Supporting Information

Synthesis of Oxaboranes via Nickel-Catalyzed Dearylative Cyclocondensation

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General Experimental Details

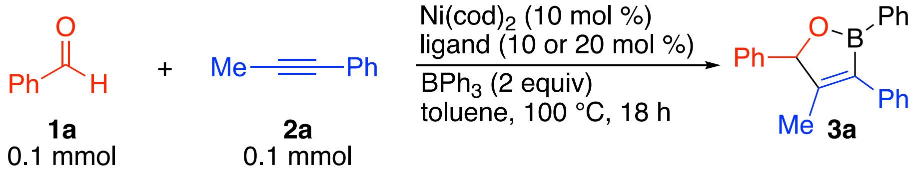
All air-sensitive procedures were conducted under inert atmosphere in a nitrogen-filled dry box or by standard Schlenk techniques. All reactions were performed under nitrogen unless otherwise stated. All glassware for moisture sensitive reactions were dried at 140 °C in an oven. Tetrahydrofuran (THF) and dimethylformamide (DMF) were degassed by purging with argon for 45 minutes and dried with a solvent purification system by passing through a one-meter column of activated alumina. Anhydrous 1,4-dioxane was purchased from Sigma-Aldrich and used as received. Methanol was purchased from Sigma-Aldrich and vacuumed distilled into a receiving flask with 4Å molecular sieves before use. Flash column chromatography was performed on SiliFlash® P60 silica gel (40-63 μm, 60Å) or using a Teledyne Isco Combiflash® Rf system with RediSep GoldTM columns using hexane/ethyl acetate. Reaction products were visualized on TLC under UV light. Prep-HPLC was performed on a Waters HPLC System. Detection was acquired on a 2998 Photodiode Array Detector and a SunFire C18 column 5 μm.

HRMS (ESI and EI) analysis was performed at the Iowa State University Chemical Instrumentation Facility on an Agilent 6540 QTOF spectrometer and an Agilent 7250 GC Q-TOF spectrometer. NMR spectra were acquired on Varian MR-400, Bruker Avance NEO 400, and Bruker Avance III 600 spectrometers at the Iowa State University Chemical Instrumentation Facility. Chemical shifts are reported in ppm relative to a residual solvent peak ($CDCl_3$ = 7.26 ppm for ^1H and 77.16 ppm for ^{13}C). ^{19}F NMR shifts are reported based on indirect reference to $CDCl_3$.[1] Carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation.[2]

Materials

1-Phenyl-1-propyne, 3-hexyne, 4-octyne, benzaldehyde, p-tolualdehyde, 4-(dimethylamino)benzaldehyde, 3-methylbenzaldehyde, 3-methoxybenzaldehyde, furfural, thiophene-2-carbaldehyde, pivaldehyde, sodium perborate tetrahydrate, ethanol, copper(I) iodide, 1,10-phenanthroline, 4-iodotoluene, potassium carbonate, potassium fluoride, palladium(II) acetate, methyl 4-iodobenzoate, and bis(triphenylphosphine)palladium(II) dichloride were purchased from Sigma Aldrich. Diphenylacetylene and 2-methoxybenzaldehyde were purchased from Alfa Aesar. 4-Methoxybenzaldehyde and 2-naphthaldehyde were purchased from AK Scientific. 4-Fluorobenzaldehyde, 4-(trifluoromethyl)benzaldehyde, and 1-hexyne were purchased from Oakwood Chemical. Sodium hydroxide, silver nitrate, triethylamine, potassium iodine, and potassium hydroxide were purchased from Fischer Scientific. Palladium-tetrakis(triphenylphosphine) and tri-*tert*-butylphosphine were purchased from Strem. Methyl 4-formylbenzoate and 1-fluoro-3-iodobenzene were purchased from Combi-Blocks. 5-bromobenzofuran was purchased from Carbocore. Boric acid was purchased from Baker Chemical Company. Triphenylborane is a known compound and was synthesized according to literature procedure.[3] Alkynes **2b**,[4] **2c**,[5] **2d**,[6] **2e**,[4] **2g**,[7] **2h-2i**,[8] **2j**,[7] **2l-2m**,[9] and **2o**[10] are known compounds and were synthesized according to literature procedures. Aldehyde **1y** is a known compound and was synthesized according to literature procedure.[11]

Table S1. Ligand and Solvent Screen for Ni-Catalyzed Dearylative Cyclocondensation[a]



entry	ligand	mol %	solvent	yield **3a** (%)[b]
1	PBu_3	20	toluene	70
2	P^tBu_3	20	toluene	<5
3	P(*o*-tol)$_3$	20	toluene	<10
4	PPh_3	20	toluene	37
5	PCy_3	20	toluene	10
6	P^iPr_3	20	toluene	25
7	P(2,4,6-trimethoxyphenyl)$_3$	20	toluene	<5
8	IPr	10	toluene	<5
9	dppp	10	toluene	<5
10	4,4′-di-tert-butyl-2,2′-bipyridine	10	toluene	18
11	PBu_3	20	THF	96
12	PBu_3	20	dioxane	67
13	PBu_3	20	CPME	76

[a]Reaction conditions: **1a** (0.100 mmol), **2** (0.100 mmol), BPh_3 (0.200 mmol), Ni(cod)$_2$ (0.010 mmol), PBu_3 (0.020 mmol), solvent (0.3 mL, 0.3 M), at 100 °C for 18 h. [b]Determined by ^1H NMR with dibromomethane as an internal standard.

Procedure for Ligand Screen for Ni-Catalyzed Dearylative Cyclocondensation

In a glovebox atmosphere, an oven dried 1-dram vial was charged with Ni(cod)$_2$ (2.8 mg, 0.010 mmol), benzaldehyde **1a** (10.6 mg, 0.100 mmol), 1-phenyl-1-propyne (11.6 mg, 0.100 mmol), ligand (0.010 or 0.020 mmol), triphenylborane (48.4 mg, 0.200 mmol), and solvent (0.3 mL, 0.3 M). The resulting solution was stirred at 100 °C in a block heater for 18 h. The reaction was cooled to room temperature, diluted with DCM, and concentrated. The residue was dissolved in 70:30 hexane/EtOAc and filtered through a short plug of silica gel, eluting with additional 70:30

hexane/EtOAc. The yield of the reaction was determined by ^{1}H NMR with dibromomethane as an internal standard.

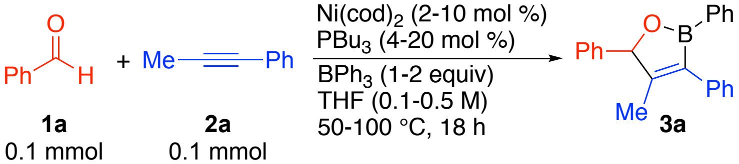
Design of Experiment (DoE) Factor Screen for Dearylative Cyclocondensation

-Response: Percent Yield

-Factors: Catalyst loading, BPh$_3$ loading, concentration, temperature

-JMP Pro 16® software was used to generate and analyze the 14 reactions of the D-optimal design.

Table S2. Design of Experiment Factor Screen for Dearylative Cyclocondensation[a]



entry	catalyst loading (mol %)	BPh$_3$ loading (equiv)	concentration (M)	temperature (°C)	yield **3a** (%)[b]
1	10	2	0.1	100	99
2	2	1.5	0.1	100	63
3	10	1.5	0.5	50	71
4	6	1.5	0.3	75	88
5	2	1	0.5	50	33
6	10	2	0.5	75	83
7	2	1	0.1	75	70
8	6	2	0.1	50	97
9	2	2	0.5	100	81
10	6	1	0.5	100	60
11	6	1.5	0.3	75	80
12	10	1	0.1	50	77
13	10	1	0.3	100	44
14	2	2	0.3	50	74

[a] Reaction conditions: **1a** (0.100 mmol), **2** (0.100 mmol), BPh$_3$ (0.100-0.200 mmol), Ni(cod)$_2$ (0.002-0.010 mmol), PBu$_3$ (0.004-0.020 mmol), THF (0.2-1.0 mL, 0.1-0.5 M), at 50-100 °C for 18 h. [b] Determined by ^{1}H NMR with dibromomethane as an internal standard.

Procedure for Design of Experiment Factor Screen

In a glovebox atmosphere, an oven dried 1-dram vial was charged with Ni(cod)$_2$ (0.002-0.010 mmol), benzaldehyde **1a** (10.6 mg, 0.100 mmol), 1-phenyl-1-propyne (11.6 mg, 0.100 mmol), PBu$_3$ (0.004-0.020 mmol), triphenylborane (0.100-0.200 mmol), and THF (0.1-0.5 M). The resulting solution was stirred at the specified temperature in a block heater for 18 h. The reaction was cooled to room temperature, diluted with DCM, and concentrated. The residue was dissolved in 70:30 hexane/EtOAc and filtered through a short plug of silica gel, eluting with additional 70:30 hexane/EtOAc. The yield of the reaction was determined by ^{1}H NMR with dibromomethane as an internal standard.

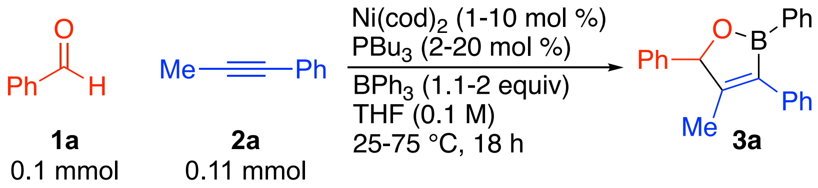
Design of Experiment (DoE) Response Surface Optimization for Dearylative Cyclocondensation

-Response: Percent Yield

-Factors: Catalyst loading, BPh_3 loading, temperature

-JMP Pro 16® software was used to generate and analyze the 15 reactions of the Box-Behnken Design.

Table S3. Design of Experiment Response Surface Optimization for Dearylative Cyclocondensation[a]



entry	catalyst loading (mol %)	BPh_3 loading (equiv)	temperature (°C)	yield **3a** (%)[b]
1	1	2	50	99
2	1	1.1	50	58
3	5	2	25	99
4	1	1.5	25	33
5	1	1.5	75	99
6	10	1.5	75	31
7	5	1.1	25	82
8	10	2	50	99
9	5	1.5	50	98
10	10	1.1	50	76
11	10	1.5	25	80
12	5	1.5	50	93
13	5	1.1	75	55
14	5	2	75	77
15	5	1.5	50	89

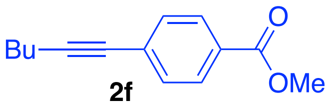
[a] Reaction conditions: **1a** (0.100 mmol), **2** (0.110 mmol), BPh_3 (0.110-0.200 mmol), $Ni(cod)_2$ (0.001-0.010 mmol), PBu_3 (0.002-0.020 mmol), THF (1.0 mL, 0.1 M), at 25-75 °C for 18 h.

[b] Determined by ^1H NMR with dibromomethane as an internal standard.

Procedure for Design of Experiment Response Surface Optimization

In a glovebox atmosphere, an oven dried 1-dram vial was charged with Ni(cod)$_2$ (0.001-0.010 mmol), benzaldehyde **1a** (10.6 mg, 0.100 mmol), 1-phenyl-1-propyne (12.8 mg, 0.110 mmol), PBu$_3$ (0.002-0.020 mmol), triphenylborane (0.110-0.200 mmol), and THF (1.0 mL, 0.1 M). The resulting solution was stirred at the specified temperature in a block heater for 18 h. The reaction was cooled to room temperature, diluted with DCM, and concentrated. The residue was dissolved in 70:30 hexane/EtOAc and filtered through a short plug of silica gel, eluting with additional 70:30 hexane/EtOAc. The yield of the reaction was determined by ^{1}H NMR with dibromomethane as an internal standard.

Experimental Procedure and Characterization Data for Alkynes 2f, 2k, and 2n:

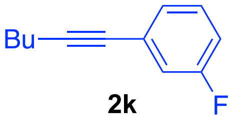


2f

methyl 4-(hex-1-yn-1-yl)benzoate (2f): Prepared according to a modified literature procedure.[4] To an oven dried 50 mL round bottom with stir bar was added methyl 4-iodobenzoate (786.1 mg, 3.0 mmol, 1.00 equiv), 1-hexyne (271.1 mg, 3.3 mmol), Pd(PPh$_3$)$_2$Cl$_2$ (105.3 mg, 0.15 mmol), CuI (57.1 mg, 0.3 mmol), triethylamine (6.0 mL, 0.5 M). The resulting solution was sealed and stirred 60 °C in an oil bath in 18 h. The crude reaction mixture was quenched with aqueous NH$_4$Cl and extracted with Et$_2$O x3. The combined organic layers were washed with brine, dried with MgSO$_4$, and concentrated under reduced pressure. The crude product was purified by column chromatography (95:5 hexanes:EtOAc) to give **2f** as a dark oil in 73% yield (471.5 mg, 2.2 mmol).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.94 (d, J = 7.6 Hz, 2H), 7.43 (d, J = 7.6 Hz, 2H), 3.90 (s, 3H), 2.42 (t, J = 7.4 Hz, 2H), 1.63 – 1.55 (m, 2H), 1.53 – 1.43 (m, 2H), 0.95 (t, J = 7.3 Hz, 3H).

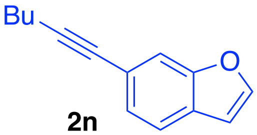
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.7, 131.5, 129.4, 129.0, 128.8, 94.0, 80.1, 52.1, 30.7, 22.0, 19.2, 13.6. **HRMS** (ESI) m/z: ($[M+H]^+$) Calc. for $C_{14}H_{17}O_2^+$ 217.1223; Found: 217.1223.



2k

1-fluoro-3-(hex-1-yn-1-yl)benzene (2k): Prepared according to a modified literature procedure.[4] To an oven dried 50 mL round bottom with stir bar was added 1-fluoro-3-iodobenzene (599.4 mg, 2.7 mmol, 1.00 equiv), 1-hexyne (239.0 mg, 2.97 mmol), $Pd(PPh_3)_2Cl_2$ (94.8 mg, 0.135 mmol), CuI (51.4 mg, 0.27 mmol), triethylamine (5.4 mL, 0.5 M). The resulting solution was sealed and stirred 60 °C in an oil bath in 18 h. The crude reaction mixture was quenched with aqueous NH_4Cl and extracted with Et_2O x3. The combined organic layers were washed with brine, dried with $MgSO_4$, and concentrated under reduced pressure. The crude product was purified by column chromatography (100% hexanes) to give **2k** as a pale yellow oil in 62% yield (322.4 mg, 1.7 mmol).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.23 (m, 1H), 7.22 – 7.18 (m, 1H), 7.12 (dd, *J* = 9.6, 2.5, 1.3 Hz, 1H), 7.03 – 6.96 (m, 1H), 2.44 (t, *J* = 7.0 Hz, 2H), 1.68 – 1.58 (m, 2H), 1.57 – 1.47 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H) **^{13}C NMR** (151 MHz, $CDCl_3$) δ 162.4 (d, *J* = 245.9 Hz), 129.7 (d, *J* = 8.7 Hz), 127.4 (d, *J* = 3.0 Hz), 126.0 (d, *J* = 9.5 Hz), 118.4 (d, *J* = 22.5 Hz), 114.8 (d, *J* = 21.2 Hz), 91.6, 79.5 (d, *J* = 3.4 Hz), 30.7, 22.0, 19.1, 13.6. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -113.5. **HRMS** (EI) m/z: (M^+) Calc. for $C_{12}H_{13}F$ 176.1001; Found: 176.0995.

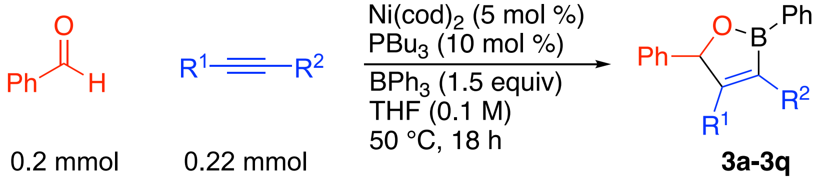


2n

6-(hex-1-yn-1-yl)benzofuran (2n): Prepared according to a modified literature procedure.[4] To an oven dried 50 mL round bottom with stir bar was added 5-bromobenzofuran (591.1 mg, 3.0 mmol, 1.00 equiv), 1-hexyne (271.1 mg, 3.3 mmol), $Pd(PPh_3)_2Cl_2$ (105.3 mg, 0.15 mmol), CuI (57.1 mg, 0.3 mmol), triethylamine (6.0 mL, 0.5 M). The resulting solution was sealed and stirred 60 °C in an oil bath in 18 h. The crude reaction mixture was quenched with aqueous NH_4Cl and extracted with Et_2O x3. The combined organic layers were washed with brine, dried with $MgSO_4$, and concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography (100% hexanes) to give **2n** as a yellow oil in 35% yield (206.9 mg, 1.0 mmol).

