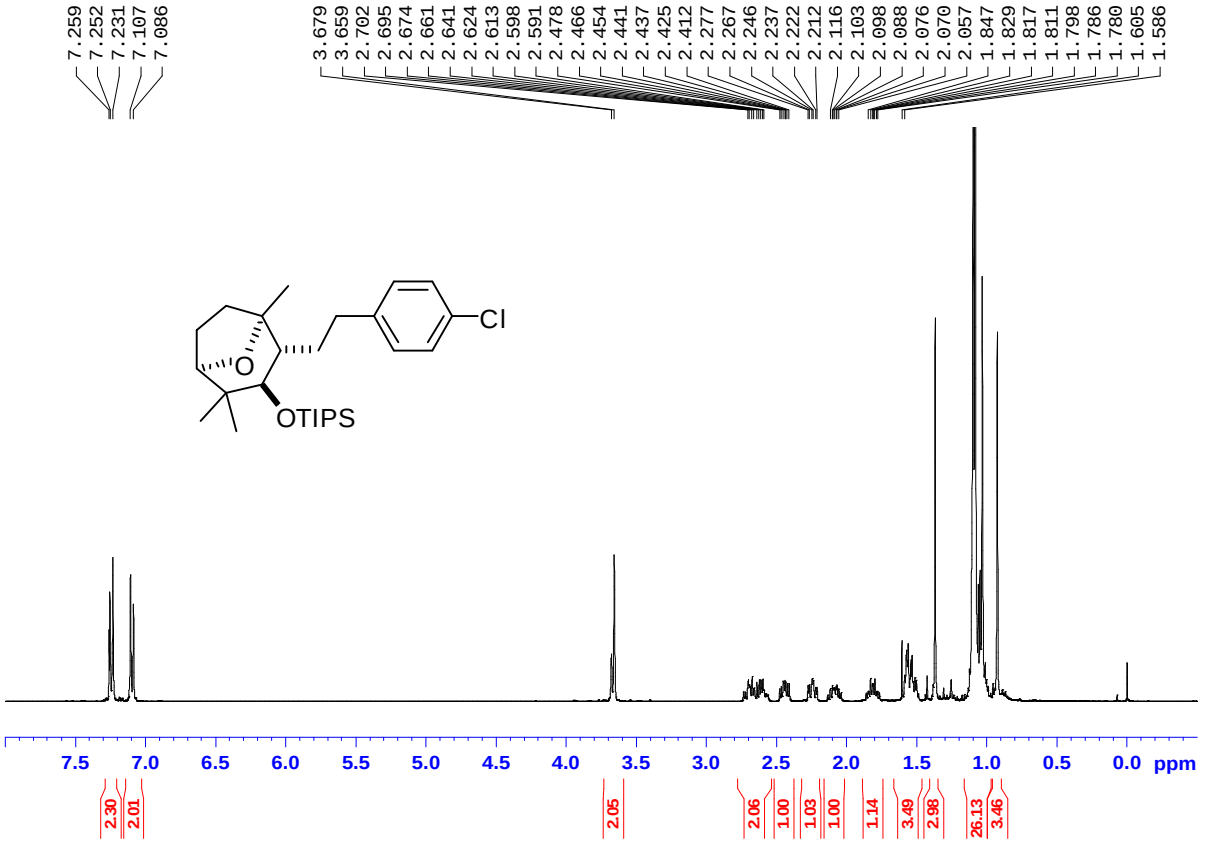
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Cl
O
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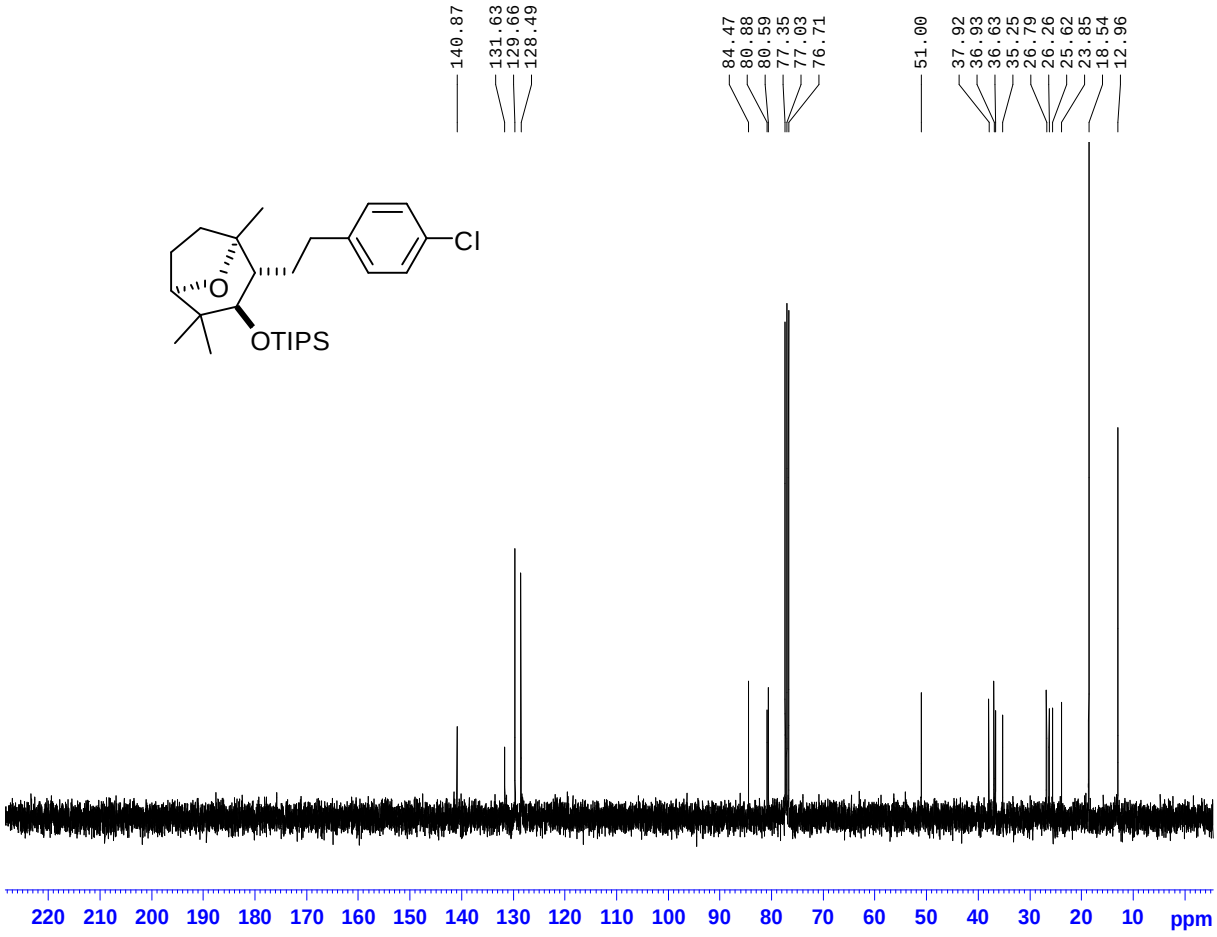
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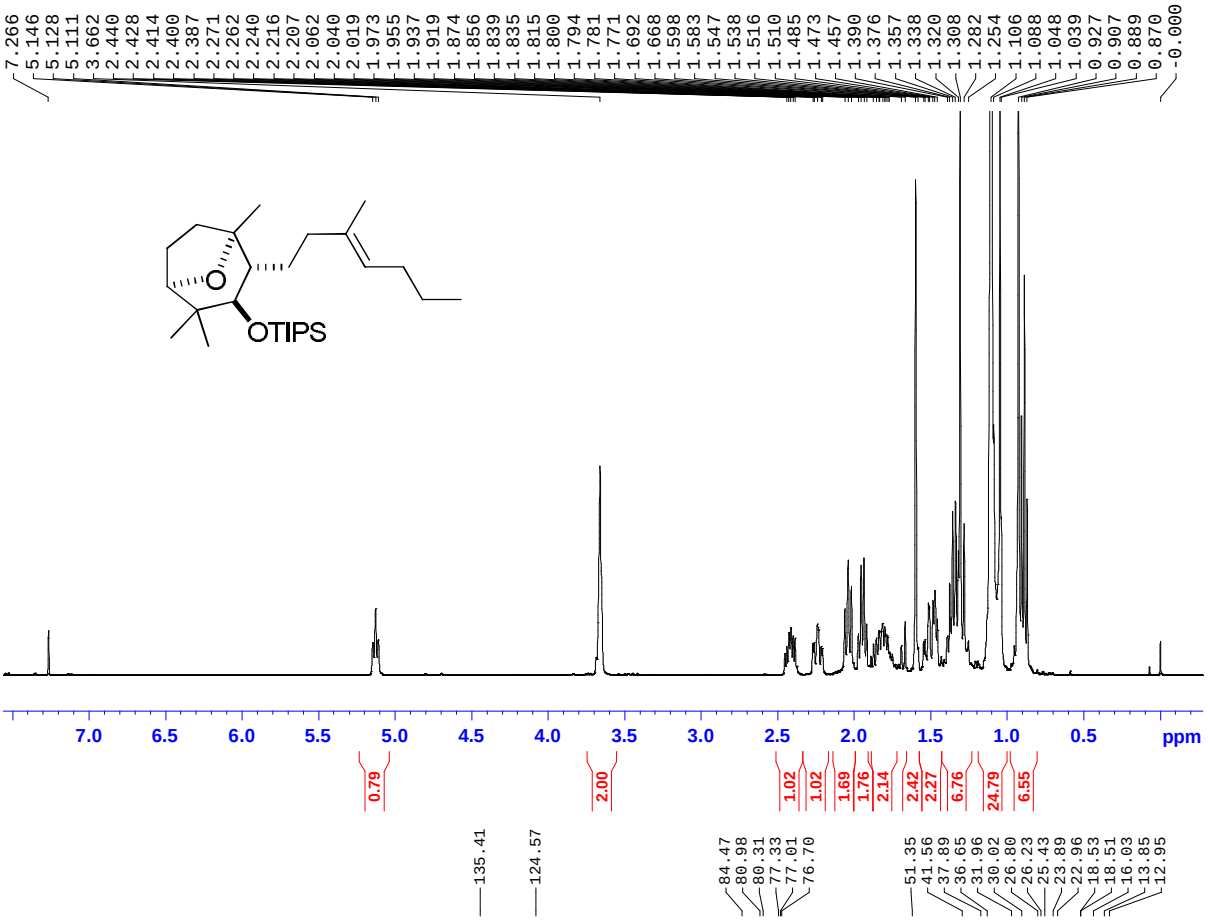
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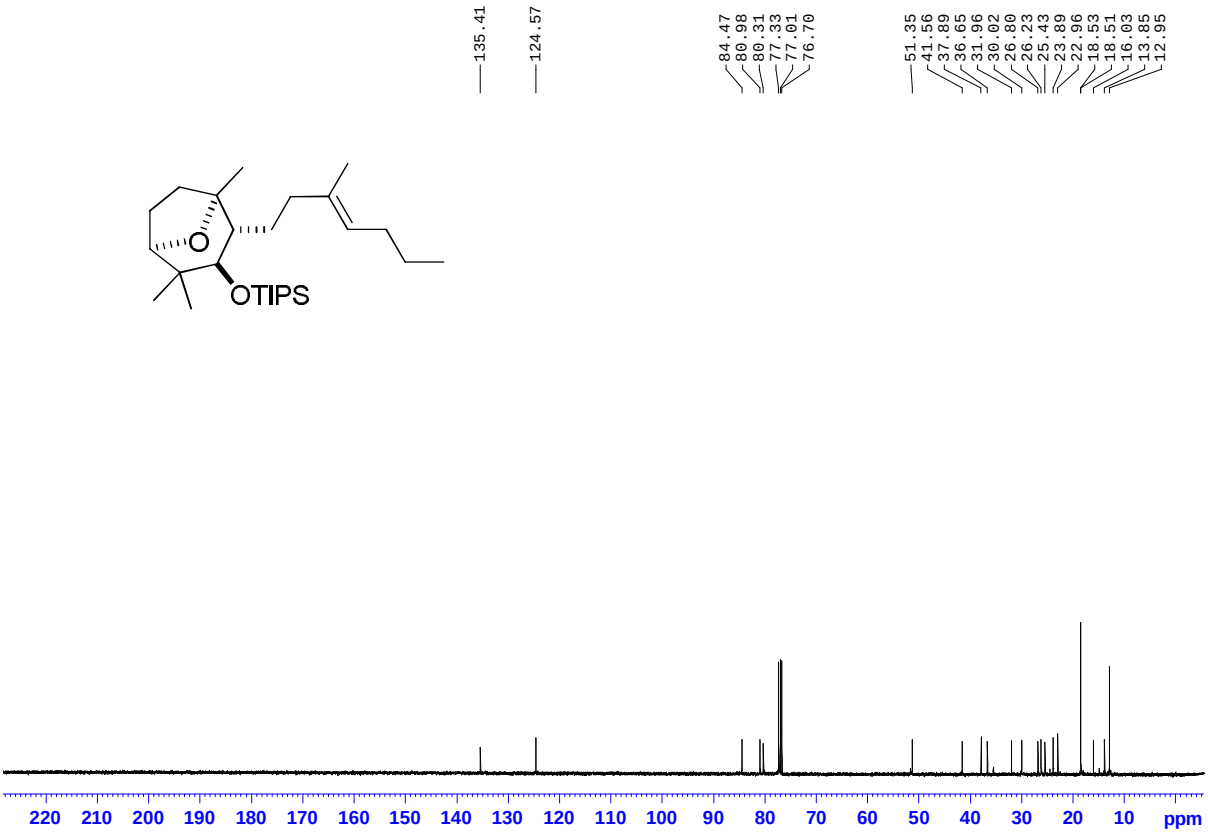
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18.51
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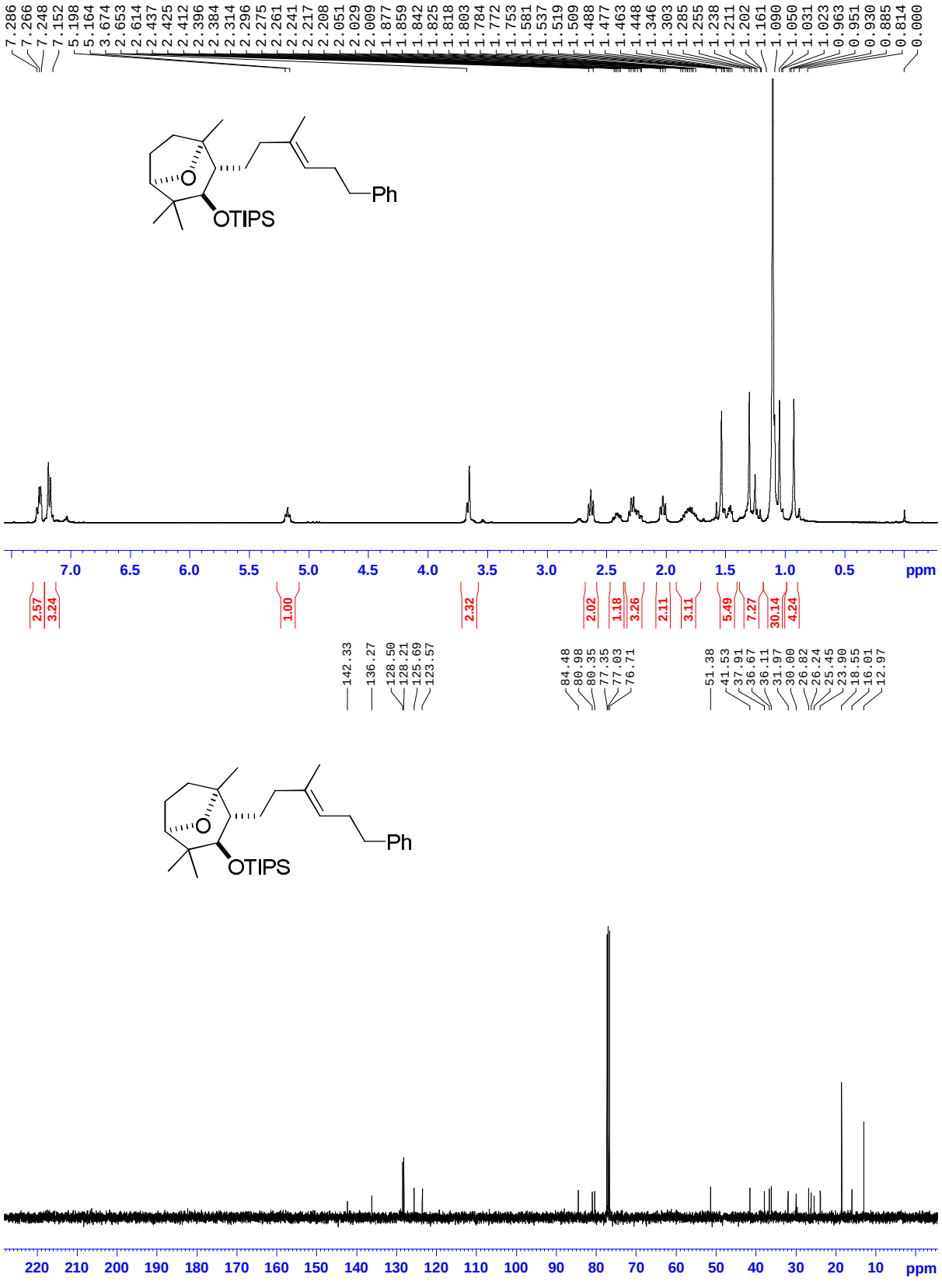
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0.000
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OTIPS
Ph
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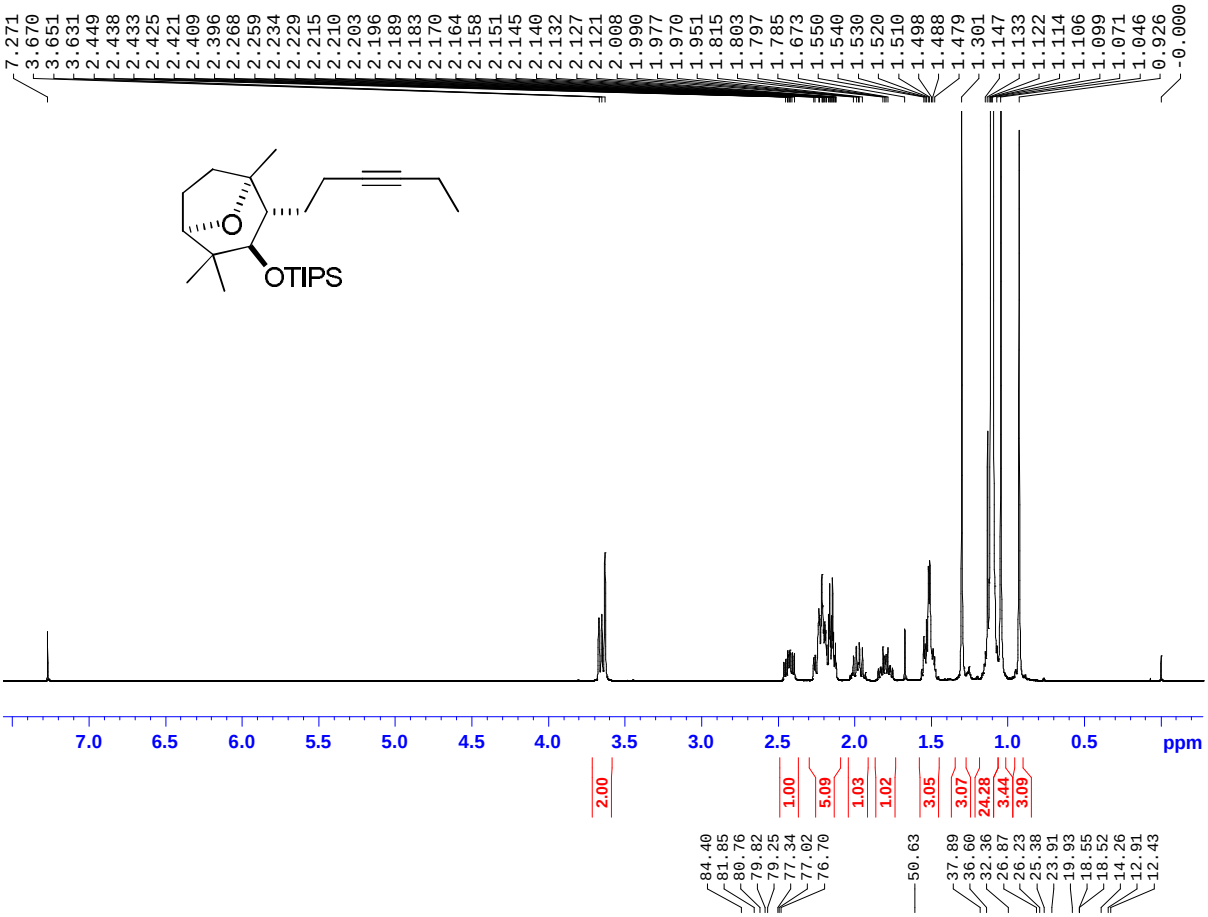
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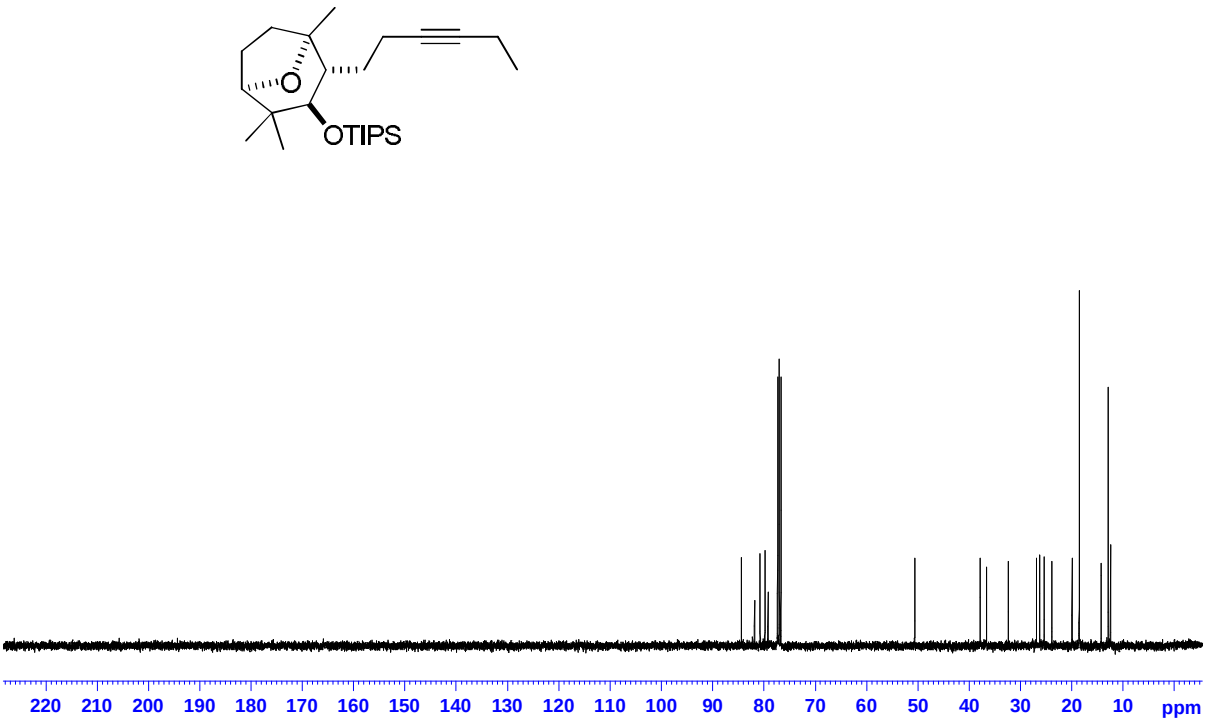
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18.52
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12.43
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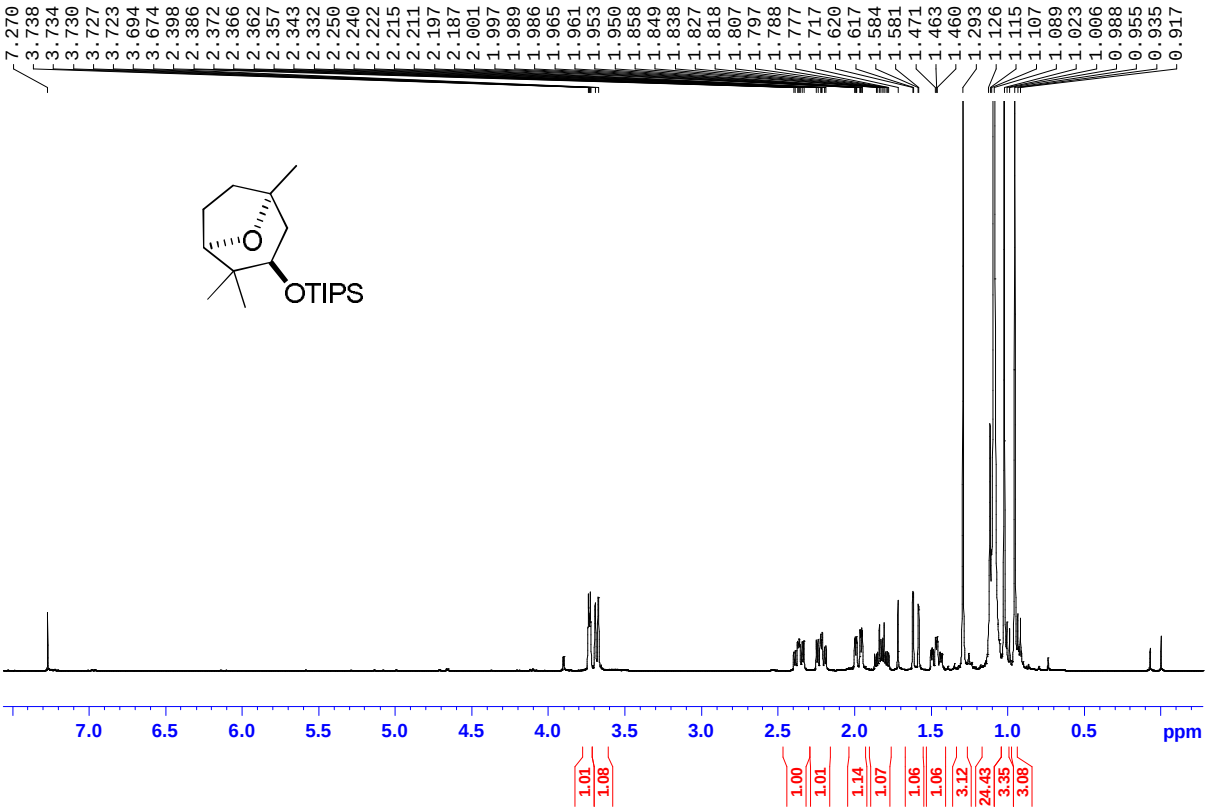
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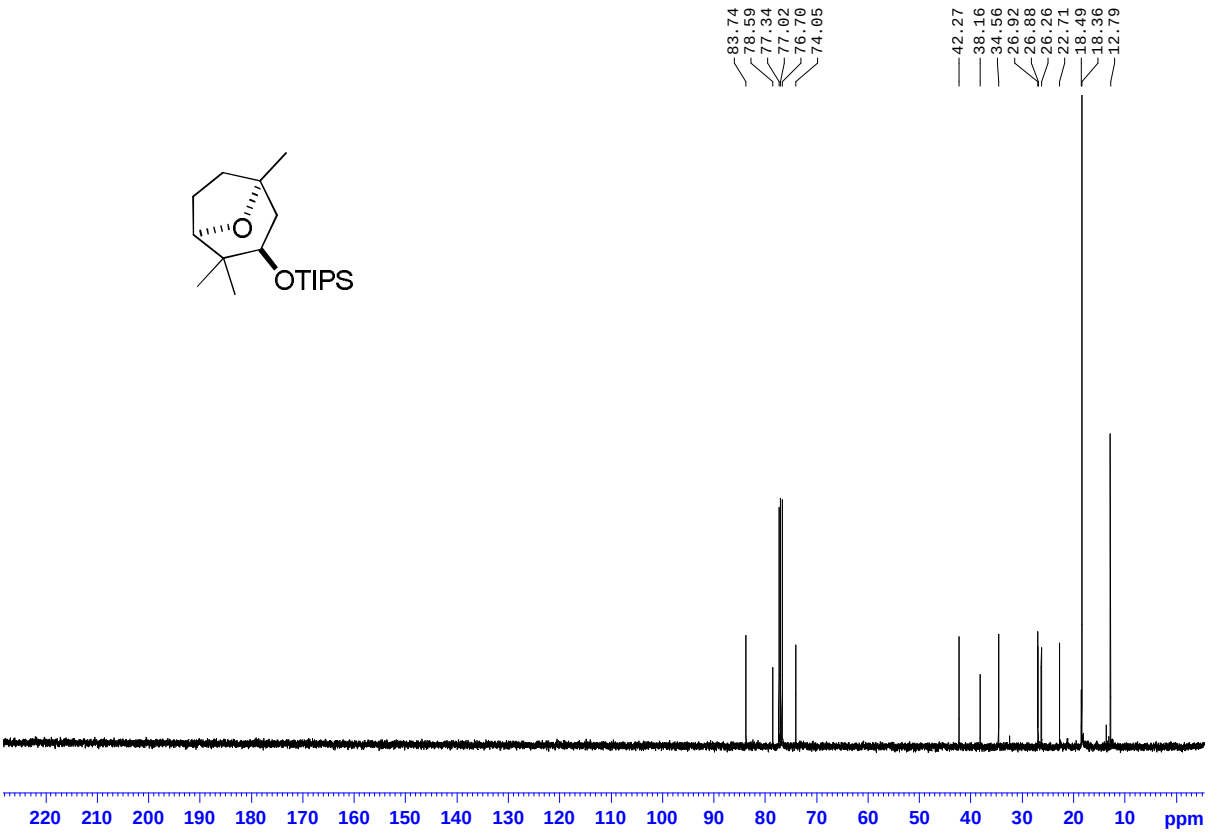
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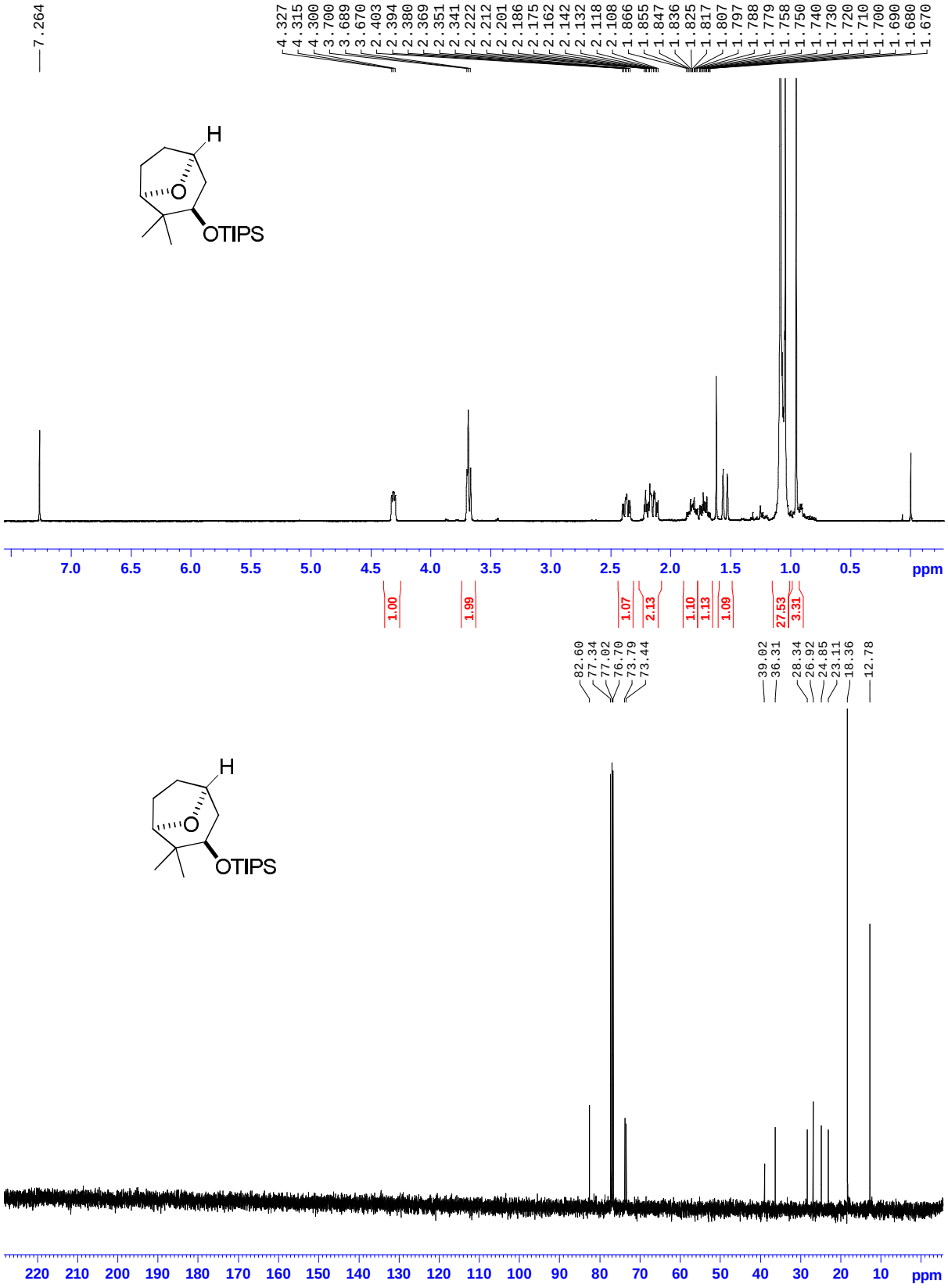
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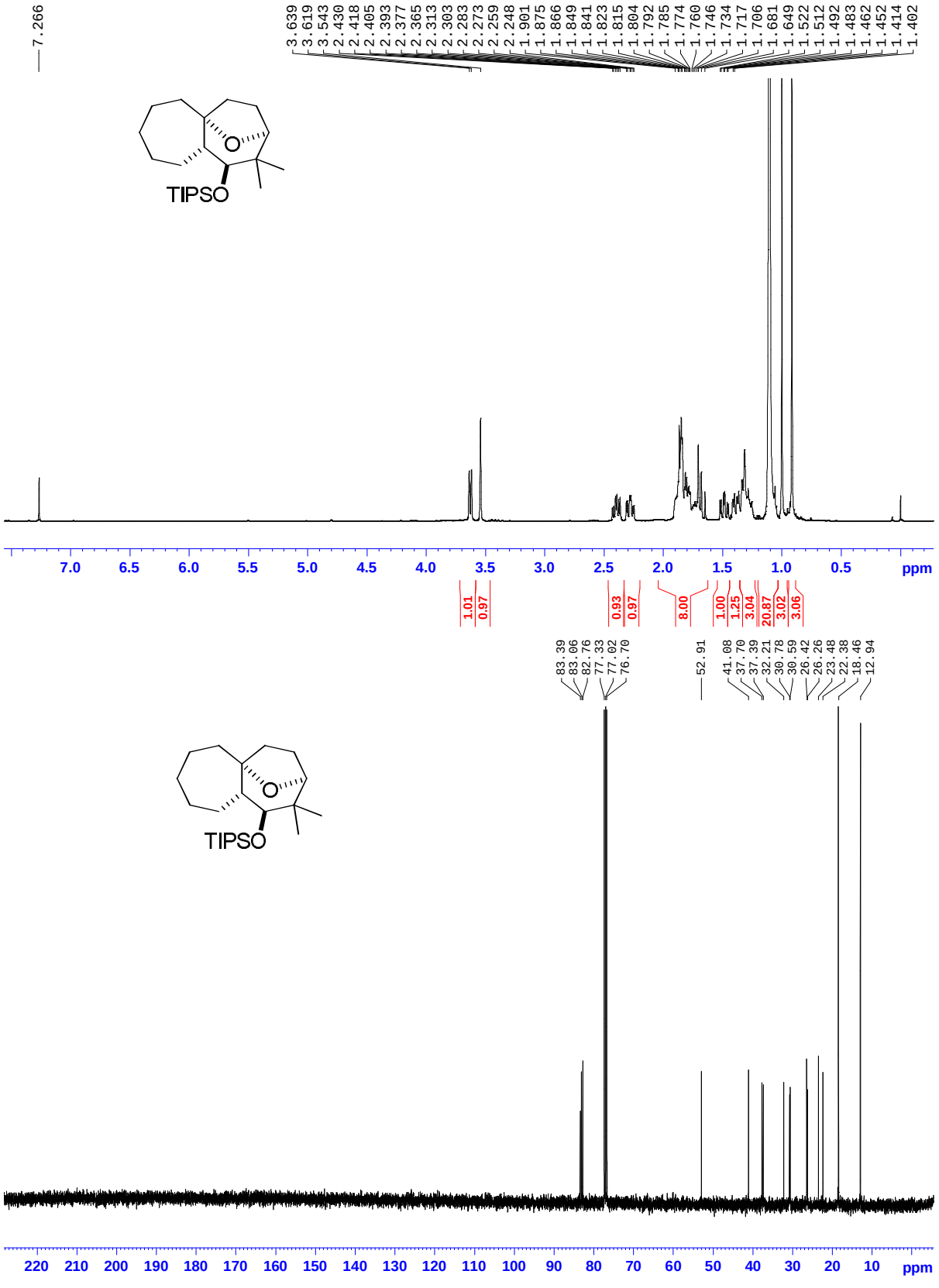
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TIPSO
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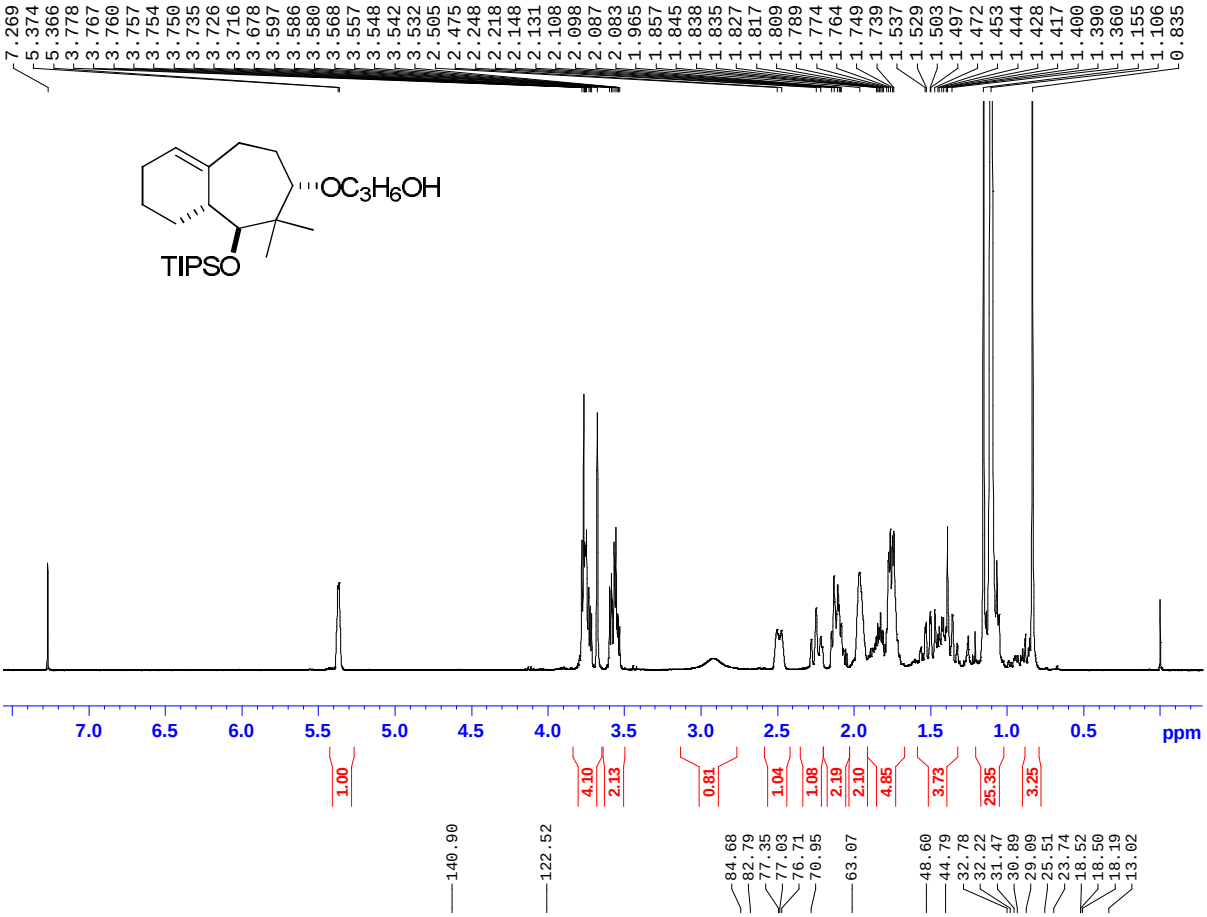
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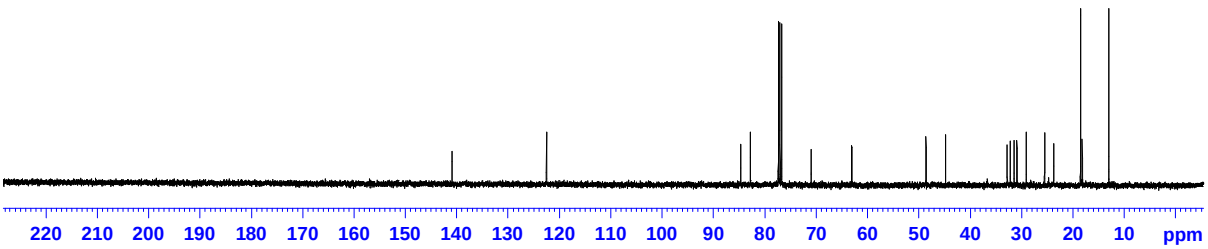
OC3H6OH
TIPSO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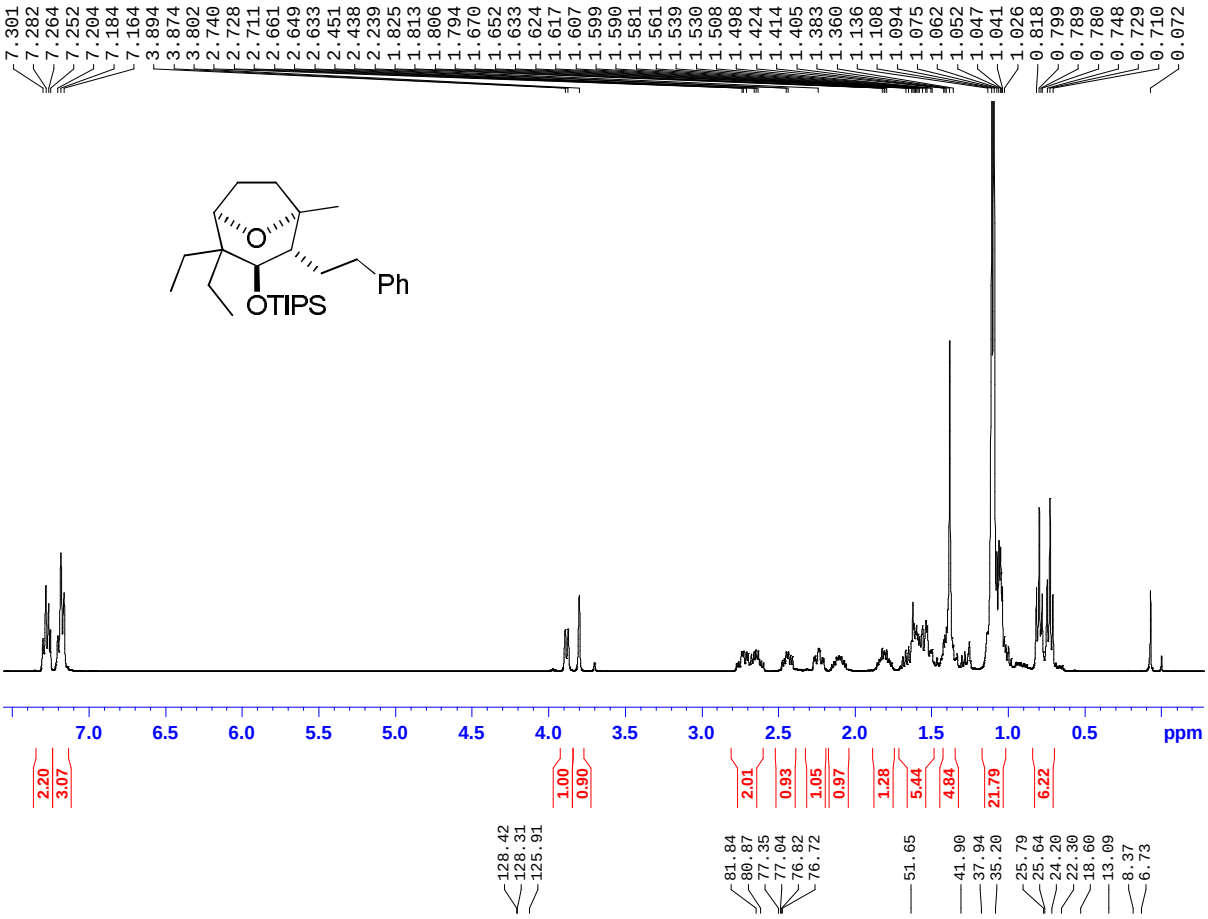
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Ph
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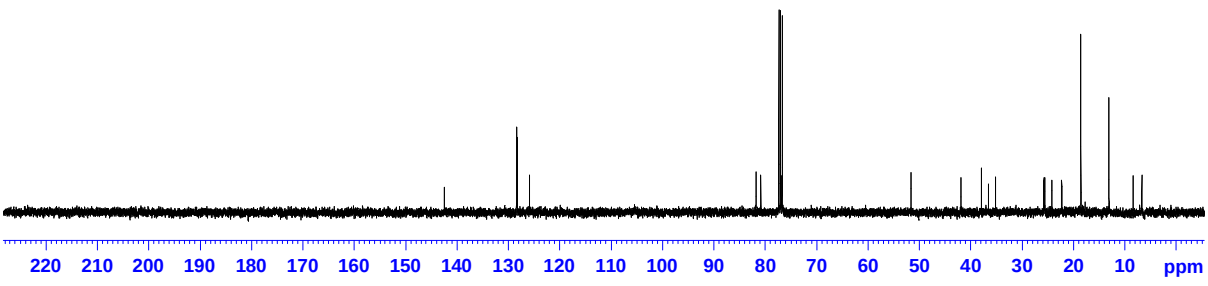
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O
Ph
OTIPS

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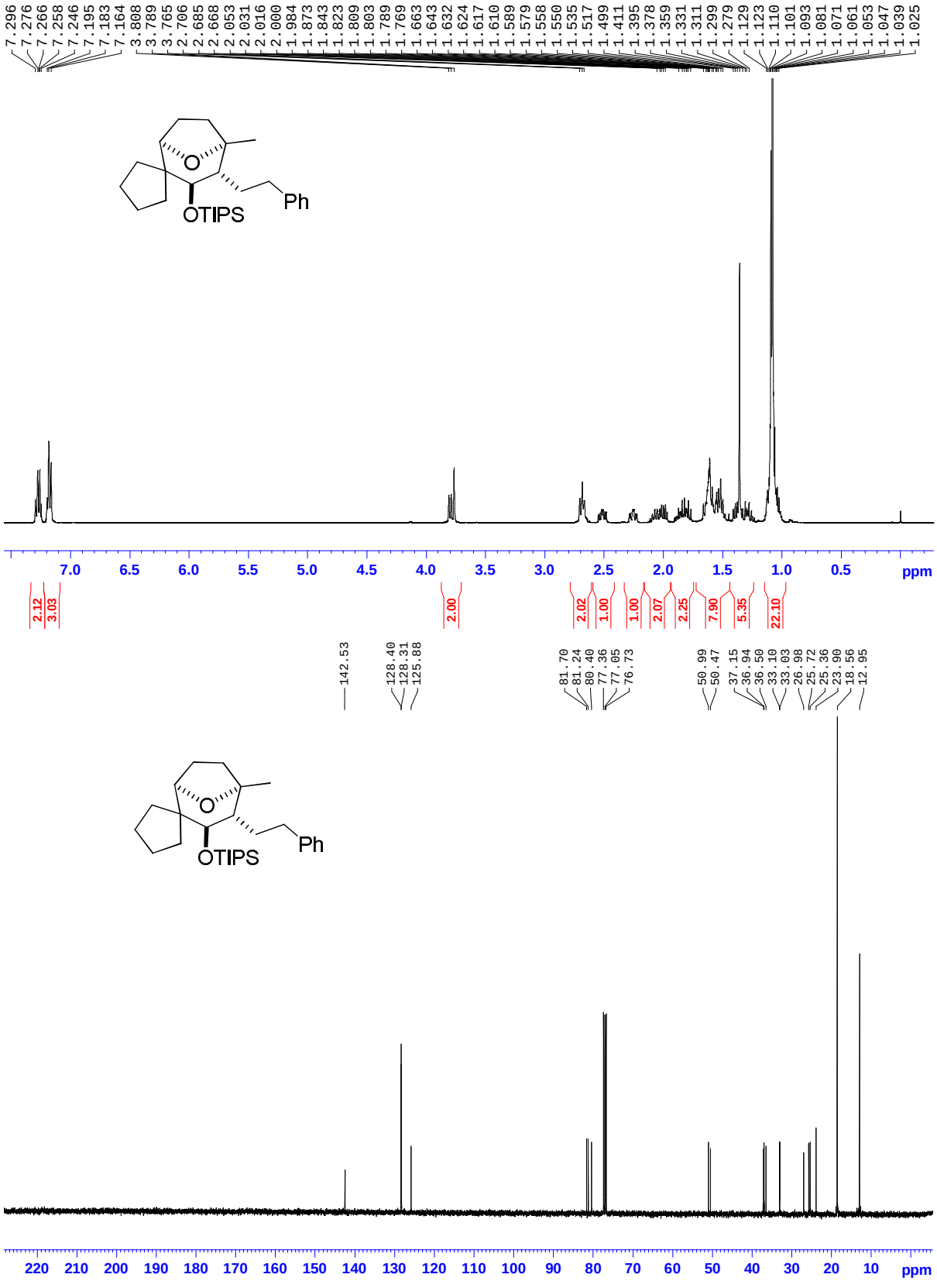
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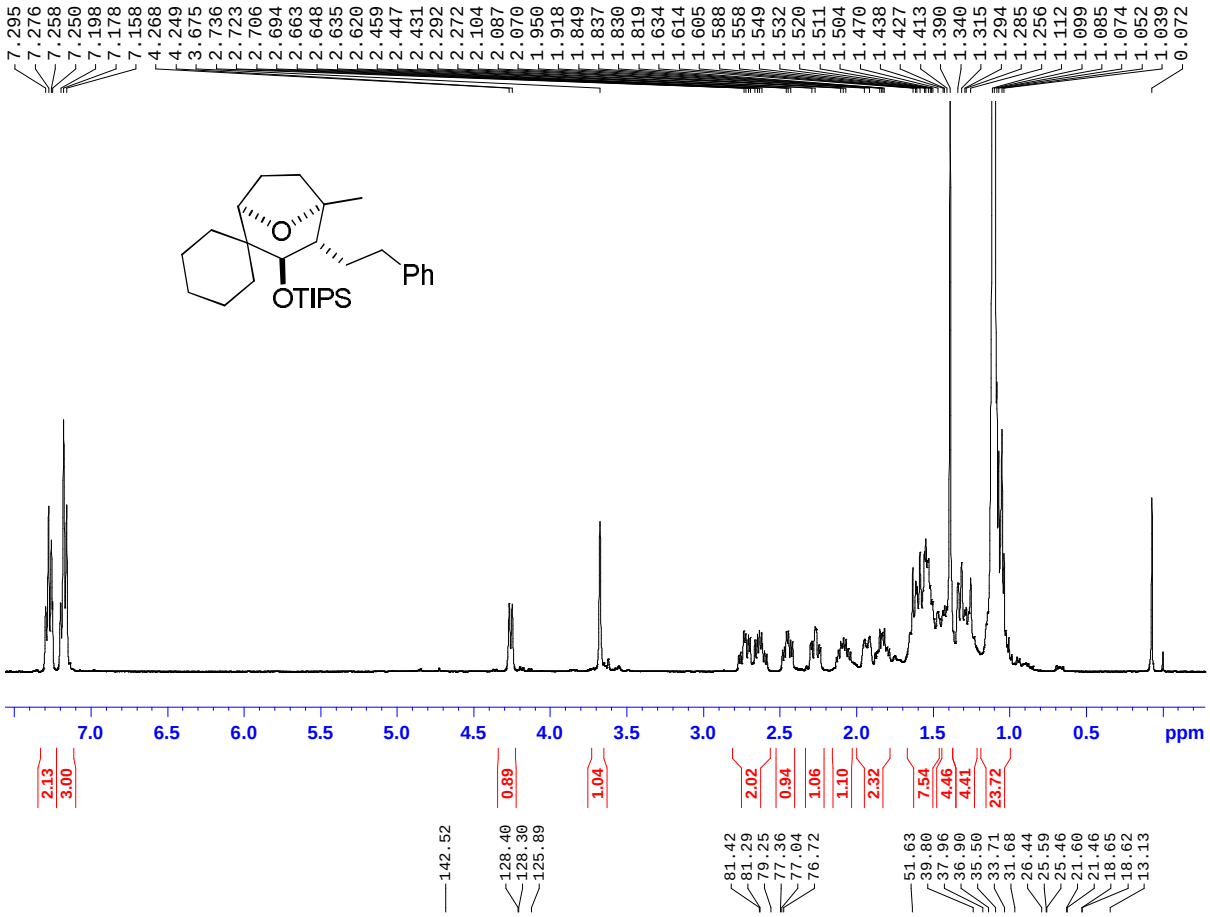
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Ph
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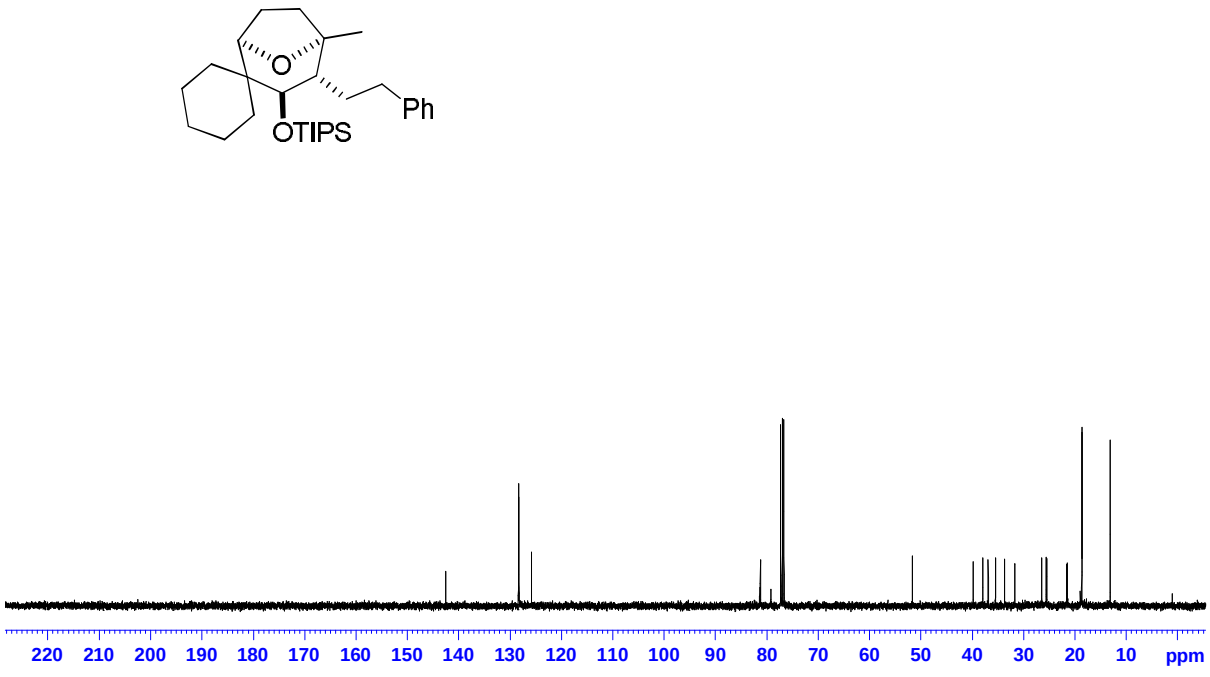
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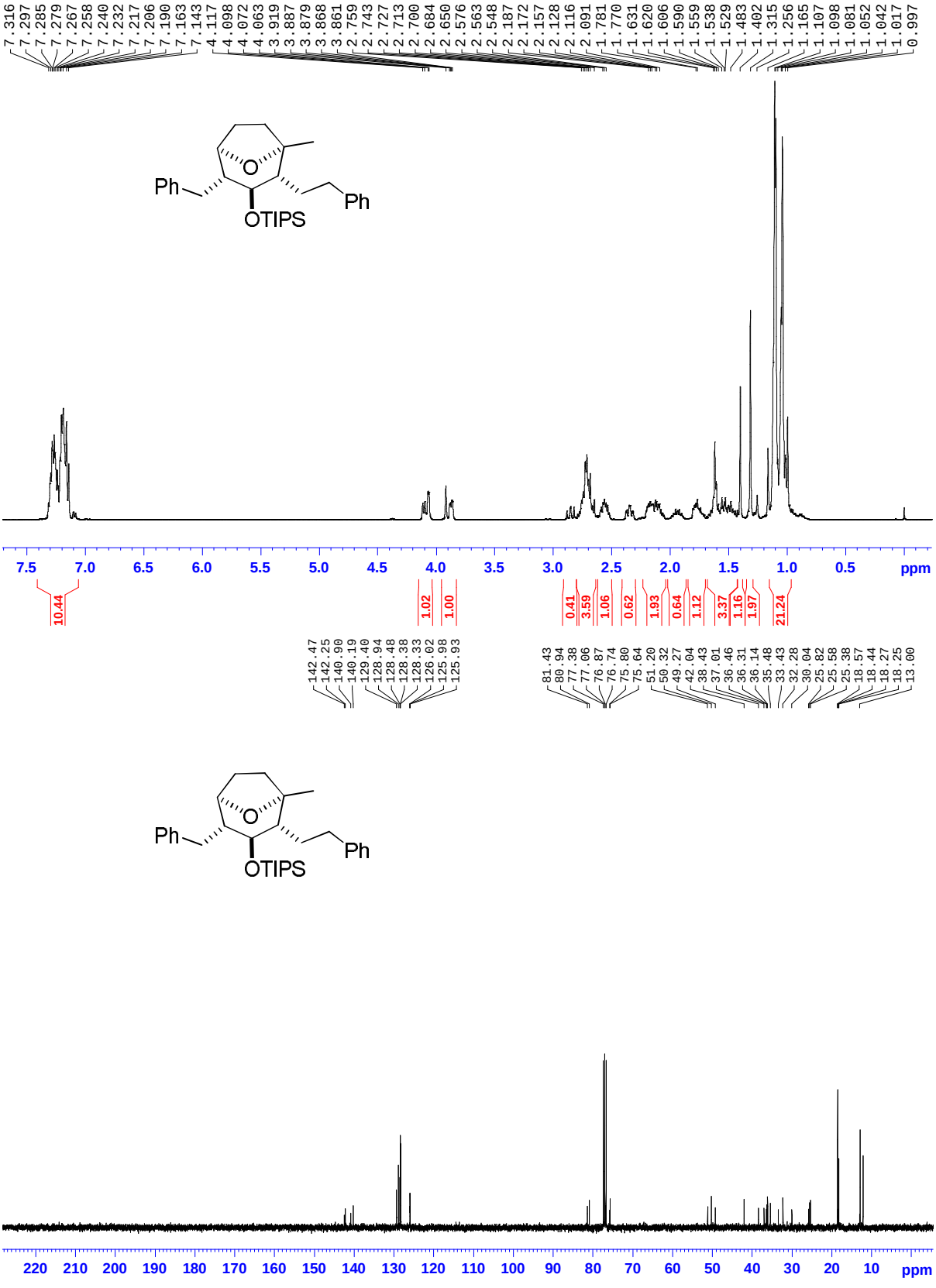
10
ppm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Ph
OTIPS
Ph
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10.44 1.02 1.00 0.41 3.59 1.06 0.62 1.93 0.64 1.12 3.37 1.16 1.97 21.24
142.47 142.25 140.90 140.19 129.40 128.94 128.48 128.38 128.33 126.02 125.98 125.93
