Electronic Supplementary Material (ESI) for Chemical Communications

- **Supporting Information** -

Z-Selective Ring Opening of Vinyl Oxetanes with Dialkyl Dithiophosphate Nucleophiles

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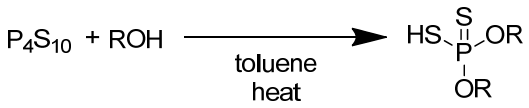
General Information:

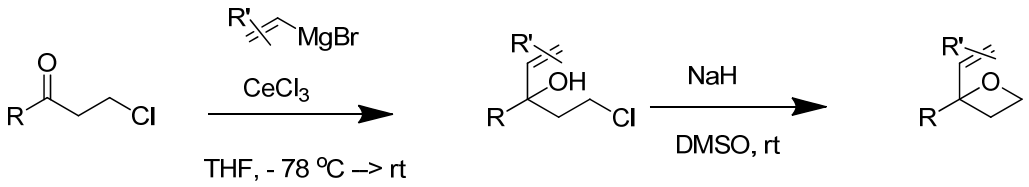
All reactions were carried out under a nitrogen atmosphere with flame-dried glassware. Commercially available reagents were used as received without further purification unless specified. Toluene, benzene, diethyl ether, dichloromethane, and tetrahydrofuran were dried through activated alumina columns. Flash chromatography was done with Silicycle SiliaFlash® F60 silica, and thin layer chromatography (TLC) was performed with EMD 250μm silica gel 60-F254 plates. ^{1}H NMR data were recorded on Bruker (400, 500 or 600 MHz) spectrometers with tetramethylsilane (TMS) (0 ppm) as internal reference. Signals are reported as follows: s (singlet), d (doublet), t (triplet), dd (doublet of doublets), dt (doublet of triplets), m (multiplet). Coupling constants are reported in hertz (Hz). ^{13}C NMR spectra were recorded on spectrometers operating at 100 or 125 MHz with chloroform ($CDCl_3$) (77.0 ppm) as internal reference. Infrared spectra were obtained on an FT-IR spectrometer. High-resolution mass (HRMS) spectra were performed at the University of Arizona Mass Spectrometry Facility.

General Procedure A: Preparation of *O,O*-Dialkyl Dithiophosphates



The known *O,O*-dialkyl dithiophosphoric acids were prepared according to the reported procedures with minor modifications[1]. To a flame dried three-necked 250 mL rbf charged with condenser, 15 mL addition funnel and magnetic stir bar was introduced dry toluene (100 mL) and P_4S_{10} (13.36 g, 30 mmol). The above mixture was heated to 40 °C with stirring. At the same temperature, dry methanol (12.2 mL, 300 mmol) was added through addition funnel during a course of 30 minutes. The mixture was heated at 65 °C for 10 hours and then reflux for 1 hour at which point a clear homogenous solution was obtained. The excessive amount of toluene was removed under reduced pressure and the residue was distilled under high vacuum.

General Procedure B: Synthesis of vinyl oxetanes from ß-chloro ketone



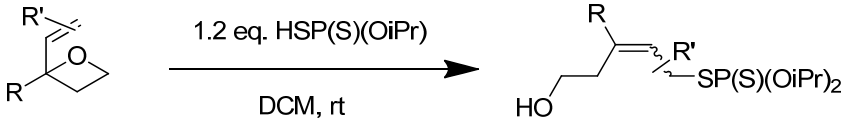
The preparation of vinyl oxetanes from the corresponding ß-chloro ketone as precursor has been described in our previous publication[2]. The same procedures were followed for the preparation of vinyl oxetanes here except for **1r.**

Cerium chloride ($CeCl_3 \cdot 7H_2O$) (3.55mmol, 1.2 eq) was finely ground into a powder in a mortar and transferred into a 50 mL two-necked flame dried round bottom flask charged with a stir bar. The flask was heated gradually to 140 °C in an oil bath under high vacuum and maintained at the same temperature for 2 hs. Then the oil bath was removed and the flask was cooled to room temperature. THF (10 mL) was introduced with vigorous stirring. The suspension was well stirred under nitrogen at room temperature for 2 h. Then the flask placed in an acetone-dry ice bath for another 30 mins. The corresponding vinyl magnesium bromide (3.55 mmol, 1.2 eq) was added via syringe. After being stirred for 45 mins at -78 °C, the corresponding ß-chloro ketone (2.96 mmol, 1 eq) in THF (15 mL) was added via syringe slowly. The reaction was maintained at -78 °C for 3 hrs, TLC indicated that all of the starting material was consumed. The reaction was quenched with 50 mL sat. $NaHCO_3$. After separation, the

aqueous layer was extracted with ether (4 x 10 mL). The combined organic layers were washed sequentially with sat. $NaHCO_3$ (1 x 5 mL), water (1 x 5mL) and brine (1 x 2 mL) then dried over $MgSO_4$. The crude product was purified by flash column chromatography on silica gel.

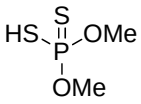
To a 50 mL flame-dried round bottom flask charged with a magnetic stir bar, was introduced 3 mL freshly distilled DMSO and the corresponding allylic alcohol (0.647 mmol, 1 eq). NaH (60% suspension in mineral oil, 1.30 mmol, 2 eq) was added in one portion. Reaction was stirred at room temperature for 30 minutes, during which course, a yellowish or brownish color appeared. The reaction was then chilled to 0 °C by standing in an ice bath, 3 mL sat. $NaHCO_3$ was added carefully. The organic layer was separated and washed with sat. $NaHCO_3$ (3 x 5 mL), H_2O (1 x 2 mL) and dried with $MgSO_4$. The crude product was purified by flash column chromatography.

General procedure C: Ring opening with dialkyl dithiophosphate



To a flame dried 25 mL rbf charged with magnetic stir bar was introduced 5 mL dry DCM, vinyl oxetane (1 mmol), and *O,O*-diisopropyl dithiophosphoric acid (1.2 mmol, 1.2 eq.). The reaction was stirred at room temperature and monitored by TLC till all of oxetane was consumed. DCM was then removed under reduced pressure and the crude residue was purified by flash chromatography to afford the corresponding alcohol product.

_O,O_-dimethyl dithiophosphoric acid (A_1)

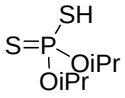


A1

Known acid $\mathbf{A_1}$ (reference 1) was prepared according to General Procedure **A** using P_4S_{10} (13.36 g, 30 mmol) and MeOH (12.2 mL, 300 mmol). Distillation under high vacuum afforded $\mathbf{A_1}$ (86%) as a clear liquid.

^{1}H NMR (400 MHz, CDCl3) δ 3.90 (s, 3H), 3.86 (s, 3H); **^{13}C NMR** (101 MHz, CDCl3) δ 54.33, 54.27; **^{31}P NMR** (162 MHz, CDCl3) δ 118.50.

O,O-diisopropyl dithiophosphoric acid (A_3)

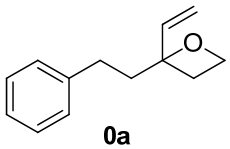


A3

Known acid $\mathbf{A_3}$ (reference 1) was prepared according to general procedure **A** using P_4S_{10} (10 g. 22.5 mmol) and *i*-PrOH (15.1 mL, 198 mmol). Distillation under high vacuum afforded $\mathbf{A_3}$ (75%) as a clear liquid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.94 – 4.87 (m, 2H), 1.38 (dd, *J* = 6.2, 2.4 Hz, 12H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 73.95, 73.88, 23.54, 23.50; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 81.76.

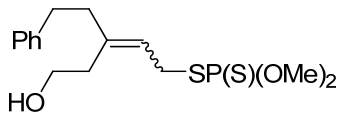
2-phenethyl-2-vinyloxetane (0a)



0a

For the preparation of oxetane **0a**, please see reference 2

(5-hydroxy-3-phenethylpent-2-en-1-yl) _O,O_-dimethyl phosphorodithioate (1aa)



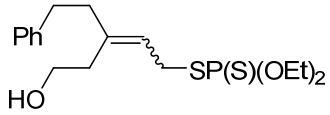
1aa

Compound **1aa** was prepared according to General Procedure **C** using oxetane **0a** (15 mg, 0.08 mmol) and acid $\mathbf{A_1}$ (15 mg, 0.096 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **1aa** (24 mg, 88%) as a clear liquid. The

Z/E isomers were inseparable and characterized as mixture. Only NMR-data was collected for this mixture since the isopropyl esters became the focus of our studies.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.31 – 7.26 (m, 2H), 7.22 – 7.14 (m, 3H), 5.43 (dt, *J* = 34.3, 7.9 Hz, 1H), 3.76 (dd, *J* = 15.0, 6.9 Hz, 6H), 3.73 – 3.68 (m, 2H), 3.45 (ddd, *J* = 23.2, 14.7, 7.9 Hz, 2H), 2.72 (td, *J* = 9.6, 7.1 Hz, 2H), 2.46 – 2.39 (m, 2H), 2.39 – 2.28 (m, 2H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 141.38, 140.44, 128.39, 128.34, 128.27, 125.95, 122.29, 122.23, 77.25, 77.00, 76.75, 60.73, 53.94, 53.90, 38.52, 34.47, 33.74, 31.33, 31.30;**^{31}P NMR** (162 MHz, $CDCl_3$) δ 99.21(*E* isomer) 99.16 (*Z* isomer).

(5-hydroxy-3-phenethylpent-2-en-1-yl) *O,O*-diethylphosphorodithioate (1ab)

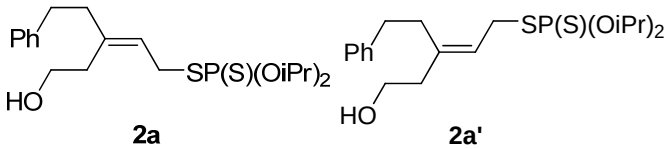


1ab

Compound **1ab** was prepared according to General Procedure C using oxetane **0a** (23 mg,0.122 mmol) and acid $\mathbf{A_2}$ (27 mg, 0.15 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **1ab** (37.5 mg, 83%) as a clear liquid. The Z/E isomers were inseparable and characterized as mixture. Only NMR-data was collected for this mixture since the isopropyl esters became the focus of our studies.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2H), 7.21 – 7.13 (m, 3H), 5.44 (t, 7.8 Hz, 1H), 4.27 – 4.06 (m, 4H), 3.70 (q, *J* = 6.6 Hz, 2H), 3.57 (dd, *J* = 13.8, 8.0 Hz, 2H), 2.72 (dt, *J* = 8.1, 6.6 Hz, 2H), 2.48 – 2.39 (m, 2H), 2.39 – 2.32 (m, 2H), 1.36 (dq, *J* = 7.1, 0.8 Hz, 6H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.48, 140.30, 128.40, 128.32, 125.99, 122.52, 122.45, 63.98, 63.92, 60.79, 38.60, 34.50, 33.80, 31.30, 31.27, 15.92, 15.84;**^{31}P NMR** (162 MHz, $CDCl_3$) δ 94.14(*E* isomer) 93.99(*Z* isomer).

(5-hydroxy-3-phenethylpent-2-en-1-yl) *O,O*-diisopropyl phosphorodithioate (2a)



2a **2a'**

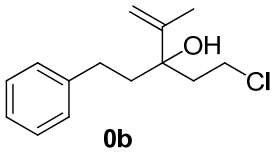
Coumpound **2a** was prepared according to General Procedure **C** using oxetane **1a** (20 mg, 0.1 mmol) and acid $\mathbf{A_3}$ (27 mg, 0.12 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2a +2a'** (35 mg, 87%) as a clear liquid.

2a: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.21 – 7.15 (m, 3H), 5.50 (t, *J* = 8.0 Hz, 1H), 4.83 (dhept, *J* = 12.4, 6.2 Hz, 2H), 3.70 (q, *J* = 6.5 Hz, 2H), 3.60 (dd, *J* = 13.2, 8.0 Hz, 2H), 2.73 (dd, *J* = 9.3, 6.9 Hz, 2H), 2.43 (t, *J* = 6.7 Hz, 2H), 2.35 (dd,

J = 11.7, 4.6 Hz, 2H), 1.36 (dt, *J* = 4.9, 2.5 Hz, 13H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 141.53, 140.21, 128.37, 128.31, 125.95, 122.45, 122.39, 73.48, 73.42, 60.83, 38.65, 34.54, 33.88, 31.50, 23.80, 23.76, 23.54, 23.49; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.23; **IR** (neat) ν_{max}/cm^{-1} 3520, 3464, 3024, 2945, 1454, 1417, 1176, 1012, 921, 813, 700, 657;**HRMS** [ESI] 402.1450 [$(M)^+$; calcd for $C_{19}H_{31}O_3PS_2$ 402.1452].

2a': **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.22 – 7.17 (m, 3H), 5.43 (t, *J* = 7.9 Hz, 1H), 4.86 – 4.77 (m, 2H), 3.71 (t, *J* = 6.3 Hz, 2H), 3.41 (dd, *J* = 13.9, 7.9 Hz, 2H), 2.72 (dd, *J* = 9.0, 7.0 Hz, 2H), 2.41 (dd, *J* = 9.0, 7.0 Hz, 2H), 2.31 (t, *J* = 6.1 Hz, 2H), 1.34 (dd, *J* = 7.5, 6.2 Hz, 12H);**^{13}C NMR** (126 MHz, $CDCl_3$) δ 141.32, 139.93, 128.41, 126.09, 123.00, 122.94, 73.44, 73.39, 60.49, 39.86, 34.74, 32.41, 31.11, 23.78, 23.75, 23.52, 23.48;**^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.07.

5-chloro-2-methyl-3-phenethylpent-1-en-3-ol (0b)

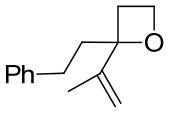


0b

Alcohol **0b** was prepared according to General Procedure **B** using 1-chloro-5-phenylpentan-3-one[2] (300 mg, 1.53 mmol) and isopropenylmagnesium bromide (3.67 mL, 0.5M, 1.84 mmol). Purification by flash chromatography [1% EA/Hexanes] afforded **0b** [313 mg, 86%] as a clear liquid.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.20 – 7.15 (m, 3H), 5.10 (dd, *J* = 1.4, 0.7 Hz, 1H), 5.04 (p, *J* = 1.4 Hz, 1H), 3.60 (ddd, *J* = 10.8, 9.2, 7.0 Hz, 1H), 3.47 (ddd, *J* = 10.8, 9.1, 6.1 Hz, 1H), 2.63 (ddd, *J* = 13.7, 11.2, 6.2 Hz, 1H), 2.50 (ddd, *J* = 13.7, 11.1, 5.3 Hz, 1H), 2.13 – 2.07 (m, 2H), 1.93 – 1.85 (m, 2H), 1.79 – 1.76 (m, 3H), 1.74 (s, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 146.47, 141.88, 128.49, 128.31, 125.93, 112.33, 77.60, 42.12, 41.62, 40.17, 29.45, 19.67; **IR** (neat) ν_{max}/cm^{-1} 3548, 3070, 3035, 2943, 2890, 1498, 1460, 1227, 1029, 990, 970, 757, 690; **HRMS** [ESI] 238.1120, 240.1071 [$(M)^+$; calcd for $C_{14}H_{19}ClO$ 238.1124, 240.1095].

2-phenethyl-2-(prop-1-en-2-yl) oxetane (1b)

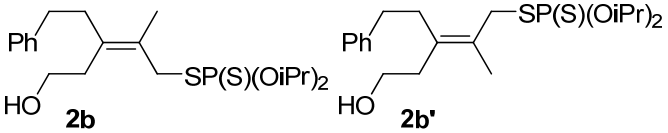


1b

Oxetane **1b** was prepared according to General Procedure **B** alcohol **0b** (150 mg, 0.63 mmol) and NaH (60% suspension in mineral oil, 51 mg, 1.26 mmol). Purification by flash chromatography [0.5:0.5:99 EA/TEA/Hexanes] gave vinyl oxetane **1b** as a clear liquid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.24 (m, 2H), 7.21 – 7.14 (m, 3H), 5.20 – 5.18 (m, 1H), 4.98 (dq, *J* = 3.1, 1.5 Hz, 1H), 4.47 (m, 2H), 2.70 – 2.59 (m, 1H), 2.55 – 2.41 (m, 3H), 2.16 (ddd, *J* = 13.7, 12.3, 4.8 Hz, 1H), 1.99 (ddd, *J* = 13.7, 12.5, 4.8 Hz, 1H), 1.70 (dd, *J* = 1.5, 0.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.96, 142.29, 128.34, 128.32, 125.70, 110.18, 89.40, 64.48, 40.84, 31.82, 29.69, 29.28, 17.20; **IR** (neat) ν_{max}/cm^{-1} 3084, 3061, 3026, 2933, 2879, 1496, 1452, 1226, 1172, 991, 977, 750, 698; **HRMS** [ESI] 202.1360 [$(M)^+$; calcd for $C_{14}H_{18}O$ 202.1358].

(5-hydroxy-2-methyl-3-phenethylpent-2-en-1-yl) *O,O*-diisopropyl phosphorodithioate (2b)

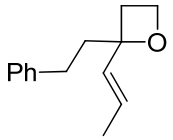


Compound **2b** was prepared according to General Procedure **C** using oxetane **1b** (57 mg, 0.28 mmol) and acid $\mathbf{A_3}$ (90 mg, 0.42 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2b + 2b'** [107 mg, 92%] as a clear liquid.

2b: **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.29 – 7.24 (m, 2H), 7.20 – 7.13 (m, 3H), 4.91 – 4.77 (m, 2H), 3.69 (t, *J* = 6.8 Hz, 2H), 3.63 (d, *J* = 9.6 Hz, 2H), 2.65 (dd, *J* = 9.3, 6.8 Hz, 2H), 2.42 (t, *J* = 6.8 Hz, 2H), 2.34 (dd, *J* = 9.3, 6.8 Hz, 2H), 1.71 (s, 3H), 1.37 (dd, *J* = 6.2, 1.2 Hz, 13H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.63, 134.45, 128.39, 127.63, 127.54, 125.96, 73.48, 73.41, 61.27, 38.02, 35.75, 34.78, 34.48, 23.79, 23.74, 23.52, 23.47, 17.92;**^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.56; **IR** (neat) ν_{max}/cm^{-1} 3450, 3024, 2945**,** 2887, 1494, 1452, 1438, 1226, 1176, 1014, 910, 813, 732, 700, 655; **HRMS** [ESI] 416.1608 [$(M)^+$; calcd for $C_{20}H_{33}O_3PS_2$ 416.1609]

2b': **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.29 – 7.24 (m, 2H), 7.21 – 7.15 (m, 3H), 4.83 (dhept, *J* = 12.4, 6.2 Hz, 2H), 3.66 (dt, *J* = 16.9, 8.3 Hz, 2H), 3.50 (d, *J* = 10.1 Hz, 2H), 2.70 (dd, *J* = 9.5, 6.7 Hz, 2H), 2.45 – 2.35 (m, 4H), 1.82 (s, 3H), 1.34 (dd, *J* = 6.2, 4.9 Hz, 12H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.62, 134.27, 128.42, 128.37, 127.69, 127.61, 126.00, 73.40, 73.34, 61.07, 37.60, 35.84, 35.36, 34.82, 23.77, 23.73, 23.51, 23.46, 18.13; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.09.

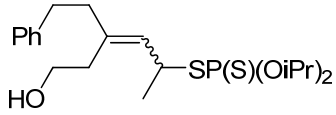
(*E*)-2-phenethyl-2-(prop-1-en-1-yl) oxetane (1c)



1c

For the preparation of **1c** please see reference 2.

(6-hydroxy-4-phenethylhex-3-en-2-yl) *O*,*O*-diisopropyl phosphorodithioate (2c)

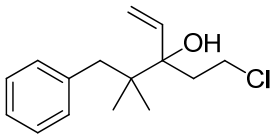


2c

Compound **2c** was prepared according to General Procedure C using oxetane **1c** (80 mg, 0.4 mmol) and acid $\mathbf{A_3}$ (102 mg, 0.47 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2c** [129 mg, 78%] as a clear liquid. The E/Z isomers were inseparable by flash chromatography and characterized as a mixture.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.20 – 7.14 (m, 3H), 5.29 (dd, J = 15.7, 10.3 Hz, 1H), 4.90 – 4.73 (m, 2H), 4.37 – 4.09 (m, 1H), 3.83 – 3.66 (m, 2H), 2.78 – 2.62 (m, 2H), 2.56 – 2.45 (m, 2H), 2.41 – 2.26 (m, 2H), 1.44 (t, J = 7.4 Hz, 3H), 1.33 (ddd, J = 10.9, 6.1, 2.3 Hz, 12H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.62, 141.50, 137.47, 136.78, 130.55, 130.47, 129.71, 129.62, 128.56, 128.40, 128.37, 128.33, 126.02, 125.91, 73.68, 73.60, 73.52, 73.48, 73.46, 73.41, 60.79, 60.33, 43.87, 43.84, 43.41, 43.37, 39.55, 38.35, 34.60, 34.44, 32.80, 24.23, 24.18, 23.78, 23.73, 23.70, 23.65, 23.61, 23.55, 23.51, 23.46, 23.43; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.87, 90.93; **IR** (neat) ν_{max}/cm^{-1} 3453, 3022, 2941**,** 2884, 1495, 1451, 1428, 1226, 1177, 1010, 916, 810, 728, 700, 650; **HRMS** [ESI] 416.1600 [$(M)^+$; calcd for $C_{20}H_{33}O_3PS_2$ 416.1609]

3-(2-chloroethyl)-4,4-dimethyl-5-phenylpent-1-en-3-ol (0d)

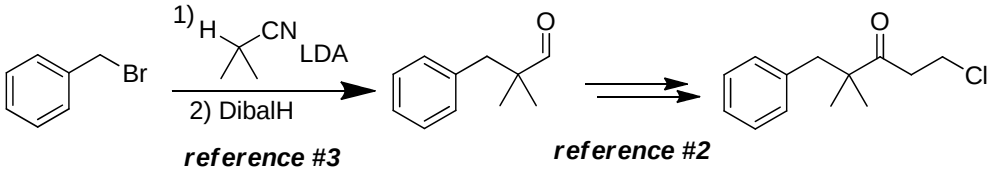


0d

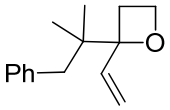
Alcohol **0d** was prepared according to General Procedure **B** using 5-chloro-2,2-dimethyl-1-phenylpentan-3-one (synthesized as shown below) (200 mg, 0.89 mmol) and vinylmagnesium bromide (1.53 mL, 0.7M, 1.07 mmol). Purification by flash chromatography afforded **0d** [204 mg, 91%] as a clear liquid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.22 (ddt, *J* = 6.4, 5.1, 2.5 Hz, 1H), 7.13 (dt, *J* = 3.3, 1.9 Hz, 2H), 6.02 (dd, *J* = 17.3, 11.0 Hz, 1H), 5.33 (m, 2H), 3.63 (td, *J* = 10.5, 6.0 Hz, 1H), 3.53 (td, *J* = 10.6, 4.9 Hz, 1H), 2.69 – 2.61 (m, 2H), 2.27 (ddd, *J* = 13.7, 10.6, 6.1 Hz, 1H), 2.15 – 2.07 (m, 1H), 1.72 (d, *J* = 1.1 Hz, 1H), 0.87 (s, 3H), 0.82 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.38, 138.81, 131.07, 127.76, 126.01, 115.11, 79.95, 42.10, 41.72, 41.33, 37.14, 21.51, 21.15; **IR** (neat) ν_{max}/cm^{-1} 3423, 3418, 2927, 1274, 1177, 1032, 909, 733, 704; **HRMS** [ESI] 252.1278 [$(M)^+$; calcd for $C_{15}H_{21}ClO$ 252.1281]

Synthesis of ß-Chloro Ketone Precursor:



2-(2-methyl-1-phenylpropan-2-yl)-2-vinyloxetane (1d)

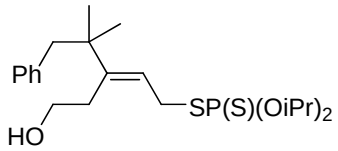


1d

Oxetane **1d** was prepared according to General Procedure **B** using alcohol **0d** (133 mg, 0.53 mmol) and NaH (60% suspension in mineral oil, 42 mg, 1.05 mmol). Purification by flash chromatography [1% EA/Hexanes] gave oxetane **1d** [104 mg, 93%] as a clear liquid.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.28 – 7.23 (m, 2H), 7.22 – 7.17 (m, 1H), 7.13 (dt, *J* = 8.1, 2.5 Hz, 2H), 6.15 (dd, *J* = 17.0, 10.8 Hz, 1H), 5.49 (dd, *J* = 17.0, 2.0 Hz, 1H), 5.30 (dd, *J* = 10.8, 2.0 Hz, 1H), 4.47 (ddd, *J* = 8.8, 7.2, 5.8 Hz, 1H), 4.36 (dt, *J* = 9.2, 5.9 Hz, 1H), 2.78 (ddd, *J* = 11.1, 9.2, 7.2 Hz, 1H), 2.57 (s, 2H), 2.26 (ddd, *J* = 11.1, 8.8, 6.0 Hz, 1H), 0.90 (s, 3H), 0.86 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 139.95, 138.90, 130.92, 127.64, 125.80, 113.58, 92.44, 64.43, 41.09, 40.45, 27.96, 20.14, 20.09; **IR** (neat) ν_{max}/cm^{-1} 3080, 3071, 3020, 2930, 2881, 1495, 1450, 1226, 1171, 977, 750, 698; **HRMS** [ESI] 216.1520 [$(M)^+$; calcd for $C_{15}H_{20}O$ 216.1514].

(3-(2-hydroxyethyl)-4,4-dimethyl-5-phenylpent-2-en-1-yl) O,O-diisopropyl phosphorodithioate (2d)

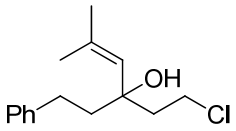


2d

Compound **2d** was prepared according to General Procedure **C** using oxetane **1d** (7 mg, 0.032 mmol) and acid $\mathbf{A_3}$ (8.3 mg,0.038 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2d** [13 mg, 95%] as a clear liquid.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.25 – 7.15 (m, 3H), 7.03 – 6.99 (m, 2H), 5.31 (t, *J* = 8.1 Hz, 1H), 4.82 (dhept, *J* = 12.4, 6.2 Hz, 2H), 3.70 – 3.63 (m, 2H), 3.60 (dd, *J* = 12.2, 8.1 Hz, 2H), 2.62 (s, 2H), 2.56 – 2.49 (m, 2H), 1.35 (dd, *J* = 6.2, 3.9 Hz, 12H), 1.03 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 146.12, 138.43, 130.45, 127.60, 125.99, 121.36, 121.30, 73.43, 73.38, 62.50, 47.47, 40.73, 32.35, 32.32, 32.04, 26.54, 23.75, 23.71, 23.49, 23.45;**^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.66; **IR** (neat) ν_{max}/cm^{-1} 3516, 3403, 3386, 2977, 2931, 1465, 1452, 1384, 1373, 1176, 1102, 1037, 989, 966, 775, 702, 651;**HRMS** [ESI] 430.1760 [$(M)^+$; calcd for $C_{21}H_{35}O_3PS_2$ 430.1765].

1-chloro-5-methyl-3-phenethylhex-4-en-3-ol (0e)

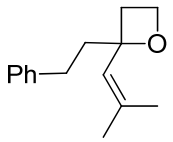


0e

Alcohol **0e** was prepared according to General Procedure **B** using 1-chloro-5-phenylpentan-3-one (300 mg, 1.53 mmol) and 2-methyl propenylmagnesium bromide (3.67 mL, 0.5 M, 1.84 mmol). Purification by flash chromatography afforded **0e** [340 mg, 88%] as a clear liquid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.20 – 7.16 (m, 3H), 5.16 – 5.15 (m, 1H), 3.70 – 3.65 (m, 1H), 3.64 – 3.58 (m, 1H), 2.75 – 2.63 (m, 2H), 2.12 (dd, *J* = 14.9, 7.2 Hz, 2H), 1.91 – 1.87 (m, 2H), 1.86 (d, *J* = 1.3 Hz, 3H), 1.76 (d, *J* = 1.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.16, 135.57, 128.46, 128.33, 128.06, 125.87, 75.60, 44.89, 44.49, 40.66, 30.15, 27.59, 18.86; **IR** (neat) ν_{max}/cm^{-1}3555, 3518, 3448, 3442, 3025, 2965, 1700, 1250, 1186, 1035, 928, 907, 736, 704; **HRMS** [ESI] 252.1280, 254.1244 [$(M)^+$; calcd for $C_{15}H_{21}ClO$ 252.1281, 154.1251]

2-(2-methylprop-1-en-1-yl)-2-phenethyloxetane (1e)

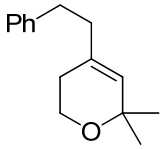


1e

Oxetane **1e** was prepared according to General Procedure **B** from using alcohol 0e (30 mg, 0.12 mmol) and NaH (60% suspension in mineral oil, 9.6 mg, 0.24 mmol). Purification by flash chromatography [0.5:0.5:99 EA:TEA:Hexanes] afforded oxetane **1b** [24 mg, 96%] as a clear liquid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.23 – 7.16 (m, 3H), 5.41 (dq, *J* = 2.7, 1.3 Hz, 1H), 4.52 (dd, *J* = 8.4, 7.1 Hz, 2H), 2.80 – 2.64 (m, 3H), 2.61 – 2.55 (m, 1H), 2.19 (ddd, *J* = 13.5, 12.2, 4.8 Hz, 1H), 2.04 (ddd, *J* = 13.5, 12.4, 5.0 Hz, 1H), 1.74 (d, *J* = 1.4 Hz, 3H), 1.66 (d, *J* = 1.2 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 142.50, 132.04, 130.57, 128.32, 125.66, 87.86, 65.30, 43.88, 33.60, 29.78, 26.14, 18.67; **IR** (neat) ν_{max}/cm^{-1} 3419, 3413, 1469, 1399, 1250, 1231, 1035, 994, 968, 922; **HRMS** [ESI] 216.1510 $[(M)^+$; calcd for $C_{15}H_{21}O$ 216.1514]

6, 6-dimethyl-4-phenethyl-3,6-dihydro-2*H*-pyran (2e')

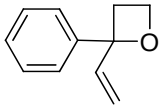


2e'

Pyran **2e'** was prepared according to General Procedure **C** using oxetane **1e** (15 mg, 0.07 mmol) and acid $\mathbf{A_3}$ (17.8 mg, 0.083 mmol). Purification by flash chromatography [1% EA/Hexanes] afforded **2e'** [13 mg, 86%] as a clear sticky oil.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 – 7.27 (m, 2H), 7.21 – 7.16 (m, 3H), 5.26 (p, *J* = 1.5 Hz, 1H), 3.79 (t, *J* = 5.5 Hz, 2H), 2.73 (dd, *J* = 8.9, 6.9 Hz, 2H), 2.30 – 2.25 (m, 2H), 2.03 – 1.98 (m, 2H), 1.19 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.93, 133.26, 129.15, 128.43, 128.24, 125.78, 71.69, 59.56, 38.95, 33.92, 28.31, 27.64; **IR** (neat) ν_{max}/cm^{-1} 3058, 3033, 2977, 2957, 2848, 1492, 1444, 1386, 1360, 1237, 1135, 968, 913, 699, 668; **HRMS** [ESI] 216.1514 $[(M)^+$; calcd for $C_{15}H_{21}O$ 216.1514]

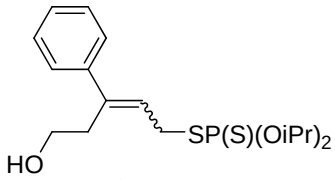
2-phenyl-2-vinyloxetane (1f)



1f

For the preparation of oxetane 1f, please see reference 2.

(5-hydroxy-3-phenylpent-2-en-1-yl) *O,O*-diisopropyl phosphorodithioate (2f)

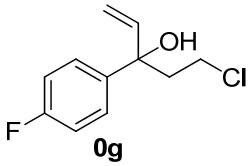


2f

Compound **2f** was prepared according to General Procedure **C** using oxetane **1f** (6 mg, 0.037 mmol) and acid $\mathbf{A_3}$ (9.6 mg, 0.045 mmol). Purification by flash chromatography afforded **2f** (12 mg, 90%). The *E* isomer was lost in mixture and only *Z* isomer was characterized.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.37 – 7.26 (m, 6H), 5.95 (t, *J* = 8.1 Hz, 1H), 4.90 – 4.81 (m, 2H), 3.78 (dd, *J* = 14.0, 8.1 Hz, 2H), 3.65 (d, *J* = 5.9 Hz, 2H), 2.88 (t, *J* = 6.7 Hz, 2H), 1.36 (dd, *J* = 6.2, 4.6 Hz, 13H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.35, 140.43, 128.46, 127.63, 126.44, 125.28, 125.21, 73.61, 73.54, 61.15, 33.39, 32.14, 32.10, 23.79, 23.74, 23.52, 23.47; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.08; **IR** (neat) ν_{max}/cm^{-1} 3441, 3418, 2979, 2933, 1602, 1509, 1385, 1373, 1225, 1102, 1041, 989, 837, 775, 651; **HRMS** [ESI] 374.1130 [$(M)^+$; calcd for $C_{17}H_{27}O_3PS_2$ 374.1139]

5-chloro-3-(4-fluorophenyl) pent-1-en-3-ol (0g)



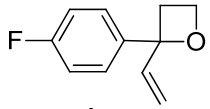
0g

Alcohol **0g** was prepared according to General Procedure **B** using 3-chloro-4'-fluoropropiophenone (commercially available from Acros) (300 mg, 1.61 mmol) and vinylmagnesium bromide (2.76 mL, 1.93 mmol). Purification by flash chromatography afforded **0g** [294 mg, 86%] as a clear liquid.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.42 – 7.37 (m, 2H), 7.07 – 7.01 (m, 2H), 6.14 (dd, *J* = 17.2, 10.7 Hz, 1H), 5.32 (ddd, *J* = 17.3, 2.4, 0.8 Hz, 1H), 5.23 (dd, *J* = 10.7, 0.8 Hz, 1H), 3.60 – 3.54 (m, 1H), 3.44 – 3.37 (m, 1H), 2.42 (ddd, *J* = 14.0, 10.3, 5.6 Hz, 1H),

2.33 (ddd, *J* = 14.0, 10.3, 5.5 Hz, 1H), 2.06 (s, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 162.94, 160.98, 142.86, 140.04, 140.01, 127.00, 126.93, 115.31, 115.14, 113.81, 76.17, 44.54, 39.99; **IR** (neat) ν_{max}/cm^{-1} 3559, 3551, 3518, 3064, 3025, 2965, 1460, 1489, 1250, 1186, 1035, 928, 907, 735, 703; **HRMS** [ESI] 214.0560, 216.0525 $[(M)^+$; calcd for $C_{11}H_{12}ClFO$ 214.0561, 216.0531]

2-(4-fluorophenyl)-2-vinyloxetane (1g)

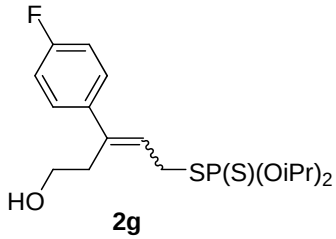


1g

Oxetane **1g** was prepared according to General Procedure **B** using alcohol **0g** (160 mg, 0.75 mmol) and NaH (60% suspension in mineral oil, 45 mg, 1.12 mmol). Purification by flash chromatography [0.5:0.5:99 EA/TEA/Hexanes] afforded **1g** [131 mg, 98%] as a clear liquid.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.38 – 7.32 (m, 2H), 7.08 – 7.01 (m, 2H), 6.18 (dd, *J* = 17.1, 10.5 Hz, 1H), 5.32 (dd, *J* = 17.1, 1.4 Hz, 1H), 5.21 (dt, *J* = 2.7, 1.3 Hz, 1H), 4.57 (m, 2H), 2.99 – 2.90 (m, 1H), 2.81 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 162.80, 160.84, 142.03, 141.04, 141.02, 126.27, 126.21, 115.10, 114.93, 113.29, 87.38, 64.93, 34.48; **IR** (neat) ν_{max}/cm^{-1} 3559, 3555, 3521, 3057, 3020, 2964, 1471, 1470, 1249, 1185, 1055, 934, 921, 730, 713; **HRMS** [ESI] 178.0793 $[(M)^+$; calcd for $C_{11}H_{11}FO$ 178.0794]

(3-(4-fluorophenyl)-5-hydroxypent-2-en-1-yl) O,O-diisopropyl phosphorodithioate (2g)

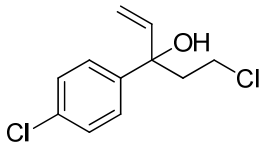


2g

Compound **2g** was prepared according to General Procedure **C** using oxetane **1g** (67 mg, 0.37 mmol) and acid $\mathbf{A_3}$ (96.6 mg, 0.45 mmol). Purification by flash chromatography afforded **2g** [137 mg, 93%] as a clear liquid. The E/Z isomers were inseparable and characterized as a mixture.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34 – 7.30 (m, 2H), 7.03 – 6.99 (m, 2H), 5.89 (dd, *J* = 9.9, 6.3 Hz, 1H), 4.85 (tq, *J* = 12.4, 6.2 Hz, 2H), 3.77 (dd, *J* = 14.2, 8.1 Hz, 2H), 3.64 (dd, *J* = 8.3, 5.2 Hz, 2H), 2.84 (t, *J* = 6.7 Hz, 2H), 1.35 (t, *J* = 6.0 Hz, 12H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 163.55, 161.09, 139.43, 137.43, 137.40, 128.08, 128.00, 125.29, 125.23, 115.39, 115.17, 73.64, 73.57, 61.00, 33.47, 32.05, 32.02, 23.75, 23.71, 23.49, 23.44; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.08; **IR** (neat) ν_{max}/cm^{-1}3418, 3406, 2979, 2933, 1509, 1385, 1373, 1225, 1178, 1161, 1102, 989, 970, 888, 835, 775, 651; **HRMS** [ESI] 392.1040 [$(M)^+$; calcd for $C_{17}H_{26}FO_3PS_3$ 392.1045]

5-chloro-3-(4-chlorophenyl) pent-1-en-3-ol (0h)