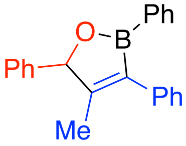
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 1.0 Hz, 1H), 7.61 (d, *J* = 2.2 Hz, 1H), 7.42 (d, *J* = 8.5 Hz, 1H), 7.35 (dd, *J* = 8.5, 1.6 Hz, 1H), 6.72 (dd, *J* = 2.2, 1.0 Hz, 1H), 2.43 (t, *J* = 7.0 Hz, 2H), 1.66 – 1.57 (m, 2H), 1.56 – 1.46 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 154.2, 145.6, 128.0, 127.5, 124.5, 118.7, 111.3, 106.4, 88.8, 80.7, 31.0, 22.1, 19.1, 13.7. **HRMS** (ESI) m/z: ([M+H]$^+$) Calc. for $C_{14}H_{15}O^+$ 199.1117; Found: 199.1116.

General Procedure A: Synthesis of Oxaborane Products 3a-3q:



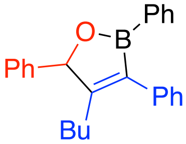
In a glovebox atmosphere, an oven dried 1-dram vial was charged with $Ni(cod)_2$ (2.8 mg, 0.010 mmol), benzaldehyde (21.2 mg, 0.200 mmol), alkyne (0.220 mmol), PBu_3 (5.0 µL, 0.020 mmol), triphenylborane (72.6 mg, 0.300 mmol), and THF (2.0 mL, 0.1 M). The resulting solution was stirred at 50 °C in a block heater for 18 h. The reaction was cooled to room temperature, diluted with DCM, and concentrated. The residue was dissolved in 70:30 hexane/EtOAc and filtered through a short plug of silica gel, eluting with additional 70:30 hexane/EtOAc. The crude product was purified by flash column chromatography to give oxaborane products **3a-3q**.

Characterization Data for Oxaboranes Products 3a-3q:



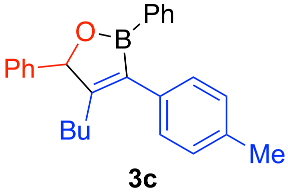
3a

4-methyl-2,3,5-triphenyl-2,5-dihydro-1,2-oxaborole (3a): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 1-phenyl-1-propyne **2a** (25.6 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3a** as an off-white solid in 91% yield (62.2 mg, 0.18 mmol) and 11.1:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (dd, *J* = 6.5, 1.5 Hz, 2H), 7.46 – 7.27 (m, 11H), 7.26 – 7.23 (m, 2H), 5.87 (s, 1H), 1.78 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.62, 138.71, 138.15, 135.48, 128.74, 128.70, 128.43, 128.26, 127.70, 126.91, 126.22, 91.76, 13.26 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 45.7. **HRMS** (EI) m/z: (M^+) Calc. for $C_{22}H_{19}BO$ 310.1529; Found: 310.1529.



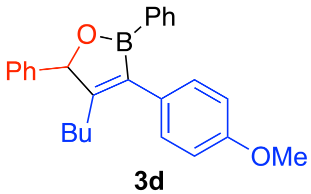
3b

4-butyl-2,3,5-triphenyl-2,5-dihydro-1,2-oxaborole (3b): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 1-phenyl-1-hexyne **2b** (34.8 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3b** as a yellow oil in 81% yield (57.4 mg, 0.16 mmol) and 12.0:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (dd, *J* = 8.1, 1.4 Hz, 2H), 7.45 – 7.27 (m, 11H), 7.24 – 7.20 (m, 2H), 5.99 (s, 1H), 2.36 (ddd, *J* = 13.8, 9.6, 6.6 Hz, 1H), 1.87 (ddd, *J* = 13.8, 9.6, 5.3 Hz, 1H), 1.47 – 1.36 (m, 1H), 1.36 – 1.10 (m, 3H), 0.76 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.1, 139.0, 138.2, 135.5, 131.3, 128.8, 128.6, 128.5, 128.3, 127.7, 127.0, 126.1, 90.2, 31.2, 27.2, 22.7, 13.8 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 45.3. **HRMS** (EI) m/z: (M^+) Calc. for $C_{25}H_{25}BO$ 352.1998; Found: 352.2002.



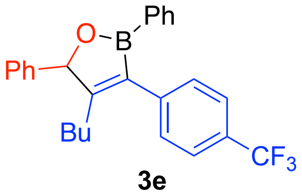
3c

4-butyl-3-(4-methylphenyl)-2,5-diphenyl-2,5-dihydro-1,2-oxaborole (3c): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 1-(hex-1-yn-1-yl)-4-methylbenzene **2c** (37.9 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3c** as a yellow oil in 83% yield (60.5 mg, 0.17 mmol) and 10.6:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (dd, *J* = 7.9, 1.2 Hz, 2H), 7.44 – 7.27 (m, 8H), 7.20 (d, *J* = 7.9, 2H), 7.11 (d, *J* = 7.9 Hz, 2H), 5.97 (s, 1H), 2.41 (s, 3H), 2.40 – 2.33 (m, 1H), 1.85 (ddd, *J* = 13.9, 9.6, 5.3 Hz, 1H), 1.47 – 1.36 (m, 1H), 1.35 – 1.10 (m, 3H), 0.77 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.0, 158.0, 136.0, 135.7, 135.7, 131.4, 129.1, 128.9, 128.6, 128.6, 127.8, 127.2, 90.3, 31.4, 27.3, 22.8, 21.4, 14.0 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.7. **HRMS** (EI) m/z: (M^+) Calc. for $C_{26}H_{27}BO_2$ 366.2155; Found: 366.2156.



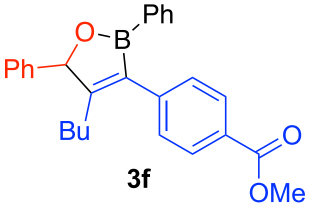
3d

4-butyl-3-(4-methoxyphenyl)-2,5-diphenyl-2,5-dihydro-1,2-oxaborole (3d): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 1-(hex-1-yn-1-yl)-4-methoxybenzene **2d** (41.4 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3d** as a yellow oil in 94% yield (69.3 mg, 0.19 mmol) and 9.4:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (d, J = 8.0 Hz, 2H), 7.48 – 7.28 (m, 8H), 7.17 (dd, J = 8.7, 1.2 Hz, 2H), 6.97 (dd, J = 8.7, 1.2 Hz, 2H), 6.00 (s, 1H), 3.90 (s, 3H), 2.41 (ddd, J = 14.6, 9.6, 6.7 Hz, 1H), 1.90 (ddd, J = 14.6, 9.6, 5.3 Hz, 1H), 1.51 – 1.14 (m, 4H), 0.80 (t, J = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.8, 158.0, 138.3, 135.5, 131.2, 131.1, 129.6, 128.7, 128.4, 127.7, 127.0, 113.7, 90.2, 55.3, 31.2, 27.2, 22.7, 13.8 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 47.0. **HRMS** (EI) m/z: (M^+) Calc. for $C_{26}H_{27}BO$ 382.2104; Found: 382.2104.



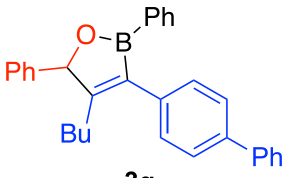
3e

4-butyl-2,5-diphenyl-3-(4-(trifluoromethyl)phenyl)-2,5-dihydro-1,2-oxaborole (3e): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 1-(hex-1-yn-1-yl)-4-(trifluoromethyl)benzene **2e** (49.8 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3e** as a clear oil in 73% yield (61.3 mg, 0.15 mmol) and 10.2:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70 (dd, *J* = 6.6, 1.4 Hz, 2H), 7.66 (dd, *J* = 8.6, 0.7 Hz, 2H), 7.47 – 7.29 (m, 10H), 6.01 (s, 1H), 2.30 (ddd, *J* = 13.8, 9.6, 6.7 Hz, 1H), 1.90 (ddd, *J* = 13.8, 9.3, 5.2 Hz, 1H), 1.47 – 1.34 (m, 1H), 1.34 – 1.09 (m, 3H), 0.79 – 0.73 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.4, 143.1 (q, *J* = 1.6 Hz), 137.7, 135.4, 131.5, 128.8, 128.6, 128.3, 127.8, 126.9, 127.1 (q, *J* = 272.8 Hz), 125.3 (q, *J* = 3.7 Hz), 90.3, 31.1, 27.2, 22.6, 13.7 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.2 (3F). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.9. **HRMS** (EI) m/z: (M^+) Calc. for $C_{26}H_{24}BF_3O$ 420.1872; Found: 420.1877.



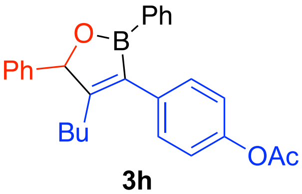
3f

1-(4-(4-butyl-2,5-diphenyl-2,5-dihydro-1,2-oxaborol-3-yl)phenyl)ethan-1-one (3f): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), methyl 4-(hex-1-yn-1-yl)benzoate **2f** (47.6 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3f** as a yellow oil in 80% yield (65.7 mg, 0.16 mmol) and 10.8:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.08 (dd, *J* = 8.4, 1.8 Hz, 2H), 7.73 – 7.68 (m, 2H), 7.45 – 7.27 (m, 10H), 6.00 (s, 1H), 3.95 (s, 3H), 2.31 (ddd, *J* = 13.7, 9.6, 6.5 Hz, 1H), 1.94 – 1.85 (m, 1H), 1.45 – 1.07 (m, 4H), 0.75 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 172.0, 167.3, 144.5, 137.8, 135.4, 131.5, 129.7, 128.8, 128.6, 128.1, 127.8, 127.0, 90.4, 52.1, 31.1, 27.2, 22.6, 13.7 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, CDCl$_3$) δ 46.0. **HRMS** (EI) m/z: (M$^+$) Calc. for $C_{27}H_{27}BO_3$ 410.2053; Found: 410.2059.



3g

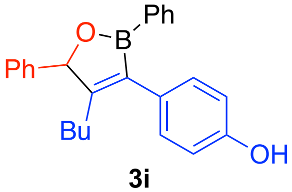
3-([1,1'-biphenyl]-4-yl)-4-butyl-2,5-diphenyl-2,5-dihydro-1,2-oxaborole (3g): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 4-(hex-1-yn-1-yl)-1,1'-biphenyl **2g** (51.2 mg, 0.220 mmol), and BPh$_3$ (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3g** as a yellow oil in 98% yield (84.0 mg, 0.20 mmol) and 11.3:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, CDCl$_3$) δ 7.81 (dd, J = 8.1, 1.4 Hz, 2H), 7.72 – 7.68 (m, 2H), 7.65 (ddd, J = 8.4, 2.0, 1.9 Hz, 2H), 7.50 – 7.45 (m, 2H), 7.45 – 7.28 (m, 11H), 6.01 (s, 1H), 2.43 (ddd, J = 13.8, 9.8, 6.7 Hz, 1H), 1.91 (ddd, J = 13.8, 9.5, 5.3 Hz, 1H), 1.51 – 1.13 (m, 4H), 0.78 (t, J = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, CDCl$_3$) δ 171.3, 140.9, 138.8, 138.2, 138.1, 135.5, 131.3, 129.0, 128.8, 128.8, 128.5, 127.7, 127.2, 127.0, 127.0, 126.9, 90.3, 31.2, 27.3, 22.7, 13.8 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, CDCl$_3$) δ 46.0. **HRMS** (EI) m/z: (M$^+$) Calc. for $C_{31}H_{29}BO$ 428.2311; Found: 428.2328.



3h

4-(4-butyl-2,5-diphenyl-2,5-dihydro-1,2-oxaborol-3-yl)phenyl acetate (3h): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 4-(hex-1-yn-1-yl)phenyl acetate **2h** (47.6 mg, 0.220 mmol), and BPh$_3$ (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h.

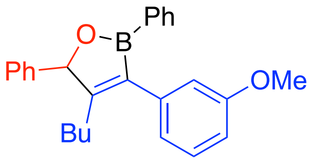
The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3h** as a yellow oil in 91% yield (74.6 mg, 0.18 mmol) and 8.7:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (dd, *J* = 8.1, 1.4 Hz, 2H), 7.46 – 7.34 (m, 4H), 7.34 – 7.28 (m, 4H), 7.23 (ddd, *J* = 9.0, 2.5, 2.2 Hz, 2H), 7.14 (ddd, *J* = 9.0, 2.5, 2.2 Hz, 2H), 5.98 (s, 1H), 2.42 – 2.34 (m, 1H), 2.33 (s, 3H), 1.87 (ddd, *J* = 13.8, 9.5, 5.3 Hz, 1H), 1.48 – 1.10 (m, 4H), 0.77 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.6, 169.5, 149.1, 138.0, 136.5, 131.3, 129.5, 128.8, 128.5, 127.7, 127.0, 121.3, 90.2, 31.2, 27.2, 22.6, 21.3, 13.7 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 45.9. **HRMS** (EI) m/z: (M^+) Calc. for $C_{27}H_{27}BO_3$ 410.2053; Found: 410.2054.



3i

4-(4-butyl-2,5-diphenyl-2,5-dihydro-1,2-oxaborol-3-yl)phenol (3i): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 4-(hex-1-yn-1-yl)phenol **2i** (52.3 mg, 0.300 mmol), and BPh_3 (96.8 mg, 0.400 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3i** as a red oil in 85% yield (62.5 mg, 0.17 mmol) and 9.1:1 r.r. The regioisomers were not separable by Reverse-Phase Prep HPLC due to degradation. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.31 (d, *J* = 6.7 Hz, 2H, minor regioisomer integral value = 0.3), 8.12 (d, *J* = 6.7 Hz, 2H, minor regioisomer integral value = 0.3), 7.83 (d, *J* = 6.9 Hz, 2H, major regioisomer integral value = 2.1), 7.65 (t, *J* = 7.4 Hz, 1H, minor regioisomer integral value = 0.2), 7.56 (dd, *J* = 7.4, 7.0 Hz, 2H, minor regioisomer integral value = 0.7), 7.49 – 7.34 (m, major + minor regioisomer value =

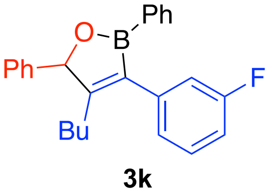
9.6), 7.26 (dd, *J* = 7.3, 5.7 Hz, 2H, minor regioisomer integral value = 0.6), 7.14 (d, *J* = 8.3 Hz, 2H, major regioisomer integral value = 2.3), 6.98 (d, *J* = 8.4 Hz, 2H, minor regioisomer integral value = 0.3), 6.91 (d, *J* = 8.3 Hz, 2H, major regioisomer integral value = 2.3), 6.77 (d, *J* = 8.4 Hz, 2H, minor regioisomer integral value = 0.3), 6.19 (s, 1H, minor regioisomer integral value = 0.1), 6.02 (s, 1H, major regioisomer integral value = 1.0), 5.24 (bs, 1H, major + minor regioisomer integral value = 1.3), 2.64 – 2.57 (m, 1H, minor regioisomer integral value = 0.2), 2.43 (ddd, *J* = 13.9, 9.6, 6.6 Hz, 1H, major regioisomer integral value = 1.1), 1.92 (ddd, *J* = 13.9, 9.6, 5.3 Hz, 1H, major regioisomer integral value = 1.2), 1.50 – 1.17 (m, major + minor regioisomer integral value = 6.6), 0.92 (t, *J* = 7.3 Hz, 3H, minor regioisomer integral value = 0.7), 0.82 (t, *J* = 7.2 Hz, 3H, major regioisomer integral value = 3.5). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.9, 154.0, 138.2, 135.5, 131.3, 129.8, 128.8, 128.5, 128.0, 127.7, 127.0, 115.3, 90.3, 31.2, 27.2, 22.7, 13.8 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). Peaks observed for minor regioisomer: 171.9, 166.5, 155.2, 135.7, 134.9, 132.8, 131.2, 129.8, 128.4, 128.0, 115.3, 90.9, 32.9, 27.5, 23.0, 14.0 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 45.9. **HRMS** (EI) m/z: (M^+) Calc. for $C_{25}H_{25}BO_2$ 368.1948; Found: 368.1950.