81.43 80.94 77.38 77.06 76.87 76.74 75.80 75.64 51.20 50.32 49.27 42.04 38.43 37.01 36.46 36.31 36.14 35.48 33.43 32.28 30.04 25.82 25.58 25.38 18.57 18.44 18.27 18.25 13.00
Ph
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Ph
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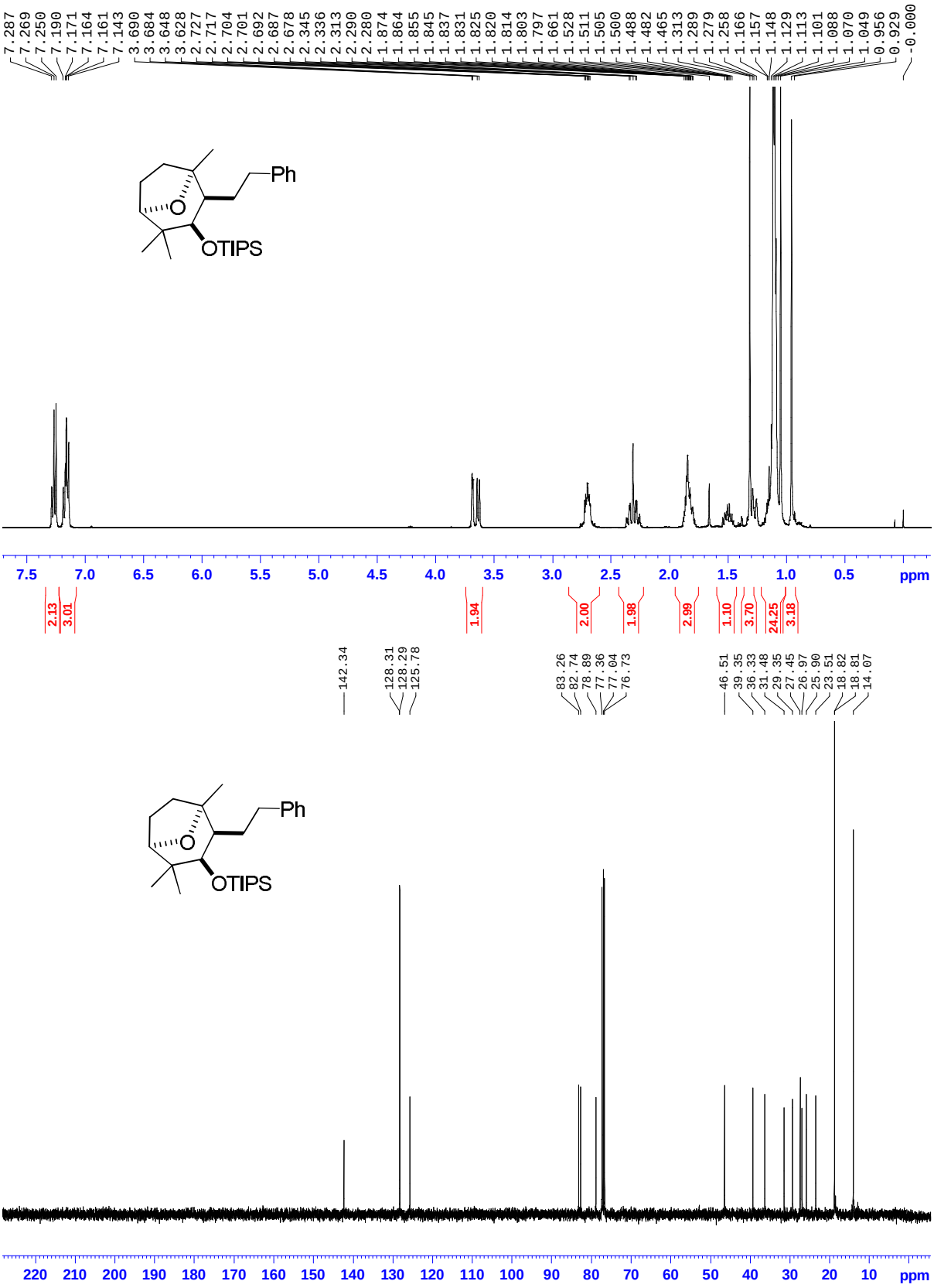
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OTIPS
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3.18
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77.36
77.04
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Ph
O
OTIPS
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210
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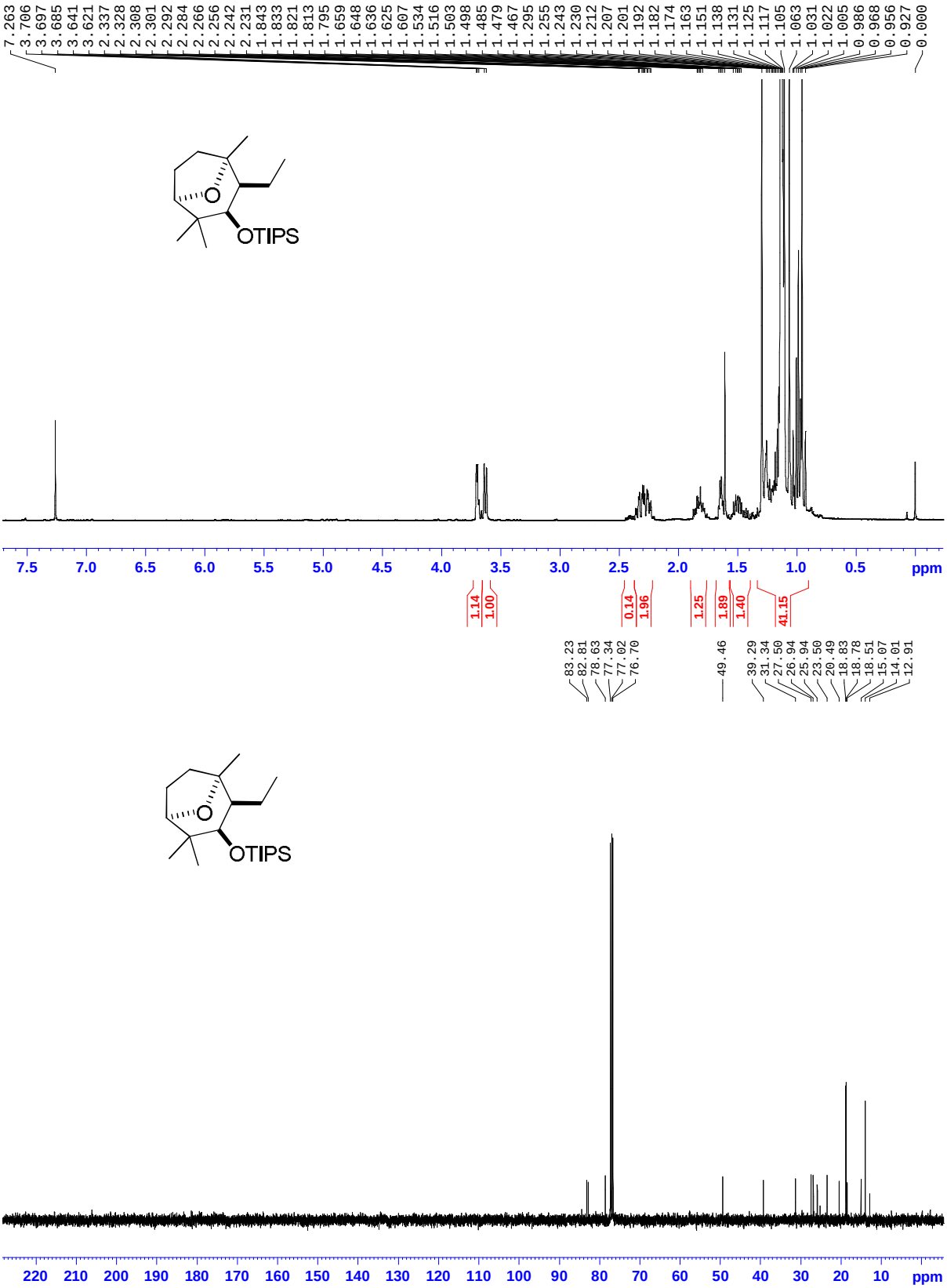
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1.516
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OTIPS
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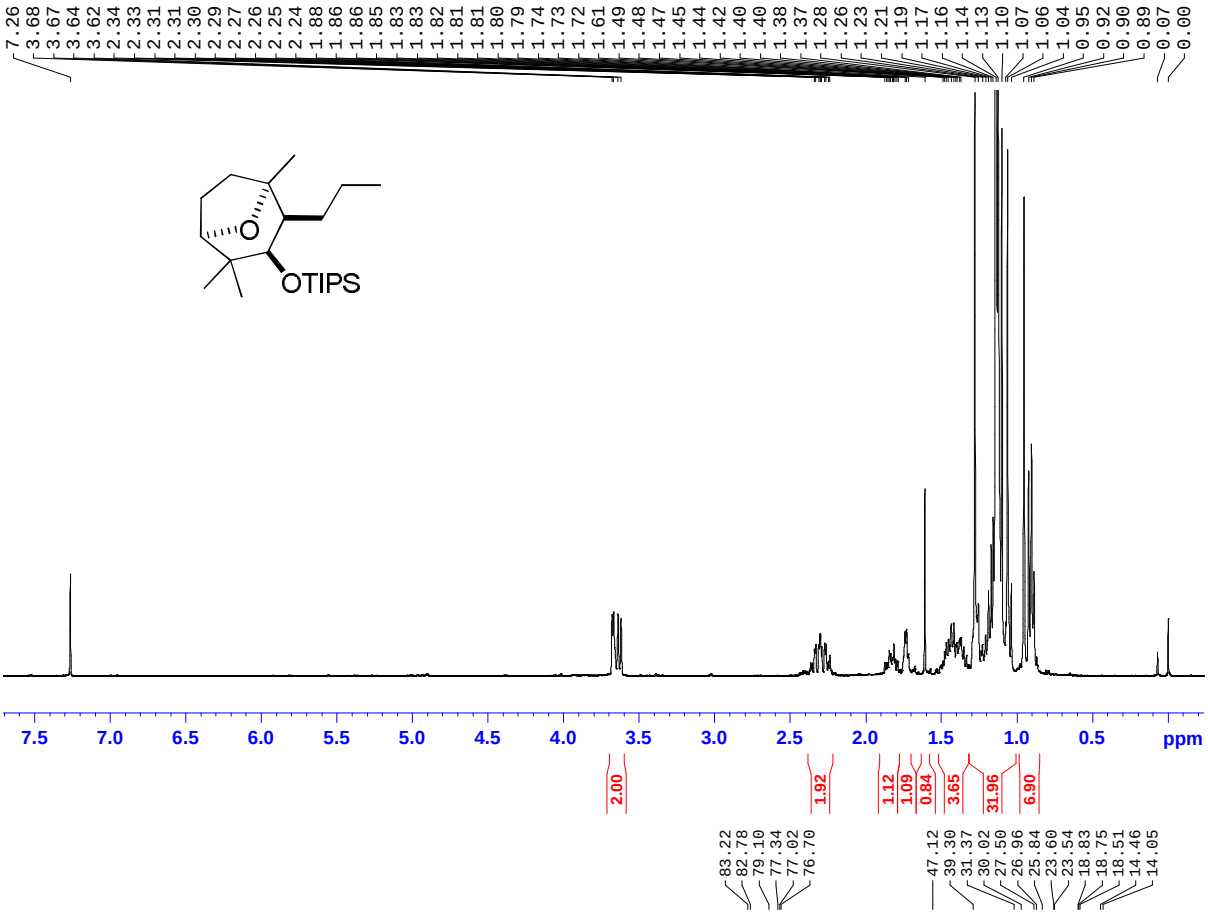
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O
OTIPS
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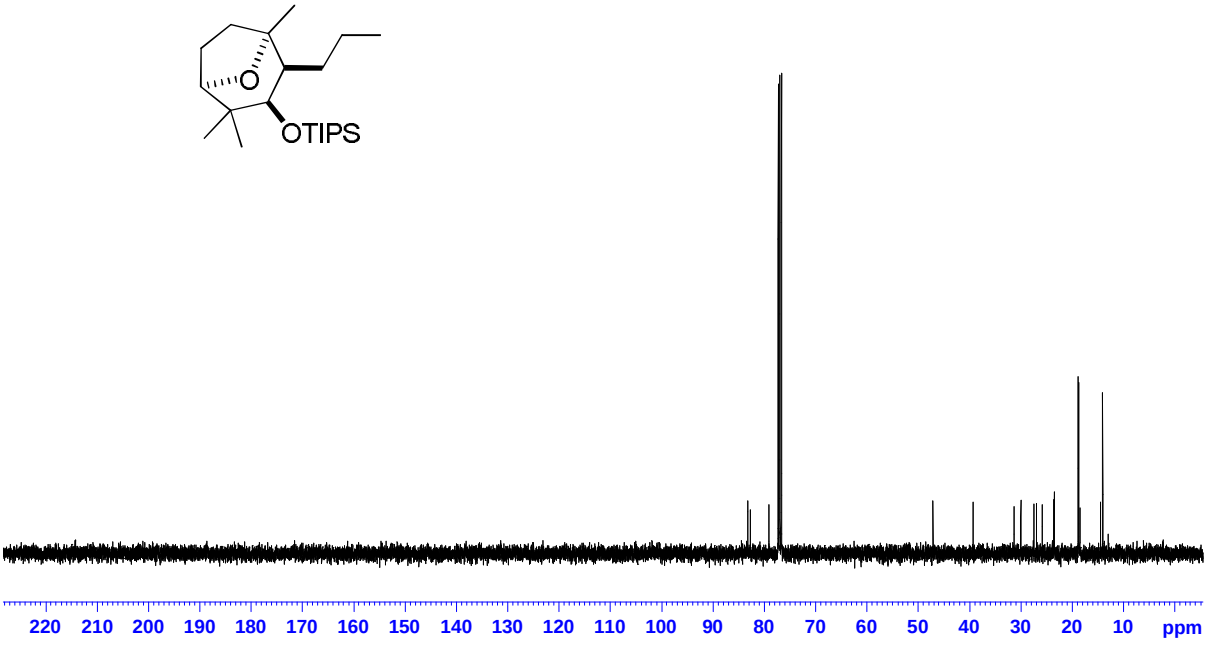
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OTIPS
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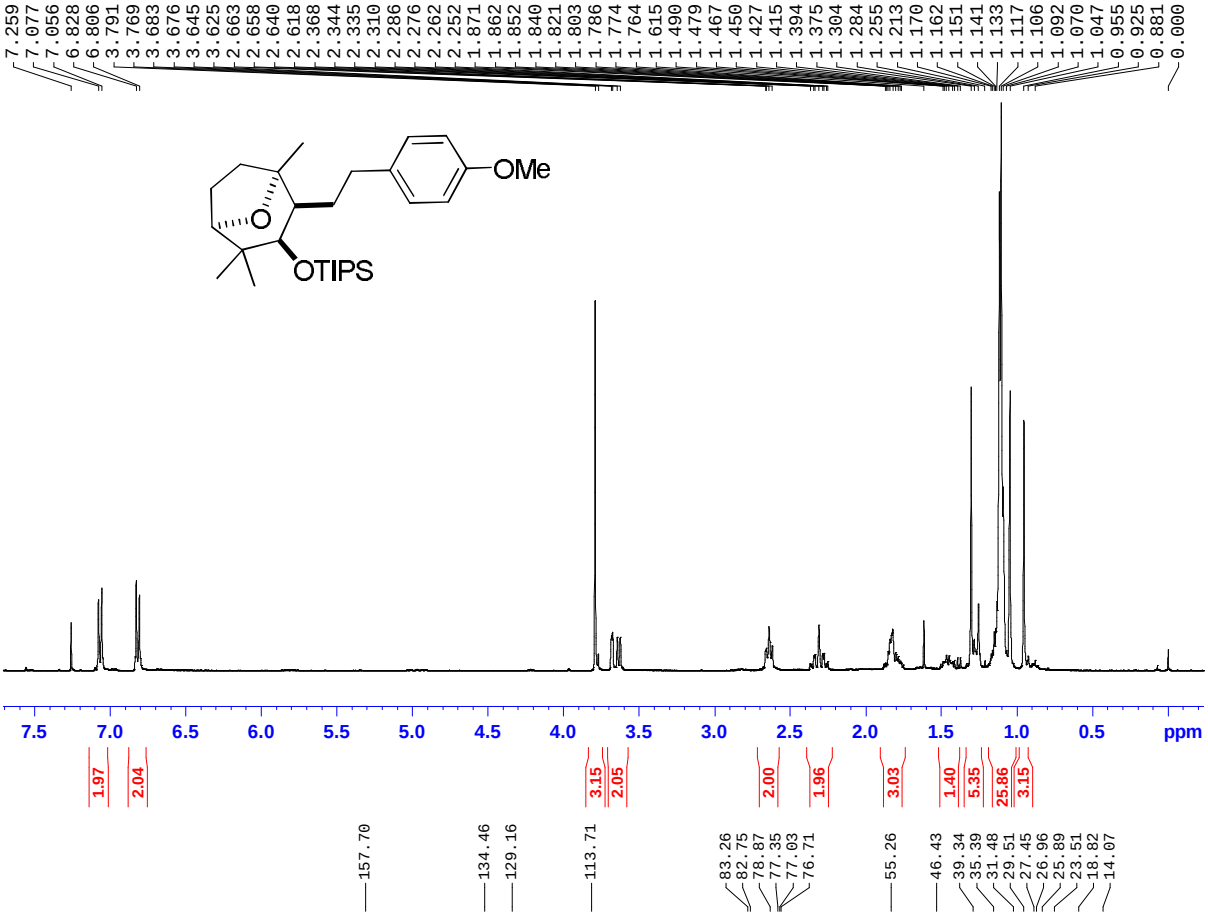
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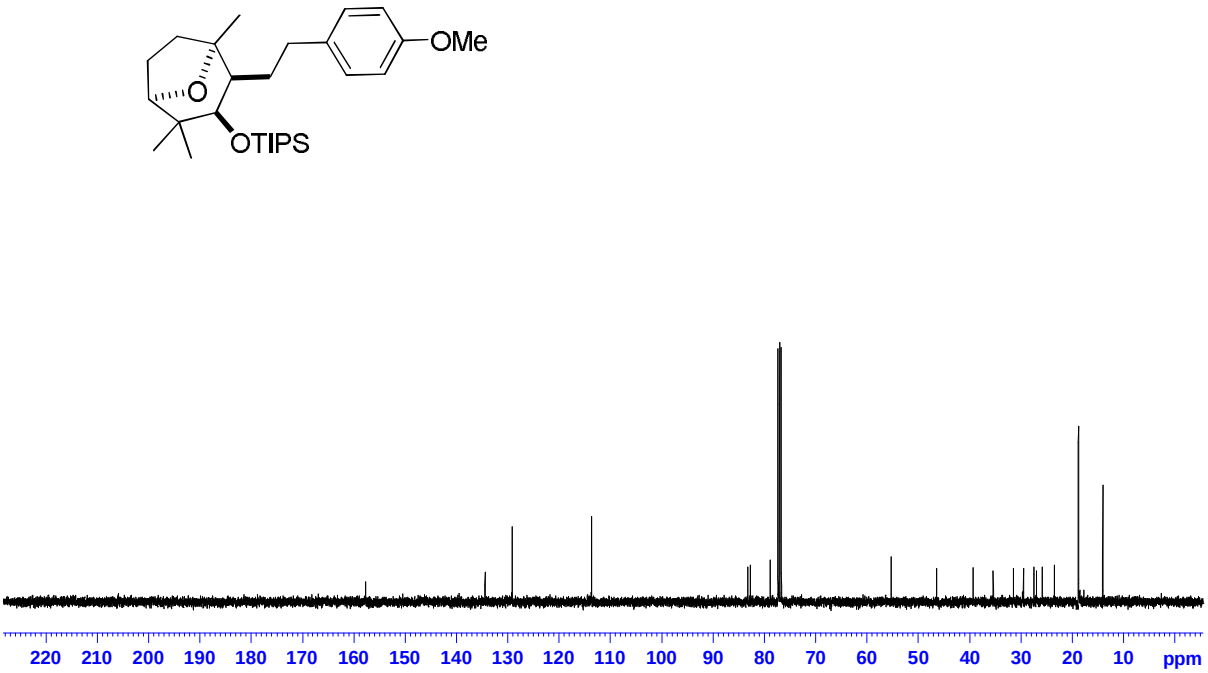
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OTIPS
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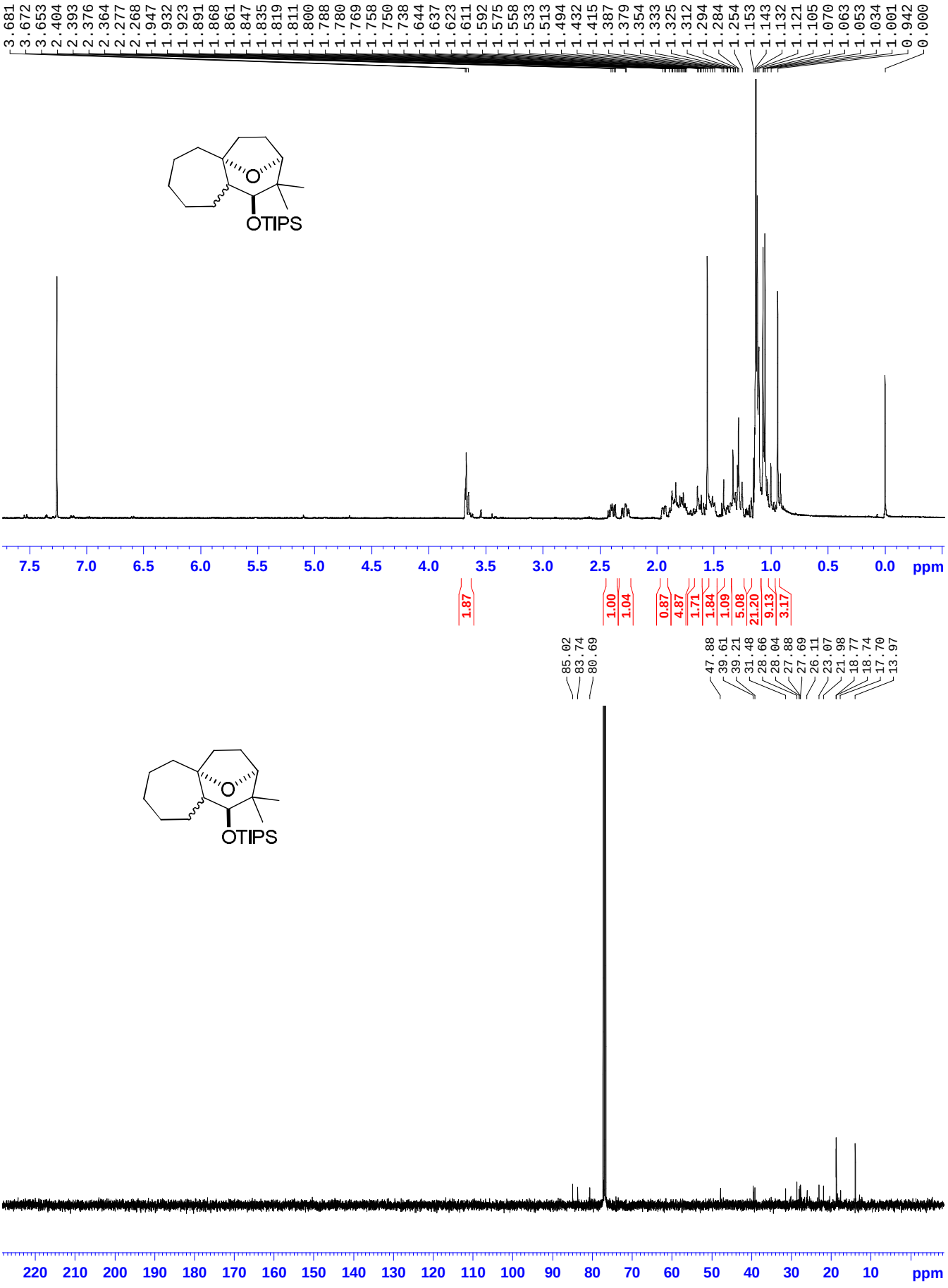
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OTIPS
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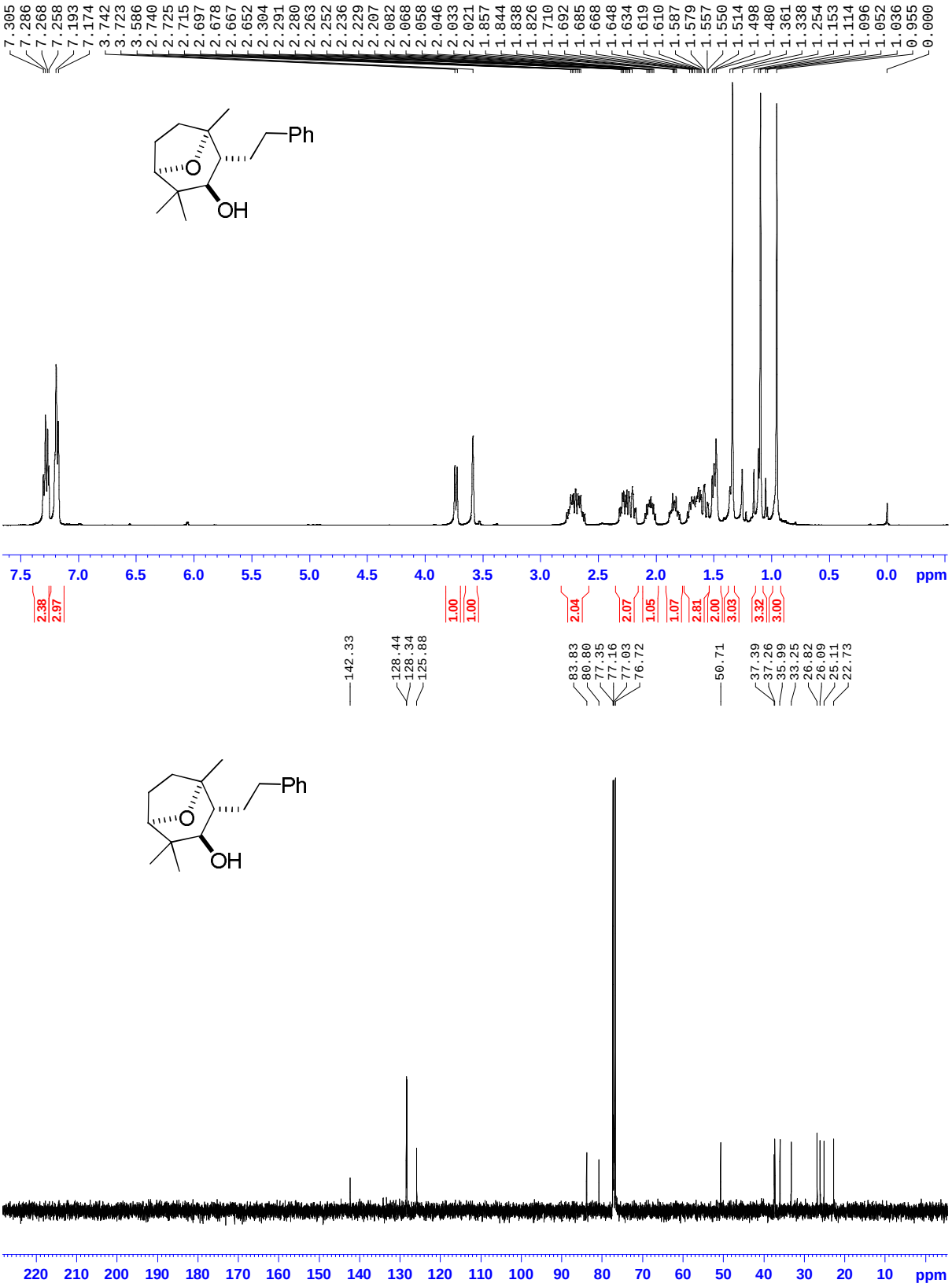
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Ph
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OH
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35.99
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26.82
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22.73
Ph
O
OH
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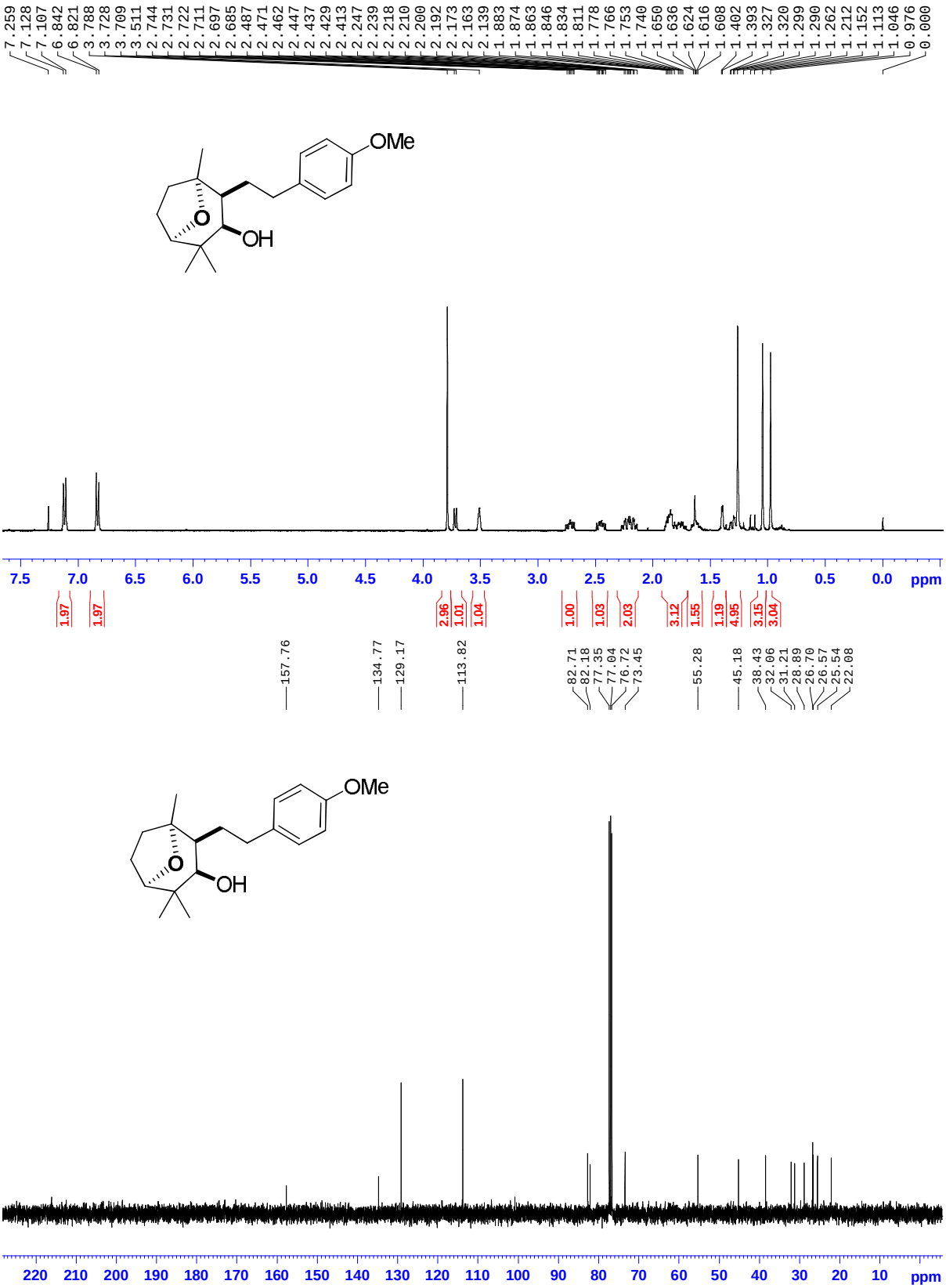
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O
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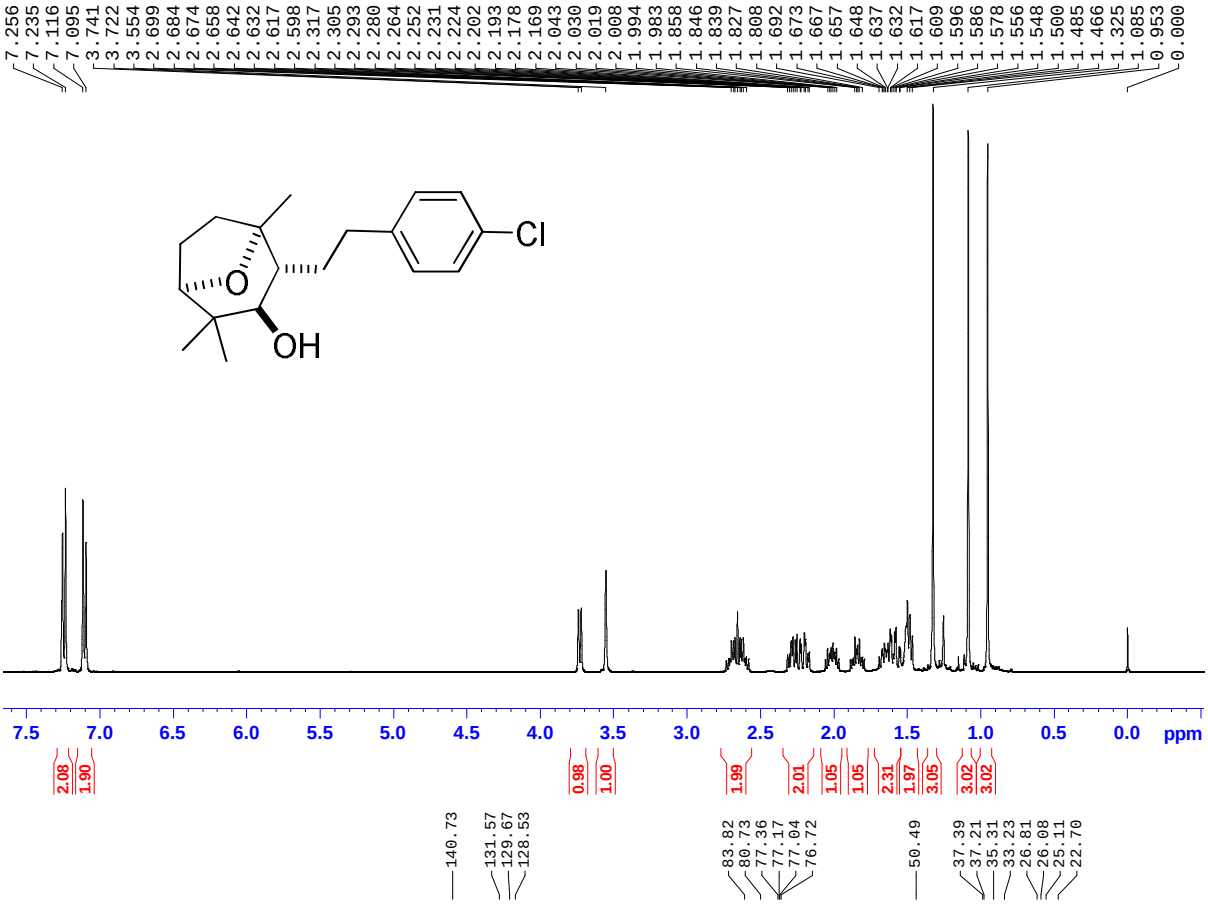
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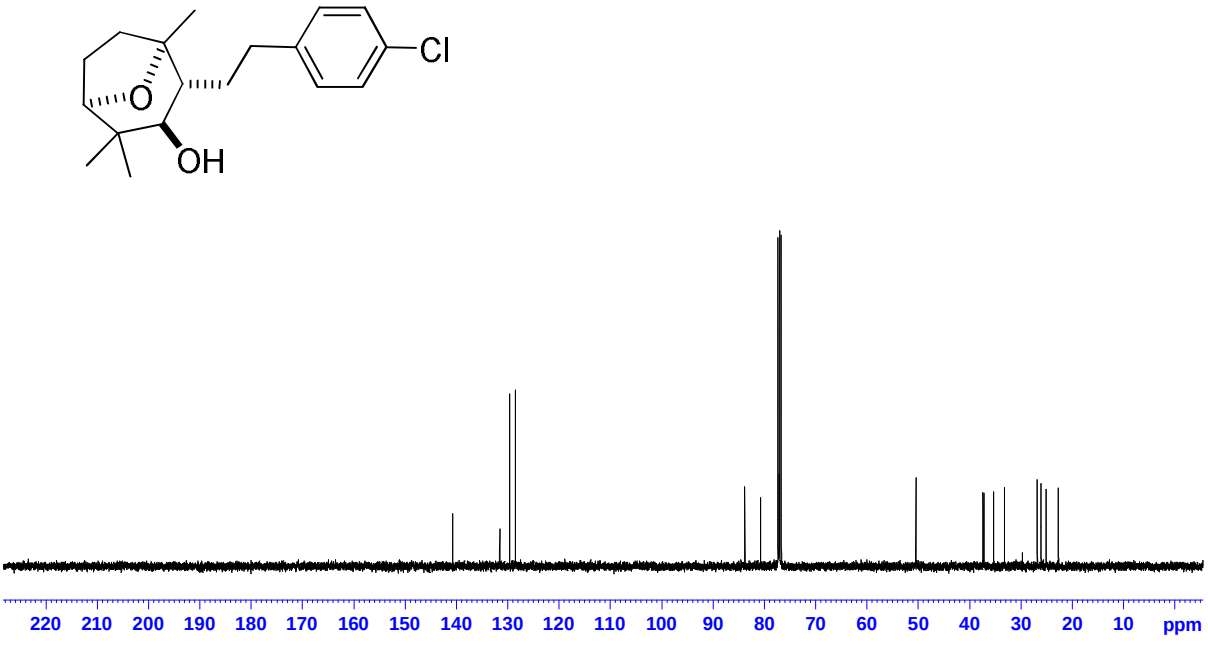
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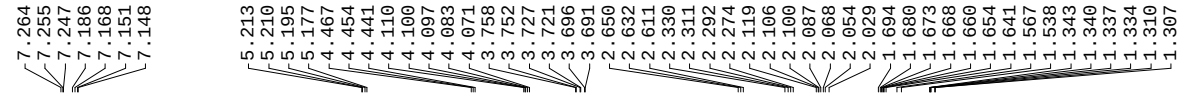
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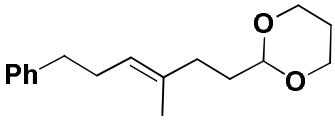
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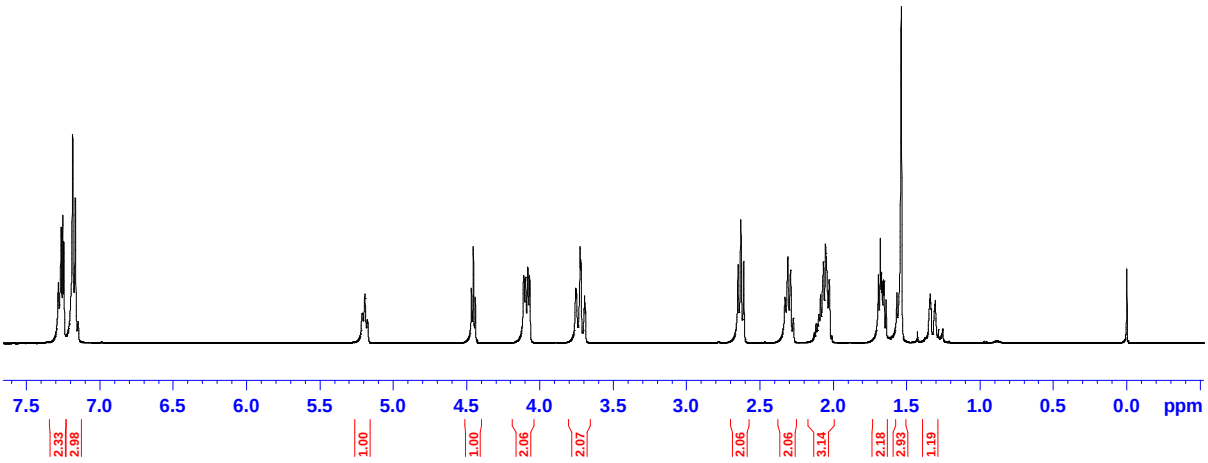