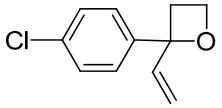


0h

Alcohol **0h** was prepared according to General Procedure **B** using 3,4'-dichloropropiophenone (commercially available from Aldrich) (300 mg, 1.48 mmol) and vinylmagnesium bromide (2.54 mL, 0.7 M, 1.78 mmol). Purification by flash chromatography afforded **0h** [272 mg, 78%] as a clear liquid.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.38 – 7.31 (m, 4H), 6.13 (dd, *J* = 17.2, 10.7 Hz, 1H), 5.33 (dt, *J* = 17.3, 1.4 Hz, 1H), 5.24 (dd, *J* = 10.7, 0.4 Hz, 1H), 3.61 – 3.54 (m, 1H), 3.43 – 3.36 (m, 1H), 2.37 (dddd, *J* = 39.0, 14.0, 10.2, 5.6 Hz, 2H), 2.04 (d, *J* = 1.7 Hz, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 142.77, 142.68, 133.22, 128.58, 126.69, 114.01, 76.20, 44.44, 39.90; **IR** (neat) ν_{max}/cm^{-1}3563, 3558, 3523, 3070, 3012, 2960, 1570, 1461, 1452, 1248, 1155, 1025, 927, 912, 730, 702; **HRMS** [ESI] 230.0261, 232.0228 [$(M)^+$; calcd for $C_{11}H_{12}Cl_2O$ 230.0265, 232.0236]

2-(4-chlorophenyl)-2-vinyloxetane (1h)

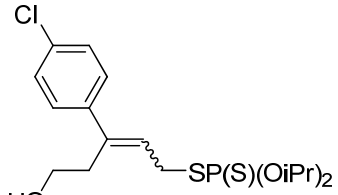


1h

Oxetane **1h** was prepared according to General Procedure **B** using alcohol **0h** (73 mg, 0.32 mmol) and NaH (60% suspension in mineral oil, 19 mg, 0.47 mmol). Purification by flash chromatography [0.5:0.5:99 EA/TEA/Hexanes] afforded **1h** [56 mg, 92%] as a clear liquid.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.35 – 7.30 (m, 4H), 6.17 (dd, *J* = 17.1, 10.5 Hz, 1H), 5.32 (dd, *J* = 17.1, 1.3 Hz, 1H), 5.22 (dd, *J* = 10.5, 1.3 Hz, 1H), 4.57 (m, 2H), 2.95 (ddd, *J* = 10.7, 8.6, 6.6 Hz, 1H), 2.80 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 143.82, 141.79, 132.79, 128.38, 125.97, 113.54, 87.34, 65.01, 34.49; **IR** (neat) v_{max}/cm^{-1}3561, 3553, 3517, 3043, 2960, 1478, 1471, 1244, 1255, 1034, 933, 920, 728, 713; **HRMS** [ESI] 194.0490 [(M)$^+$; calcd for $C_{11}H_{11}ClO$ 194.0498]

(3-(4-chlorophenyl)-5-hydroxypent-2-en-1-yl) *O,O*-diisopropyl phosphorodithioate (2h)

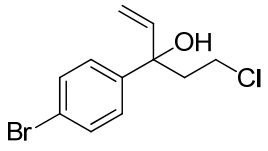


2h

Compound **2h** was prepared according to General Procedure **C** using oxetane **1h** (46 mg, 0.23 mmol) and acid $\mathbf{A_3}$ (60 mg, 0.28 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2h** [89 mg, 92%] as a clear liquid. The E/Z isomers were inseparable and characterized as a mixture.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.32 (s, 4H), 5.96 (t, *J* = 8.1 Hz, 1H), 4.93 – 4.82 (m, 2H), 3.80 (dd, *J* = 14.5, 8.1 Hz, 2H), 3.67 (t, *J* = 6.7 Hz, 2H), 2.87 (t, *J* = 6.7 Hz, 2H), 1.38 (dd, *J* = 6.1, 5.1 Hz, 13H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 139.83, 139.28, 133.44, 128.60, 127.74, 125.87, 125.82, , 73.67, 73.61, 61.02, 33.29, 32.04, 32.02, 23.77, 23.73, 23.51, 23.47; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.06; **IR** (neat) v_{max}/cm^{-1}3616, 3508, 3445, 3418, 1388, 1373, 1251, 1177, 1101, 989, 967, 775, 651; **HRMS** [ESI] 408.0749 [(M)$^+$; calcd for $C_{17}H_{26}ClO_3PS_2$ 408.0750]

3-(4-bromophenyl)-5-chloropent-1-en-3-ol (0i)

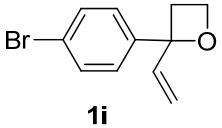


0i

Alcohol **0i** was prepared according to General Procedure **B** using 3-chloro-4'-bromopropiophenone (commercially available from Acros) (300 mg, 1.2 mmol) and vinylmagnesium bromide (2 mL, 1.4 mmol). Purification by flash chromatography [1% EA/Hexanes] afforded 0i [264 mg, 80%] as a clear liquid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.50 – 7.47 (m, 2H), 7.32 – 7.29 (m, 2H), 6.13 (dd, *J* = 17.2, 10.7 Hz, 1H), 5.33 (dd, *J* = 17.2, 2.7 Hz, 1H), 5.25 (dd, *J* = 9.2, 5.9 Hz, 1H), 3.57 (td, *J* = 10.5, 5.5 Hz, 1H), 3.42 – 3.36 (m, 1H), 2.44 – 2.29 (m, 2H), 2.04 (s, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.31, 142.63, 131.54, 127.05, 121.37, 114.05, 76.25, 44.39, 39.88; **IR** (neat) ν_{max}/cm^{-1}3545, 3550, 3512, 3049, 2958, 2948, 1546, 1459, 1444, 1240, 1154, 1023, 925, 910, 728, 714; **HRMS** [ESI] 273.9758, 275.9738 [$(M)^+$; calcd for $C_{11}H_{12}BrClO$ 273.9760, 275.9740]

2-(4-bromophenyl)-2-vinyloxetane (1i)

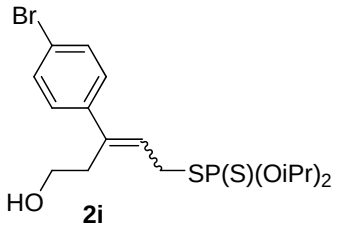


1i

Oxetane **1i** was prepared according to General Procedure **B** using alcohol **0i** (52 mg, 0.19 mmol) and NaH (60% suspension in mineral oil, 15 mg, 0.37 mmol). Purification by flash chromatography [0.5:0.5:99 EA/TEA/Hexanes] afforded **1i** [42 mg, 94%] as a clear liquid.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.51 – 7.45 (m, 2H), 7.27 – 7.23 (m, 2H), 6.16 (dd, *J* = 17.1, 10.6 Hz, 1H), 5.32 (dd, *J* = 17.1, 1.3 Hz, 1H), 5.21 (dd, *J* = 10.5, 1.3 Hz, 1H), 4.59 (ddd, *J* = 8.6, 6.8, 5.8 Hz, 1H), 4.53 (ddd, *J* = 8.7, 6.5, 5.8 Hz, 1H), 2.95 (ddd, *J* = 10.7, 8.6, 6.6 Hz, 1H), 2.79 (ddd, *J* = 10.8, 8.7, 6.8 Hz, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 144.45, 141.81, 131.36, 126.36, 120.95, 113.56, 87.39, 64.99, 34.54; **IR** (neat) ν_{max}/cm^{-1}3550, 3548, 3502, 3043, 2960, 1468, 1461, 1254, 1251, 1024, 932, 921, 725, 712; **HRMS** [ESI] 237.9990 [$(M)^+$; calcd for $C_{11}H_{11}BrO$ 237.9993]

(3-(4-bromophenyl)-5-hydroxypent-2-en-1-yl) O,O-diisopropyl phosphorodithioate (2i)

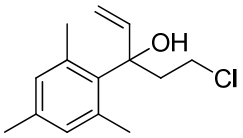


2i

Compound **2i** was prepared according to General Procedure **C** using oxetane 1i (25 mg, 0.1 mmol) and acid $\mathbf{A_3}$ (27 mg, 0.12 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2i** [43 mg, 96%] as a clear liquid. The E/Z isomers were inseparable and characterized as a mixture.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.46 – 7.43 (m, 2H), 7.25 – 7.21 (m, 2H), 5.94 (dd, *J* = 10.5, 5.6 Hz, 1H), 4.89 – 4.80 (m, 2H), 3.76 (dd, *J* = 14.5, 8.1 Hz, 2H), 3.64 (dd, *J* = 11.8, 6.5 Hz, 2H), 2.84 (t, *J* = 6.7 Hz, 2H), 1.35 (dd, *J* = 8.0, 4.4 Hz, 13H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.31, 139.33, 131.55, 128.08, 125.94, 125.87, 121.58, 73.68, 73.61, 61.02, 33.24, 32.04, 32.01, 23.77, 23.72, 23.51, 23.46; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.05, 90.88; **IR** (neat) ν_{max}/cm^{-1}3418, 2978,2931, 2876, 1486, 1466, 1379, 1373, 1250, 1177, 1141, 1076, 1042, 1007, 989, 969, 775, 651; **HRMS** [ESI] 452.0238 [$(M)^+$; calcd for $C_{17}H_{26}BrO_3PS_2$ 452.0244]

5-chloro-3-mesitylpent-1-en-3-ol (0j)



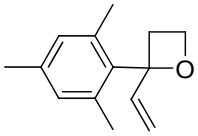
0j

Alcohol **0j** was prepared according to General Procedure **B** using 3-chloro-1-mesitylpropan-1-one (synthesized from mesitylene and 3-chloropropionyl chloride) (322 mg, 1.53 mmol) and vinylmagnesium bromide (2.6 mL, 1.8 mmol). Purification by flash chromatography [1% EA/Hexanes] afforded **0j** [280 mg, 77%] as a clear liquid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.80 (dd, *J* = 1.1, 0.6 Hz, 2H), 6.35 (dd, *J* = 17.4, 10.6 Hz, 1H), 5.18 (ddd, *J* = 6.8, 4.1, 0.7 Hz, 2H), 3.72 (ddd, *J* = 11.1, 10.5, 5.4 Hz, 1H), 3.37 (ddd, *J* = 11.1, 10.5, 5.0 Hz, 1H), 2.68 (dddd, *J* = 13.8, 11.1, 5.0, 0.6 Hz, 1H), 2.44 – 2.41 (m, 7H), 2.41 – 2.35 (m, 1H), 2.23 (s, 3H), 1.71 (d, *J* = 0.5 Hz, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.34, 136.68, 136.34, 136.10, 131.98, 114.12, 79.40,

43.99, 40.57, 24.74, 20.35; **IR** (neat) ν_{max}/cm^{-1} 3543, 3439, 3088, 3061, 2976, 2873, 1492, 1446, 1105, 925, 769, 700; **HRMS** [ESI] 238.1121, 240.1087 $[(M)^+$; calcd for $C_{11}H_{19}ClO$ 238.1124, 240.1095]

2-mesityl-2-vinyloxetane (1j)

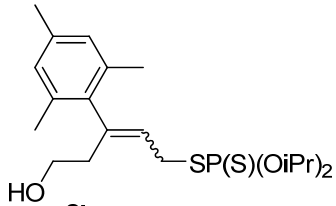


1j

Oxetane **1j** was prepared according to General Procedure **B** using alcohol **0j** (300 mg, 1.27 mmol) and NaH (60% suspension in mineral oil, 202 mg, 2.55 mmol). Purification by flash chromatography [0.5:0.5:99 EA/TEA/Hexanes] afforded **1i** [230 mg, 90%] as a clear liquid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.78 (dt, *J* = 1.2, 0.6 Hz, 2H), 6.37 (dd, *J* = 17.0, 10.4 Hz, 1H), 5.18 (ddd, *J* = 13.7, 11.3, 1.6 Hz, 2H), 4.62 (ddd, *J* = 8.4, 7.7, 5.6 Hz, 1H), 4.38 (ddd, *J* = 9.4, 5.6, 4.8 Hz, 1H), 3.24 (ddd, *J* = 10.8, 9.4, 8.5 Hz, 1H), 2.80 (ddd, *J* = 10.8, 7.7, 4.8 Hz, 1H), 2.24 (s, 3H), 2.19 (s, 6H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.45, 137.89, 136.13, 129.98, 113.33, 91.12, 65.28, 36.65, 21.13, 20.60; **IR** (neat) ν_{max}/cm^{-1}3084, 3059, 3024, 3001, 2956, 2883, 1490, 1446, 985, 966, 759, 700; **HRMS** [ESI] 202.1355 $[(M)^+$; calcd for $C_{14}H_{18}O$ 202.1358]

(5-hydroxy-3-mesitylpent-2-en-1-yl) O,O-diisopropyl phosphorodithioate (2j)



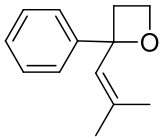
2j

Thioate **2j** was prepared according to General Procedure **C** using oxetane 1j (40 mg, 0.19 mmol) and acid $\mathbf{A_3}$ (50 mg, 0.24 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2j** [67 mg, 85%] as a clear liquid. The E/Z isomers were inseparable and characterized as a mixture.

^{1}H NMR (600 MHz, $CDCl_3$) δ 6.86 (s, 2H), 5.76 (tt, *J* = 7.8, 1.3 Hz, 1H), 4.76 (dhept, *J* = 12.4, 6.2 Hz, 2H), 3.72 (t, *J* = 6.6 Hz, 2H), 3.21 (dd, *J* = 13.3, 7.8 Hz, 2H), 2.50 (td, *J* = 6.5, 0.9 Hz, 2H), 2.26 (s, 3H), 2.15 (s, 6H), 1.30 (d, *J* = 6.2 Hz, 6H), 1.24 (d, *J* = 6.2 Hz, 6H);**^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.69, 136.64, 135.02, 128.51, 123.14, 123.07, 73.36, 73.30, 60.45, 40.93, 32.28, 23.73, 23.69, 23.29, 23.24, 20.89, 19.82;

^{31}P NMR (162 MHz, $CDCl_3$) δ 90.58, 90.32; **IR** (neat) v_{max}/cm^{-1} 3516, 3508, 3470, 3442, 3384, 2979, 2921, 1384, 1373, 1178, 1141, 1102, 1034, 990, 966, 889, 850, 777, 652; **HRMS** [ESI] 416.1600 [$(M)^+$; calcd for $C_{20}H_{33}O_3PS_2$ 416.1609]

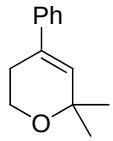
2-(2-methylprop-1-en-1-yl)-2-phenyloxetane (1k)



1k

For the synthesis of oxetane **1k**, please see reference 2.

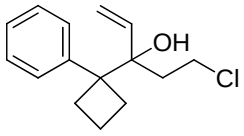
6, 6-dimethyl-4-phenyl-3,6-dihydro-2H-pyran (2k)



2k

Pyran **2k** was prepared according to General Procedure **C** using oxetane **1k** (15 mg, 0.8 mmol) and acid $\mathbf{A_3}$ (20 mg, 0.95 mmol). Purification by flash chromatography [1% EA/Hexanes] afforded 2k [13.5 mg, 90%] as a clear liquid. For the spectroscopic data please see ***ref.2***.

5-chloro-3-(1-phenylcyclobutyl)pent-1-en-3-ol (0l)



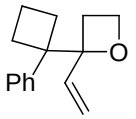
0l

Alcohol **0l** was prepared according to General Procedure **B** using 3-chloro-1-(1-phenylcyclobutyl) propan-1-one (prepared from 1-phenylcyclobutanecarbonitrile as described in **0d**) (300 mg, 1.35 mmol) and vinylmagnesium bromide (2.3 mL, 1.62 mmol). Purification by flash chromatography [1% EA/Hexanes] afforded **0l** [260 mg, 79%] as a clear liquid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34 – 7.30 (m, 2H), 7.24 – 7.20 (m, 3H), 5.81 (dd, *J* = 17.3, 10.9 Hz, 1H), 5.32 – 5.25 (m, 2H), 3.51 (td, *J* = 10.6, 6.0 Hz, 1H), 3.40 (td, *J* = 10.6, 5.1 Hz, 1H), 2.67 – 2.60 (m, 1H), 2.52 – 2.45 (m, 1H), 2.37 (m, 2H), 1.98 (ddd, *J* = 13.8, 10.5, 6.0 Hz, 1H), 1.91 – 1.85 (m, 2H), 1.79 (dtt, *J* = 11.6, 10.0, 5.9 Hz, 1H), 1.68 (s, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.23, 138.41, 128.49, 127.61, 126.12, 115.27, 77.55, 53.65, 41.01, 37.44, 29.77, 29.57, 15.62; **IR** (neat) v_{max}/cm^{-1}3581,

3567, 3081, 3021, 2984, 2947, 2864, 1700, 1492, 1444, 1412, 1342, 1186, 1006, 925, 786, 754, 706; **HRMS** [ESI] 250.1120, 252.1088 $[(M)^+$; calcd for $C_{15}H_{19}ClO$ 250.1124, 252.1095]

2-(1-phenylcyclobutyl)-2-vinyloxetane (1l)

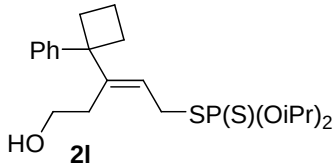


1l

Oxetane **1l** was prepared according to General Procedure B using alcohol 0l(220 mg, 0.87 mmol) and NaH (60% suspension in mineral oil, 70 mg, 1.75 mmol). Purification by flash chromatography [0.5:0.5:99 EA/TEA/Hexanes] afforded **1l** [178 mg, 95%] as a clear oil.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 2H), 7.24 – 7.18 (m, 3H), 5.64 (dd, *J* = 17.0, 10.7 Hz, 1H), 5.36 (dd, *J* = 17.0, 2.1 Hz, 1H), 5.14 (dd, *J* = 10.7, 2.1 Hz, 1H), 4.45 (td, *J* = 8.1, 5.7 Hz, 1H), 4.36 (dt, *J* = 9.3, 5.5 Hz, 1H), 2.97 – 2.88 (m, 1H), 2.85 (ddd, *J* = 10.9, 9.3, 7.9 Hz, 1H), 2.50 – 2.36 (m, 2H), 2.32 – 2.23 (m, 1H), 2.08 (ddd, *J* = 10.9, 8.4, 5.3 Hz, 1H), 1.93 (dtt, *J* = 11.2, 9.8, 6.0 Hz, 1H), 1.80 (dtt, *J* = 11.2, 9.8, 6.7 Hz, 1H);**^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.20, 138.44, 127.81, 127.51, 125.83, 113.57, 90.02, 64.27, 52.24, 29.74, 27.90, 27.54, 15.32; **IR** (neat) ν_{max}/cm^{-1} 3087, 3058, 3021, 2984, 2932, 2878, 1700, 1489, 1444, 1278, 1253, 1225, 1081, 980, 964, 924, 747, 703, 668, 651; **HRMS** [ESI] 214.1358 $[(M)^+$; calcd for $C_{15}H_{18}O$ 214.1358]

(5-hydroxy-3-(1-phenylcyclobutyl)pent-2-en-1-yl) *O,O*-diisopropyl phosphorodithioate (2l)



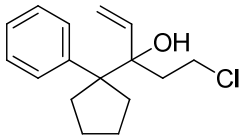
2l

Compound **2l** was prepared according to General Procedure **C** using oxetane **1l** (110 mg, 0.51 mmol) and acid $\mathbf{A_3}$ (132 mg, 0.62 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2l** [198 mg, 91%] as a clear liquid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33 – 7.28 (m, 4H), 7.21 – 7.18 (m, 1H), 5.78 (t, *J* = 8.0 Hz, 1H), 4.86 (dhept, *J* = 12.4, 6.2 Hz, 2H), 3.66 (dd, *J* = 13.3, 8.0 Hz, 2H), 3.07 (d, *J* = 2.7 Hz, 2H), 2.45 – 2.34 (m, 4H), 2.21 (t, *J* = 7.4 Hz, 2H), 1.88 – 1.75 (m, 2H), 1.38 (d, *J* = 6.2 Hz, 12H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.34, 145.74, 128.30, 126.38, 126.06, 120.20, 120.12, 73.47, 73.40, 61.63, 53.57, 32.58, 32.07, 31.79, 23.79,

23.75, 23.55, 23.49, 15.83; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.44; **IR** (neat) ν_{max}/cm^{-1}3423, 3419, 3406, 3384, 3055, 3021, 2977, 2871, 1492, 1444, 1384, 1178, 1141, 1103, 965, 936, 888, 797, 759, 701, 651; **HRMS** [ESI] 428.1600 $[(M)^+$; calcd for $C_{21}H_{33}O_3PS_2$ 428.1609]

5-chloro-3-(1-phenylcyclopentyl)pent-1-en-3-ol (0m)

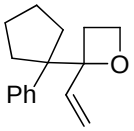


0m

Alcohol **0m** was prepared according to General Procedure **B** using 3-chloro-1-(1-phenylcyclopentyl) propan-1-one (prepared from 1-phenylcyclopentanecarbonitrile as described in **0d**) (300 mg, 1.26 mmol) and vinylmagnesium bromide (2.1 mL, 1.5 mmol). Purification by flash chromatography [1% EA/Hexanes] afforded **0m** [250 mg, 75%] as a clear liquid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42 (dt, *J* = 8.6, 1.8 Hz, 2H), 7.35 – 7.31 (m, 2H), 7.26 – 7.22 (m, 1H), 5.81 (dd, *J* = 17.2, 11.0 Hz, 1H), 5.20 (ddd, *J* = 13.6, 9.2, 1.4 Hz, 2H), 3.49 – 3.42 (m, 1H), 3.36 (td, *J* = 10.5, 5.4 Hz, 1H), 2.27 – 2.14 (m, 2H), 2.04 (dt, *J* = 12.9, 8.7 Hz, 1H), 2.01 – 1.89 (m, 3H), 1.68 – 1.59 (m, 3H), 1.45 – 1.32 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.80, 139.61, 129.05, 127.88, 126.45, 114.81, 78.24, 59.60, 41.08, 39.30, 34.23, 32.83, 32.81, 23.77, 23.69, 23.66; **IR** (neat) ν_{max}/cm^{-1}3555, 3551, 3518, 3442, 3091, 3025, 2965, 2959, 2874, 1700, 1250, 1186, 928, 907, 735, 704; **HRMS** [ESI] 264.1280, 266.1244 $[(M)^+$; calcd for $C_{16}H_{21}ClO$ 264.1281, 266.1251]

2-(1-phenylcyclopentyl)-2-vinyloxetane (1m)

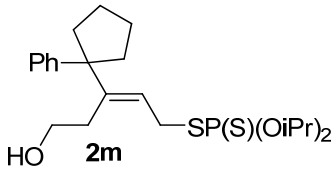


1m

Oxetane **1m** was prepared according to General Procedure **B** using alcohol 0m (45 mg, 0.17 mmol) and NaH (60% suspension in mineral oil, 13 mg, 0.34 mmol). Purification by flash chromatography [0.5:0.5:99 EA/TEA/Hexanes] afforded **1m** [36 mg, 93%] as a clear liquid.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.40 – 7.36 (m, 2H), 7.30 – 7.26 (m, 2H), 7.21 – 7.17 (m, 1H), 5.68 (dd, *J* = 17.0, 10.7 Hz, 1H), 5.26 (dd, *J* = 17.0, 2.0 Hz, 1H), 5.04 (dd, *J* = 10.7, 2.0 Hz, 1H), 4.40 (ddd, *J* = 8.3, 7.8, 5.6 Hz, 1H), 4.28 (dt, *J* = 9.3, 5.5 Hz, 1H), 2.93 (ddd, *J* = 10.9, 9.3, 7.7 Hz, 1H), 2.28 – 2.14 (m, 3H), 2.10 – 1.93 (m, 2H), 1.81 – 1.63 (m, 2H), 1.55 – 1.47 (m, 2H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 143.89, 140.47, 127.99, 127.71, 125.99, 112.64, 109.98, 91.10, 64.22, 57.67, 33.06, 31.28, 29.87, 24.13, 24.05; **IR** (neat) ν_{max}/cm^{-1}3419, 3413, 2960, 2875, 1250, 1231, 1035, 994, 968, 922; **HRMS** [ESI] 228.1514 [$(M)^+$; calcd for $C_{16}H_{20}O$ 228.1514]

(5-hydroxy-3-(1-phenylcyclopentyl)pent-2-en-1-yl) *O,O*-diisopropyl phosphorodithioate (2m)

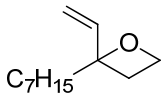


2m

Compound **2m** was prepared according to General Procedure C using oxetane **1m** (20 mg, 0.088 mmol) and acid **A_3** (22 mg, 0.11 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2m** [37 mg, 95%] as a clear liquid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 (s, 2H), 7.27 (s, 2H), 7.18 (hept, *J* = 3.7 Hz, 1H), 5.77 (t, *J* = 8.0 Hz, 1H), 4.85 (dhept, *J* = 12.4, 6.2 Hz, 2H), 3.65 (dd, *J* = 12.9, 8.0 Hz, 2H), 3.13 (t, *J* = 7.6 Hz, 2H), 2.28 – 2.20 (m, 2H), 2.06 – 1.99 (m, 2H), 1.99 – 1.93 (m, 2H), 1.73 – 1.64 (m, 2H), 1.64 – 1.53 (m, 3H), 1.37 (d, *J* = 6.2 Hz, 12H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.63, 145.34, 128.12, 126.97, 126.15, 120.31, 120.23, 73.46, 73.40, 62.13, 57.93, 36.25, 33.27, 32.21, 23.79, 23.75, 23.55, 23.49, 22.40; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.48; **IR** (neat) ν_{max}/cm^{-1}3419, 3058, 3021, 2976, 2959, 1492, 1466, 1454, 1444, 1384, 1378, 1176, 1141, 1103, 1023, 989, 962, 888, 776, 702, 651; **HRMS** [ESI] 442.1760 [$(M)^+$; calcd for $C_{22}H_{35}O_3PS_2$ 442.1765]

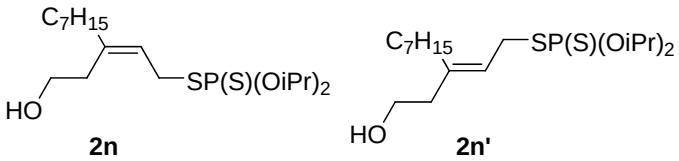
2-heptyl-2-vinyloxetane (1n)



1n

For the preparation of oxetane 1n, please see reference 2.

(3-(2-hydroxyethyl)dec-2-en-1-yl) *O,O*-diisopropyl phosphorodithioate (2n)

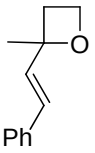


Compound **2n** was prepared according to General Procedure **C** using oxetane **1n** (18 mg, 0.045 mmol) and acid $\mathbf{A_3}$ (11 mg, 0.054 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2n** + **2n'** [14 mg, 80%] as a clear liquid.

2n: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 5.46 (t, *J* = 8.1 Hz, 1H), 4.83 (dhept, *J* = 12.4, 6.2 Hz, 2H), 3.69 (t, *J* = 6.7 Hz, 2H), 3.60 (dd, *J* = 13.1, 8.1 Hz, 2H), 2.39 (t, *J* = 6.7 Hz, 2H), 2.05 – 2.00 (m, 2H), 1.40 (dd, *J* = 14.7, 7.4 Hz, 3H), 1.38 – 1.34 (m, 12H), 1.33 – 1.21 (m, 10H), 0.88 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 141.12, 121.75, 121.69, 73.46, 73.41, 60.92, 36.89, 33.67, 31.84, 31.65, 31.62, 29.34, 29.20, 27.99, 23.83, 23.80, 23.56, 23.52, 22.69, 14.13; **^{31}P NMR** (202 MHz, $CDCl_3$) δ 100.00; **IR** (neat) ν_{max}/cm^{-1}3421, 3405, 3054, 2998, 2967, 1499, 1480, 1454, 1444, 1321, 1122, 1100 , 989, 960, 878, 773, 700, 650; **HRMS** [ESI] 396.1921 [$(M)^+$; calcd for $C_{18}H_{37}O_3PS_2$ 396.1922]

2n': **^{1}H NMR** (600 MHz, $CDCl_3$) δ 5.39 (t, *J* = 7.8 Hz, 1H), 4.84 (tt, *J* = 12.3, 6.2 Hz, 2H), 3.68 (t, *J* = 6.3 Hz, 2H), 3.56 (dd, *J* = 13.3, 7.9 Hz, 2H), 2.29 (t, *J* = 6.3 Hz, 2H), 2.12 – 2.07 (m, 2H), 1.39 (s, 3H), 1.36 (dd, *J* = 6.2, 1.7 Hz, 12H), 1.28 (dd, *J* = 13.8, 7.5 Hz, 10H), 0.91 – 0.86 (m, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 141.41, 121.87, 121.82, 73.40, 73.34, 60.47, 39.84, 31.80, 31.28, 31.24, 30.21, 29.71, 29.17, 28.55, 23.78, 23.74, 23.49, 23.45, 22.64, 14.09; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.27

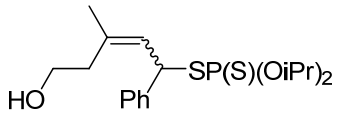
(*E*)-2-methyl-2-styryloxetane (1o)



1o

For the preparation of oxetane 1o, please see reference 2.

(5-hydroxy-3-methyl-1-phenylpent-2-en-1-yl) O,O-diisopropyl phosphorodithioate (2o)

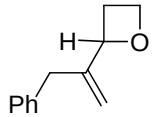


2o

Compound **2o** was prepared according to General Procedure **C** using oxetane 1o (33 mg, 0.19 mmol) and acid $\mathbf{A_3}$ (48 mg, 0.22 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2o** [45 mg, 65%] as a clear liquid. The E/Z isomers were inseparable and characterized as a mixture.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.43 – 7.39 (m, 2H), 7.33 – 7.29 (m, 2H), 7.25 – 7.21 (m, 1H), 5.73 – 5.67 (m, 1H), 5.42 – 5.29 (m, 1H), 4.91 – 4.55 (m, 2H), 3.81 – 3.65 (m, 2H), 2.62 – 2.24 (m, 2H), 1.85 – 1.78 (m, 3H), 1.32 (m ,3H), 1.25 (m,, 4H), 1.16 (m,, 6H); **^{13}C NMR** (126 MHz, CDCl3) δ 141.64, 141.52, 137.49, 136.80, 130.57, 129.73, 129.65, 128.58, 128.42, 128.39, 128.35, 126.04, 125.93, 73.70, 73.63, 73.55, 73.51, 73.48, 60.81, 60.35, 43.89, 43.86, 43.39, 39.57, 38.37, 34.62, 34.46, 32.82, 24.25, 24.20, 23.80, 23.75, 23.72, 23.67, 23.63, 23.58, 23.53, 23.49, 23.45; **IR** (neat) ν_{max}/cm^{-1} 3556, 3503, 3486, 2957, 2941, 1469, 1465, 1455, 1453, 1342, 1235, 1102, 1037, 989, 966, 775, 702, 651;**HRMS** [ESI] 388.1290 [$(M)^+$; calcd for $C_{18}H_{29}O_3PS_2$ 388.1296].

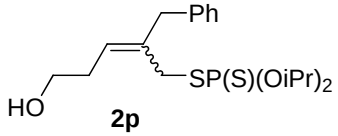
2-(3-phenylprop-1-en-2-yl)oxetane (1p)



1p

For the preparation of oxetane 1p, please see reference 2.

(2-benzyl-5-hydroxypent-2-en-1-yl) *O,O*-diisopropyl phosphorodithioate (2p)



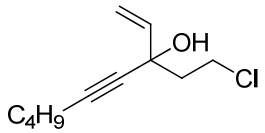
2p

Compound **2p** was prepared according to General Procedure **C** using oxetane **1p** (40 mg, 0.24 mmol) and acid $\mathbf{A_3}$ (60 mg, 0.27 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2p** (42 mg, 48%) as a clear liquid.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.23 – 7.17 (m, 3H), 5.57 (t, *J* =7.4 Hz, 1H), 4.85 – 4.76 (m, 2H), 3.71 (dt, *J* = 16.9, 6.4 Hz, 2H), 3.56 (s, 1H), 3.48 (ddd, *J* = 14.6, 11.8, 6.0 Hz, 3H), 2.45 (td, *J* = 13.8, 6.5 Hz, 2H), 1.35 – 1.29 (m, 12H);**^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.91, 138.75, 136.18, 129.06, 128.62, 128.56,

128.45, 127.61, 127.38, 126.41, 126.34, 73.55, 73.48, 73.42, 73.35, 62.20, 62.15, 42.66, 40.07, 34.76, 32.83, 31.82, 29.69, 23.74, 23.69, 23.47, 23.45, 23.39;**^{31}P NMR** (162 MHz, $CDCl_3$) δ 91.53; **IR** (neat) ν_{max}/cm^{-1}3433, 3425, 3067, 2993, 2966, 1489, 1484, 1452, 1434, 1122, 1100 , 996, 987, 878, 771, 745, 650; **HRMS** [ESI] 388.1281 $[(M)^+$; calcd for $C_{18}H_{29}O_3PS_2$ 388.1296]

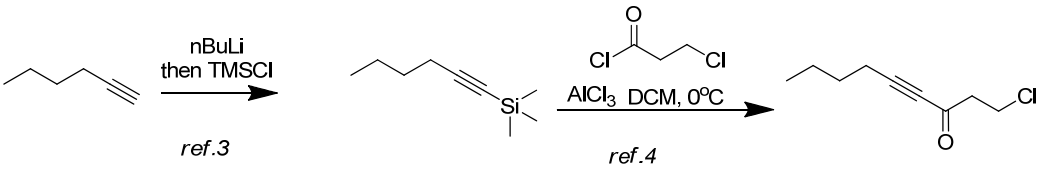
3-(2-chloroethyl)non-1-en-4-yn-3-ol (0q)



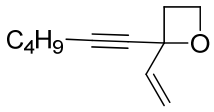
0q

Alcohol **0q** was prepared according to General Procedure **B** using 1-chlorononon-4-yn-3-one (prepared according to known procedure) (153 mg, 0.87 mmol) and vinylmagnesium bromide (1.7 mL, 0.7M, 1.22 mmol). Purification by flash chromatography [1% EA/Hexanes] afforded **0q** (128 mg, 74%) as a clear liquid.

^{1}H NMR (499 MHz, $CDCl_3$) δ 5.90 (dd, *J* = 17.0, 10.2 Hz, 1H), 5.53 (ddd, *J* = 17.0, 2.8, 1.1 Hz, 1H), 5.18 (dd, *J* = 10.2, 1.1 Hz, 1H), 3.74 – 3.62 (m, 2H), 2.28 – 2.10 (m, 4H), 1.54 – 1.46 (m, 2H), 1.46 – 1.35 (m, 2H), 0.92 (dd, *J* = 8.0, 6.6 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.89, 114.87, 87.81, 79.68, 70.52, 44.96, 40.13, 30.63, 21.95, 18.31, 13.55; **IR** (neat) ν_{max}/cm^{-1}3508, 3501, 3418, 3406, 3333, 3323, 2959, 2933, 1275, 1173, 1052, 986, 932, 689; **HRMS** [ESI] 200.0959, 202.0937 $[(M)^+$; calcd for $C_{11}H_{17}ClO$ 200.0968, 202.0938]



2-(hex-1-yn-1-yl)-2-vinyloxetane (1q)

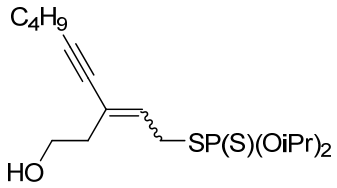


1q

Oxetane **1q** was prepared according to General Procedure **B** using alcohol 0q (70 mg, 0.35 mmol) and NaH (60% suspension in mineral oil, 28 mg, 0.7 mmol). Purification by flash chromatography [0.5:0.5:99 EA/TEA/Hexanes] afforded **1q** (56 mg, 98%) as a clear liquid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.16 (dd, *J* = 16.9, 10.2 Hz, 1H), 5.54 (dd, *J* = 16.9, 1.3 Hz, 1H), 5.30 – 5.14 (m, 1H), 4.63 (ddd, *J* = 8.7, 7.0, 5.8 Hz, 1H), 4.46 (ddd, *J* = 8.7, 6.7, 5.8 Hz, 1H), 2.93 (ddd, *J* = 10.9, 8.7, 6.7 Hz, 1H), 2.74 (ddd, *J* = 10.9, 8.7, 7.0 Hz, 1H), 2.35 – 2.25 (m, 2H), 1.59 – 1.50 (m, 2H), 1.50 – 1.36 (m, 2H), 0.92 (t, *J* = 7.2 Hz, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.75, 114.61, 89.81, 80.09, 79.55, 65.39, 35.88, 30.61, 21.95, 18.62, 13.57; **IR** (neat) ν_{max}/cm^{-1}2958, 2932, 2886, 1598, 1588, 1274, 1228, 1183, 1033, 984, 959, 929; **HRMS** [ESI] 124.1200 [$(M)^+$; calcd for $C_{11}H_{16}O$ 124.1201]

(3-(2-hydroxyethyl)non-2-en-4-yn-1-yl) *O,O*-diisopropyl phosphorodithioate (2q)

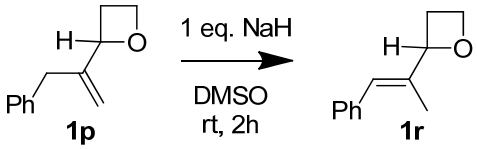


2q

Compound **2q** was prepared according to General Procedure C using oxetane 1q (25 mg, 0.15 mmol) and acid **A_3** (39 mg, 0.18 mmol). Purification by flash chromatography [30% EA/Hexanes] afforded **2q** [48 mg, 85%] as a clear liquid. The E/Z mixture was inseparable and characterized as a mixture.