3j

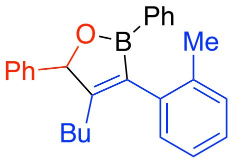
4-butyl-3-(3-methoxyphenyl)-2,5-diphenyl-2,5-dihydro-1,2-oxaborole (3j): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 1-(hex-1-yn-1-yl)-3-methoxybenzene **2j** (41.4 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100%

hexanes followed by 97:3 hexanes:EtOAc) to give **3j** as a yellow oil in 89% yield (68.3 mg, 0.18 mmol) and 11.3:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (dd, *J* = 8.1, 1.4 Hz, 2H), 7.47 – 7.25 (m, 9H), 6.86 (ddd, *J* = 8.3, 2.6, 1.0 Hz, 1H), 6.81 (dd, *J* = 7.5, 1.3, Hz, 1H), 6.76 (dd, *J* = 2.6, 1.3 Hz, 1H), 5.98 (s, 1H), 3.81 (s, 3H), 2.37 (ddd, *J* = 13.8, 9.6, 6.6 Hz, 1H), 1.87 (ddd, *J* = 13.8, 9.6, 5.3 Hz, 1H), 1.49 – 1.35 (m, 1H), 1.38 – 1.09 (m, 3H), 0.77 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.3, 159.6, 140.7, 138.3, 135.7, 131.5, 129.4, 128.9, 128.6, 127.9, 127.2, 121.3, 114.1, 111.9, 90.3, 55.4, 31.4, 27.4, 22.8, 14.0 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.0. **HRMS** (EI) m/z: (M^+) Calc. for $C_{26}H_{27}BO_2$ 382.2104; Found: 382.2115.



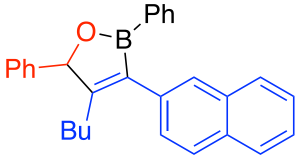
3k

4-butyl-3-(3-fluorophenyl)-2,5-diphenyl-2,5-dihydro-1,2-oxaborole (3k): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 1-(hex-1-yn-1-yl)-3-fluorobenzene **2k** (38.8 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3k** as a clear oil in 72% yield (53.7 mg, 0.14 mmol) and 11.6:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.7 Hz, 2H), 7.48 – 7.26 (m, 9H), 7.01 (dd, *J* = 9.6, 7.7 Hz, 2H), 6.94 (d, *J* = 9.3 Hz, 1H), 5.99 (s, 1H), 2.34 (ddd, *J* = 15.3, 9.6, 7.1 Hz, 1H), 1.89 (ddd, *J* = 14.5, 9.6, 5.4 Hz, 1H), 1.47 – 1.35 (m, 1H), 1.35 – 1.10 (m, 3H), 0.77 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.9, 162.8 (d, *J* = 244.8 Hz), 141.5 (d, *J* = 8.0 Hz), 137.9, 135.4, 131.4, 129.8 (d, *J* = 10.1 Hz), 128.8, 128.6, 127.8, 127.0, 124.3, 115.3 (d, *J* = 21.7 Hz), 113.1 (d, *J* = 21.0), 90.3, 31.1, 27.2, 22.6, 13.7 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{19}F NMR** (376 MHz, CDCl$_3$) δ -113.6 (1F). **^{11}B NMR** (193 MHz, CDCl$_3$) δ 44.8. **HRMS** (EI) m/z: (M$^+$) Calc. for $C_{25}H_{24}BFO$ 370.1904; Found: 370.1913.



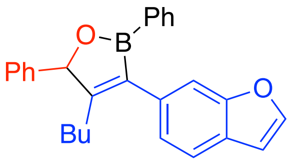
3l

4-butyl-2,5-diphenyl-3-(*o*-tolyl)-2,5-dihydro-1,2-oxaborole (3l): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 1-(hex-1-yn-1-yl)-2-methylbenzene **2l** (37.9 mg, 0.220 mmol), and BPh$_3$ (72.6 mg, 0.300 mmol) at 70 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3l** as a yellow oil in 80% yield (58.6 mg, 0.16 mmol) and >20:1 r.r. as a 1.1:1 ratio of diastereomers. Attempts to isolate the two diastereomers to determine the major product were not successful. The presence of atropisomers was confirmed by protodeboronation of **3l** to generate the corresponding allylic alcohol. **^{1}H NMR** (400 MHz, CDCl$_3$) δ 7.76 (d, *J* = 7.9 Hz, major + minor isomer integral value = 2), 7.49 – 7.38 (m, major + minor isomer integral value = 6), 7.36 – 7.27 (m, major + minor isomer integral value = 5), 7.20 – 7.11 (m, major + minor isomer integral value = 1), 6.10 (s, 1H, minor isomer integral value = 0.4), 6.08 (s, 1H, major isomer integral value = 0.6), 2.33 (ddd, *J* = 14.0, 9.5, 6.5 Hz, minor isomer integral value = 0.5), 2.24 (s, major isomer integral value = 1.6), 2.23 (s, minor isomer integral value = 1.2), 2.21 – 2.13 (m, major isomer integral value = 0.7), 1.96 – 1.85 (m, major + minor integral value = 1), 1.49 – 1.11 (m, major + minor integral value = 4), 0.83 (t, *J* = 7.3 Hz, 3H, major isomer integral value = 2), 0.79 (t, *J* = 7.2 Hz, 3H, minor isomer integral value = 1.3). **^{13}C NMR** (101 MHz, CDCl$_3$) Peaks observed for major diastereomer: δ 171.1, 138.7, 138.3, 135.2, 135.2, 131.5, 129.9, 128.8, 128.5, 128.5, 127.9, 127.0, 126.5, 125.9, 90.5, 31.0, 27.5, 22.7, 20.7, 13.8 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). Peaks observed for minor diastereomer: δ 170.97, 138.81, 135.50, 135.25, 129.96, 128.81, 128.49, 127.09, 126.52, 90.39, 30.54, 27.35, 22.66, 20.69, 13.72 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 45.9. **HRMS** (EI) m/z: (M^+) Calc. for $C_{26}H_{27}BO$ 366.2155; Found: 366.2159.



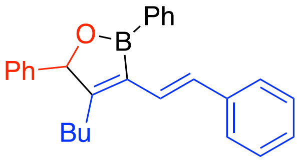
3m

4-butyl-3-(naphthalen-2-yl)-2,5-diphenyl-2,5-dihydro-1,2-oxaborole (3m): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 2-(hex-1-yn-1-yl)naphthalene **2m** (45.8 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3m** as a yellow oil in 99% yield (79.3 mg, 0.20 mmol) and 11.2:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.92 – 7.87 (m, 2H), 7.86 – 7.81 (m, 1H), 7.78 – 7.74 (m, 2H), 7.70 – 7.67 (m, 1H), 7.54 – 7.47 (m, 2H), 7.45 – 7.34 (m, 7H), 7.29 – 7.23 (m, 2H), 6.04 (s, 1H), 2.41 (ddd, J = 13.7, 9.6, 6.7 Hz, 1H), 1.92 (ddd, J = 13.7, 9.6, 5.4 Hz, 1H), 1.49 – 1.28 (m, 2H), 1.28 – 1.09 (m, 2H), 0.75 (t, J = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.6, 138.2, 136.7, 135.6, 133.6, 132.1, 131.3, 128.8, 128.5, 127.8, 127.8, 127.8, 127.7, 127.6, 127.0, 126.6, 126.0, 125.5, 90.3, 31.2, 27.2, 22.6, 13.7 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, CDCl$_3$) δ 45.4. **HRMS** (EI) m/z: (M$^+$) Calc. for $C_{29}H_{27}BO$ 402.2155; Found: 402.2165.



3n

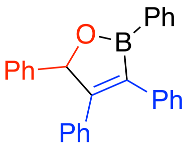
3-(benzofuran-6-yl)-4-butyl-2,5-diphenyl-2,5-dihydro-1,2-oxaborole (3n): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 6-(hex-1-yn-1-yl)benzofuran **2n** (43.6 mg, 0.220 mmol), and BPh$_3$ (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3n** as a yellow oil in 90% yield (70.4 mg, 0.18 mmol) and 9.0:1 r.r. An analytically pure sample of the major regioisomer was obtained by Reverse-Phase Prep HPLC (85:15 to 95:5 acetonitrile:water). **^{1}H NMR** (400 MHz, CDCl$_3$) δ 7.75 (dd, *J* = 8.1, 1.4 Hz, 2H), 7.66 (d, *J* = 2.2 Hz, 1H), 7.54 (dt, *J* = 8.4, 0.8 Hz, 1H), 7.44 – 7.38 (m, 4H), 7.38 – 7.33 (m, 3H), 7.30 – 7.25 (m, 2H), 7.16 (dd, *J* = 8.4, 1.7 Hz, 1H), 6.78 (dd, *J* = 2.2, 1.0 Hz, 1H), 6.02 (s, 1H), 2.39 (ddd, *J* = 13.8, 9.6, 6.7 Hz, 1H), 1.89 (ddd, *J* = 13.8, 9.6, 5.2 Hz, 1H), 1.48 – 1.09 (m, 4H), 0.75 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, CDCl$_3$) δ 171.1, 153.8, 145.1, 138.3, 135.5, 133.6, 131.3, 128.7, 128.5, 127.7, 127.6, 127.0, 125.2, 120.6, 111.1, 106.7, 90.2, 31.2, 27.2, 22.6, 13.7 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, CDCl$_3$) δ 46.0. **HRMS** (EI) m/z: (M$^+$) Calc. for $C_{27}H_{25}BO_2$ 392.1948; Found: 392.1957.



3o

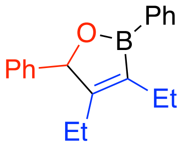
(*E*)-4-butyl-2,5-diphenyl-3-styryl-2,5-dihydro-1,2-oxaborole (3o): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), (*E*)-oct-1-en-3-yn-1-ylbenzene **2o** (40.5 mg, 0.220

mmol), BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3o** as a yellow oil in 40% yield (30.1 mg, 0.08 mmol) and 3.3:1 r.r. The regioisomers were not separable by Reverse-Phase Prep HPLC due to degradation. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.06 – 8.03 (m, major + minor regioisomer integral value = 2.7), 7.54 – 7.25 (m, major + minor regioisomer integral value = 22.3), 7.18 (d, *J* = 16.3 Hz, 1H, major regioisomer)), 7.16 (d, *J* = 16.6 Hz, major + minor regioisomer integral value = 2.1), 6.90 (d, *J* = 16.3 Hz, 1H, major regioisomer integral value = 1.1), 6.50 (d, *J* = 16.6 Hz, 1H, minor regioisomer integral value = 0.3), 6.18 (s, 1H, minor regioisomer integral value = 0.3), 5.94 (s, 1H, major regioisomer integral value = 1.0), 2.82 (t, *J* = 7.8 Hz, 1H, minor regioisomer integral value = 0.3), 2.69 – 2.58 (m, 1H, major regioisomer integral value = 1.2), 2.08 (ddd, *J* = 14.0, 9.0, 5.5 Hz, 1H, major regioisomer integral value = 1.1), 1.71 – 1.62 (m, 1H, minor regioisomer integral value = 0.9), 1.57 – 1.46 (m, major + minor regioisomer integral value = 2.5), 1.43 – 1.28 (m, major + minor regioisomer integral value = 4.4), 1.02 (t, *J* = 7.3 Hz, 3H, minor regioisomer integral value = 1.2), 0.90 (t, *J* = 7.2 Hz, 3H, major regioisomer integral value = 3.5). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.0, 138.1, 138.0, 134.7, 132.9, 130.9, 128.7, 128.7, 128.5, 127.8, 127.4, 127.1, 126.3, 124.4, 90.6, 31.2, 27.3, 22.8, 13.9 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). Peaks observed for minor regioisomer: 160.8, 139.7, 137.0, 134.9, 134.0, 131.2, 128.8, 128.3, 127.9, 127.7, 126.7, 119.6, 88.9, 33.1, 27.3, 23.0, 14.1 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 47.1. **HRMS** (EI) m/z: (M^+) Calc. for $C_{27}H_{27}BO$ 378.2155; Found: 378.2159.



3p

2,3,4,5-tetraphenyl-2,5-dihydro-1,2-oxaborole (3p): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), diphenylacetylene (39.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3p** as a yellow solid in 84% yield (62.6 mg, 0.17 mmol). ^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 8.0, 1.3 Hz, 2H), 7.49 (tt, *J* = 7.0, 1.3 Hz, 1H), 7.40 – 7.25 (m, 13H), 7.15 (m, 2H), 7.04 (m, 2H), 6.49 (s, 1H). ^{13}**C NMR** (101 MHz, $CDCl_3$) δ 166.3, 138.7, 138.0, 135.5, 134.4, 131.5, 128.9, 128.8, 128.6, 128.4, 128.4, 128.1, 128.0, 127.8, 127.4, 126.4, 90.9 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). ^{11}**B NMR** (193 MHz, $CDCl_3$) δ 46.0. **HRMS** (EI) m/z: (M^+) Calc. for $C_{27}H_{21}BO$ 372.1685; Found: 372.1693.

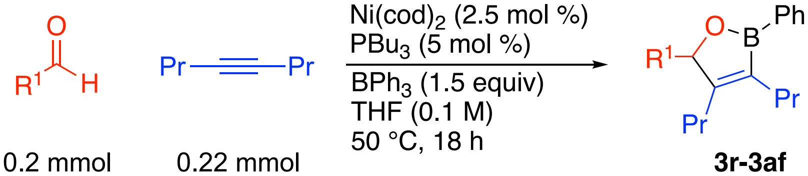


3q

3,4-diethyl-2,5-diphenyl-2,5-dihydro-1,2-oxaborole (3q): Prepared according to general procedure **A** from benzaldehyde (21.2 mg, 0.200 mmol), 3-hexyne (18.0 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3q** as a yellow oil in 80% yield (43.9 mg, 0.16 mmol). ^{1}**H NMR** (400 MHz, $CDCl_3$) δ 8.05 (dd, *J* = 7.9, 1.4 Hz, 2H), 7.54 – 7.49 (m, 1H), 7.50 – 7.41 (m, 2H), 7.40 – 7.29 (m, 3H), 7.28 – 7.23 (m, 2H), 5.83 (s, 1H), 2.57 (q, *J* = 7.6 Hz, 2H), 2.44 (dq, *J* = 15.2, 7.6 Hz, 1H), 1.95 (dq, *J* = 15.2, 7.6 Hz, 1H), 1.18 (t, *J* = 7.6 Hz, 3H), 0.99 (t, *J* = 7.6 Hz, 3H). ^{13}**C NMR** (101 MHz, $CDCl_3$) δ 170.21, 138.63, 134.85, 131.15, 128.60, 128.25, 127.94, 126.97, 89.76, 20.29, 19.70, 15.48, 13.90 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.7.

HRMS (EI) m/z: (M^+) Calc. for $C_{19}H_{21}BO$ 276.1685; Found: 276.1688.

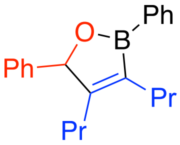
General Procedure B: Synthesis of Oxaborane Products 3r-3af:



3r-3af

In a glovebox atmosphere, an oven dried 1-dram vial was charged with $Ni(cod)_2$ (1.4 mg, 0.005 mmol), aldehyde (0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), PBu_3 (2.5 µL, 0.010 mmol), triphenylborane (72.6 mg, 0.300 mmol), and THF (2.0 mL, 0.1 M). The resulting solution was stirred at 50 °C in a block heater for 18 h. The reaction was cooled to room temperature, diluted with DCM, and concentrated. The residue was dissolved in 70:30 hexane/EtOAc and filtered through a short plug of silica gel, eluting with additional 70:30 hexane/EtOAc. The crude product was purified by flash column chromatography treated with 1% Et_3N to give oxaborane products **3r-3af**.

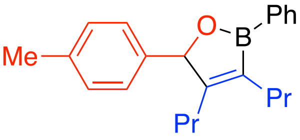
Characterization Data for Oxaborole Products 3r-3af:



3r

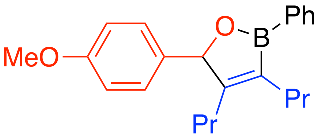
2,5-diphenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborole (3r): Prepared according to general procedure **B** from benzaldehyde (21.2 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3r** as a light yellow oil in 94% yield (57.4 mg, 0.19 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 7.5 Hz, 2H), 7.49 (t, *J* = 7.1 Hz, 1H), 7.43 (dd, *J* = 7.5, 7.1 Hz, 2H), 7.38 – 7.28 (m, 3H), 7.23 (d, *J* = 7.6 Hz, 2H), 5.80 (s, 1H), 2.57 – 2.44 (m, 2H), 2.38 (ddd, *J* = 14.3, 9.5, 7.0 Hz, 1H), 1.87 (ddd, *J*

= 14.3, 9.5, 5.6 Hz, 1H), 1.63 – 1.44 (m, 3H), 1.40 – 1.27 (m, 1H), 0.99 (t, *J* = 7.3 Hz, 3H), 0.90 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 169.3, 138.9, 134.9, 131.2, 128.7, 128.3, 128.0, 127.1, 90.1, 29.5, 29.0, 24.0, 22.5, 14.5, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.6. **HRMS** (EI) m/z: (M^+) Calc. for $C_{21}H_{25}BO$ 304.1998; Found: 304.2005.