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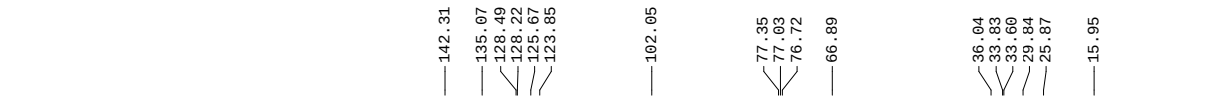
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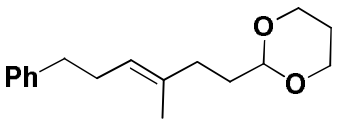
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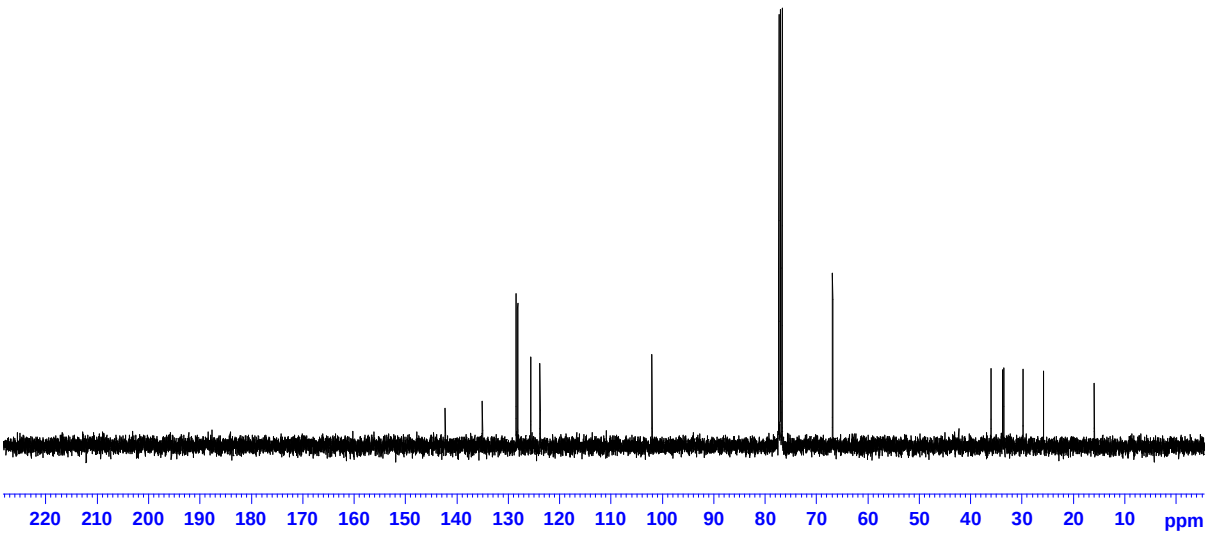
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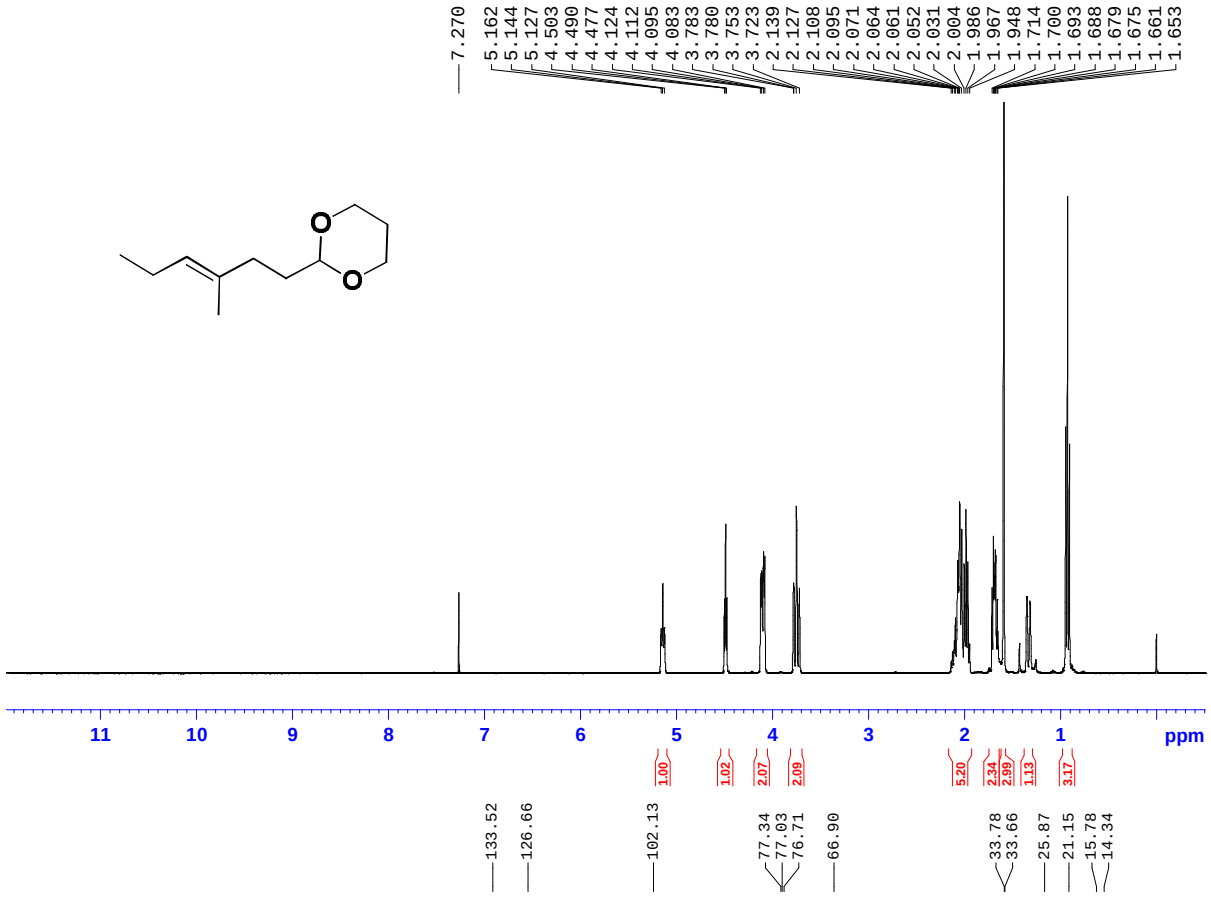
ppm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5
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2.07
2.09
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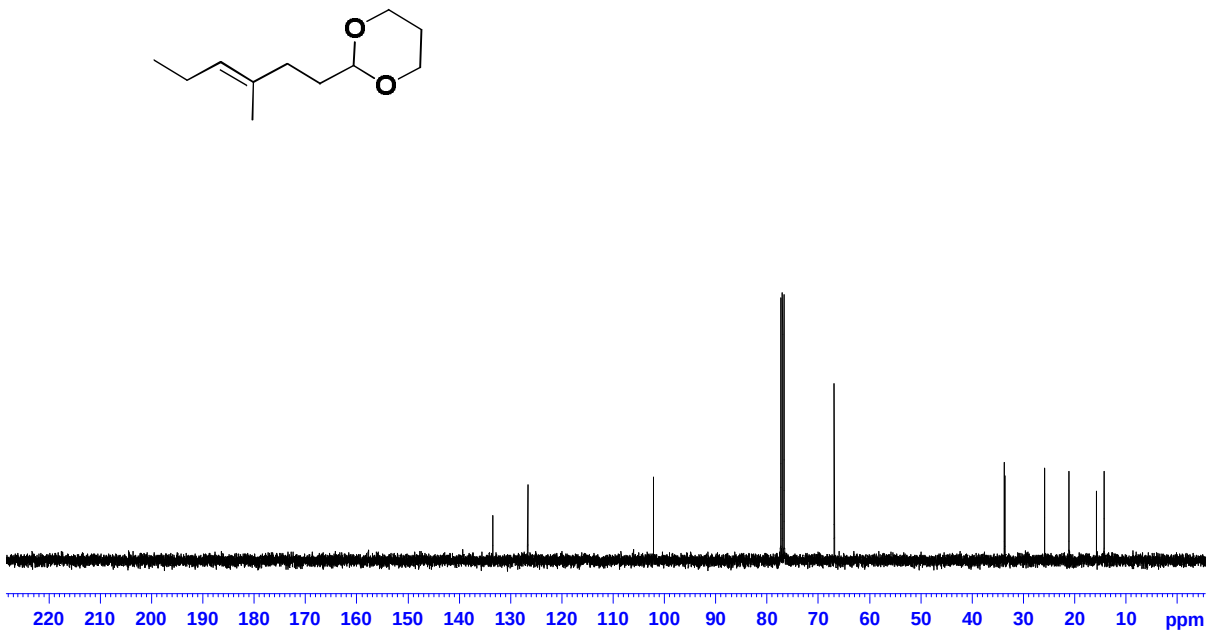
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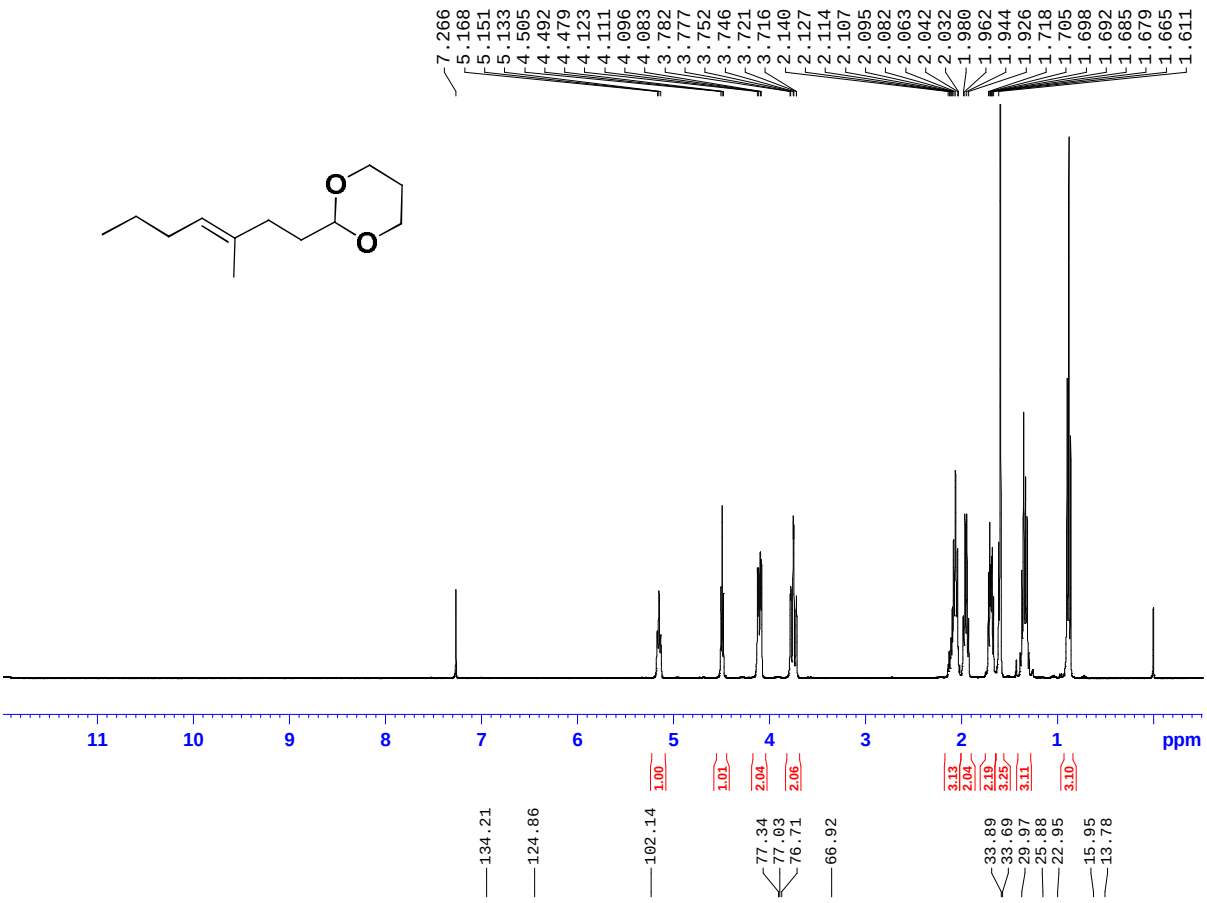
ppm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4
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ppm
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2.04
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3.10

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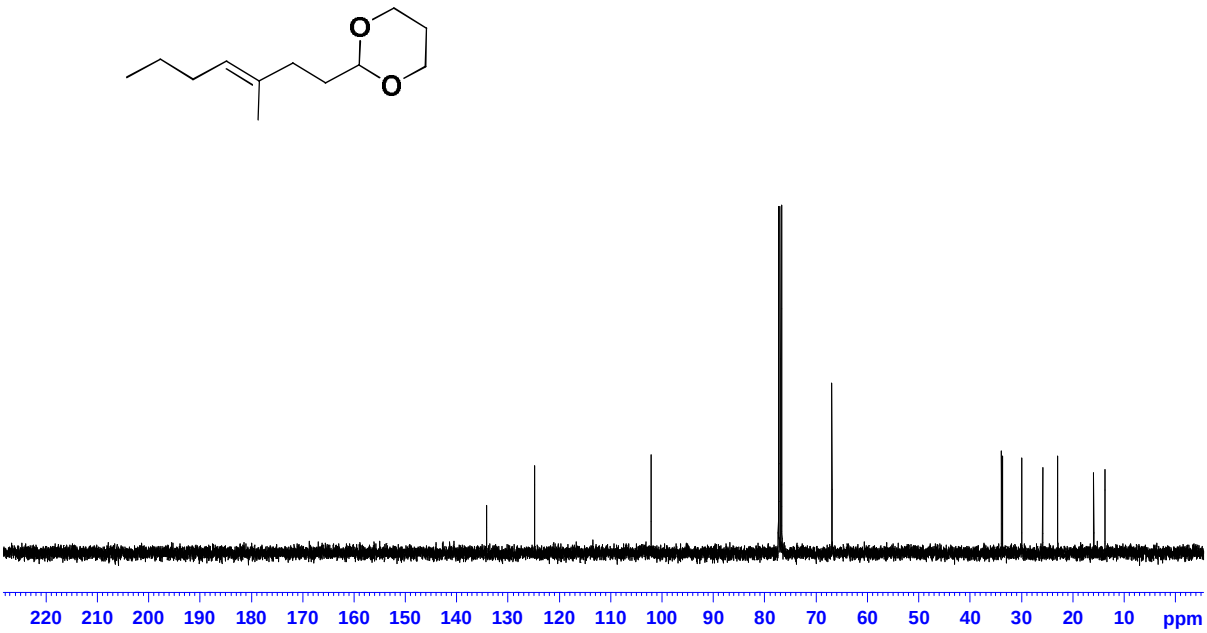
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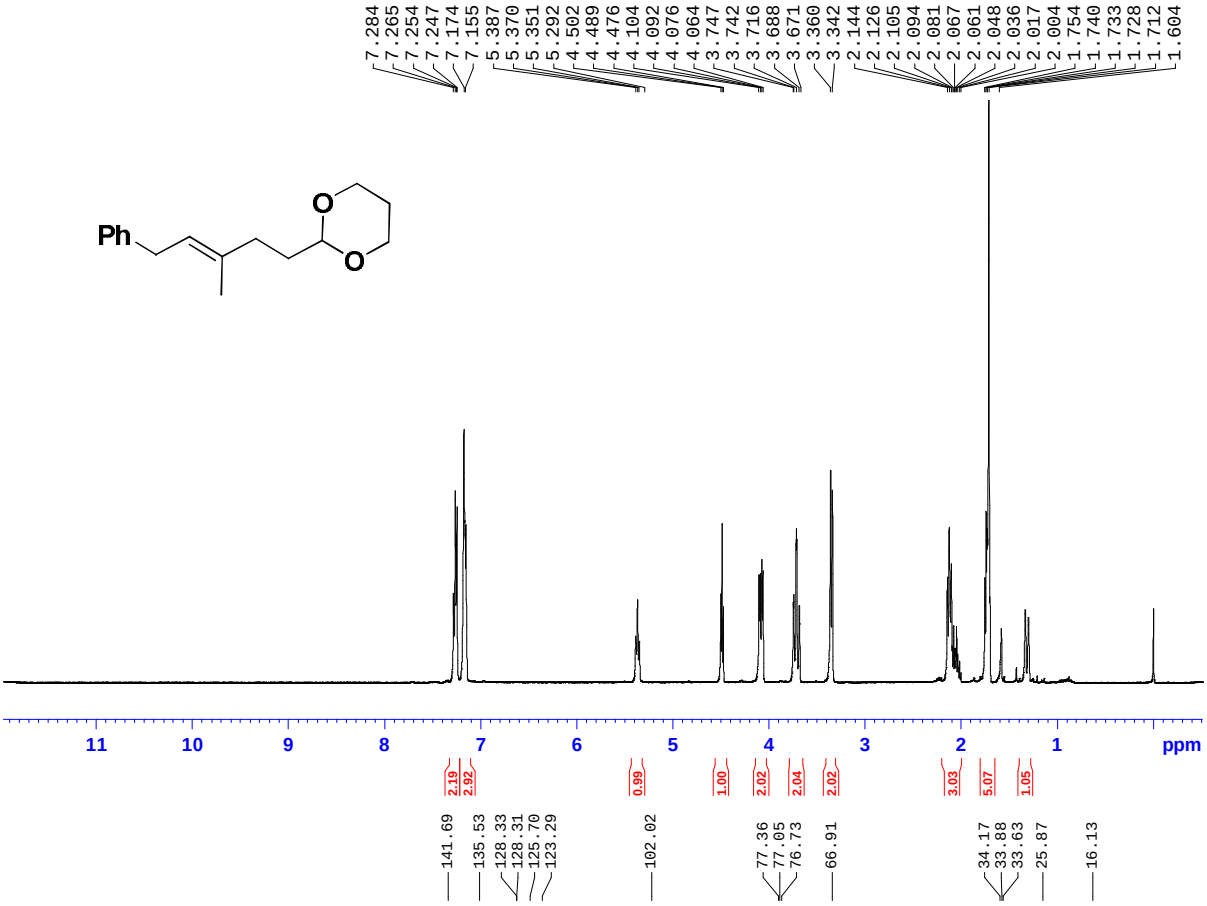
ppm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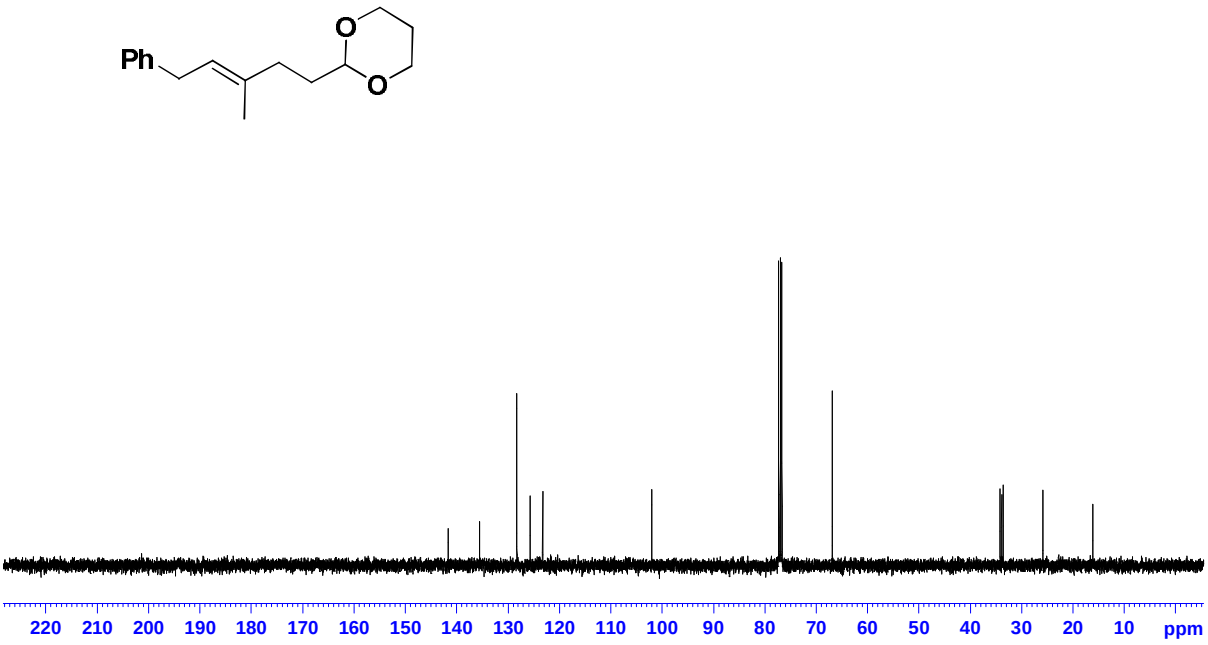
Ph
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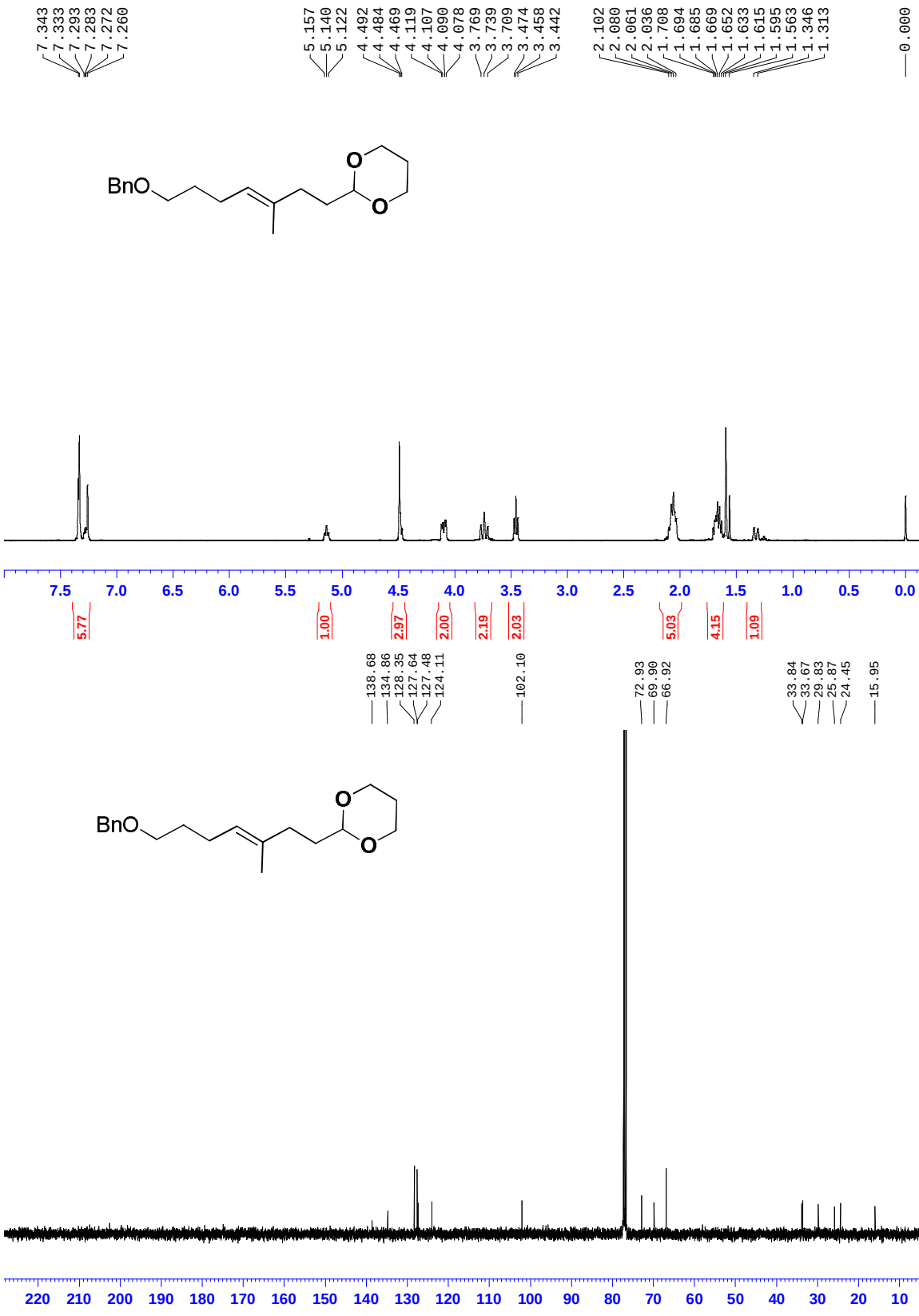
ppm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24.45
15.95
BnO
O
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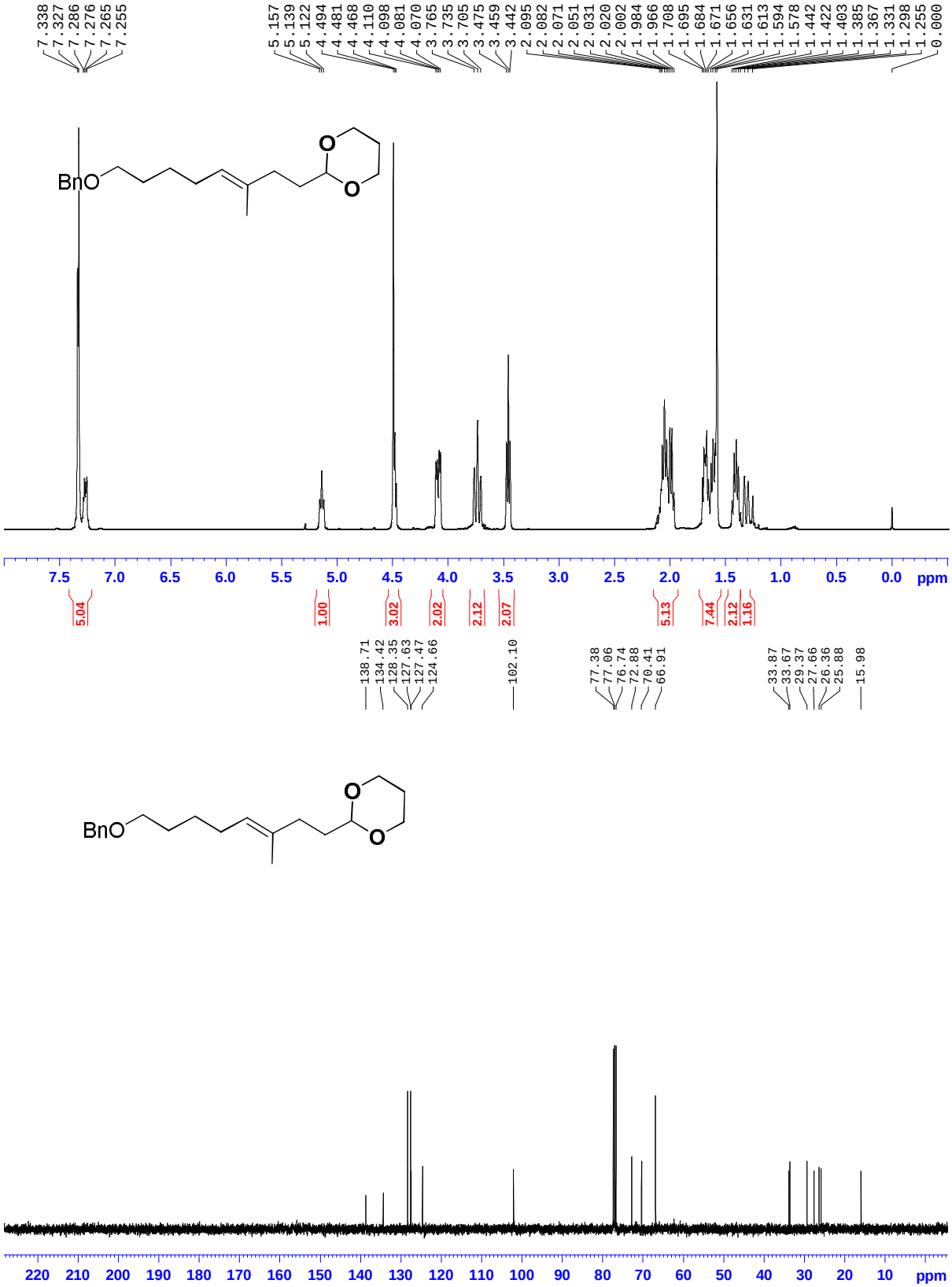
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BnO
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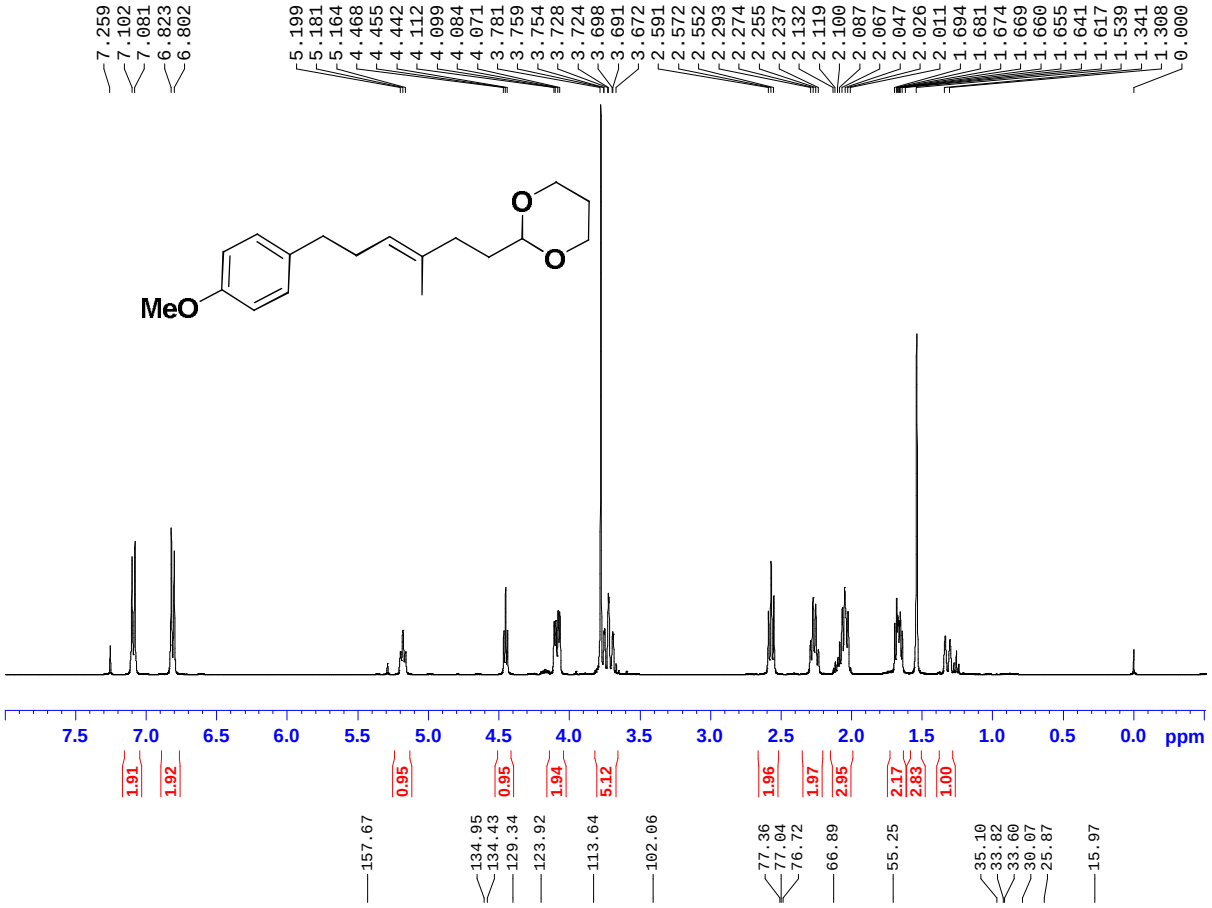
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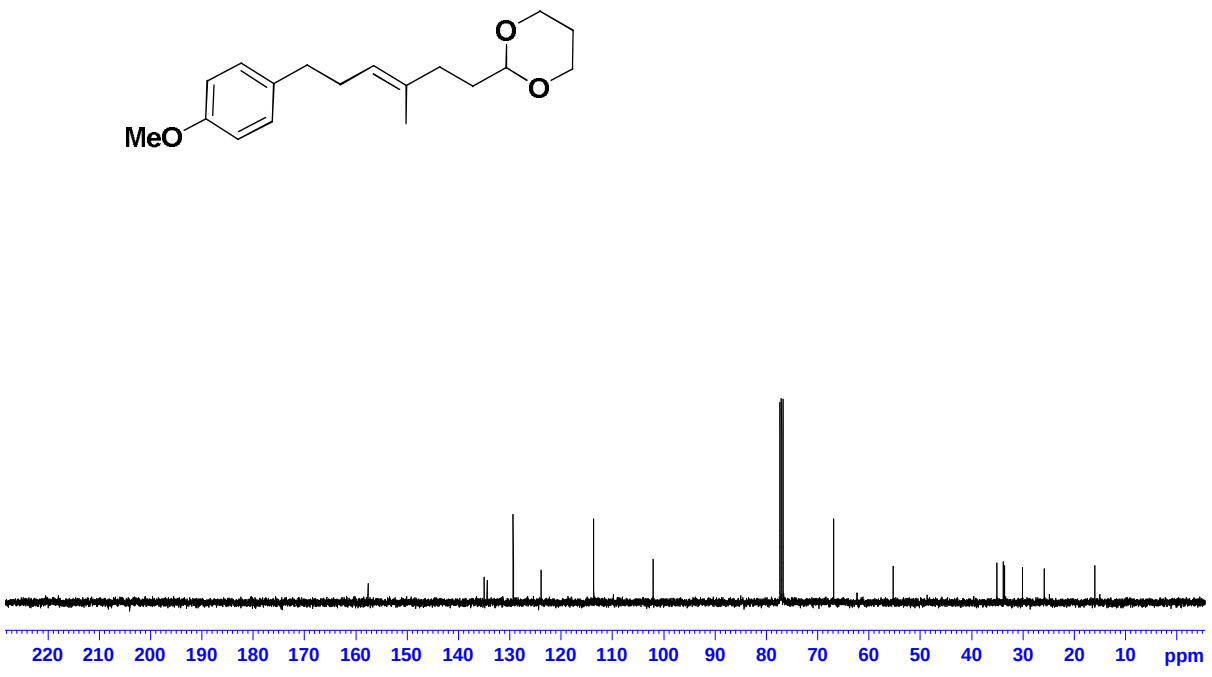
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O
O
MeO
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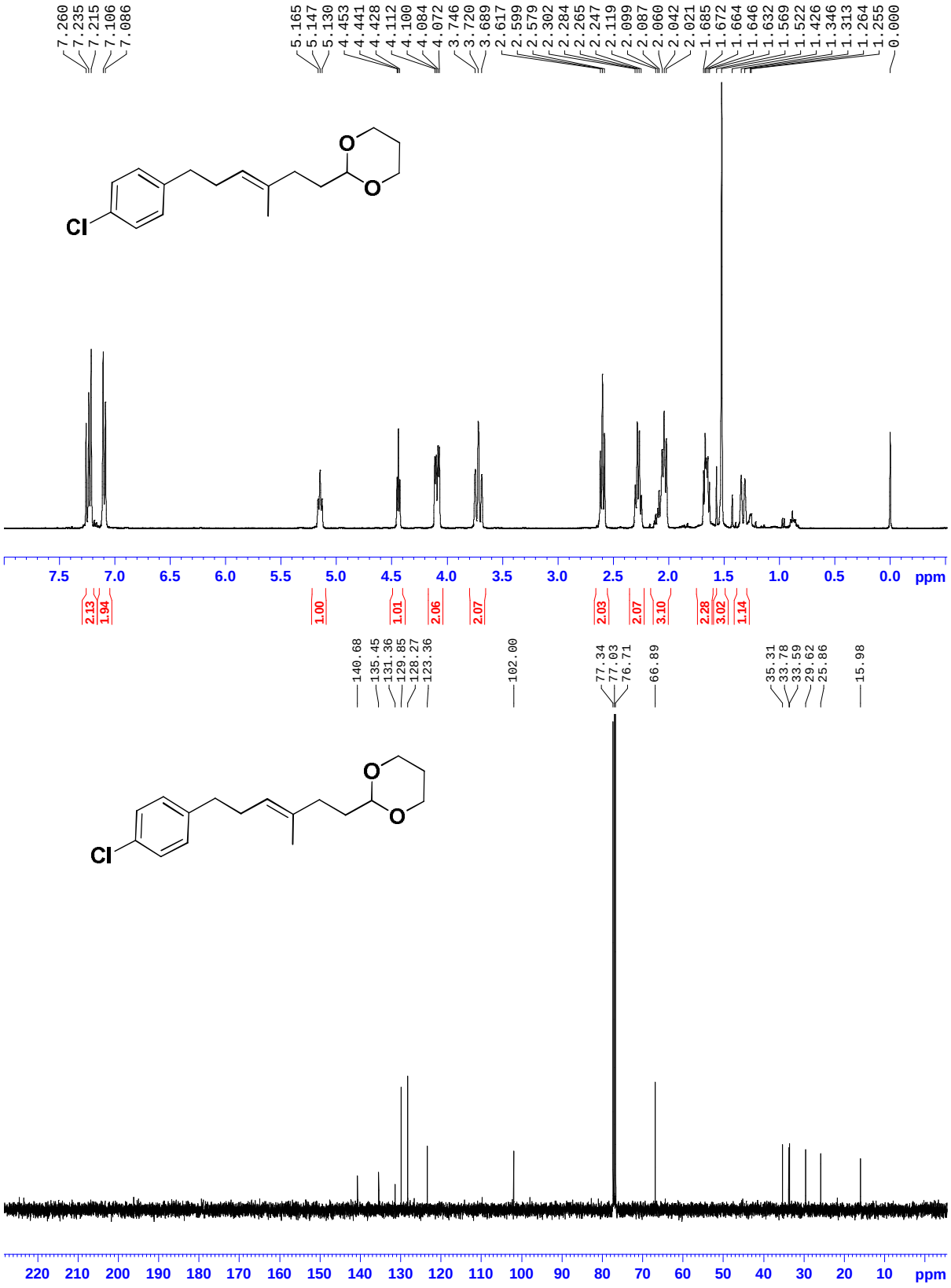
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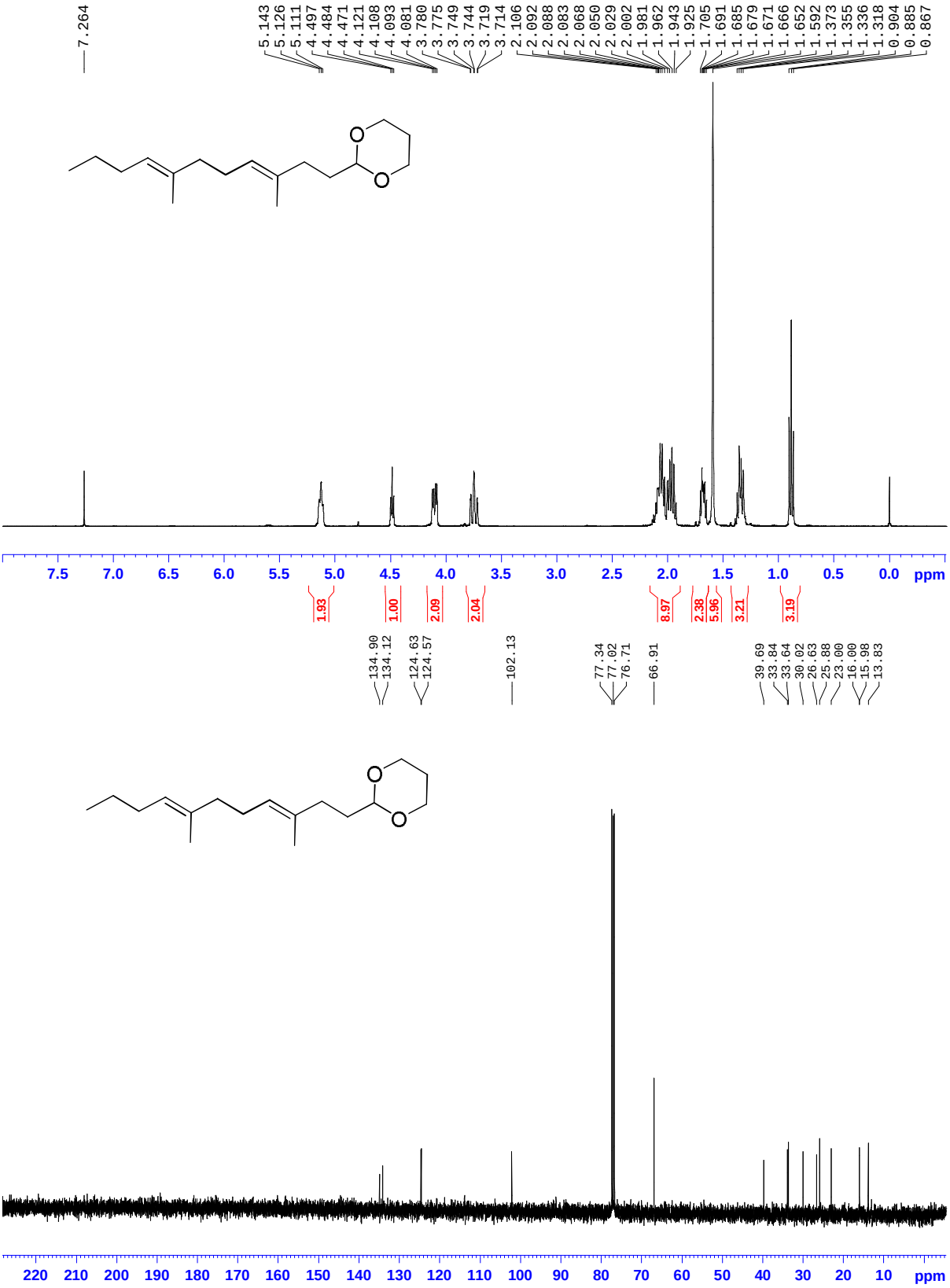
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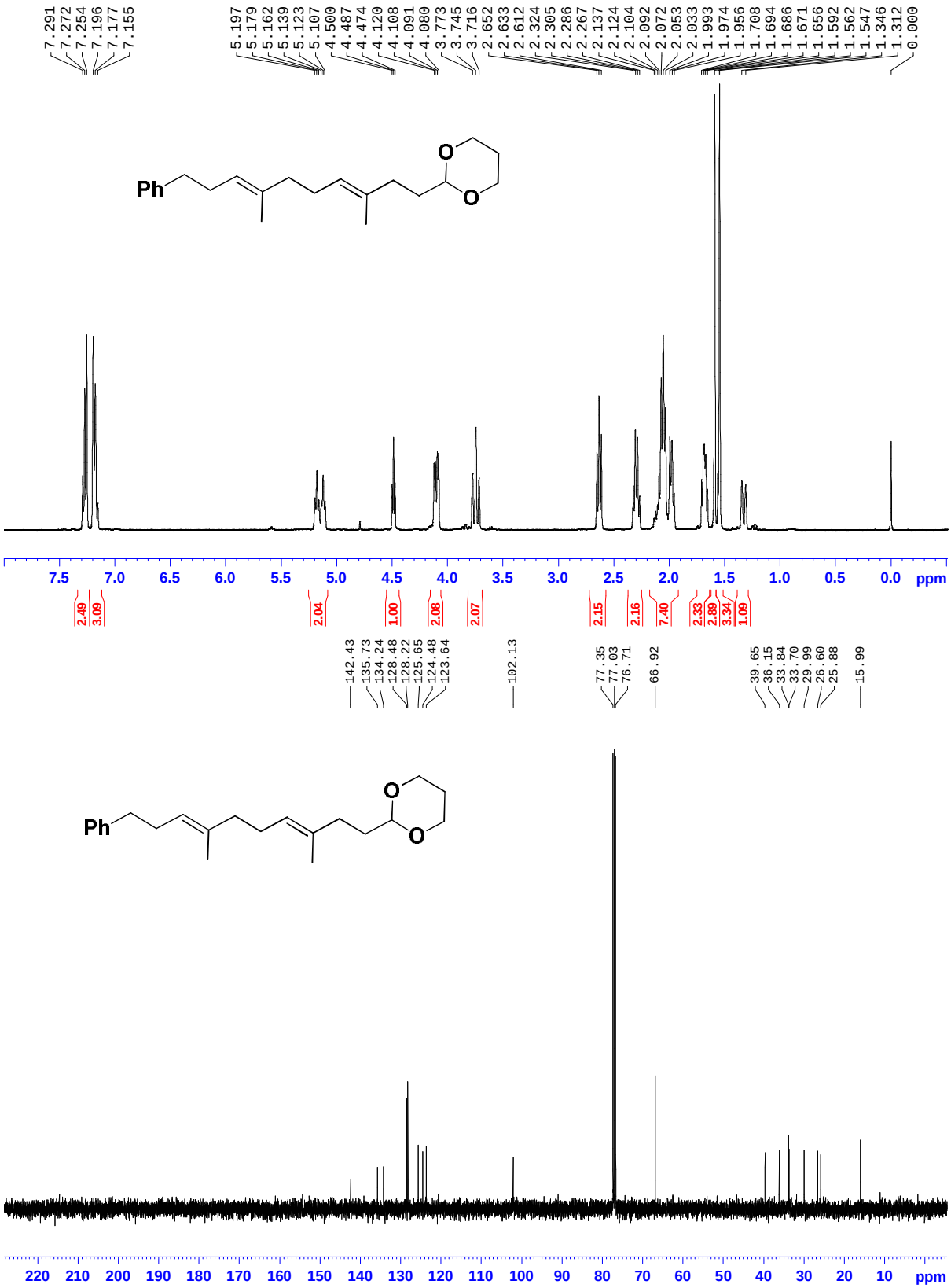
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1.956
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1.671
1.656
1.592
1.562
1.547
1.346
1.312
0.000
Ph
O
O
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6.0
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2.08
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3.34
1.09
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134.24
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102.13
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77.03
76.71
66.92
39.65
36.15
33.84
33.70
29.99
26.60
25.88
15.99
Ph
O
O