^{1}H NMR (600 MHz, $CDCl_3$) δ 5.82 (t, *J* = 7.7 Hz, 1H), 4.84 (tq, *J* = 12.4, 6.2 Hz, 3H), 3.80 – 3.73 (m, 4H), 2.39 – 2.34 (m, 5H), 1.57 – 1.53 (m, 2H), 1.47 – 1.41 (m, 2H), 1.37 – 1.36 (m, 12H), 0.97 – 0.90 (m, 3H);**^{13}C NMR** (126 MHz, $CDCl_3$) δ 132.11, 132.06, 123.81, 98.27, 73.43, 73.38, 60.96, 40.50, 33.27, 33.24, 30.75, 29.69, 23.78, 23.74, 23.47, 23.43, 22.02, 19.25, 13.59; **^{31}P NMR** (162 MHz, $CDCl_3$) δ 90.86; **IR** (neat) ν_{max}/cm^{-1}3441, 3418, 3406, 2976, 2958, 2931, 1466, 1458, 1384, 1373, 1178, 1141, 1102, 989, 970, 889, 777, 652; **HRMS** [ESI] 378.2450 [$(M)^+$; calcd for $C_{17}H_{31}O_3PS_2$ 378.1452]

(*E*)-2-(1-phenylprop-1-en-2-yl) oxetane (1r)

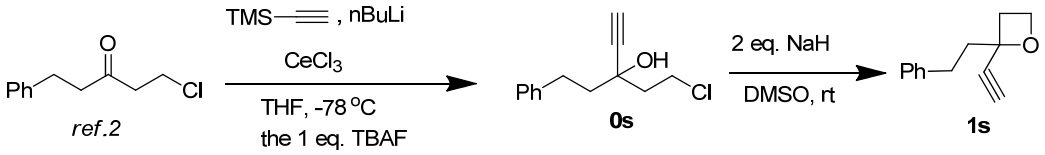


Oxetane **1r** was prepared from oxetane **1p** (15 mg, 0.086 mmol) and NaH (60% suspension in mineral oil, 3.4 mg, 0.086 mmol) in DMSO as shown above.

Purification by flash chromatography [1% EA/Hexanes] afforded **1r** (13.5 mg, 90%) as a clear liquid.

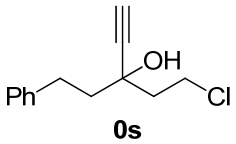
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 4H), 7.24 – 7.18 (m, 1H), 6.61 (s, 1H), 5.27 (t, *J* = 7.6 Hz, 1H), 4.73 (ddd, *J* = 8.3, 7.7, 5.9 Hz, 1H), 4.55 (ddd, *J* = 8.3, 7.6, 5.9 Hz, 1H), 2.89 – 2.79 (m, 1H), 2.62 – 2.51 (m, 1H), 1.94 – 1.90 (m, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.61, 137.47, 128.96, 128.12, 126.45, 124.51, 85.66, 67.93, 27.48, 12.62; **IR** (neat) ν_{max}/cm^{-1}3058, 3024, 2962, 2932, 2879, 1449, 1440, 1250, 1228, 1035, 990, 968, 754, 747; **HRMS** [ESI] 174.1041 [$(M)^+$; calcd for $C_{12}H_{14}O$ 174.1045]

2-ethynyl-2-phenethyloxetane (1s)



Oxetane 1s was prepared from 1-chloro-5-phenylpentan-3-one as shown above.

5-chloro-3-phenethylpent-1-yn-3-ol (0s)

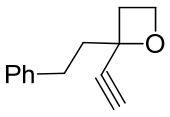


0s

Clear liquid, yield 72%.

^{1}H NMR (499 MHz, $CDCl_3$) δ 7.32 – 7.26 (m, 2H), 7.24 – 7.17 (m, 3H), 3.81 (m, 2H), 2.93 – 2.84 (m, 2H), 2.60 (s, 1H), 2.25 – 2.15 (m, 3H), 2.04 – 1.92 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.31, 128.55, 128.41, 126.10, 84.69, 74.15, 70.26, 44.31, 44.26, 40.20, 30.49; **IR** (neat) ν_{max}/cm^{-1} 3550, 3078, 3029, 2943, 2894, 1500, 1461, 1228, 1029, 991, 977, 750, 698; **HRMS** [ESI] 222.0810 [$(M)^+$; calcd for $C_{13}H_{15}ClO$ 222.0811]

2-ethynyl-2-phenethyloxetane (1s)



1s

Clear liquid, yield 88%

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 2H), 7.25 – 7.18 (m, 3H), 4.70 (ddd, *J* = 8.8, 7.2, 6.0 Hz, 1H), 4.51 – 4.46 (m, 1H), 2.87 – 2.82 (m, 2H), 2.82 (s, 1H), 2.77

(ddd, *J* = 13.8, 11.5, 5.6 Hz, 1H), 2.30 – 2.24 (m, 1H), 2.17 (ddd, *J* = 13.4, 11.7, 5.6 Hz, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.36, 128.43, 128.40, 125.96, 85.11, 80.05, 75.78, 66.03, 43.68, 34.16, 29.94; **IR** (neat) ν_{max}/cm^{-1} 2944, 2890, 1520, 1489, 1230, 1031, 987, 972, 754, 698; **HRMS** [ESI] 186.1040 [$(M)^+$; calcd for $C_{13}H_{14}O$ 186.1045]

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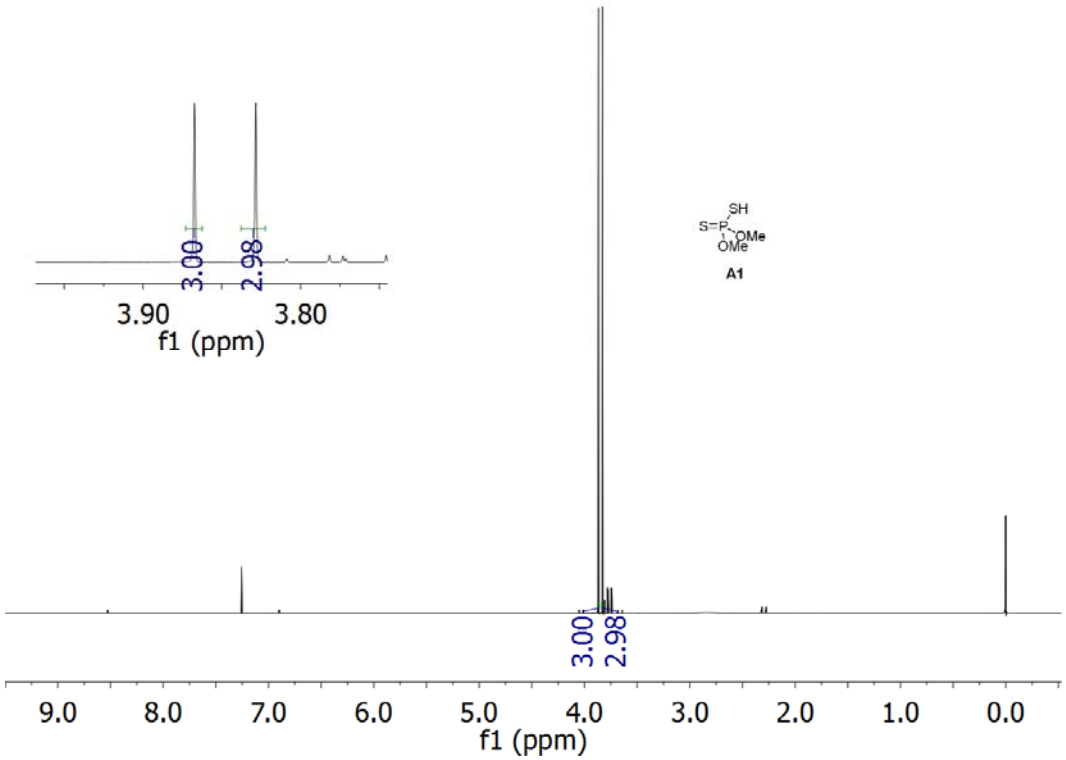
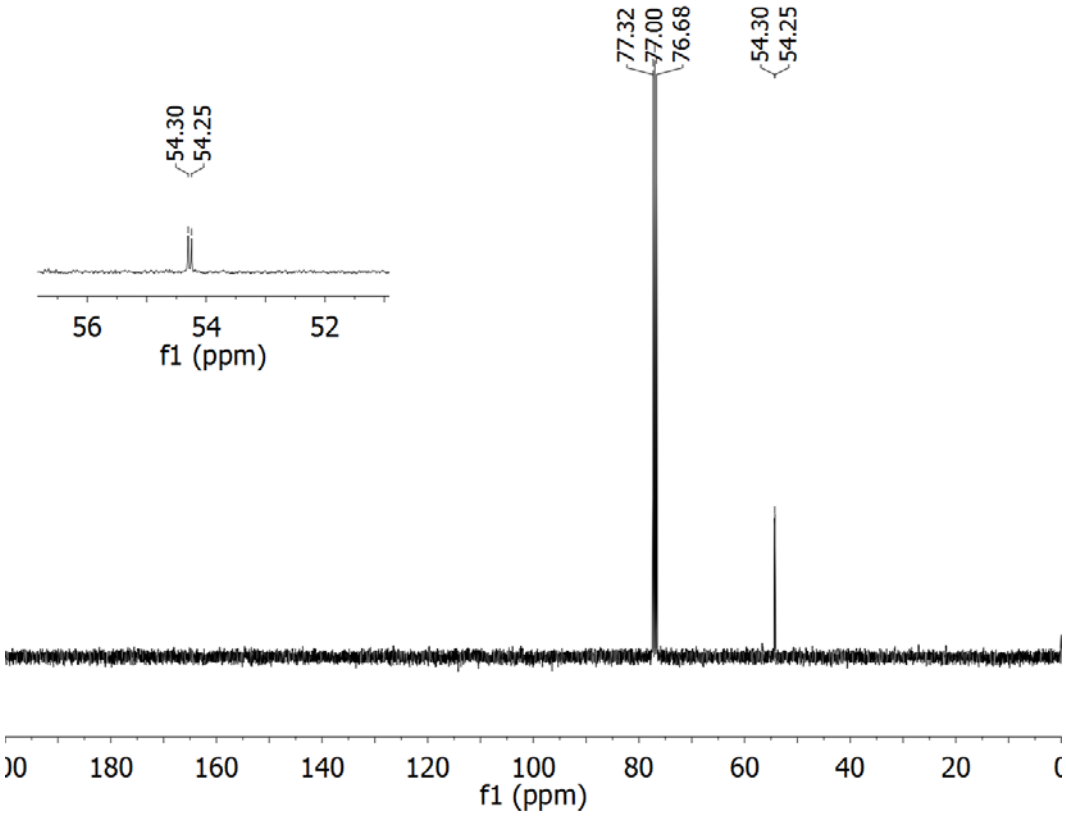
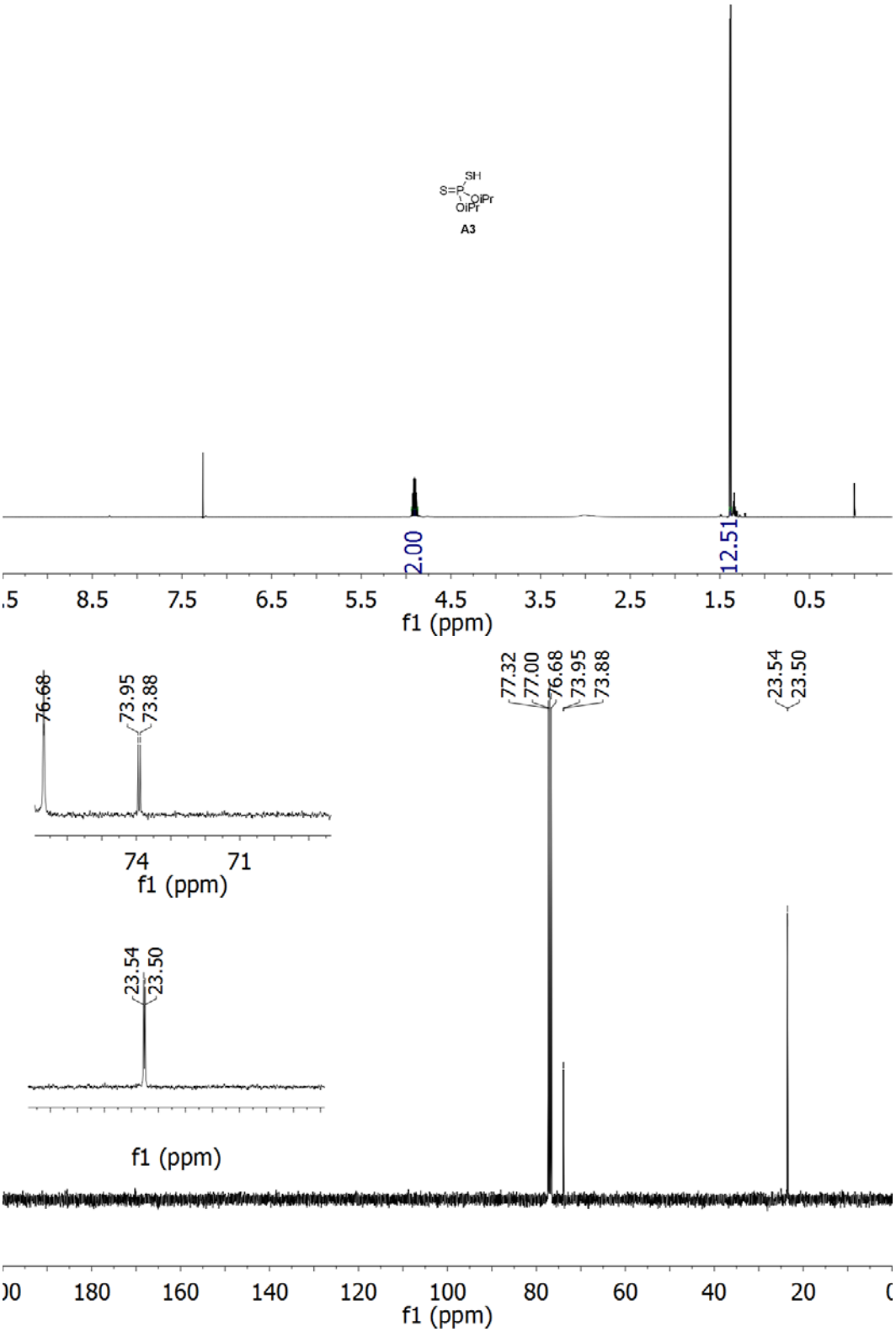
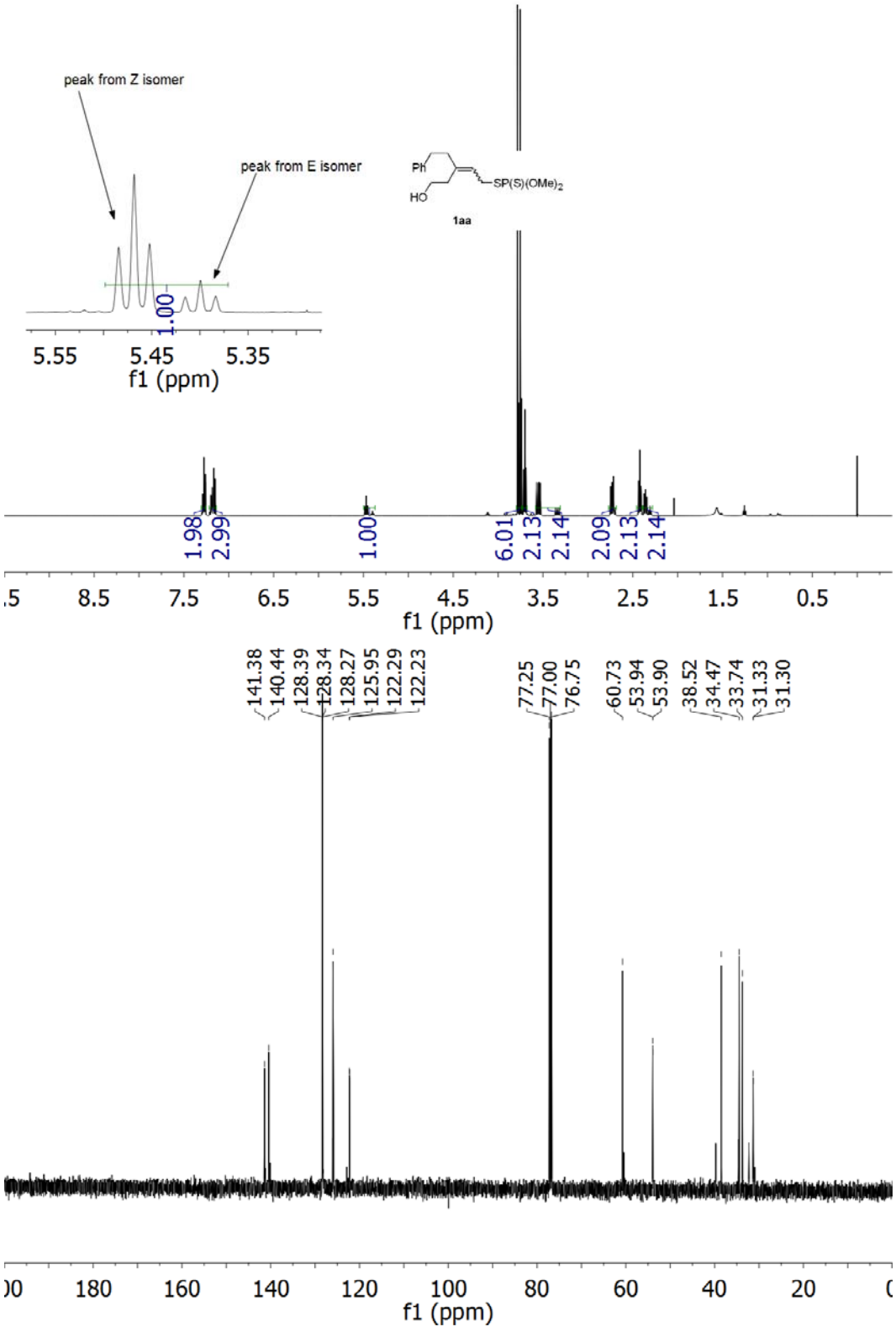
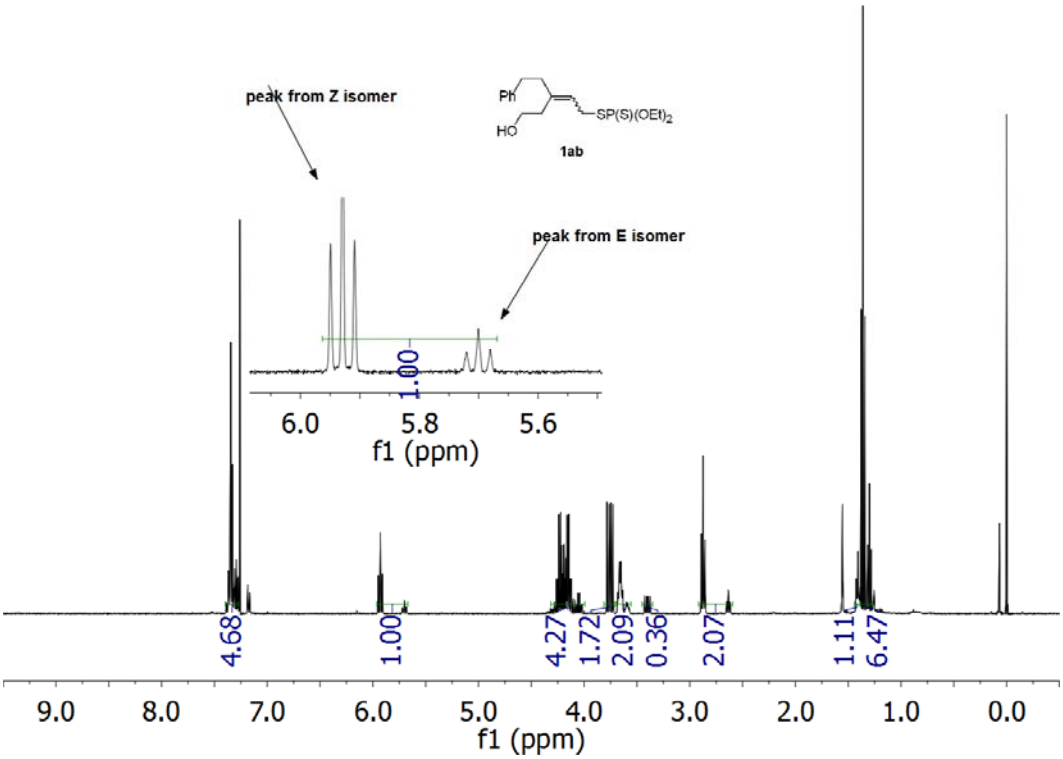
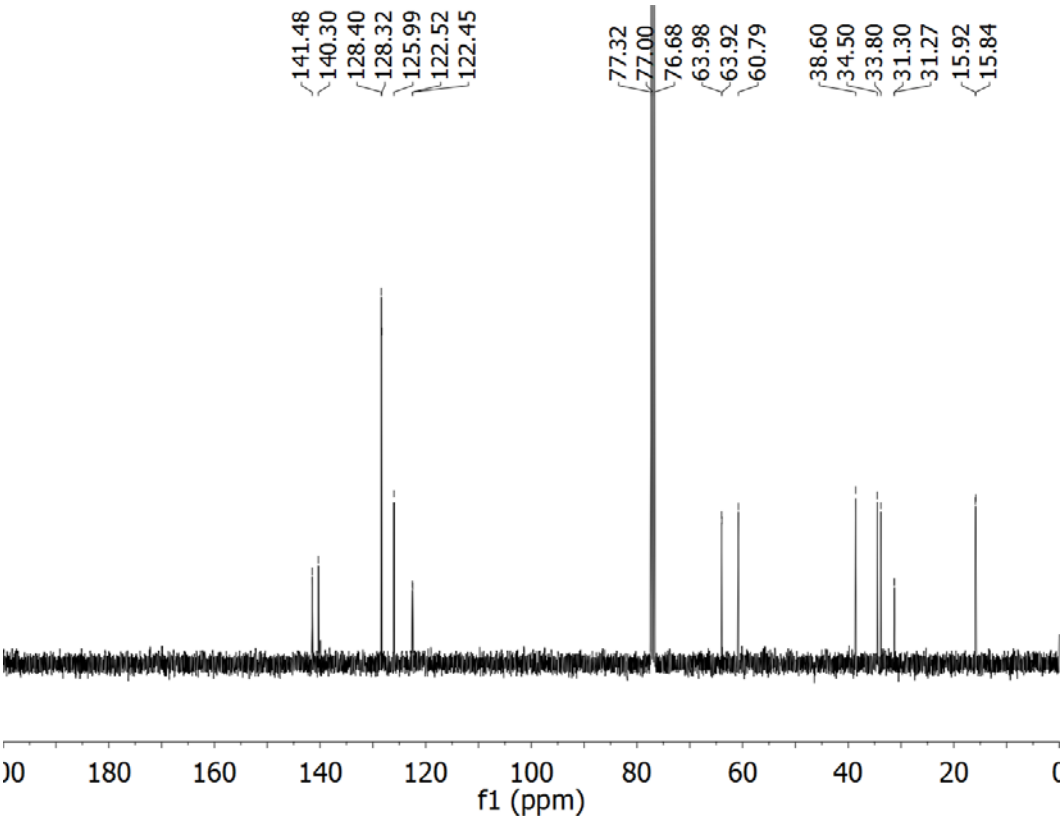

3.00
2.98
3.90
3.80
f1 (ppm)
SH
S=P
OMe
OMe
A1
3.00
2.98
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

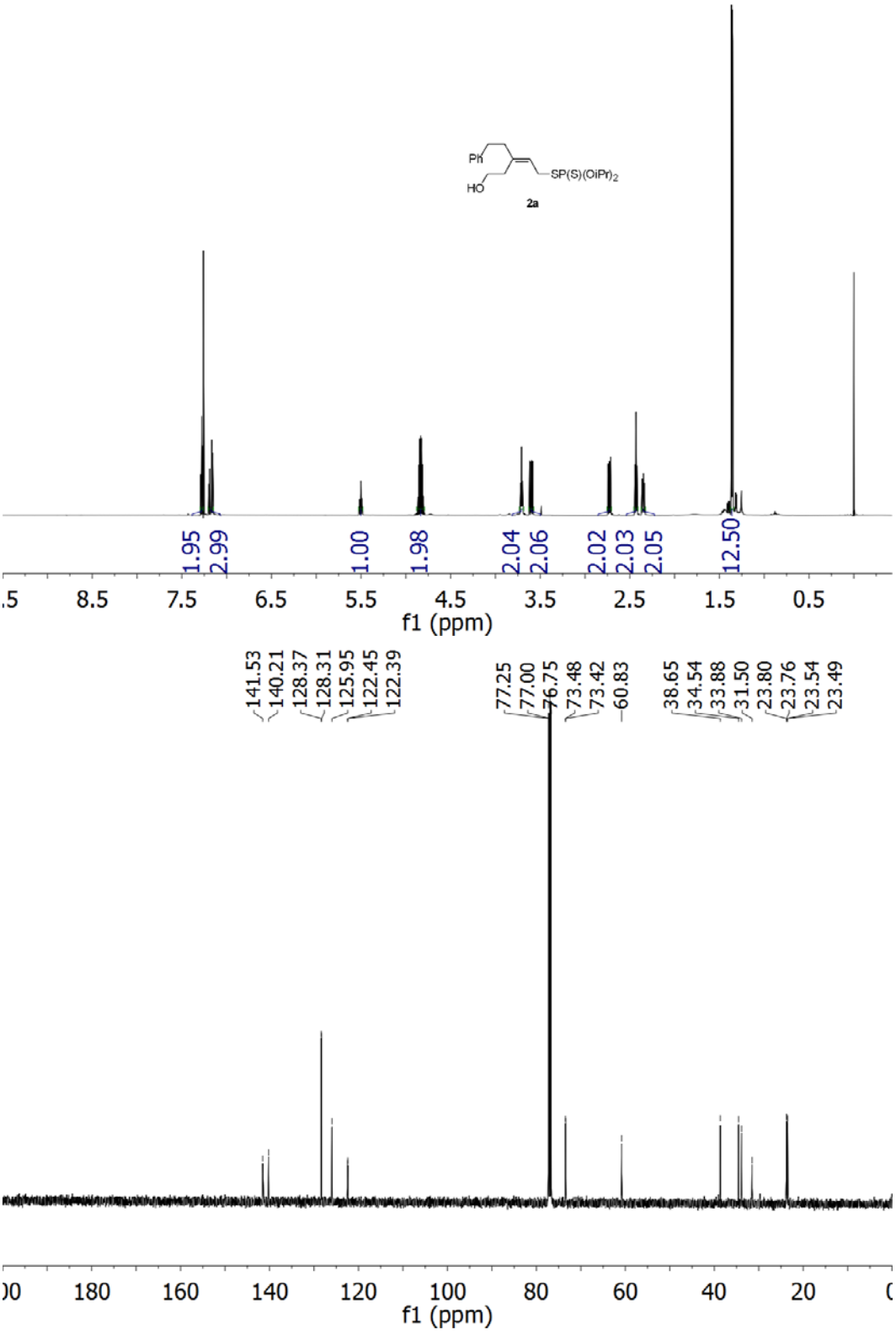

77.32
77.00
76.68
54.30
54.25
54.30
54.25
56
54
52
f1 (ppm)
180
160
140
120
100
80
60
40
20
f1 (ppm)

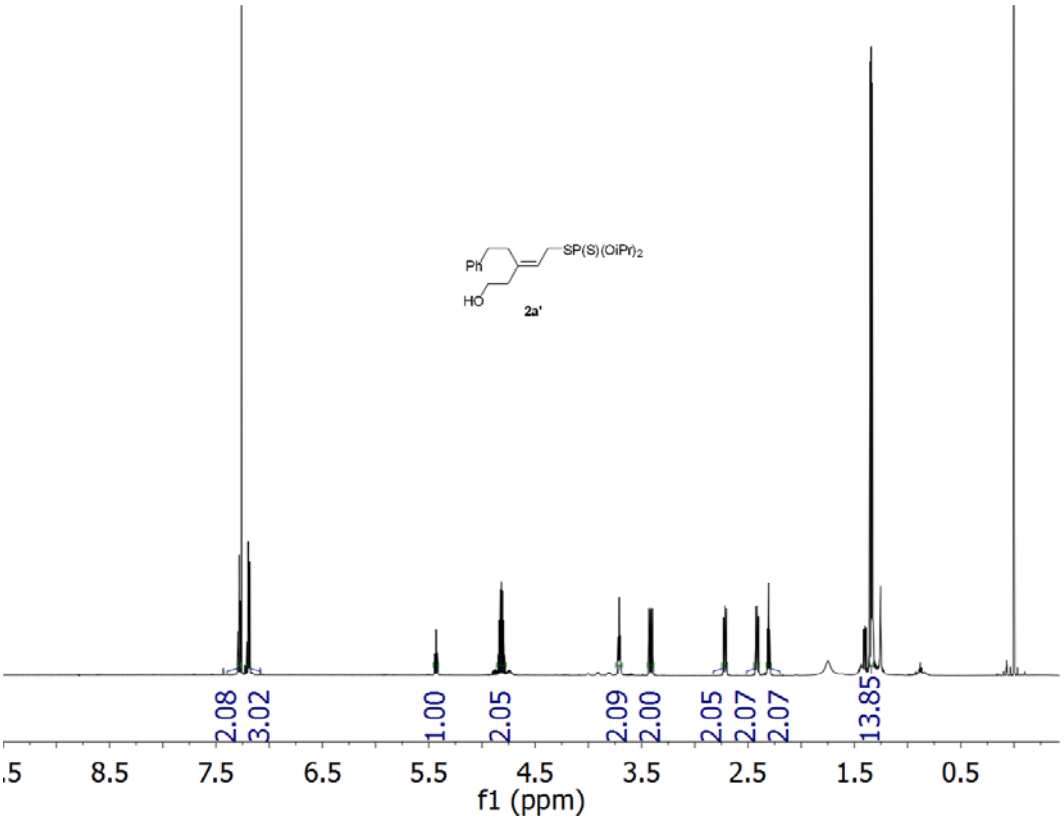

SH
S=P
OiPr
OiPr
A3
2.00
12.51
f1 (ppm)
76.68
73.95
73.88
74
71
f1 (ppm)
77.32
77.00
76.68
73.95
73.88
23.54
23.50
23.54
23.50
f1 (ppm)
f1 (ppm)

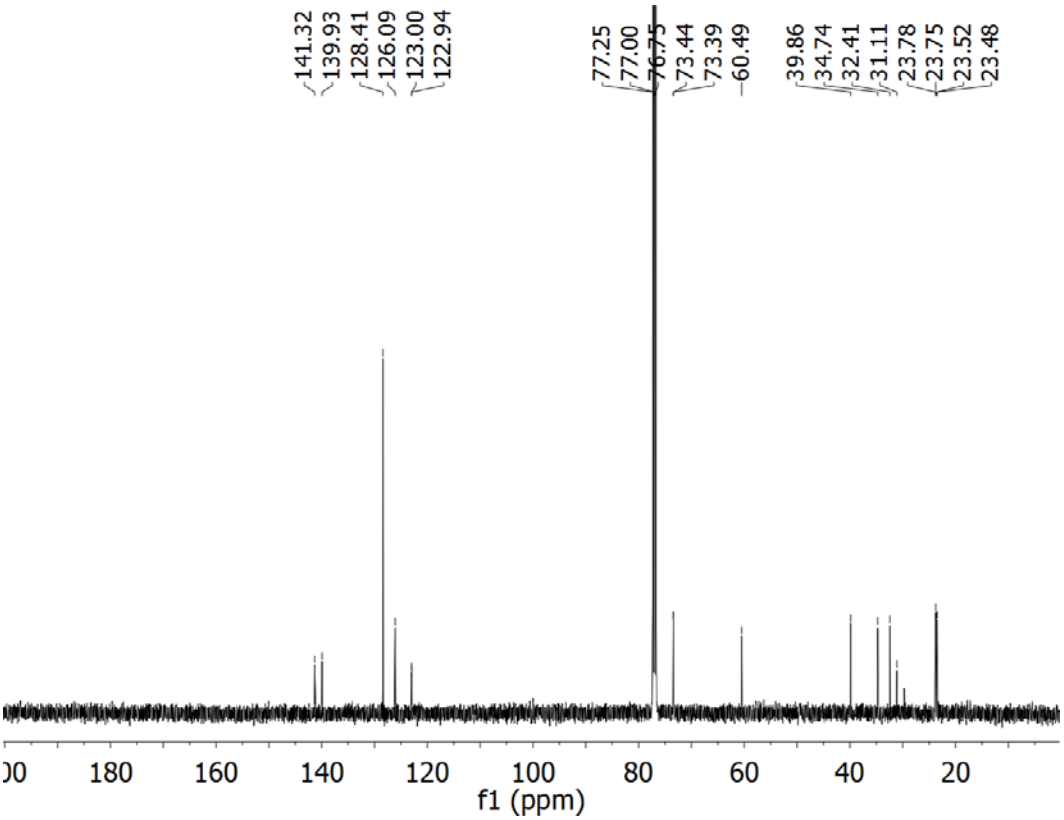

peak from Z isomer
peak from E isomer
1.00
5.55
5.45
5.35
f1 (ppm)
Ph
HO
SP(S)(OMe)2
1aa
1.98
2.99
1.00
6.01
2.13
2.14
2.09
2.13
2.14
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)
141.38
140.44
128.39
128.34
128.27
125.95
122.29
122.23
77.25
77.00
76.75
60.73
53.94
53.90
38.52
34.47
33.74
31.33
31.30
180
160
140
120
100
80
60
40
20
f1 (ppm)

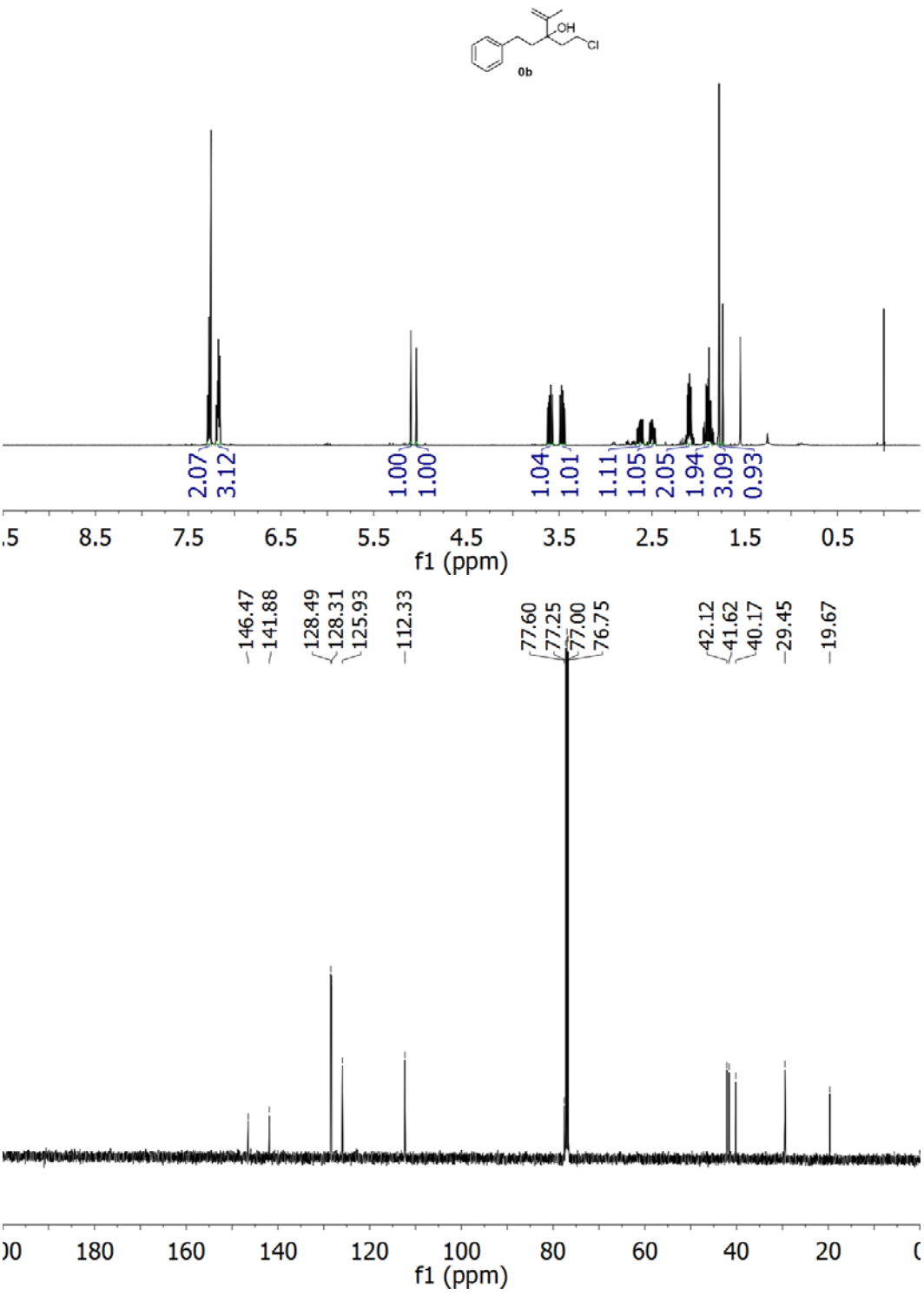

peak from Z isomer
peak from E isomer
Ph
HO
SP(S)(OEt)2
1ab
1.00
6.0
5.8
5.6
f1 (ppm)
4.68
1.00
4.27
1.72
2.09
0.36
2.07
1.11
6.47
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

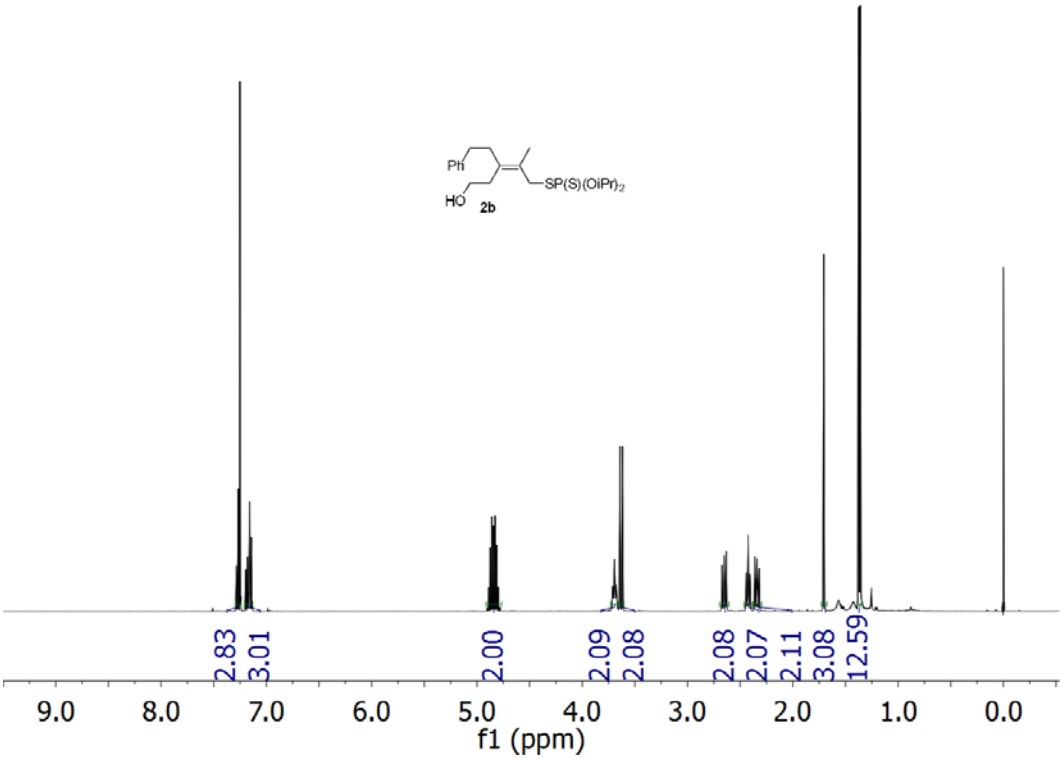

141.48
140.30
128.40
128.32
125.99
122.52
122.45
77.32
77.00
76.68
63.98
63.92
60.79
38.60
34.50
33.80
31.30
31.27
15.92
15.84
180
160
140
120
100
80
60
40
20
f1 (ppm)

