Electronic Supplementary Material (ESI) for ChemComm.

Supporting information

Synergistic Pd/Cu-catalysed regio- and stereoselective borylation of allenylic carbonates

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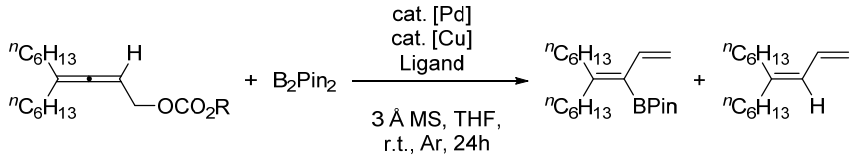
General Information. ^{1}H NMR and ^{13}C NMR spectra were recorded on BRUKER AVANCE III (500 MHz) spectrometers. ^{11}B NMR spectra were recorded on BRUKER AVANCE III (400 MHz). ^{1}H NMR spectra are reported as follows: chemical shift in ppm (δ) relative to the chemical shift of $CDCl_3$ at 7.26 ppm, integration, multiplicities (s = singlet, d =doublet, t = triplet, m = multiplet, appt = apparently triplet), and coupling constants (Hz). ^{13}C NMR spectra were recorded on BRUKER AVANCE III (126MHz) spectrometers with complete proton decoupling, and chemical shift reported in ppm (δ) relative to the central line of triplet for $CDCl_3$ at 77.0 ppm. ^{11}B NMR chemical shifts were quoted relative to $BF_3 \cdot Et_2O$ as external standard. High-resolution mass spectra (HRMS) were obtained on a BRUKER autoflex maX MALDI-TOF(TOF) instrument. Column chromatography and filtration via silica plug were carried out employing silica gel (Qingdao Haiyang Chem, neutral, 300-400 Mesh). Analytical thin-layer chromatography (TLC) was performed on 0.2 mm precoated plate Kieselgel 60 F254 (Merck).

Materials. Unless otherwise noted, commercially available chemicals were used as received. The products **3e** and **3k** were unambiguously confirmed by ^{1}H- and ^{13}C NMR comparing with the reported literature.[1] The structures of new products were determined by ^{1}H, ^{13}C , ^{11}B NMR, and high-resolution mass.

Table S1. Detailed optimization of reaction conditions[a]



1
R = ^{i}Pr, **1a**
R = Me,**1a-Me**

2a
(1.5 equiv)

3a

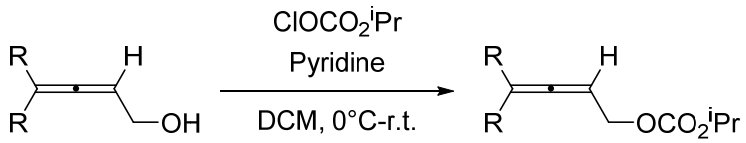
6

Entry	**1**	[Pd]/ 2.5 mol%	[Cu]/ 5 mol%	Ligand/ 15 mol%	Conversion of **1** / %[b]	Yield of **3a** / %[b]	Yield of **6** / %[b]
1	**1a**	Pd_2dba_3	CuI	PPh_3	100	50	18
2	**1a**	Pd_2dba_3	CuI	$P(2\text{-furyl})_3$	89	61	12
3	**1a**	Pd_2dba_3	CuI	$P(C_6H_4\text{-}p\text{-}OMe)_3$	100	29	7
4[c]	**1a**	Pd_2dba_3	CuI	dppbz	60	34	19
5[c]	**1a**	Pd_2dba_3	CuI	dppe	92	49	43
6	**1a**	Pd_2dba_3	CuI	$P(OMe)_3$	100	81	11
7	**1a**	Pd_2dba_3	CuI	$P(OEt)_3$	100	86 (71)	7
8	**1a**	Pd_2dba_3	CuI	$P(O^{i}Pr)_3$	100	78	20
9	**1a**	Pd_2dba_3	CuI	$P(OPh)_3$	64	19	2
10[d]	**1a**	$PdCl_2$	CuI	$P(OEt)_3$	11	0	0
11[d]	**1a**	$Pd(OAc)_2$	CuI	$P(OEt)_3$	100	5	13
12	**1a**	Pd_2dba_3	CuCl	$P(OEt)_3$	100	15	5
13	**1a**	Pd_2dba_3	CuCN	$P(OEt)_3$	87	6	2
14[e]	**1a**	Pd_2dba_3	CuI	$P(OEt)_3$	100	9	24
15[f]	**1a**	Pd_2dba_3	CuI	$P(OEt)_3$	100	43	11
16[g]	**1a**	Pd_2dba_3	CuI	$P(OEt)_3$	25	14	4
17[h]	**1a**	Pd_2dba_3	CuI	$P(OEt)_3$	100	21	18
18[i]	**1a**	Pd_2dba_3	CuI	$P(OEt)_3$	100	83	10
19[j]	**1a**	Pd_2dba_3	CuI	$P(OEt)_3$	100	40	20
20[k]	**1a**	Pd_2dba_3	-	$P(OEt)_3$	6	0	0
21[k]	**1a**	-	CuI	$P(OEt)_3$	4	0	0
22[k,l]	**1a**	-	CuI/ *t*-BuOK	$P(OEt)_3$	10	7	1
23	**1a-Me**	Pd_2dba_3	CuI	$P(OEt)_3$	100	79	9

[a] General reaction condition: a mixture of **1** (0.2 mmol), B_2Pin_2 (**2a,** 0.3 mmol, 1.5 equiv), Pd catalyst (2.5 mol%), Cu catalyst (5 mol%), Ligand (15 mol%) and 3Å MS in THF (0.1 M, 2 mL) was reacted at r.t. for 24 h under Ar. [b] The conversions of **1** and yields of **3a**/**6** were determined by crude ^{1}H NMR spectrum with naphthalene as an internal standard, the yield of isolated **3a** is given in parentheses. [c] 10 mol% ligand was used in the reaction. [d] 5 mol% Pd catalyst was used in the reaction. [e] Toluene was used in the reaction. [f] 1,4-Dioxane was used in the reaction. [g] The reaction was carried out at 0 C. [h] The reaction was carried out at 50 C. [i] 3Å MS was omitted in the reaction. [j] 10 μL water was added in the reaction. [k] 10 mol% $P(OEt)_3$ was used in the reaction. [l] 10 mol% *t*-BuOK was used in the reaction. dppbz = 1,2 - bis(diphenylphosphino)benzene. dppe = 1,2 - bis(diphenylphosphino)ethane.

Experimental Procedures and Spectral Data

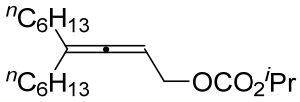
1. Synthesis of Allenylic Carbonates 1



S1 **1**

The allenylic carbonates **1** were synthesized according to reported procedures by Glorius and co-works.[2] To a round bottom-flask equipped with a Teflon coated stirrer bar were added 2,3-allenol (**S1**, 1.0 equiv), pyridine (3.0 equiv) and dichloromethane (0.25 M) under Ar atmosphere. The mixture was cooled to 0 °C and the isopropyl chloroformate (2.0 equiv) was slowly added via syringe. The reaction was allowed to stir at room temperature until completion (monitored by TLC). The mixture was diluted with dichloromethane and washed successively with saturated NH_4Cl solution and brine. The organic phase was dried over $MgSO_4$, filtered and evaporated under reduced pressure. Then, the residue was purified by flash column chromatography using a mixture of petroleum ether/EtOAc as eluent to yield the desired products **1**.

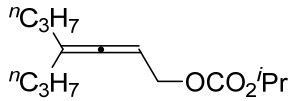
4-Hexyldeca-2,3-dien-1-yl isopropyl carbonate (1a)



1a

Yield, 86%. Colorless oil. NMR data:^{1}H NMR (500 MHz, $CDCl_3$) δ 5.30-5.20 (m, 1H), 4.87 (septet, *J* = 6.3 Hz, 1H), 4.55 (d, *J* = 6.8 Hz, 2H), 1.98-1.88 (m, 4H), 1.44-1.34 (m, 4H), 1.34-1.21 (m, 18H), 0.88 (t, *J* = 6.7 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 202.8, 154.5, 106.7, 87.1, 71.7, 66.6, 32.2, 31.6, 28.9, 27.4, 22.6, 21.7, 14.0; HRMS (ESI): m/z calcd. for $[C_{20}H_{36}O_3Na]^+$ 347.25567, found 347.25470.

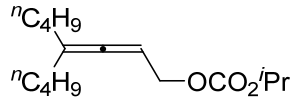
Isopropyl (4-propylhepta-2,3-dien-1-yl) carbonate (1b)



1b

Yield, 81%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.30-5.23 (m, 1H), 4.87 (septet, *J* = 6.3 Hz, 1H), 4.56 (d, *J* = 6.8 Hz, 2H), 1.92 (appt, *J* = 7.3 Hz, 4H), 1.43 (tq, *J* = 7.3, 7.3 Hz, 4H), 1.30 (d, *J* = 6.2 Hz, 6H), 0.90 (t, *J* = 7.3 Hz, 6H);^{13}C NMR (126 MHz, $CDCl_3$) δ 202.8, 154.5, 106.2, 87.1, 71.7, 66.5, 34.3, 21.7, 20.6, 13.7; HRMS (ESI): m/z calcd. for $[C_{14}H_{24}O_3Na]^+$ 263.16177, found 263.16092.

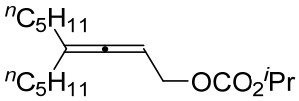
4-Butylocta-2,3-dien-1-yl isopropyl carbonate (1c)



1c

Yield, 84%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.30-5.22 (m, 1H), 4.87 (septet, *J* = 6.3 Hz, 1H), 4.56 (d, *J* = 6.8 Hz, 2H), 1.98-1.90 (m, 4H), 1.43-1.34 (m 4H), 1.34-1.26 (m, 4H), 1.30 (d, *J* = 6.4 Hz, 6H), 0.89 (t, *J* = 7.2 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 202.7, 154.5, 106.6, 87.1, 71.7, 66.5, 31.9, 29.6, 22.3, 21.7, 13.8; HRMS (ESI): m/z calcd. for $[C_{16}H_{28}O_3Na]^+$ 291.19307, found 291.19223.

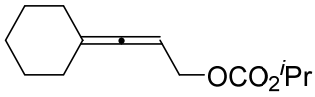
Isopropyl (4-pentylnona-2,3-dien-1-yl) carbonate (1d)



1d

Yield, 89%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.29-5.22 (m, 1H), 4.87 (septet, *J* = 6.3 Hz, 1H), 4.56 (d, *J* = 6.9 Hz, 2H), 1.96-1.90 (m, 4H), 1.45-1.35 (m, 4H), 1.33-1.23 (m, 8H), 1.30 (d, *J* = 6.2 Hz, 6H), 0.88 (t, *J* = 6.8 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 202.8, 154.5, 106.6, 87.1, 71.7, 66.6, 32.1, 31.4, 27.1, 22.4, 21.7, 14.0; HRMS (ESI): m/z calcd. for $[C_{18}H_{32}O_3Na]^+$ 319.22437, found 319.22348.

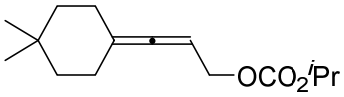
3-Cyclohexylideneallyl isopropyl carbonate (1e)



1e

Yield, 79%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.18-5.11 (m, 1H), 4.87 (septet, *J* = 6.3 Hz, 1H), 4.55 (d, *J* = 6.9 Hz, 2H), 2.15-2.08 (m, 4H), 1.62-1.54 (m, 4H), 1.54-1.47 (m, 2H), 1.30 (d, *J* = 6.3 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 200.3, 154.4, 104.4, 84.2, 71.7, 66.6, 32.4, 31.0, 27.1, 25.9, 21.7; HRMS (ESI): m/z calcd. for $[C_{13}H_{20}O_3Na]^+$ 247.13047, found 247.12973.

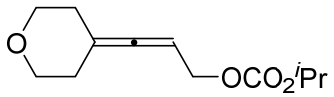
3-(4,4-Dimethylcyclohexylidene)allyl isopropyl carbonate (1f)



1f

Yield, 87%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.18-5.09 (m, 1H), 4.86 (septet, *J* = 6.3 Hz, 1H), 4.54 (d, *J* = 6.9 Hz, 2H), 2.20-2.07 (m, 4H), 1.37 (t, *J* = 6.3 Hz, 4H), 1.29 (d, *J* = 6.3 Hz, 6H), 0.92 (s, 3H), 0.91 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 200.2, 154.4, 103.9, 84.2, 71.7, 66.6, 39.5, 29.9, 28.0(6), 28.0(4), 26.8, 21.7; HRMS (ESI): m/z calcd. for $[C_{15}H_{24}O_3Na]^+$ 275.16177, found 275.16086.

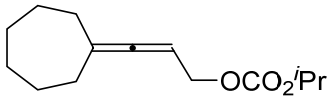
Isopropyl (3-(tetrahydro-4H-pyran-4-ylidene)allyl) carbonate (1g)



1g

Yield, 82%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.30-5.20 (m, 1H), 4.87 (septet, *J* = 6.3 Hz, 1H), 4.57 (d, *J* = 6.7 Hz, 2H), 3.78-3.69 (m, 4H), 2.30-2.20 (m, 4H), 1.30 (dd, *J* = 6.3 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 200.3, 154.4, 100.0, 85.8, 71.8, 68.3, 65.9, 31.0, 21.7; HRMS (ESI): m/z calcd. for $[C_{12}H_{18}O_4Na]^+$ 249.10973, found 249.10918.

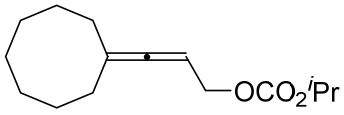
3-Cycloheptylideneallyl isopropyl carbonate (1h)



1h

Yield, 92%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.15-5.09 (m, 1H), 4.87 (septet, *J* = 6.2 Hz, 1H), 4.55 (d, *J* = 6.8 Hz, 2H), 2.32-2.16 (m, 4H), 1.65-1.58 (m, 4H), 1.57-1.50 (m, 4H), 1.30 (d, *J* = 6.2 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 203.5, 154.4, 106.5, 84.0, 71.7, 66.5, 32.0, 29.2, 28.2, 21.7; HRMS (ESI): m/z calcd. for $[C_{14}H_{22}O_3Na]^+$ 261.14612, found 261.14536.

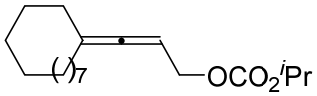
3-Cyclooctylideneallyl isopropyl carbonate (1i)



1i

Yield, 93%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.19-5.11 (m, 1H), 4.87 (septet, *J* = 6.2 Hz, 1H), 4.56 (d, *J* = 6.9 Hz, 2H), 2.22-2.11 (m, 4H), 1.68-1.48(m, 10H), 1.30 (d, *J* = 6.3 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 203.7, 154.5, 106.5, 84.7, 71.7, 66.1, 31.2, 26.7, 26.6, 26.0, 21.7; HRMS (ESI): m/z calcd. for $[C_{15}H_{24}O_3Na]^+$ 275.16177, found 275.16119.

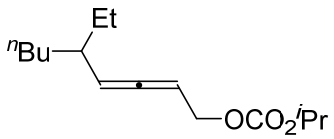
3-Cyclododecylideneallyl isopropyl carbonate (1j)



1j

Yield, 89%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.28-5.21 (m, 1H), 4.88 (septet, *J* = 6.3 Hz, 1H), 4.58 (d, *J* = 6.9 Hz, 2H), 2.08-1.97 (m, 4H), 1.51-1.43 (m, 4H), 1.37-1.24 (m, 14H), 1.30 (d, *J* = 6.3 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 203.8, 154.5, 103.6, 86.4, 71.7, 66.3, 29.3, 24.4, 24.2, 23.8, 22.9, 22.2, 21.7; HRMS (ESI): m/z calcd. for $[C_{19}H_{32}O_3Na]^+$ 331.22437, found 331.22342.

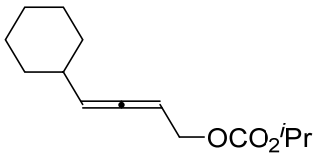
5-Ethylnona-2,3-dien-1-yl isopropyl carbonate (1k)



1k

Yield, 91%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.30-5.25 (m, 1H), 5.13-5.04 (m, 1H), 4.87 (septet, *J* = 6.3 Hz, 1H), 4.58 (d, *J* = 6.5 Hz, 2H), 2.00-1.90 (m, 1H), 1.47-1.19 (m, 14H), 0.93-0.80 (m, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 205.3(8), 205.3(6), 154.4, 96.9(6), 96.9(4), 86.4(5), 86.4(1), 71.8, 66.1, 40.8, 40.7, 34.4, 34.2, 29.3, 29.2, 27.9, 27.8, 22.7, 22.6, 21.7, 14.0, 11.5, 11.4; HRMS (ESI): m/z calcd. for $[C_{15}H_{26}O_3Na]^+$ 277.17742, found 277.17648.

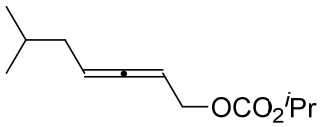
4-Cyclohexylbuta-2,3-dien-1-yl isopropyl carbonate (1l)



1l

Yield, 87%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.35-5.23 (m, 2H), 4.87 (septet, *J* = 6.3 Hz, 1H), 4.58 (dd, *J* = 6.7, 1.6 Hz, 2H), 2.06-1.93 (m, 1H), 1.74 (appt, *J* = 14.5 Hz, 4H), 1.66-1.59 (m, 1H), 1.30 (d, *J* = 6.3 Hz, 6H), 1.27-1.02 (m, 5H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 204.5, 154.4, 99.0, 87.4, 71.8, 66.0, 36.7, 32.8(5), 32.8(2), 26.0, 25.9, 21.7; HRMS (ESI): m/z calcd. for $[C_{14}H_{22}O_3Na]^+$ 261.14612, found 261.14548.

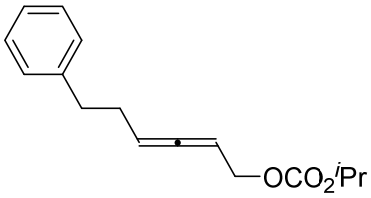
Isopropyl (6-methylhepta-2,3-dien-1-yl) carbonate (1m)



1m

Yield, 85%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.30-5.19 (m, 2H), 4.87 (septet, *J* = 6.3 Hz, 1H), 4.62-4.54 (m, 2H), 1.96-1.85 (m, 2H), 1.66 (septet, *J* = 6.6 Hz, 1H), 1.30 (d, *J* = 6.3 Hz, 6H), 0.91 (d, *J* = 6.5 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 206.1, 154.4, 91.4, 85.8, 71.8, 65.9, 37.6, 28.2, 22.1, 22.0, 21.7; HRMS (ESI): m/z calcd. for $[C_{12}H_{20}O_3Na]^+$ 235.13047, found 235.13020.

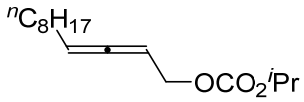
Isopropyl (6-phenylhexa-2,3-dien-1-yl) carbonate (1n)



1n

Yield, 88%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30-7.26 (m, 2H), 7.21-7.15 (m, 3H), 5.36-5.25 (m, 2H), 4.87 (septet, *J* = 6.4 Hz, 1H), 4.52 (dd, *J* = 6.8, 2.4 Hz, 2H), 2.80-2.64 (m, 2H), 2.38-2.27 (m, 2H), 1.29 (d, *J* = 6.3 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 205.7, 154.4, 141.3, 128.4, 128.2, 125.9, 92.2, 87.0, 71.9, 65.7, 35.1, 29.8, 21.7; HRMS (ESI): m/z calcd. for $[C_{16}H_{20}O_3Na]^+$ 283.13047, found 283.12955.

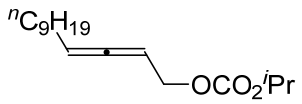
Dodeca-2,3-dien-1-yl isopropyl carbonate (1o)



1o

Yield, 84%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.30-5.23 (m, 2H), 4.87 (septet, *J* = 6.5 Hz, 1H), 4.58 (dd, *J* = 4.5, 4.5 Hz, 2H), 2.05-1.96 (m, 2H), 1.42-1.34 (m, 2H), 1.34-1.20 (m, 16H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 205.6, 154.4, 93.0, 86.4, 71.8, 65.9, 31.8, 29.3, 29.2, 29.0, 28.9, 28.2, 22.6, 21.7, 14.0; HRMS (ESI): m/z calcd. for $[C_{16}H_{28}O_3Na]^+$ 291.19307, found 291.19205.

Isopropyl trideca-2,3-dien-1-yl carbonate (1p)



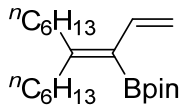
1p

Yield, 87%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.34-5.21 (m, 2H), 4.87 (septet, *J* = 6.2 Hz, 1H), 4.58 (dd, *J* = 4.5, 4.5 Hz, 2H), 2.06-1.96 (m, 2H), 1.44-1.35 (m, 2H), 1.35-1.20 (m, 18H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 205.6, 154.4, 93.0, 86.5, 71.9, 66.0, 31.8, 29.5, 29.4, 29.3, 29.0, 28.9, 28.2, 22.6, 21.7, 14.0; HRMS (ESI): m/z calcd. for $[C_{17}H_{30}O_3Na]^+$ 305.20872, found 305.20795.

2. Synthesis of 2-Bory 1,3-Butadienes 3

An oven-dried Schlenk flask was equipped with a Teflon coated stirrer bar and charged with activated 3Å molecular sieves (3-4 pellets), Pd_2dba_3 (0.005 mmol), CuI (0.01 mol), $P(OEt)_3$ (0.03 mmol), B_2pin_2 **2a** (0.30 mmol), and THF (1 mL) under Ar atmosphere. The resulted solution was stirred for 10 min, then a THF solution (1 mL) of **1** (0.2 mmol) was added to the reaction mixture via syringe. The final reaction mixture was stirred at room temperature for 24 hours. Completion of the reaction was checked by TLC and/or crude ^{1}H NMR spectrum. After a complete conversion of **1**, the reaction mixture was diluted by petroleum ether (about 5 mL). The precipitate was filtered off by a short silica pad (Qingdao Haiyang Chem, neutral, 300-400 Mesh, about 2-3 cm in pipette) using EtOAc/petroleum ether (1:10 v/v) as an eluent. The solvent was removed and the residue was purified by a rapid silica gel chromatography (within 10 min) using a mixture of petroleum ether/EtOAc as eluent affording pure product **3**.

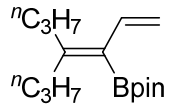
2-(4-Hexyldeca-1,3-dien-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3a)



3a

Yield, 71%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.69 (dd, *J* = 17.6, 10.8 Hz, 1H), 5.16 (dd, *J* = 17.6, 1.8 Hz, 1H), 5.01 (dd, *J* = 10.7, 1.8 Hz, 1H), 2.20-2.12 (m, 4H), 1.43-1.26 (m, 16H), 1.32 (s, 12H), 0.88 (t, *J* = 6.6 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.7, 136.2, 114.3, 83.2, 37.7, 32.0, 31.7(9), 31.7(0), 29.7, 29.6, 29.5, 29.0, 24.9, 22.6, 22.5, 14.0(7), 14.0(5); Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 31. HRMS (ESI): m/z calcd. for $[C_{22}H_{42}BO_2]^+$ 349.32724, found 349.32672.

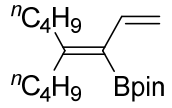
4,4,5,5-Tetramethyl-2-(4-propylhepta-1,3-dien-3-yl)-1,3,2-dioxaborolane (3b)



3b

Yield, 84%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.70 (dd, *J* = 17.6, 10.8 Hz, 1H), 5.17 (dd, *J* = 17.5, 1.8 Hz, 1H), 5.01 (dd, *J* = 10.9, 1.8 Hz, 1H), 2.19-2.11 (m, 4H), 1.51-1.38 (m, 4H), 1.33 (s, 12H), 0.91 (t, *J* = 7.3 Hz, 3H), 0.89 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.1, 136.3, 114.3, 83.2, 39.6, 34.1, 24.9, 22.7, 22.2, 14.4, 14.2; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 31. HRMS (ESI): m/z calcd. for $[C_{16}H_{30}BO_2]^+$ 265.23334, found 265.23306.

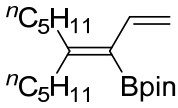
2-(4-Butylocta-1,3-dien-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3c)



3c

Yield, 64%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.70 (dd, *J* = 17.4, 10.9 Hz, 1H), 5.16 (dd, *J* = 17.6, 1.9 Hz, 1H), 5.01 (dd, *J* = 10.9, 1.9 Hz, 1H), 2.22-2.13 (m, 4H), 1.48-1.20 (m, 8H), 1.32 (s, 12H), 0.92-0.86 (m, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.6, 136.3, 114.3, 83.3, 37.4, 31.7(6), 31.7(2), 31.1, 24.9, 23.0, 14.0, 13.9; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 31. HRMS (ESI): m/z calcd. for $[C_{18}H_{33}BO_2Na]^+$ 315.24658, found 315.24628.

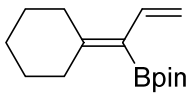
4,4,5,5-Tetramethyl-2-(4-pentylnona-1,3-dien-3-yl)-1,3,2-dioxaborolane (3d)



3d

Yield, 65%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.69 (dd, *J* = 17.5, 10.8 Hz, 1H), 5.17 (dd, *J* = 17.5, 1.8 Hz, 1H), 5.01 (dd, *J* = 10.8, 1.8 Hz, 1H), 2.22-2.10 (m, 4H), 1.46-1.35 (m, 4H), 1.33 (s, 12H), 1.31-1.24 (m, 8H), 0.92-0.84(m, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.7, 136.2, 114.3, 83.2, 37.6, 32.1, 31.9, 29.2, 28.7, 24.9, 22.5(8), 22.5(2), 14.0, 13.9; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 31. HRMS (ESI): m/z calcd. for $[C_{20}H_{38}BO_2]^+$ 321.29594, found 321.29578.

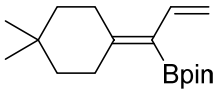
2-(1-Cyclohexylideneallyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3e)



3e

Yield, 61%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.76 (dd, *J* = 17.5, 10.8 Hz, 1H), 5.17 (dd, *J* = 17.5, 1.8 Hz, 1H), 5.01 (dd, *J* = 10.8, 1.8 Hz, 1H), 2.36-2.26 (m, 4H), 1.70-1.53 (m, 6H), 1.38 (s, 12H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.4, 135.5, 114.5, 83.3, 36.3, 30.3, 28.6, 27.9, 26.7, 24.9; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 31. HRMS (ESI): m/z calcd. for $[C_{15}H_{25}BO_2Na]^+$ 271.18398, found 271.18350.

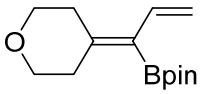
2-(1-(4,4-Dimethylcyclohexylidene)allyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3f)



3f

Yield, 66%. Colorless oil. NMR data:^{1}H NMR (500 MHz, $CDCl_3$) δ 6.76 (dd, *J* = 17.6, 10.8 Hz, 1H), 5.17 (d, *J* = 17.5 Hz, 1H), 5.01 (d, *J* = 10.8 Hz, 1H), 2.37-2.27 (m, 4H), 1.40 (t, *J* = 6.2 Hz, 1H), 1.36 (t, *J* = 6.2 Hz, 1H), 1.32 (s, 12H), 0.94 (s, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.4, 135.5, 114.6, 83.3, 40.9, 40.2, 31.9, 30.4, 28.1, 26.1, 24.9; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 31. HRMS (ESI): m/z calcd. for $[C_{17}H_{29}BO_2Na]^+$ 299.21528, found 299.21512.