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210
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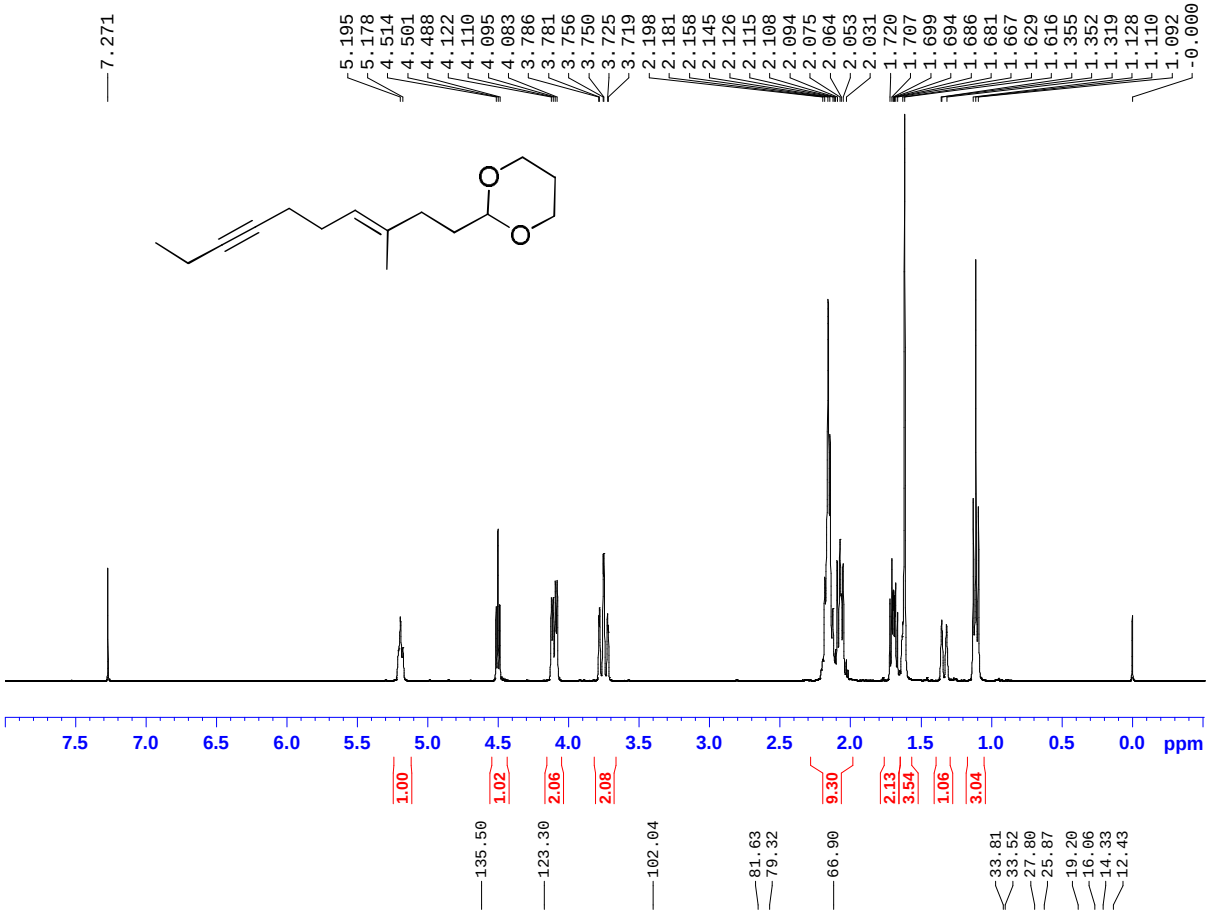
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3.725
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2.158
2.145
2.126
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2.075
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1.667
1.629
1.616
1.355
1.352
1.319
1.128
1.110
1.092
-0.000
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2.08
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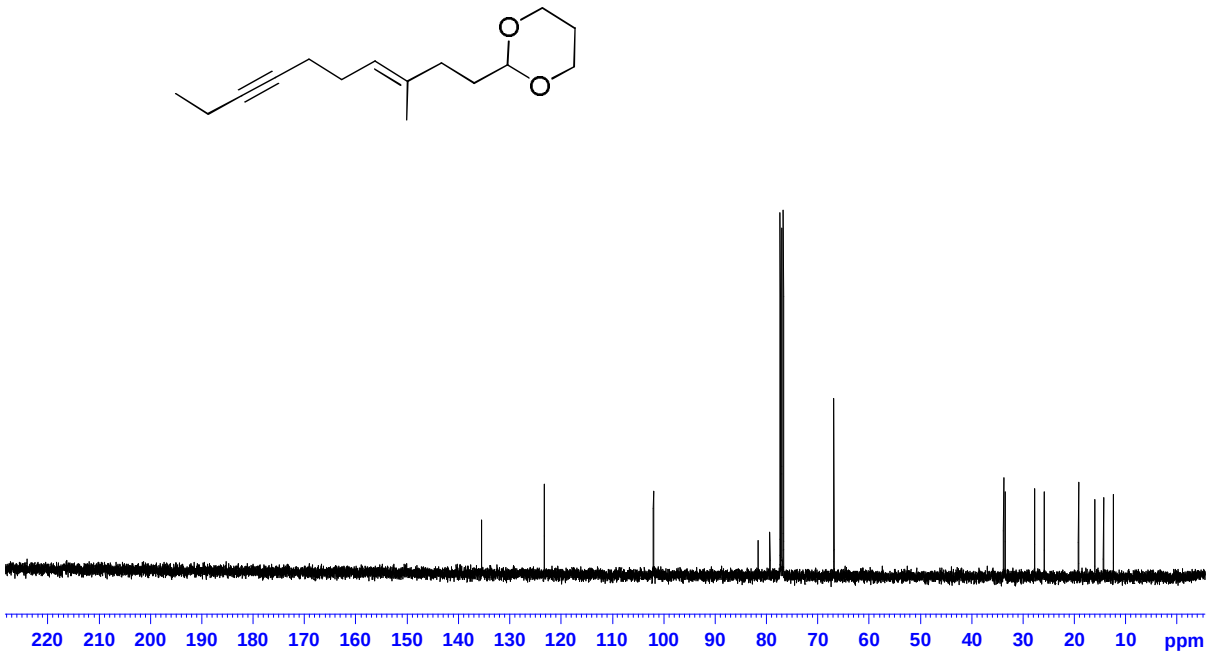
1.06
3.04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14.33
12.43
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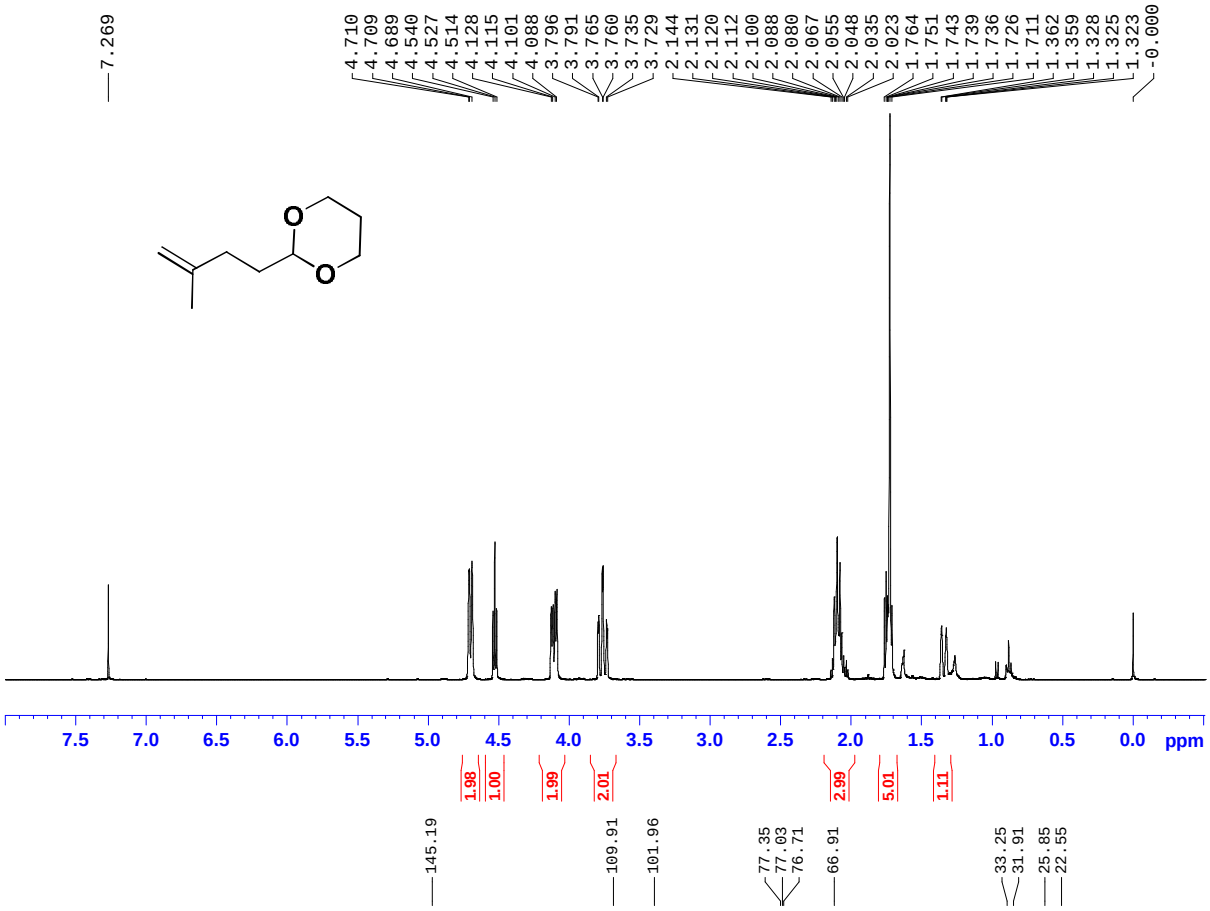
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3.765
3.760
3.735
3.729
2.144
2.131
2.120
2.112
2.100
2.088
2.080
2.067
2.055
2.048
2.035
2.023
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1.751
1.743
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1.736
1.726
1.711
1.362
1.359
1.328
1.325
1.323
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O
O
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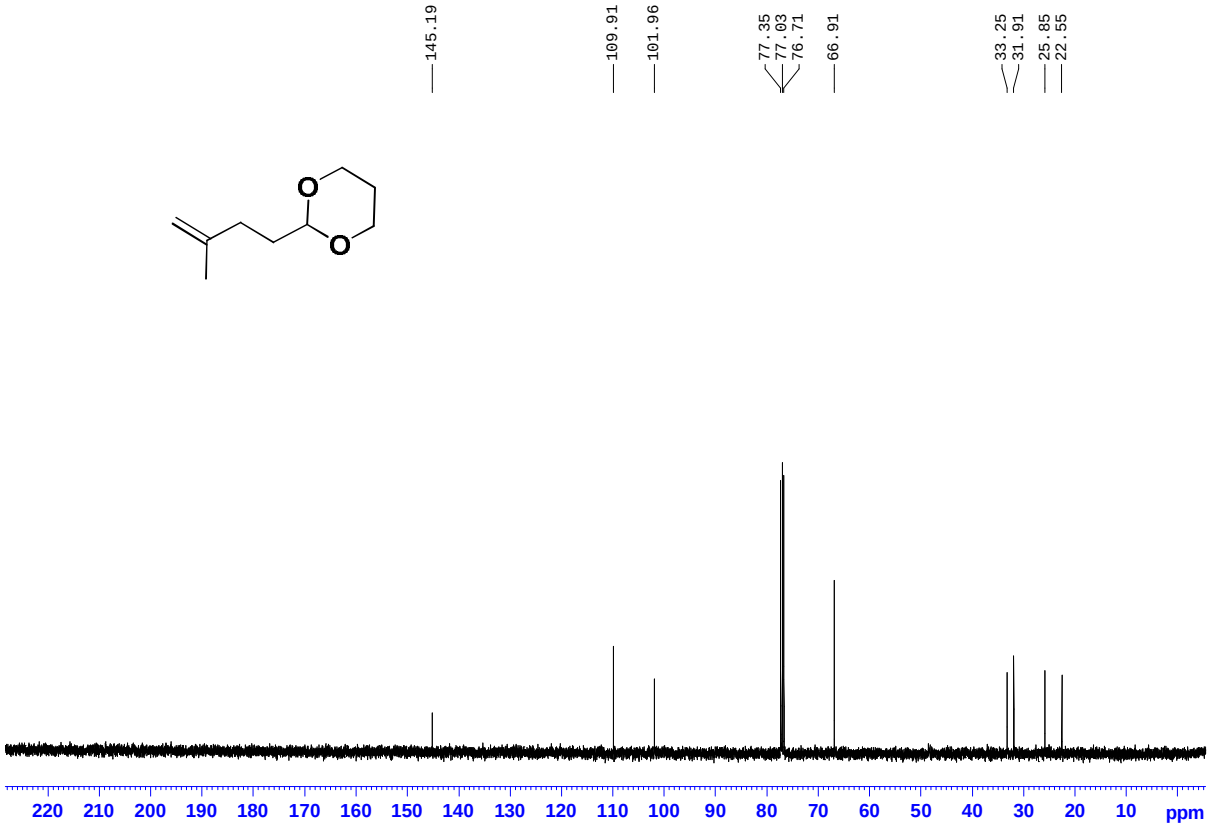
1.11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109.91
101.96
77.35
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31.91
25.85
22.55
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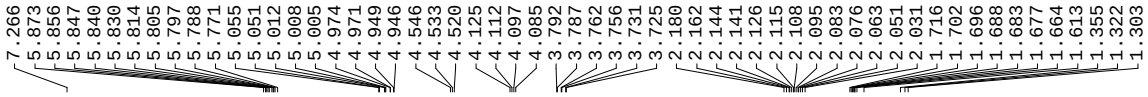
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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5.051
5.012
5.008
5.005
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4.946
4.546
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2.076
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2.051
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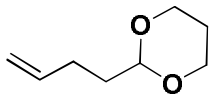
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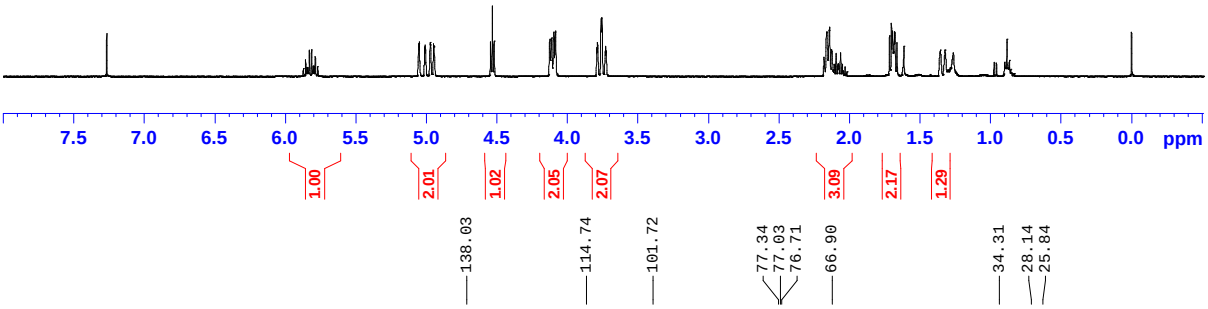
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4.0
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3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
2.01
1.02
2.05
2.07
3.09
2.17
1.29

138.03
114.74
101.72
77.34
77.03
76.71
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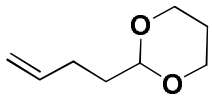
34.31
28.14
25.84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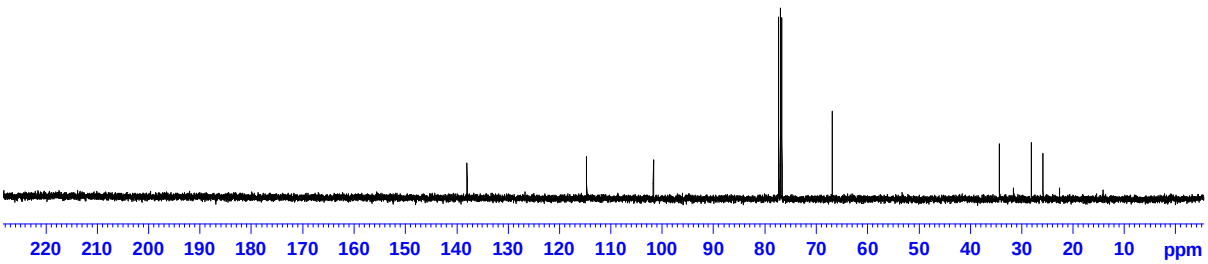
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180
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140
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120