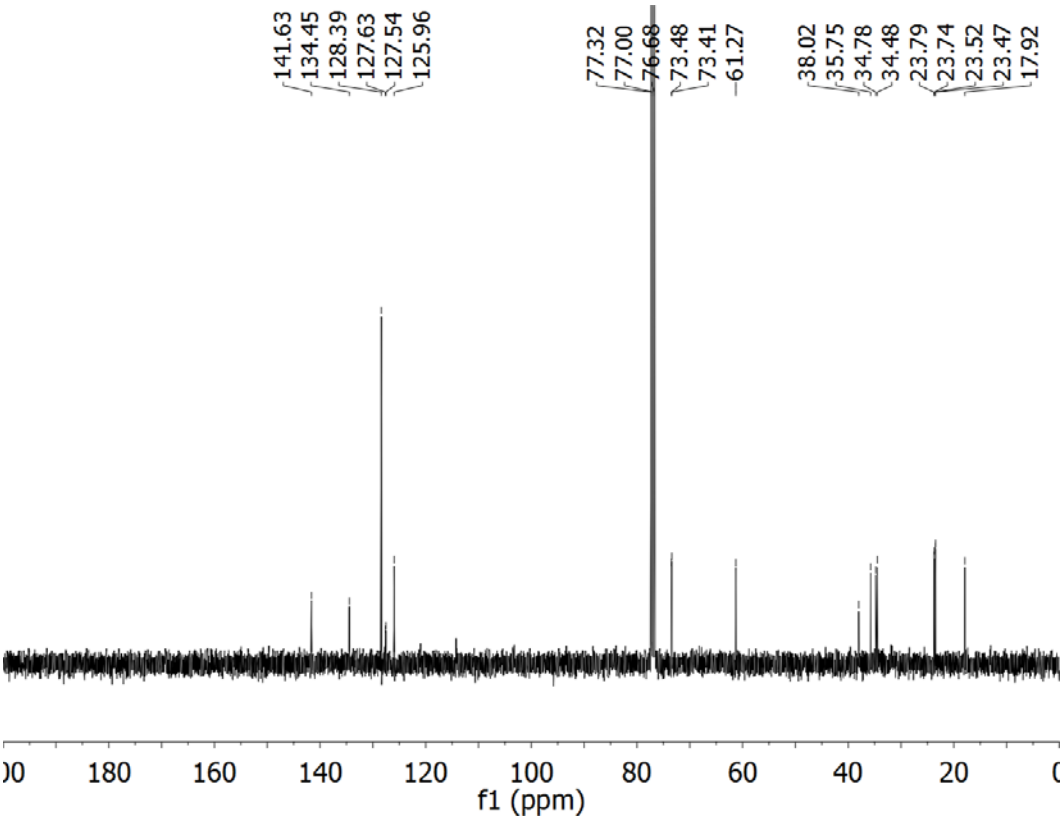

Ph
HO
SP(S)(OiPr)2
2a
1.95
2.99
1.00
1.98
2.04
2.06
2.02
2.03
2.05
12.50
f1 (ppm)
141.53
140.21
128.37
128.31
125.95
122.45
122.39
77.25
77.00
76.75
73.48
73.42
60.83
38.65
34.54
33.88
31.50
23.80
23.76
23.54
23.49
f1 (ppm)

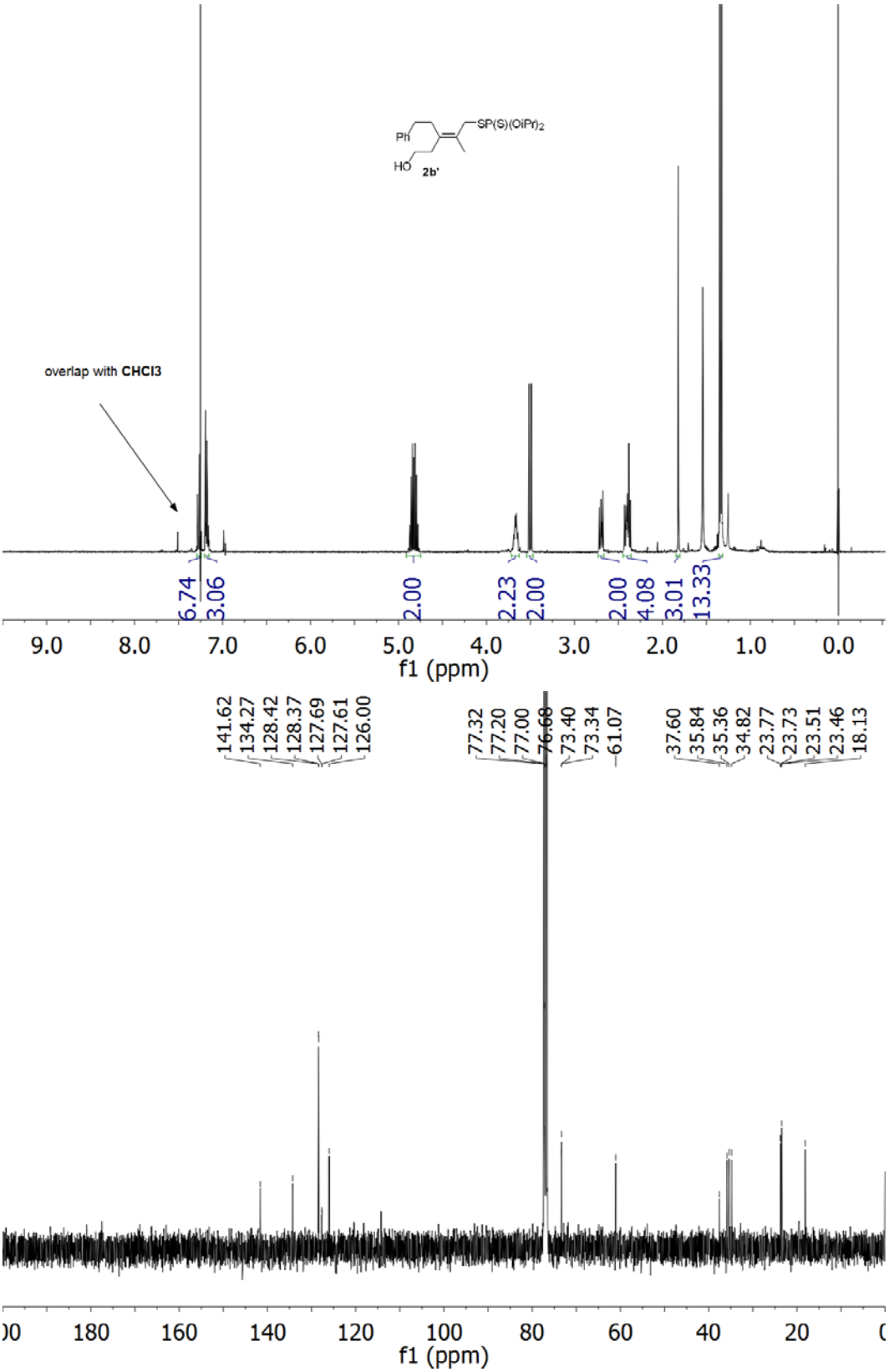

SP(S)(OiPr)2
Ph
HO
2a'
2.08
3.02
1.00
2.05
2.09
2.00
2.05
2.07
2.07
13.85
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

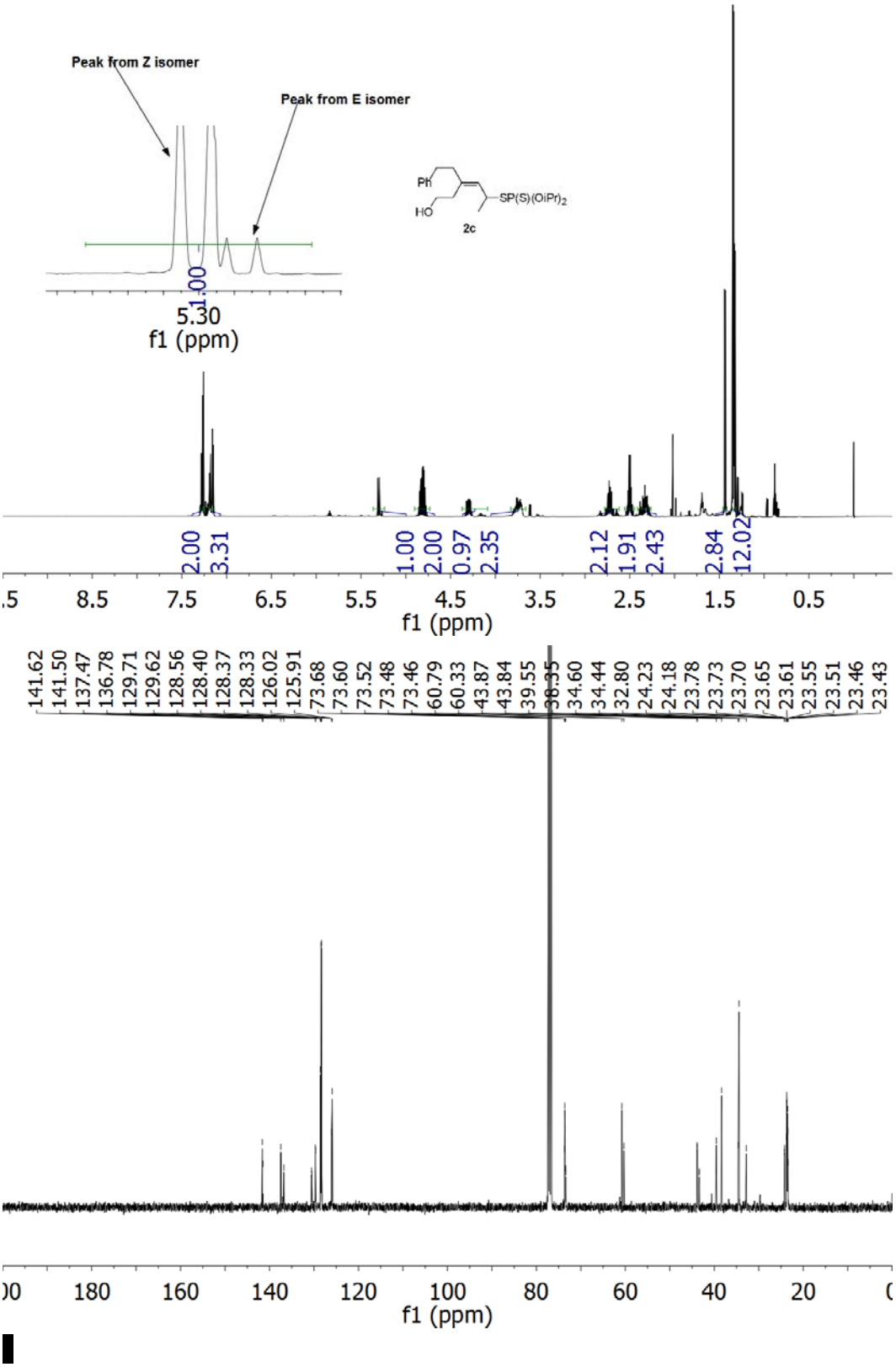

141.32
139.93
128.41
126.09
123.00
122.94
77.25
77.00
76.75
73.44
73.39
60.49
39.86
34.74
32.41
31.11
23.78
23.75
23.52
23.48
180
160
140
120
100
80
60
40
20
f1 (ppm)

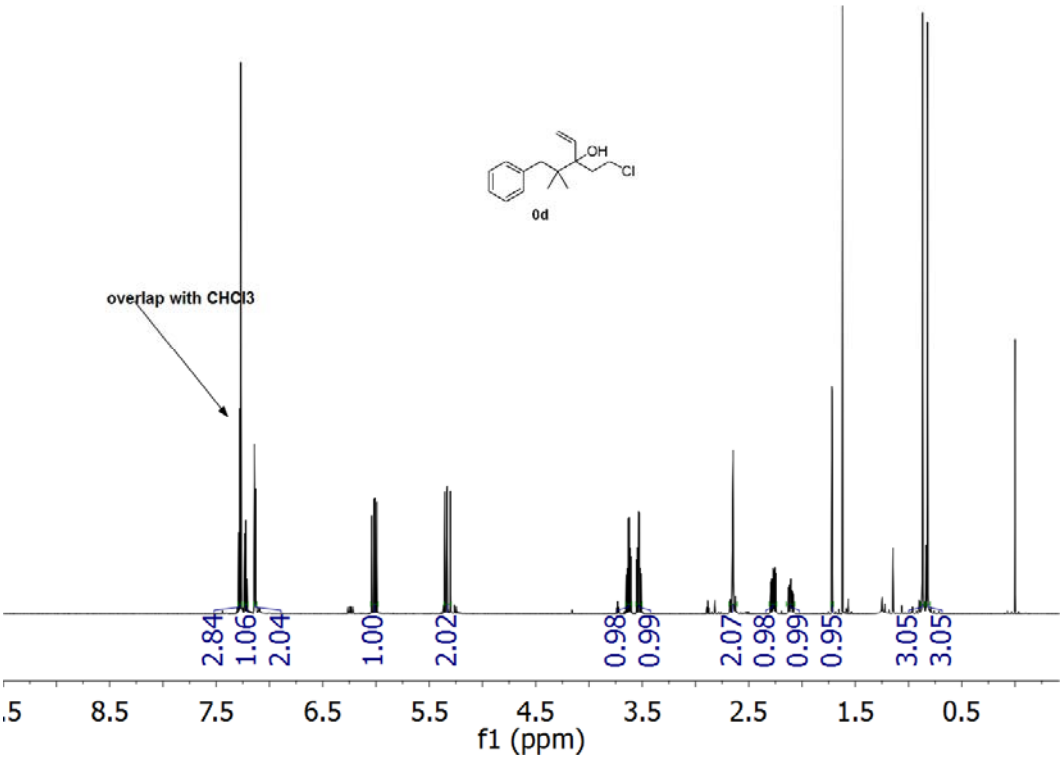


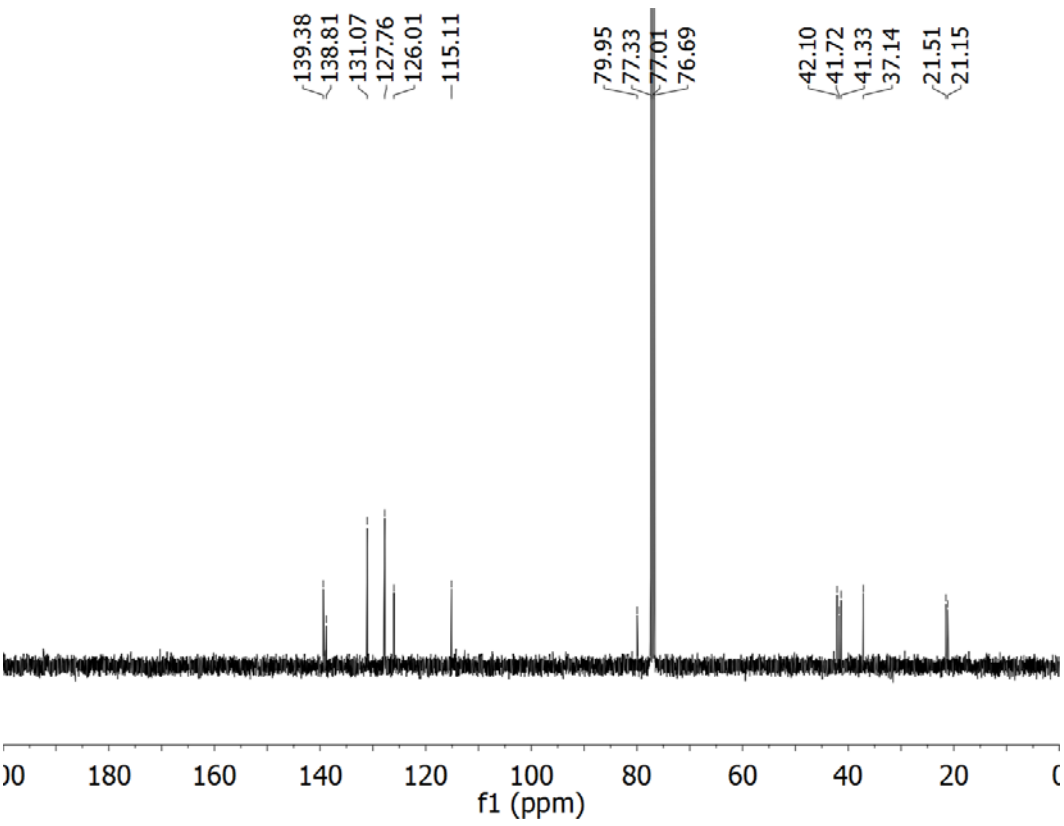

Ph
HO
SP(S)(OiPr)2
2b
2.83
3.01
2.00
2.09
2.08
2.08
2.07
2.11
3.08
12.59
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

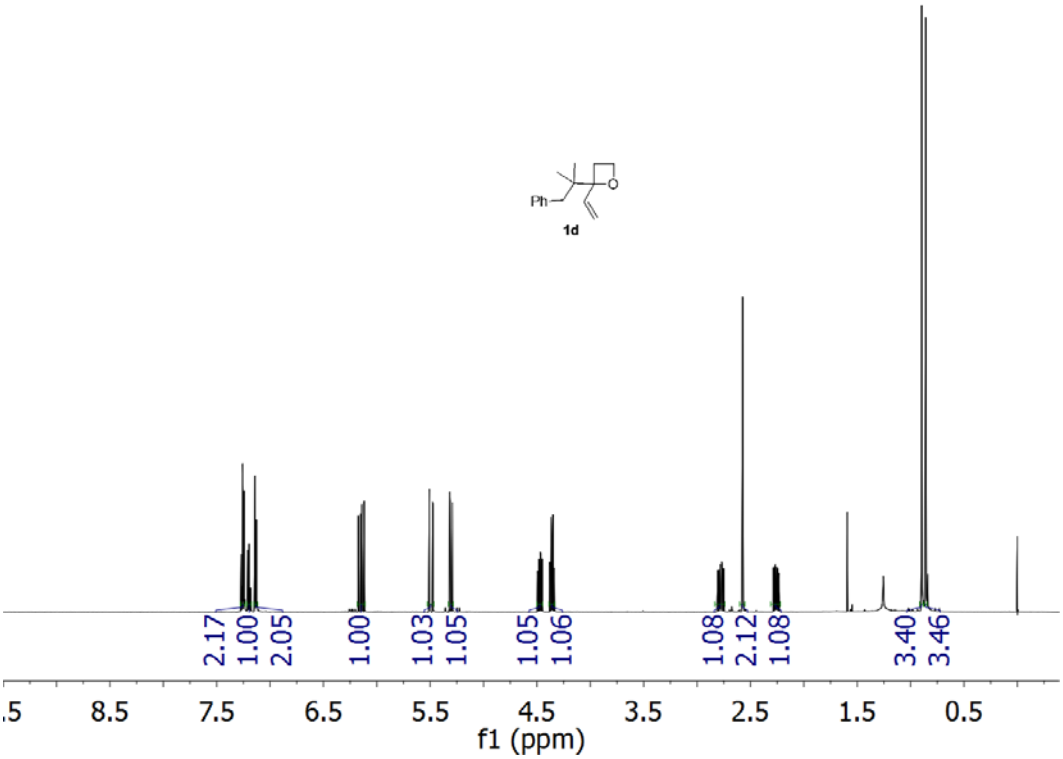

141.63
134.45
128.39
127.63
127.54
125.96
77.32
77.00
76.68
73.48
73.41
61.27
38.02
35.75
34.78
34.48
23.79
23.74
23.52
23.47
17.92
180
160
140
120
100
80
60
40
20
f1 (ppm)

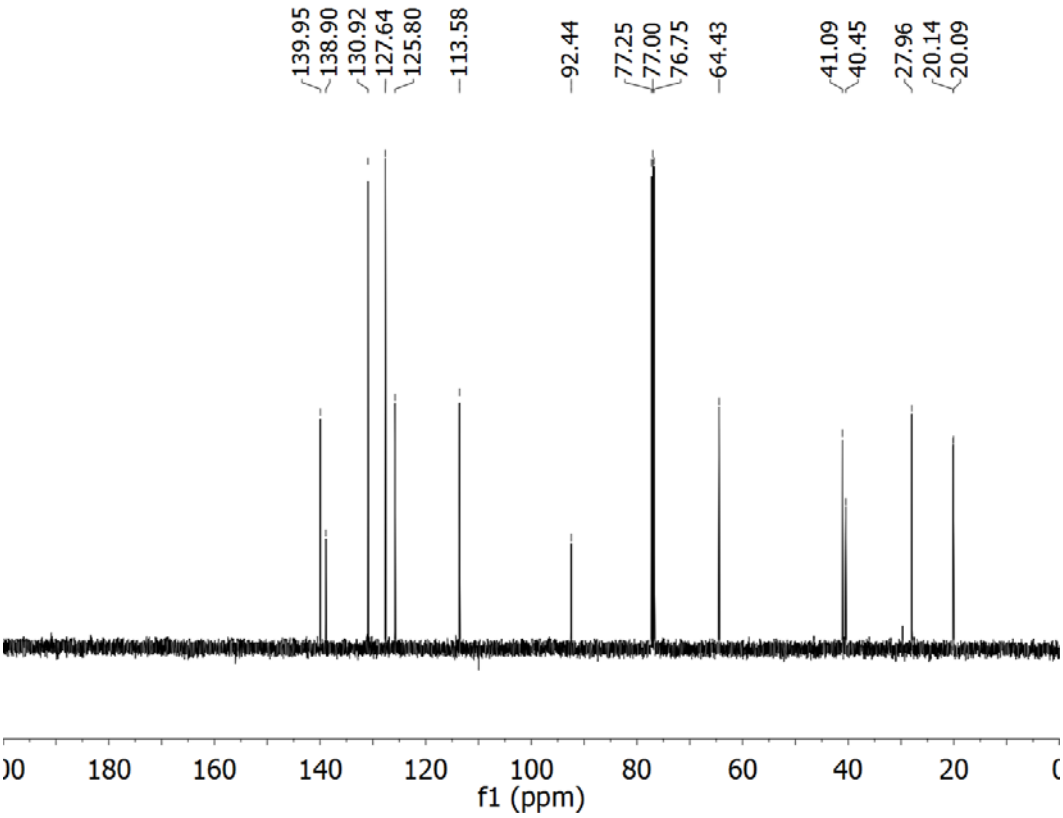

Ph
HO
SP(S)(OiPr)2
2b'
overlap with CHCl3
6.74
3.06
2.00
2.23
2.00
2.00
4.08
3.01
13.33
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)
141.62
134.27
128.42
128.37
127.69
127.61
126.00
77.32
77.20
77.00
76.68
73.40
73.34
61.07
37.60
35.84
35.36
34.82
23.77
23.73
23.51
23.46
18.13
180
160
140
120
100
80
60
40
20
f1 (ppm)

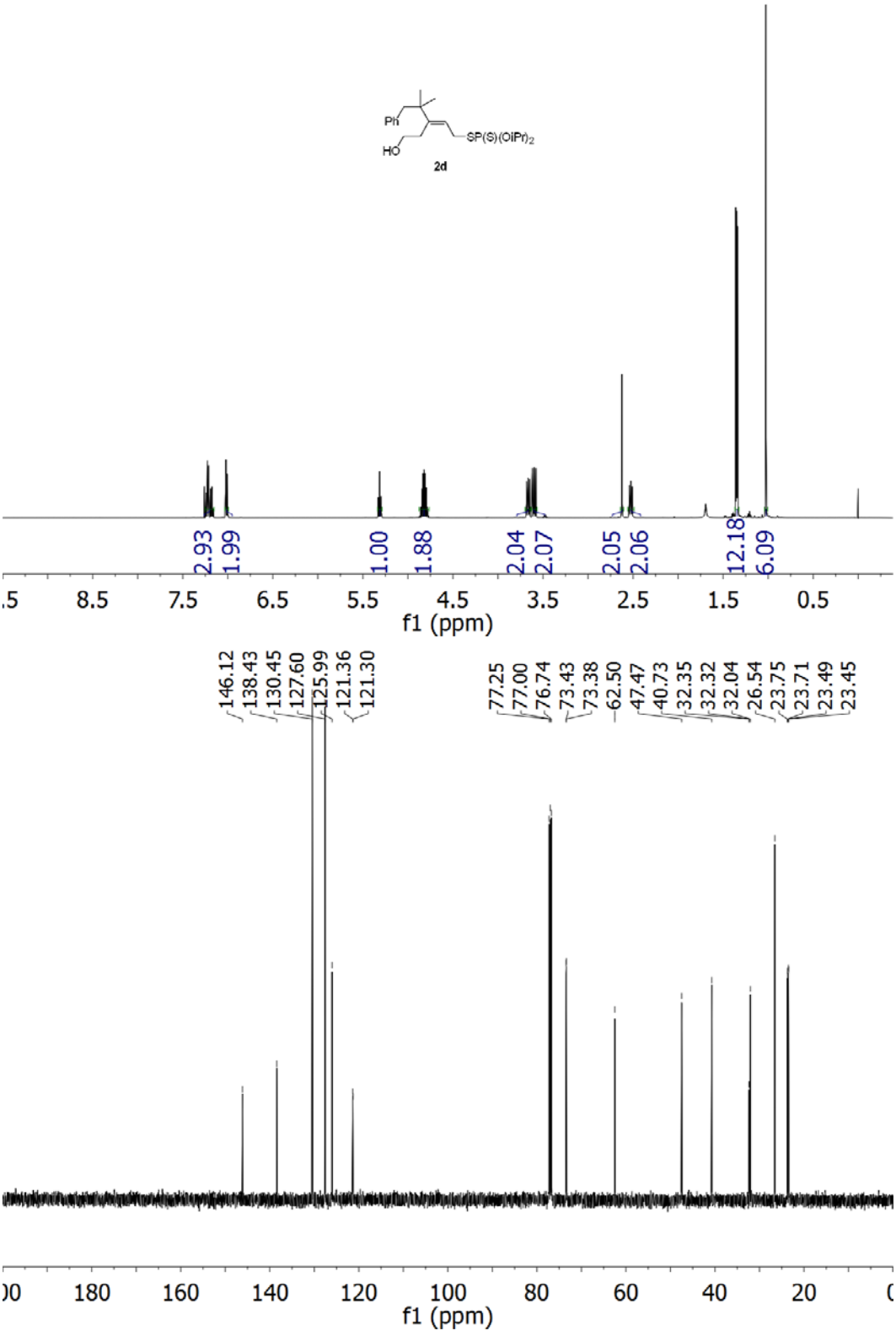

Peak from Z isomer
Peak from E isomer
Ph
HO
SP(S)(OiPr)2
2c
1.00
5.30
f1 (ppm)
2.00 3.31 1.00 2.00 0.97 2.35 2.12 1.91 2.43 2.84 12.02
8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5
f1 (ppm)
141.62 141.50 137.47 136.78 129.71 129.62 128.56 128.40 128.37 128.33 126.02 125.91 73.68 73.60 73.52 73.48 73.46 60.79 60.33 43.87 43.84 39.55 38.35 34.60 34.44 32.80 24.23 24.18 23.78 23.73 23.70 23.65 23.61 23.55 23.51 23.46 23.43
180 160 140 120 100 80 60 40 20
f1 (ppm)

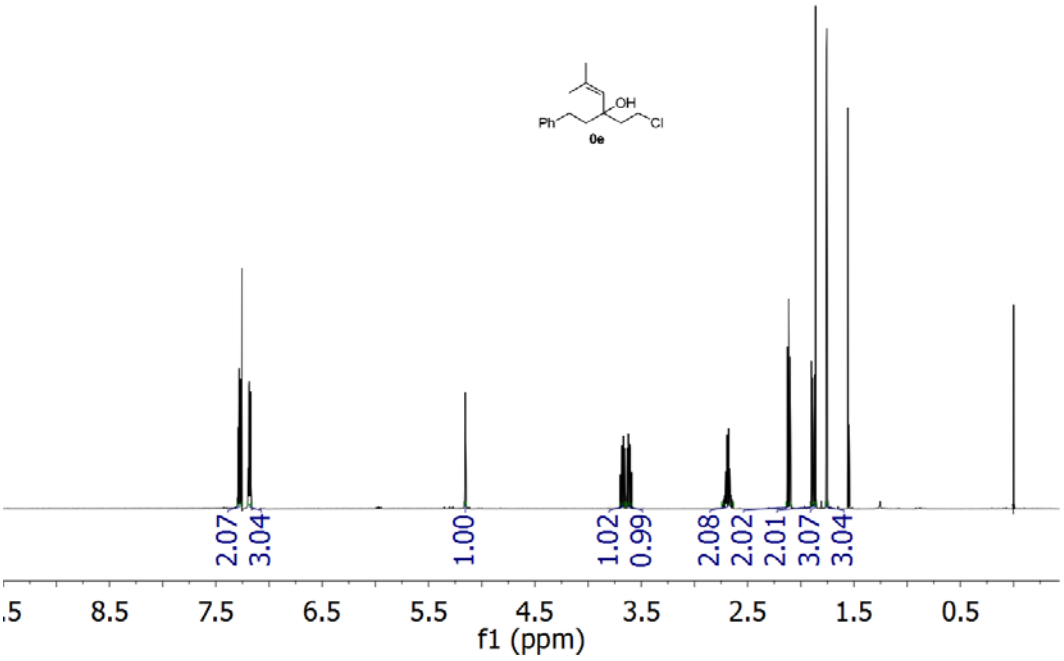

OH
Cl
0d
overlap with CHCl3
2.84
1.06
2.04
1.00
2.02
0.98
0.99
2.07
0.98
0.99
0.95
3.05
3.05
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

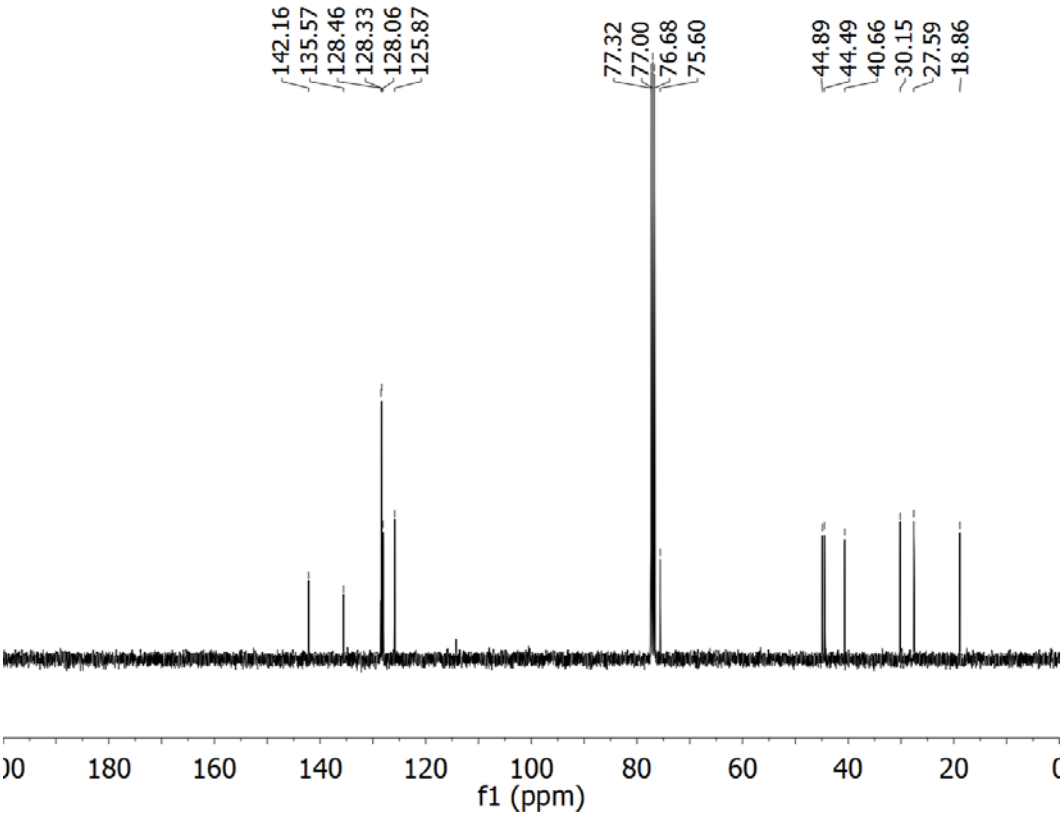

139.38
138.81
131.07
127.76
126.01
115.11
79.95
77.33
77.01
76.69
42.10
41.72
41.33
37.14
21.51
21.15
180
160
140
120
100
80
60
40
20
f1 (ppm)