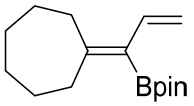
4,4,5,5-Tetramethyl-2-(1-(tetrahydro-4H-pyran-4-ylidene)allyl)-1,3,2-dioxaborolane (3g)



3g

Yield, 72%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.71 (dd, *J* = 17.5, 10.8 Hz, 1H), 5.27 (d, *J* = 17.6 Hz, 1H), 5.09 (d, *J* = 10.8 Hz, 1H), 3.73 (t, *J* = 5.4 Hz, 2H), 3.69 (t, *J* = 5.4 Hz, 2H), 2.48 (t, *J* = 5.4 Hz, 2H), 2.47 (t, *J* = 5.4 Hz, 2H), 1.32 (s, 12H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.9, 134.7, 116.0, 83.5, 69.1, 68.5, 36.0, 31.2, 24.8; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 31. HRMS (ESI): m/z calcd. for $[C_{14}H_{23}BO_3Na]^+$ 273.16325, found 273.16299.

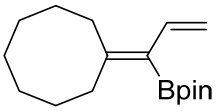
2-(1-Cycloheptylideneallyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3h)



3h

Yield, 76%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (dd, *J* = 17.6, 10.8 Hz, 1H), 5.15 (dd, *J* = 17.6, 1.9 Hz, 1H), 5.01 (dd, *J* = 10.8, 1.8 Hz, 1H), 2.49-2.33 (m, 4H), 1.66-1.55 (m, 4H), 1.53-1.44 (m, 4H), 1.32 (s, 12H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.6, 136.0, 114.3, 83.2, 36.6, 31.9, 29.0, 28.6, 26.5, 24.8; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 31. HRMS (ESI): m/z calcd. for $[C_{16}H_{28}BO_2]^+$ 263.21769, found 263.21713.

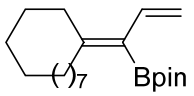
2-(1-Cyclooctylideneallyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3i)



3i

Yield, 67%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.75 (dd, *J* = 17.5, 10.8 Hz, 1H), 5.14 (dd, *J* = 17.6, 1.8 Hz, 1H), 5.01 (dd, *J* = 10.7, 1.8 Hz, 1H), 2.40-2.32 (m, 4H), 1.73-1.63 (m, 4H), 1.54-1.42 (m, 6H), 1.33 (s, 12H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 155.5, 136.3, 113.8, 83.2, 36.8, 31.0, 27.4, 27.3, 26.5, 26.4, 25.8, 24.9; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 31. HRMS (ESI): m/z calcd. for $[C_{17}H_{30}BO_2]^+$ 277.23334, found 277.23300.

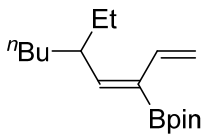
2-(1-Cyclododecylideneallyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3j)



3j

Yield, 74%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74 (dd, *J* = 17.6, 10.8 Hz, 1H), 5.16 (dd, *J* = 17.6, 1.9 Hz, 1H), 5.01 (dd, *J* = 10.8, 1.9 Hz, 1H), 2.24-2.14 (m, 4H), 1.56-1.36 (m, 18H), 1.33 (s, 12H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.2, 136.3, 114.4, 83.3, 33.1, 27.1, 26.0(8), 26.0(0), 25.9, 24.8, 24.5, 24.0, 22.9, 22.8, 22.0(9), 22.0(0); Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 31. HRMS (ESI): m/z calcd. for $[C_{21}H_{38}BO_2]^+$ 333.29594, found 333.29565.

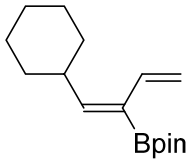
(*Z*)-2-(5-ethylnona-1,3-dien-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3k)



3k

Yield, 84%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.68 (dd, *J* = 17.5, 11.0 Hz, 1H), 6.02 (d, *J* = 10.1 Hz, 1H), 5.66 (dd, *J* = 17.6, 2.5 Hz, 1H), 5.13 (d, *J* = 11.1, 1H), 2.58-2.45 (m, 1H), 1.49-1.37 (m, 2H), 1.29 (s, 12H), 1.27-1.14 (m, 6H), 0.85 (t, *J* = 6.5 Hz, 3H), 0.82 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.1, 134.4, 117.2, 83.0, 39.8, 34.7, 29.6, 28.1, 24.7, 22.8, 14.0, 11.9; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 30. HRMS (ESI): m/z calcd. for $[C_{17}H_{31}BO_2Na]^+$ 310.23093, found 310.23077.

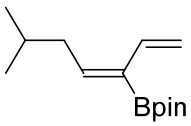
(*Z*)-2-(1-Cyclohexylbuta-1,3-dien-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3l)



3l

Yield, 80%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.69 (dd, *J* = 17.5, 11.0 Hz, 1H), 6.13 (d, *J* = 8.8 Hz, 1H), 5.66 (d, *J* = 17.4 Hz, 1H), 5.14 (d, *J* = 10.8 Hz, 1H), 2.60-2.45 (m, 1H), 1.78-1.50 (m, 6H), 1.28 (s, 12H), 1.21-1.04 (m, 4H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.2, 133.9, 117.3, 83.0, 37.4, 32.4, 25.9, 25.8, 24.7; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 30. HRMS (ESI): m/z calcd. for $[C_{16}H_{27}BO_2Na]^+$ 285.19963, found 285.19922.

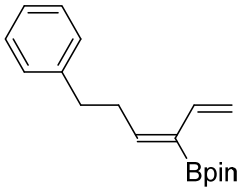
(*Z*)-4,4,5,5-Tetramethyl-2-(6-methylhepta-1,3-dien-3-yl)-1,3,2-dioxaborolane (3m)



3m

Yield, 85%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.69 (dd, *J* = 17.5, 11.0 Hz, 1H), 6.34 (t, *J* = 7.5 Hz, 1H), 5.67 (dd, *J* = 17.5, 2.4 Hz, 1H), 5.16 (d, *J* = 11.0 Hz, 1H), 2.15 (t, *J* = 7.2 Hz, 2H), 1.72 (septet, *J* = 6.7 Hz, 1H), 1.29 (s, 12H), 0.91 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.0, 133.9, 117.4, 83.0, 37.7, 28.4, 24.7, 22.5. Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 30. HRMS (ESI): m/z calcd. for $[C_{14}H_{26}BO_2]^+$ 237.20204, found 237.20161.

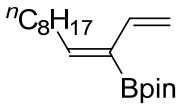
(*Z*)-4,4,5,5-Tetramethyl-2-(6-phenylhexa-1,3-dien-3-yl)-1,3,2-dioxaborolane (3n)



3n

Yield, 66%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31-7.260 (m, 2H), 7.23-7.14 (m, 3H), 6.67 (dd, *J* = 17.5, 11.0 Hz, 1H), 6.39 (t, *J* = 7.1 Hz, 1H), 5.67 (dd, *J* = 17.6, 1.0 Hz, 1H), 5.17 (d, *J* = 11.0 Hz, 1H), 2.77-2.67 (m, 2H), 2.60-2.50 (m, 2H), 1.29 (s, 12H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 146.5, 141.8, 133.5, 128.3, 128.2, 125.8, 117.9, 83.2, 35.3, 30.7, 24.7. Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 30. HRMS (ESI): m/z calcd. for $[C_{18}H_{26}BO_2]^+$ 280.20204, found 280.20154.

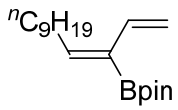
(*Z*)-2-(dodeca-1,3-dien-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3o)



3o

Yield, 72%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.69 (dd, *J* = 17.5, 11.0 Hz, 1H), 6.33 (t, *J* = 7.3 Hz, 1H), 5.66 (d, *J* = 17.6 Hz, 1H), 5.16 (d, *J* = 11.0 Hz, 1H), 2.25 (dt, *J* = 7.3, 7.3 Hz, 1H), 1.46-1.35 (m, 2H), 1.29 (s, 12H), 1.27-1.15 (m, 10H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ148.2, 133.8, 117.4, 83.1, 31.8, 29.4, 29.4, 29.2, 29.0, 28.7, 24.7, 22.6, 14.0; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 30. HRMS (ESI): m/z calcd. for $[C_{18}H_{34}BO_2]^+$ 293.26464, found 293.26422.

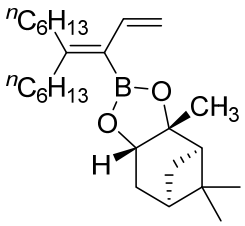
(*Z*)-4,4,5,5-tetramethyl-2-(trideca-1,3-dien-3-yl)-1,3,2-dioxaborolane (3p)



3p

Yield, 69%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.69 (dd, *J* = 17.5, 11.0 Hz, 1H), 6.33 (t, *J* = 7.1 Hz, 1H), 5.66 (d, *J* = 17.5 Hz, 1H), 5.16 (d, *J* = 11.0 Hz, 1H), 2.25 (dt, *J* = 7.3, 7.3 Hz, 2H), 1.46-1.35 (m, 2H), 1.28 (s, 12H), 1.27-1.15 (m, 12H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.2, 133.8, 117.3, 83.1, 31.8, 29.5, 29.4, 29.3, 29.0, 28.7, 24.7, 22.6, 14.0; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 30. HRMS (ESI): m/z calcd. for $[C_{19}H_{36}BO_2]^+$ 307.28029, found 307.27975.

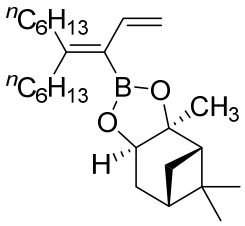
(3aR,4R,6R,7aS)-2-(4-Hexyldeca-1,3-dien-3-yl)-3a,5,5-trimethylhexahydro-4,6-methanobenzo[d][1,3,2]dioxaborole (3ab)



3ab

Yield, 83%. Colorless oil. NMR data: ^{1}H NMR (500 MHz,$CDCl_3$) δ 6.71 (dd, *J* = 17.5, 10.8 Hz, 1H), 5.21 (dd, *J* = 17.6, 1.8 Hz, 1H), 5.03 (dd, *J* = 10.8, 1.8 Hz, 1H), 4.38 (dd, *J* = 8.9, 2.1 Hz, 1H), 2.41-2.36 (m, 1H), 2.28-2.14 (m, 5H), 2.11 (dd, *J* = 5.2, 5.2 Hz, 1H), 2.00-1.85 (m, 2H), 1.44 (s, 3H), 1.42-1.36 (m, 4H), 1.35-1.20 (m, 16H), 0.95-0.80 (m, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 155.2, 136.4, 114.5, 85.6, 77.6, 51.2, 39.5, 38.1, 37.8, 35.6, 32.1, 31.7(8), 31.7(0), 29.7, 29.6, 29.5, 28.9, 28.8, 27.0, 26.7, 24.0, 22.6, 22.5, 14.0; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 30. HRMS (ESI): m/z calcd. for $[C_{26}H_{46}BO_2]^+$ 401.35854, found 401.35751.

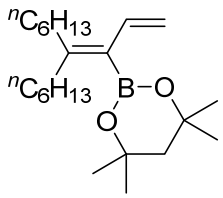
(3aS,4S,6S,7aR)-2-(4-Hexyldeca-1,3-dien-3-yl)-3a,5,5-trimethylhexahydro-4,6-methanobenzo[d][1,3,2]dioxaborole (3ac)



3ac

Yield, 85%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$ δ 6.71 (dd, *J* = 17.5, 10.8 Hz, 1H), 5.21 (d, *J* = 17.6 Hz, 1H), 5.03 (d, *J* = 10.9 Hz, 1H), 4.38 (dd, *J* = 8.5, 2.0 Hz, 1H), 2.44-2.34 (m, 1H), 2.28-2.13 (m, 5H), 2.11 (dd, *J* = 5.3, 5.3 Hz, 1H), 1.98-1.85 (m, 2H),1.44 (s, 3H), 1.43-1.34 (m, 4H), 1.33-1.16 (m, 16H), 0.96-0.76 (m, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 155.2, 136.4, 114.5, 85.6, 77.6, 51.2, 39.5, 38.2, 37.8, 35.6, 32.1, 31.7(8), 31.7(0), 29.7, 29.6, 29.5, 29.0, 28.8, 27.0, 26.7, 24.0, 22.6, 22.5, 14.0; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 30. HRMS (ESI): m/z calcd. for $[C_{26}H_{46}BO_2]^+$ 401.35854, found 401.35742.

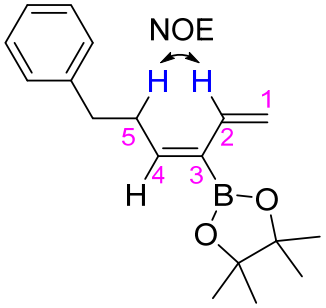
2-(4-Hexyldeca-1,3-dien-3-yl)-4,4,6,6-tetramethyl-1,3,2-dioxaborinane (3ad)



3ad

Yield, 61%. Colorless oil. NMR data: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (dd, *J* = 17.5, 10.6 Hz, 1H), 5.06 (d, *J* = 17.5 Hz, 1H), 4.97 (d, *J* = 10.6 Hz, 1H), 2.13 (t, *J* = 7.7 Hz, 2H), 2.08 (t, *J* = 7.7 Hz, 2H), 1.89 (s, 2H), 1.40 (s, 12H), 1.47-1.20 (m, 16H), 0.88 (t, *J* = 6.8 Hz, 6H);^{13}C NMR (126 MHz, $CDCl_3$) δ 150.1, 136.5, 113.6, 71.0, 49.1, 38.1, 31.9, 31.8, 31.7(6), 31.7(3), 29.9, 29.3, 29.1, 22.6, 14.0; Carbons with directly attached boron atoms were not observed, most likely due to quadrupolar relaxation. ^{11}B NMR (128 MHz, $CDCl_3$) δ 28. HRMS (ESI): m/z calcd. for $[C_{23}H_{44}BO_2]^+$ 363.34289, found 363.34210.

Determination of the stereochemistry of 3n using a NOESY spectrum



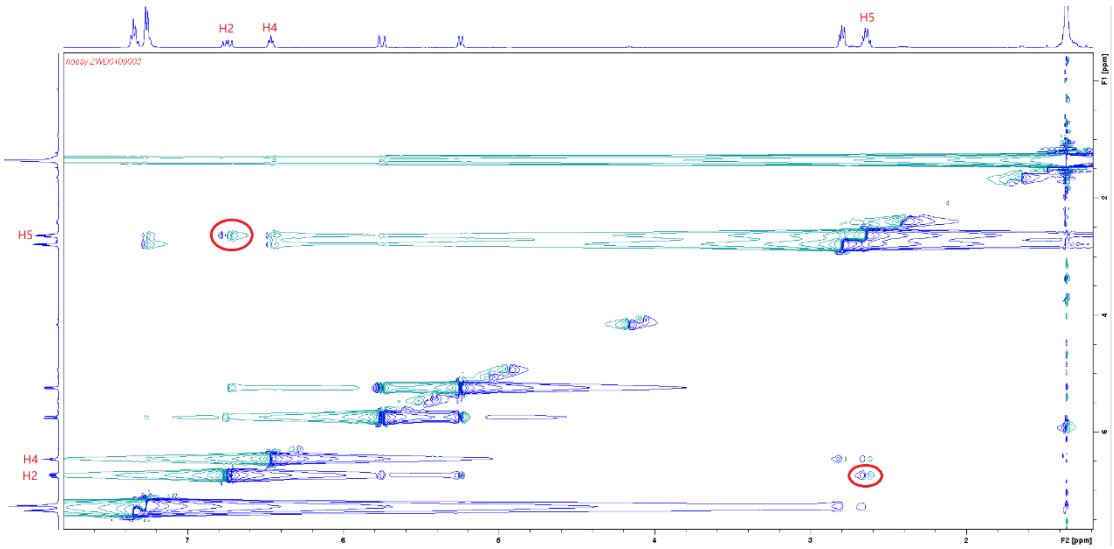


Figure S1. Observed NOESY effect between the C2 proton and C5 proton

The NOESY experiment (Figure S1) indicated a through space NOE effect between C2-H and C5-H. This indicates that C2-H and C5-H are close in space, and the C3-C4 double bond has an *Z* geometry.

References:

1. K. Semba, T. Fujihara, J. Terao and Y. Tsuji, *Angew. Chem. Int. Ed.*, 2013, **52**, 12400-12403.
2. H. Wang, B. Beiring, D.-G. Yu, K. D. Collins and F. Glorius, *Angew. Chem. Int. Ed.*, 2013, **52**, 12430-12434.

NMR Spectra of 1 and 3

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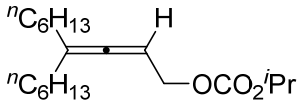

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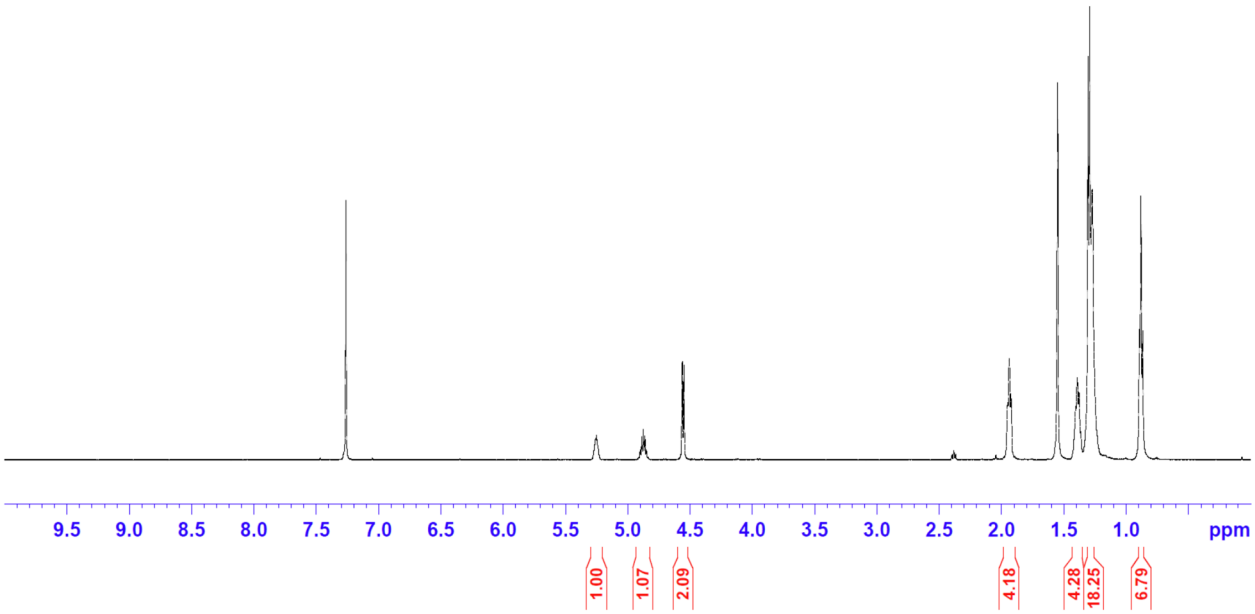

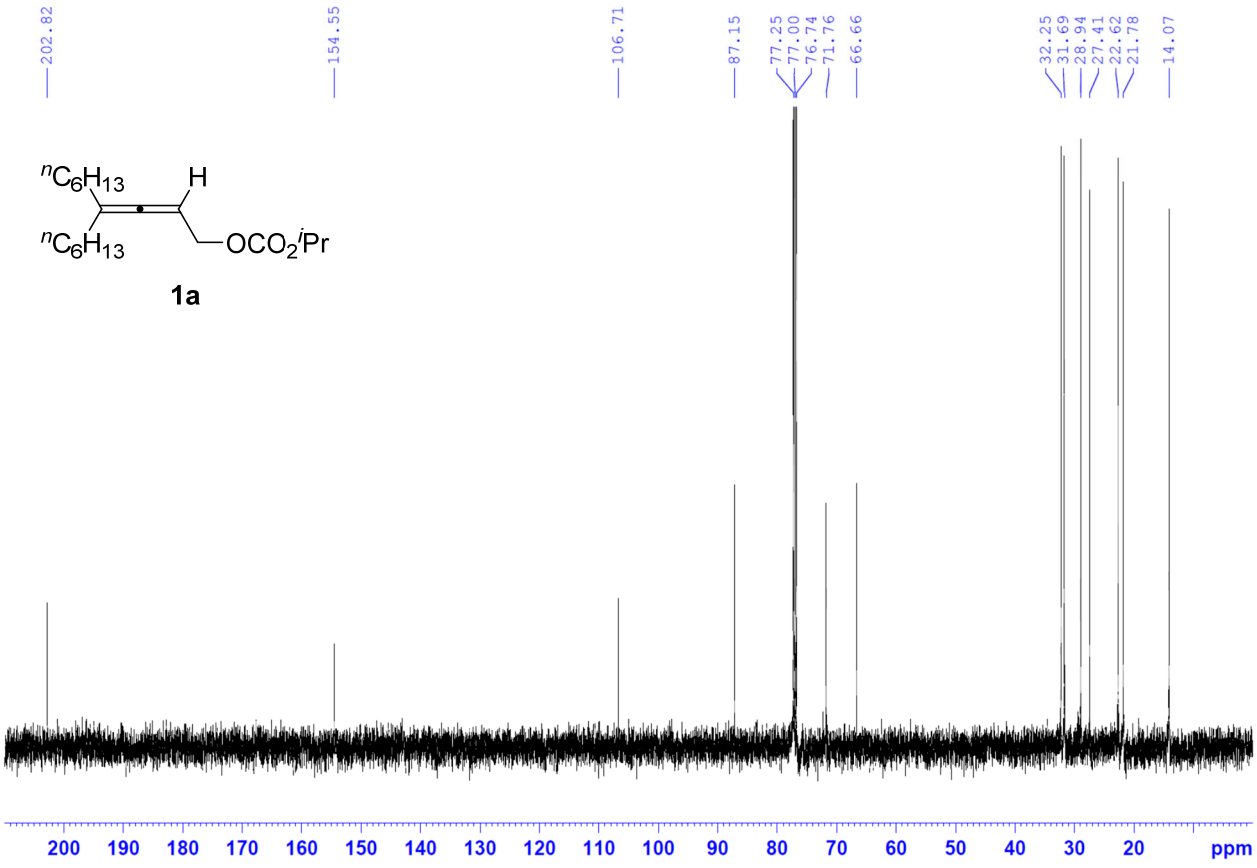

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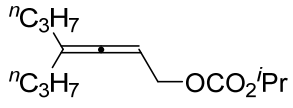

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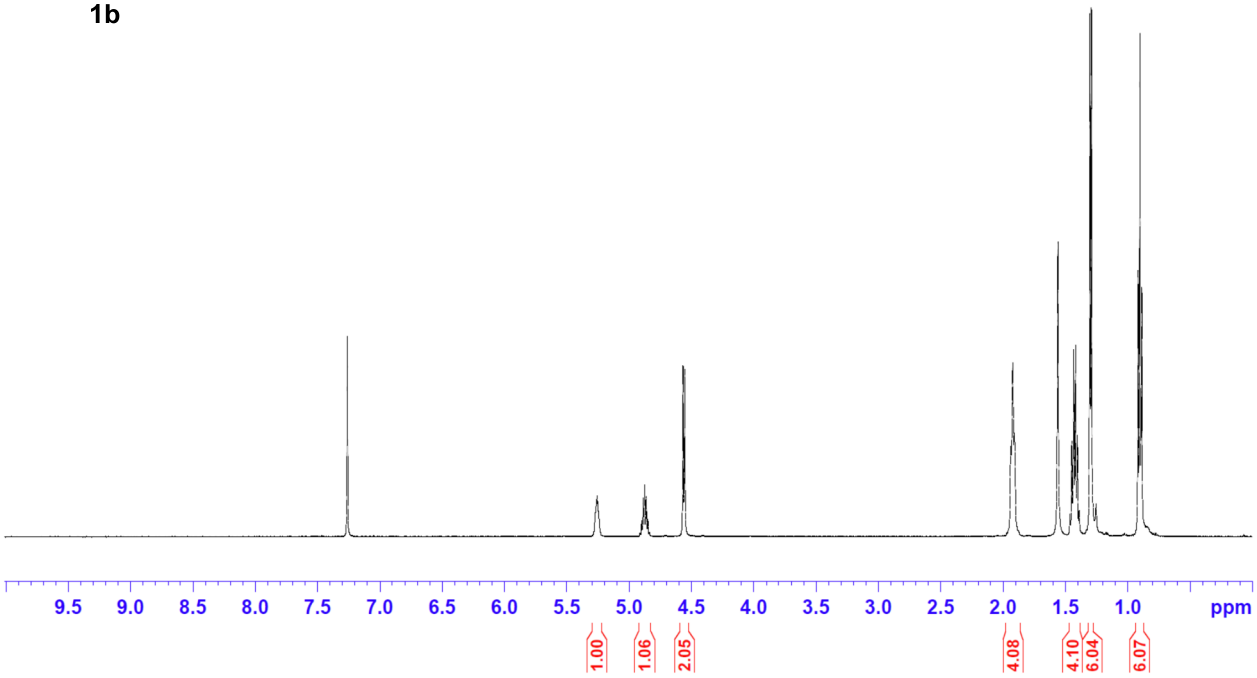

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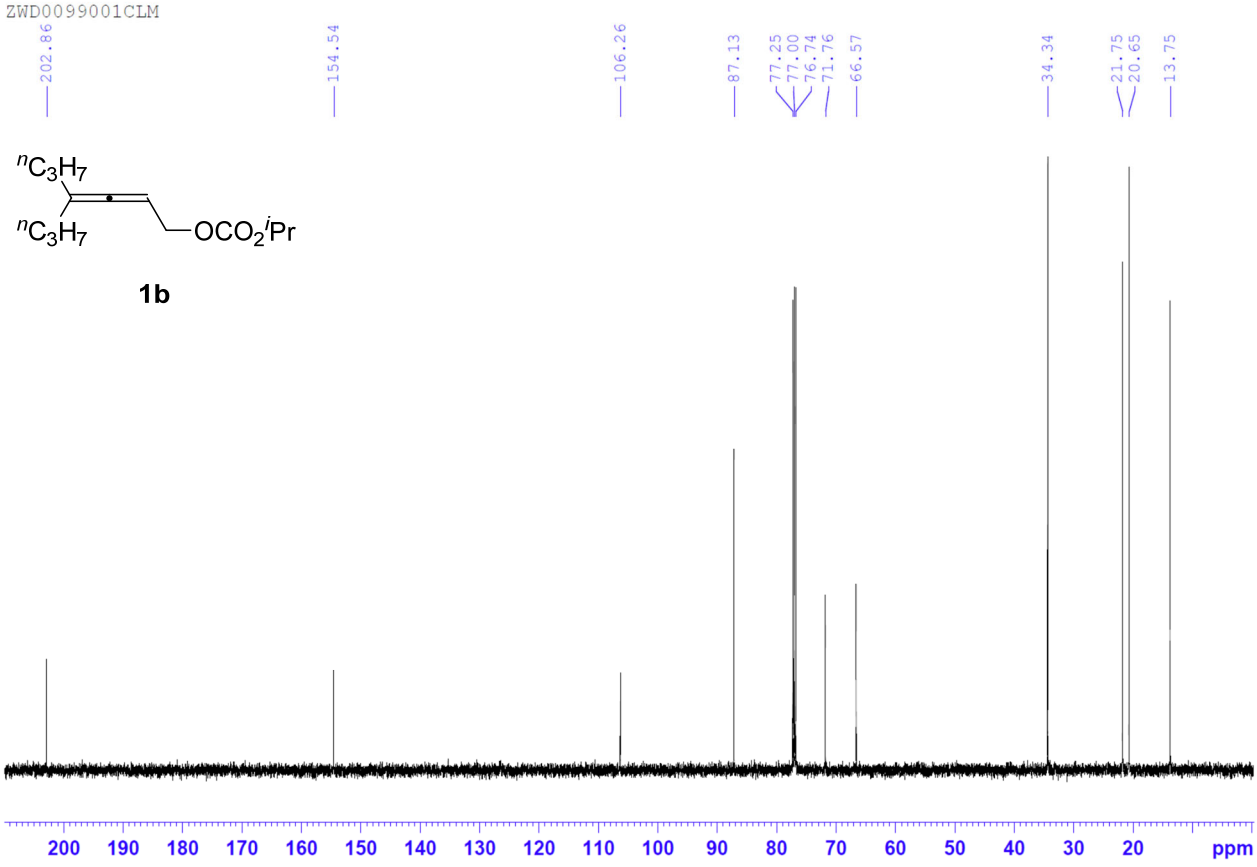