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100
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70
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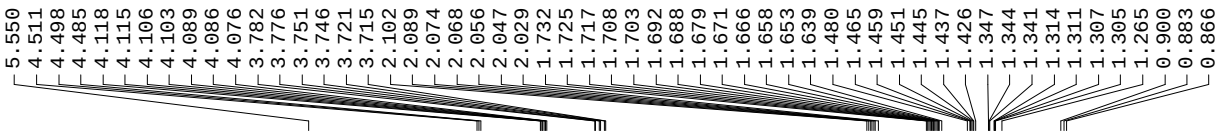
30
20
10
ppm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4.086
4.076
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3.776
3.751
3.746
3.721
3.715
2.102
2.089
2.074
2.068
2.056
2.047
2.029
1.732
1.725
1.717
1.708
1.703
1.692
1.688
1.679
1.671
1.666
1.658
1.653
1.639
1.480
1.465
1.459
1.451
1.445
1.437
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1.307
1.305
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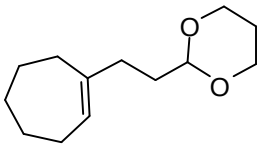
0.883
0.866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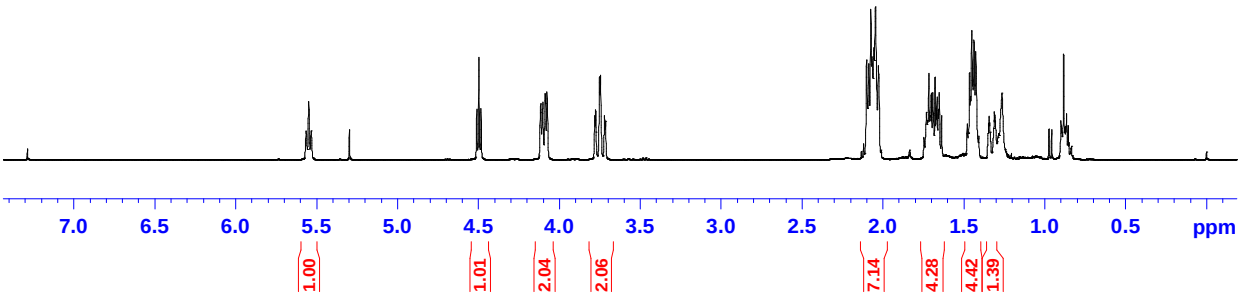
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4.0
3.5
3.0
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0.5
ppm
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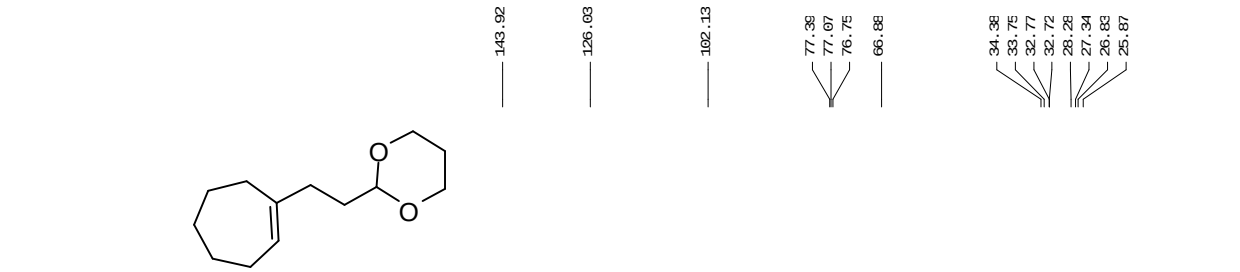
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102.13
77.38
77.07
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34.38
33.75
32.77
32.72
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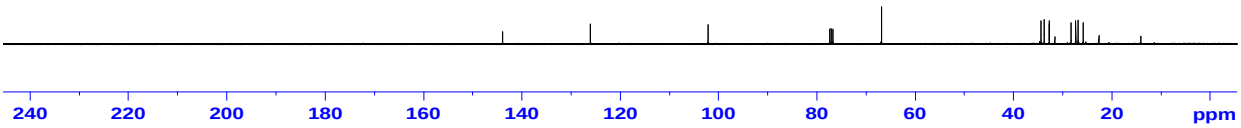
27.34
26.83
25.87

O
O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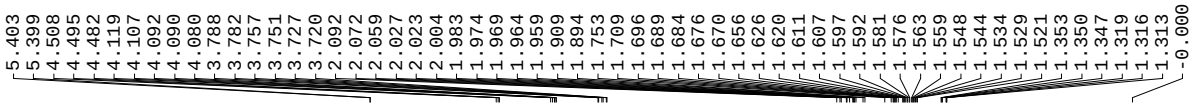
220
200
180
160
140
120
100
80
60
40
20
ppm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4.107
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4.090
4.080
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3.782
3.757
3.751
3.727
3.720
2.092
2.072
2.059
2.027
2.023
2.004
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1.969
1.964
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1.909
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1.753
1.709
1.696
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1.670
1.656
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1.620
1.611
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1.597
1.592
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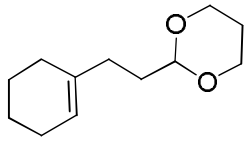
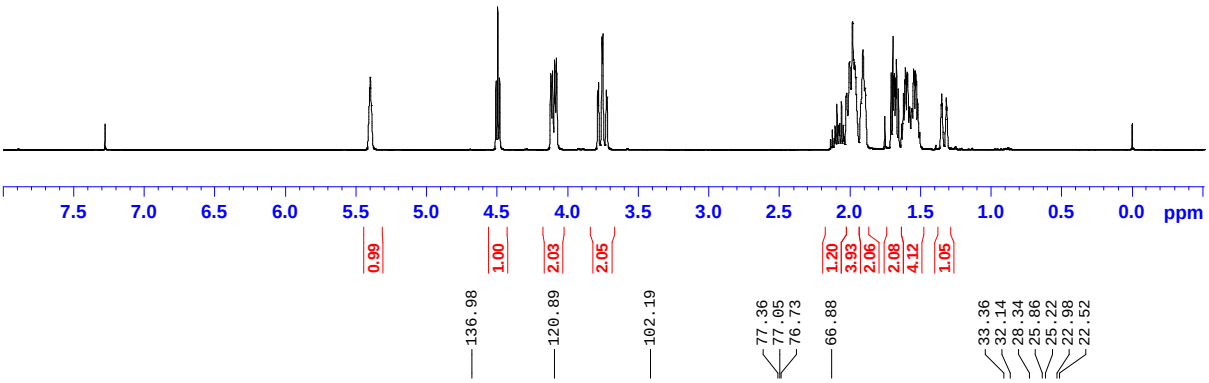
1.313
-0.000

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1.0
0.5
0.0
ppm
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2.05
1.20
3.93
2.06
2.08
4.12
1.05
136.98
120.89
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76.73
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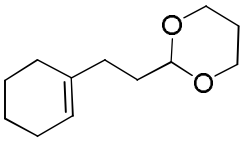
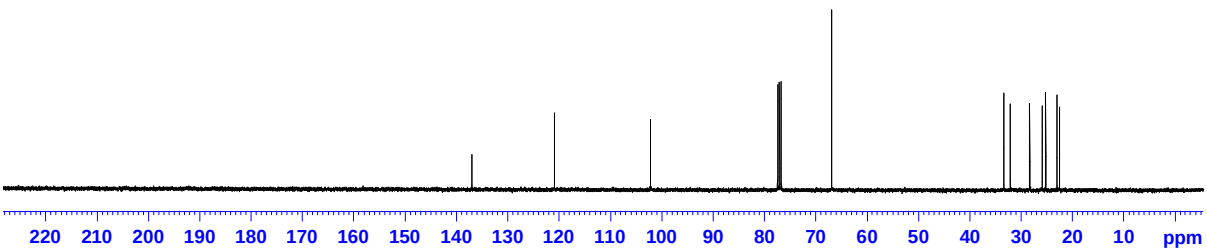
22.52

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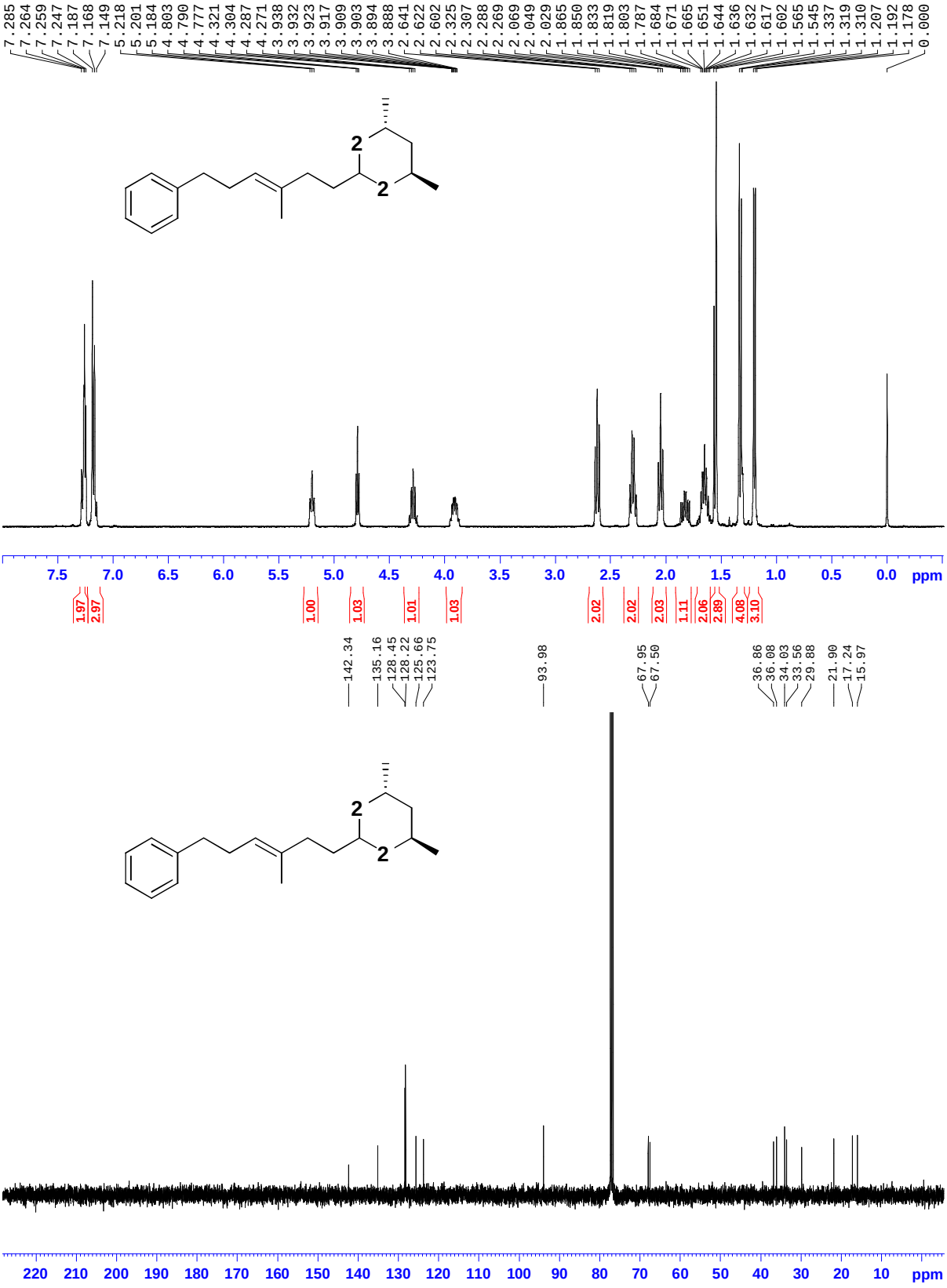