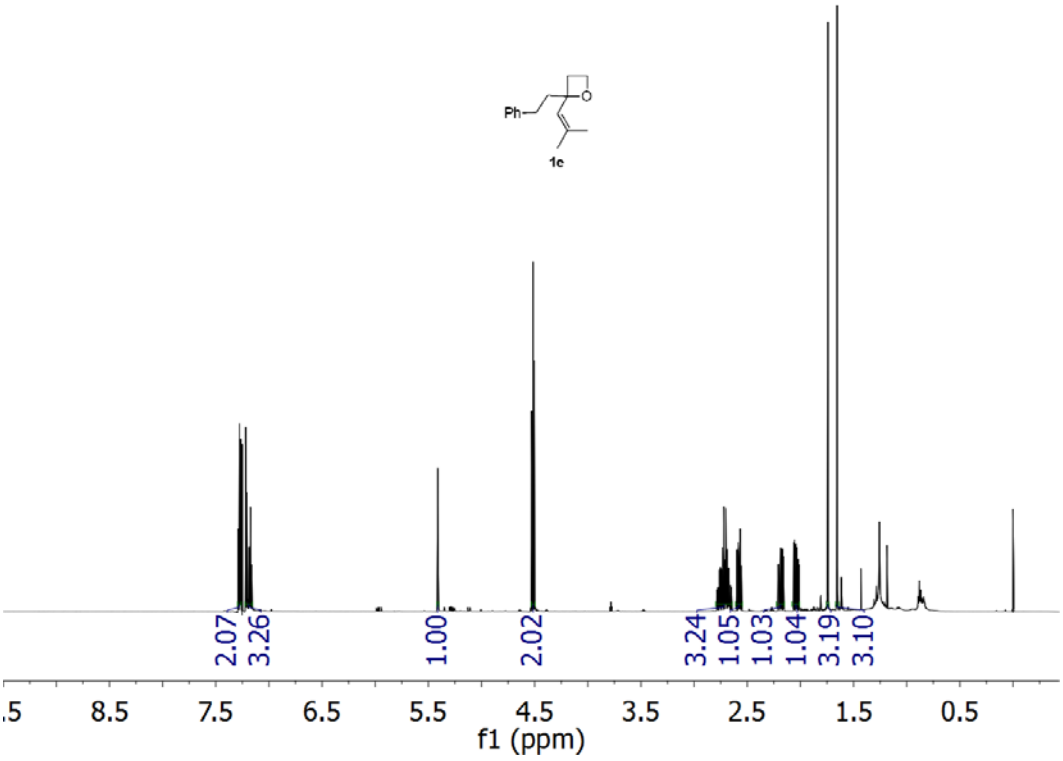


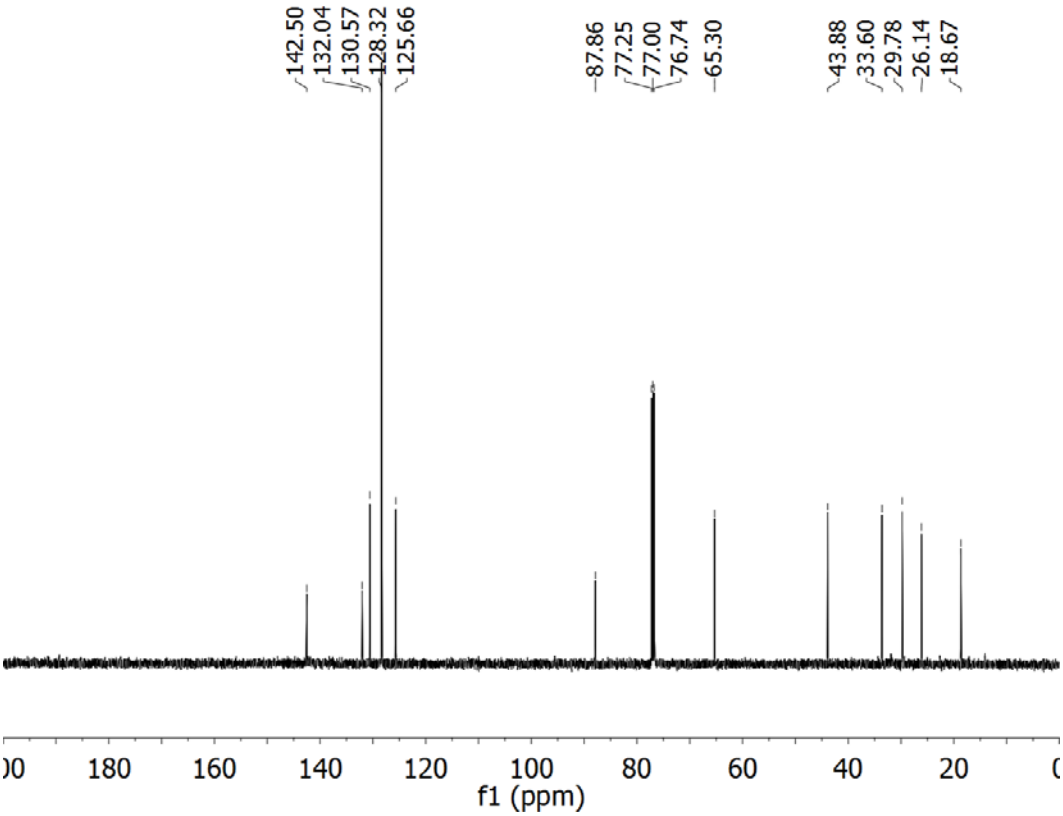


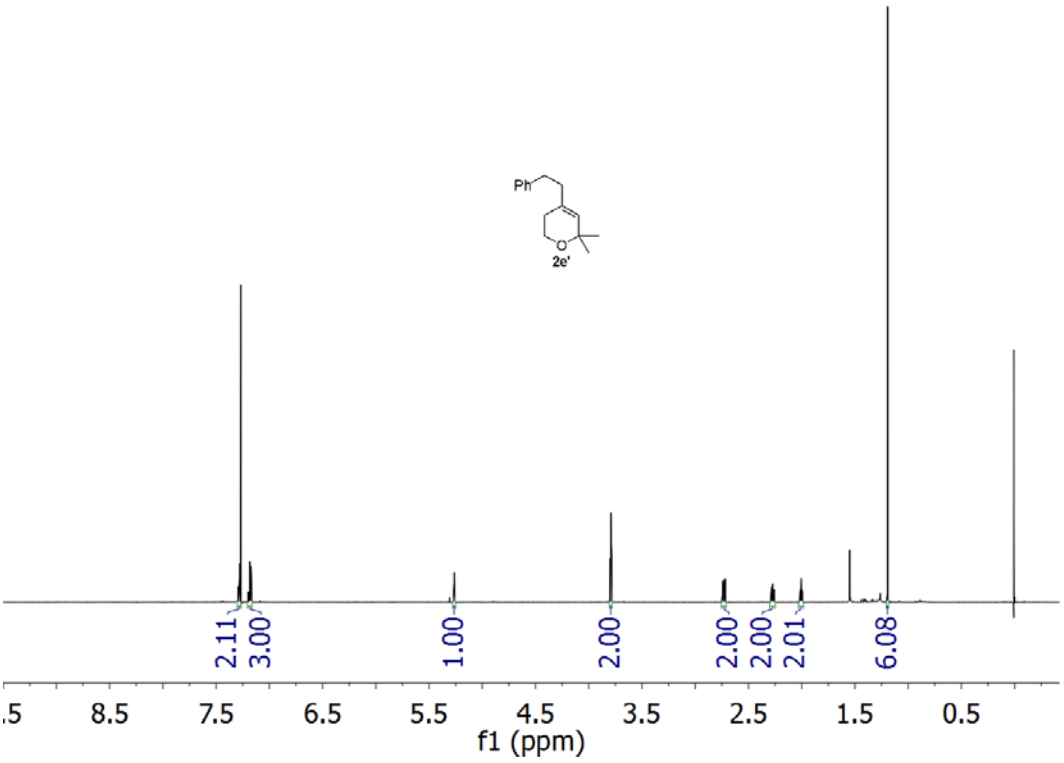


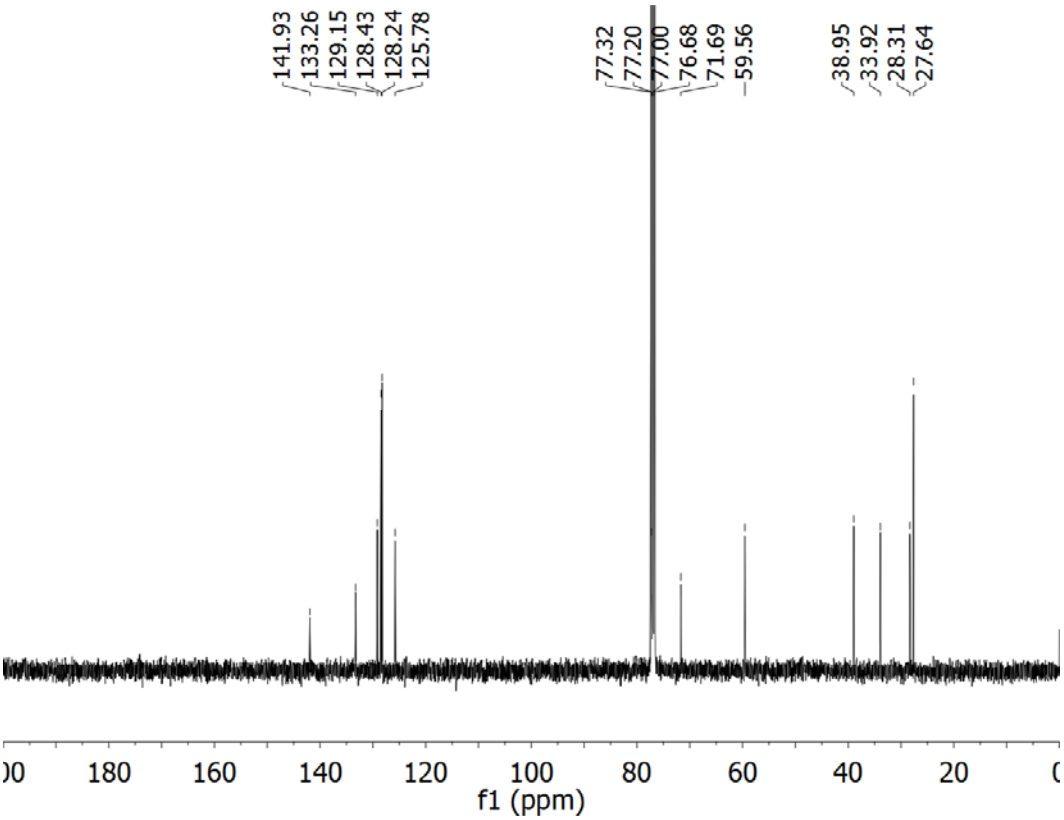

OH
Ph
Cl
0e
2.07
3.04
1.00
1.02
0.99
2.08
2.02
2.01
3.07
3.04
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

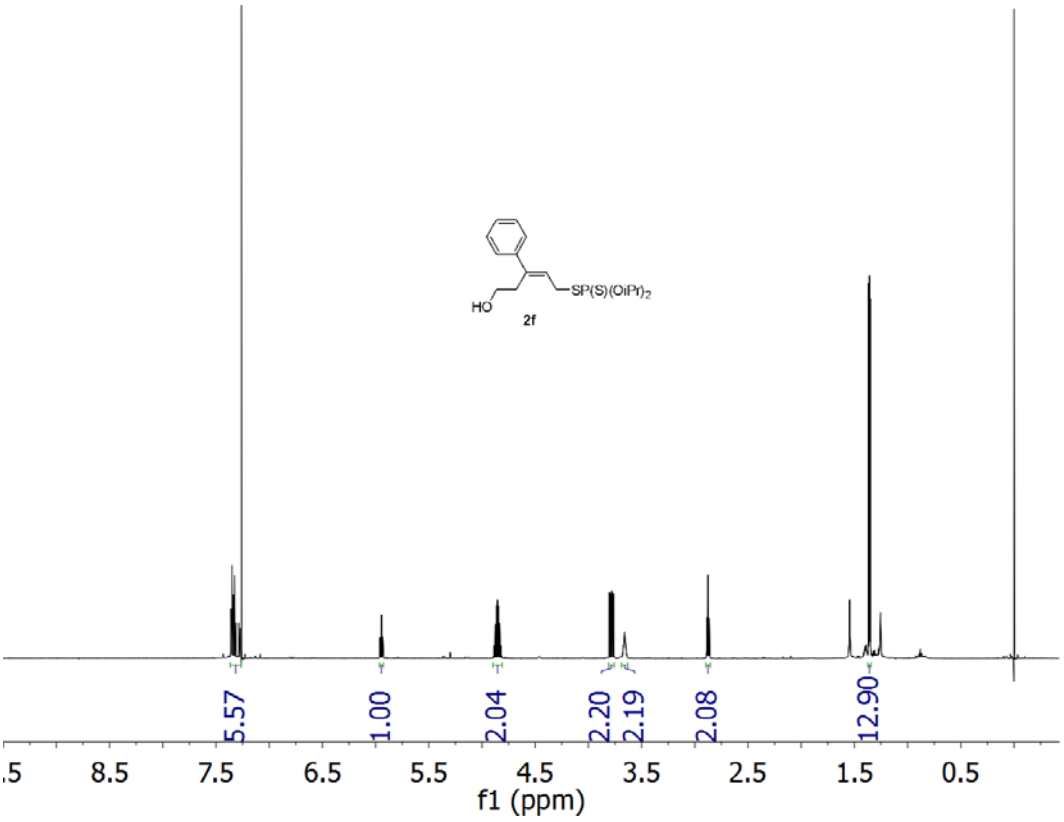

142.16
135.57
128.46
128.33
128.06
125.87
77.32
77.00
76.68
75.60
44.89
44.49
40.66
30.15
27.59
18.86
180
160
140
120
100
80
60
40
20
f1 (ppm)

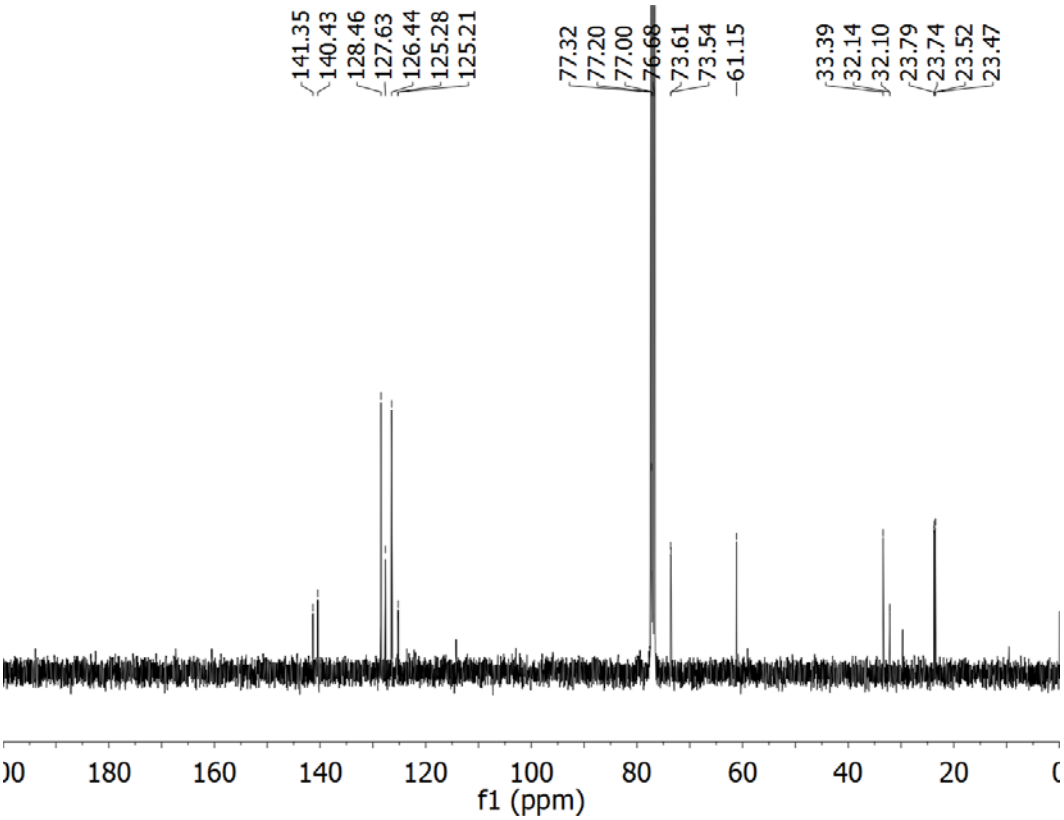

Ph
O
1e
2.07
3.26
1.00
2.02
3.24
1.05
1.03
1.04
3.19
3.10
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

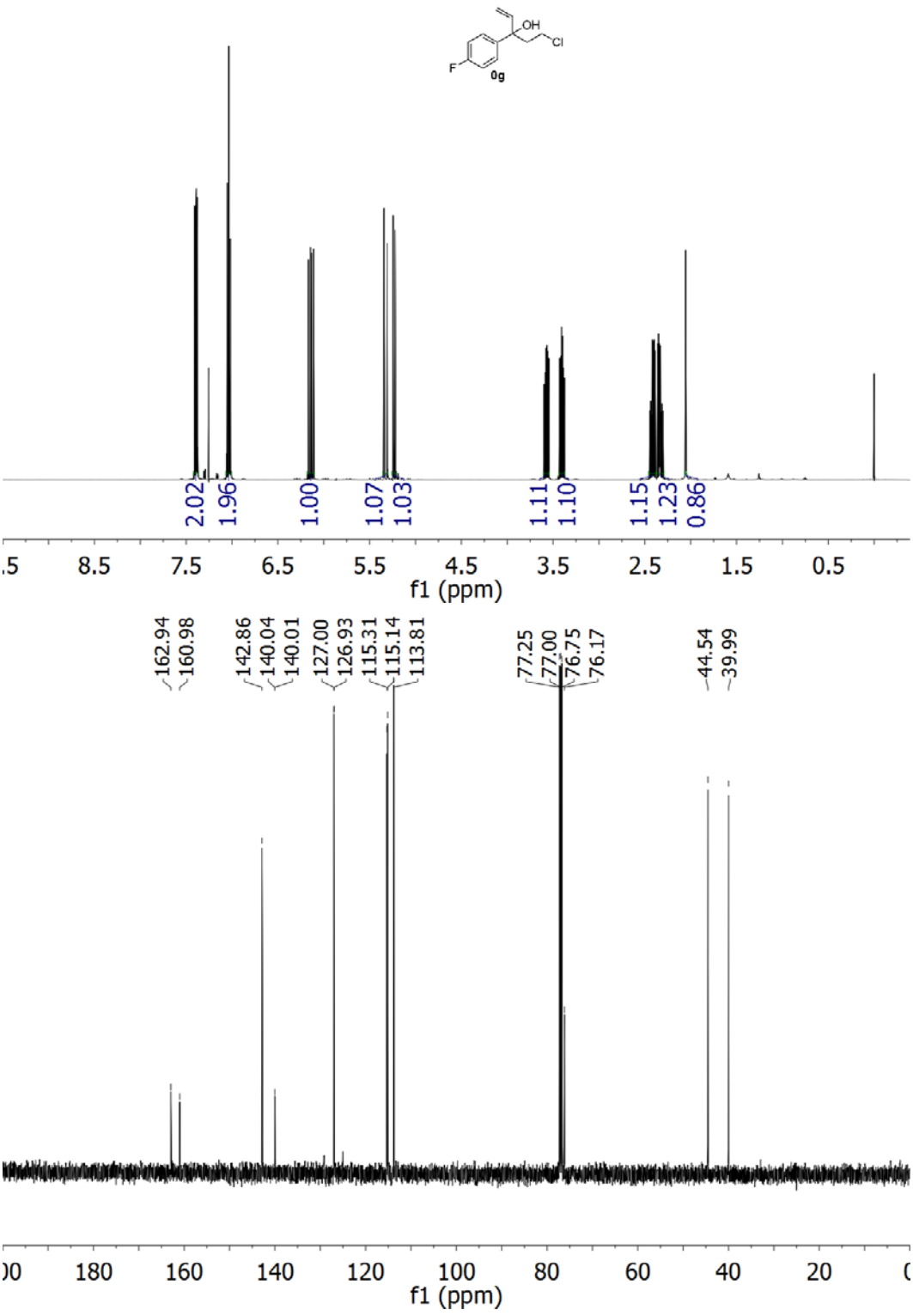

142.50
132.04
130.57
128.32
125.66
87.86
77.25
77.00
76.74
65.30
43.88
33.60
29.78
26.14
18.67
180
160
140
120
100
80
60
40
20
f1 (ppm)

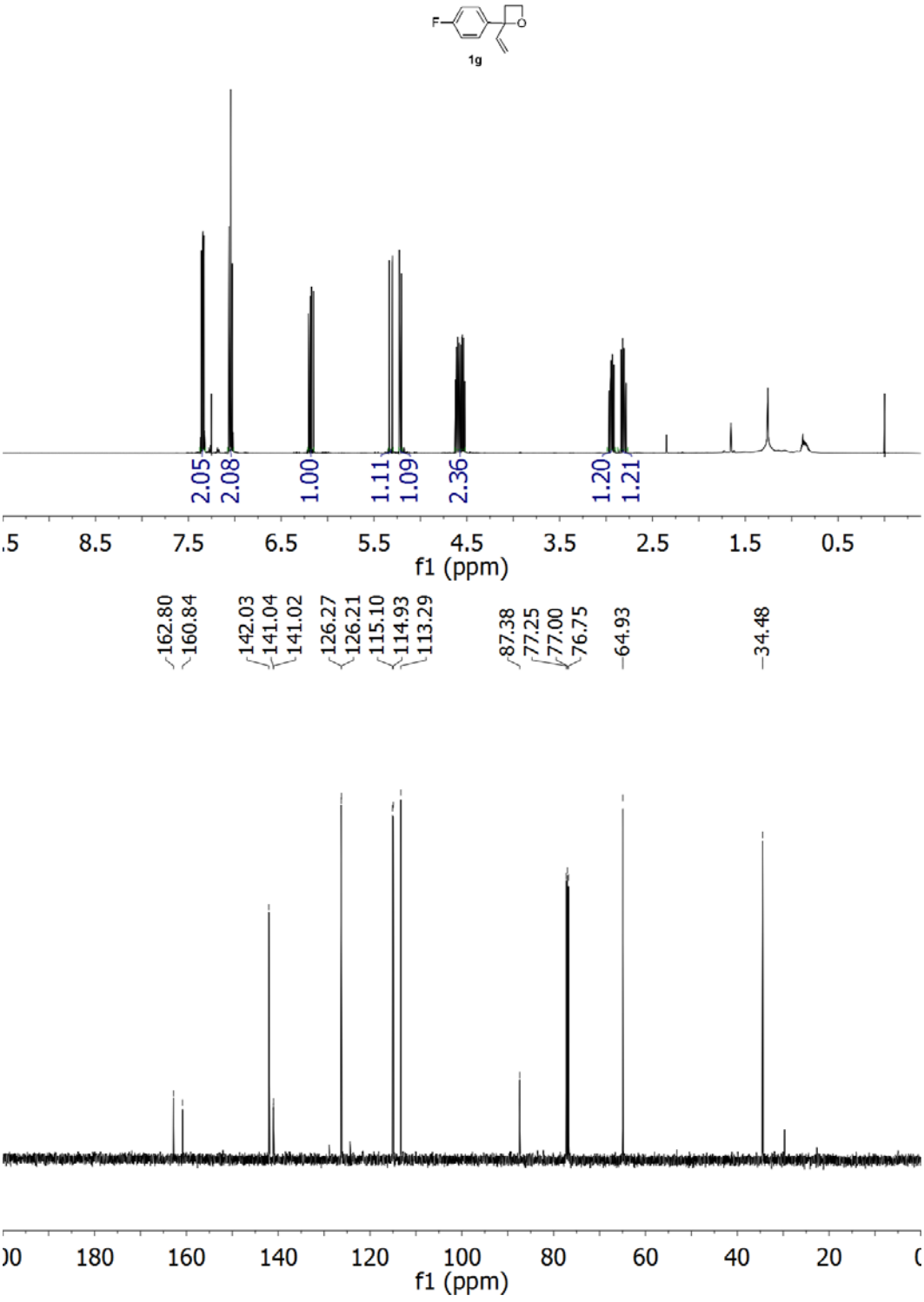

Ph
O
2e'
2.11
3.00
1.00
2.00
2.00
2.00
2.01
6.08
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

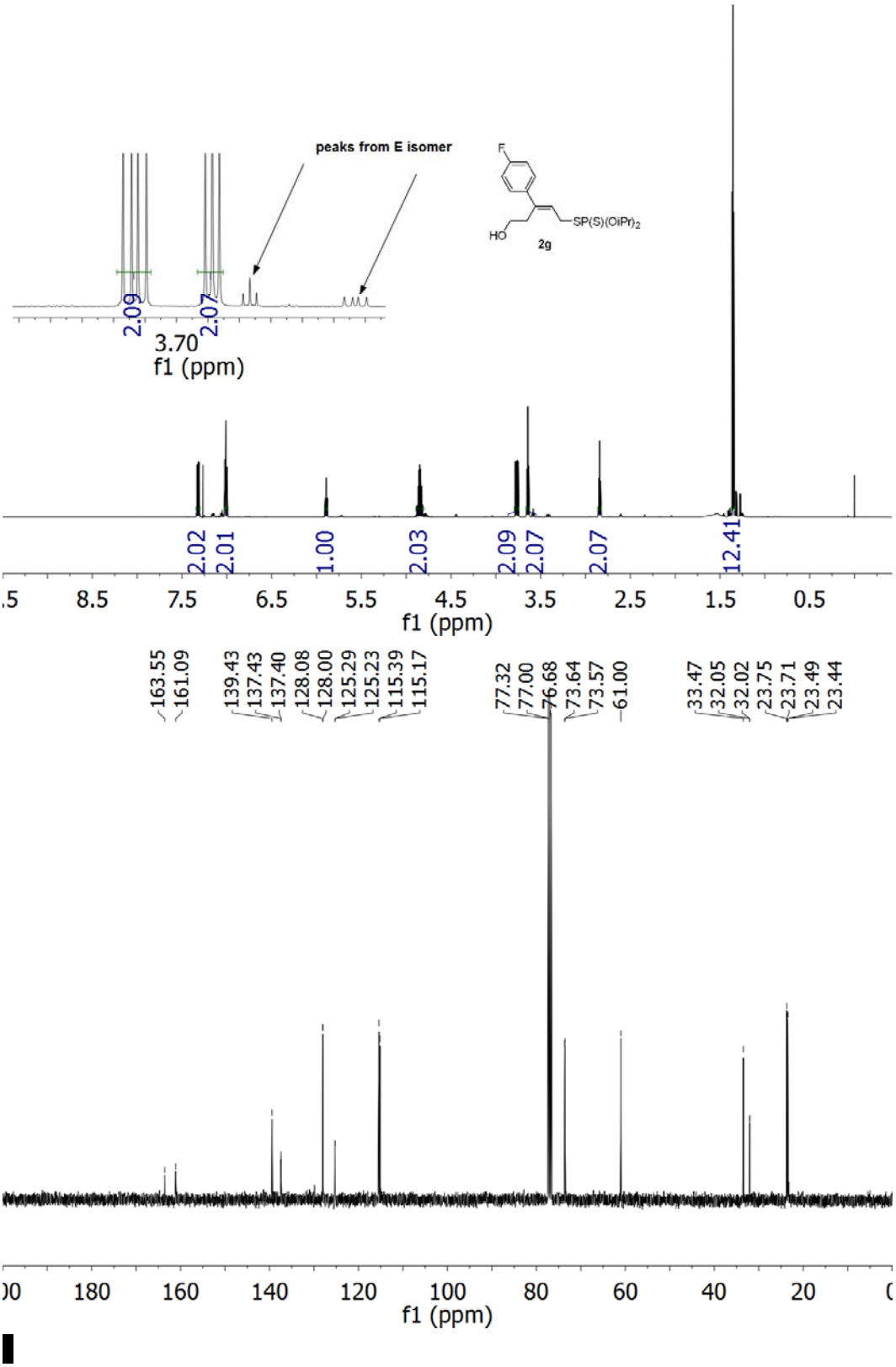

141.93
133.26
129.15
128.43
128.24
125.78
77.32
77.20
77.00
76.68
71.69
59.56
38.95
33.92
28.31
27.64
180
160
140
120
100
80
60
40
20
f1 (ppm)

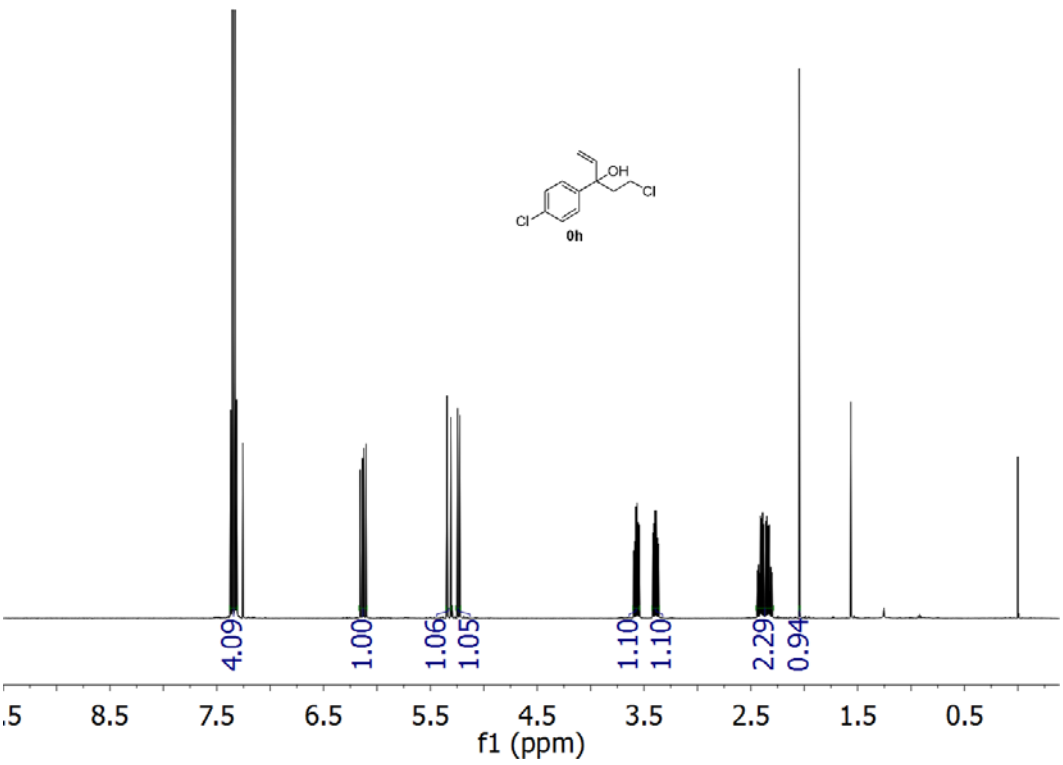

HO
SP(S)(OiPr)2
2f
5.57
1.00
2.04
2.20
2.19
2.08
12.90
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

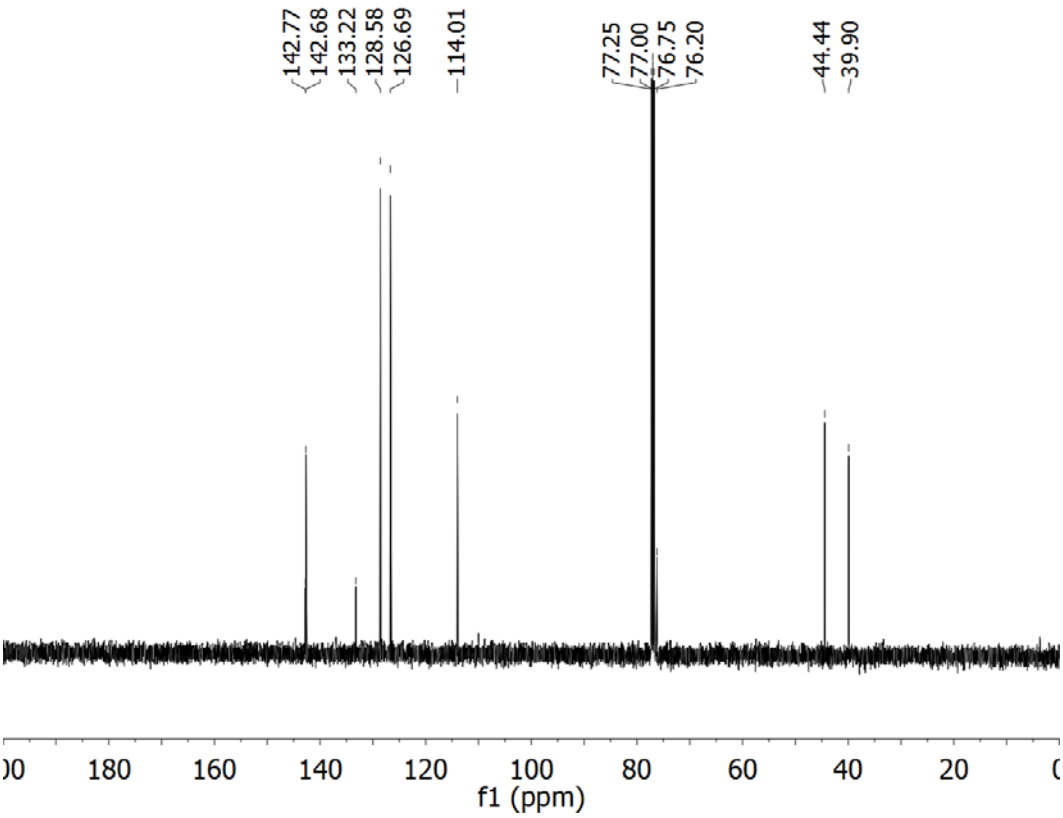

141.35
140.43
128.46
127.63
126.44
125.28
125.21
77.32
77.20
77.00
76.68
73.61
73.54
61.15
33.39
32.14
32.10
23.79
23.74
23.52
23.47
180
160
140
120
100
80
60
40
20
f1 (ppm)

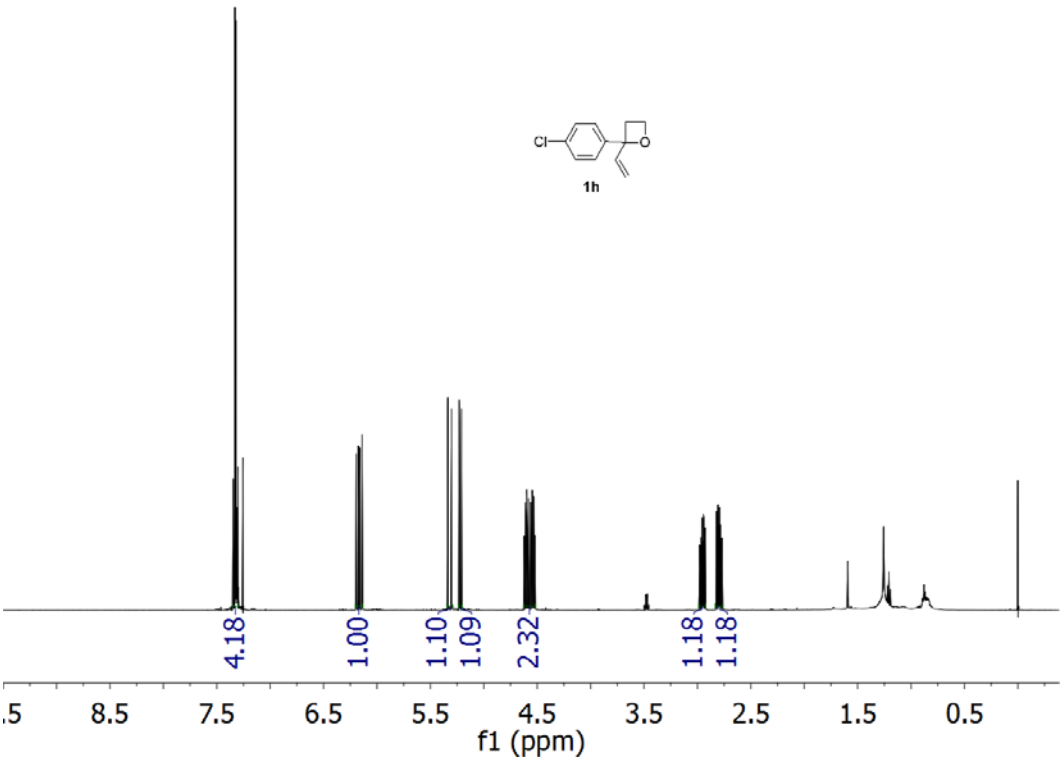

OH
Cl
F
0g
2.02
1.96
1.00
1.07
1.03
1.11
1.10
1.15
1.23
0.86
f1 (ppm)
162.94
160.98
142.86
140.04
140.01
127.00
126.93
115.31
115.14
113.81
77.25
77.00
76.75
76.17
44.54
39.99
f1 (ppm)

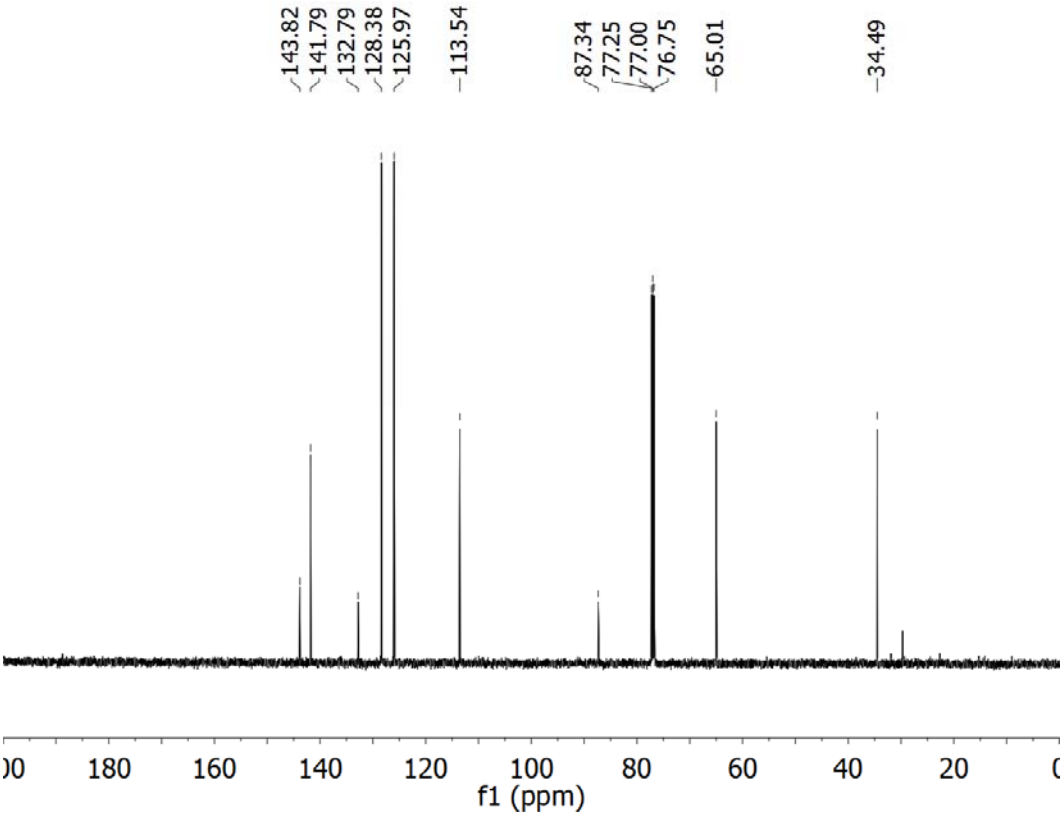

F
O
1g
2.05
2.08
1.00
1.11
1.09
2.36
1.20
1.21
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)
162.80
160.84
142.03
141.04
141.02
126.27
126.21
115.10
114.93
113.29
87.38
77.25
77.00
76.75
64.93
34.48
180
160
140
120
100
80
60
40
20
f1 (ppm)

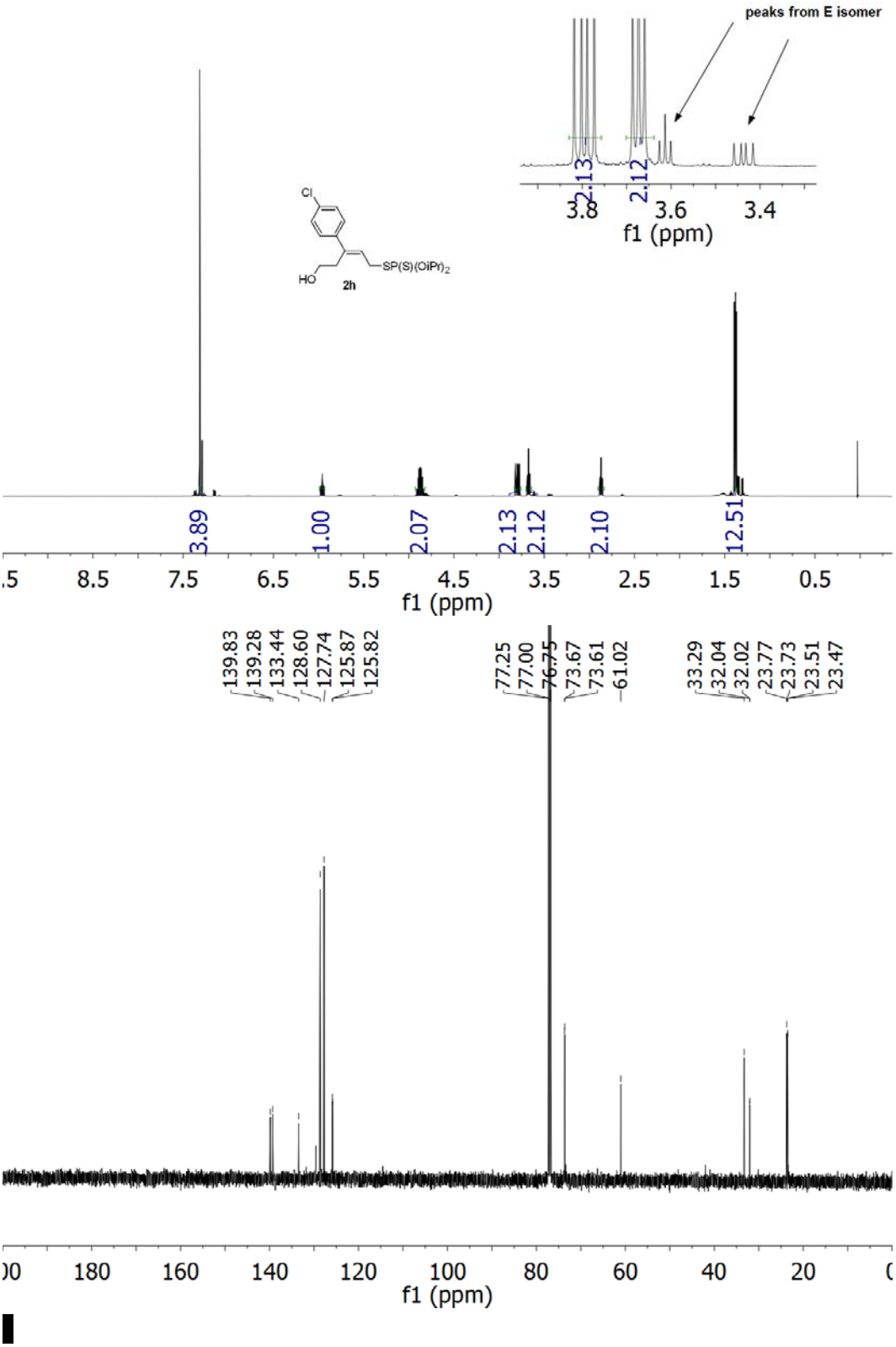

peaks from E isomer
F
HO
SP(S)(OiPr)2
2g
2.09
2.07
3.70
f1 (ppm)
2.02
2.01
1.00
2.03
2.09
2.07
2.07
12.41
f1 (ppm)
163.55
161.09
139.43
137.43
137.40
128.08
128.00
125.29
125.23
115.39
115.17
77.32
77.00
76.68
73.64
73.57
61.00
33.47
32.05
32.02
23.75
23.71
23.49
23.44
f1 (ppm)