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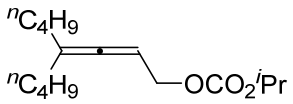

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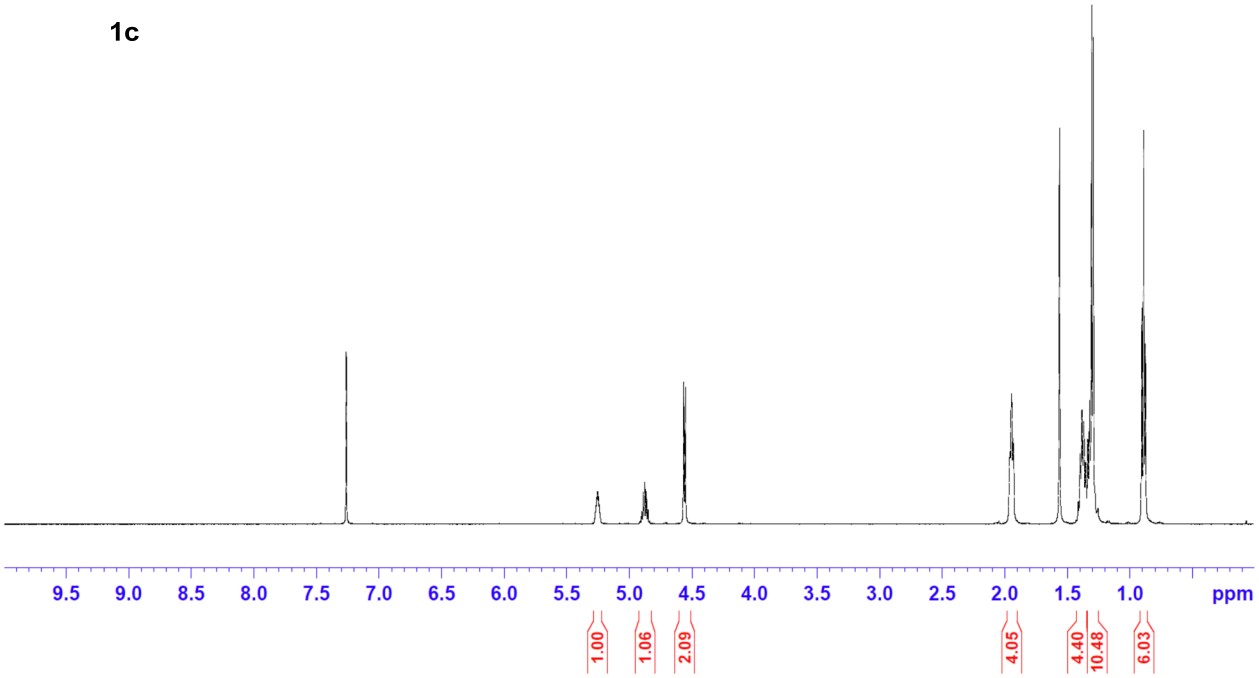

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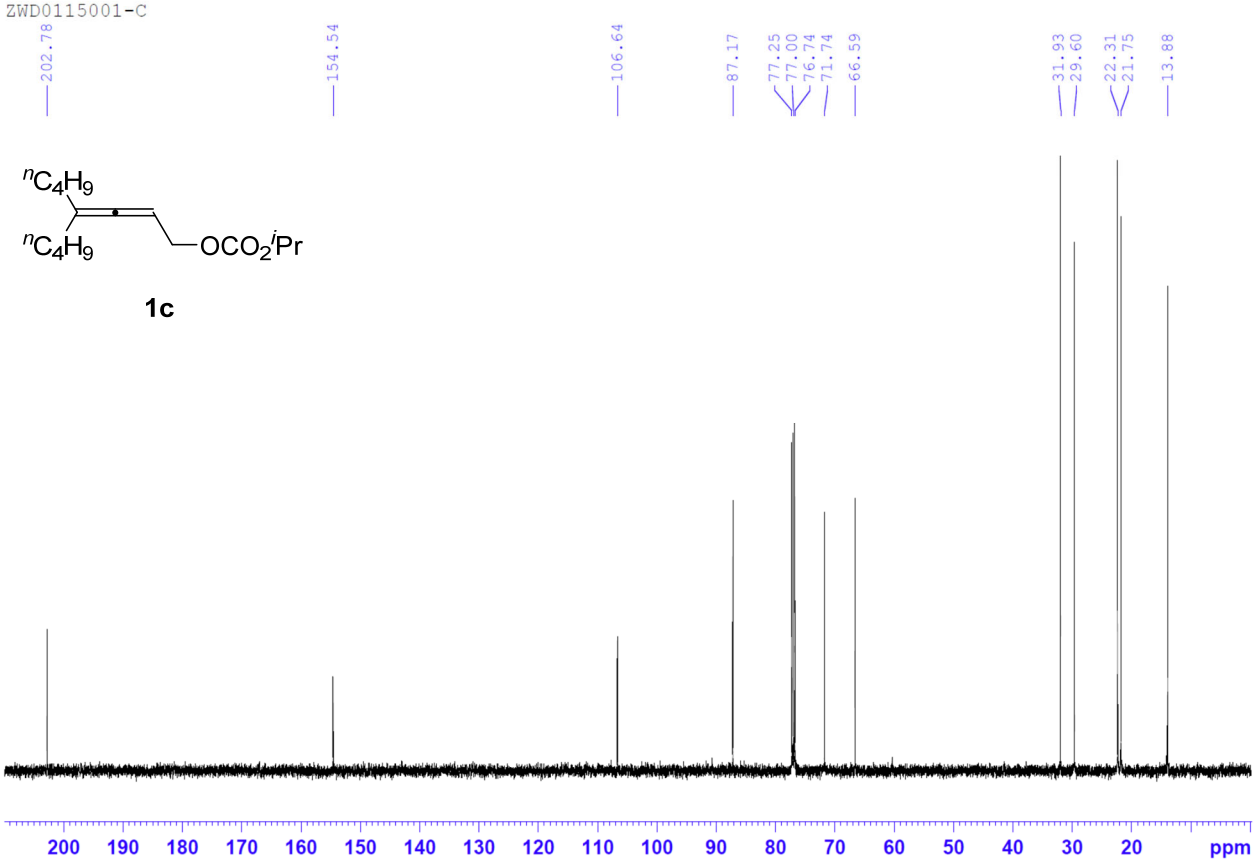

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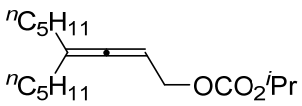

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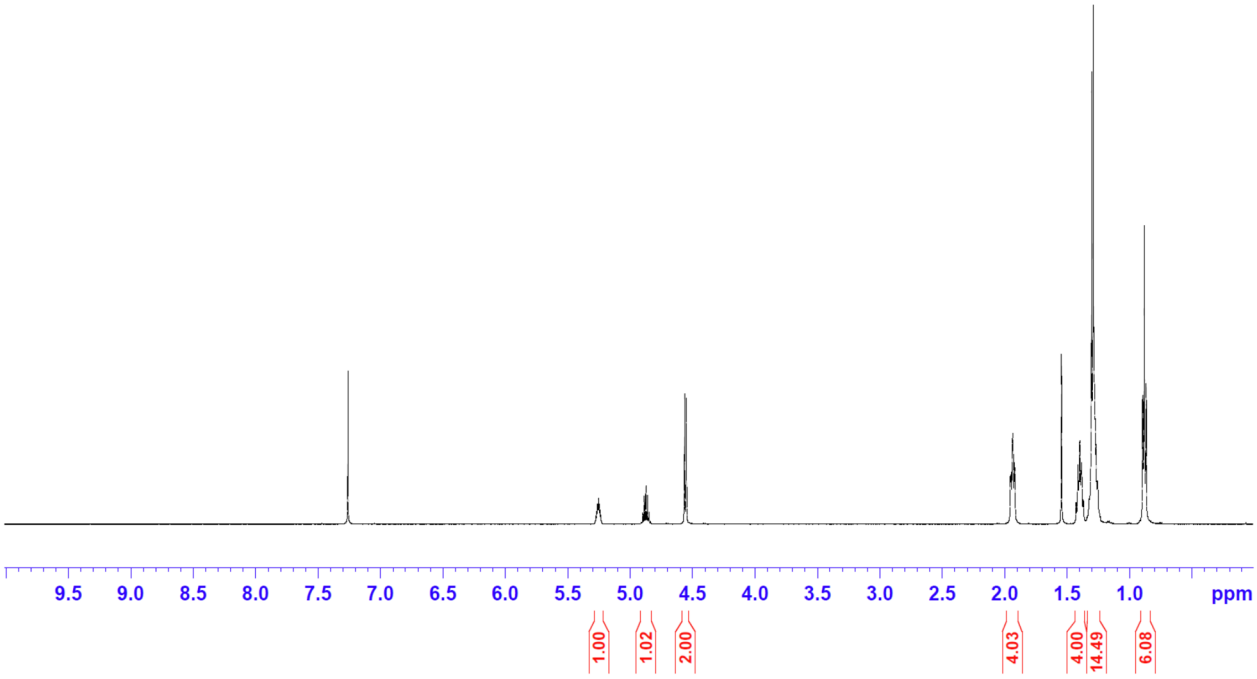

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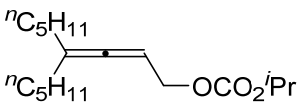

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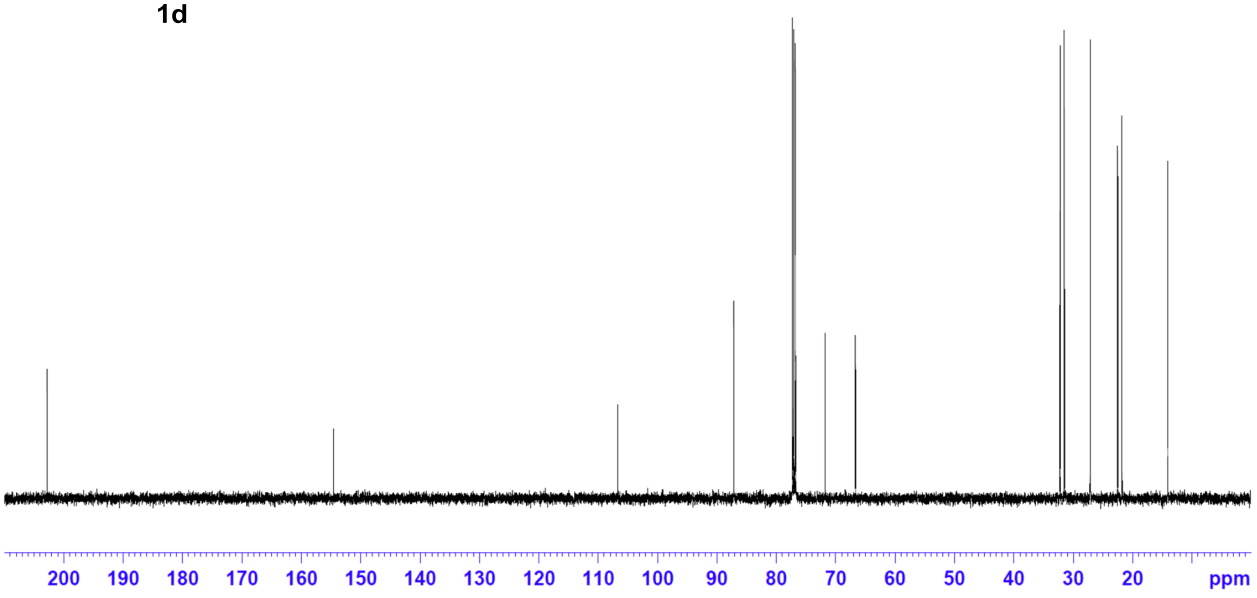

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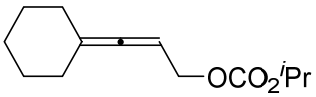

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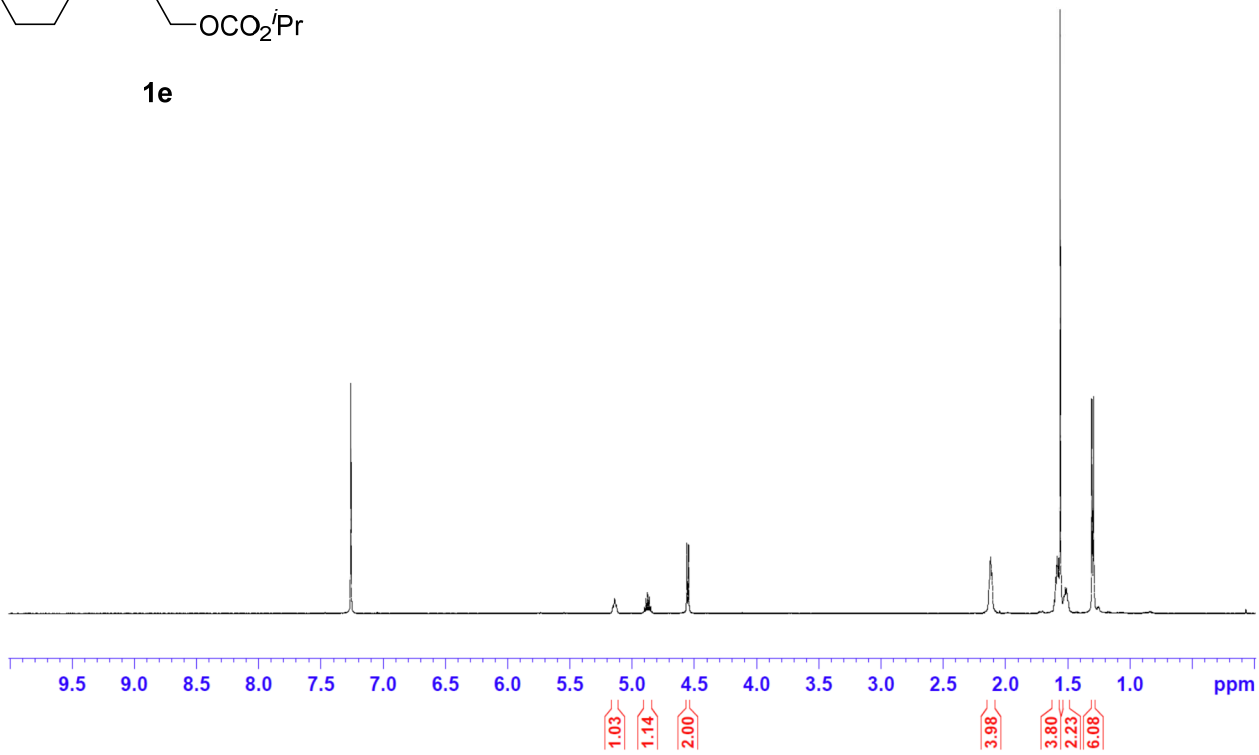

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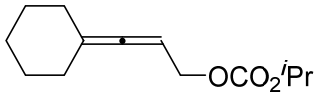

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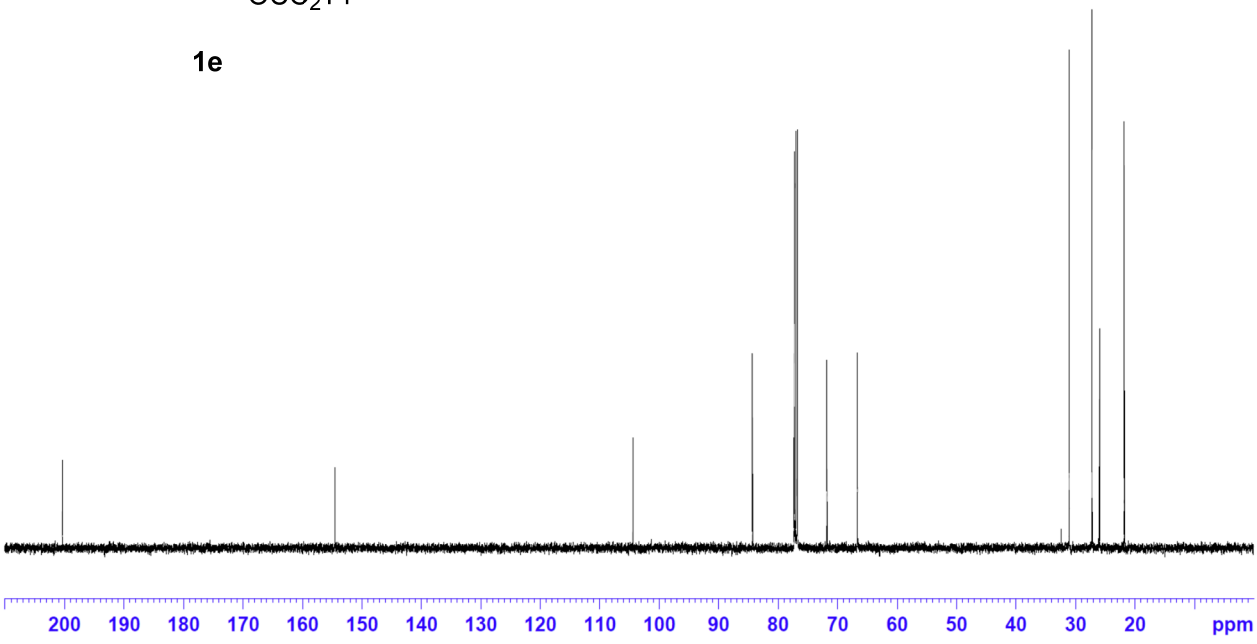

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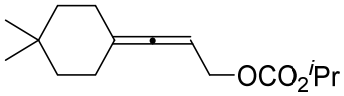

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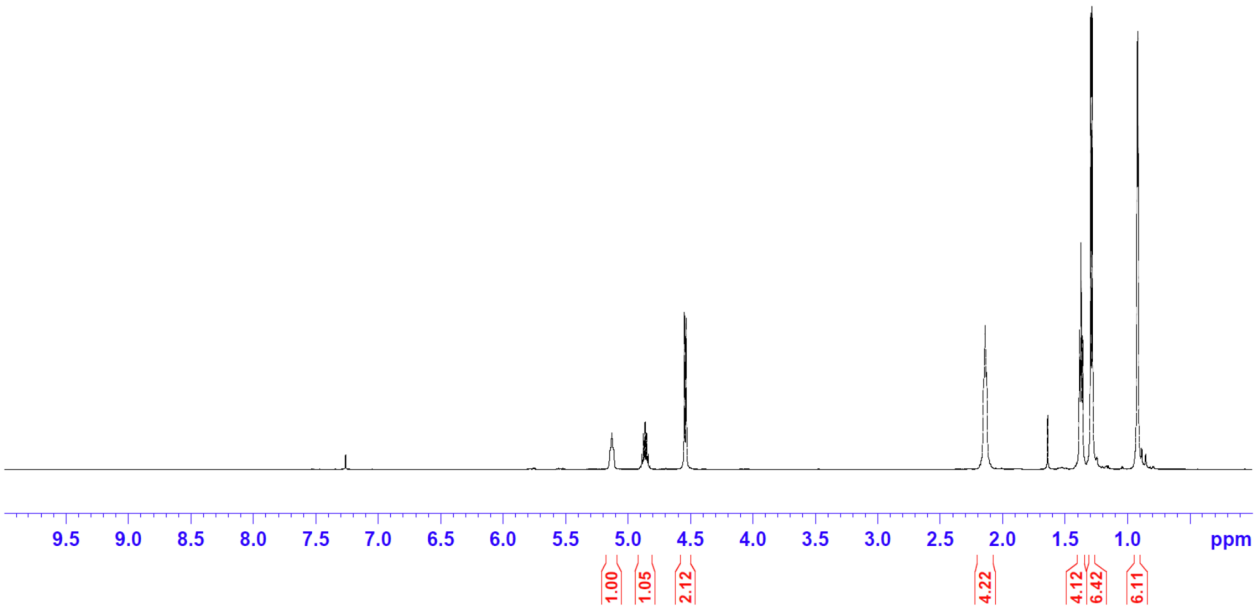

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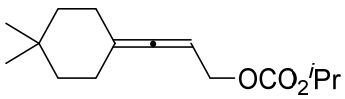

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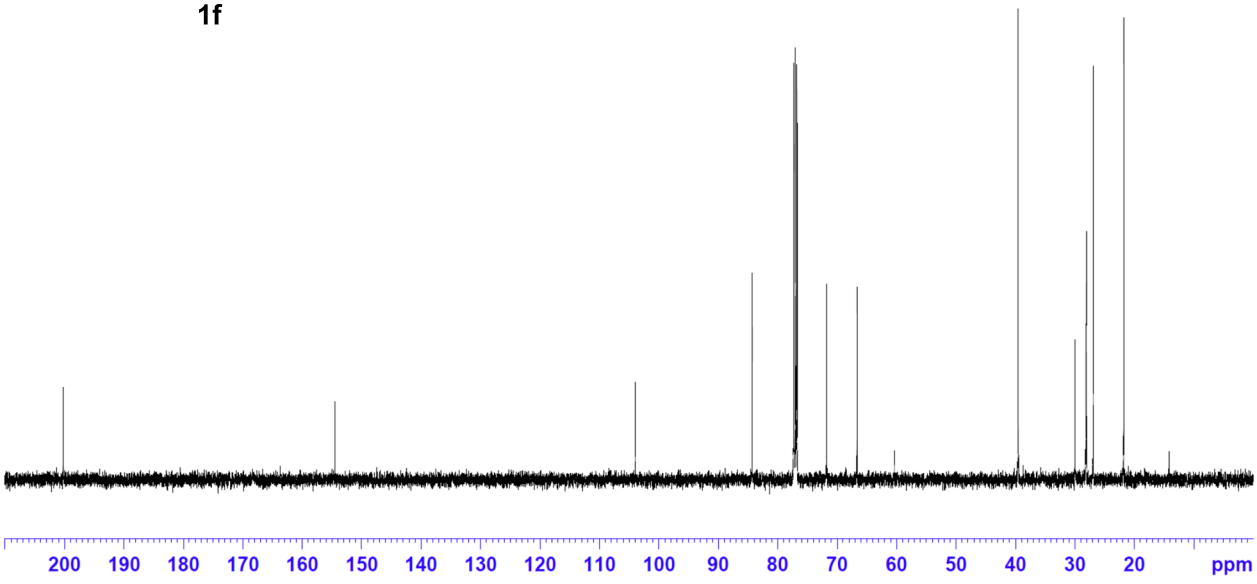

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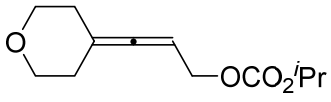

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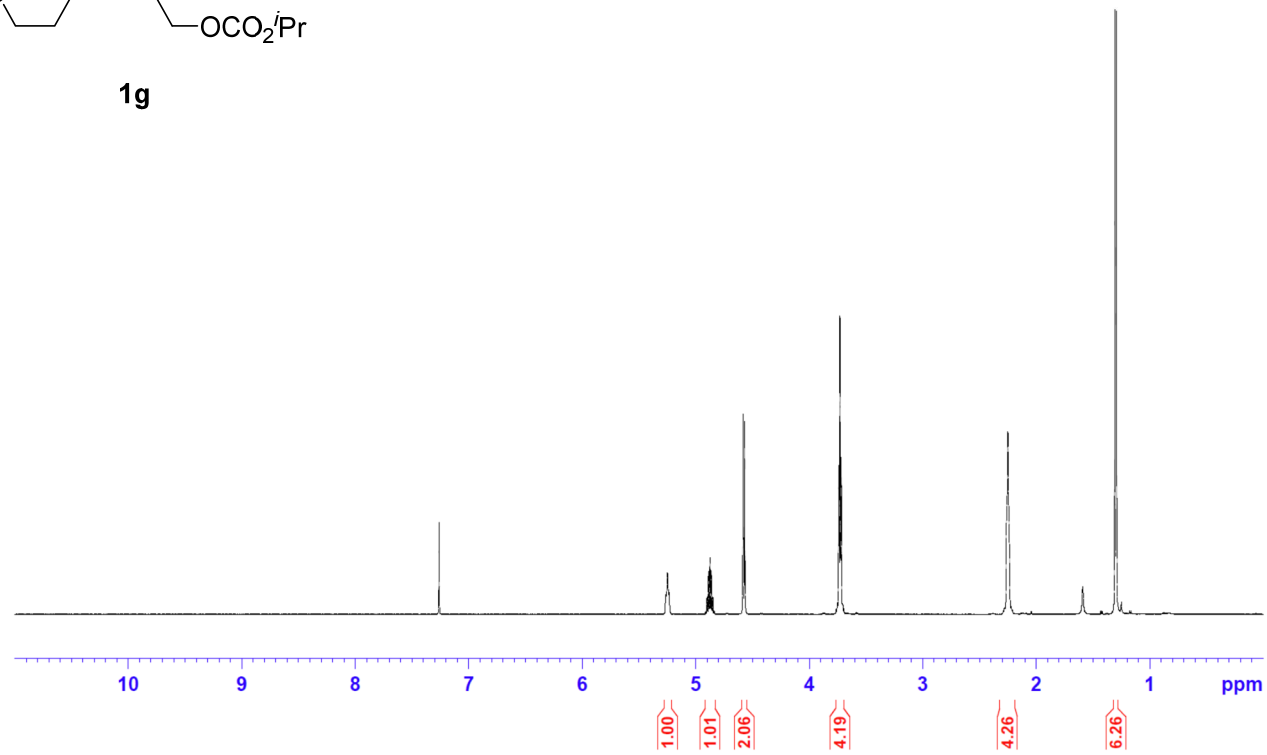

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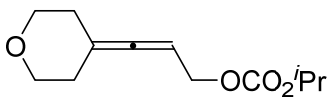

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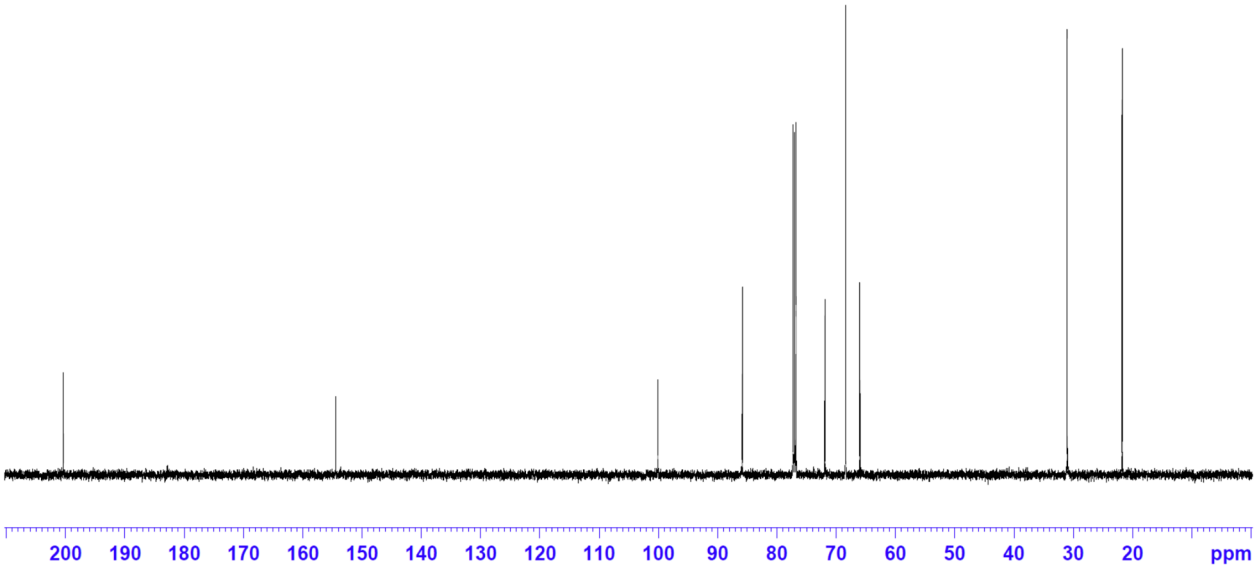

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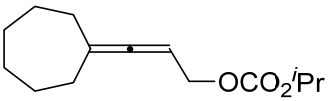