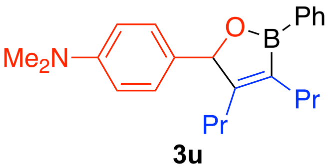
3s

2-phenyl-3,4-dipropyl-5-(*p*-tolyl)-2,5-dihydro-1,2-oxaborole (3s): Prepared according to general procedure **B** from *p*-tolualdehyde (24.0 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3s** as a light yellow oil in 89% yield (56.5 mg, 0.18 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.54 – 7.48 (m, 1H), 7.48 – 7.42 (m, 2H), 7.16 (m, 4H), 5.80 (s, 1H), 2.60 – 2.48 (m, 2H), 2.48 – 2.38 (m, 1H), 2.37 (s, 3H), 1.89 (ddd, *J* = 14.4, 9.5, 5.5 Hz, 1H), 1.67 – 1.45 (m, 3H), 1.37 (ddt, *J* = 14.4, 9.5, 7.3 Hz, 1H), 1.01 (t, *J* = 7.3 Hz, 3H), 0.93 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.3, 138.0, 135.9, 134.9, 131.1, 129.4, 128.0, 127.0, 90.0, 29.5, 29.0, 24.0, 22.5, 21.3, 14.5, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.8. **HRMS** (EI) m/z: (M^+) Calc. for $C_{22}H_{27}BO$ 318.2155; Found: 318.2155.



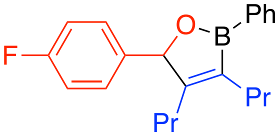
3t

5-(4-methoxyphenyl)-2-phenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborole (3t): Prepared according to general procedure **B** from 4-methoxybenzaldehyde (27.2 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3t** as a light yellow oil in 93% yield (62.5 mg, 0.19 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.53 – 7.47 (m, 1H), 7.47 – 7.41 (m, 2H), 7.19 – 7.14 (m, 2H), 6.93 – 6.84 (m, 2H), 5.78 (s, 1H), 3.82 (s, 3H), 2.59 – 2.46 (m, 2H), 2.45 – 2.35 (m, 1H), 1.89 (ddd, *J* = 13.6, 9.4, 5.5 Hz, 1H), 1.66 – 1.43 (m, 3H), 1.35 (ddd, *J* = 13.6, 9.4 7.4, Hz, 1H), 1.01 (t, *J* = 7.4 Hz, 3H), 0.92 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.3, 159.7, 134.9, 131.1, 130.9, 128.4, 128.0, 114.1, 89.8, 55.4, 29.5, 29.0, 24.0, 22.5, 14.5, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.3. **HRMS** (EI) m/z: (M^+) Calc. for $C_{22}H_{27}BO_2$ 334.2104; Found: 334.2109.



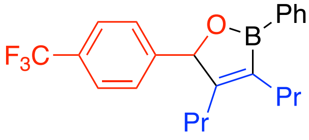
3u

***N,N*-dimethyl-4-(2-phenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborol-5-yl)aniline (3u):** Prepared according to general procedure **B** from 4-(dimethylamino)benzaldehyde (29.8 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3u** as a dark yellow oil in 69% yield (57.4 mg, 0.14 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.02 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.52 – 7.46 (m, 1H), 7.46 – 7.40 (m, 2H), 7.14 – 7.09 (m, 2H), 6.73 (d, *J* = 8.8 Hz, 2H), 5.75 (s, 1H), 2.96 (s, 6H), 2.61 – 2.45 (m, 2H), 2.47 – 2.36 (m, 1H), 1.91 (ddd, *J* = 13.6, 9.4, 5.5 Hz, 1H), 1.65 – 1.44 (m, 3H), 1.44 – 1.32 (m, 1H), 1.01 (t, *J* = 7.3 Hz, 3H), 0.92 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.4, 150.6, 134.9, 131.0, 128.1, 127.9, 112.7, 90.1, 40.8, 29.5, 29.1, 24.1, 22.5, 14.5, 14.5 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, CDCl$_3$) δ 46.7. **HRMS** (EI) m/z: (M$^+$) Calc. for $C_{23}H_{30}BNO$ 347.2420; Found: 347.2426.



3v

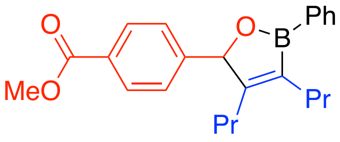
5-(4-fluorophenyl)-2-phenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborole (3v): Prepared according to general procedure **B** from 4-fluorobenzaldehyde (24.8 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh$_3$ (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et$_3$N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3v** as a yellow oil in 77% yield (49.5 mg, 0.15 mmol). **^{1}H NMR** (400 MHz, CDCl$_3$) δ 8.02 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.54 – 7.48 (m, 1H), 7.48 – 7.42 (m, 2H), 7.24 – 7.18 (m, 2H), 7.08 – 7.01 (m, 2H), 5.79 (s, 1H), 2.52 (td, *J* = 7.3, 5.5 Hz, 2H), 2.45 – 2.35 (m, 1H), 1.86 (ddd, *J* = 13.7, 9.5, 5.5 Hz, 1H), 1.65 – 1.40 (m, 3H), 1.34 (ddd, *J* = 13.7, 9.5, 7.3 Hz, 1H), 1.00 (t, *J* = 7.3 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, CDCl$_3$) δ 169.0, 162.8 (d, *J* = 246.3 Hz), 134.9, 134.7 (d, *J* = 3.2 Hz), 131.3, 128.8 (d, *J* = 8.2 Hz), 128.0, 115.6 (d, *J* = 21.6 Hz), 89.4, 29.5, 28.9, 24.0, 22.5, 14.5, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{19}F NMR** (376 MHz, CDCl$_3$) δ -114.0. **^{11}B NMR** (193 MHz, CDCl$_3$) δ 46.5. **HRMS** (EI) m/z: (M$^+$) Calc. for $C_{21}H_{24}BFO$ 322.1904; Found: 322.1907.



3w

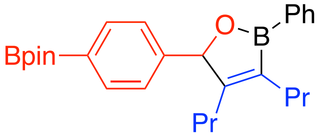
2-phenyl-3,4-dipropyl-5-(4-(trifluoromethyl)phenyl)-2,5-dihydro-1,2-oxaborole (3w): Prepared according to general procedure **B** from 4-(trifluoromethyl)benzaldehyde (34.8 mg, 0.200 mmol), 4-octyne

(24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 70 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3w** as a clear oil in 71% yield (52.9 mg, 0.14 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.02 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.62 (d, *J* = 8.0 Hz, 2H), 7.55 – 7.49 (m, 1H), 7.48 – 7.43 (m, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 5.85 (s, 1H), 2.58 – 2.45 (m, 2H), 2.41 (ddd, *J* = 14.0, 9.5, 6.4 Hz, 1H), 1.84 (ddd, *J* = 14.0, 9.4, 5.4 Hz, 1H), 1.66 – 1.46 (m, 3H), 1.41 – 1.27 (m, 1H), 0.99 (t, *J* = 7.3 Hz, 3H), 0.92 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 168.6, 143.1, 135.0, 131.5, 130.6 (q, *J* = 32.3 Hz), 128.1, 127.3, 125.7 (q, *J* = 3.8 Hz), 124.2 (q, *J* = 272.1 Hz), 89.3, 29.5, 28.8, 24.0, 22.6, 14.5, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.52 (3F). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 47.2. **HRMS** (EI) m/z: (M^+) Calc. for $C_{22}H_{24}BF_3O$ 372.1872; Found: 372.1879.



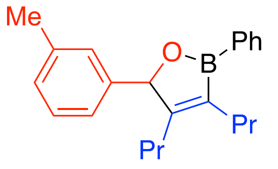
3x

methyl 4-(2-phenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborol-5-yl)benzoate (3x): Prepared according to general procedure **B** from methyl 4-formylbenzoate (32.8 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 70 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3x** as a light yellow oil in 65% yield (47.2 mg, 0.13 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 – 8.00 (m, 4H), 7.54 – 7.48 (m, 1H), 7.47 – 7.42 (m, 2H), 7.33 – 7.29 (m, 2H), 5.84 (s, 1H), 3.92 (s, 3H), 2.50 (m, 2H), 2.37 (ddd, *J* = 13.8, 9.5, 6.7 Hz, 1H), 1.84 (ddd, *J* = 13.8, 9.5, 5.4 Hz, 1H), 1.62 – 1.41 (m, 3H), 1.37 – 1.23 (m, 1H), 0.98 (t, *J* = 7.3 Hz, 3H), 0.90 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.2, 159.9, 140.5, 135.0, 131.2, 129.7, 128.0, 119.6, 113.7, 112.5, 90.0, 55.4, 29.4, 29.0, 24.0, 22.6, 14.5, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, CDCl$_3$) δ 47.6. **HRMS** (EI) m/z: (M$^+$) Calc. for $C_{23}H_{27}BO_3$ 362.2053; Found: 362.2058.



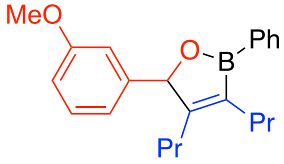
3y

4,4,5,5-tetramethyl-2-(4-(2-phenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborol-5-yl)phenyl)-1,3,2-dioxaborolane (3y): Prepared according to general procedure **B** from 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzaldehyde **1y** (46.2 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh$_3$ (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et$_3$N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3y** as a clear oil in 49% yield (41.9 mg, 0.10 mmol). **^{1}H NMR** (400 MHz, CDCl$_3$) δ 8.04 – 7.98 (m, 2H), 7.79 (d, *J* = 8.2 Hz, 2H), 7.52 – 7.46 (m, 1H), 7.43 (ddd, *J* = 7.8, 6.2, 1.4 Hz, 2H), 7.25 – 7.19 (m, 2H), 5.80 (s, 1H), 2.50 (q, *J* = 7.2 Hz, 2H), 2.36 (ddd, *J* = 13.8, 9.5, 6.9 Hz, 1H), 1.84 (ddd, *J* = 13.8, 9.5, 5.4 Hz, 1H), 1.62 – 1.43 (m, 4H), 1.34 (d, *J* = 1.9 Hz, 12H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.89 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, CDCl$_3$) δ 169.3, 142.0, 135.2, 135.0, 131.2, 128.0, 126.3, 90.1, 83.9, 29.9, 29.4, 28.9, 25.0, 24.0, 22.5, 14.5, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, CDCl$_3$) δ 47.8, 30.8. **HRMS** (EI) m/z: (M$^+$) Calc. for $C_{27}H_{36}B_2O_3$ 430.2851; Found: 430.2855.



3z

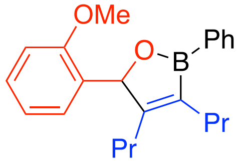
methyl 4-(2-phenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborol-5-yl)benzoate (3z): Prepared according to general procedure **B** from 3-methylbenzaldehyde (24.0 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3z** as a clear oil in 61% yield (39.1 mg, 0.12 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.01 (dd, J = 8.2, 1.9 Hz, 2H), 7.49 (m, 1H), 7.46 – 7.39 (m, 2H), 7.23 (t, J = 7.6 Hz, 1H), 7.11 (d, J = 7.1 Hz, 1H), 7.02 (m, 2H), 5.76 (s, 1H), 2.51 (m, 2H), 2.43 – 2.35 (m, 1H), 2.34 (s, 3H), 1.92 – 1.82 (m, 1H), 1.58 (m, 3H), 1.49 (m, 1H), 1.34 (m, 1H), 0.99 (t, J = 7.3 Hz, 3H), 0.90 (t, J = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.3, 138.7, 138.4, 135.0, 131.2, 129.1, 128.6, 128.0, 127.7, 124.2, 90.2, 29.5, 29.0, 24.0, 22.5, 21.6, 14.5, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.9. **HRMS** (EI) m/z: (M^+) Calc. for $C_{22}H_{27}BO$ 318.2155; Found: 318.2157.



3aa

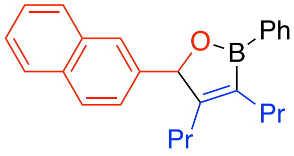
5-(3-methoxyphenyl)-2-phenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborole (3aa): Prepared according to general procedure **B** from 3-methoxybenzaldehyde (27.2 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 70 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3aa** as a light yellow oil in 73% yield (48.6 mg, 0.15 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.01 (dd, J = 8.1, 1.5 Hz, 2H), 7.52 – 7.46 (m, 1H), 7.46 – 7.40 (m, 2H), 7.29 – 7.24 (m, 1H), 6.89 – 6.81 (m, 2H), 6.78 – 6.72 (m, 1H), 5.77 (s, 1H), 3.78

(s, 3H), 2.57 – 2.43 (m, 2H), 2.39 (ddd, *J* = 13.7, 9.8, 6.8 Hz, 1H), 1.89 (ddd, *J* = 13.7, 9.8, 5.4 Hz, 1H), 1.65 – 1.42 (m, 3H), 1.34 (ddt, *J* = 14.2, 9.8, 7.3 Hz, 1H), 0.98 (t, *J* = 7.3 Hz, 3H), 0.90 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.2, 160.0, 140.5, 135.0, 131.2, 129.7, 128.0, 119.6, 113.7, 112.5, 90.0, 55.4, 29.4, 29.0, 24.0, 22.6, 14.5, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 47.0. **HRMS** (EI) m/z: (M^+) Calc. for $C_{22}H_{27}BO_2$ 334.2104; Found: 334.2107.



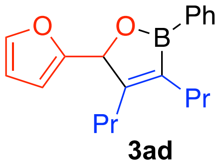
3ab

5-(2-methoxyphenyl)-2-phenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborole (3ab): Prepared according to general procedure **B** from 2-methoxybenzaldehyde (27.2 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 70 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3ab** as a light yellow oil in 83% yield (55.4 mg, 0.17 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.53 – 7.47 (m, 1H), 7.47 – 7.42 (m, 2H), 7.28 (ddd, *J* = 8.3, 7.3, 1.9 Hz, 1H), 6.99 (dd, *J* = 7.6, 1.9 Hz, 1H), 6.96 (dd, *J* = 8.3, 1.2 Hz, 1H), 6.91 (td, *J* = 7.4, 1.1 Hz, 1H), 6.47 (s, 1H), 3.91 (s, 3H), 2.58 – 2.45 (m, 2H), 2.41 (dddd, *J* = 13.4, 9.6, 6.8, 0.8 Hz, 1H), 1.89 (ddd, *J* = 13.4, 9.6, 5.4 Hz, 1H), 1.56 (ddddd, *J* = 16.3, 15.0, 9.5, 5.4, 3.5 Hz, 3H), 1.37 (ddt, *J* = 13.2, 9.5, 6.9 Hz, 1H), 1.00 (t, *J* = 7.4 Hz, 3H), 0.92 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.2, 157.3, 134.9, 131.0, 129.1, 127.9, 127.0, 126.8, 120.9, 110.9, 82.9, 55.6, 29.4, 29.0, 24.1, 22.5, 14.5 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.6. **HRMS** (EI) m/z: (M^+) Calc. for $C_{22}H_{27}BO_2$ 334.2104; Found: 334.2108.



3ac

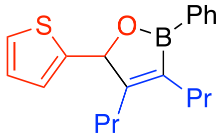
5-(2-methoxyphenyl)-2-phenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborole (3ac): Prepared according to general procedure **B** from 2-naphthaldehyde (31.2 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3ac** as a white solid in 70% yield (49.8 mg, 0.14 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.09 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.91 – 7.80 (m, 4H), 7.56 – 7.46 (m, 5H), 7.25 (dd, *J* = 8.4, 1.9 Hz, 1H), 6.00 (s, 1H), 2.65 – 2.49 (m, 2H), 2.44 (ddd, *J* = 13.7, 9.6, 7.2 Hz, 1H), 1.90 (ddd, *J* = 13.7, 9.6, 5.4 Hz, 1H), 1.71 – 1.48 (m, 3H), 1.39 (ddt, *J* = 14.1, 9.6, 7.2 Hz, 1H), 1.05 (t, *J* = 7.4 Hz, 3H), 0.92 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.2, 136.3, 135.0, 133.5, 133.5, 131.3, 128.6, 128.1, 128.0, 127.9, 127.0, 126.3, 126.2, 124.0, 90.3, 29.5, 29.0, 24.1, 22.6, 14.5, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.9. **HRMS** (EI) m/z: (M^+) Calc. for $C_{25}H_{27}BO$ 354.2155; Found: 354.2166.