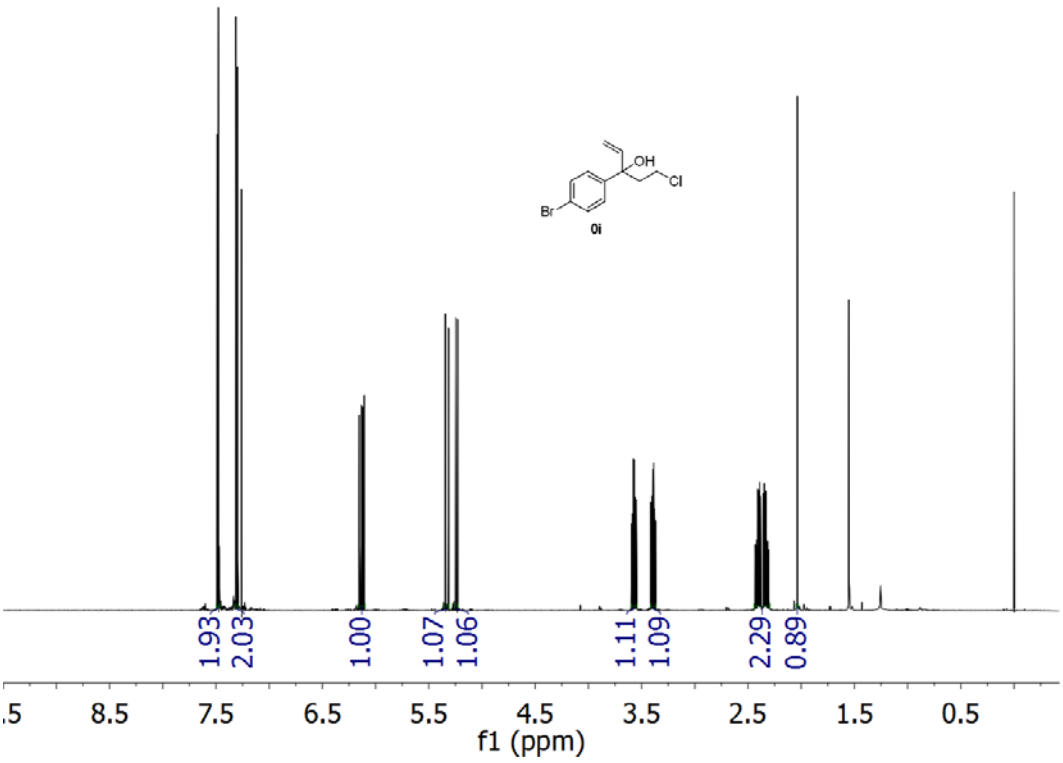


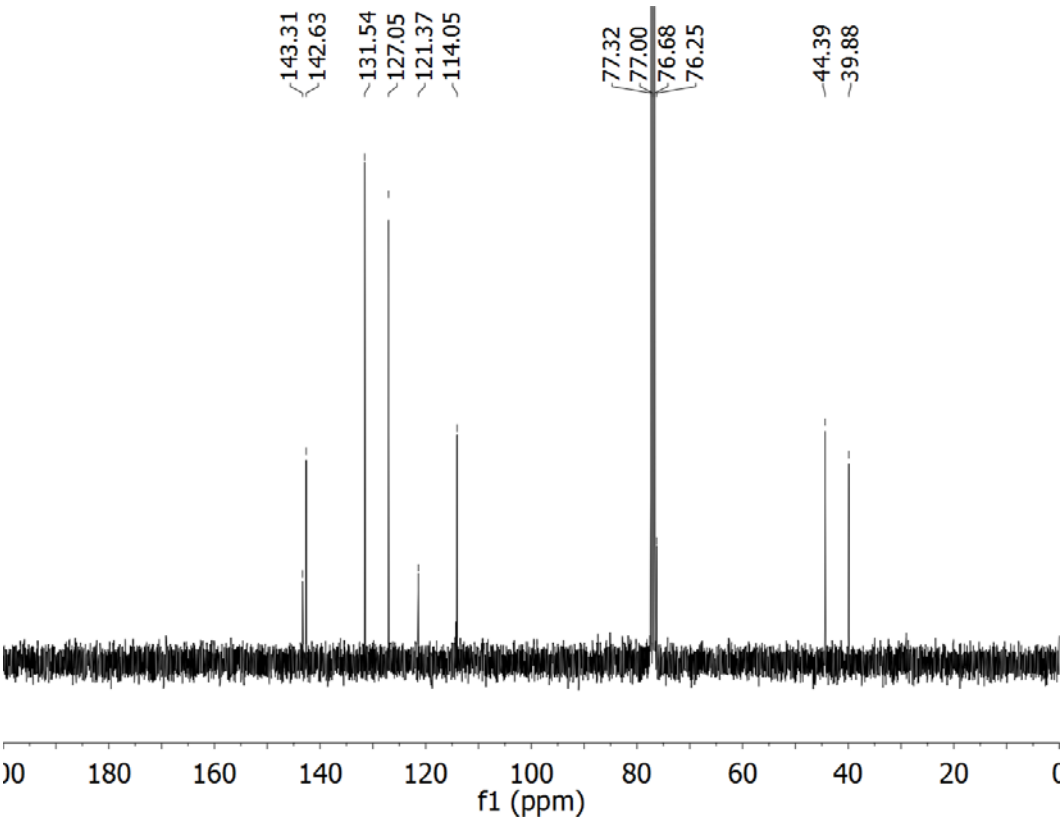


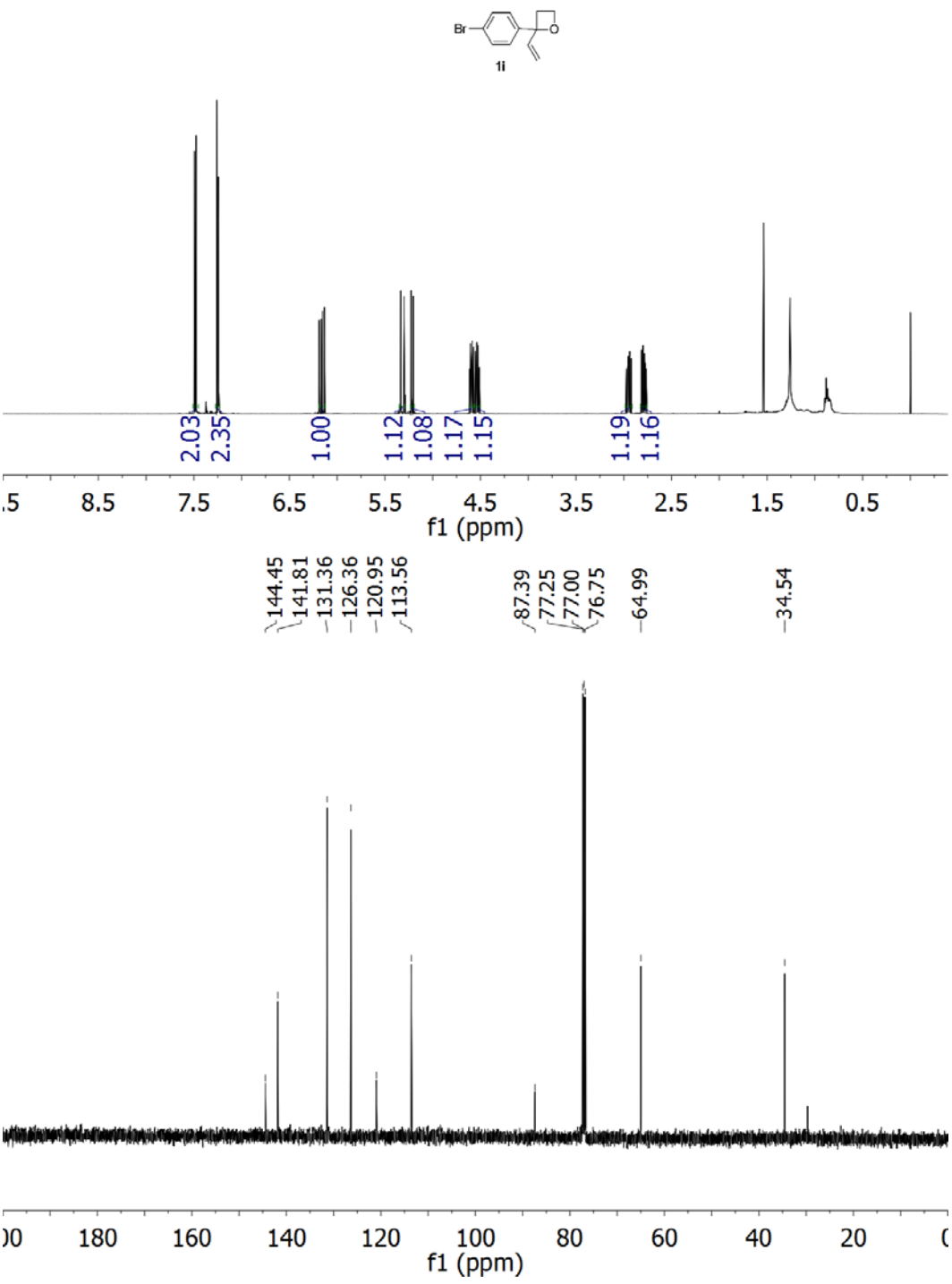

Cl
O
1h
4.18
1.00
1.10
1.09
2.32
1.18
1.18
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

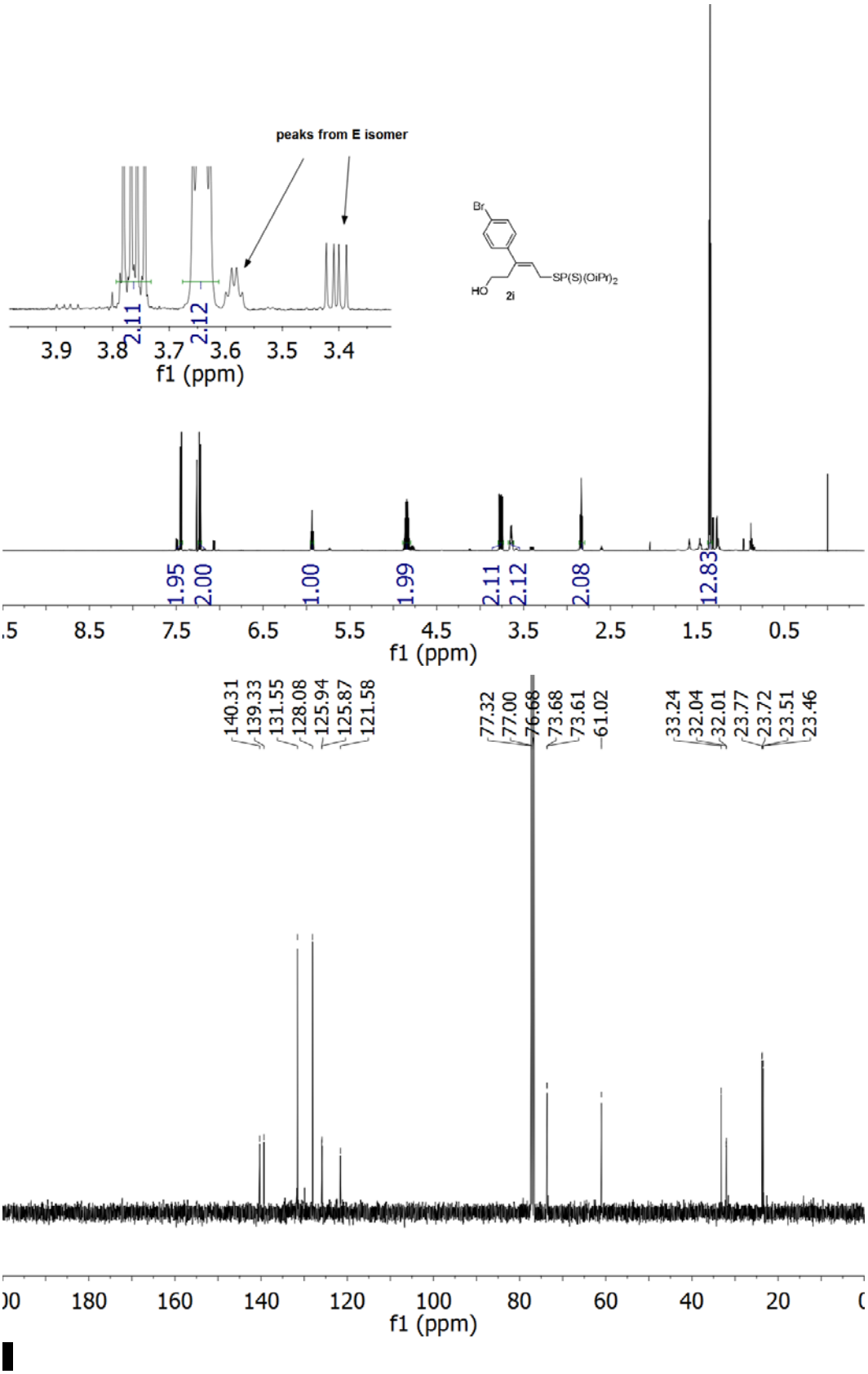

143.82
141.79
132.79
128.38
125.97
113.54
87.34
77.25
77.00
76.75
65.01
34.49
180
160
140
120
100
80
60
40
20
f1 (ppm)

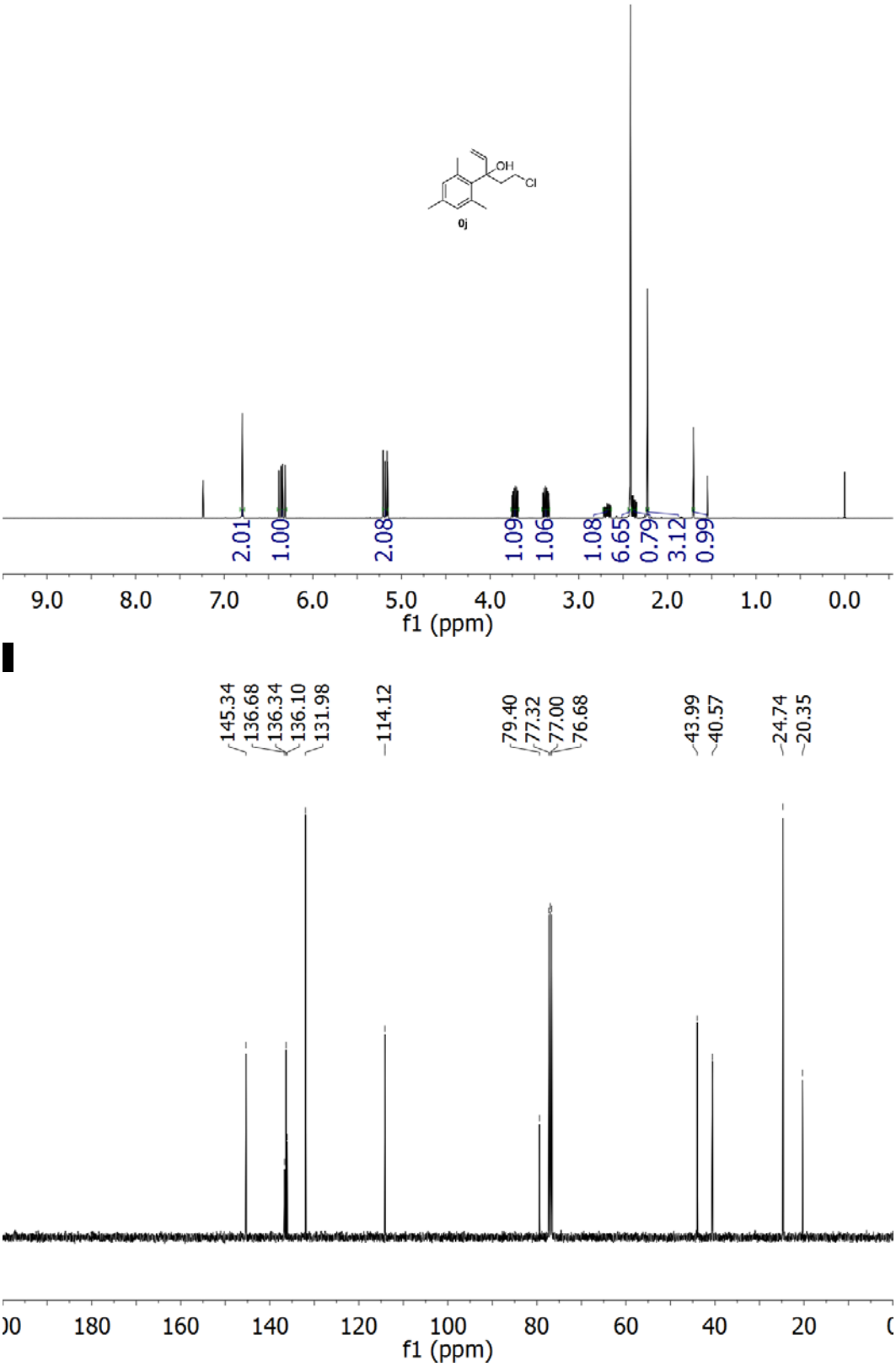

peaks from E isomer
2.13
2.12
3.8
3.6
3.4
f1 (ppm)
Cl
HO
SP(S)(OiPr)2
2h
3.89
1.00
2.07
2.13
2.12
2.10
12.51
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)
139.83
139.28
133.44
128.60
127.74
125.87
125.82
77.25
77.00
76.75
73.67
73.61
61.02
33.29
32.04
32.02
23.77
23.73
23.51
23.47
180
160
140
120
100
80
60
40
20
f1 (ppm)

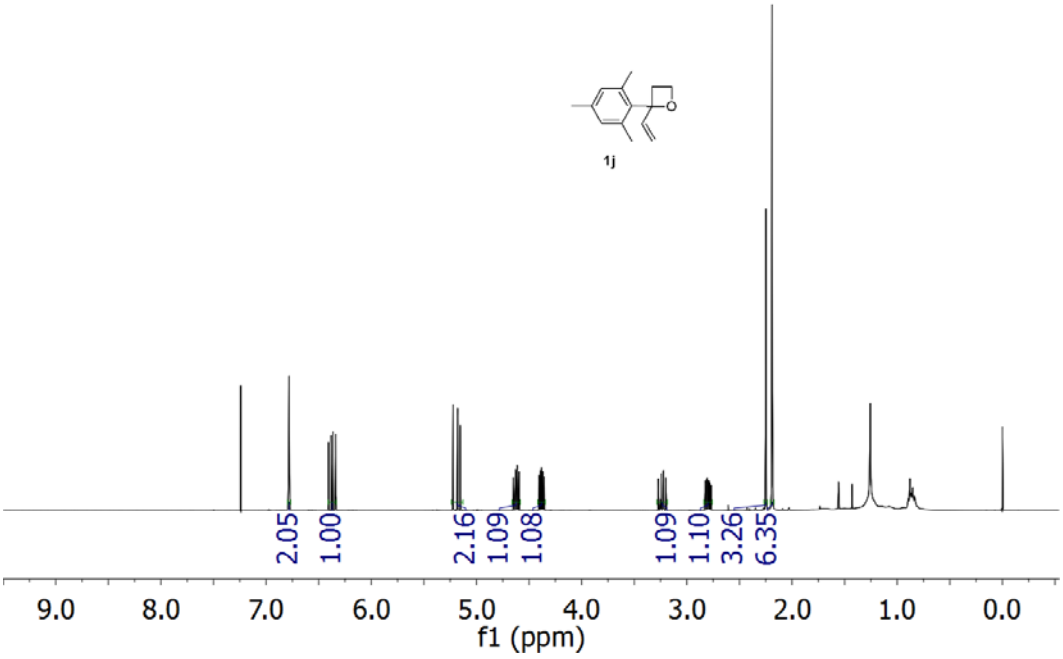

OH
Cl
Br
0i
1.93
2.03
1.00
1.07
1.06
1.11
1.09
2.29
0.89
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

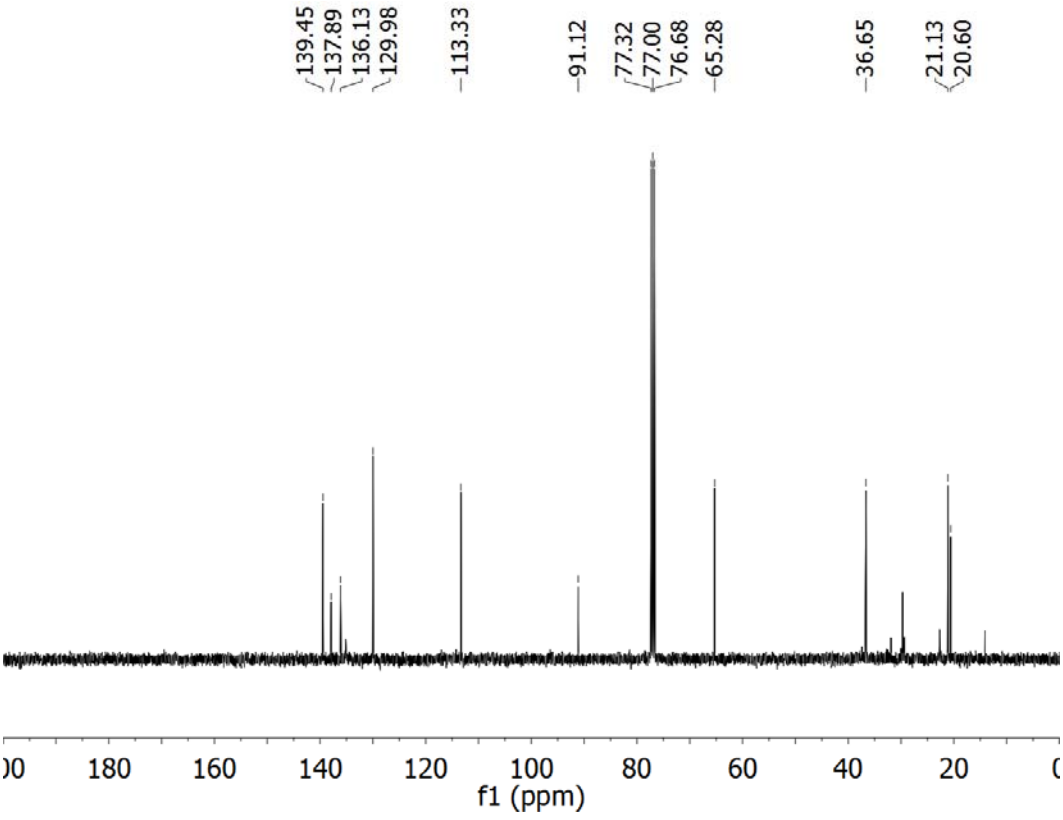

143.31
142.63
131.54
127.05
121.37
114.05
77.32
77.00
76.68
76.25
44.39
39.88
180
160
140
120
100
80
60
40
20
f1 (ppm)

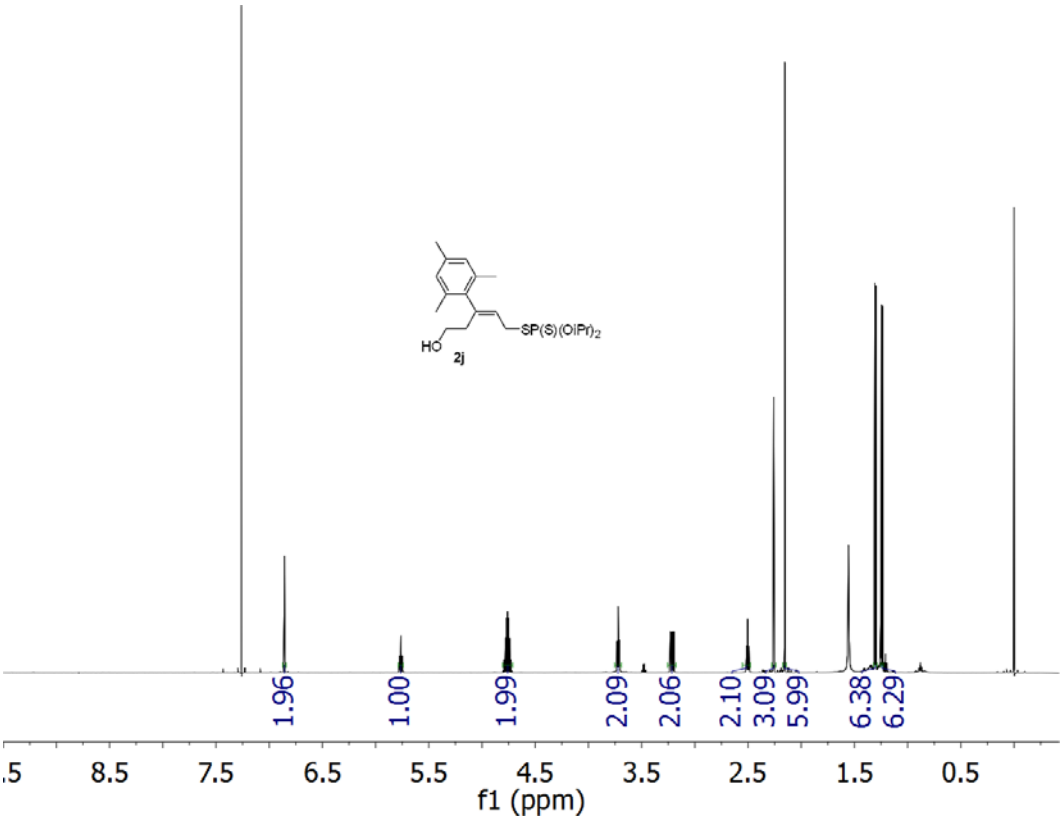


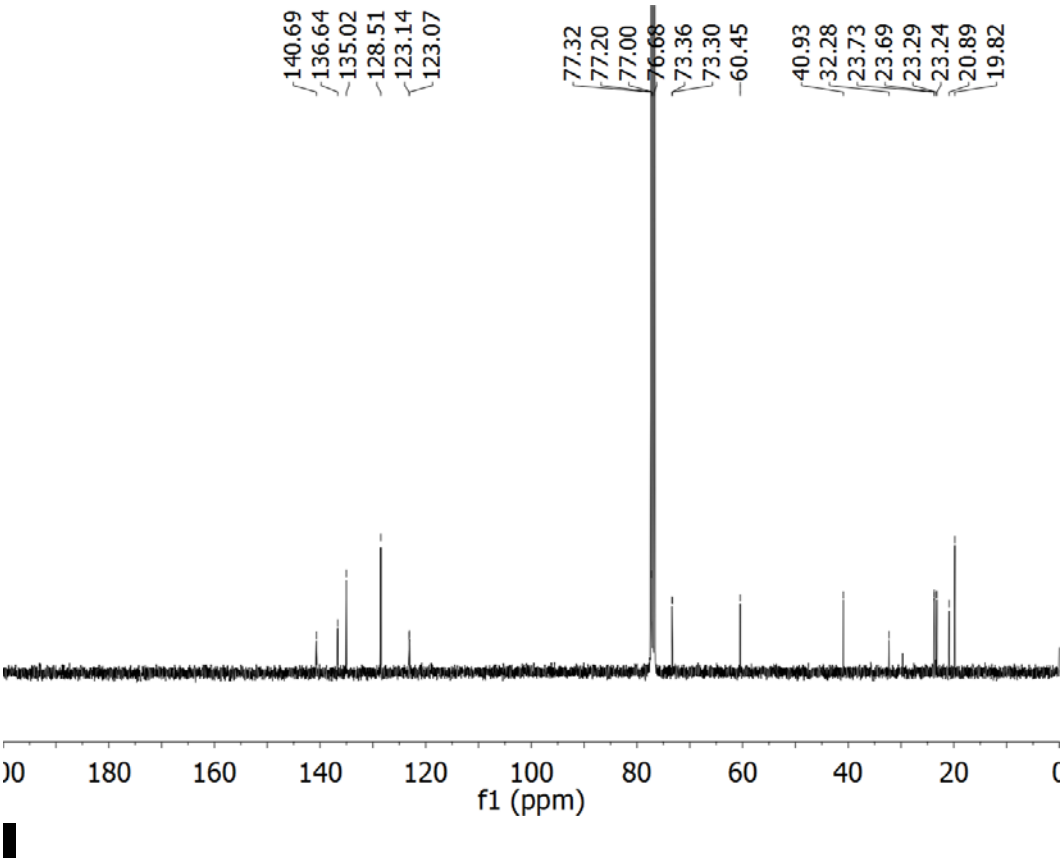

peaks from E isomer
2.11
2.12
3.9
3.8
3.7
3.6
3.5
3.4
f1 (ppm)
Br
HO
2i
SP(S)(OiPr)2
1.95
2.00
1.00
1.99
2.11
2.12
2.08
12.83
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)
140.31
139.33
131.55
128.08
125.94
125.87
121.58
77.32
77.00
76.68
73.68
73.61
61.02
33.24
32.04
32.01
23.77
23.72
23.51
23.46
180
160
140
120
100
80
60
40
20
f1 (ppm)

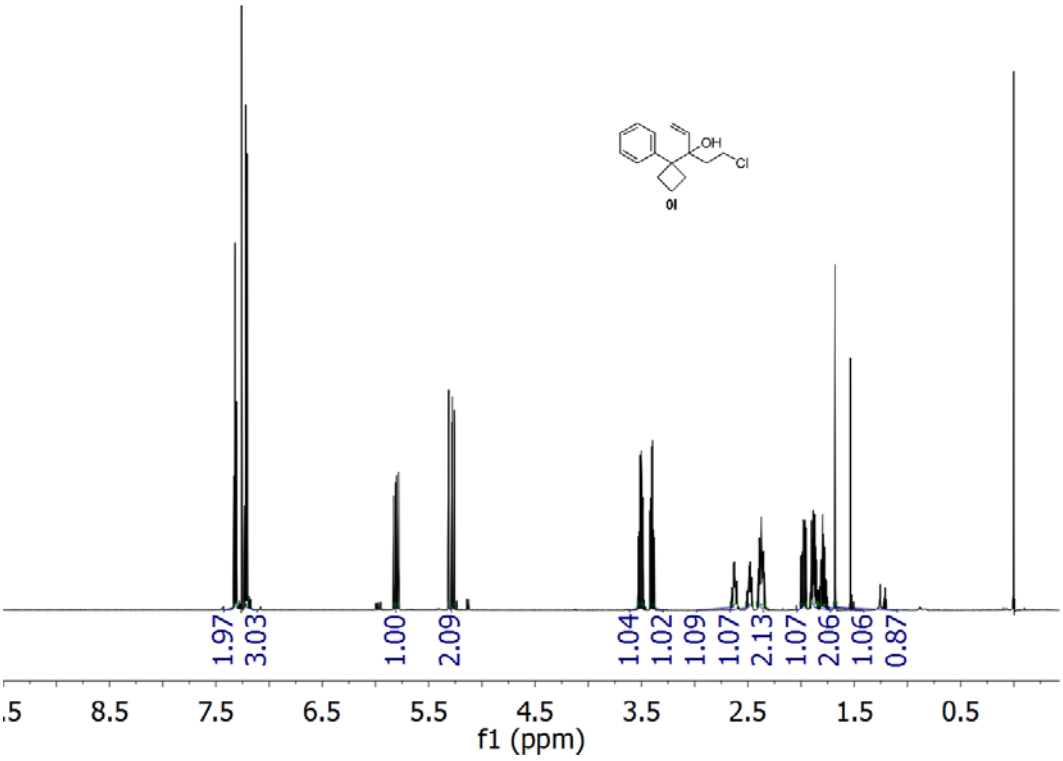


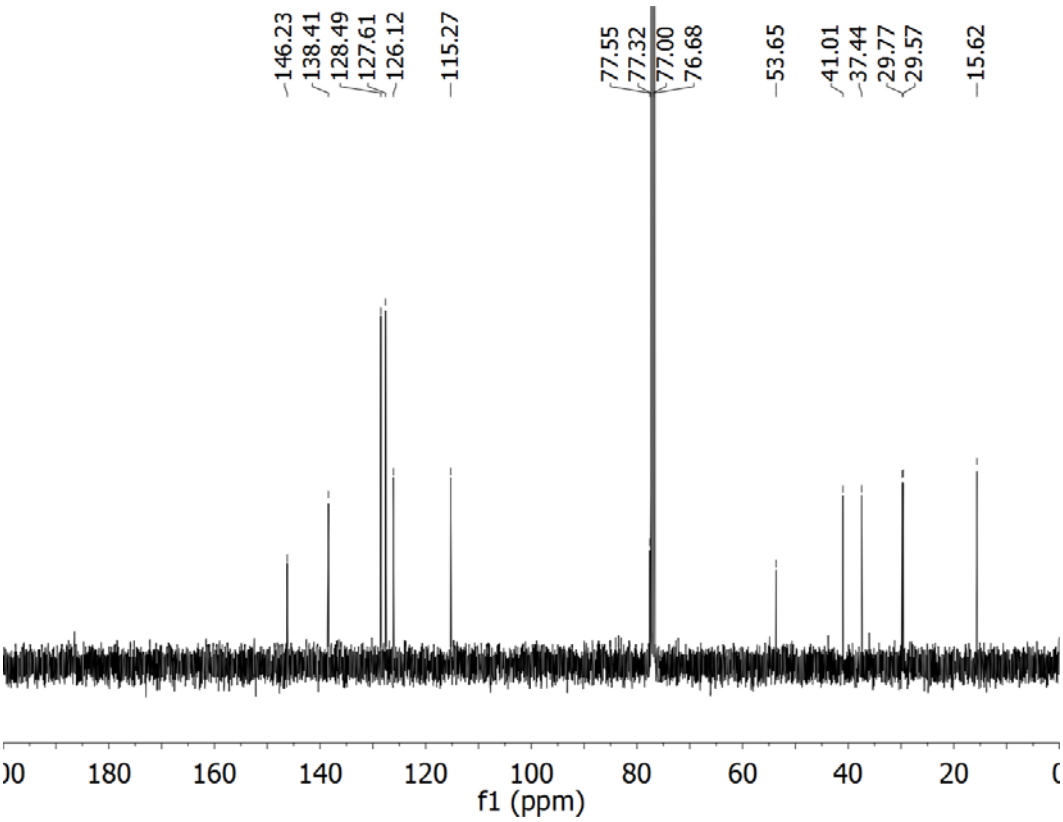

1j
2.05
1.00
2.16
1.09
1.08
1.09
1.10
3.26
6.35
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

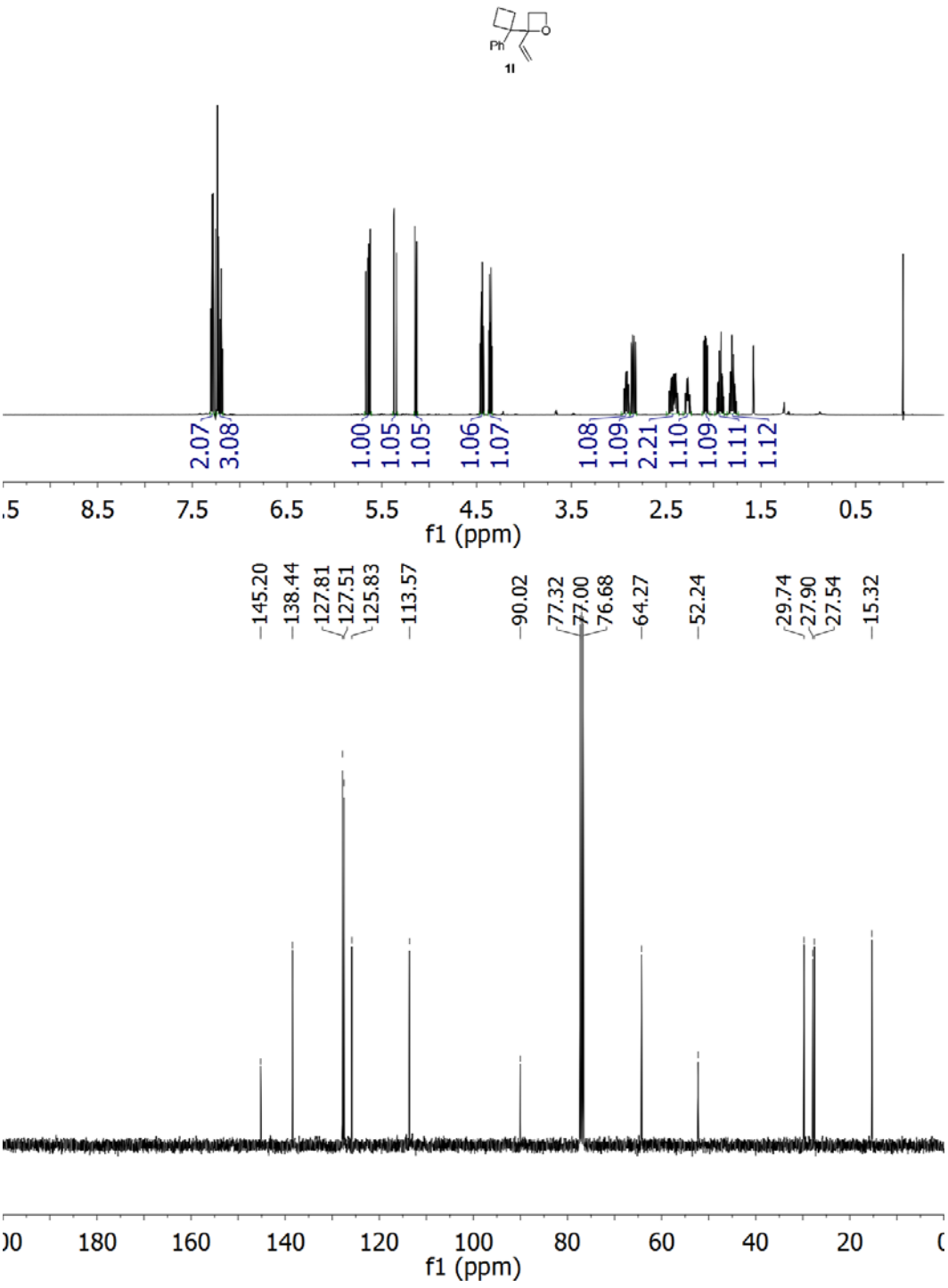

139.45
137.89
136.13
129.98
113.33
91.12
77.32
77.00
76.68
65.28
36.65
21.13
20.60
180
160
140
120
100
80
60
40
20
f1 (ppm)