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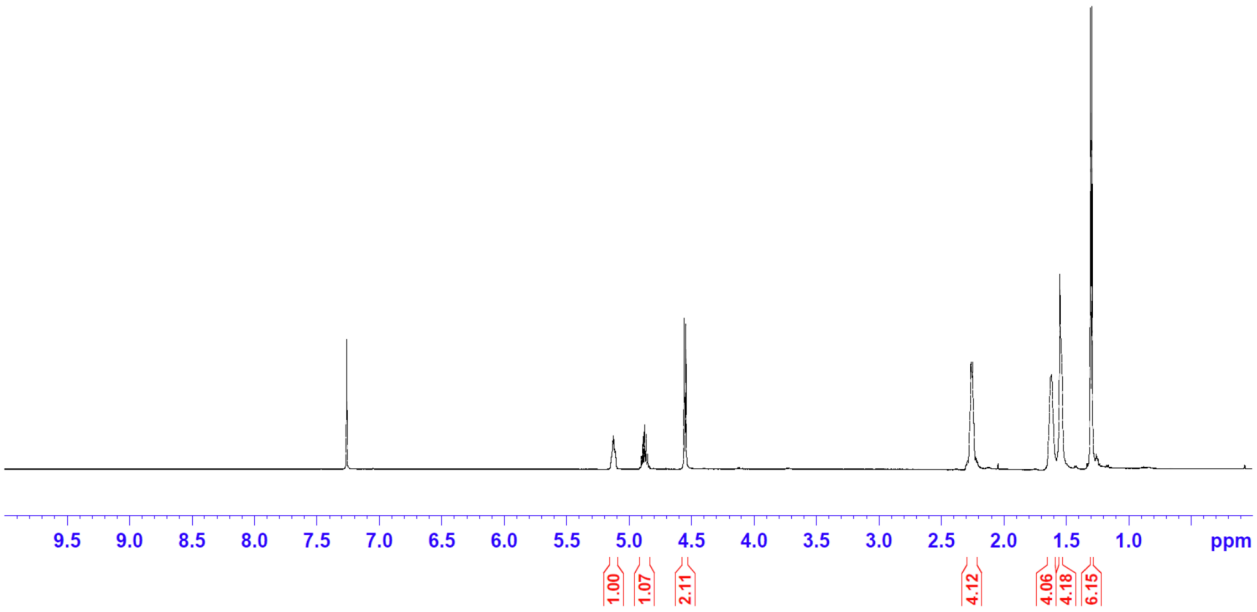

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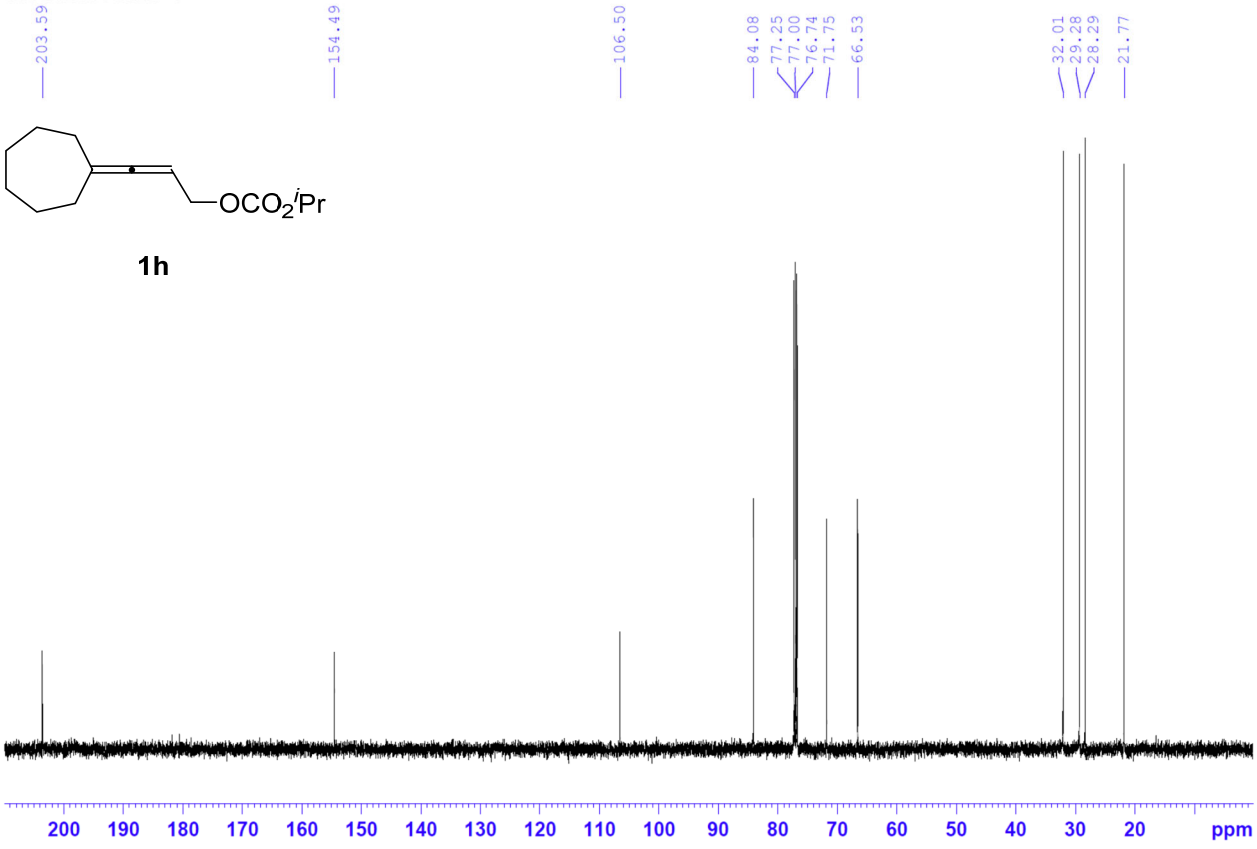

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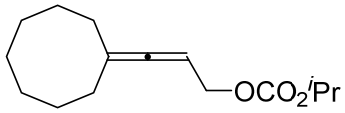

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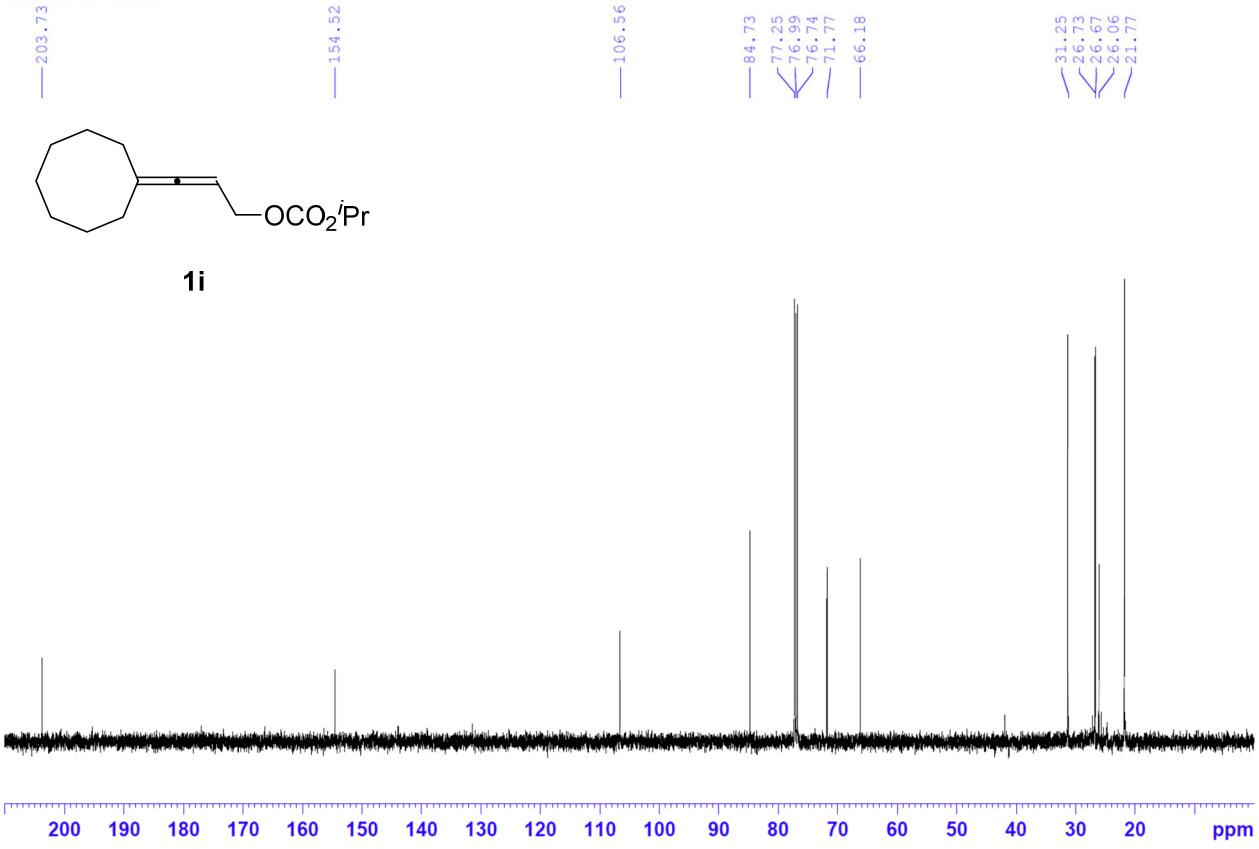

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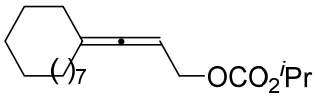

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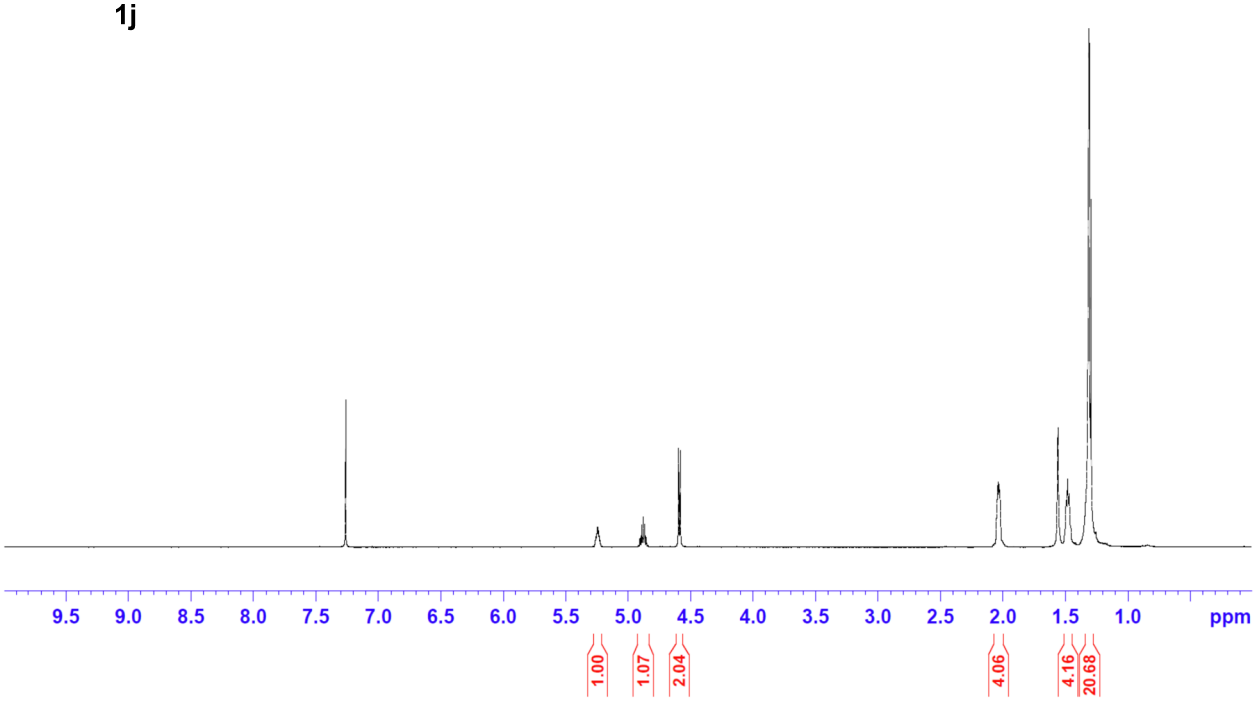

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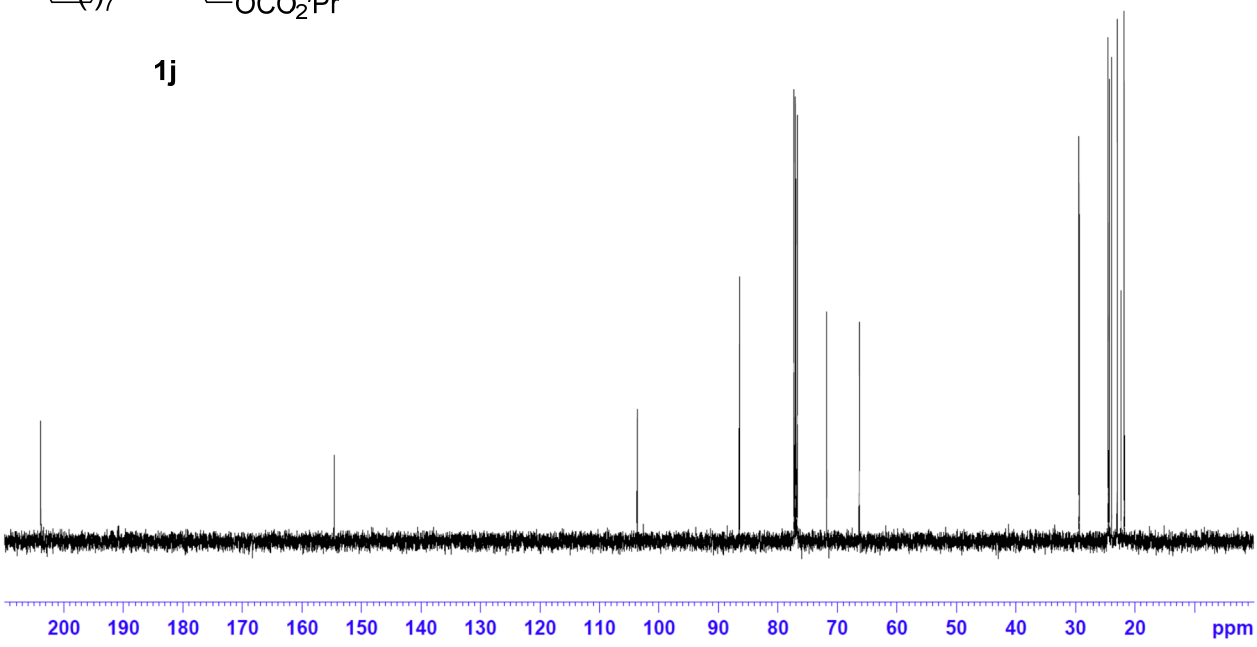

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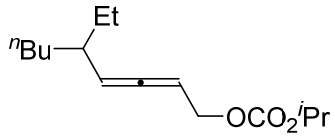

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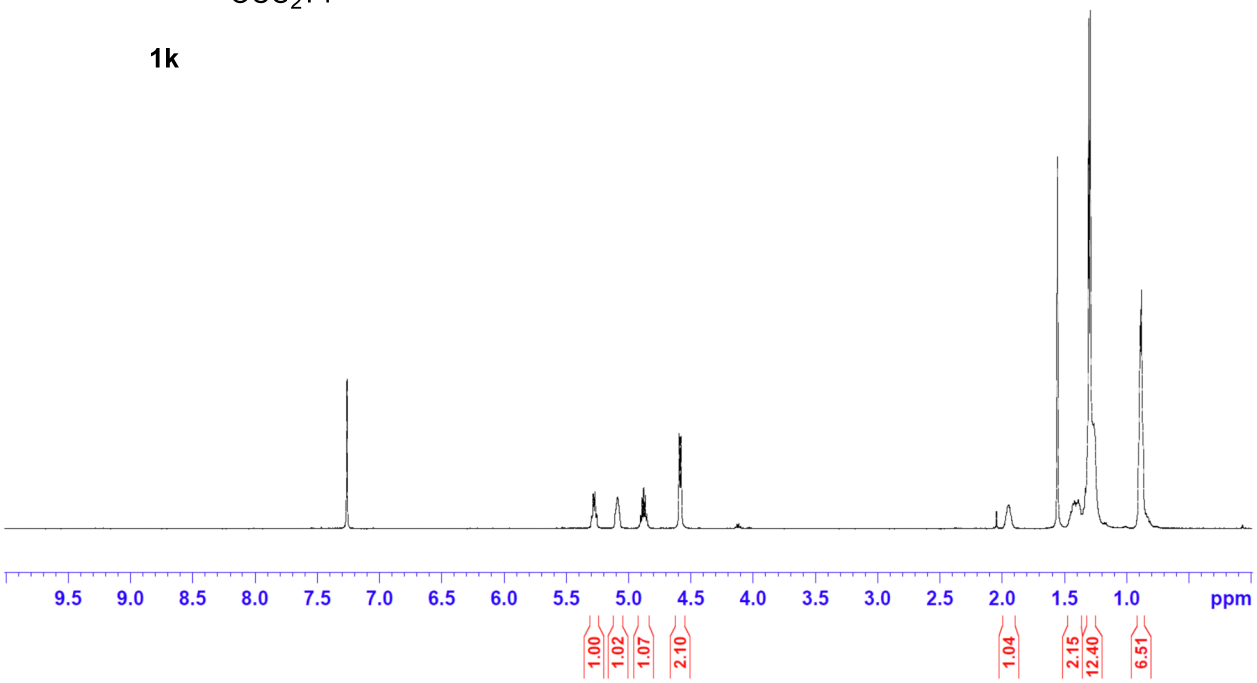

ZWD0126001-C

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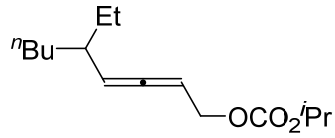

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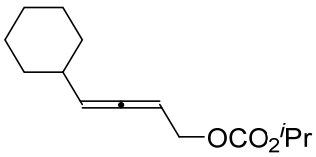

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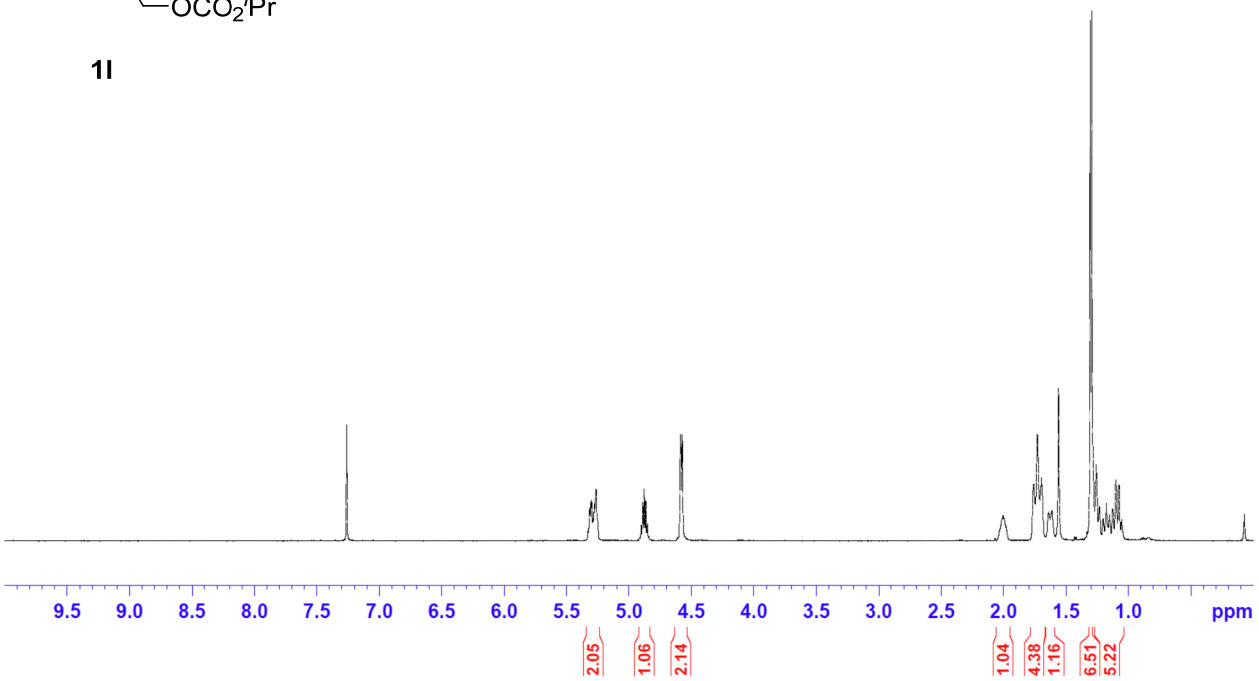

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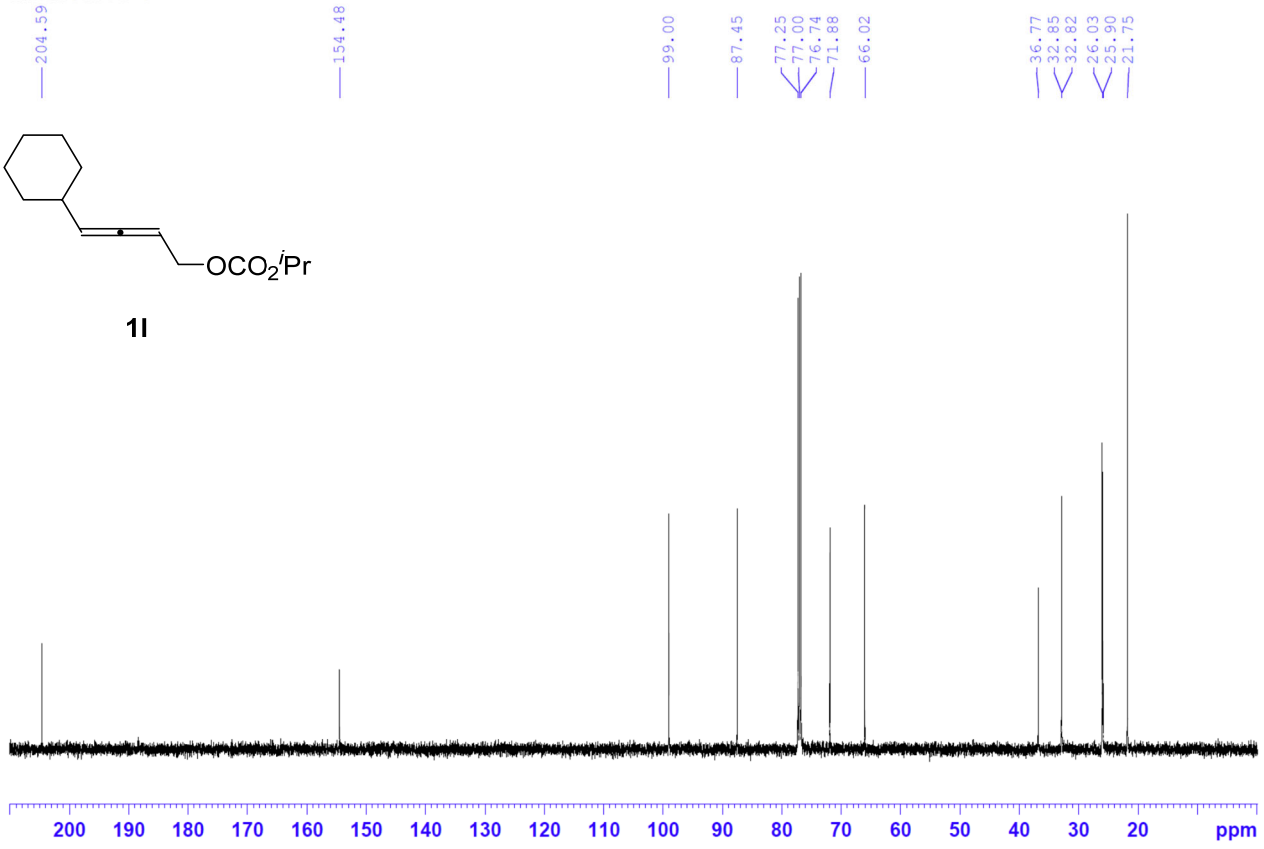

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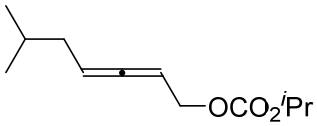

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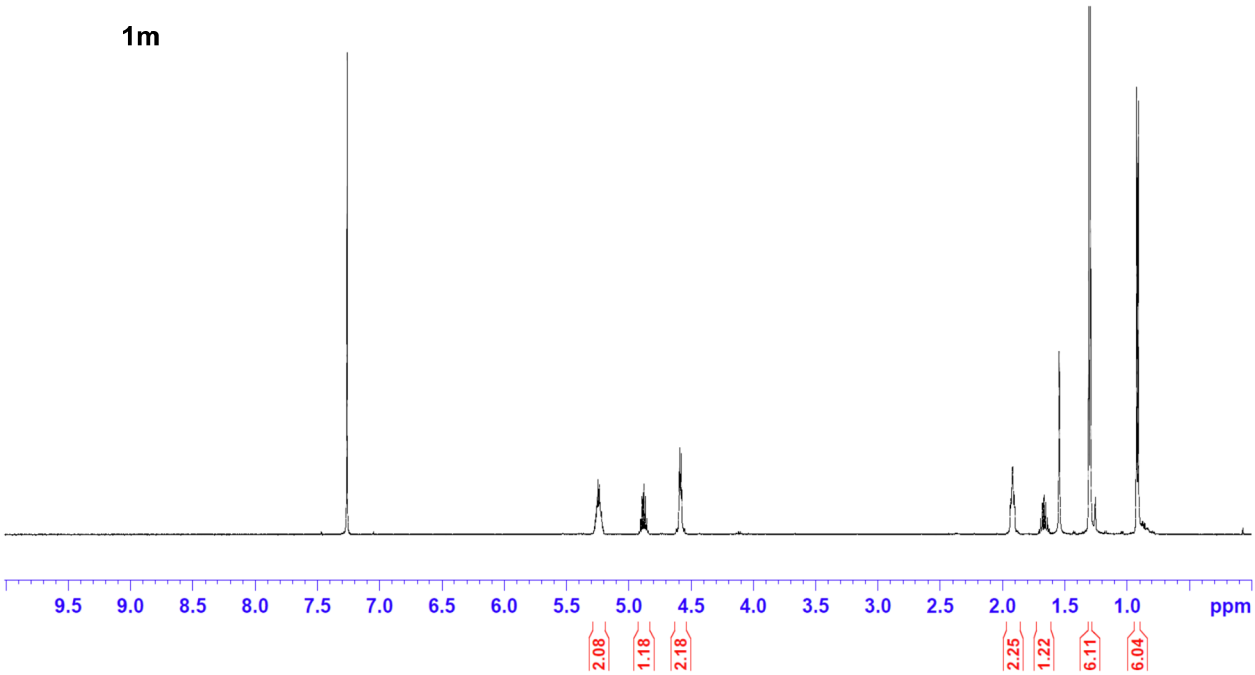