3ad

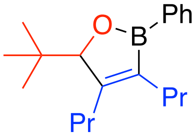
2-phenyl-3,4-dipropyl-5-(thiophen-2-yl)-2,5-dihydro-1,2-oxaborole (3ad): Prepared according to general procedure **B** from furfural (19.2 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 99:1 hexanes:EtOAc) to give **3ad** as a dark oil in 63% yield (37.1 mg, 0.13 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.99 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.51 – 7.45 (m, 1H), 7.45 – 7.38 (m, 3H), 6.37 (dd, *J* = 3.3, 1.9 Hz, 1H), 6.34

(dd, *J* = 3.3, 0.9 Hz, 1H), 5.87 (s, 1H), 2.58 – 2.42 (m, 4H), 2.05 (ddd, *J* = 13.8, 9.5, 5.5 Hz, 1H), 1.62 – 1.41 (m, 3H), 1.41 – 1.29 (m, 1H), 0.99 (t, *J* = 7.4 Hz, 3H), 0.92 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 165.8, 151.9, 142.8, 134.9, 131.2, 128.0, 110.5, 108.7, 82.9, 29.5, 29.1, 23.8, 22.3, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 47.2. **HRMS** (EI) m/z: (M^+) Calc. for $C_{19}H_{23}BO_2$ 294.1791; Found: 294.1791.



3ae

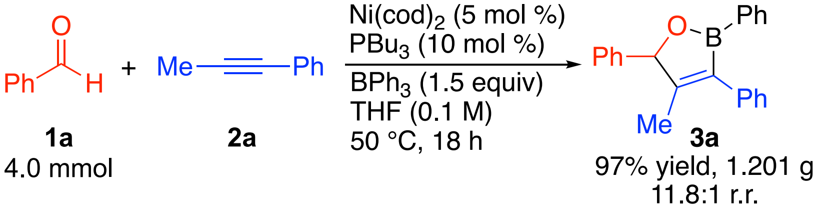
2-phenyl-3,4-dipropyl-5-(thiophen-2-yl)-2,5-dihydro-1,2-oxaborole (3ae): Prepared according to general procedure **B** from thiophene-2-carbaldehyde (22.4 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes followed by 97:3 hexanes:EtOAc) to give **3ae** as a dark yellow oil in 47% yield (29.0 mg, 0.09 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.00 (dd, *J* = 8.1, 1.6 Hz, 2H), 7.52 – 7.46 (m, 1H), 7.45 – 7.40 (m, 2H), 7.28 (ddd, *J* = 5.1, 1.3, 0.6 Hz, 1H), 7.11 (ddd, *J* = 3.5, 1.3, 0.6 Hz, 1H), 7.00 (ddd, *J* = 5.1, 3.5, 0.6 Hz, 1H), 6.08 (s, 1H), 2.58 – 2.42 (m, 3H), 2.03 (ddd, *J* = 14.2, 9.4, 5.5 Hz, 1H), 1.63 – 1.48 (m, 3H), 1.48– 1.32 (m, 1H), 0.99 (t, *J* = 7.3 Hz, 3H), 0.93 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 168.4, 142.9, 134.9, 131.3, 128.0, 126.7, 126.3, 125.8, 85.0, 29.4, 29.0, 23.8, 22.4, 14.4, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 46.7. **HRMS** (EI) m/z: (M^+) Calc. for $C_{19}H_{23}BOS$ 310.1563; Found: 310.1568.



3af

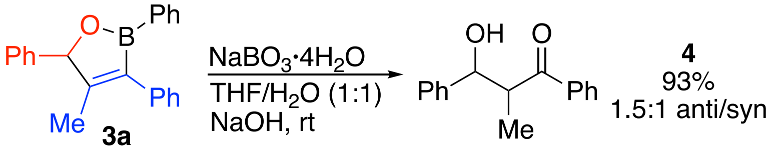
5-(tert-butyl)-2-phenyl-3,4-dipropyl-2,5-dihydro-1,2-oxaborole (3af): Prepared according to general procedure **B** from pivaldehyde (17.2 mg, 0.200 mmol), 4-octyne (24.2 mg, 0.220 mmol), and BPh_3 (72.6 mg, 0.300 mmol) at 50 °C in a block heater in 18 h. The crude reaction mixture was purified by flash column chromatography treated with 1% Et_3N (100% hexanes) to give **3af** as a clear oil in 47% yield (26.5 mg, 0.09 mmol). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.09 (dd, *J* = 6.2, 1.8 Hz, 2H), 7.56 (m, 3H), 4.77 (s, 1H), 2.72 (ddd, *J* = 12.0, 8.8, 6.1 Hz, 1H), 2.66 – 2.47 (m, 2H), 2.38 (ddd, *J* = 14.3, 9.6, 5.0 Hz, 1H), 1.83 – 1.70 (m, 1H), 1.69 – 1.50 (m, 3H), 1.14 (s, 9H), 1.08 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 168.1, 134.7, 130.8, 127.9, 94.2, 35.7, 30.7, 29.4, 26.6, 24.0, 22.9, 14.4, 14.4 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 44.6. **HRMS** (EI) m/z: (M^+) Calc. for $C_{19}H_{29}BO$ 284.2311; Found: 284.2322.

Experimental Procedure for Ni-catalyzed Dearylative Cyclocondensation – 4 mmol Scale



In a glovebox atmosphere, an oven dried 100 mL round-bottom flask was charged with $Ni(cod)_2$ (55.0 mg, 0.200 mmol), benzaldehyde **1a** (424.5 mg, 4.00 mmol), 1-phenyl-1-propyne (511.0 mg, 4.40 mmol), PBu_3 (98.7 µL, 0.400 mmol), triphenylborane (1.45 g, 6.00 mmol), and THF (40 mL, 0.1 M). The resulting solution was stirred at 50 °C in an oil bath for 18 h. The reaction was cooled to room temperature, diluted with DCM, and concentrated. The residue was dissolved in 70:30 hexane/EtOAc and filtered through a short plug of silica gel, eluting with additional 70:30 hexane/EtOAc. The crude product was purified by flash column chromatography (100% hexanes followed by 97:3 hexanes:EtOAc) to give oxaborane product **3a** in 97% yield (1.201 g) and 11.8:1 r.r.

Experimental Procedure for the Synthesis of Beta-hydroxy Ketone 4:



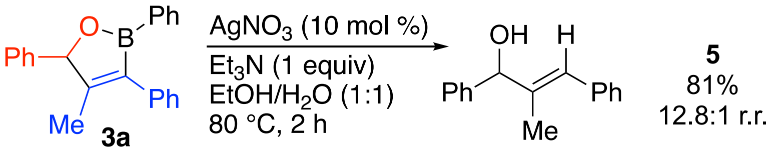
The procedure was modified from a literature procedure.[12] An oven dried 1 dram vial was charged with 4-methyl-2,3,5-triphenyl-2,5-dihydro-1,2-oxaborole **3a** (31.0 mg, 0.1 mmol, 1.00 equiv), sodium perborate tetrahydrate (76.9 mg, 0.5 mmol), NaOH (8.0 mg, 0.2 mmol), THF (0.5 mL) and H_2O (0.5 mL). The resulting mixture was stirred at room temperature for 2 hours. After reaching completion, the reaction mixture was added to water and extracted with Et_2O x3. The combined organic layers were washed with brine, dried with $MgSO_4$, and concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography (100% hexanes followed by a gradient from 100% hexanes to 90:10 hexanes:EtOAc) to give **4** as a clear oil in 93% yield (22.6 mg, 0.093 mmol) and 1.5:1 d.r.

Anti- **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.0 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 2H), 7.42 (d, *J* = 7.2 Hz, 2H), 7.37 (t, *J* = 7.6 Hz, 2H), 7.30 (t, *J* = 7.2 Hz, 1H), 5.00 (d, *J* = 8.1 Hz, 1H), 3.84 (appt. p, *J* = 7.4 Hz, 1H), 2.97 (s, 1H), 1.07 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 205.0, 142.2, 136.8, 133.3, 128.7, 128.5, 128.5, 128.0, 126.7, 76.9, 48.0, 15.8.

Syn- **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.3 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.41 (d, *J* = 7.2 Hz, 2H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.24 (m, 1H), 5.25 (d, *J* = 2.9 Hz, 1H), 3.71 (qd, *J* = 7.2, 3.1 Hz, 1H), 3.64 (bs, 1H), 1.20 (d, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 205.8, 141.8, 135.7, 133.6, 128.8, 128.5, 128.3, 127.3, 126.1, 73.1, 47.1, 11.2.

HRMS (ESI) m/z: ([M+H]$^+$) Calc. for $C_{16}H_{17}O_2^+$ 241.1223; Found: 241.1223.

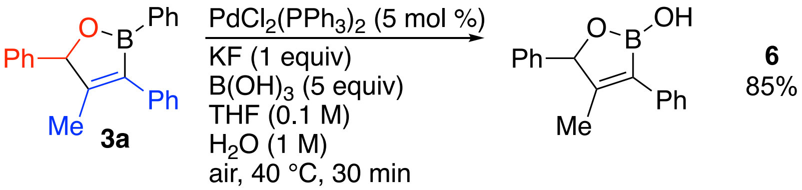
Experimental Procedure for the Synthesis of Allylic Alcohol 5:



The procedure was modified from a literature procedure.[13] An oven dried 1 dram vial was charged with 4-methyl-2,3,5-triphenyl-2,5-dihydro-1,2-oxaborole **3a** (31.0 mg, 0.1 mmol, 1.00 equiv), $AgNO_3$ (1.7 mmol, 0.01 mmol), $EtOH/H_2O$ (1.0 mL/1.0 mL, 0.05 M total), and Et_3N (13.9 µL, 0.1 mmol) under air. The vial was sealed and the mixture was stirred at 80 °C in a block heater for 2 hours. The reaction was allowed to cool to room temperature, water was added, and the mixture was extracted with Et_2O x3. The organic layers were combined and washed with brine, dried with $MgSO_4$, and concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography (91:9 hexanes:EtOAc) to give **6** as a clear oil in 81% yield (18.1 mg, 0.081 mmol) and 12.8:1 r.r.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.39 (m, 2H), 7.38 – 7.32 (m, 3H), 7.32 – 7.27 (m, 4H), 7.21 (m, 1H), 6.77 (s, 1H), 5.27 (s, 1H), 1.73 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.2, 139.7, 137.6, 129.2, 128.6, 128.3, 127.8, 126.7, 126.6, 126.1, 79.6, 14.2. **HRMS** (EI) m/z: (M$^+$) Calc. for $C_{16}H_{16}O$ 224.1201; Found: 224.1203.

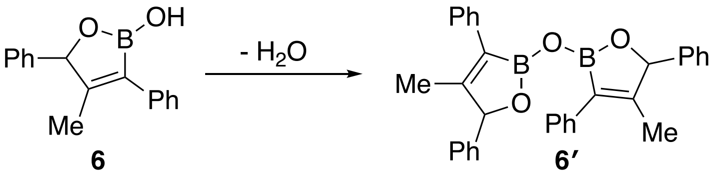
Experimental Procedure for the Palladium Catalyzed Synthesis of Oxaborole 6:



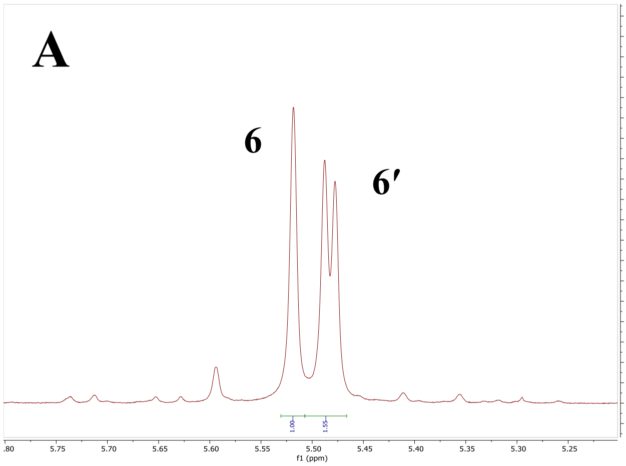
The procedure was modified from a literature procedure.[14] A 1 dram vial was charged with bis(triphenylphosphine)palladium(II) dichloride (3.5 mg, 0.005 mmol), 4-methyl-2,3,5-triphenyl-2,5-dihydro-1,2-oxaborole **3a** (31.0 mg, 0.1 mmol, 1.00 equiv), boric acid (30.9 mg, 0.5 mmol), and THF (1.0 mL, 0.1 M). The reaction mixture was stirred open to air at 40 °C in a block heater for 1 minute. Potassium fluoride (5.8 mg, 0.1 mmol) was dissolved in water (0.1 mL, 1 M) and added to the reaction mixture. The resulting mixture was stirred open to air at 40 °C in a block heater for 30 minutes. The reaction mixture was diluted with EtOAc, transferred to a round bottom flask, and concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography (95:5 to 90:10 hexanes:EtOAc) to give **6** as a yellow solid in 85% yield (21.3 mg, 0.085 mmol).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (m, 8H), 7.29 (d, *J* = 7.3 Hz, 2H), 5.51 (s, 1H), 4.46 (s, 1H), 1.86 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.3, 138.5, 136.6, 128.7, 128.5, 128.5, 128.4, 127.0, 126.6, 86.9, 14.1 (carbons directly attached to the B atom are not detected in ^{13}C NMR due to quadrupolar relaxation). **^{11}B NMR** (193 MHz, $CDCl_3$) δ 32.9. **HRMS** (ESI) m/z: ($[M+H]^+$) Calc. for $C_{16}H_{16}BO_2^+$ 251.1238; Found: 251.1243.

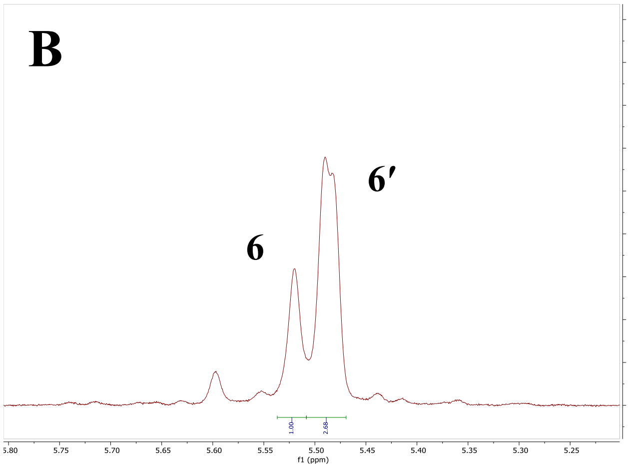
Dimerization of Oxaborole 6:



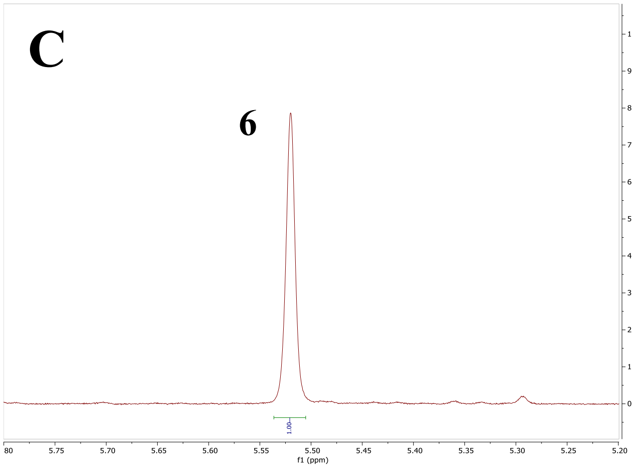
We observe dimerization of oxaborole **6** via a condensation reaction (Spectrum A). The presence of the dimer was confirmed by heating oxaborole **6** in a Kugelrohr apparatus at 100 °C for 18 hours leading to a 2.7:1 ratio of oxaborole dimer **6′** to oxaborole **6** (Spectrum B). Additionally, a mixture of oxaborole **6** with oxaborole dimer **6′** was dissolved in $CDCl_3$ and D_2O. ^1H NMR showed complete conversion to oxaborole **6** with no observable dimer (Spectrum C).



a) Oxaborole **6** and dimer **6′** in 1:1.5 ratio

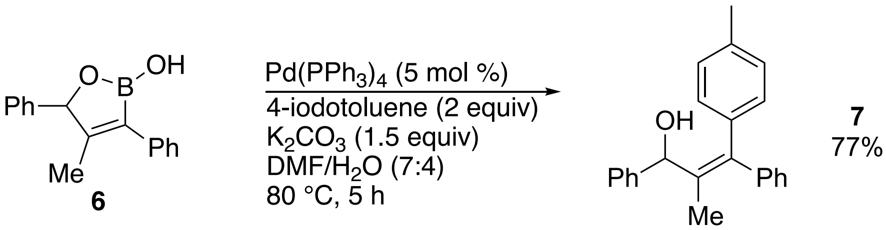


b) Oxaborole **6** and dimer **6′** in 1:2.7 ratio after heating in Kugelrohr apparatus



c) Oxaborole **6** in $CDCl_3$ and D_2O with no observable dimer

Experimental Procedure for the Synthesis of Allylic Alcohol 7:

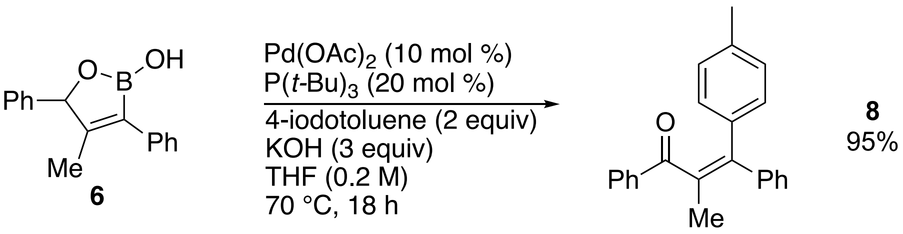


The procedure was modified from a literature procedure.[15] An oven dried 1 dram vial was charged with 4-methyl-3,5-diphenyl-1,2-oxaborol-2(5*H*)-ol **7** (25.0 mg, 0.1 mmol, 1.00 equiv), $Pd(PPh_3)_4$ (5.8 mg, 0.005 mmol), 4-iodotoluene (43.6 mg, 0.2 mmol), K_2CO_3 (20.7 mg, 0.15

mmol), DMF (1.67 mL, 0.06 M), and degassed H_2O (0.25 mL, 0.4 M). The resulting solution was stirred at 80 °C in a block heater for 5 hours. The reaction mixture was allowed to cool to room temperature, after which it was diluted with a 50:50 mixture of Et_2O/H_2O. The organic layer was removed and the aqueous layer was extracted with Et_2O x3. The organic layers were combined and washed with brine, dried with $MgSO_4$, and concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography (95:5 hexanes:EtOAc) to (90:10 hexanes:EtOAc) to give **8** as a clear oil in 77% yield (24.2 mg, 0.077 mmol).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.32 (m, 5H), 7.32 – 7.24 (m, 3H), 7.19 – 7.13 (m, 6H), 5.66 (s, 1H), 2.33 (s, 3H), 1.63 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.9, 142.6, 141.2, 139.4, 136.6, 135.2, 129.5, 129.3, 129.2, 128.3, 128.2, 127.0, 126.7, 125.8, 73.6, 21.3, 14.2. **HRMS** (EI) m/z: (M^+) Calc. for $C_{23}H_{22}O$ 314.1671; Found: 314.1667.