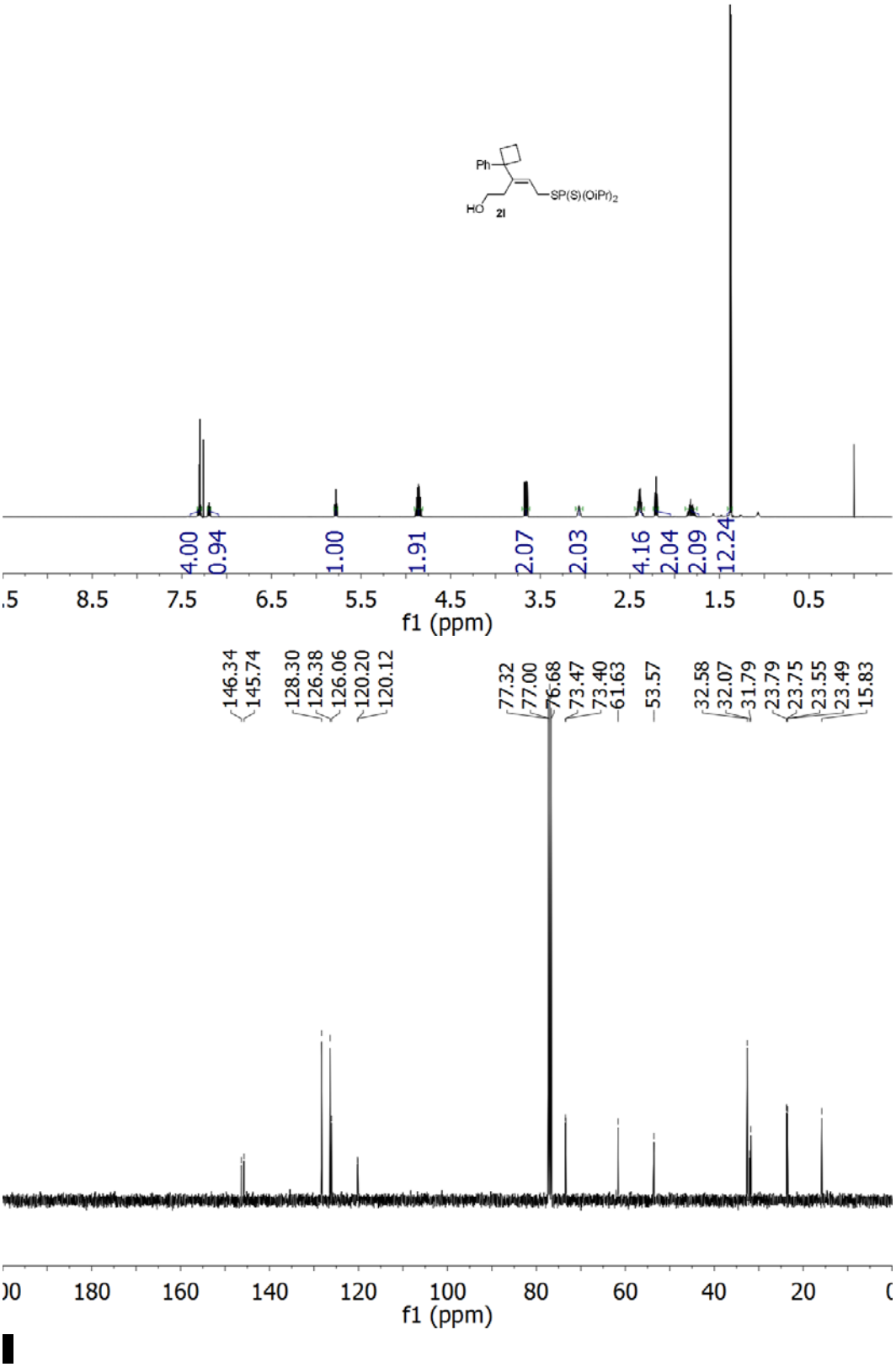

SP(S)(OiPr)2
HO
2j
1.96
1.00
1.99
2.09
2.06
2.10
3.09
5.99
6.38
6.29
f1 (ppm)

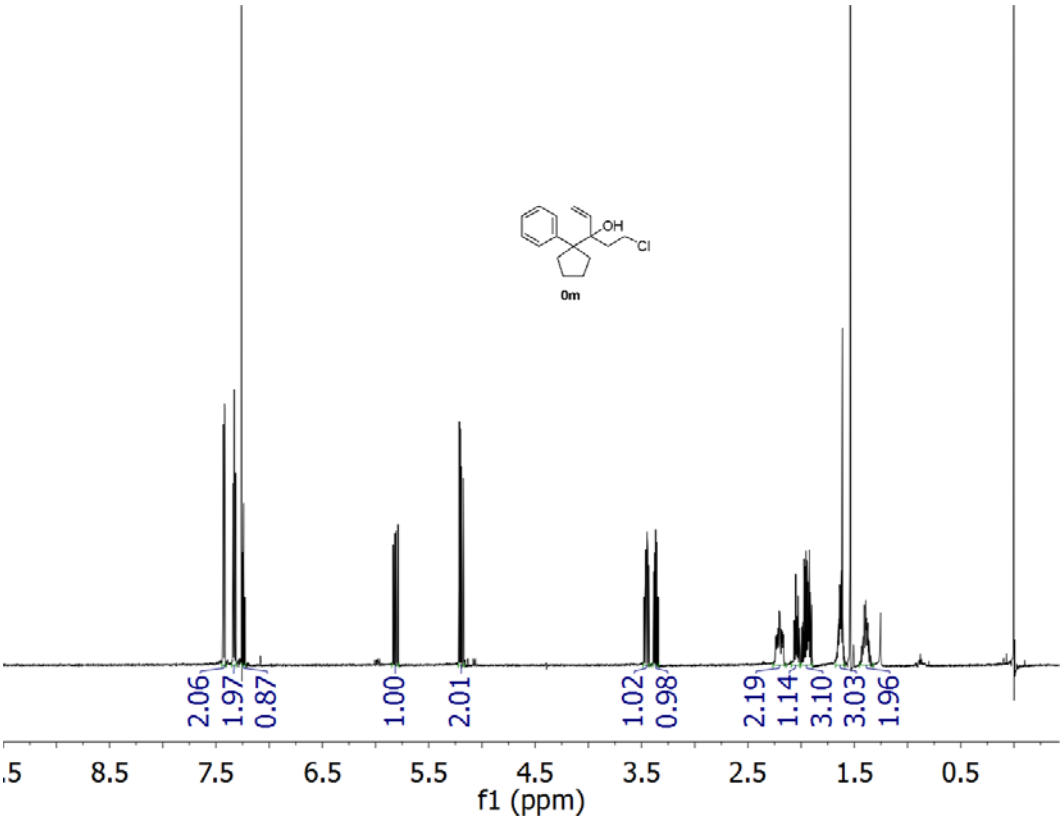

140.69
136.64
135.02
128.51
123.14
123.07
77.32
77.20
77.00
76.68
73.36
73.30
60.45
40.93
32.28
23.73
23.69
23.29
23.24
20.89
19.82
f1 (ppm)

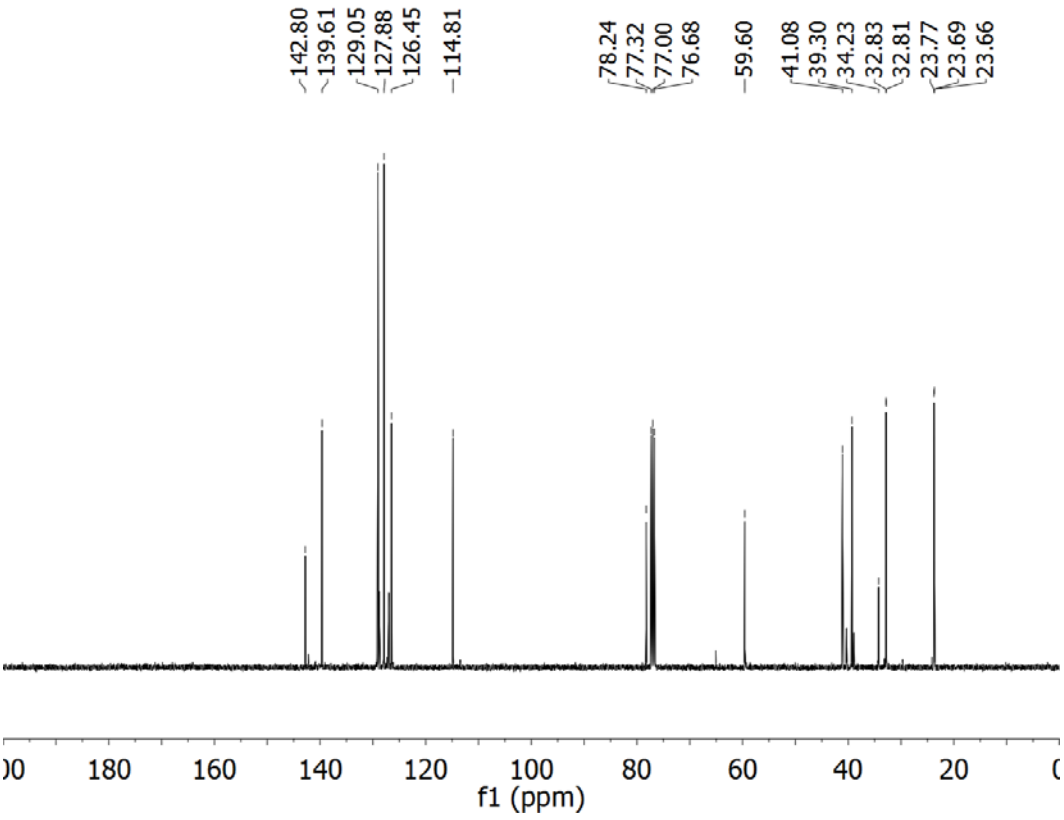


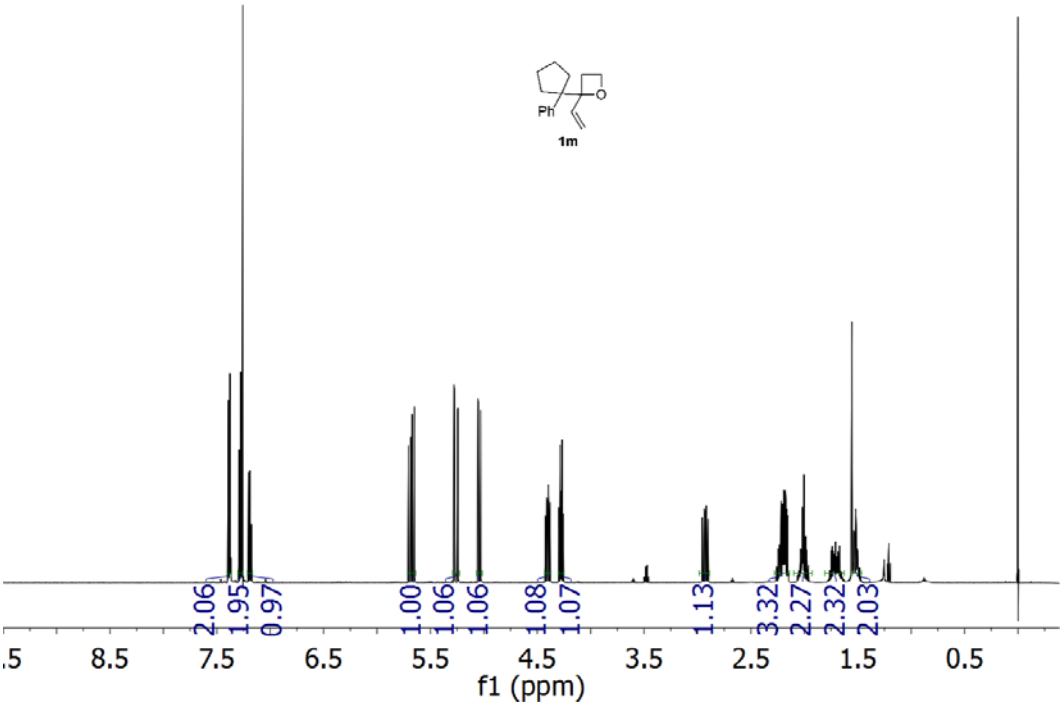


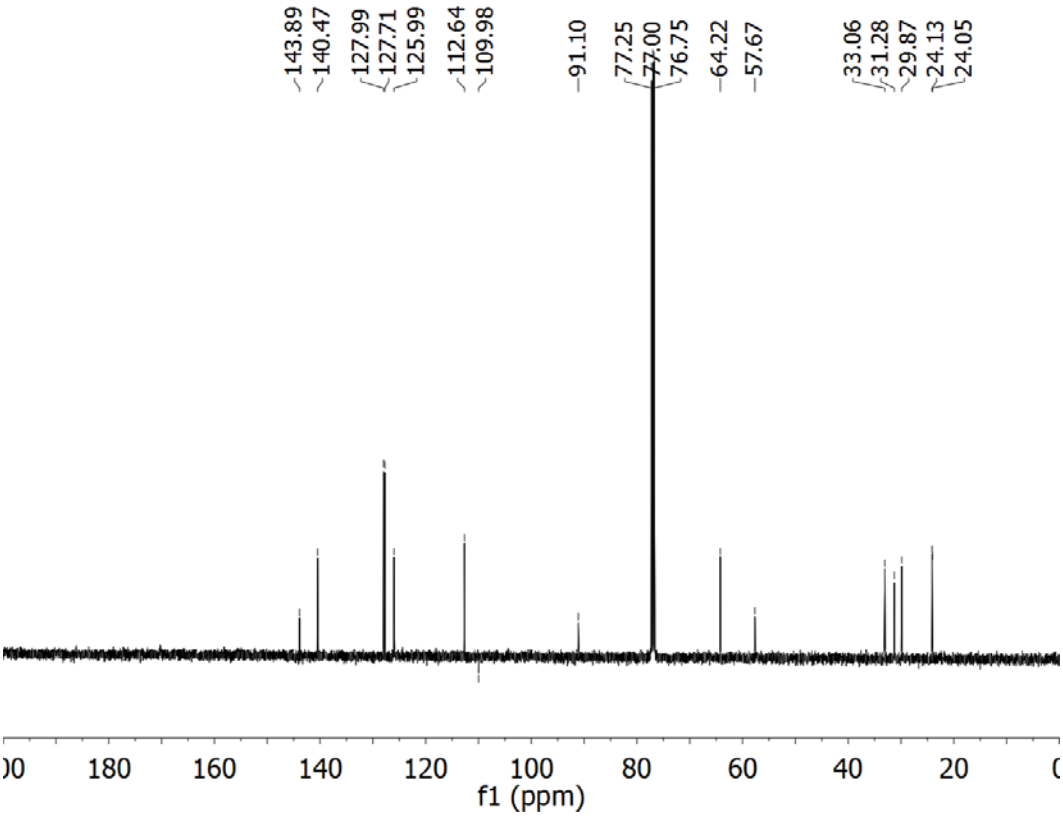

Ph
1l
2.07
3.08
1.00
1.05
1.05
1.06
1.07
1.08
1.09
2.21
1.10
1.09
1.11
1.12
f1 (ppm)
145.20
138.44
127.81
127.51
125.83
113.57
90.02
77.32
77.00
76.68
64.27
52.24
29.74
27.90
27.54
15.32
f1 (ppm)

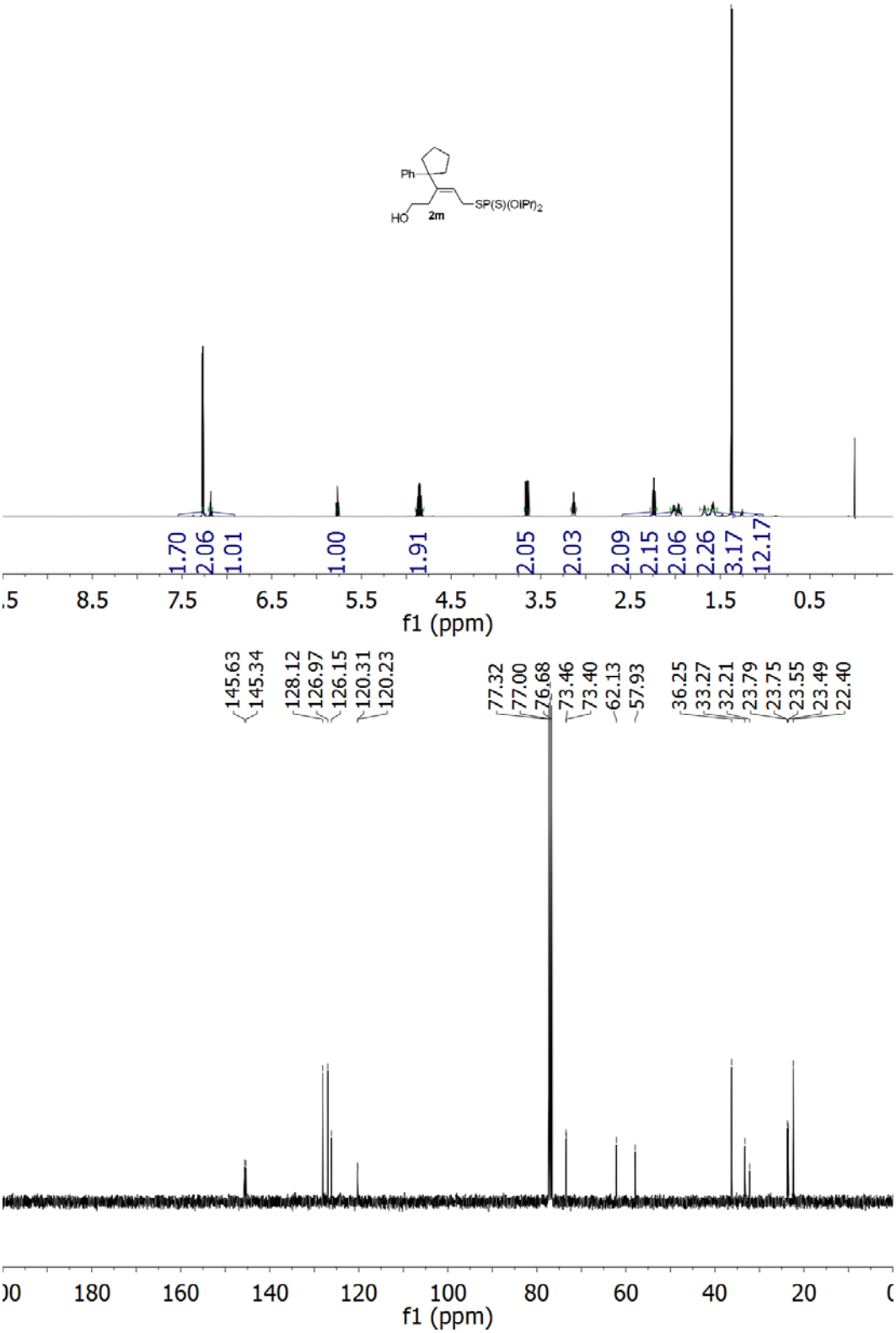

Ph
SP(S)(OiPr)2
HO
2l
4.00
0.94
1.00
1.91
2.07
2.03
4.16
2.04
2.09
12.24
f1 (ppm)
146.34
145.74
128.30
126.38
126.06
120.20
120.12
77.32
77.00
76.68
73.47
73.40
61.63
53.57
32.58
32.07
31.79
23.79
23.75
23.55
23.49
15.83
f1 (ppm)

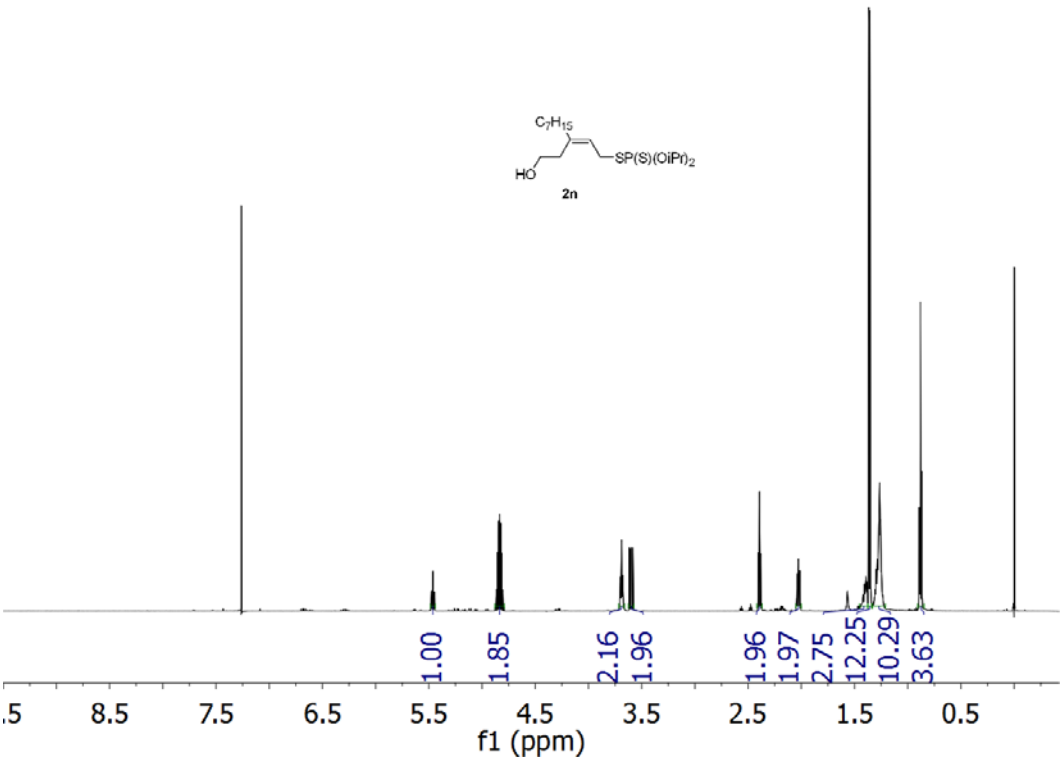

OH
Cl
0m
2.06
1.97
0.87
1.00
2.01
1.02
0.98
2.19
1.14
3.10
3.03
1.96
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

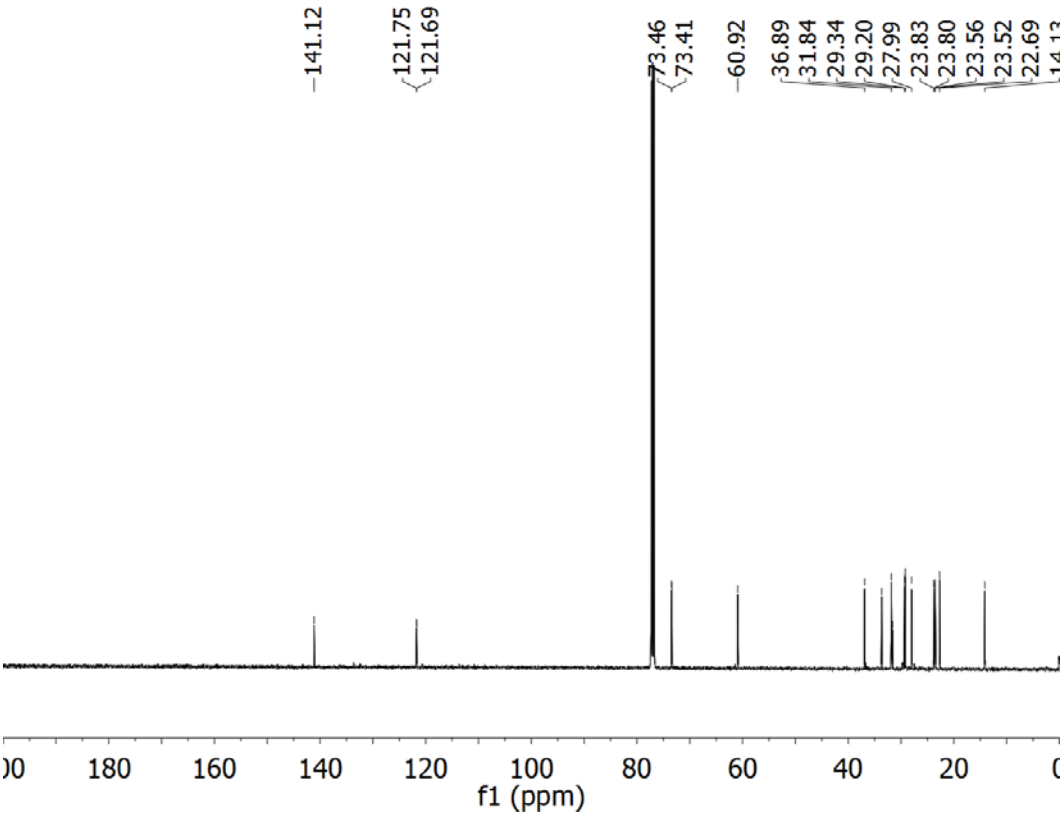

142.80
139.61
129.05
127.88
126.45
114.81
78.24
77.32
77.00
76.68
59.60
41.08
39.30
34.23
32.83
32.81
23.77
23.69
23.66
180
160
140
120
100
80
60
40
20
f1 (ppm)

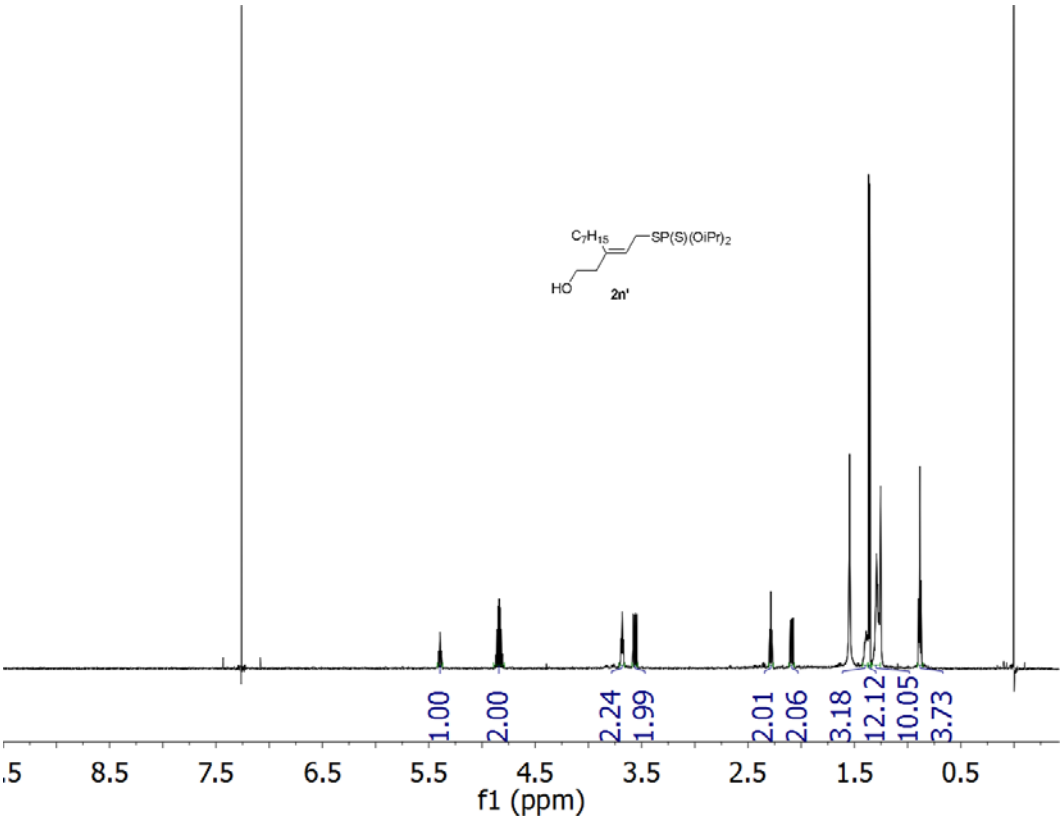

Ph
O
1m
2.06
1.95
0.97
1.00
1.06
1.06
1.08
1.07
1.13
3.32
2.27
2.32
2.03
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

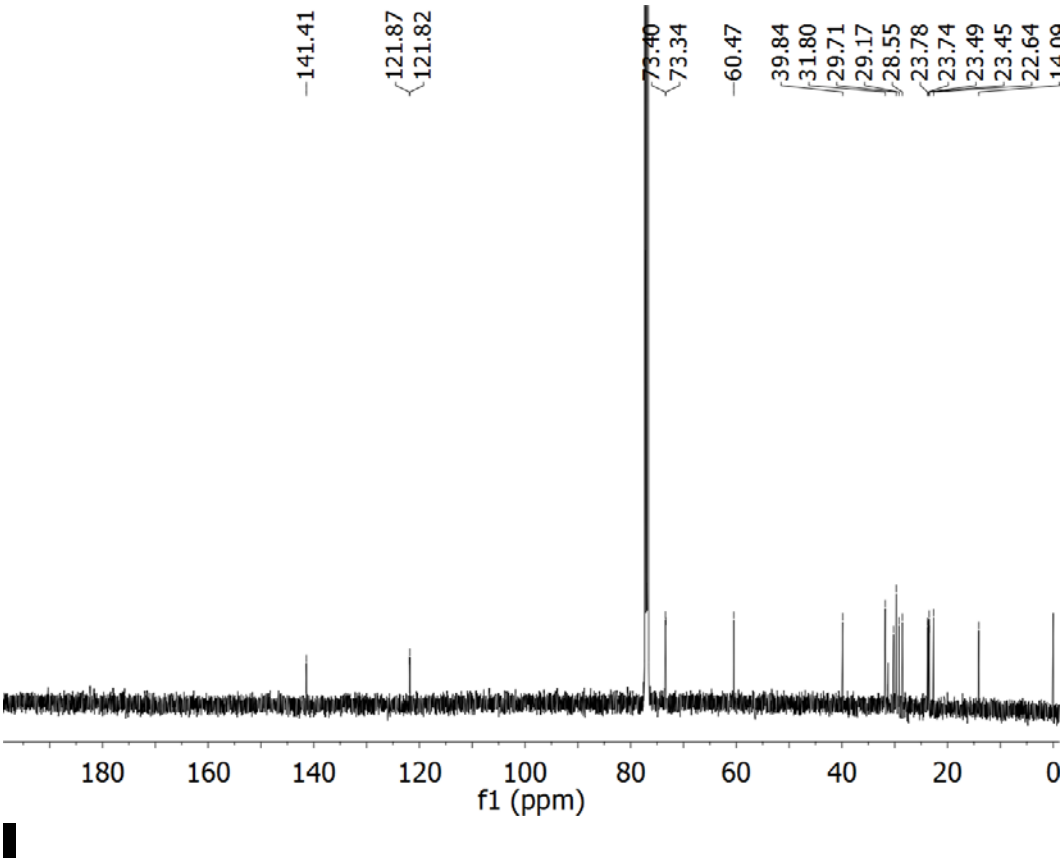

143.89
140.47
127.99
127.71
125.99
112.64
109.98
91.10
77.25
77.00
76.75
64.22
57.67
33.06
31.28
29.87
24.13
24.05
180
160
140
120
100
80
60
40
20
f1 (ppm)

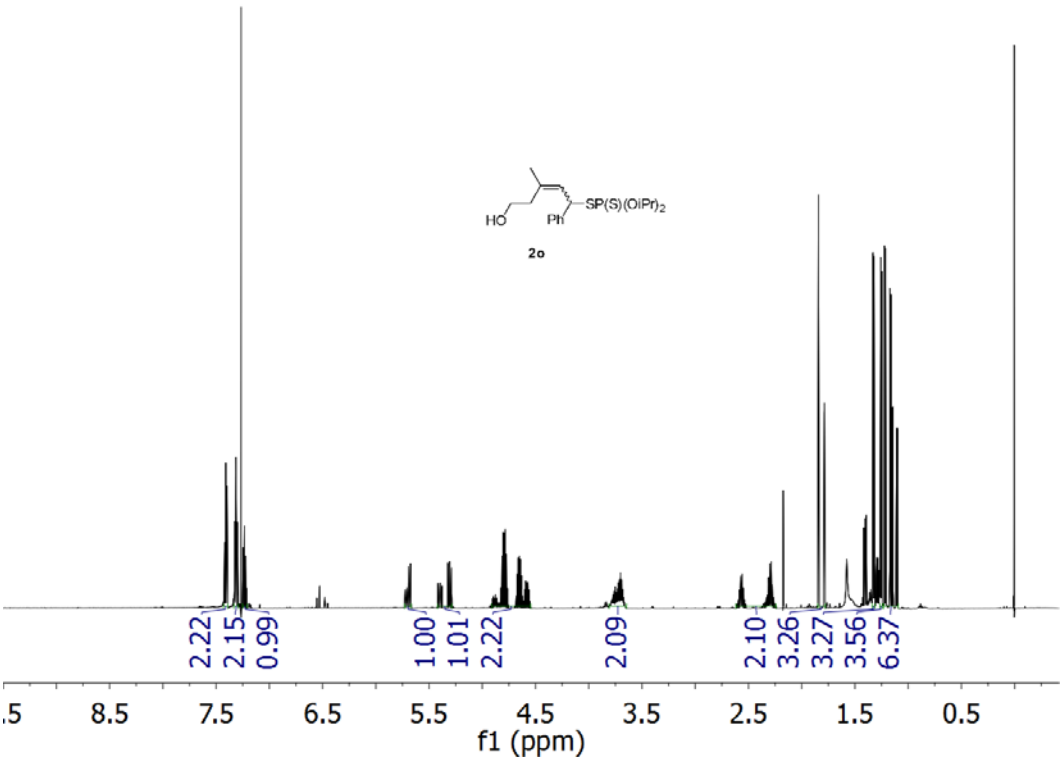

Ph
HO
2m
SP(S)(OiPr)2
1.70
2.06
1.01
1.00
1.91
2.05
2.03
2.09
2.15
2.06
2.26
3.17
12.17
f1 (ppm)
145.63
145.34
128.12
126.97
126.15
120.31
120.23
77.32
77.00
76.68
73.46
73.40
62.13
57.93
36.25
33.27
32.21
23.79
23.75
23.55
23.49
22.40
f1 (ppm)

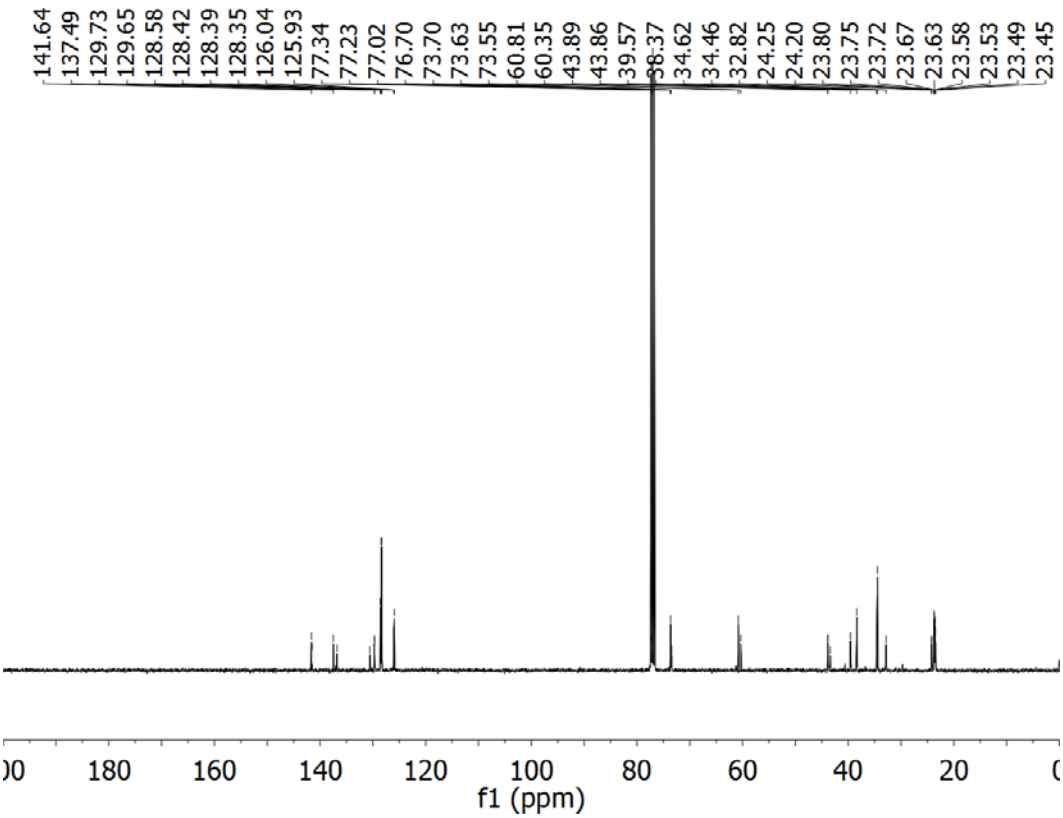

C7H15
SP(S)(OiPr)2
HO
2n
1.00
1.85
2.16
1.96
1.96
1.97
2.75
12.25
10.29
3.63
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