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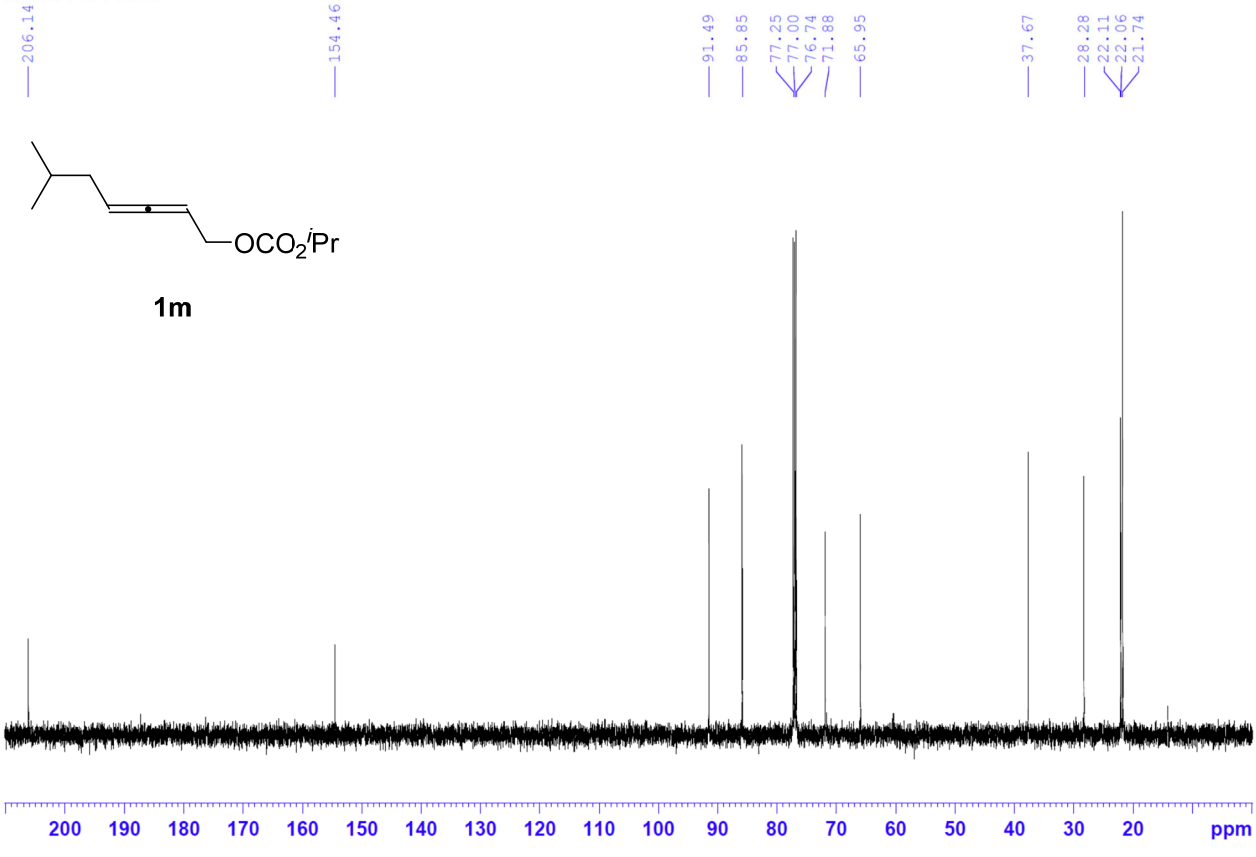

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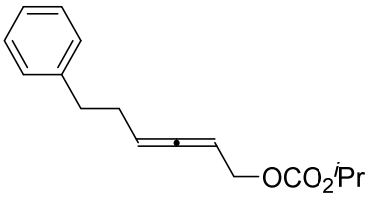

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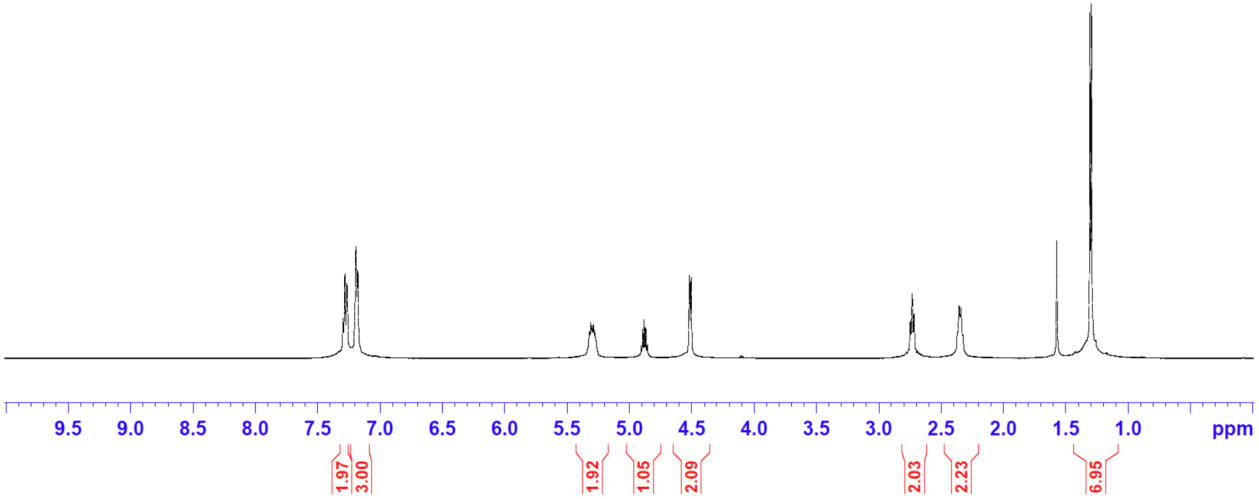

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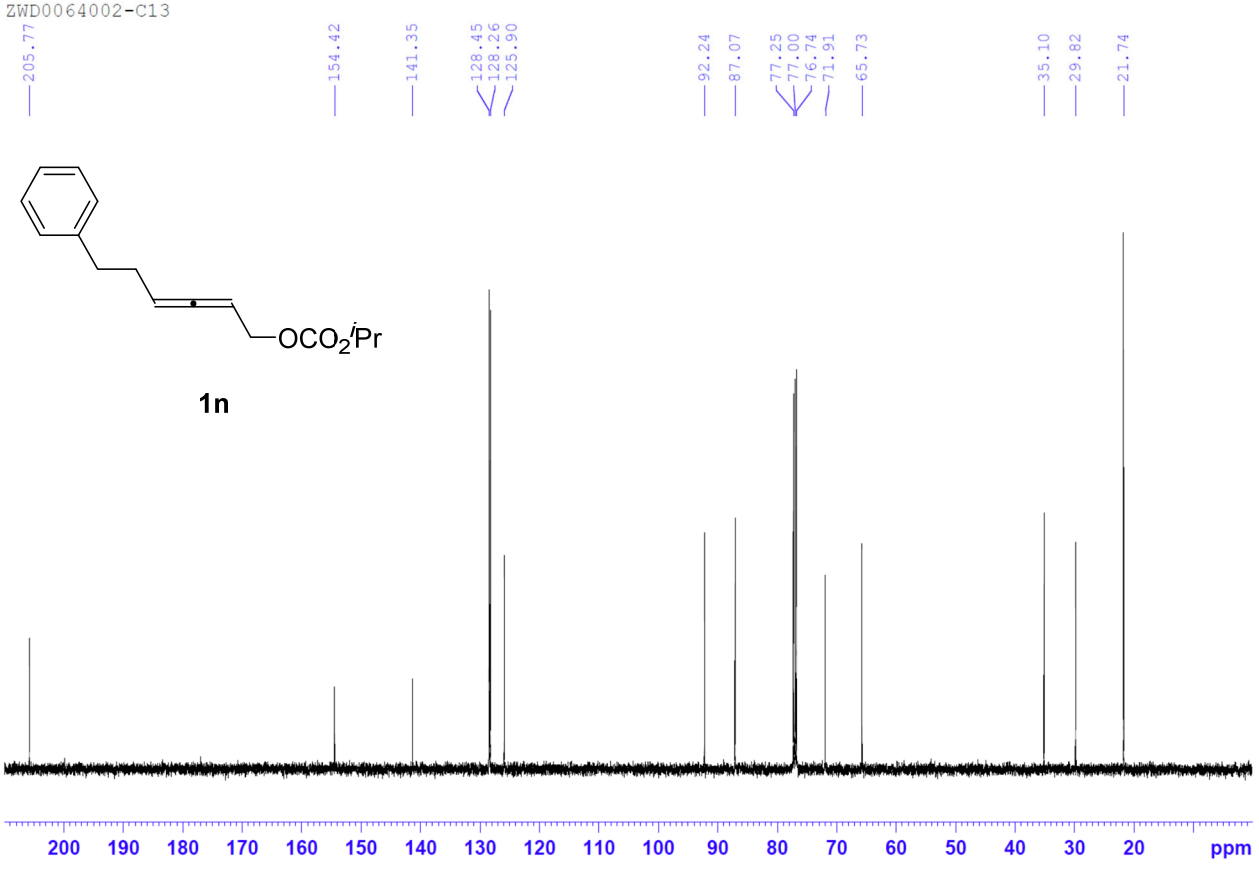

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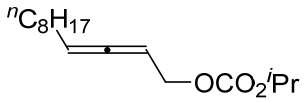

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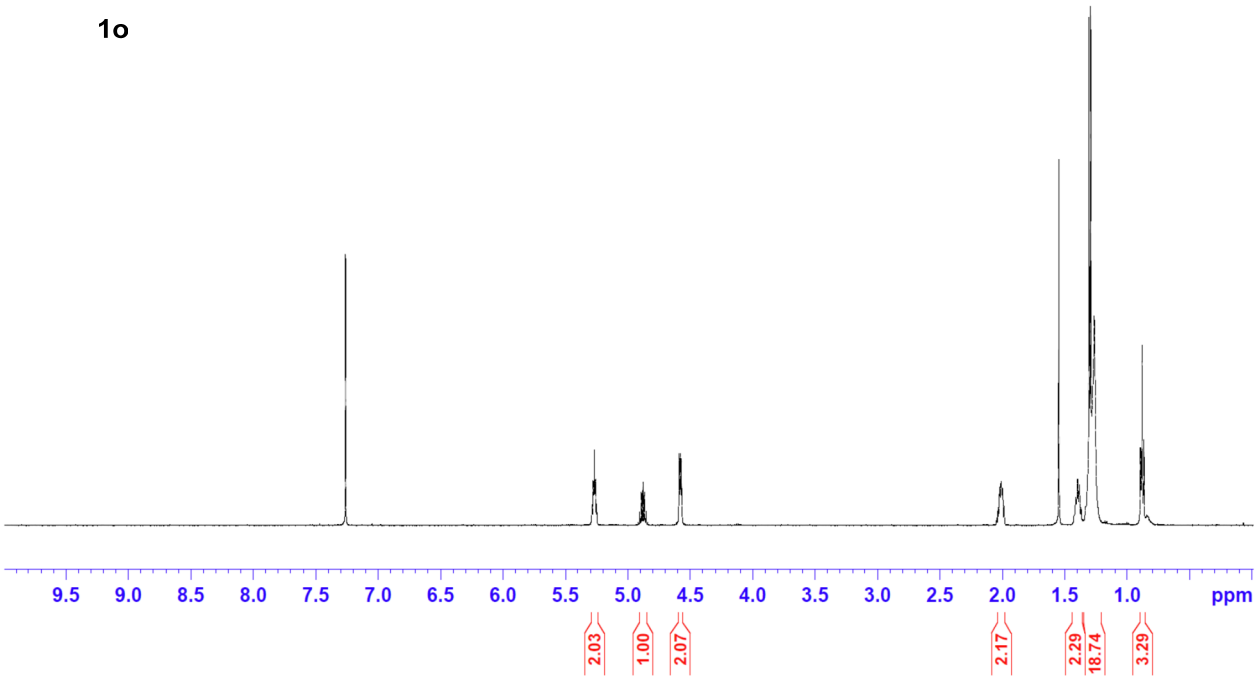

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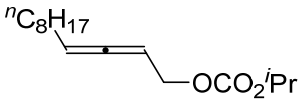

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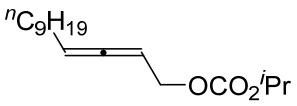

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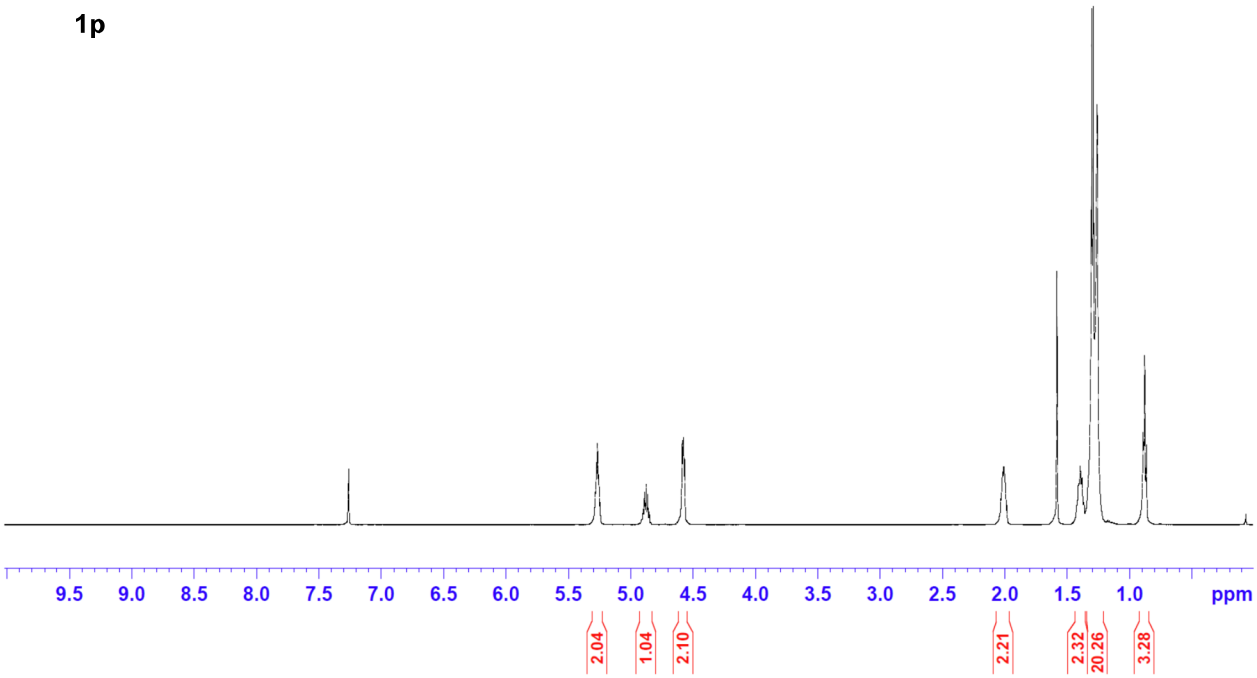

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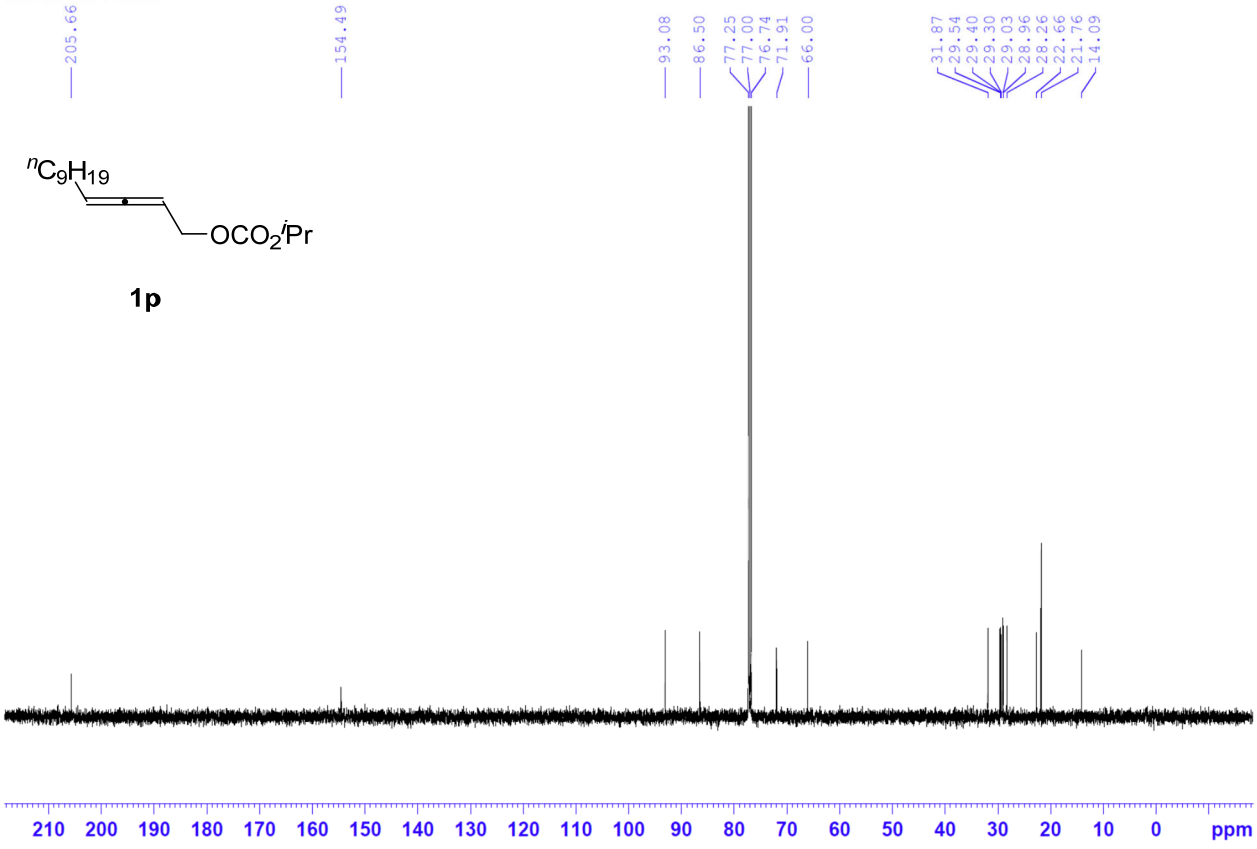

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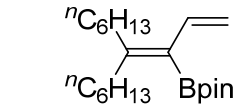
$^{n}C_6H_{13}$
$^{n}C_6H_{13}$
Bpin

3a

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
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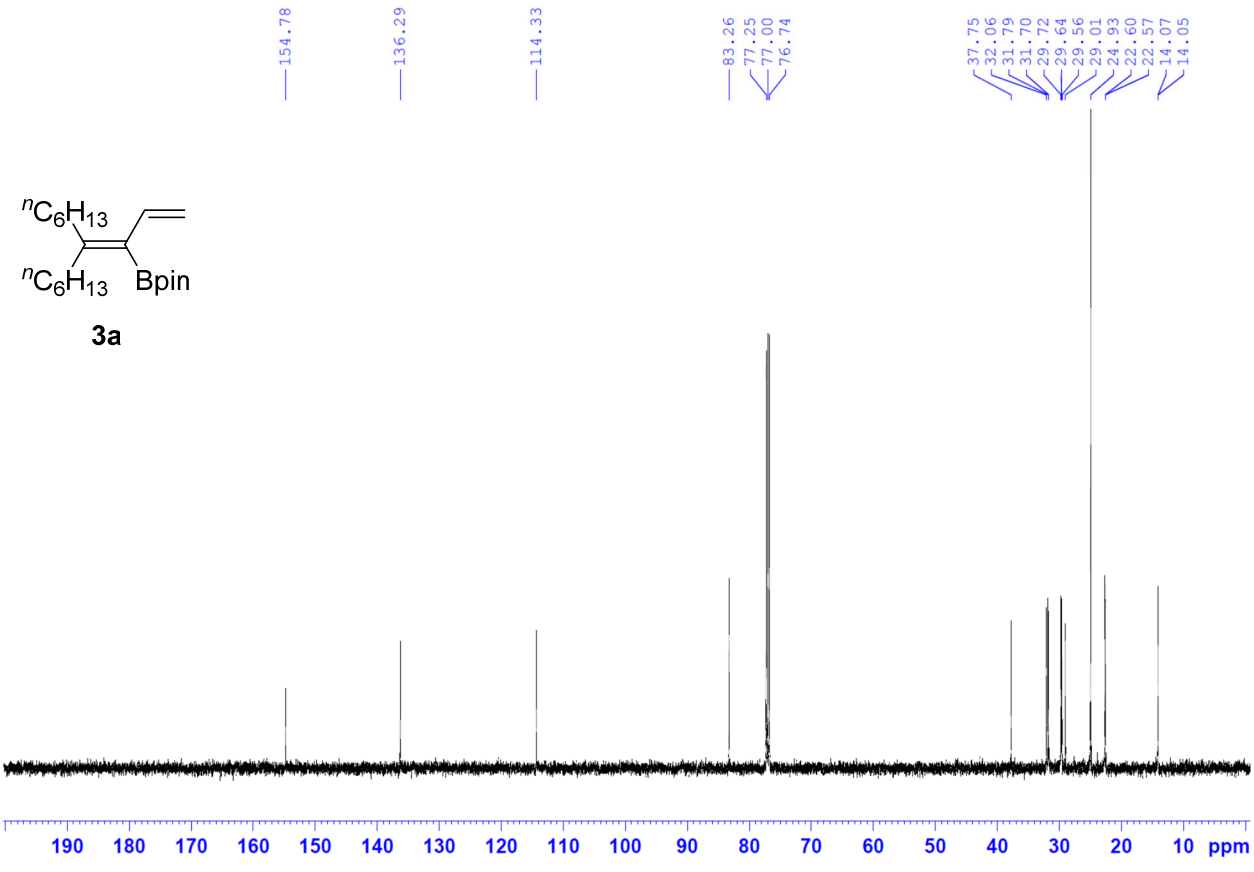
1.06
1.07
4.16
5.06
12.00
15.05
6.26


154.78
136.29
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37.75
32.06
31.79
31.70
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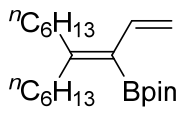
29.56
29.01
24.93
22.60
22.57
14.07
14.05
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

$^{n}C_6H_{13}$
$^{n}C_6H_{13}$
Bpin

3a

Nov14-2021-ZWD-42-17-B. 10. fid

31.21



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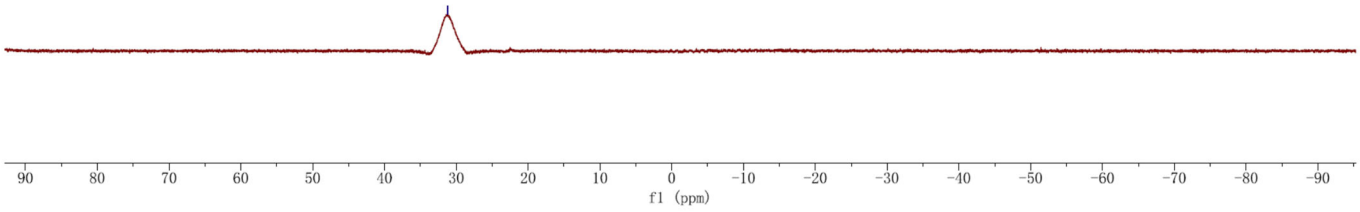

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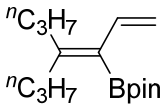

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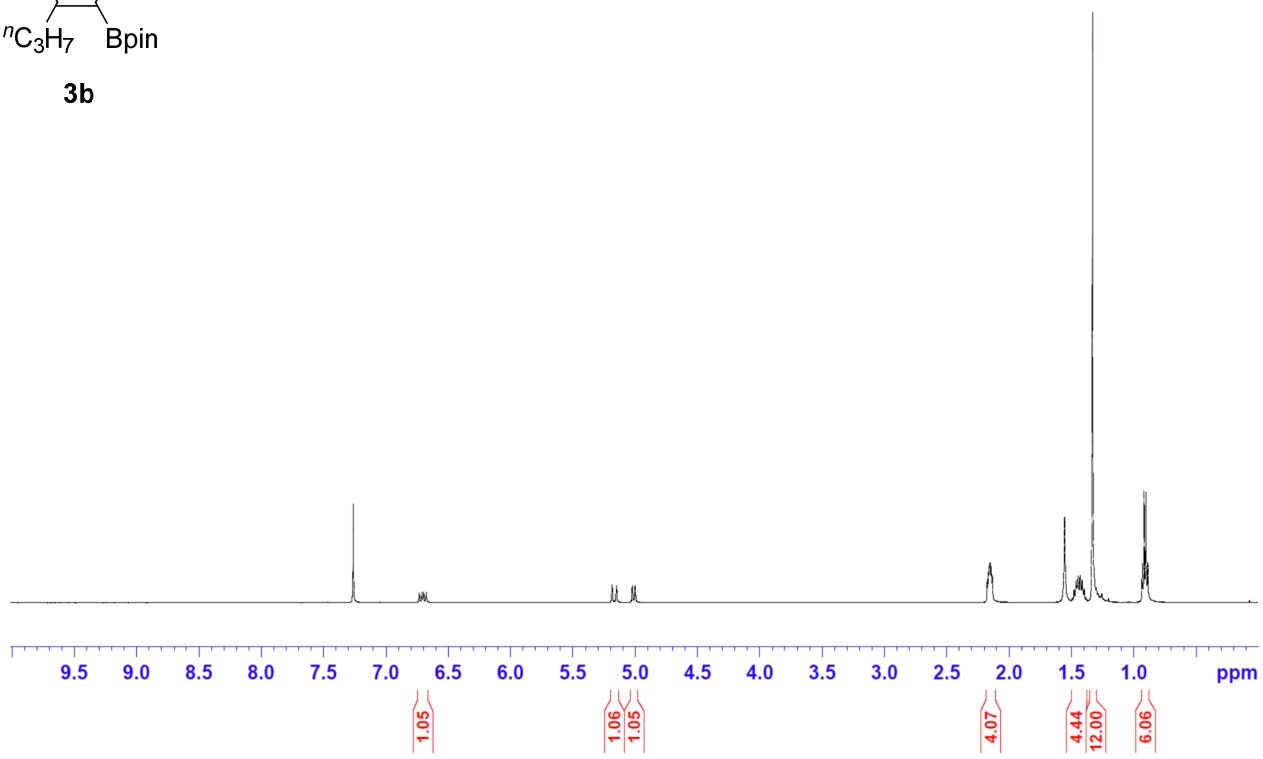

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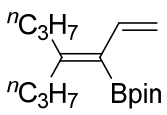

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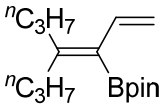
Nov20-2021-ZWD-103-B.1.fid



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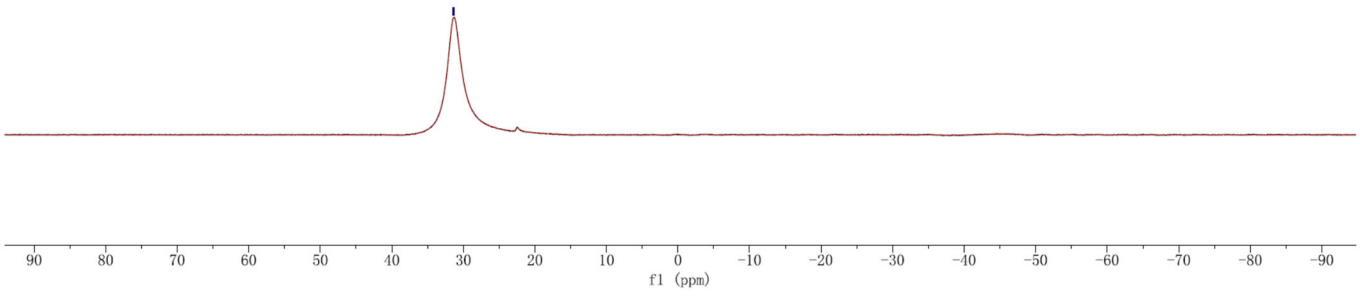

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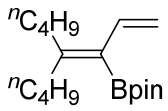