20
10
ppm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4.321
4.304
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3.932
3.923
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2.307
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1.636
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1.602
1.565
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1.337
1.319
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ppm
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1.01
1.03
2.02
2.02
2.03
1.11
2.06
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3.10
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125.66
123.75
93.98
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67.50
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2
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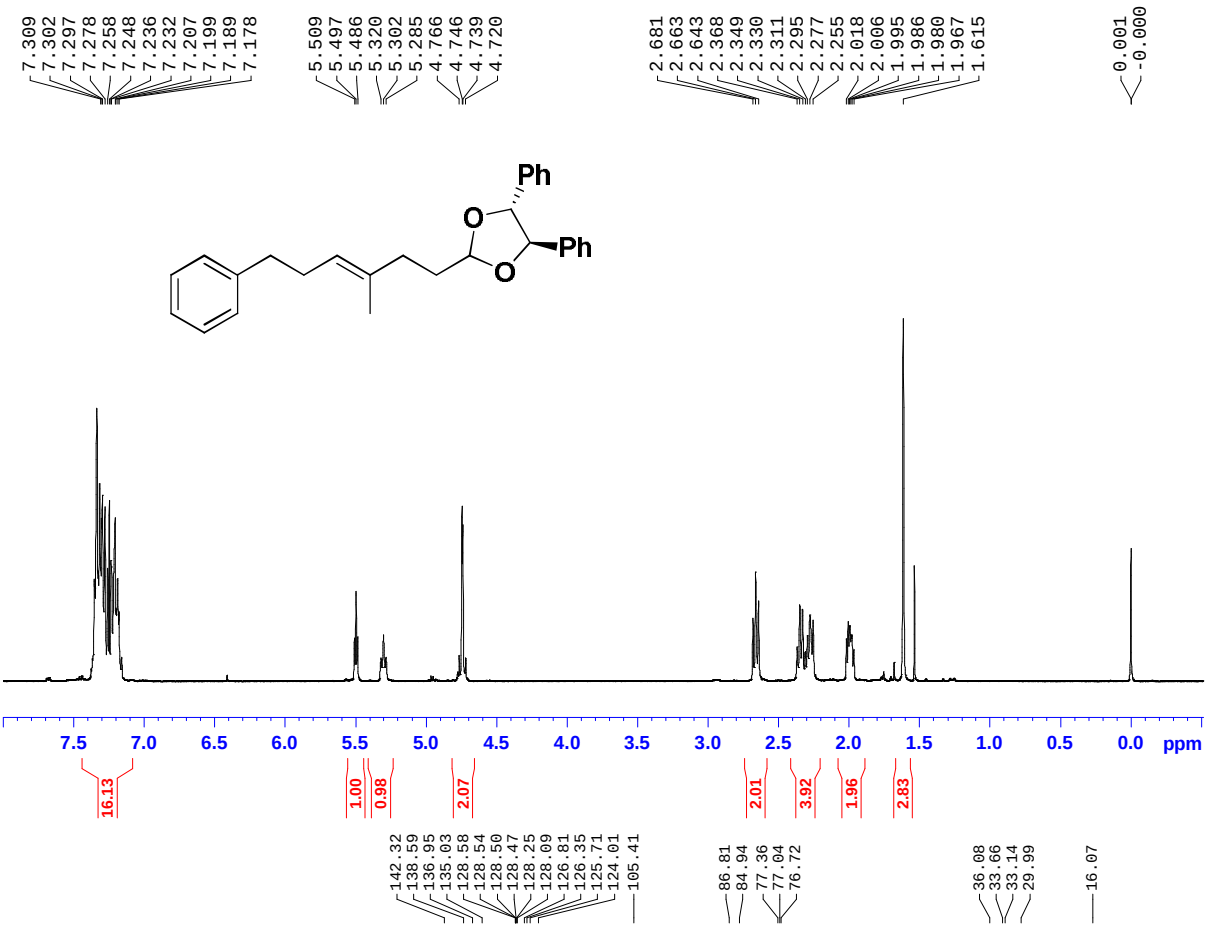
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5.486
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5.302
5.285
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4.739
4.720
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2.643
2.368
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2.330
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2.295
2.277
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2.006
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1.986
1.980
1.967
1.615
0.001
-0.000
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O
Ph
O
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6.0
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5.0
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4.0
3.5
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2.0
1.5
1.0
0.5
0.0
ppm
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3.92
1.96
2.83

142.32
138.59
136.95
135.03
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128.47
128.25
128.09
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126.35
125.71
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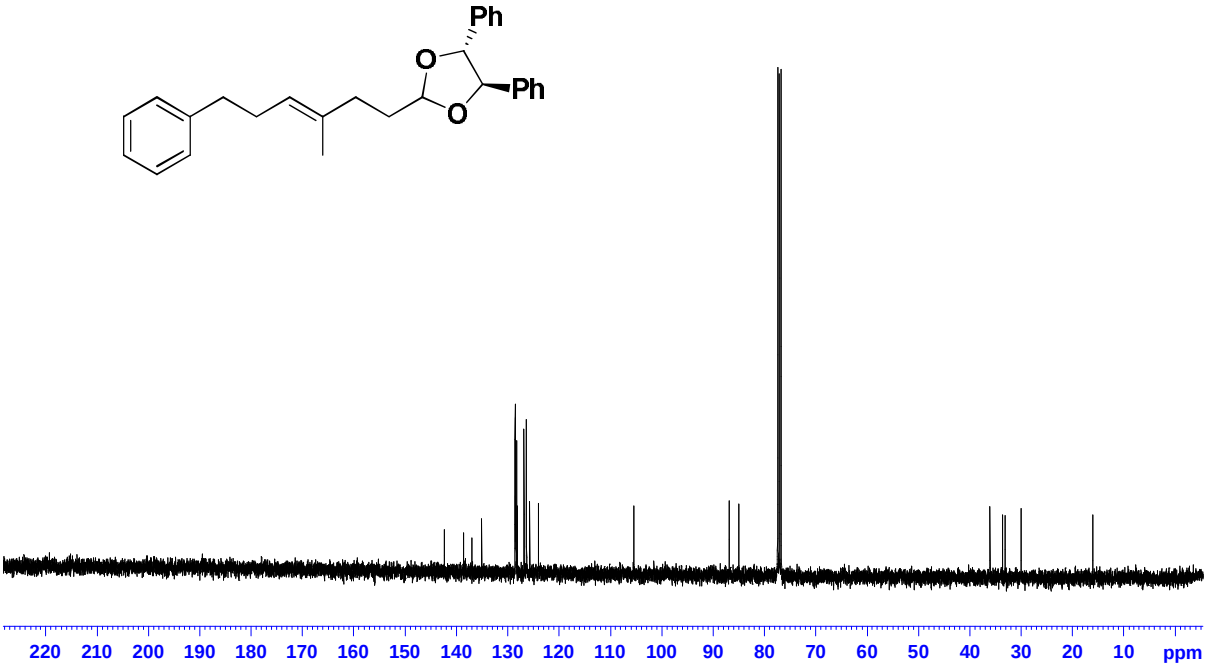
33.14
29.99
16.07


Ph
O
Ph
O
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200
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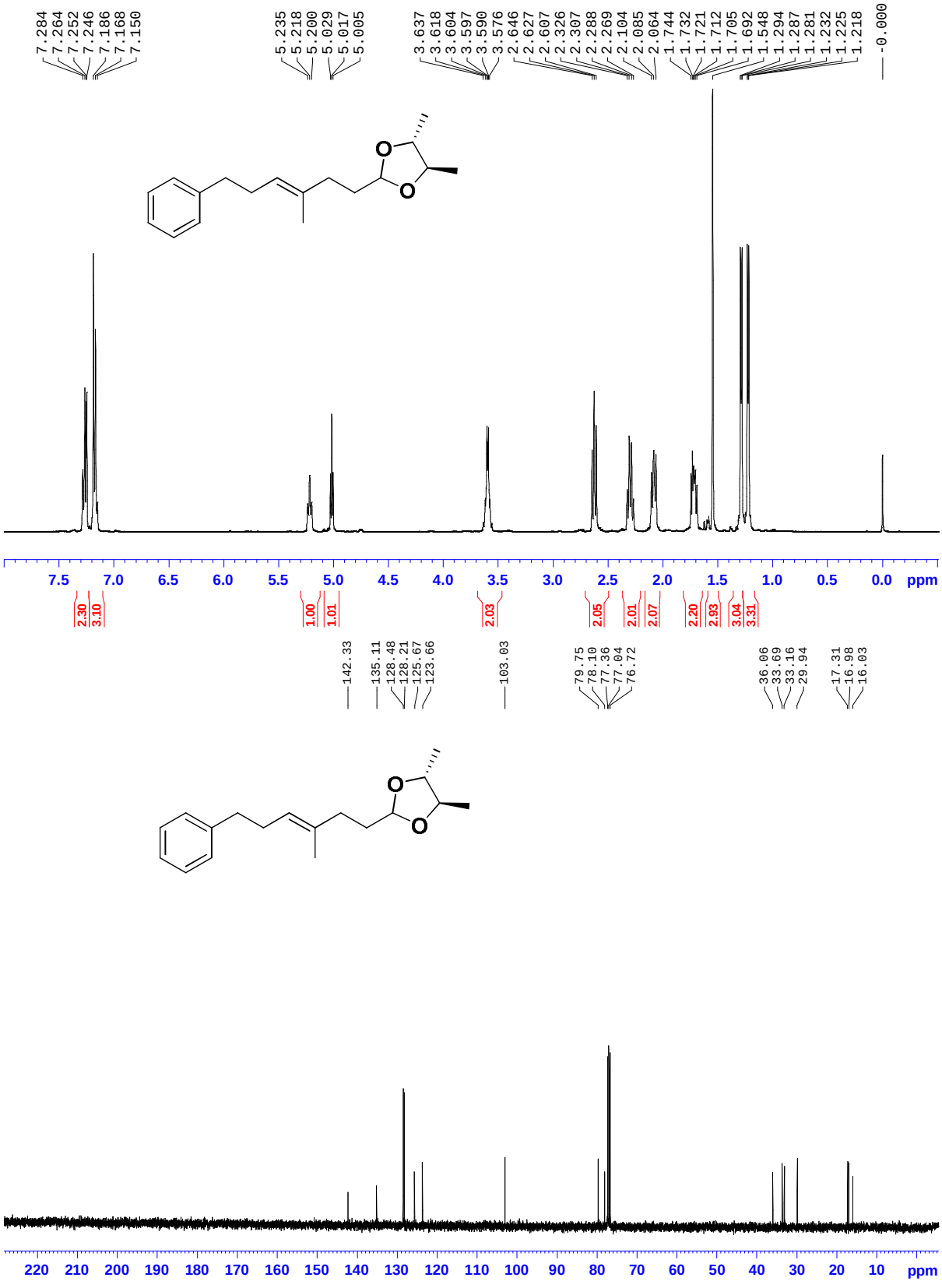
10
ppm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3.618
3.604
3.597
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3.576
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1.705
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1.232
1.225
1.218
-0.000
O
O
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6.0
5.5
5.0