Experimental Procedure for the Synthesis of Enone 8:



An oven dried 1 dram vial was charged with 4-methyl-3,5-diphenyl-1,2-oxaborol-2(5*H*)-ol **7** (25.0 mg, 0.1 mmol, 1.00 equiv), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), P(*t*-Bu)$_3$ (4.0 mg, 0.02 mmol), 4-iodotoluene (43.6 mg, 0.2 mmol), KOH (16.8 mg, 0.3 mmol), and THF (0.5 mL, 0.2 M). The resulting solution was stirred at 70 °C in a block heater for 18 hours. The reaction mixture was allowed to cool to room temperature, after which it was run through a plug of silica with DCM and concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography (100% hexanes) to (90:10 hexanes:EtOAc) to give **9** as a white solid in 95% yield (29.6 mg, 0.095 mmol).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.80 (m, 2H), 7.43 – 7.24 (m, 8H), 6.90 (d, J = 8.2 Hz, 2H), 6.82 (d, J = 8.2 Hz, 2H), 2.13 (s, 3H), 2.13 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.5, 143.8, 141.2, 138.5, 137.4, 136.9, 134.3, 132.6, 130.0, 129.9, 129.4, 128.6, 128.3, 128.2, 127.7, 21.2, 20.0. **HRMS** (EI) m/z: (M^+) Calc. for $C_{23}H_{20}O$ 312.1514; Found: 312.1509.

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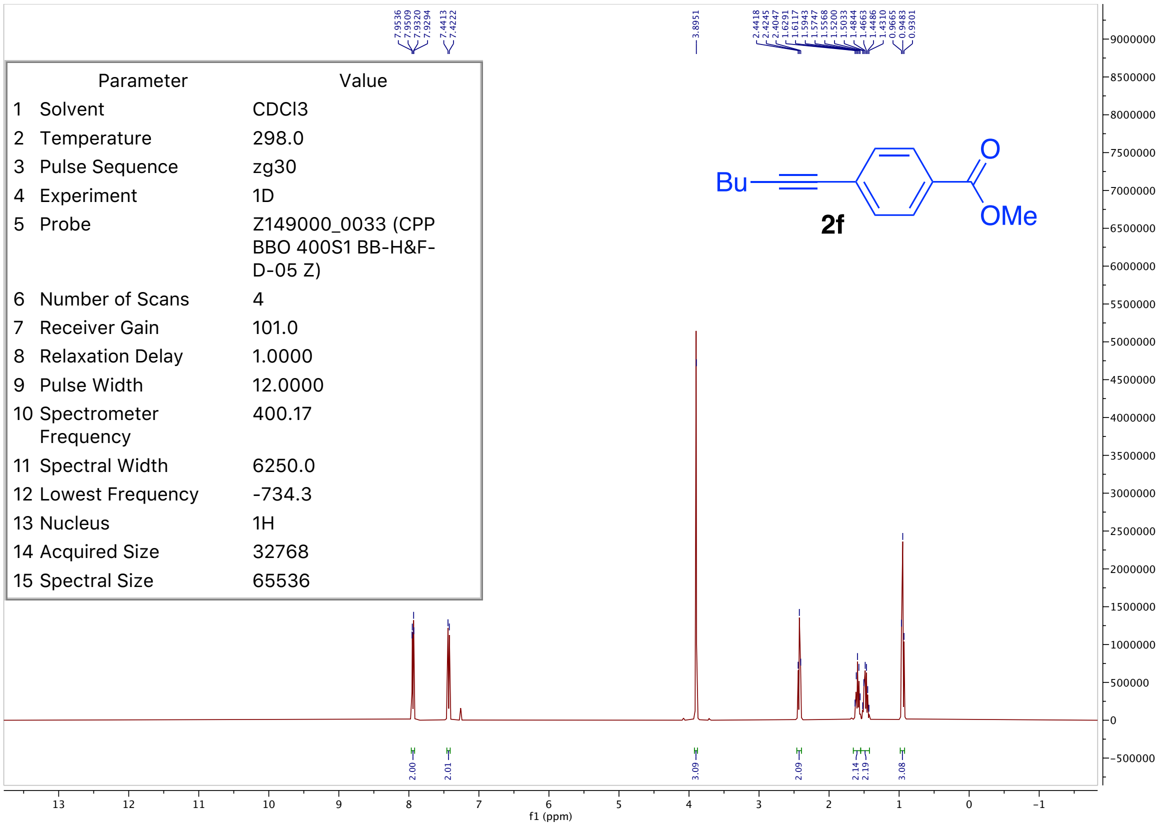
^{1}H, ^{13}C, ^{19}F, and ^{11}B NMR Spectra



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6	Number of Scans	4
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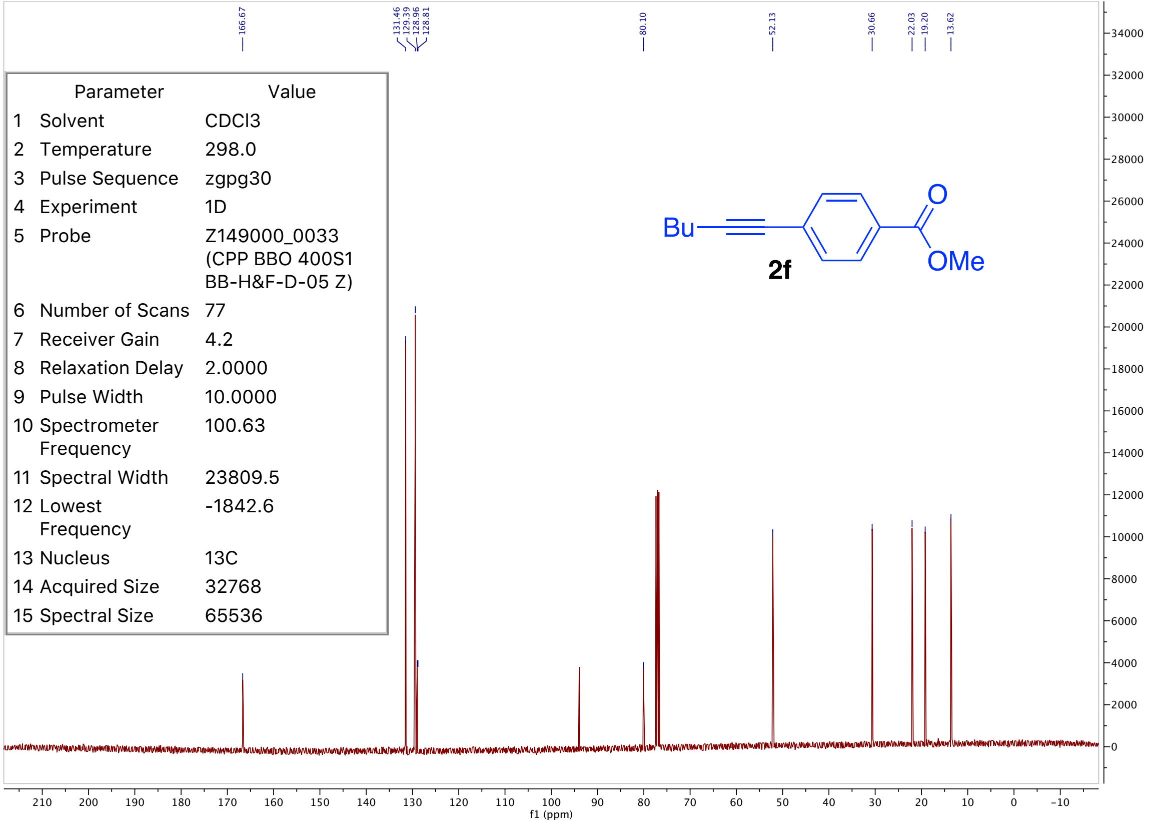

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8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
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12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
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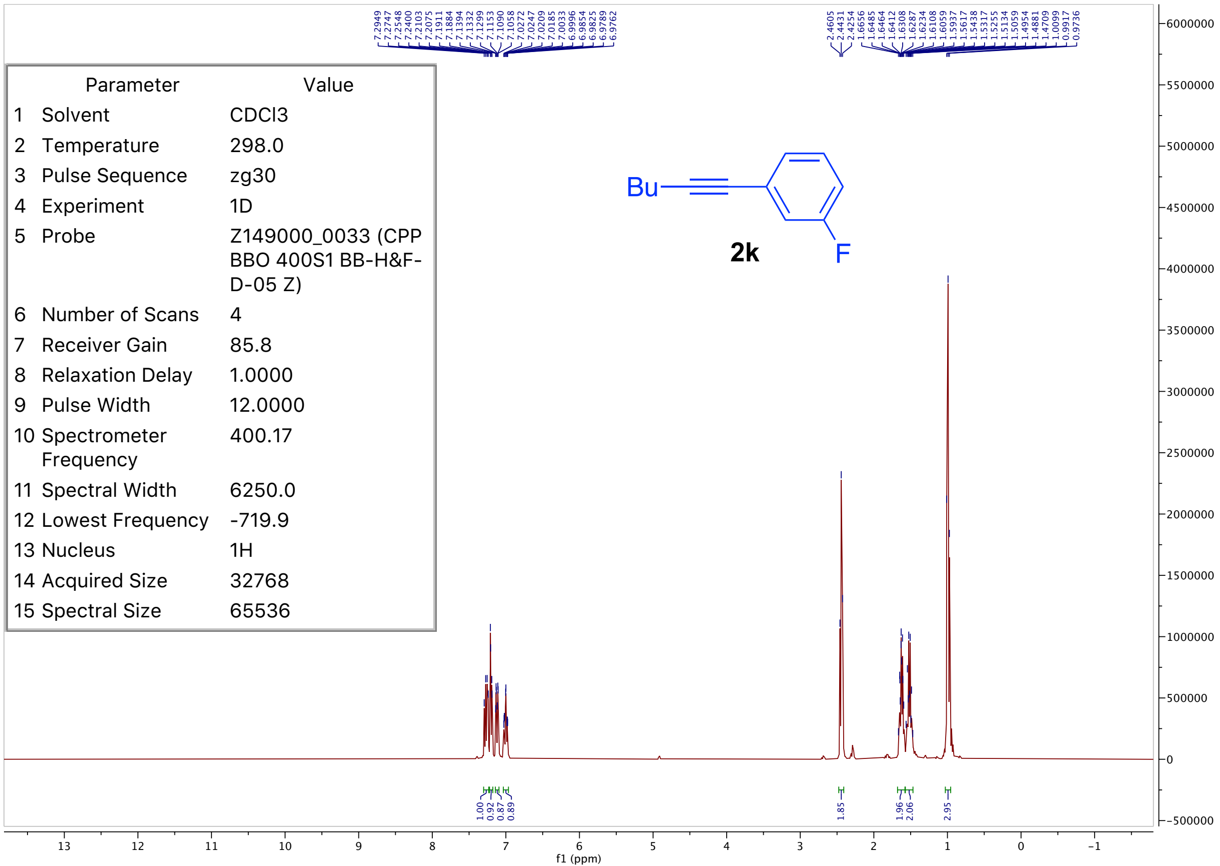

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14	Acquired Size	32768
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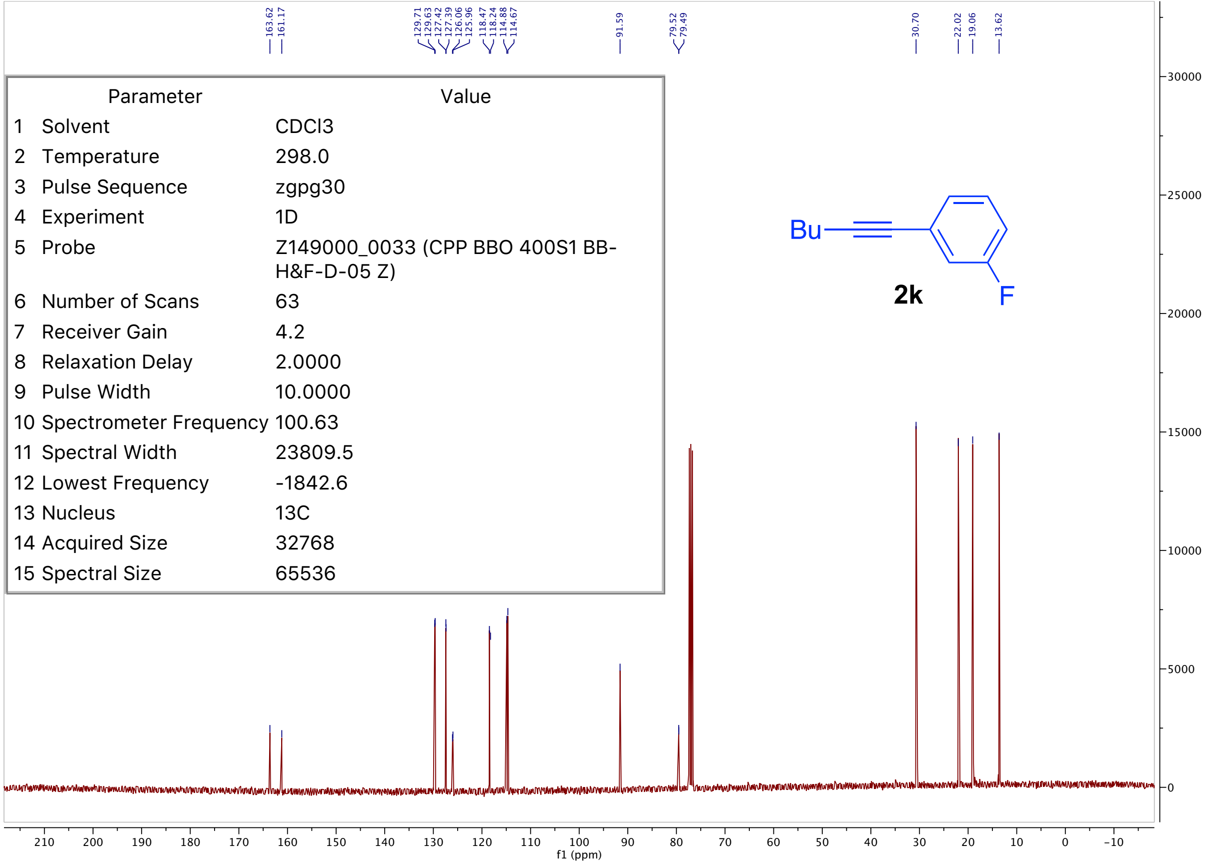