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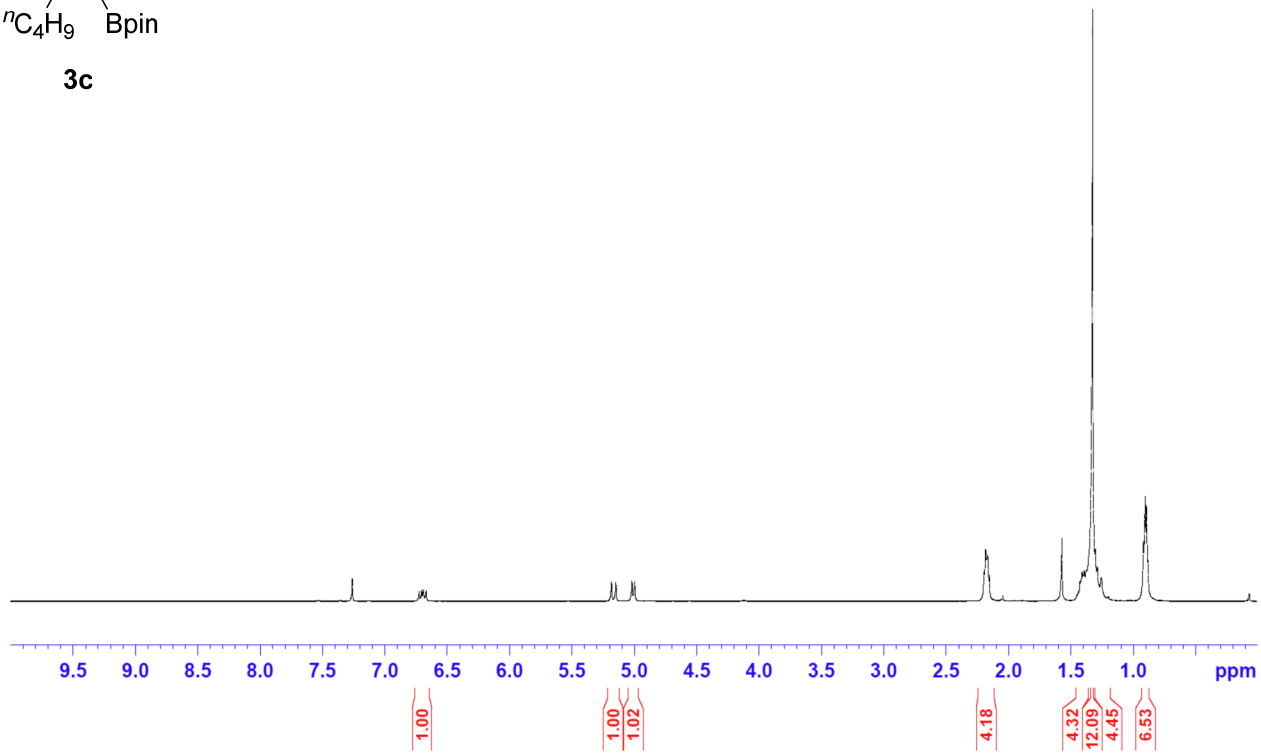

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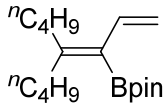

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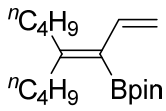
Nov14-2021-ZWD-133-B. 10. fid

31.49



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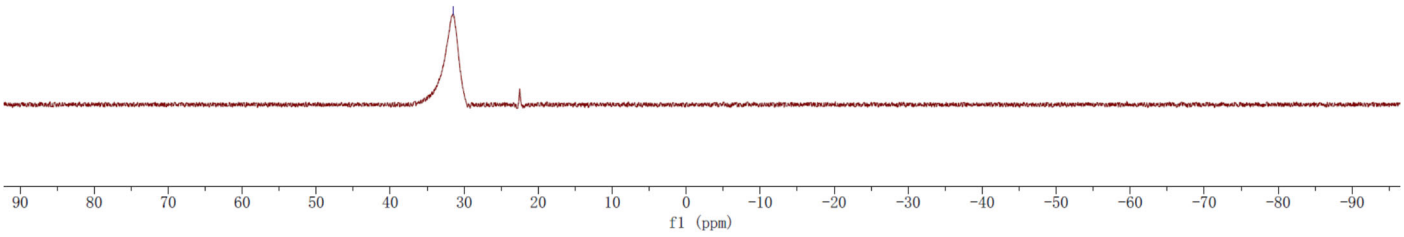

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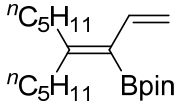

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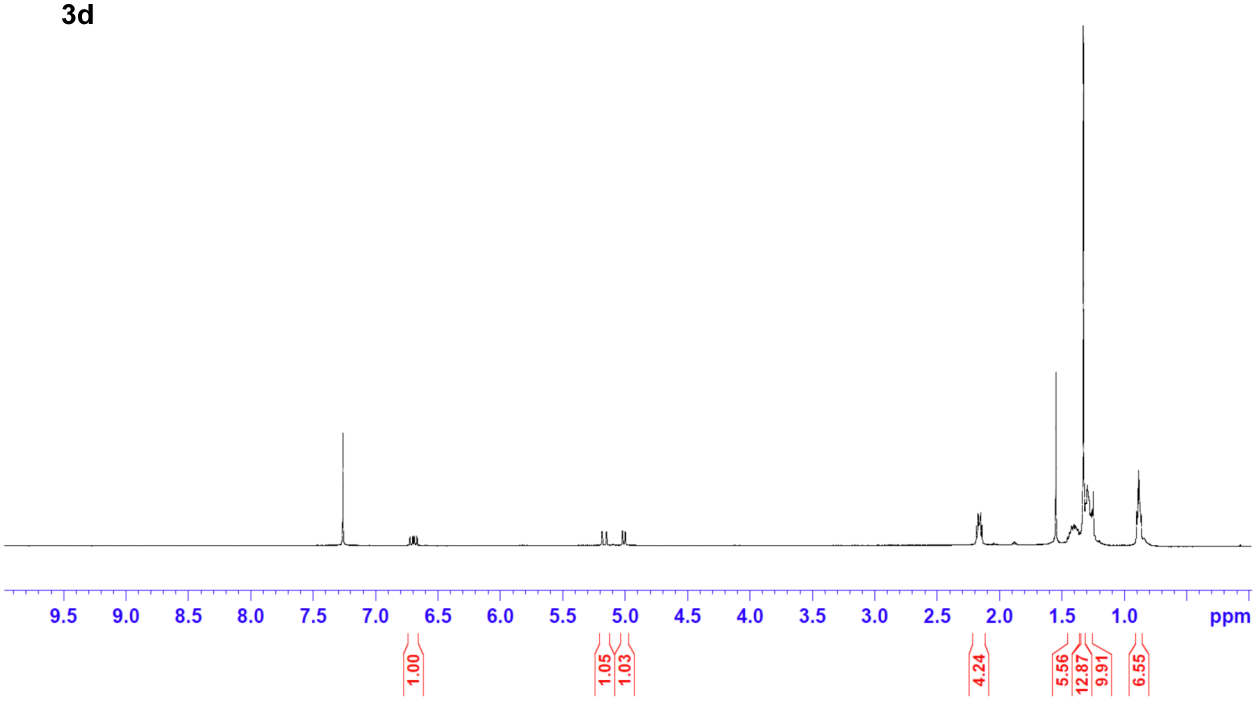

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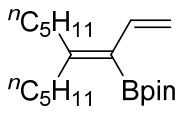

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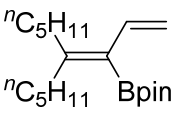
Nov20-2021-ZJY-16-8-B.1.fid



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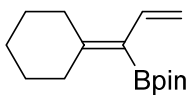

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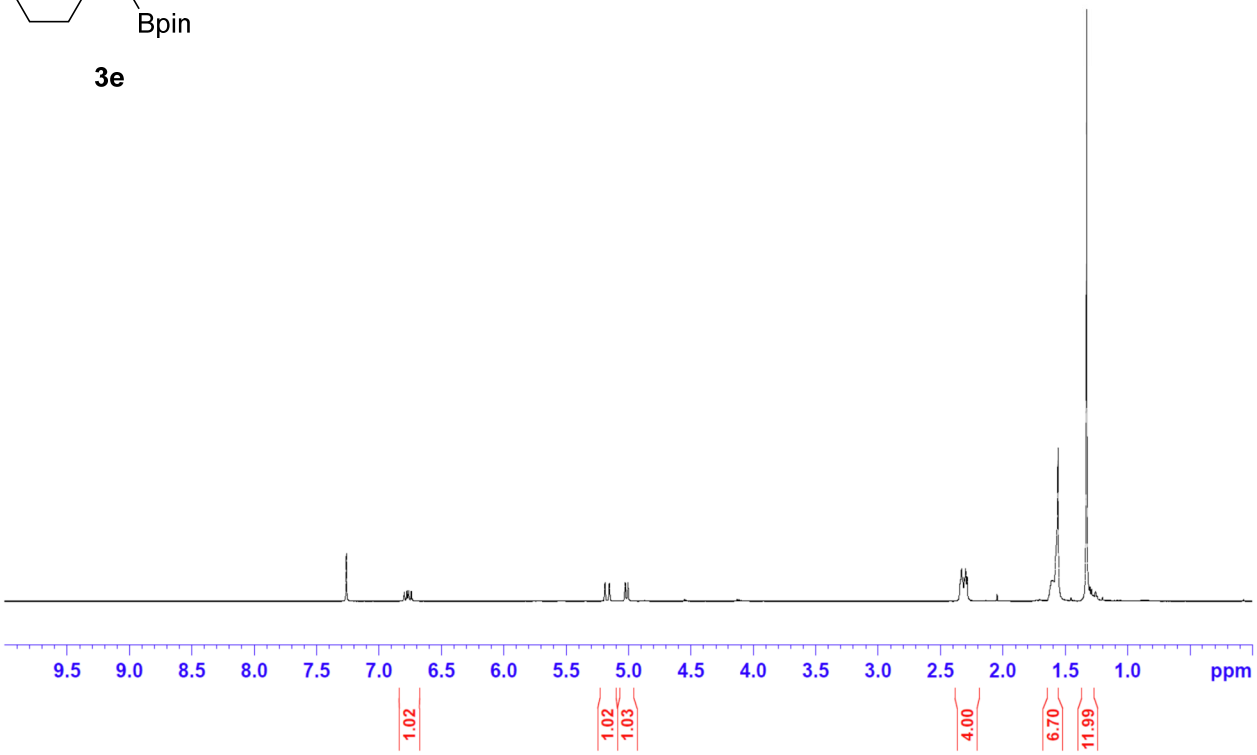

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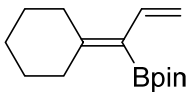

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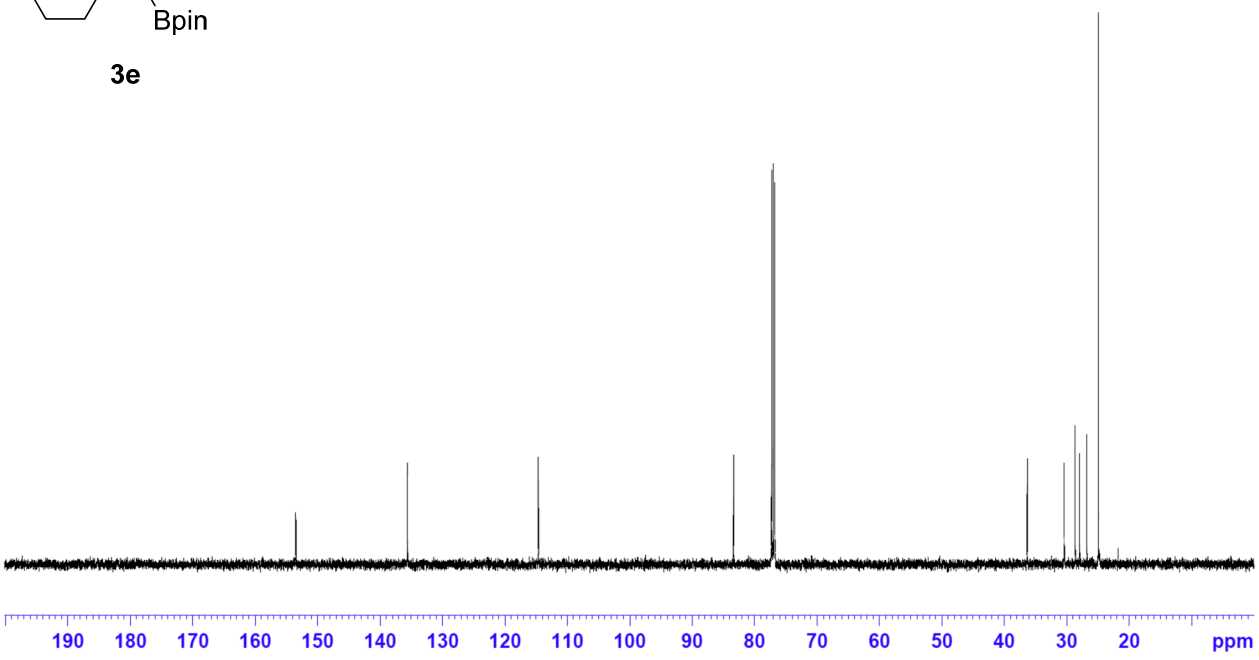
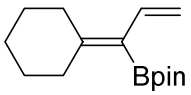


Nov14-2021-ZWD-98-B.10.fid

31.49



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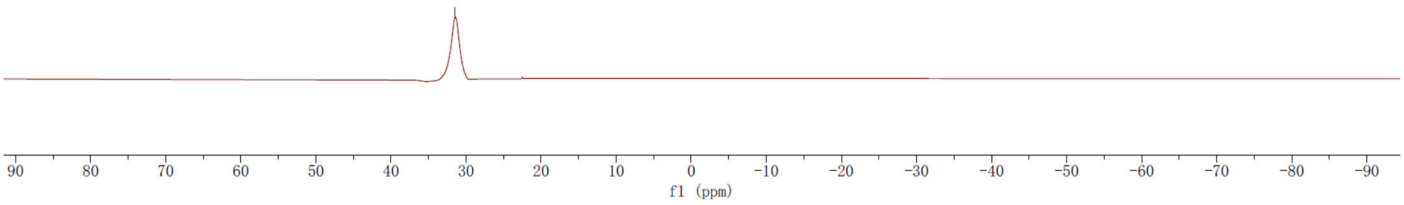

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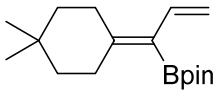

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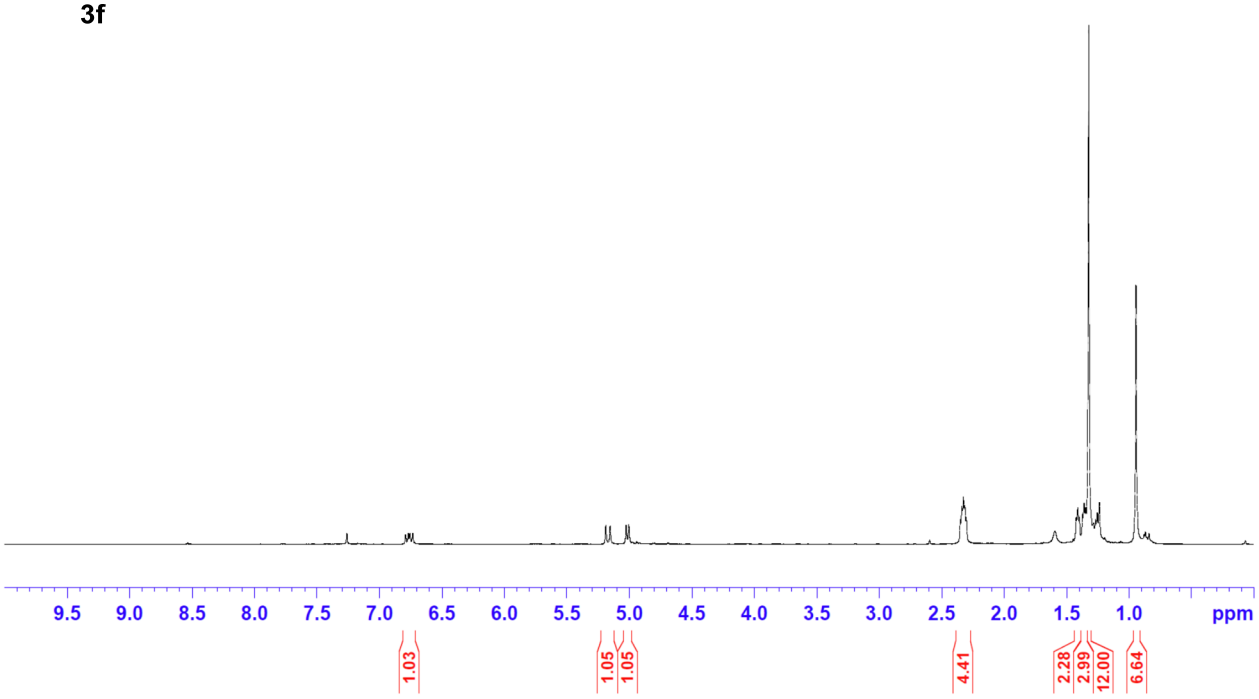

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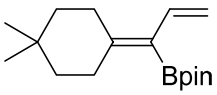

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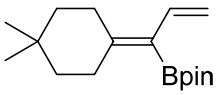
Nov20-2021-ZWD-129-B.1.fid



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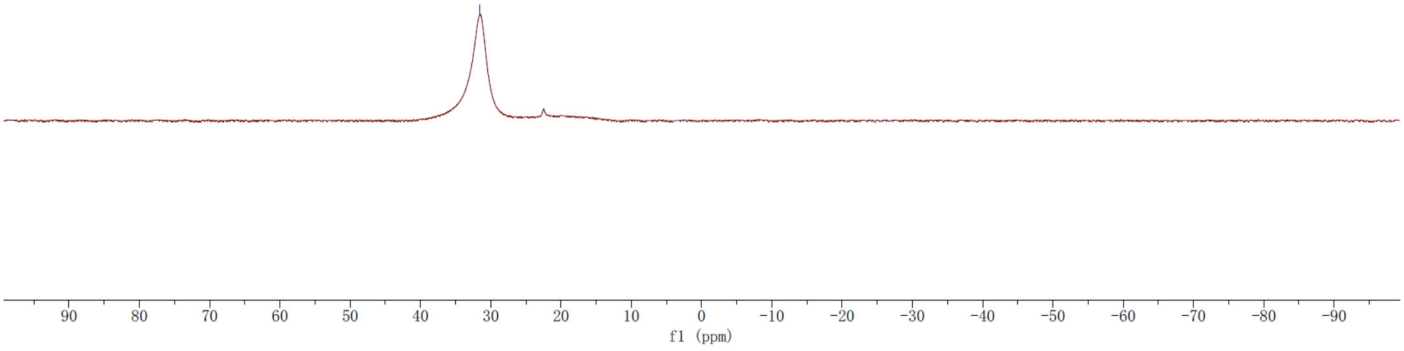

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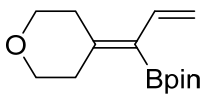

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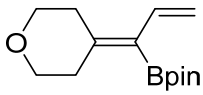

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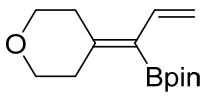
Nov27-2021-ZWD-114-B.1.fid



3g

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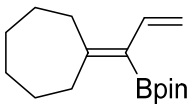

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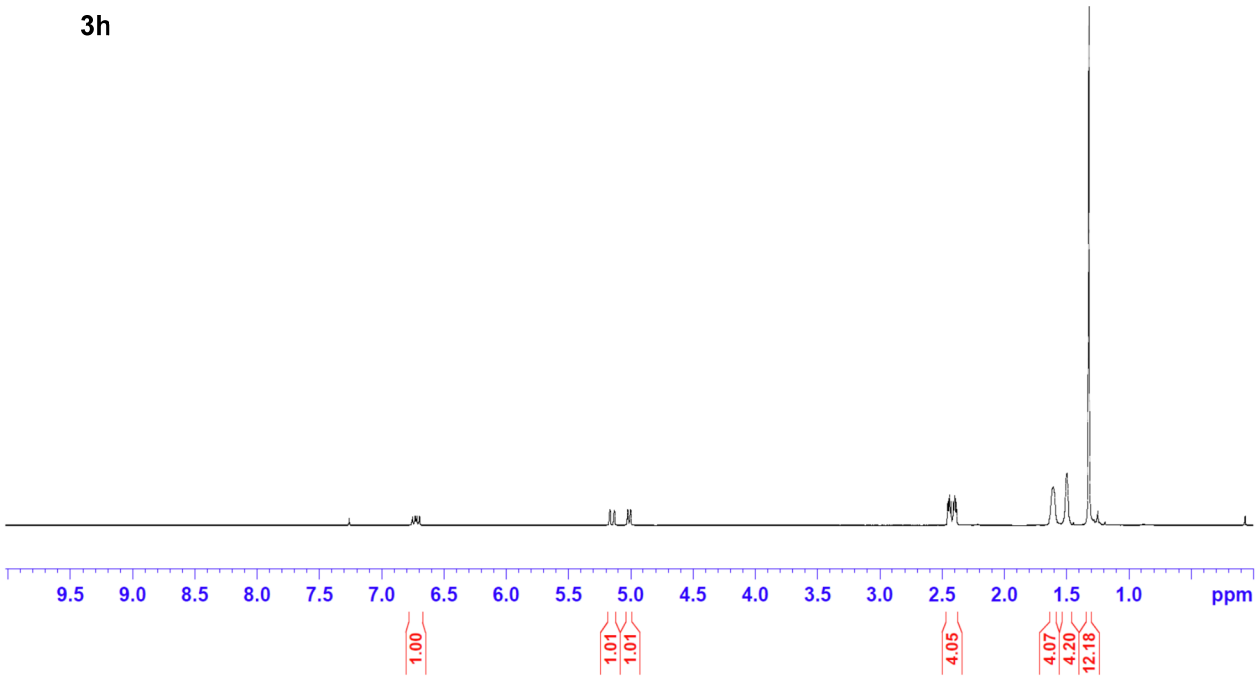

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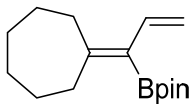

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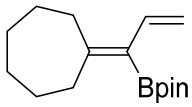

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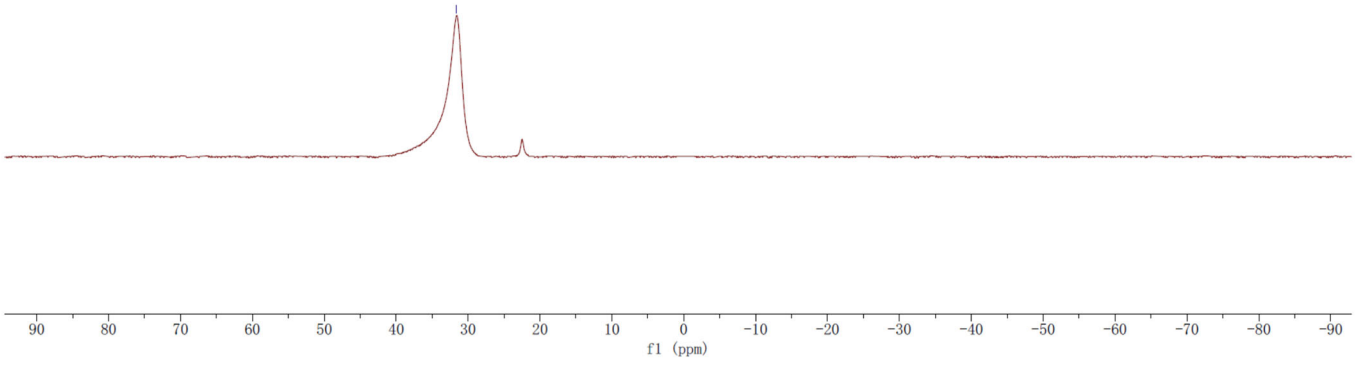
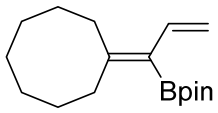


ZWD0097003-2CLM



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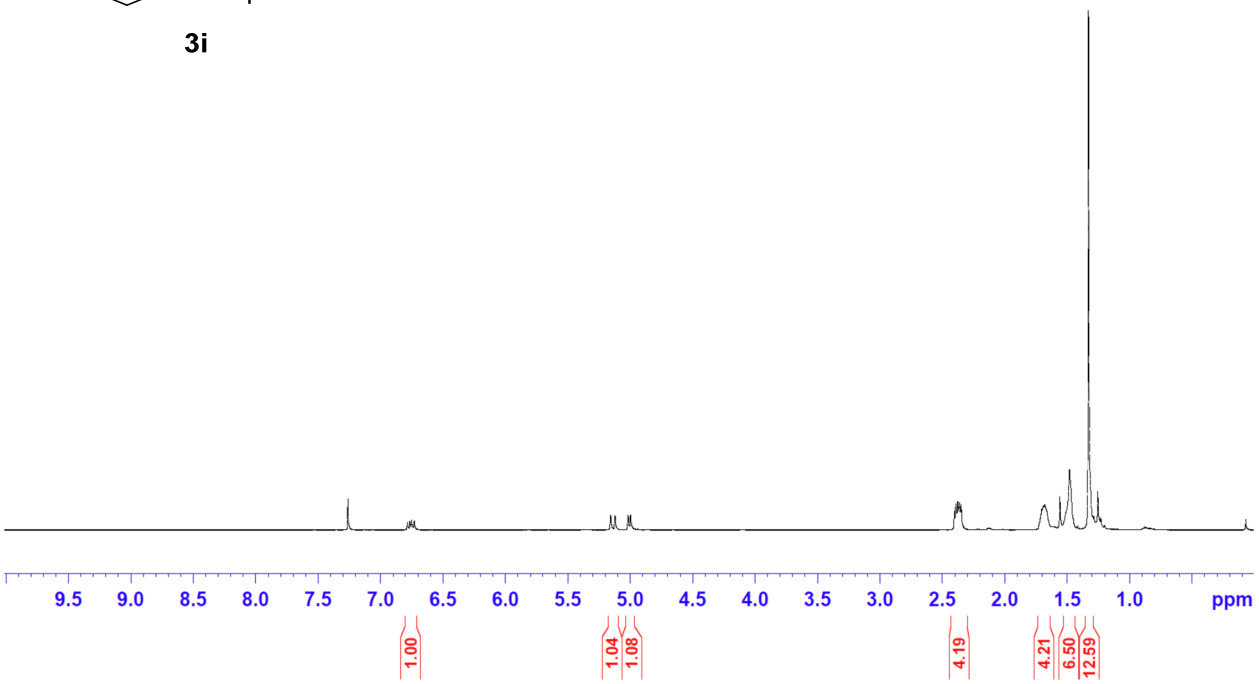
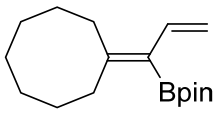


ZWD0097001-C



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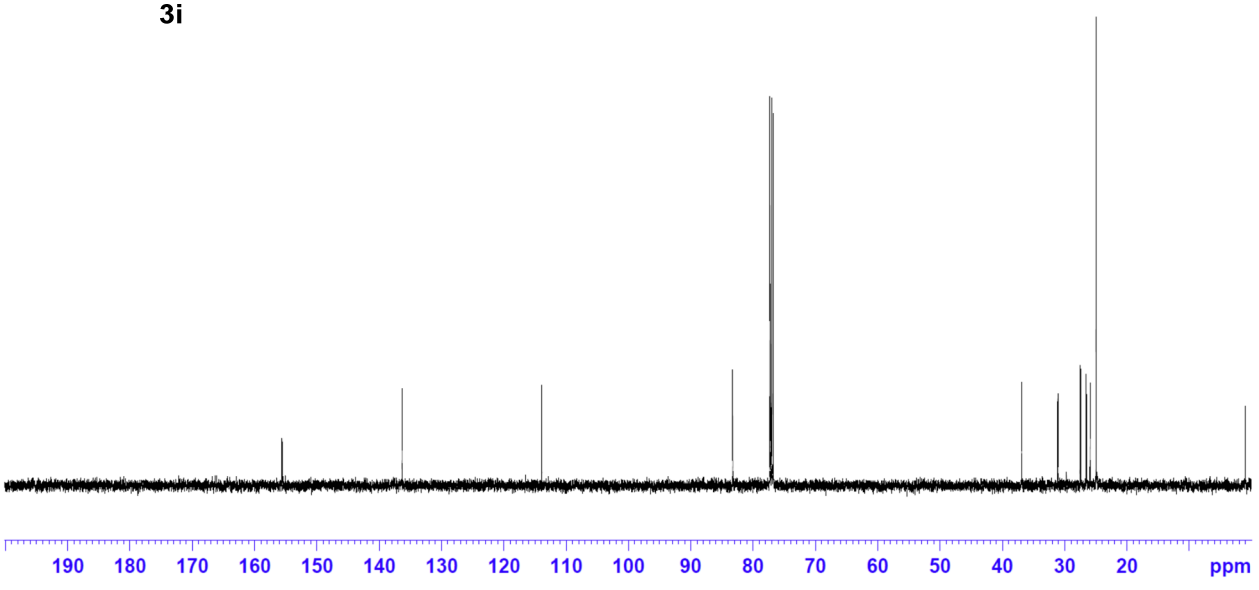
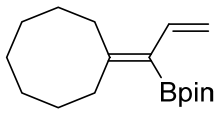