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2.0
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ppm
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1.01
2.03
2.05
2.01
2.07
2.20
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3.04
3.31
142.33
135.11
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128.21
125.67
123.66
103.03
79.75
78.10
77.36
77.04
76.72
36.06
33.69
33.16
29.94
17.31
16.98
16.03
O
O
220
210
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140
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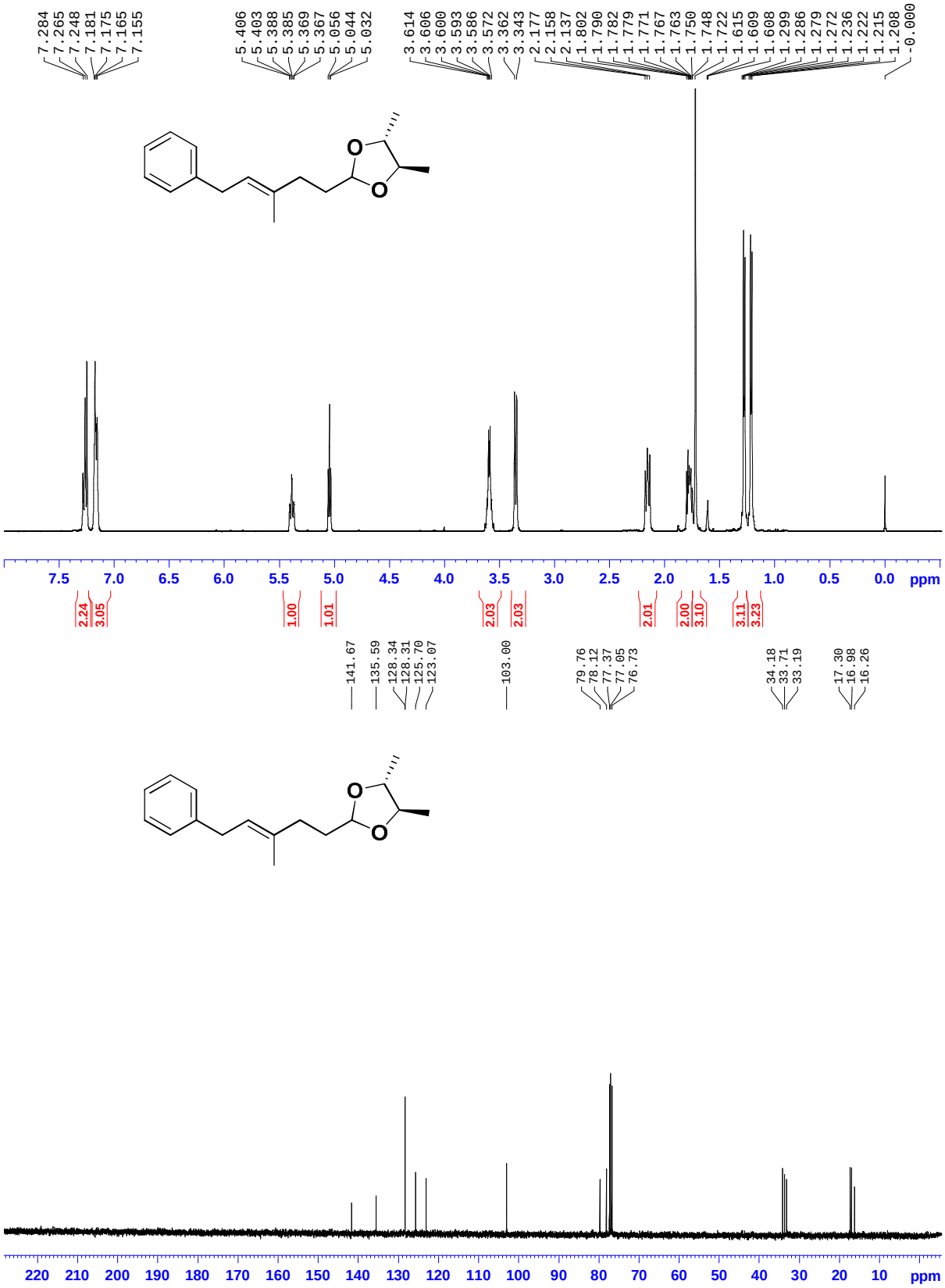
10
ppm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5.032
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O
O
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4.0
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2.0
1.5
1.0
0.5
0.0
ppm
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1.01
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2.03
2.01
2.00
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3.11
3.23
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77.37
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O
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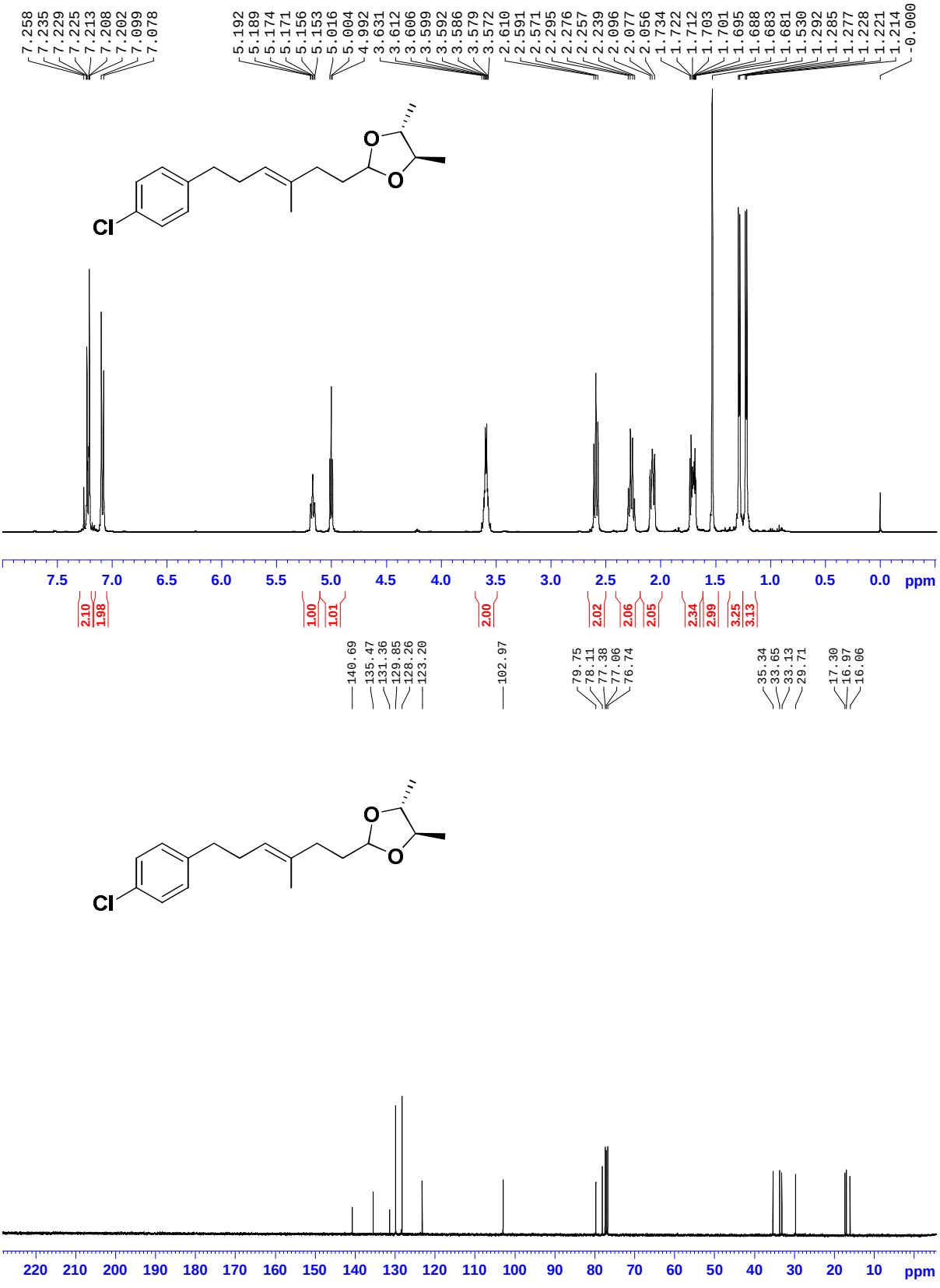
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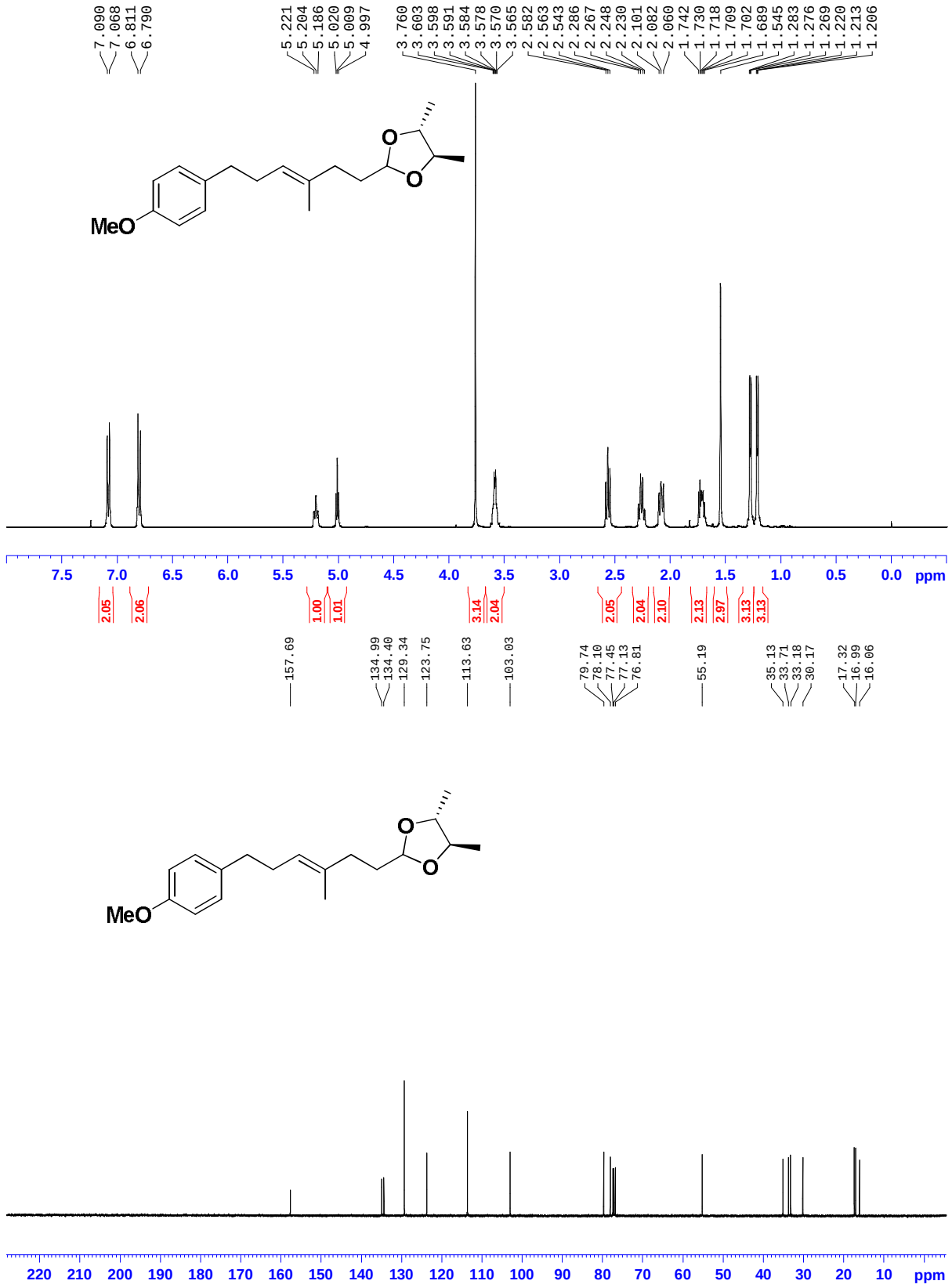
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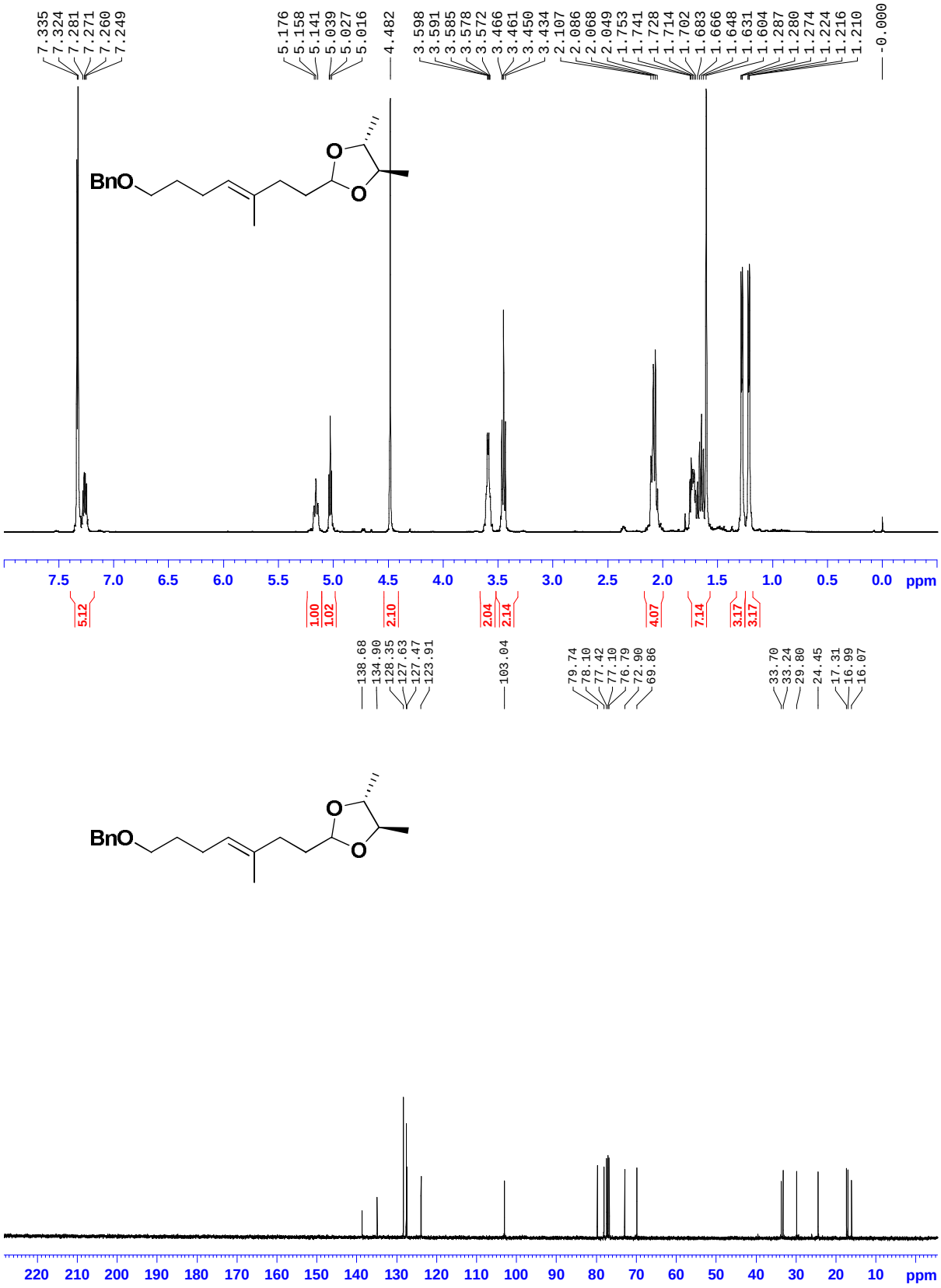
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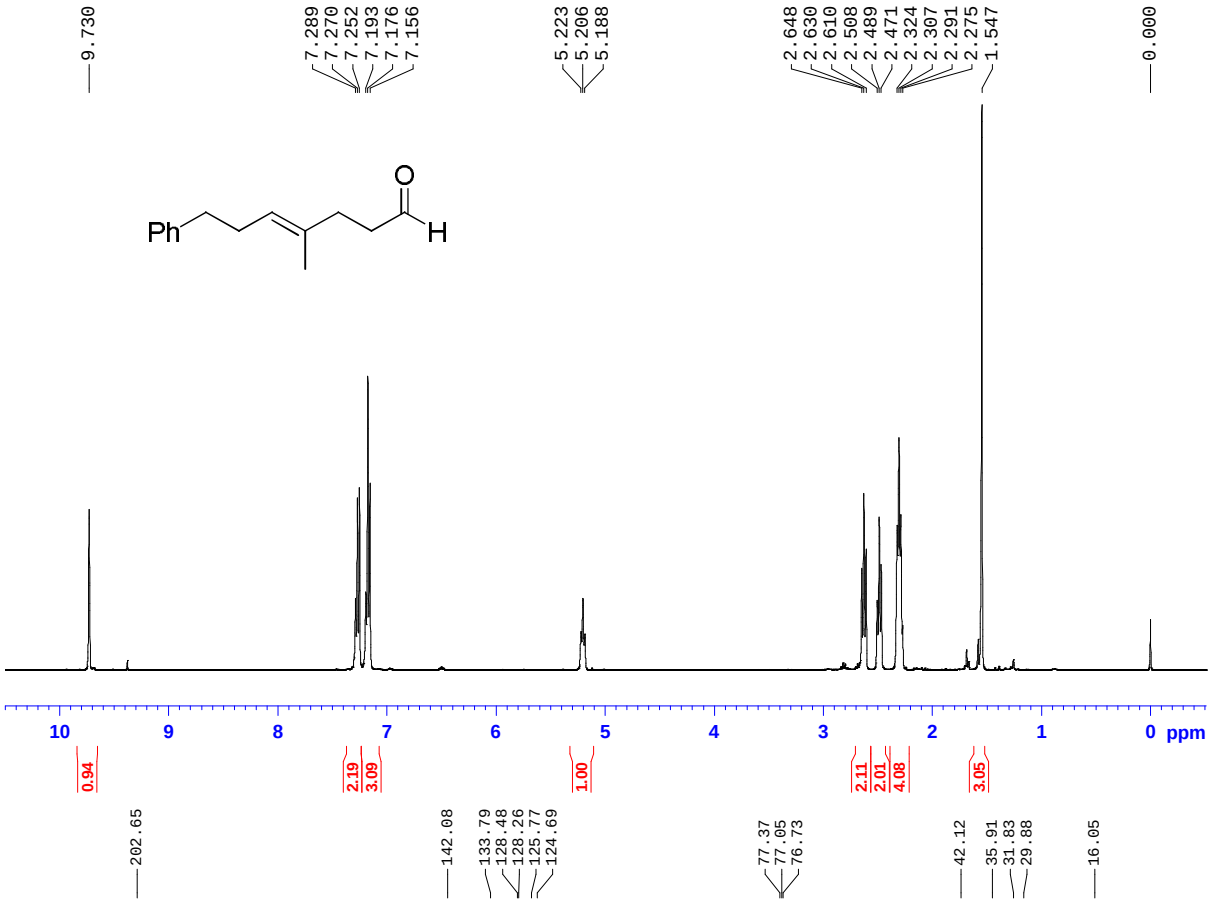
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2.610
2.508
2.489
2.471
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2.307
2.291
2.275
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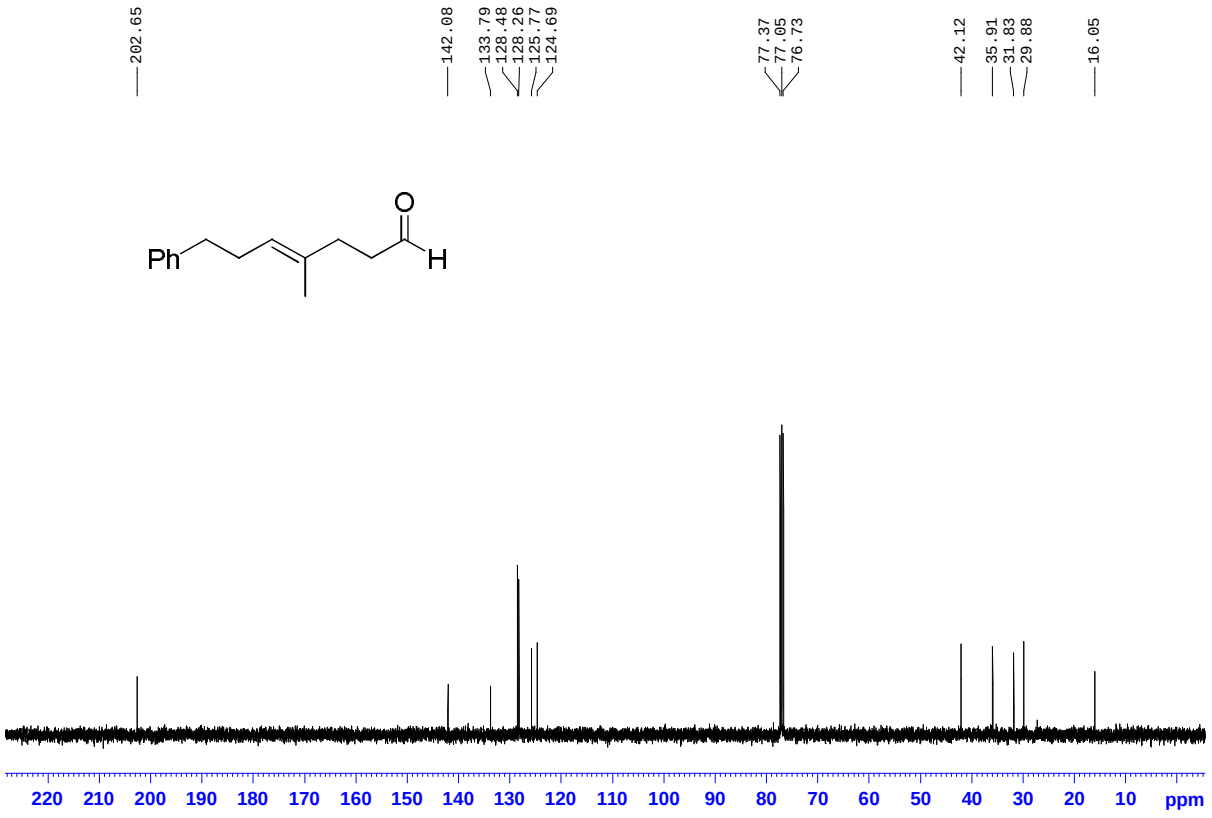
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29.88
16.05
O
Ph
H
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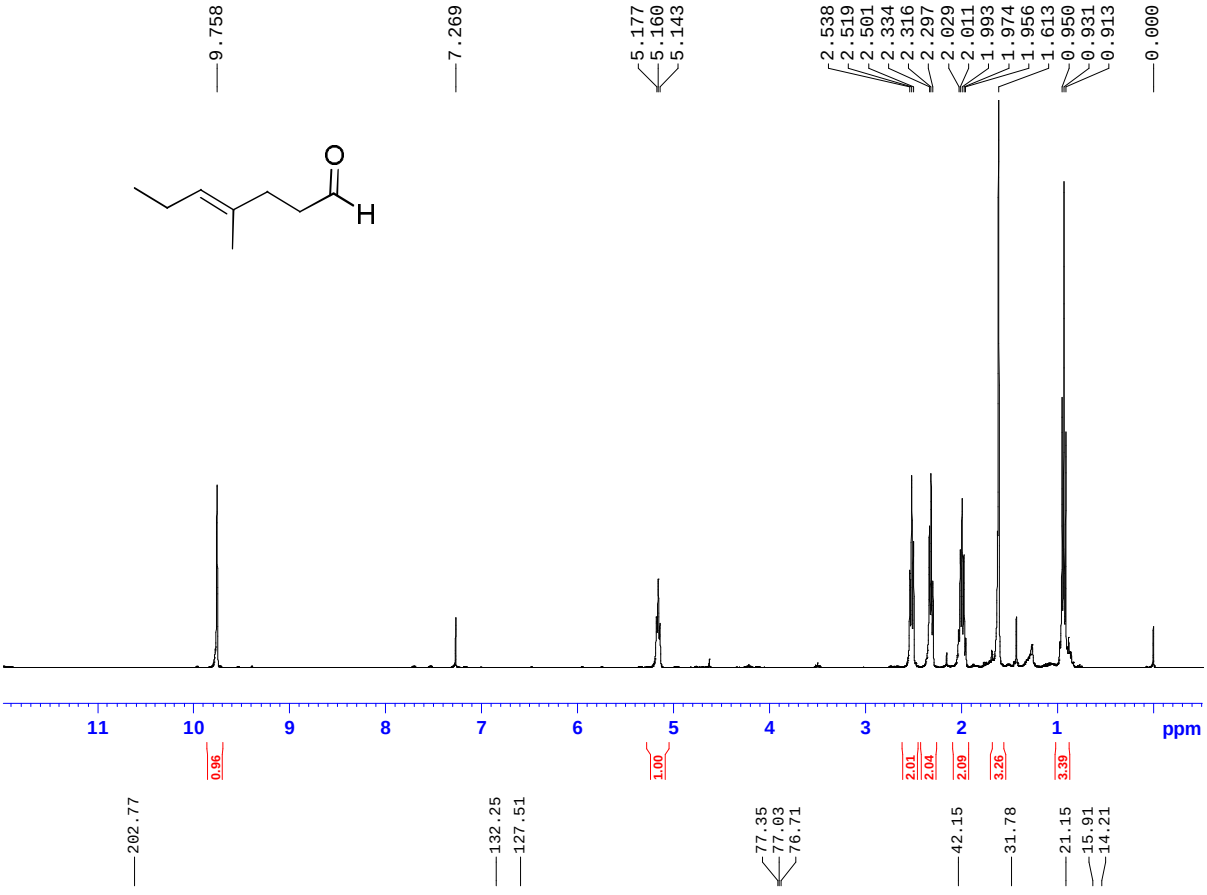
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2.297
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1.956
1.613
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0.931
0.913
0.000
O
H
11
10
9
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3
2
1
ppm
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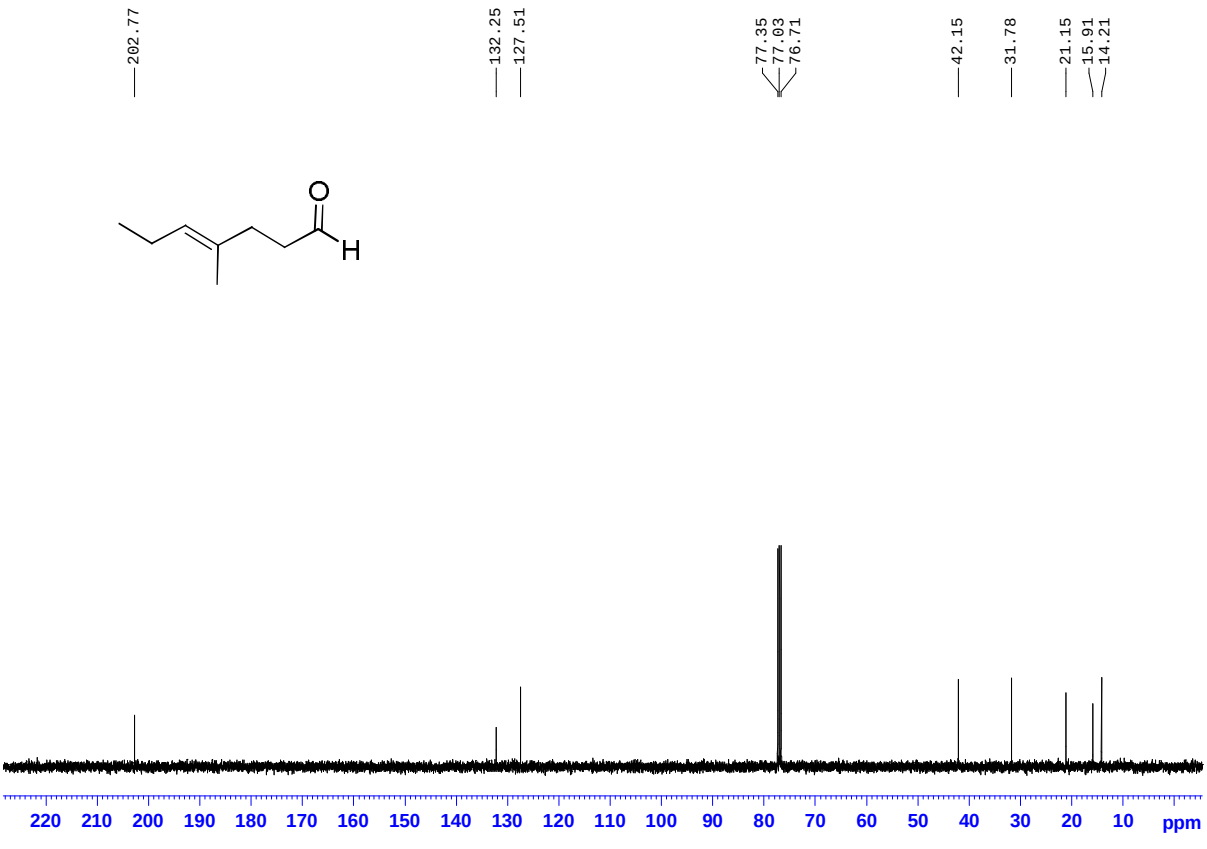
2.09
3.26
3.39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76.71
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31.78
21.15
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210
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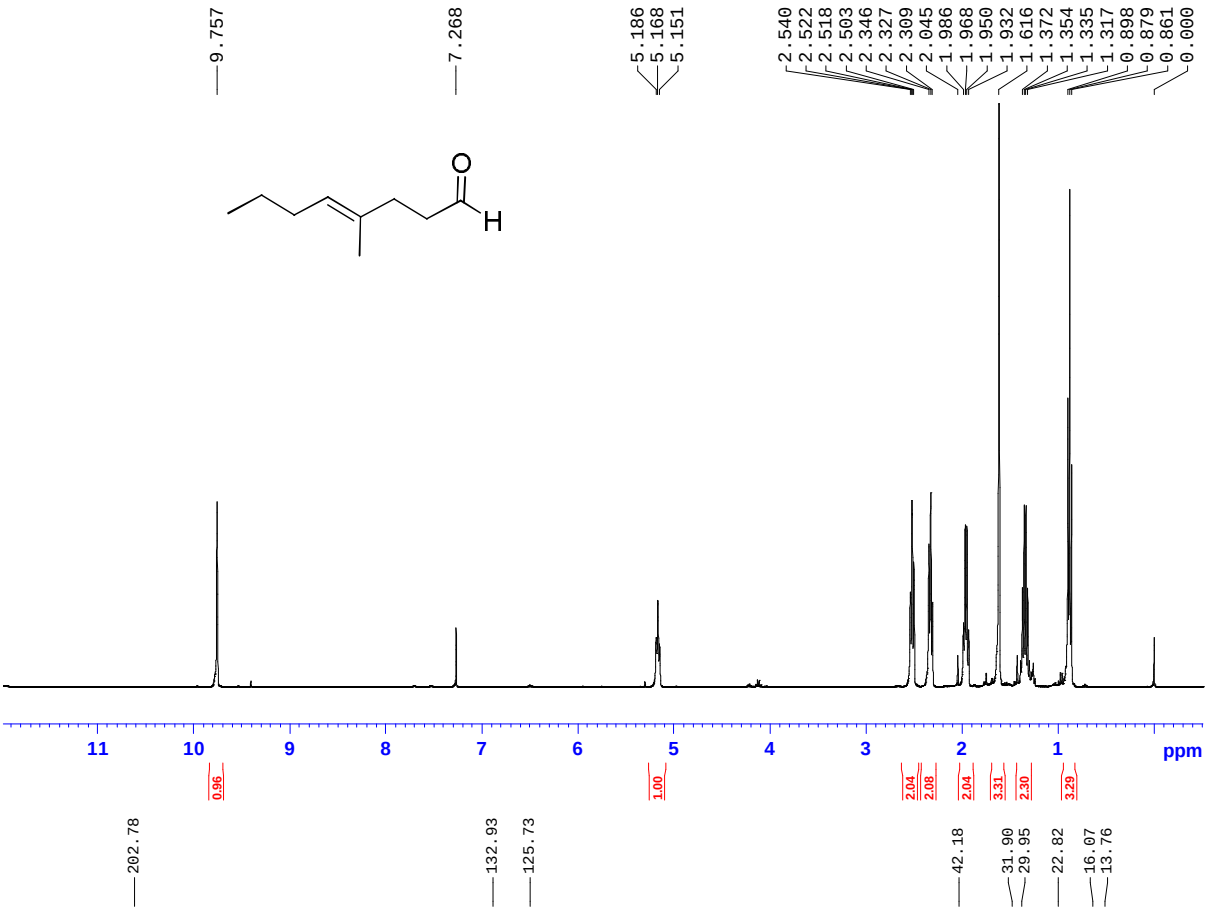