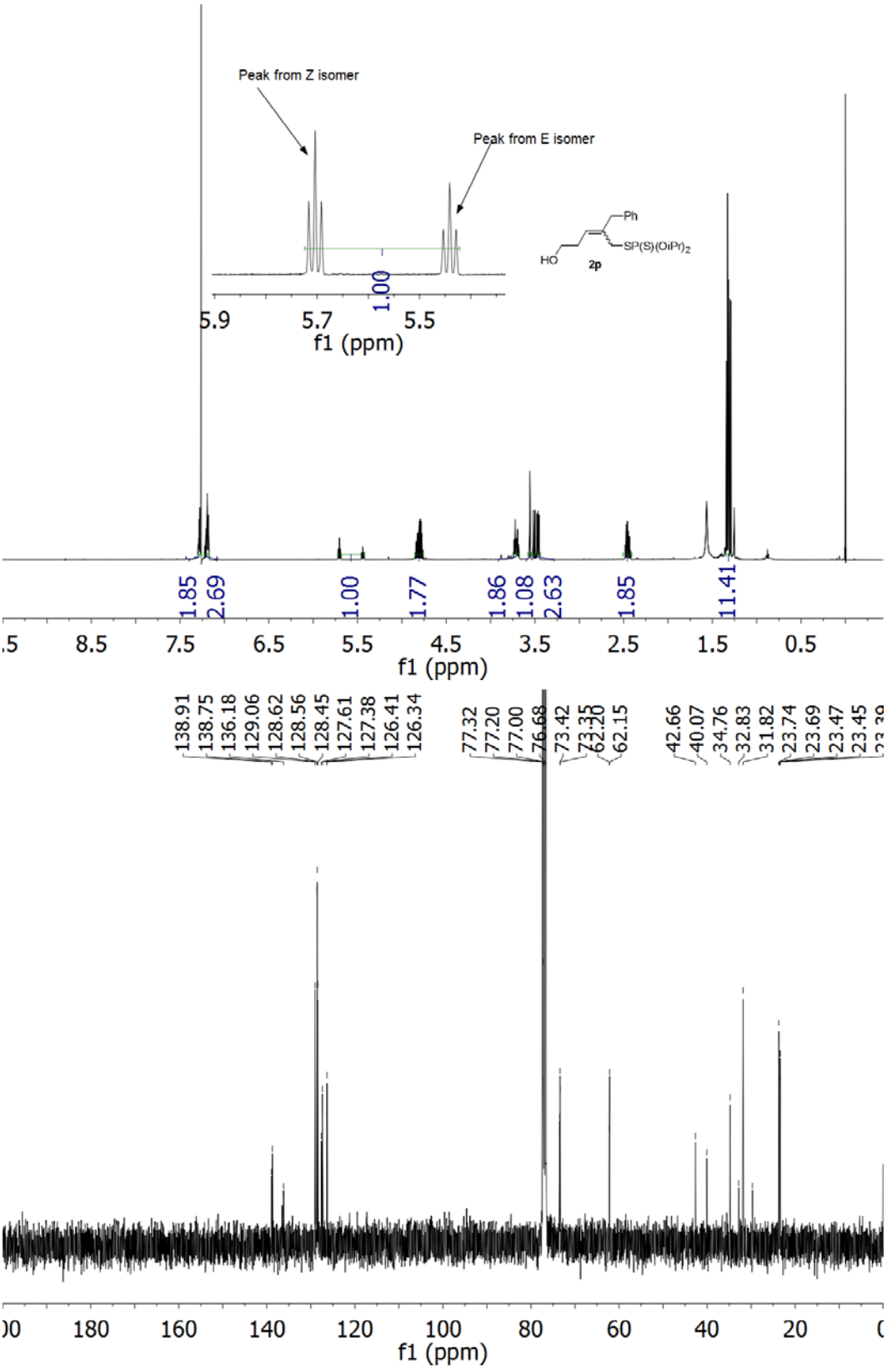

141.12
121.75
121.69
73.46
73.41
60.92
36.89
31.84
29.34
29.20
27.99
23.83
23.80
23.56
23.52
22.69
180
160
140
120
100
80
60
40
20
f1 (ppm)

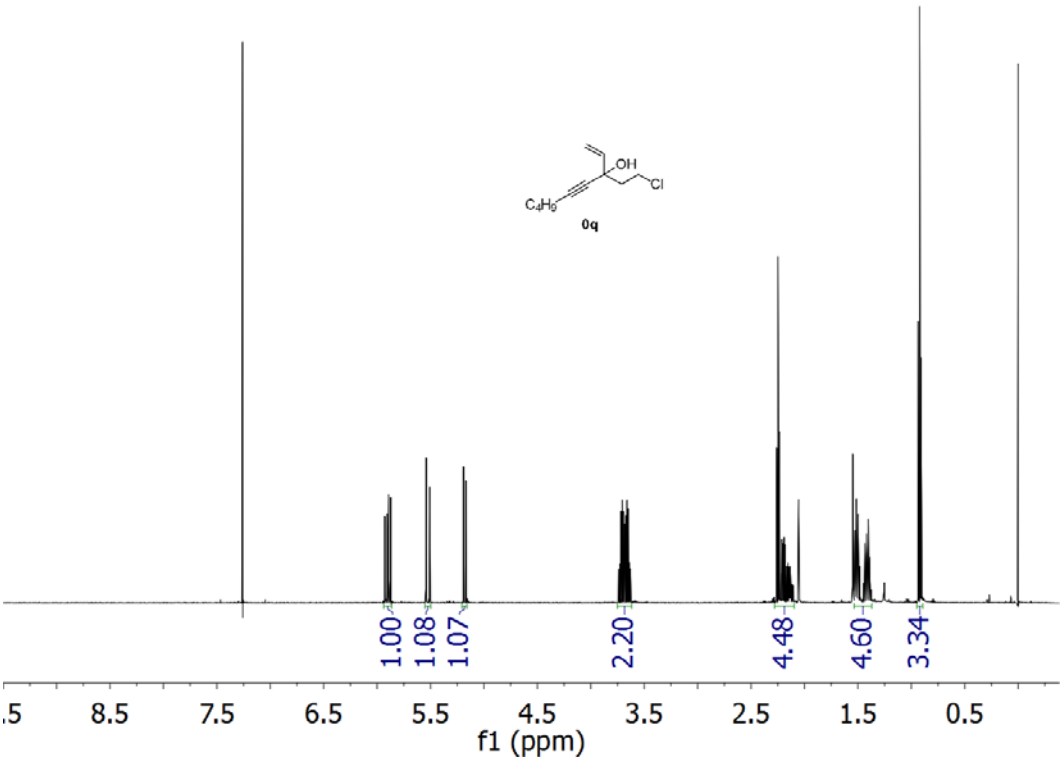

C7H15
SP(S)(OiPr)2
HO
2n'
1.00
2.00
2.24
1.99
2.01
2.06
3.18
12.12
10.05
3.73
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

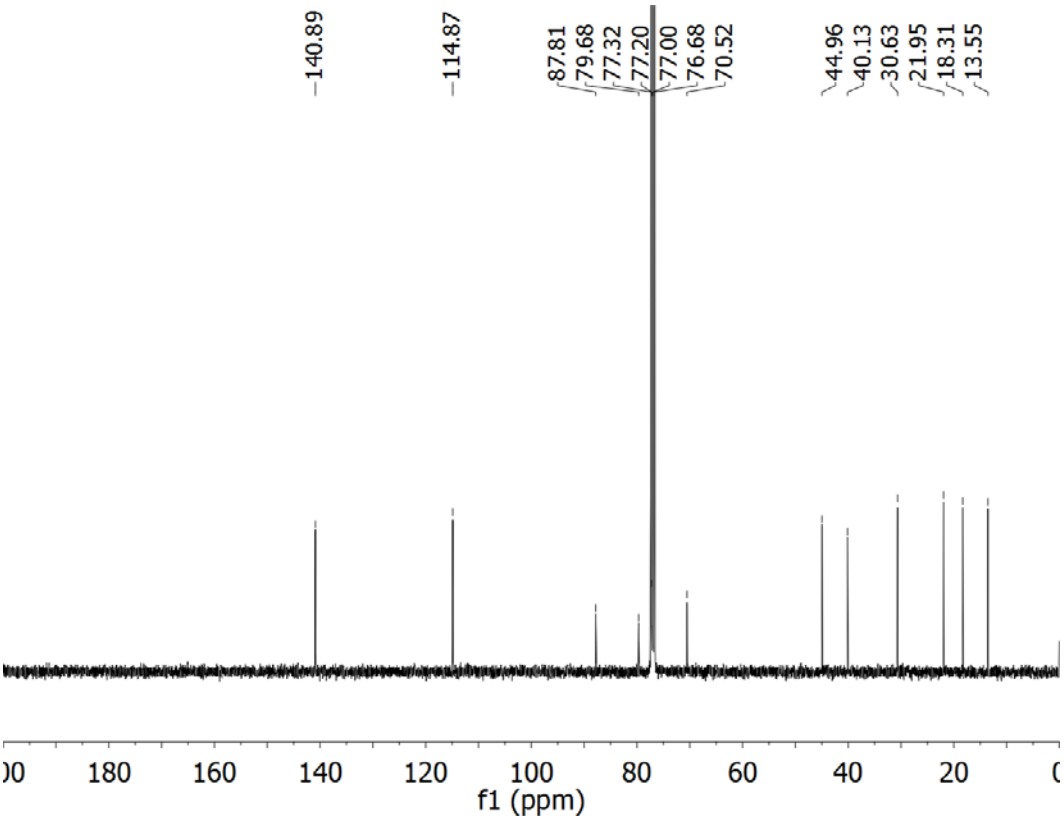

141.41
121.87
121.82
73.40
73.34
60.47
39.84
31.80
29.71
29.17
28.55
23.78
23.74
23.49
23.45
22.64
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

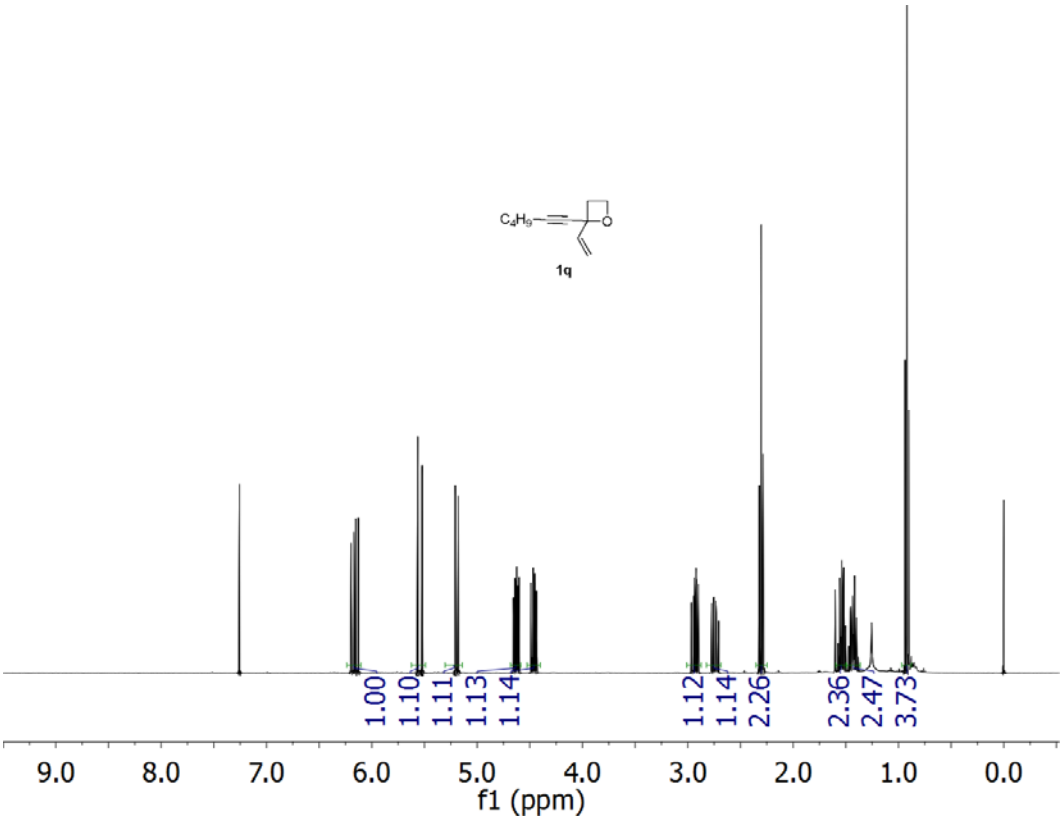

HO
Ph
SP(S)(OiPr)2
2o
2.22
2.15
0.99
1.00
1.01
2.22
2.09
2.10
3.26
3.27
3.56
6.37
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

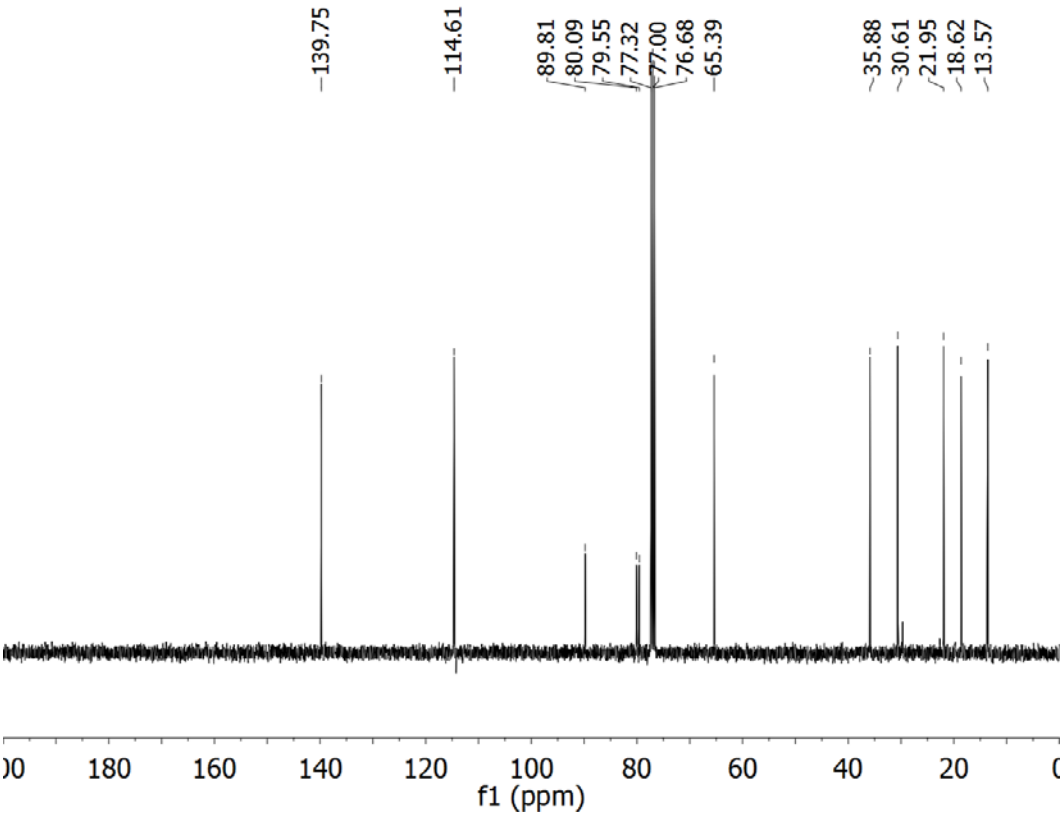

141.64
137.49
129.73
129.65
128.58
128.42
128.39
128.35
126.04
125.93
77.34
77.23
77.02
76.70
73.70
73.63
73.55
60.81
60.35
43.89
43.86
39.57
38.37
34.62
34.46
32.82
24.25
24.20
23.80
23.75
23.72
23.67
23.63
23.58
23.53
23.49
23.45
180
160
140
120
100
80
60
40
20
f1 (ppm)

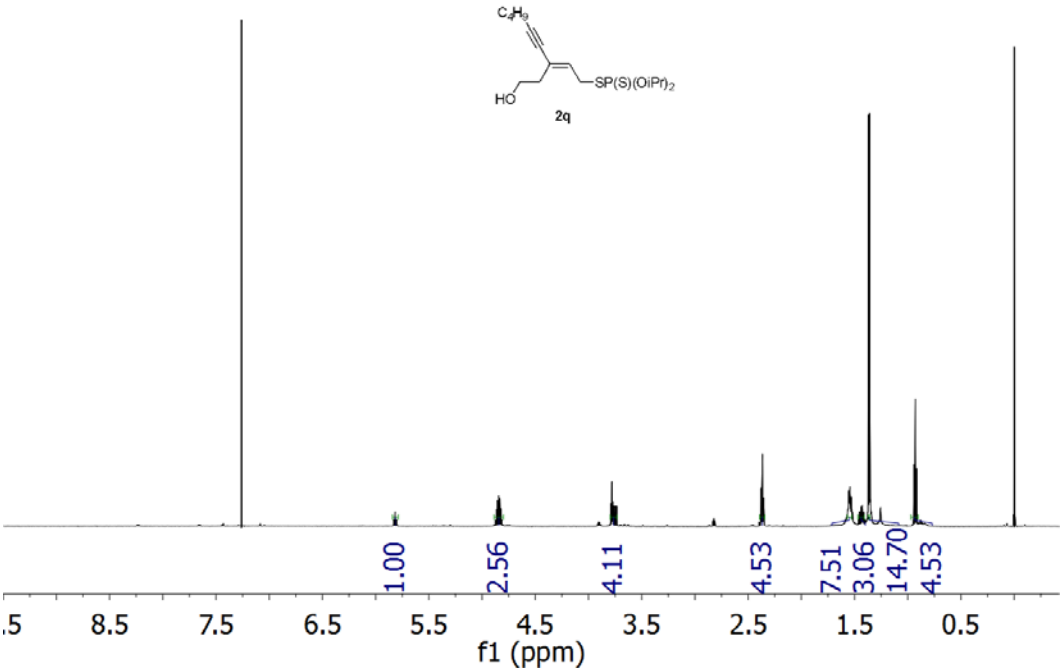

Peak from Z isomer
Peak from E isomer
Ph
SP(S)(OiPr)2
HO
2p
1.00
5.9
5.7
5.5
f1 (ppm)
1.85
2.69
1.00
1.77
1.86
1.08
2.63
1.85
11.41
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)
138.91
138.75
136.18
129.06
128.62
128.56
128.45
127.61
127.38
126.41
126.34
77.32
77.20
77.00
76.68
73.42
62.15
42.66
40.07
34.76
32.83
31.82
23.74
23.69
23.47
23.45
180
160
140
120
100
80
60
40
20
f1 (ppm)

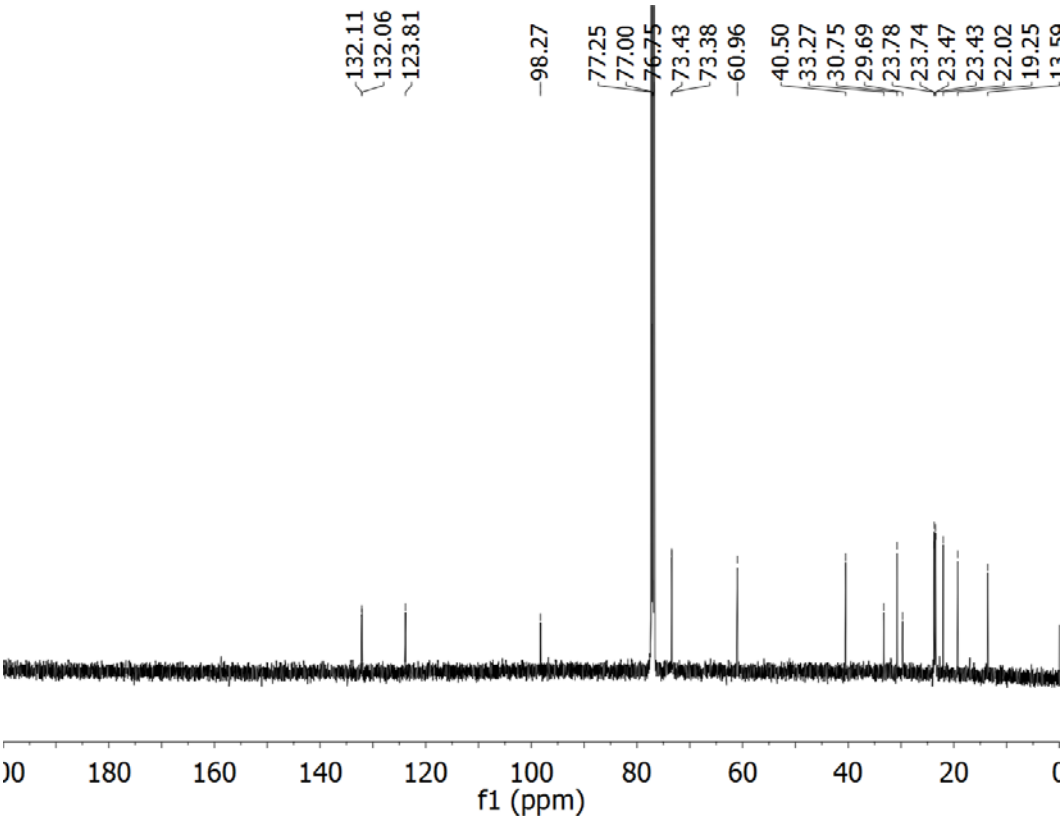

OH
Cl
0q
1.00
1.08
1.07
2.20
4.48
4.60
3.34
f1 (ppm)

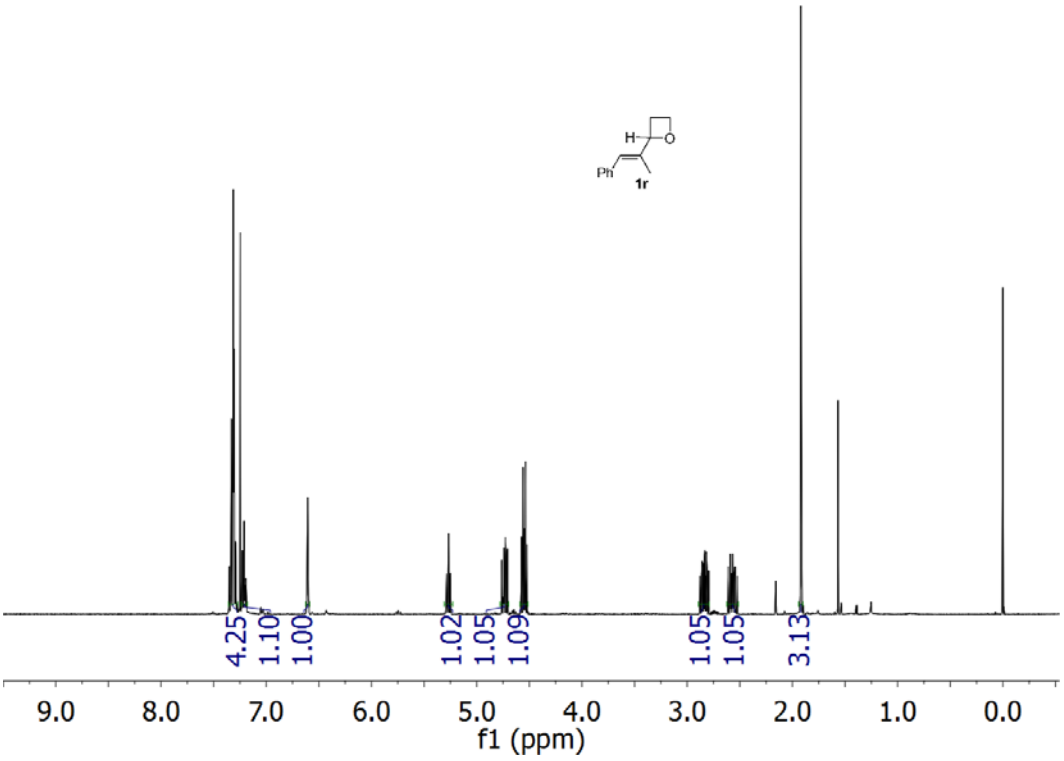

140.89
114.87
87.81
79.68
77.32
77.20
77.00
76.68
70.52
44.96
40.13
30.63
21.95
18.31
13.55
f1 (ppm)

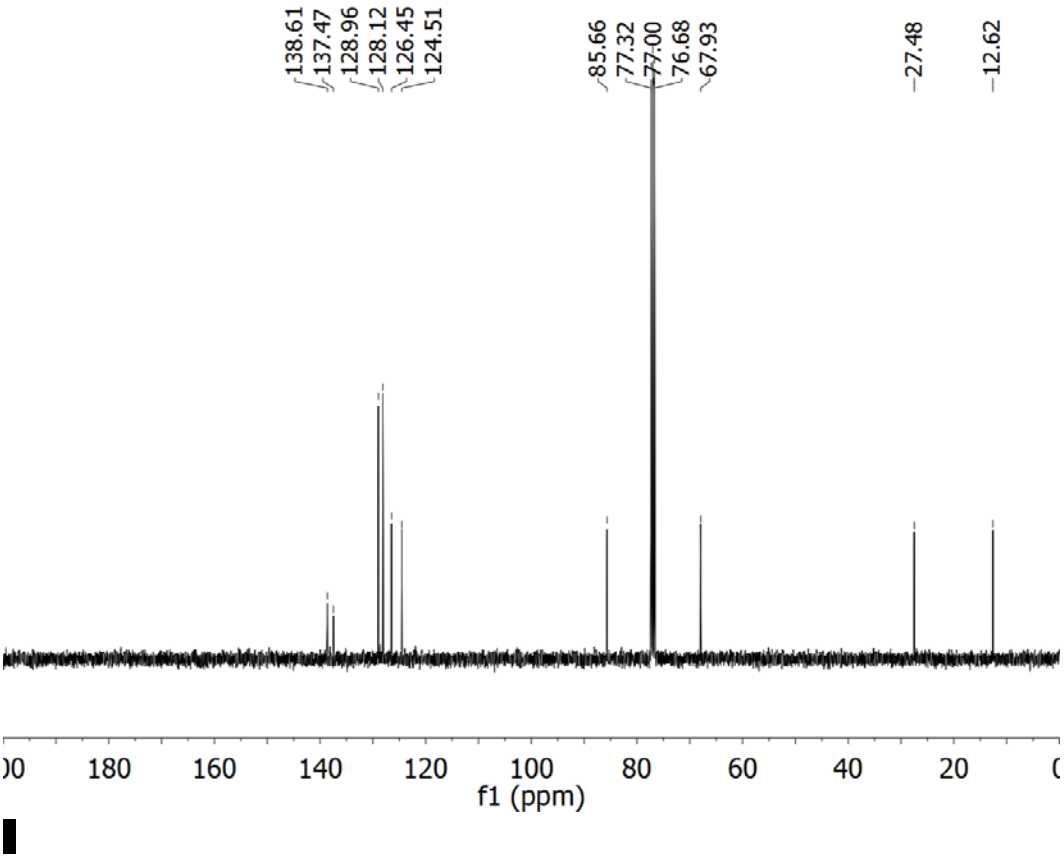


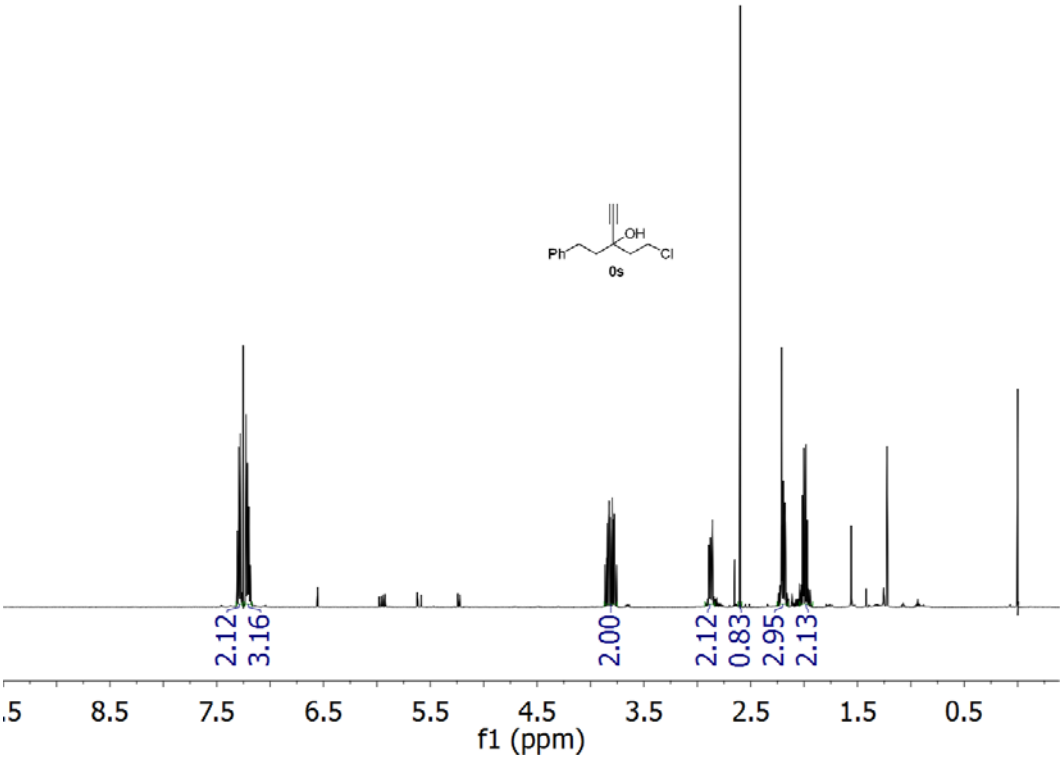


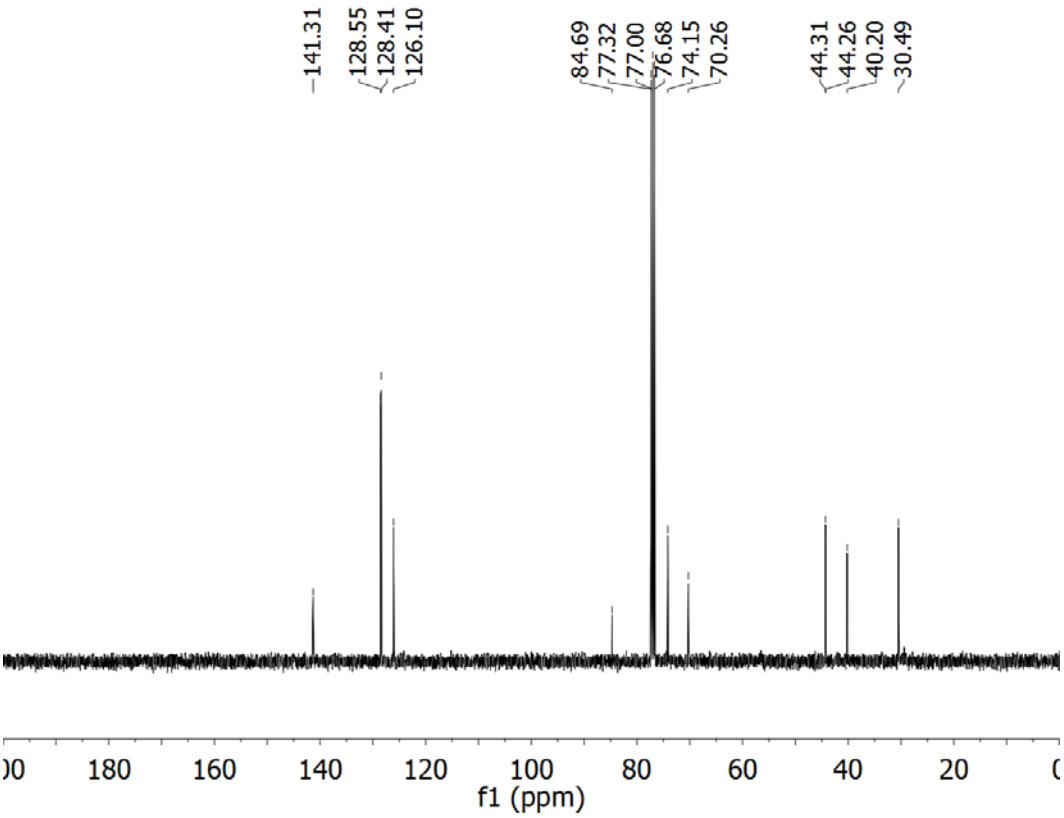

C4H9
SP(S)(OiPr)2
HO
2q
1.00
2.56
4.11
4.53
7.51
3.06
14.70
4.53
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

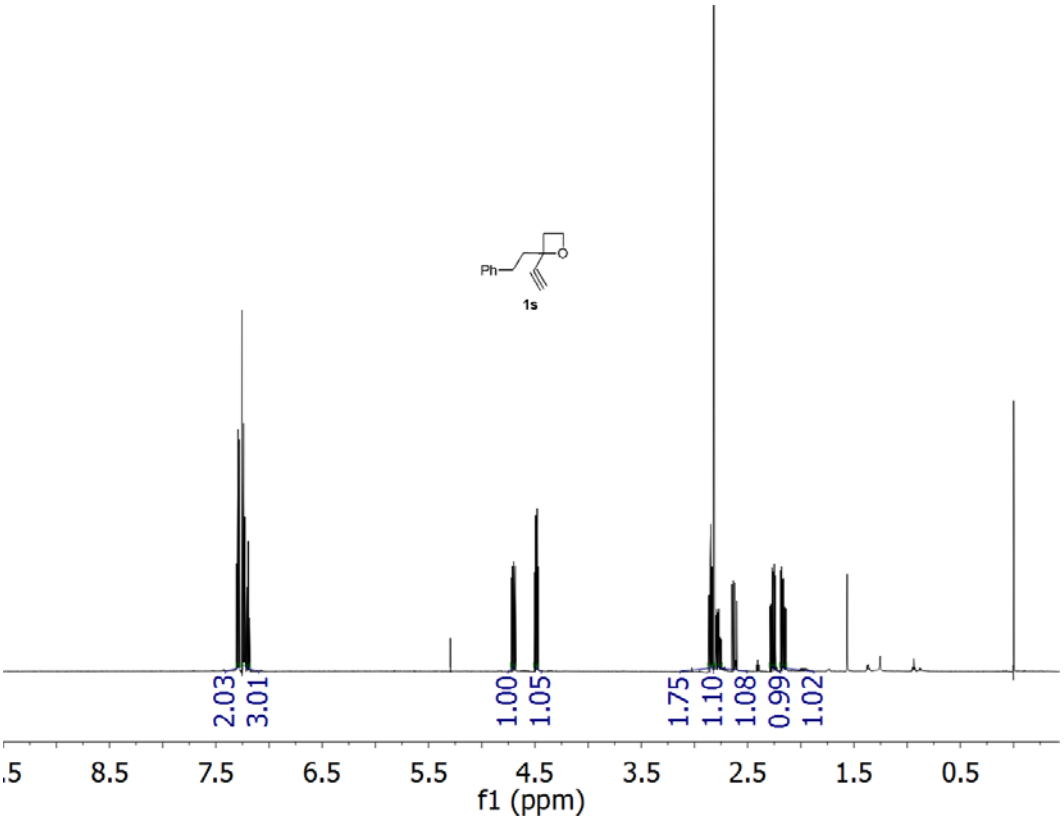

132.11
132.06
123.81
98.27
77.25
77.00
76.75
73.43
73.38
60.96
40.50
33.27
30.75
29.69
23.78
23.74
23.47
23.43
22.02
19.25
180
160
140
120
100
80
60
40
20
f1 (ppm)

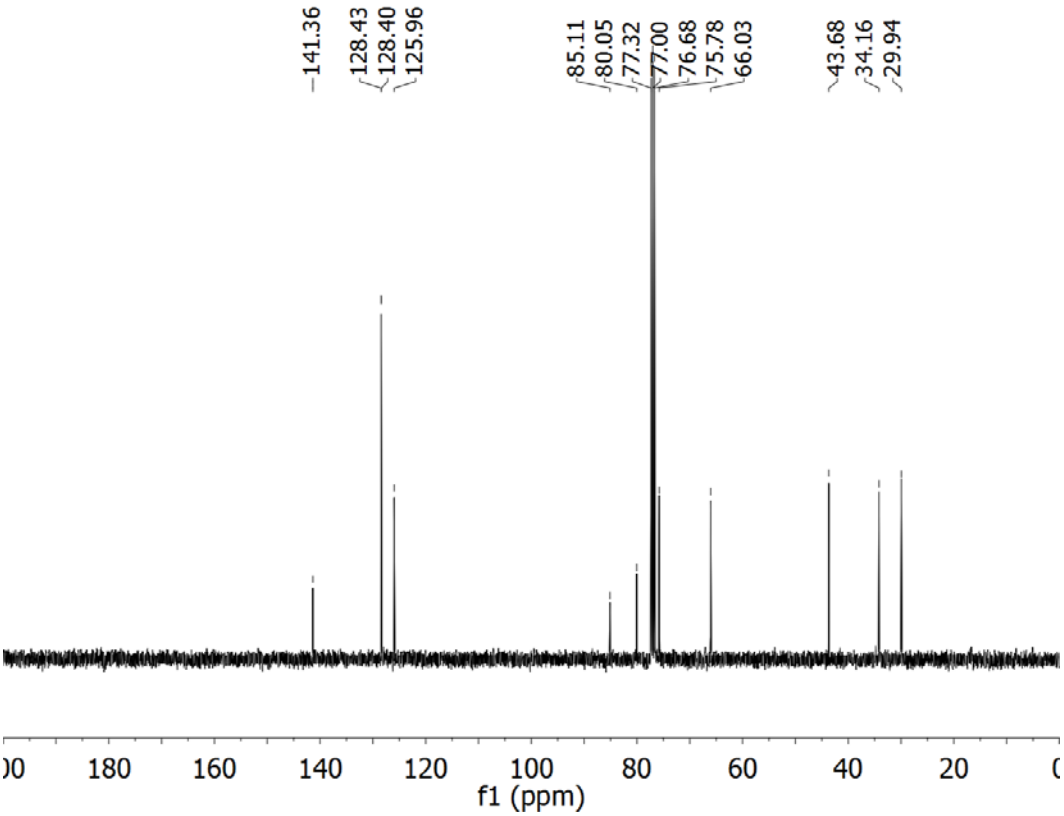

H
O
Ph
1r
4.25
1.10
1.00
1.02
1.05
1.09
1.05
1.05
3.13
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


138.61
137.47
128.96
128.12
126.45
124.51
85.66
77.32
77.00
76.68
67.93
27.48
12.62
180
160
140
120
100
80
60
40
20
f1 (ppm)






Ph
1s
2.03
3.01
1.00
1.05
1.75
1.10
1.08
0.99
1.02
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


141.36
128.43
128.40
125.96
85.11
80.05
77.32
77.00
76.68
75.78
66.03
43.68
34.16
29.94
180
160
140
120
100
80
60
40
20
f1 (ppm)