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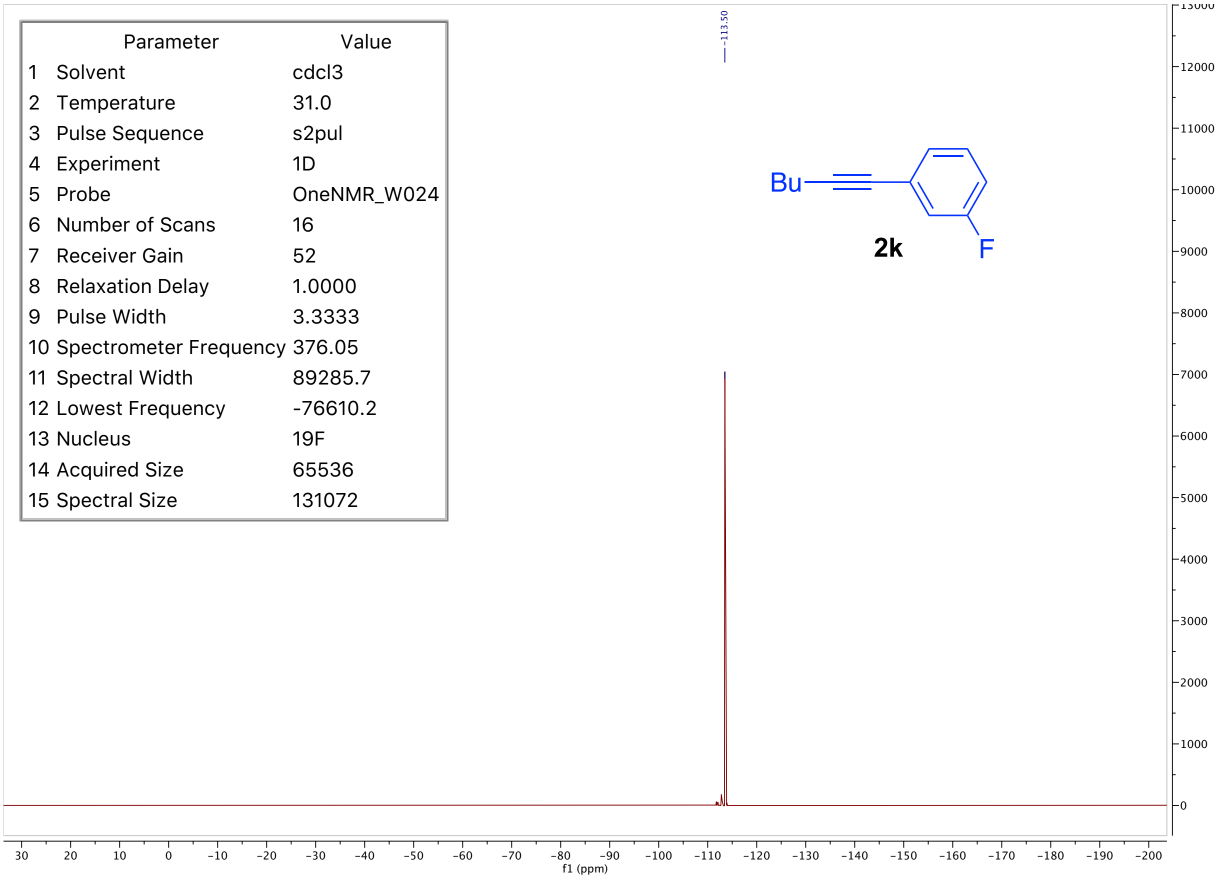
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



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13	Nucleus	19F
14	Acquired Size	65536
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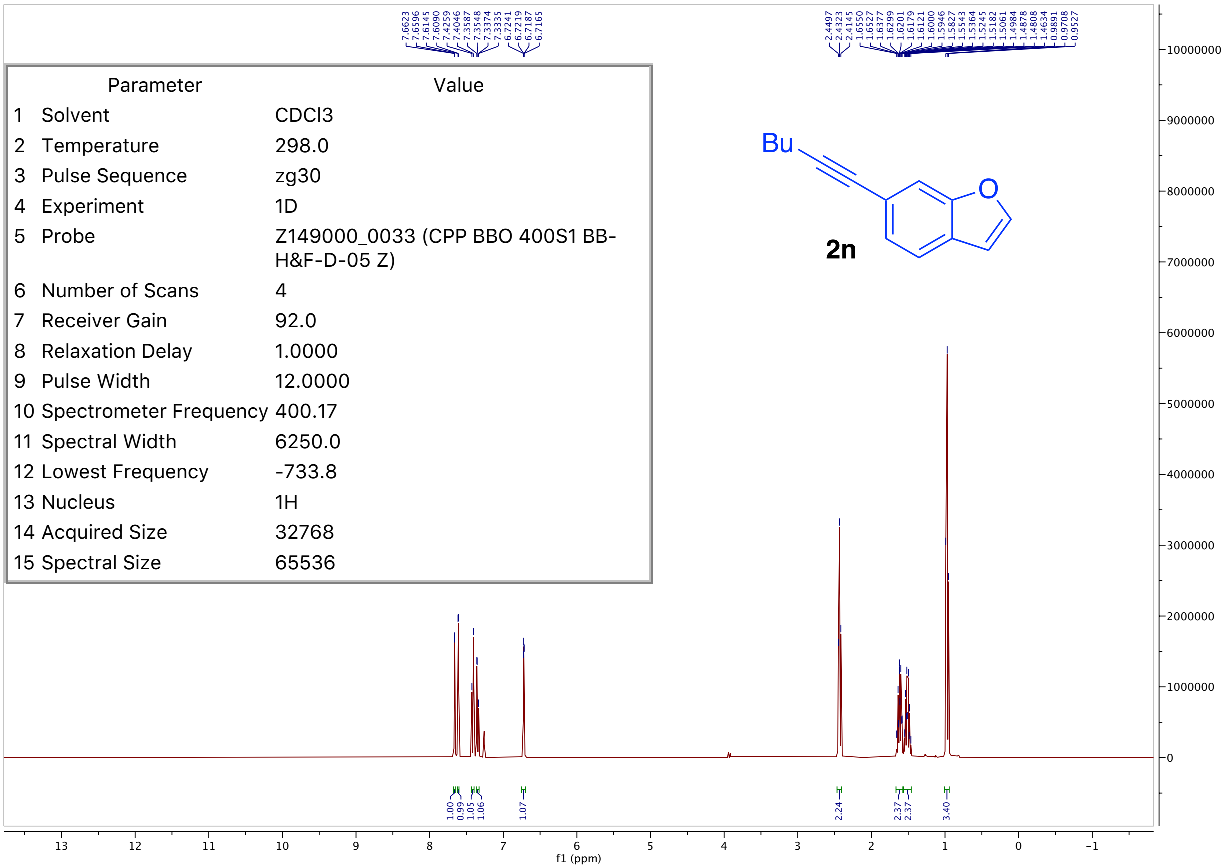

Parameter	Value
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4 Experiment	1D
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8 Relaxation Delay	1.0000
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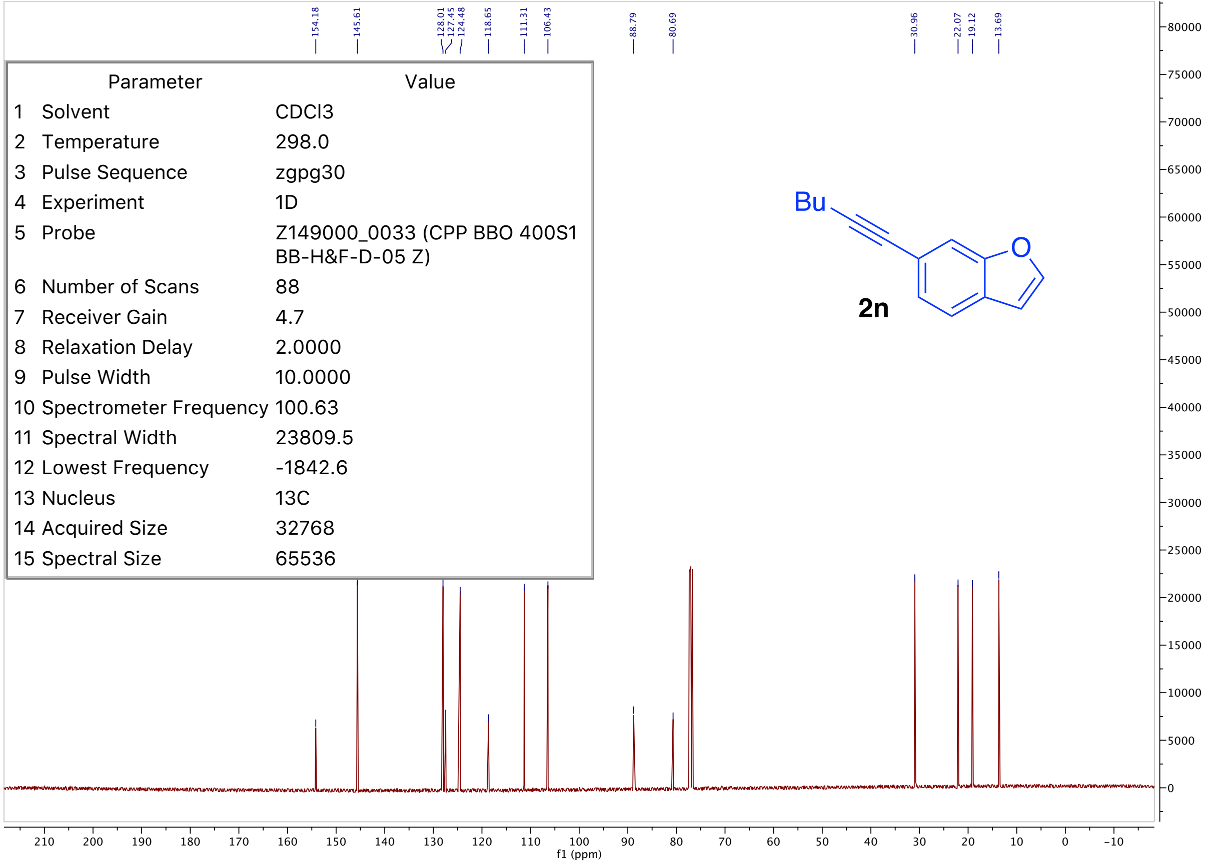
13 Nucleus	1H
14 Acquired Size	32768
15 Spectral Size	65536



Parameter	Value
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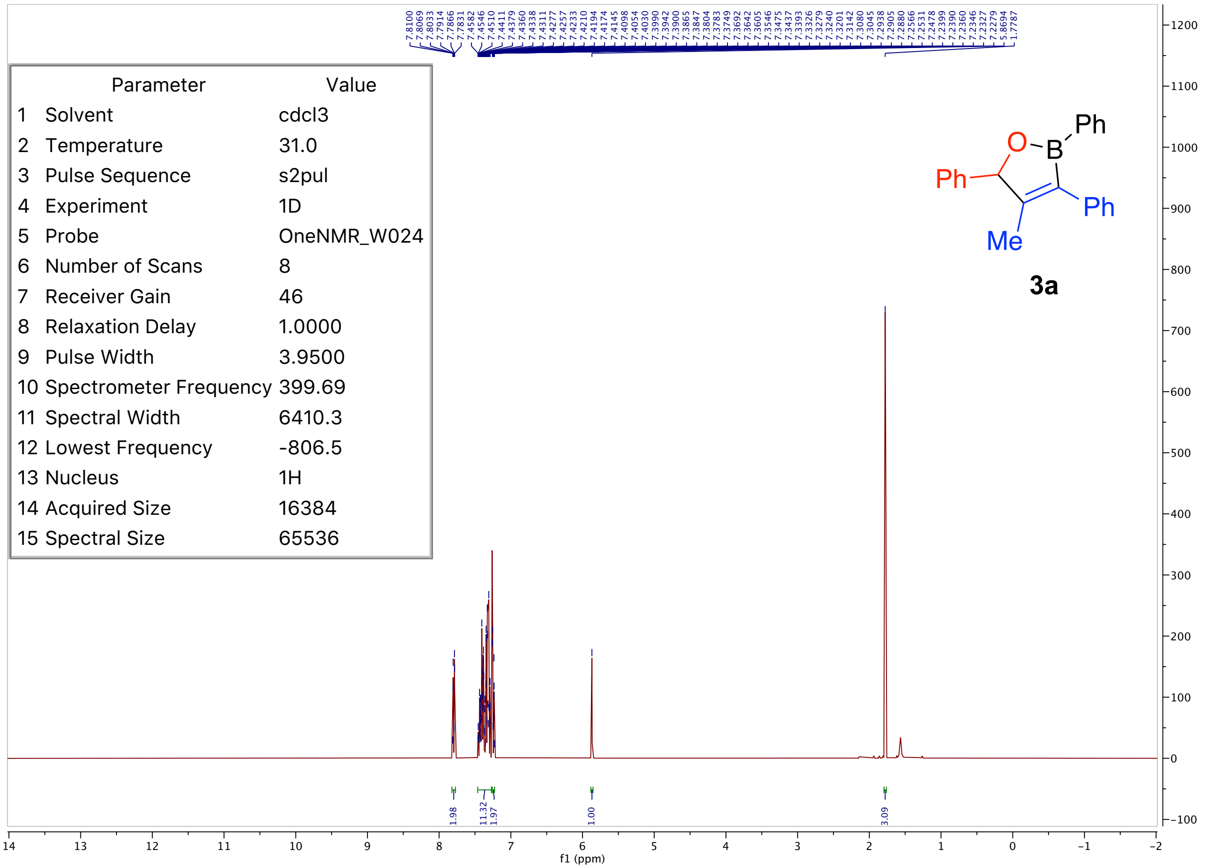
14 Acquired Size	32768
15 Spectral Size	65536



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8	Relaxation Delay	1.0000
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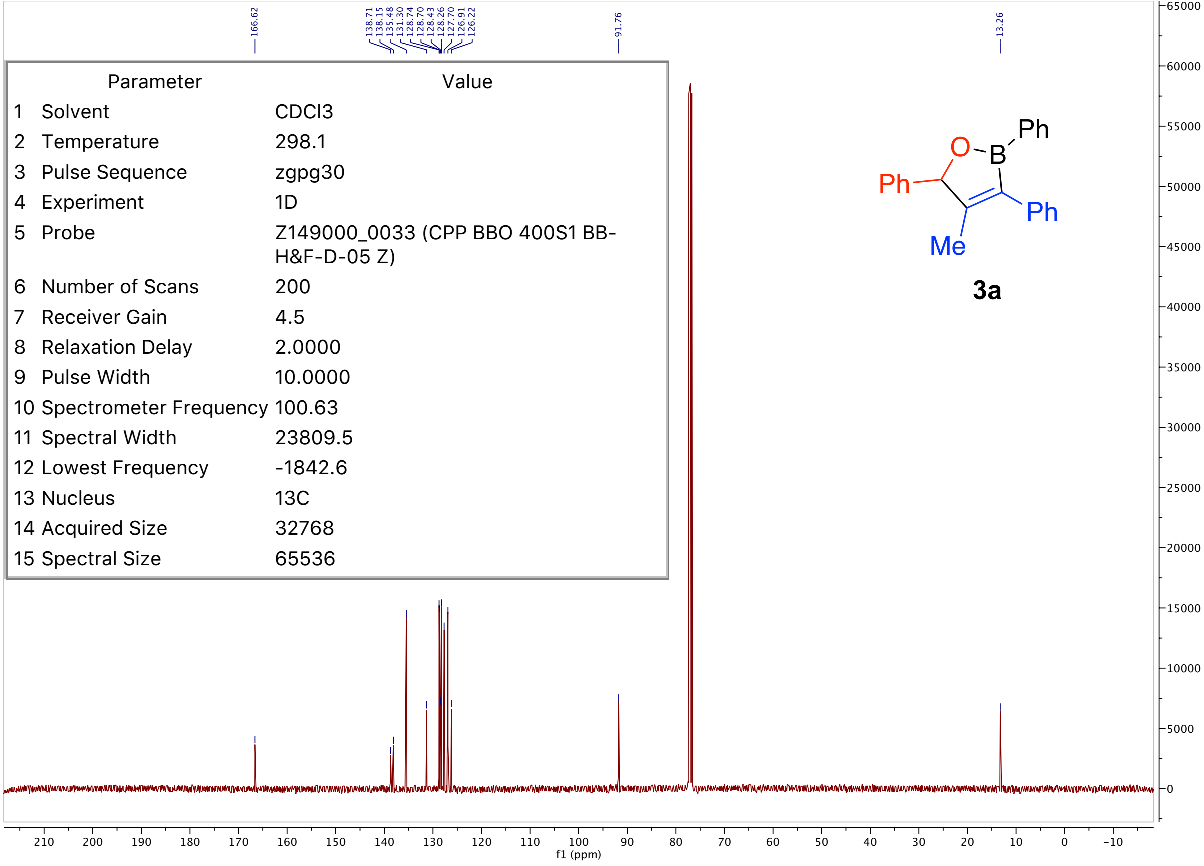
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.5
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