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1.932
1.616
1.372
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1.317
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0.879
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H
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9
8
7
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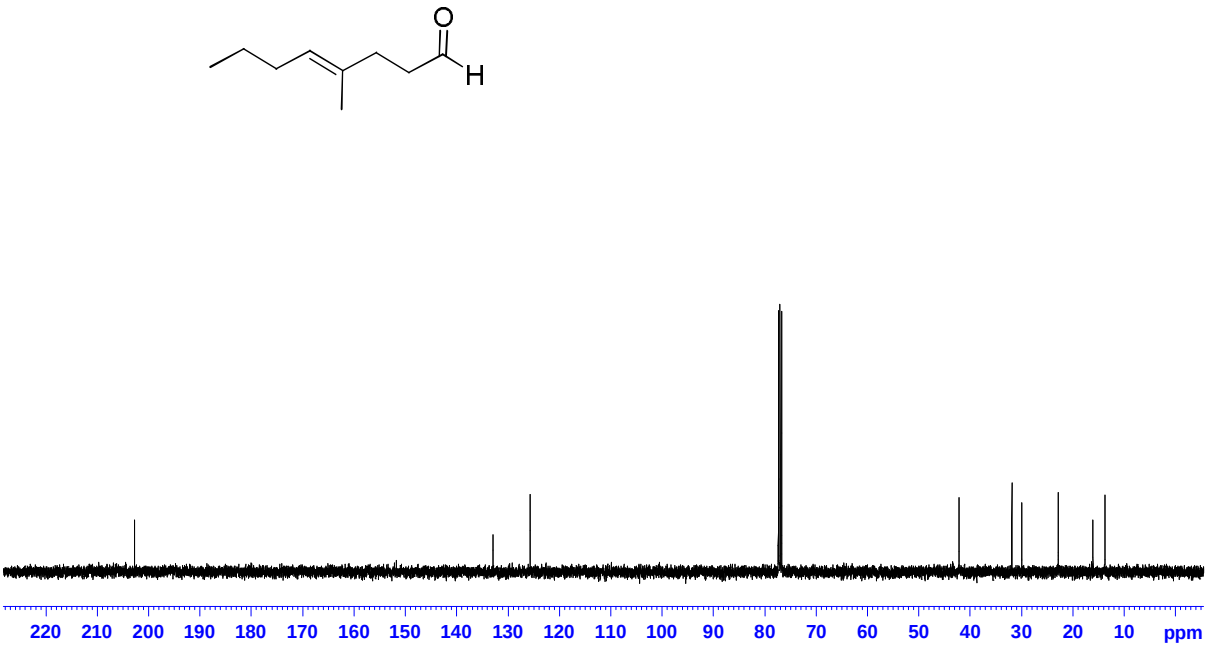
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16.07
13.76
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H
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140
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30
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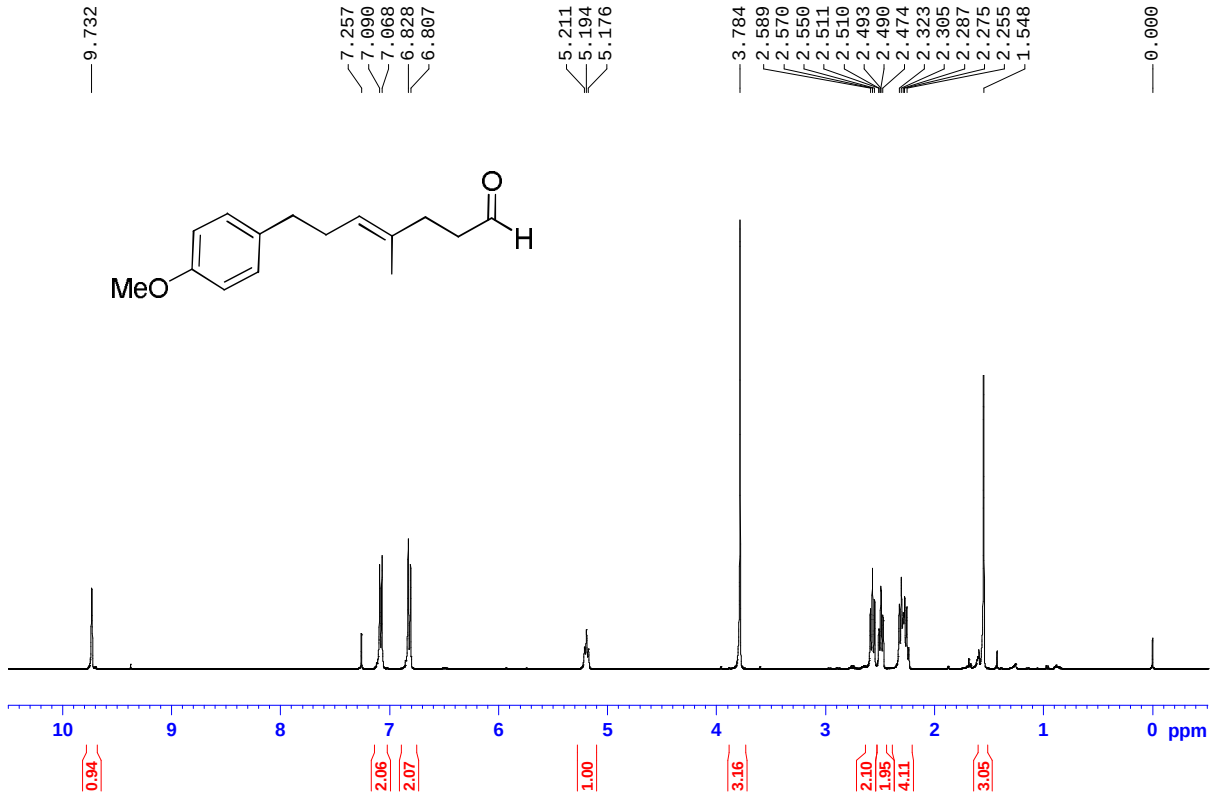
10
ppm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2.255
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0.000
O
H
MeO
10
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8
7
6
5
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3.16
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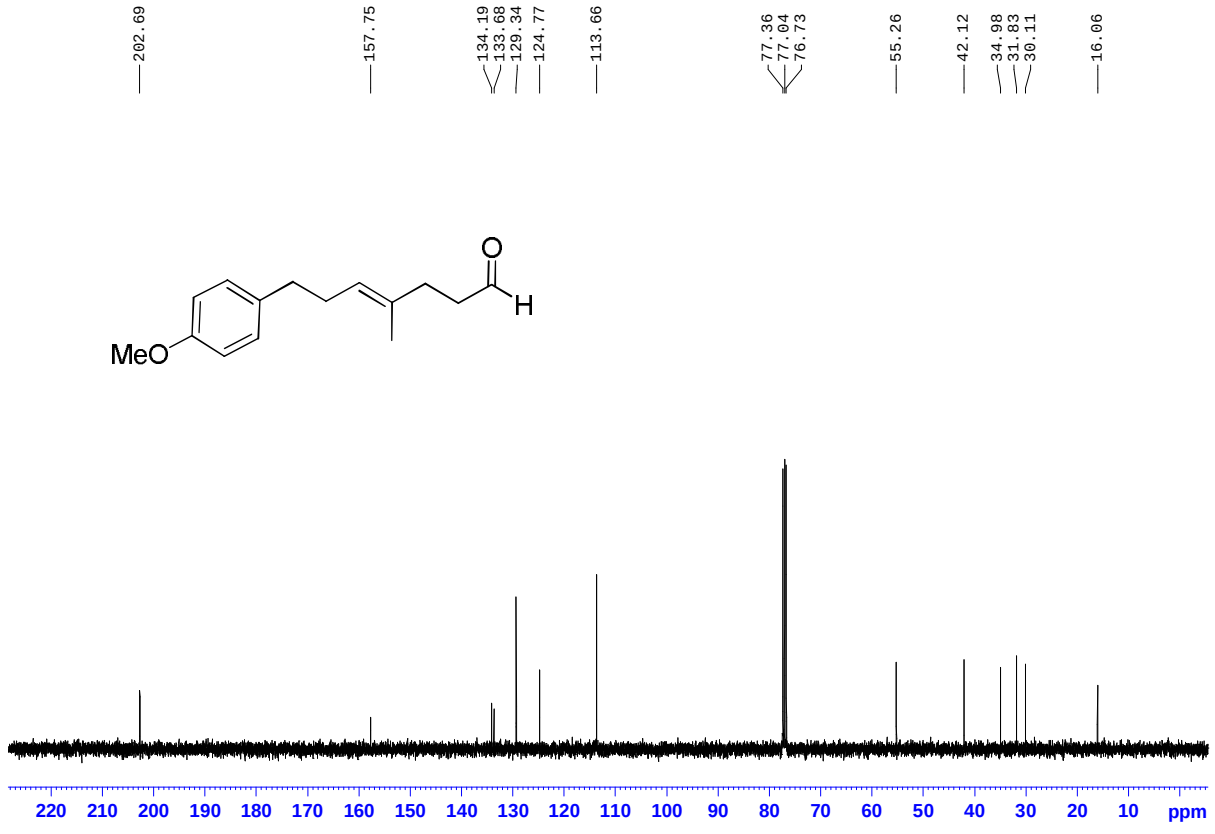
3.05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42.12
34.98
31.83
30.11
16.06
O
H
MeO
220
210
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70
60
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30
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10
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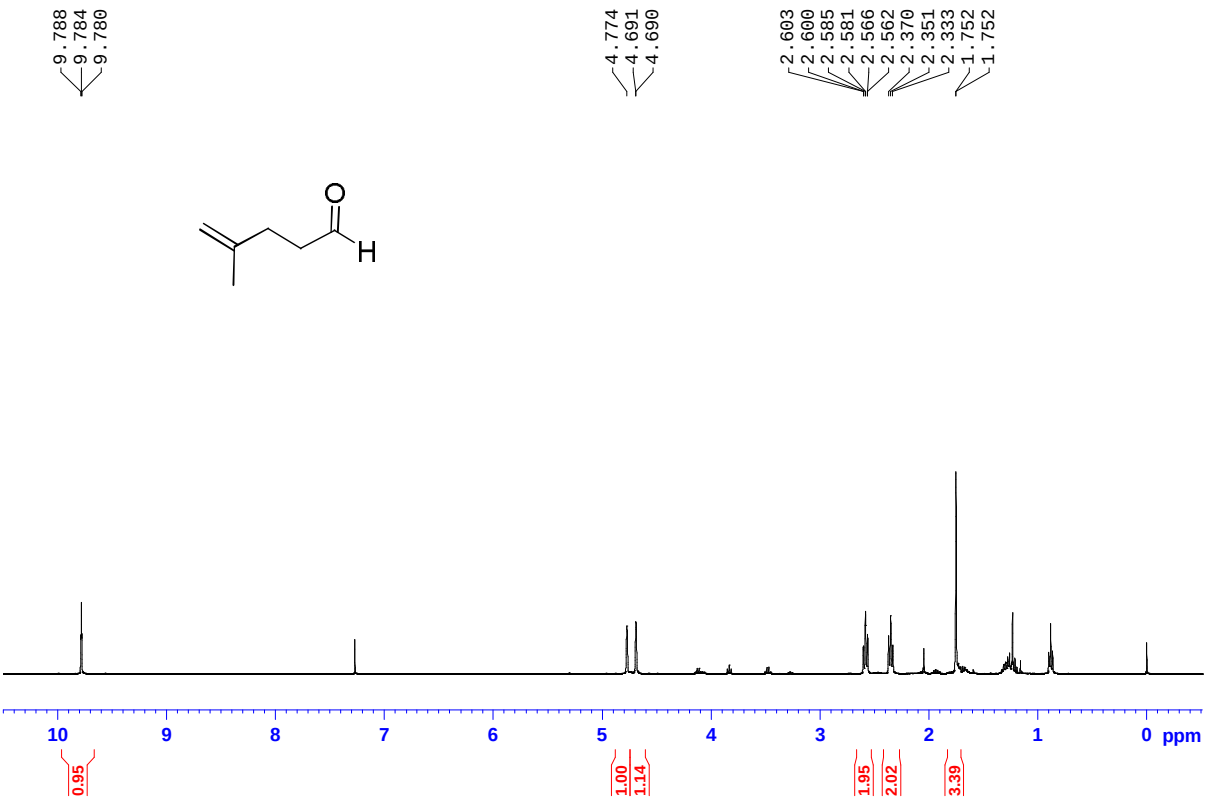
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1.752
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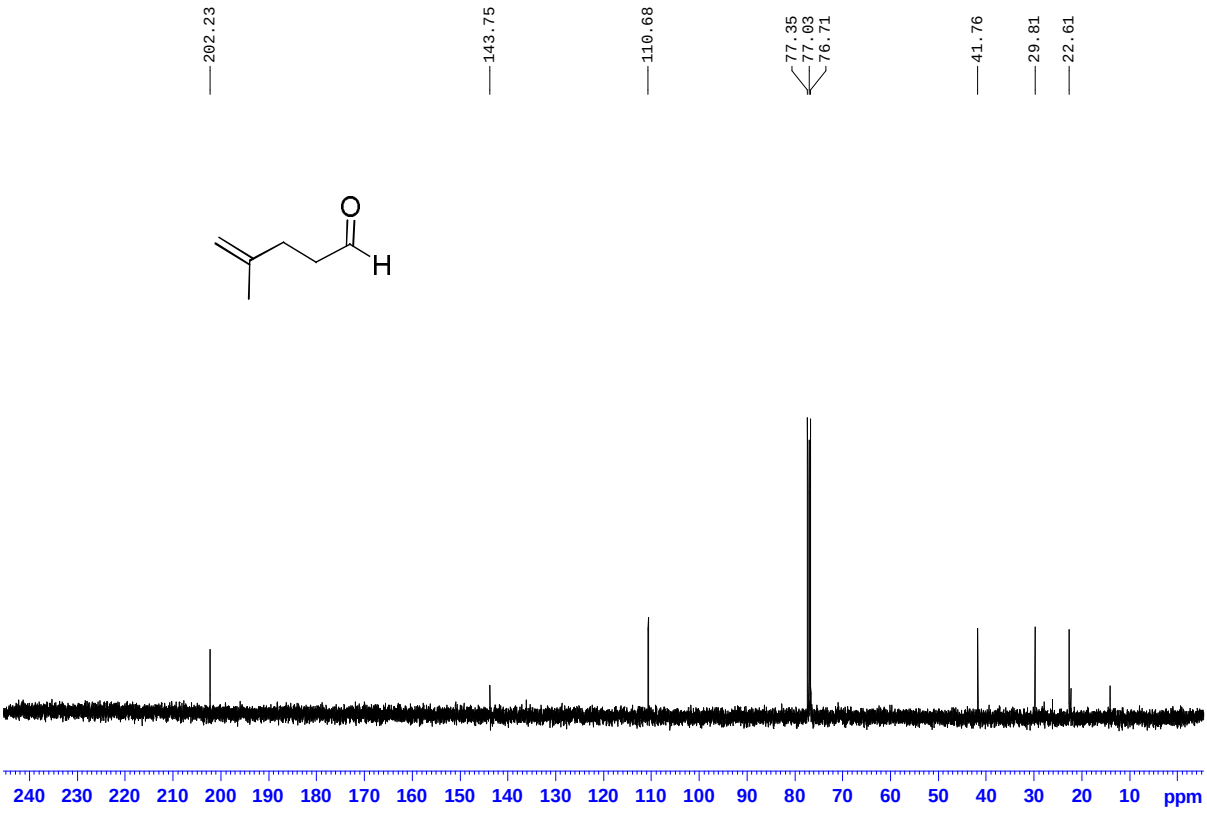
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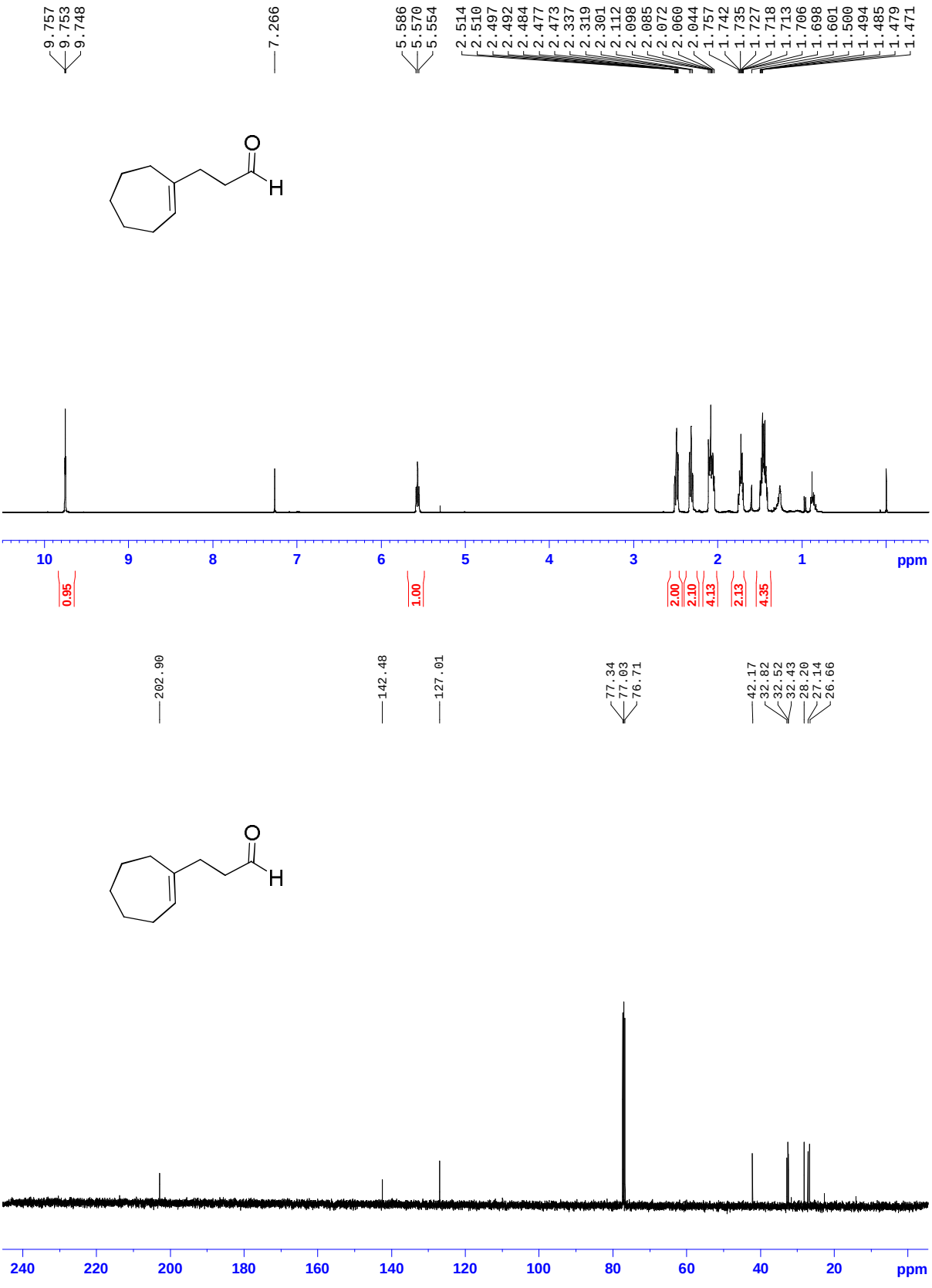
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2.060
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1.713
1.706
1.698
1.601
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1.485
1.479
1.471
O
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7
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4
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ppm
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2.00
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2.13
4.35
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77.34
77.03
76.71
42.17
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32.43
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27.14
26.66
O
H
240
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200
180
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140
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100
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60
40
20
ppm