Nov20-2021-ZWD-97-B.1.fid

31.52



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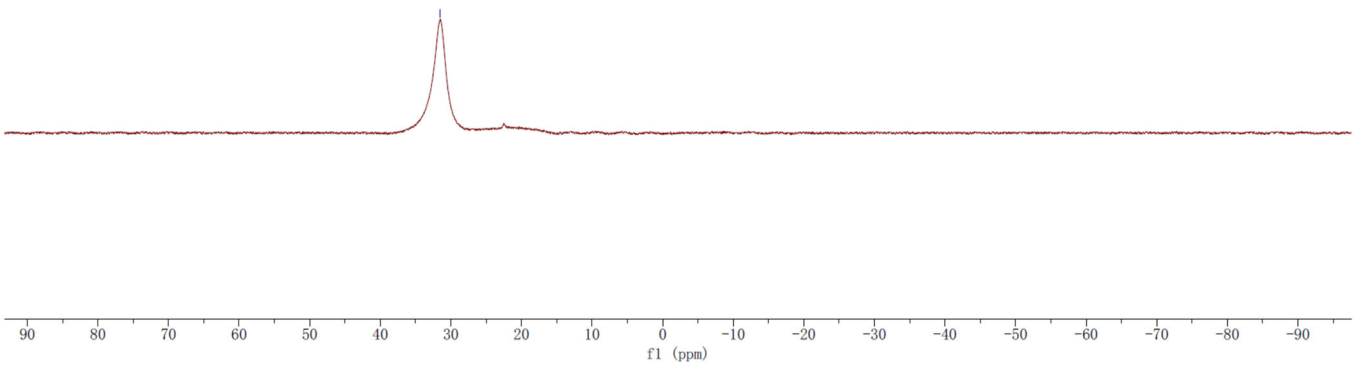
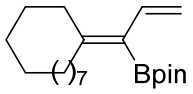


ZWD0113002-2



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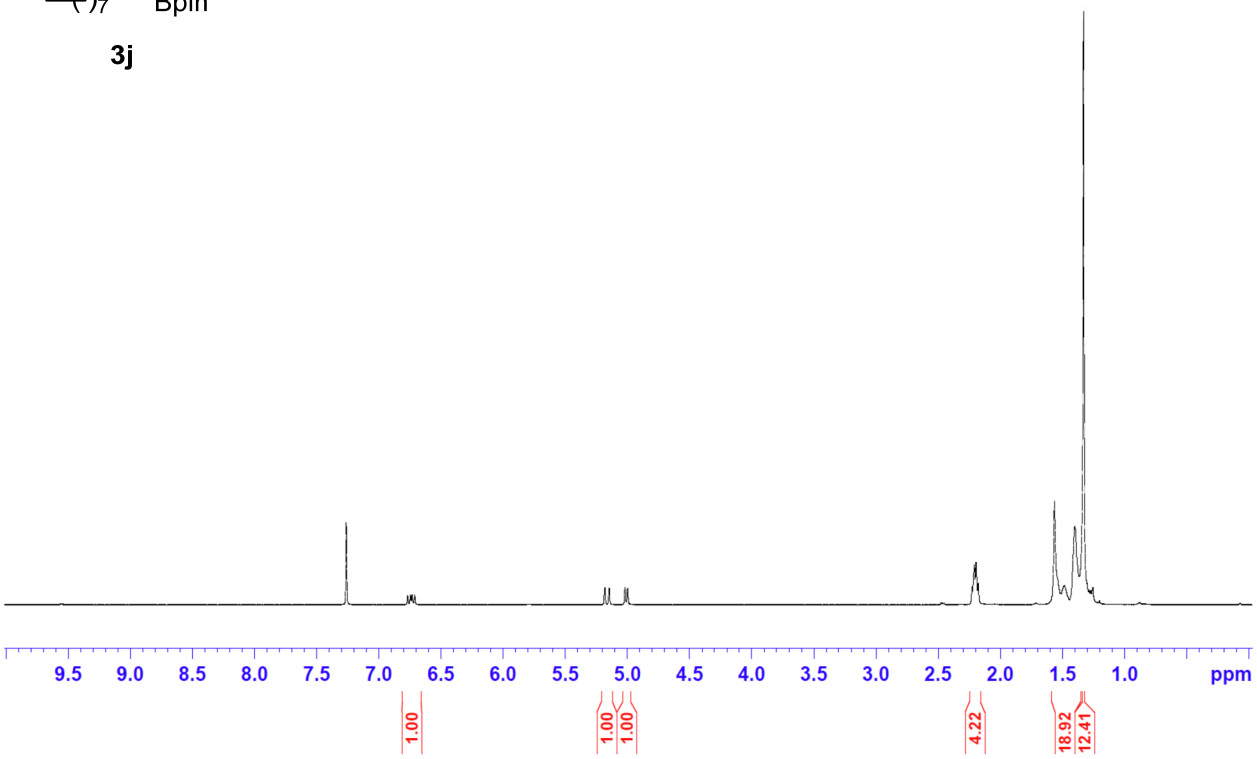
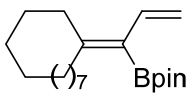
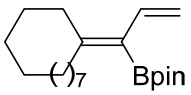


ZWD0113001-C



3j

Nov27-2021-ZWD-113-B.1.fid



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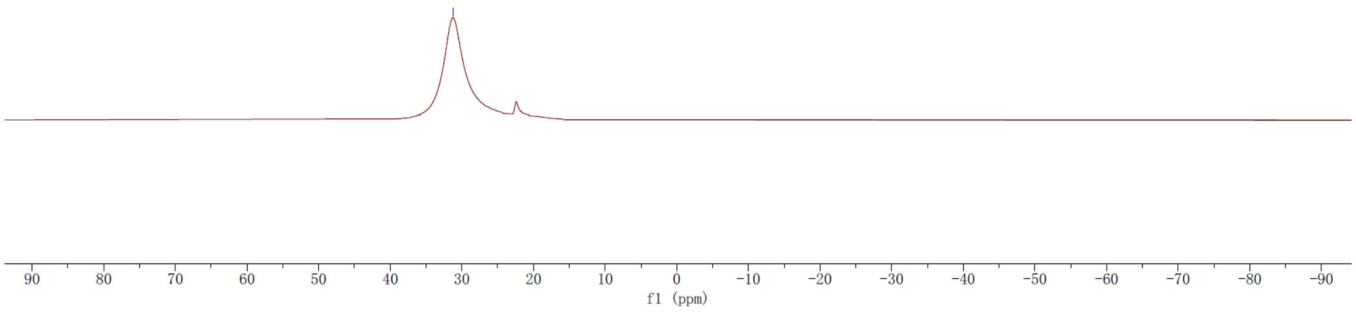
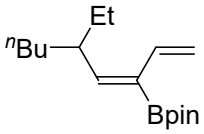


ZWD0134001CLM



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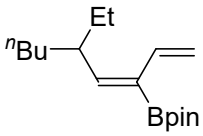

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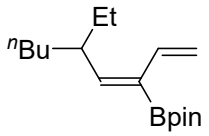
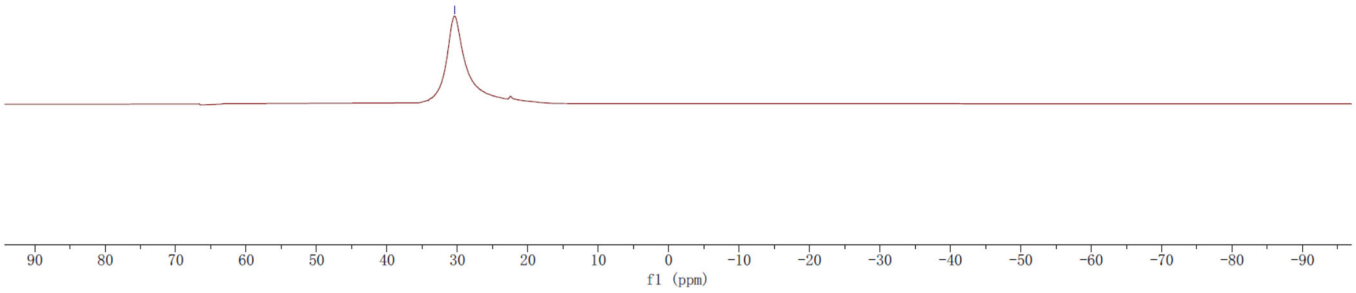
Nov20-2021-ZWD-134-B.1.fid

30.38



3k



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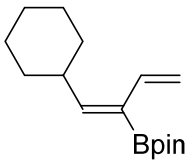

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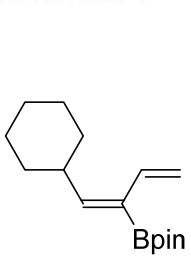
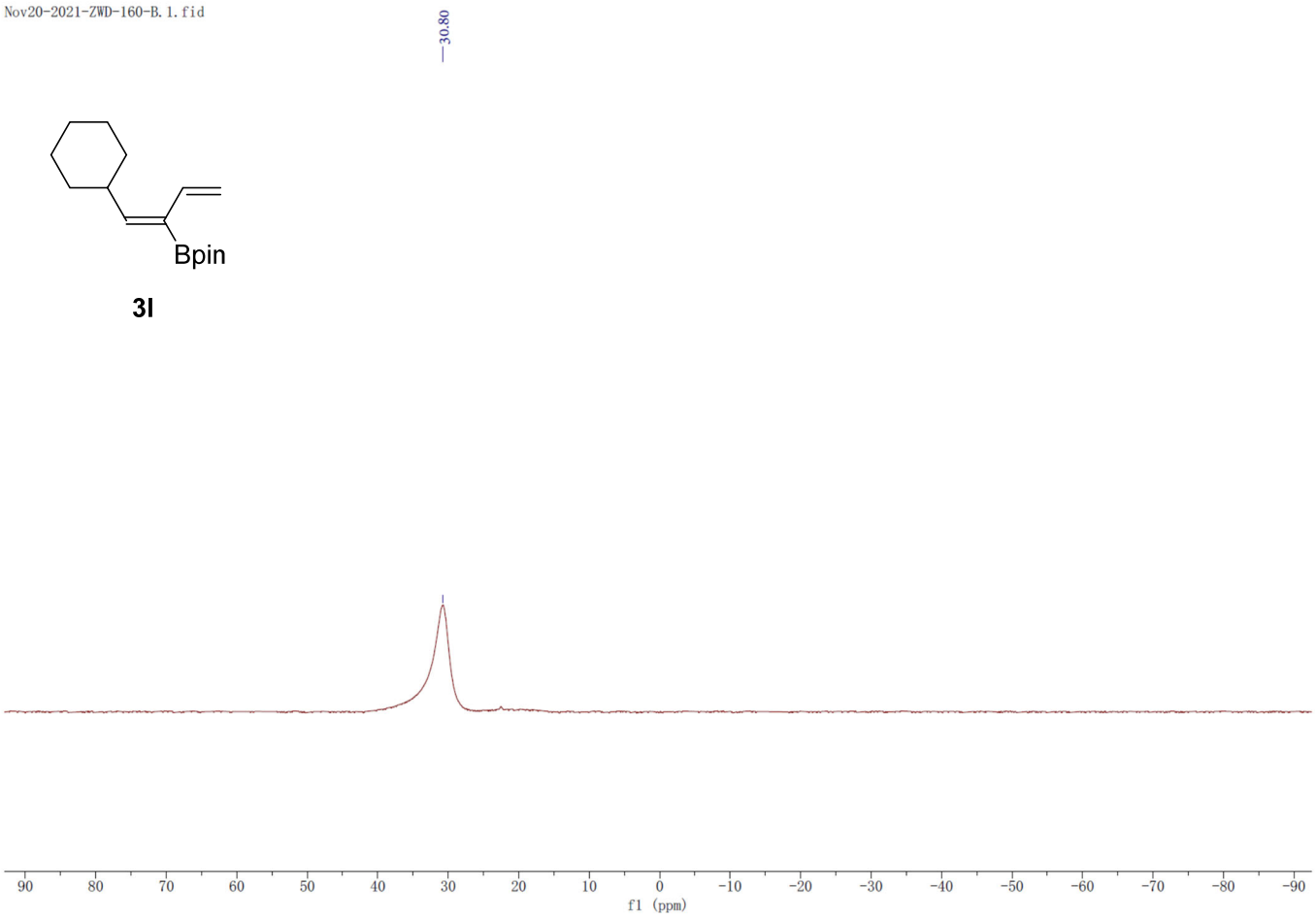


3l

Nov20-2021-ZWD-160-B.1.fid



3l

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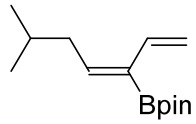
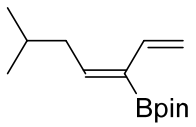


3m

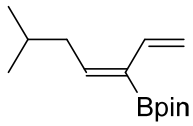
ZWD0107001CLM-C



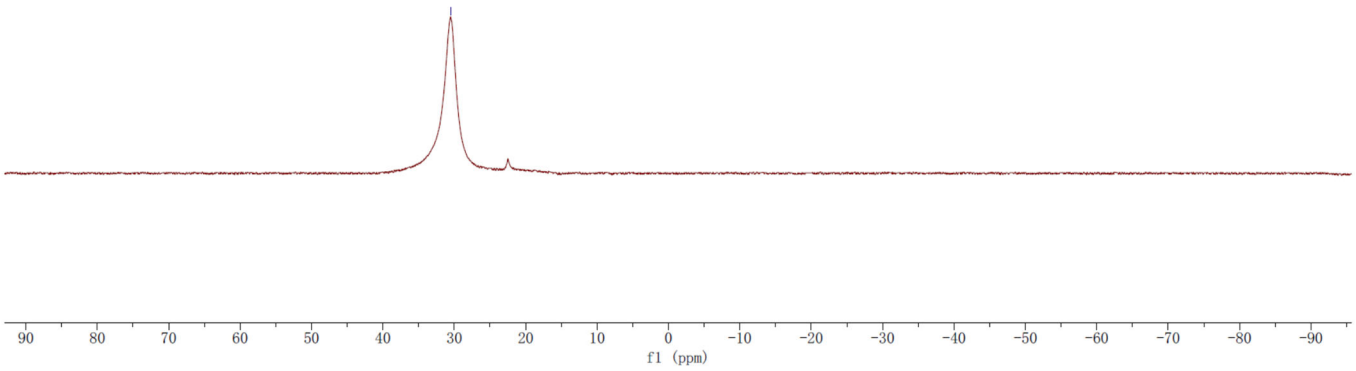
3m

Nov20-2021-ZWD-107-B.1.fid

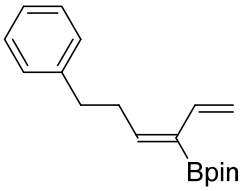
30.51



3m



ZWD0109001-2



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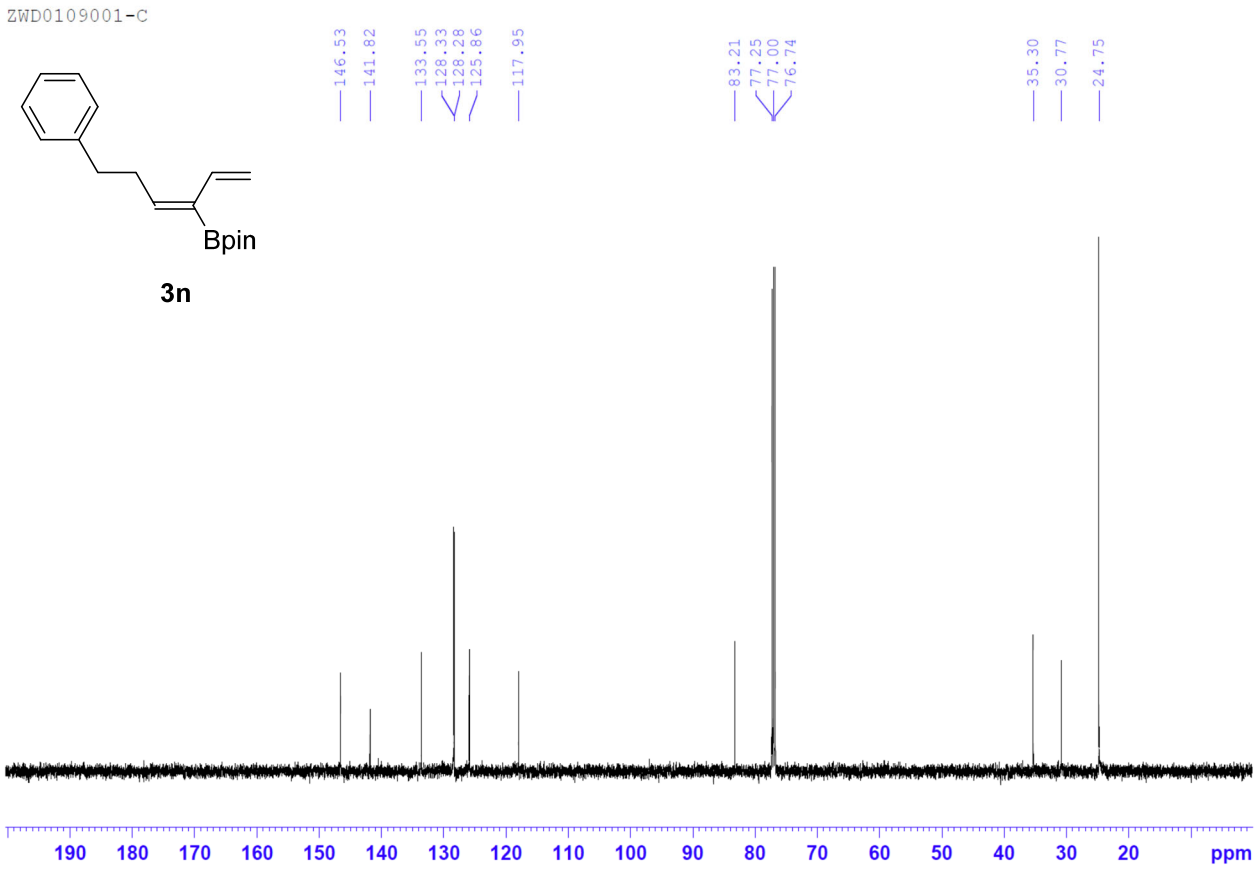
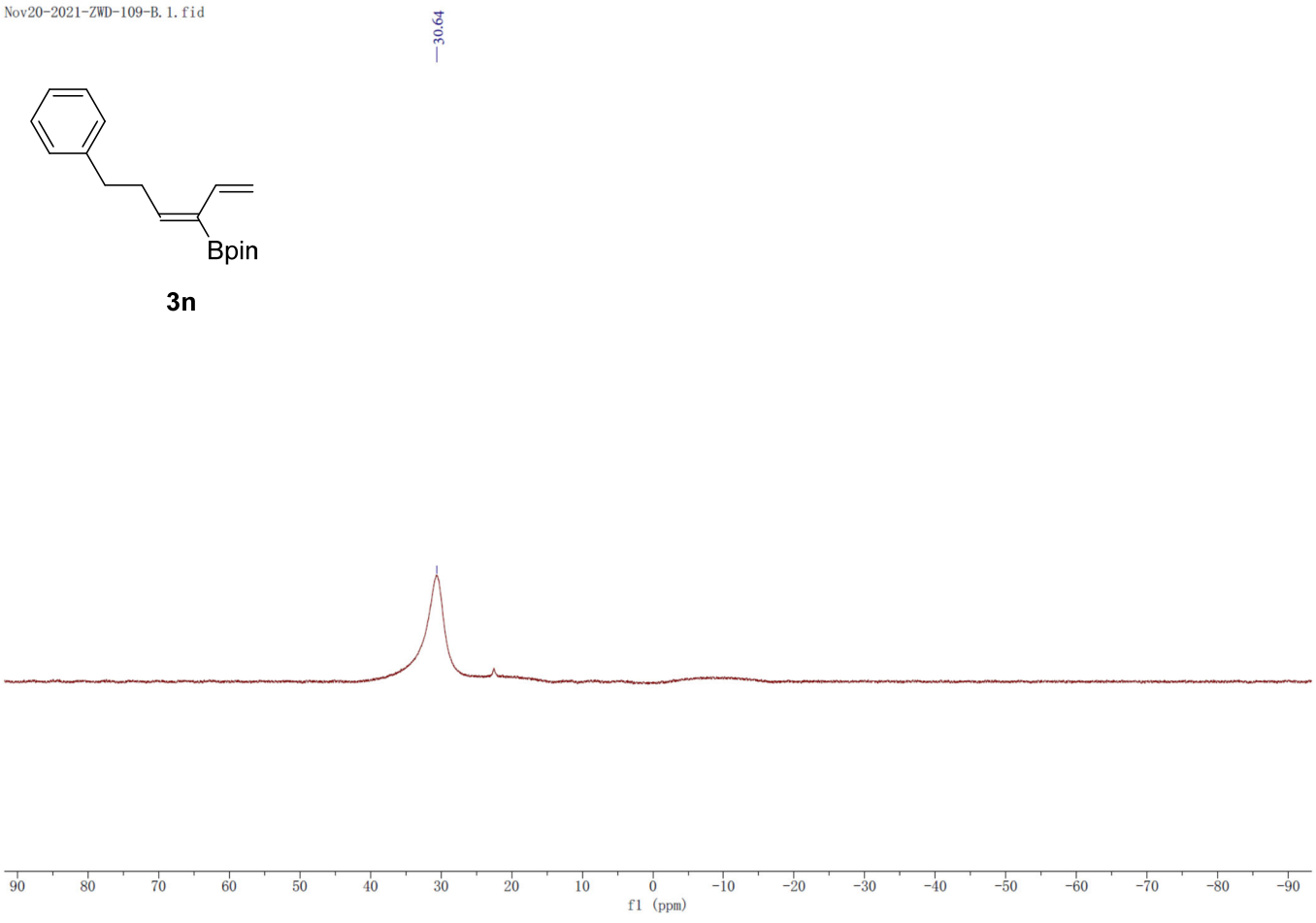
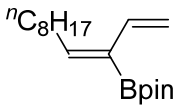
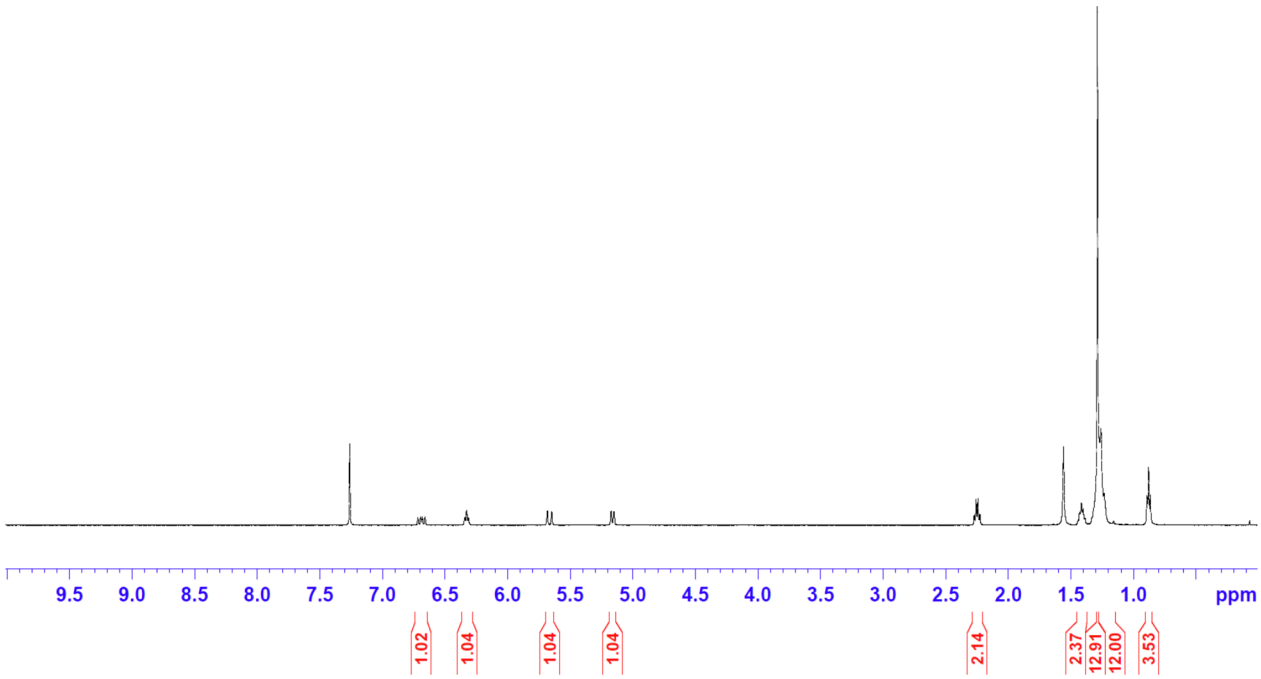

ZWD0109001-C
146.53
141.82
133.55
128.33
128.28
125.86
117.95
83.21
77.25
77.00
76.74
35.30
30.77
24.75
Bpin
3n
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


Nov20-2021-ZWD-109-B.1.fid
30.64
Bpin
3n
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
f1 (ppm)

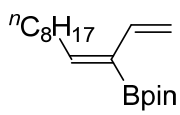
ZWD0153001CLM



3o



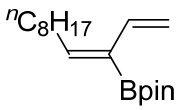
ZJY0017001CLM



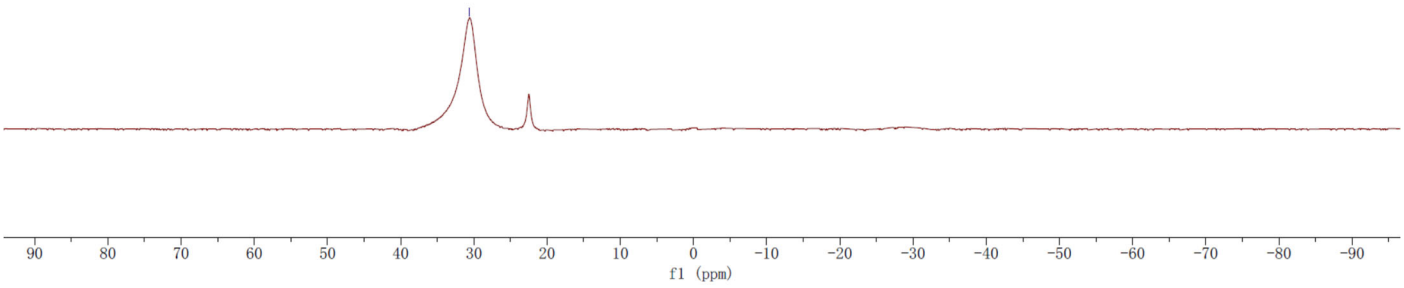
3o

Nov20-2021-ZWD-153-B.1.fid

30.57



3o



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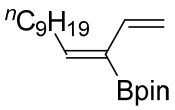
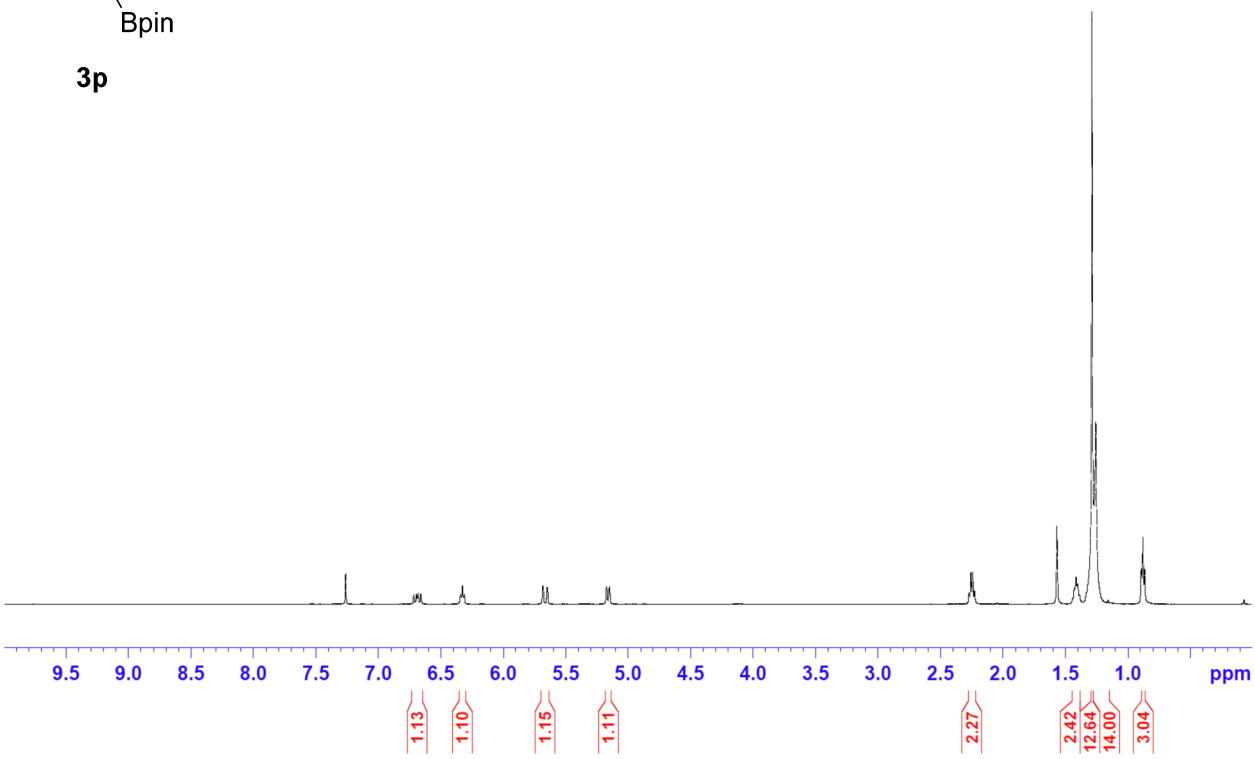
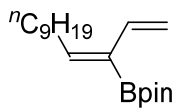


3p

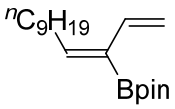


ZWD0143001-C



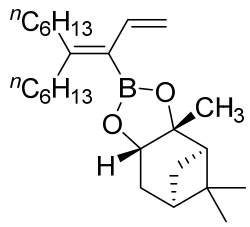
3p

Nov20-2021-ZWD-143-B.1.fid

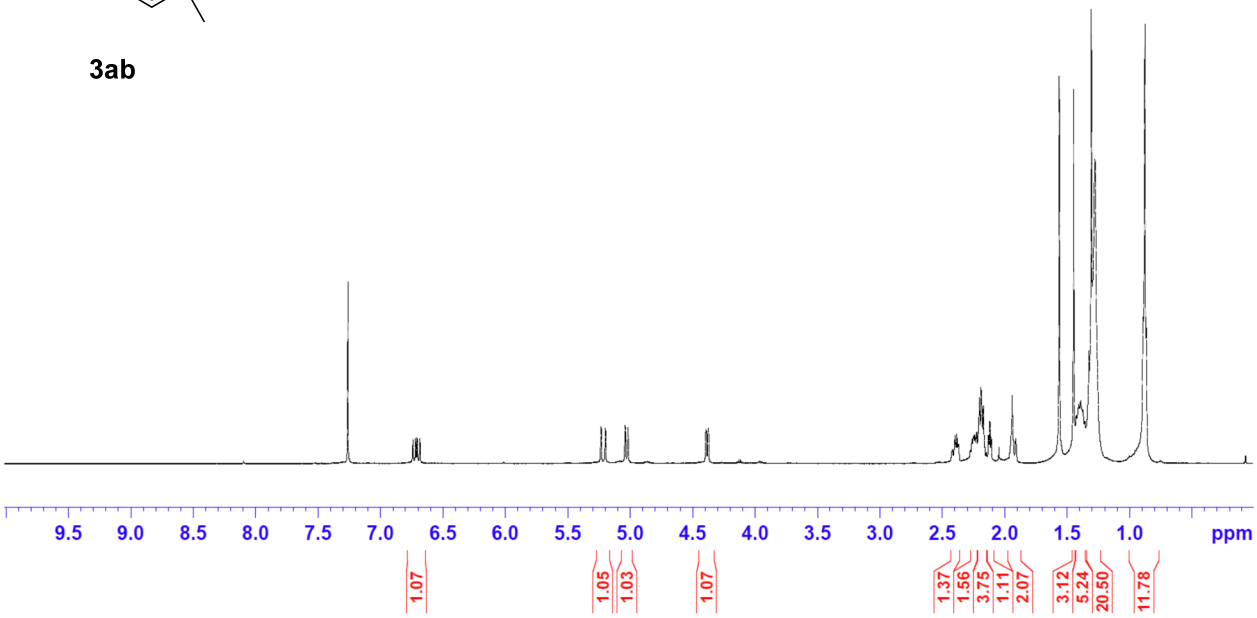


3p

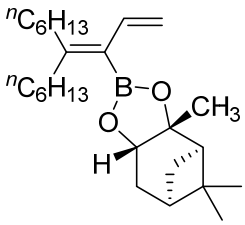
ZWD0042040-CLM3



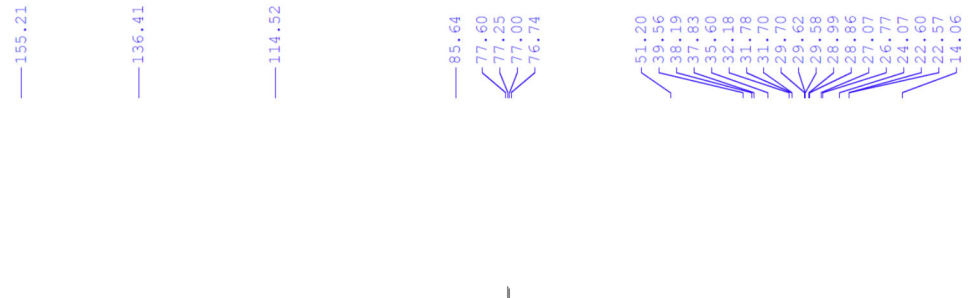
3ab

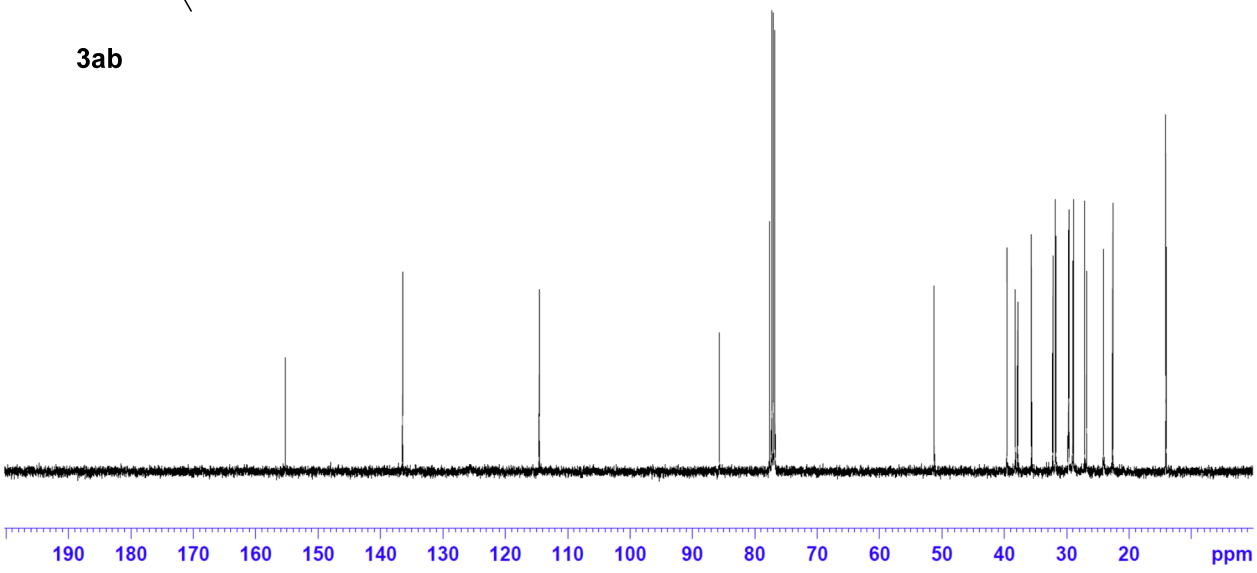


ZWD0042040-3-C



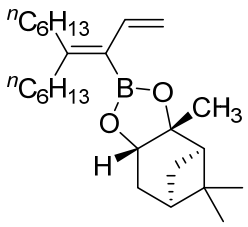
3ab



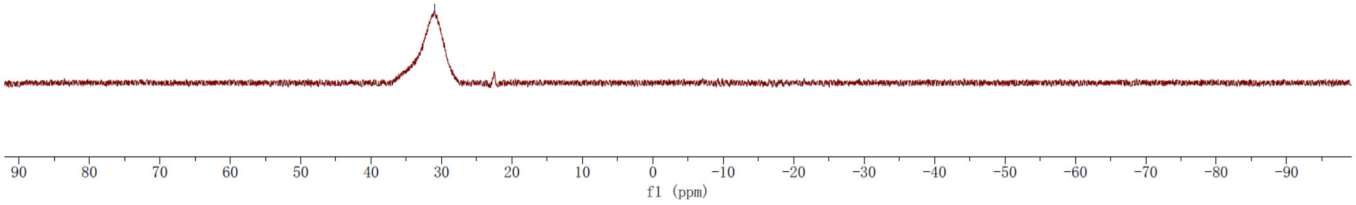


Nov14-2021-ZWD-42-40-B.10.fid

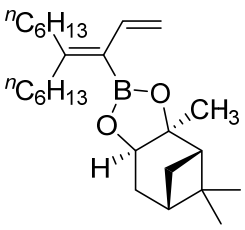
30.99



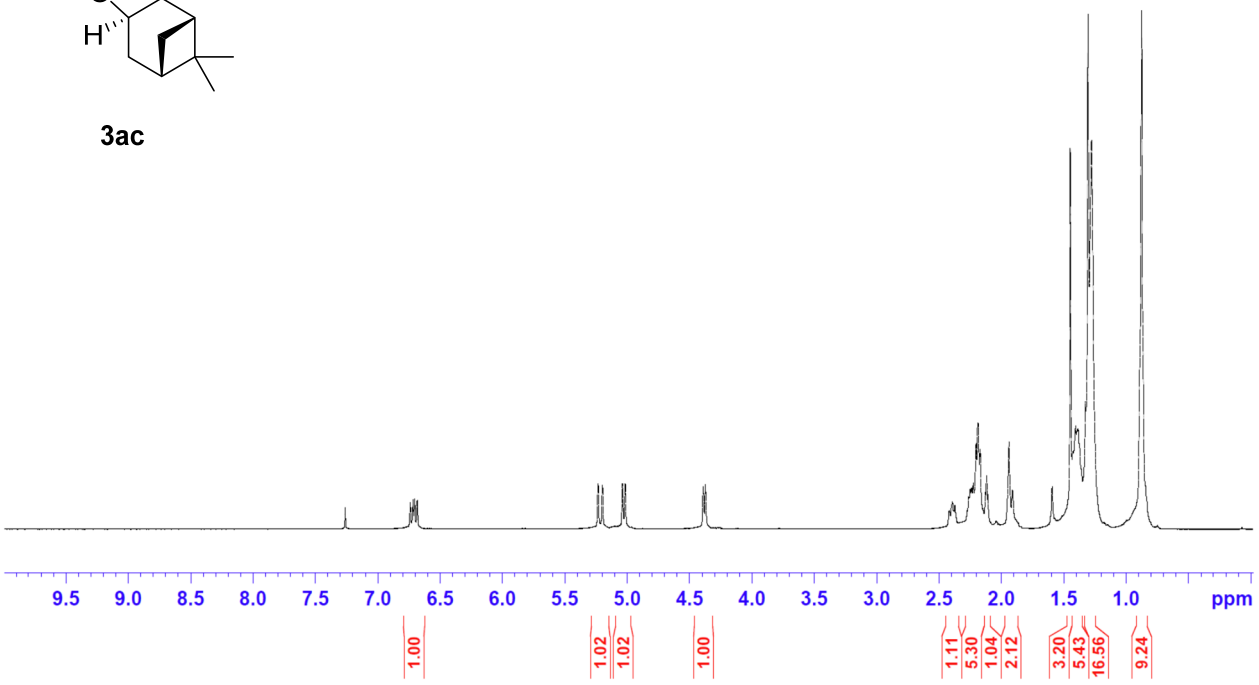
3ab

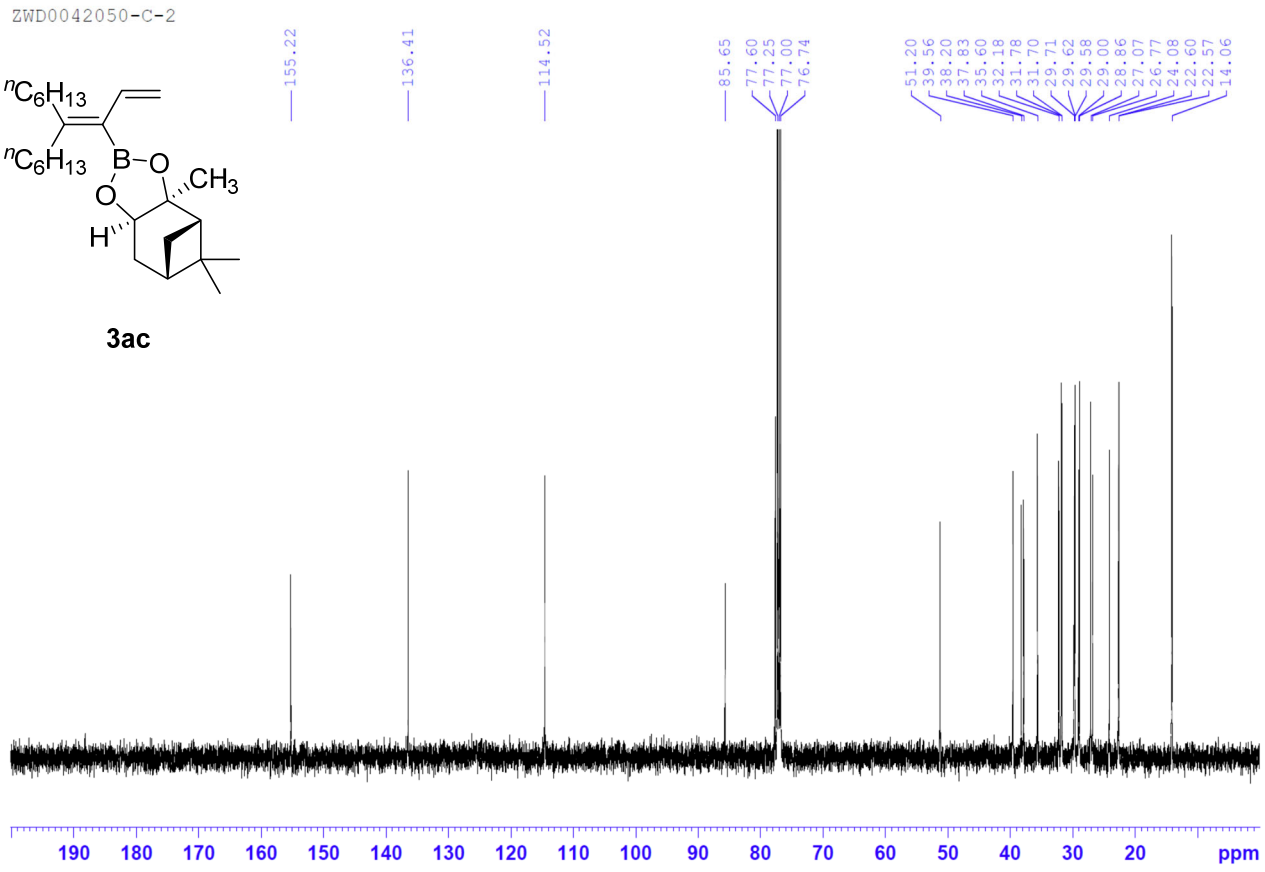


ZWD0042050CLM

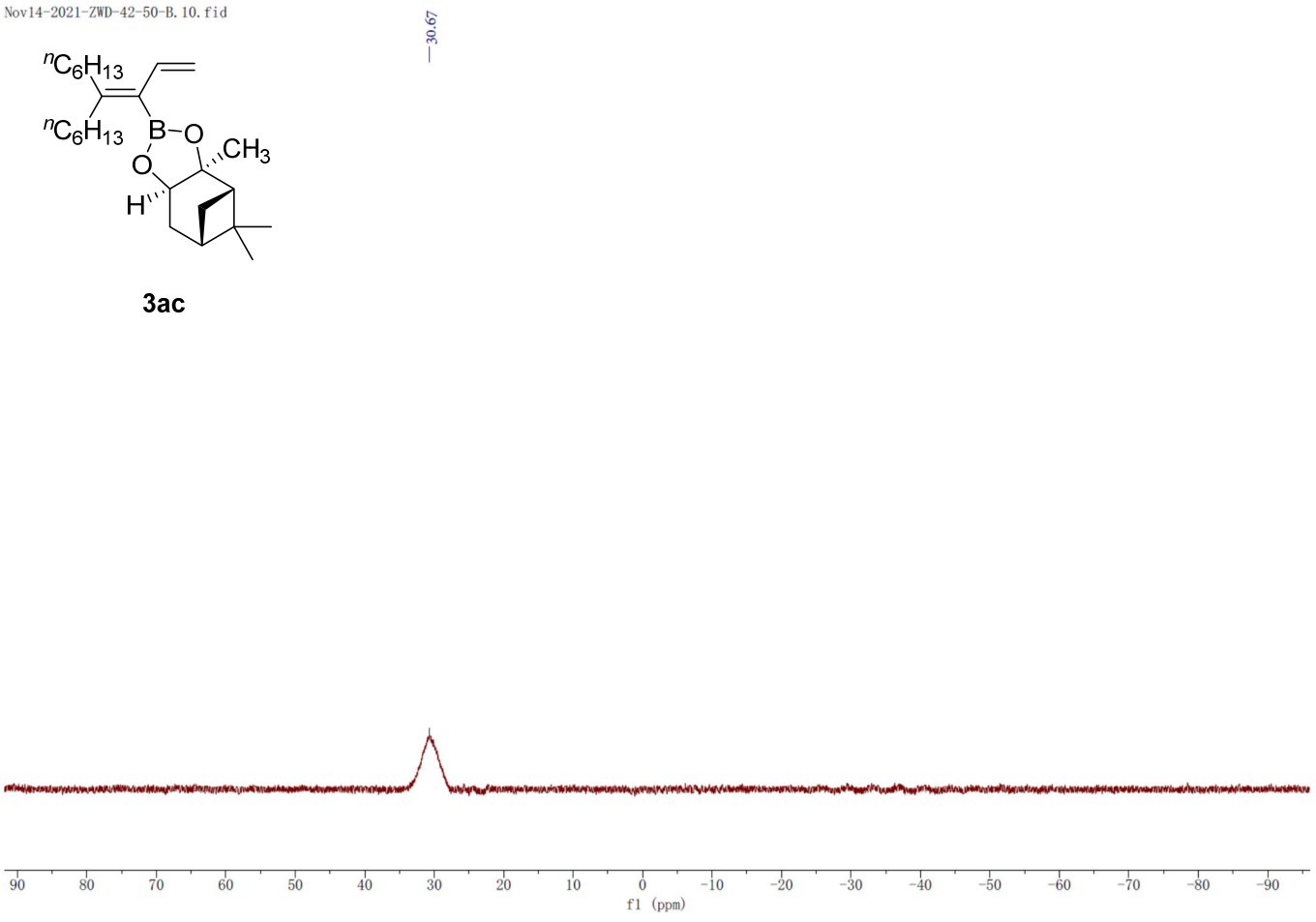


3ac



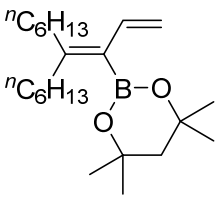

ZWD0042050-C-2
155.22
136.41
114.52
85.65
77.60
77.25
77.00
76.74
51.20
39.56
38.20
37.83
35.60
32.18
31.78
31.70
29.71
29.62
29.58
29.00
28.86
27.07
26.77
24.08
22.60
22.57
14.06
^{n}C$_6$H$_{13}$
^{n}C$_6$H$_{13}$
B–O
CH$_3$
O
H
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

3ac

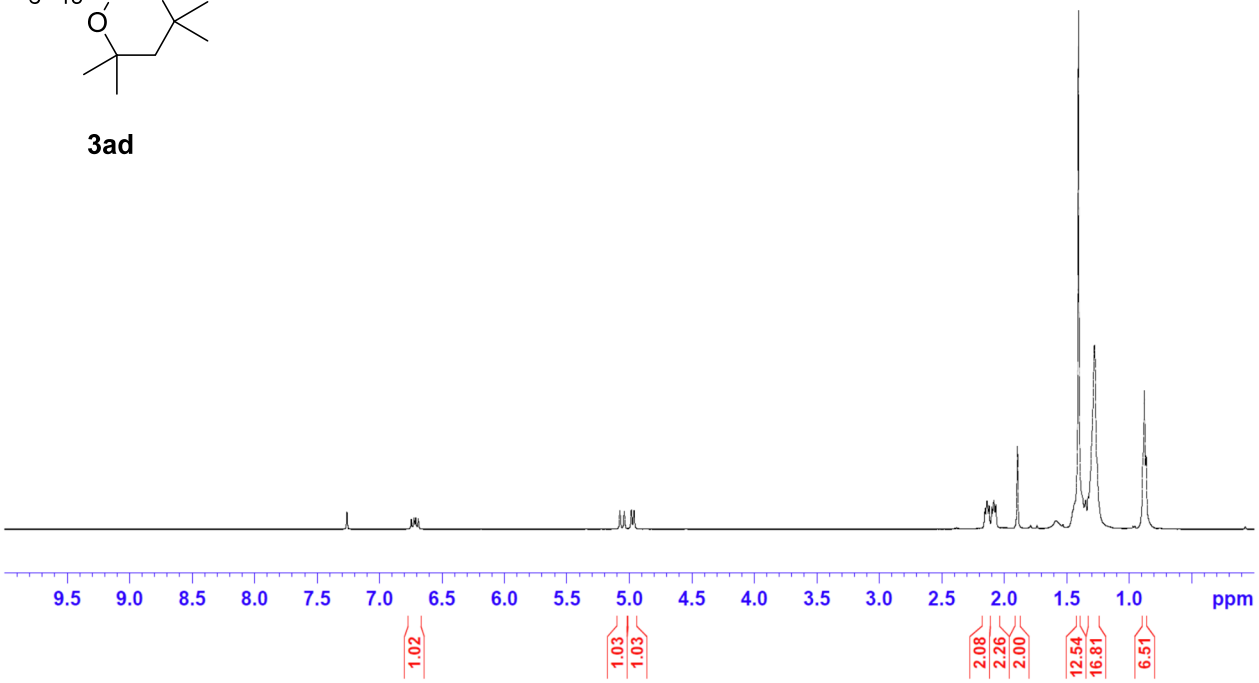

Nov14-2021-ZWD-42-50-B.10.fid
30.67
^{n}C$_6$H$_{13}$
^{n}C$_6$H$_{13}$
B–O
CH$_3$
O
H
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

3ac

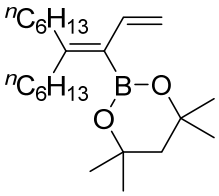
ZWD0042051CLM-2



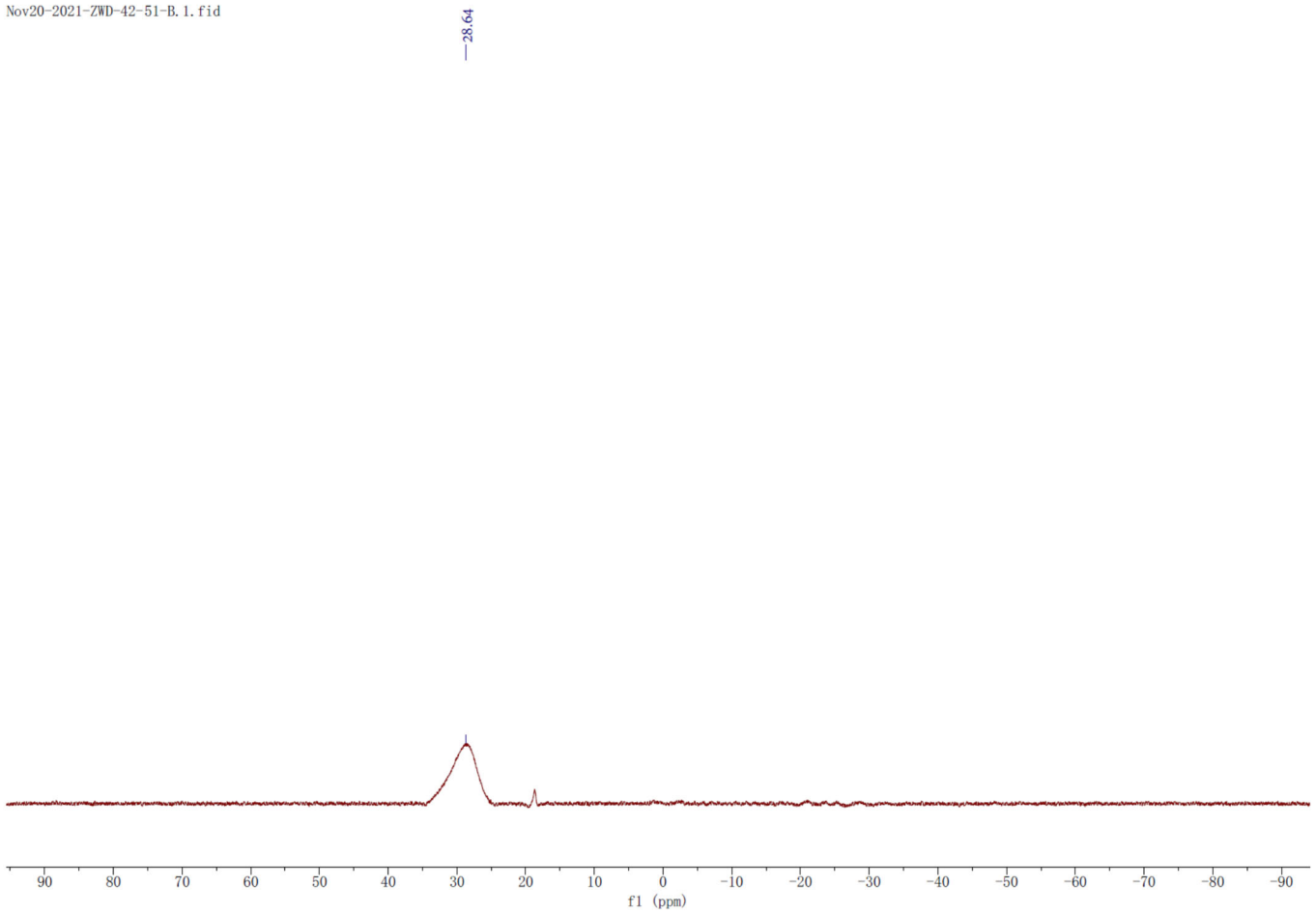
3ad



ZWD0042051-C



3ad


Nov20-2021-ZWD-42-51-B.1.fid
28.64
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
f1 (ppm)