3a



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7	Receiver Gain	4.5
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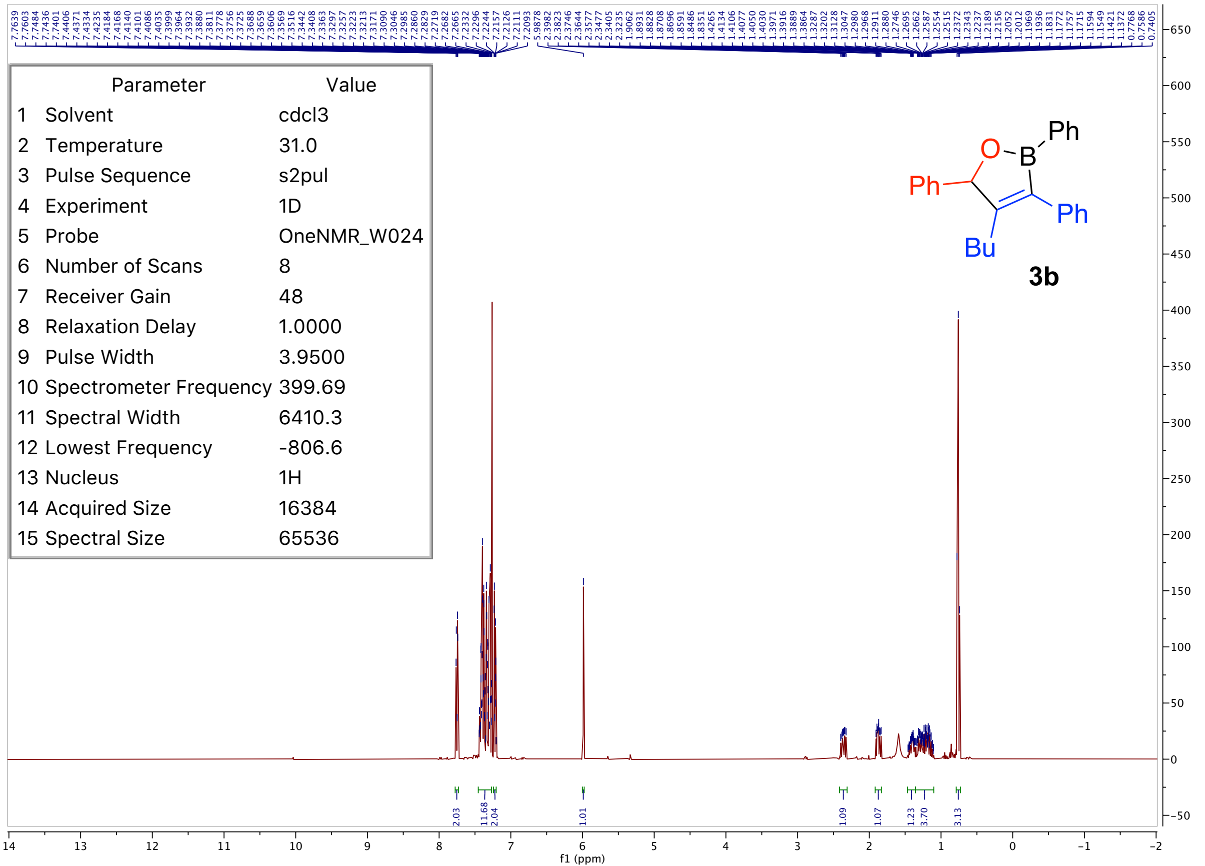
14	Acquired Size	32768
15	Spectral Size	65536

3a



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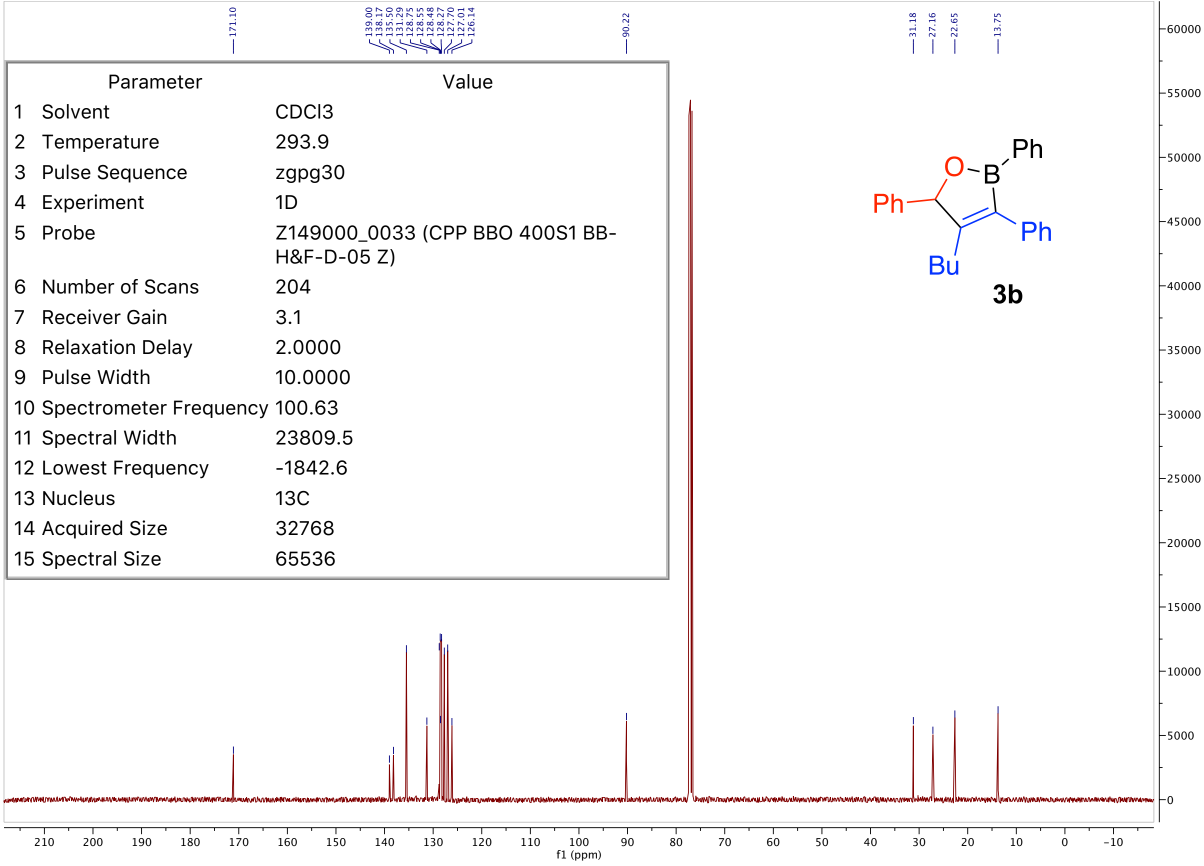

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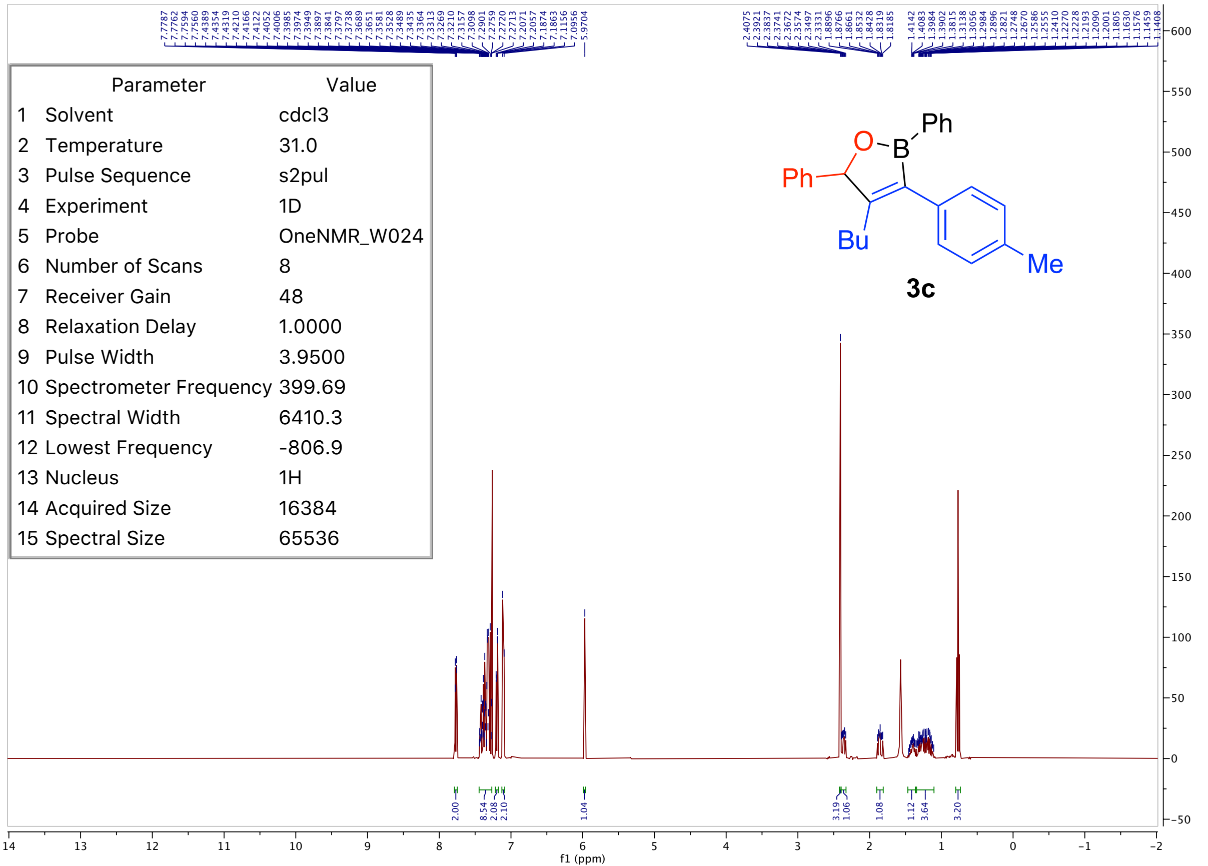
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	cdcl3
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9	Pulse Width	3.9500
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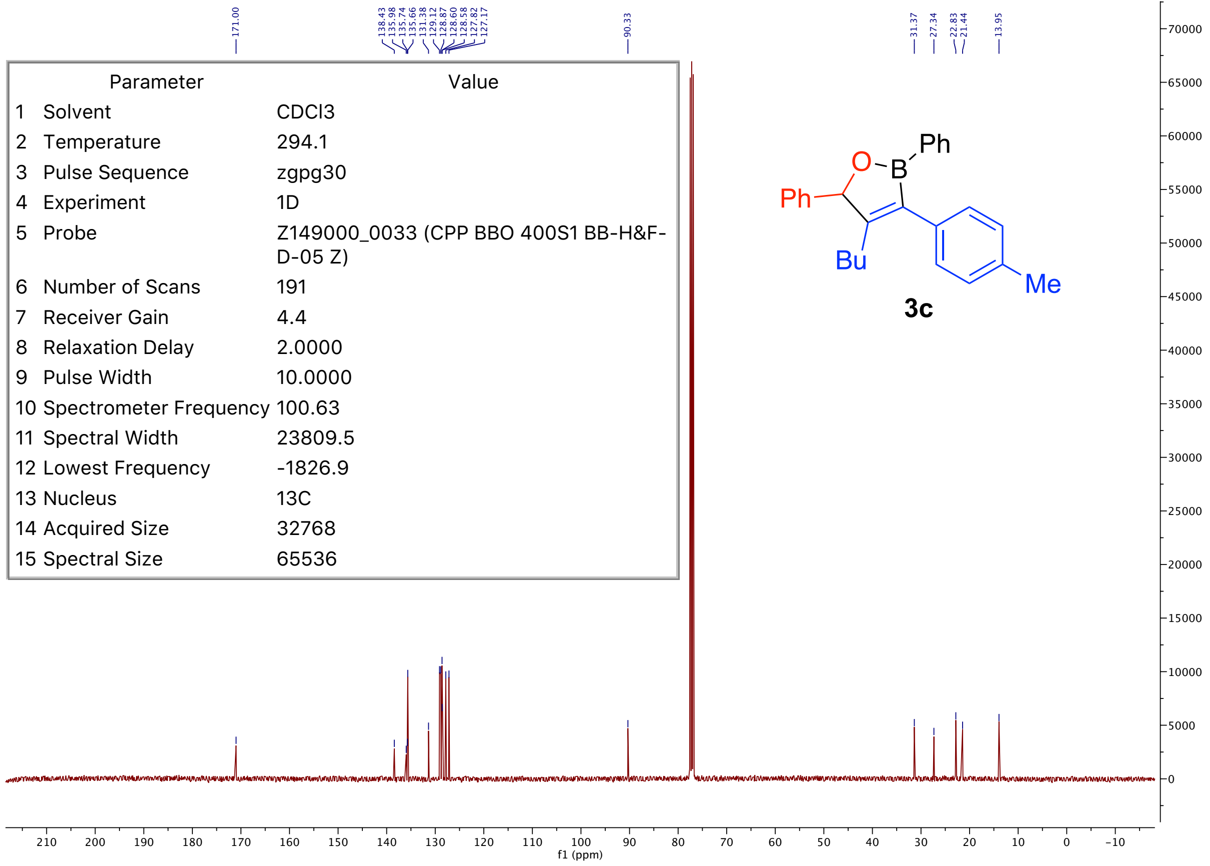
12	Lowest Frequency	-806.9
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

3c



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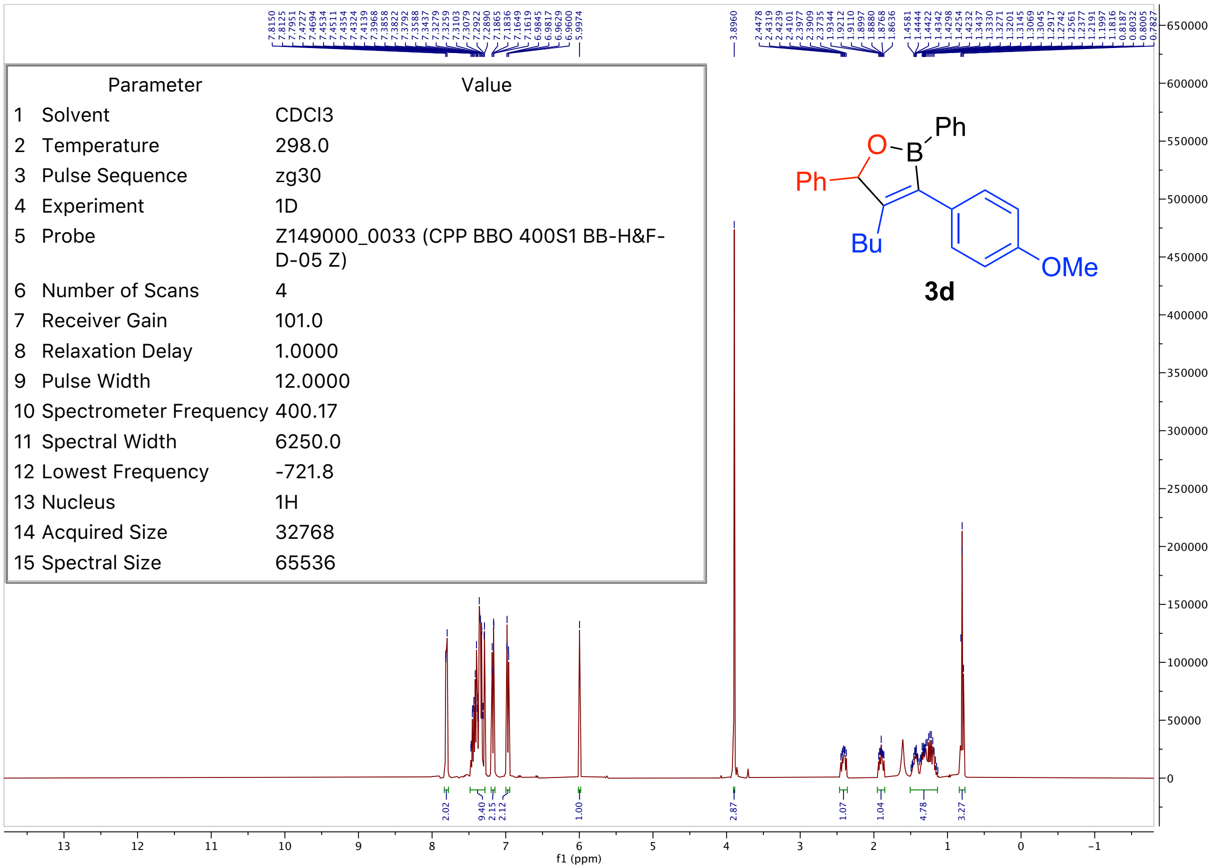
3c



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9	Pulse Width	12.0000
10	Spectrometer Frequency	400.17
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12	Lowest Frequency	-721.8
13	Nucleus	1H
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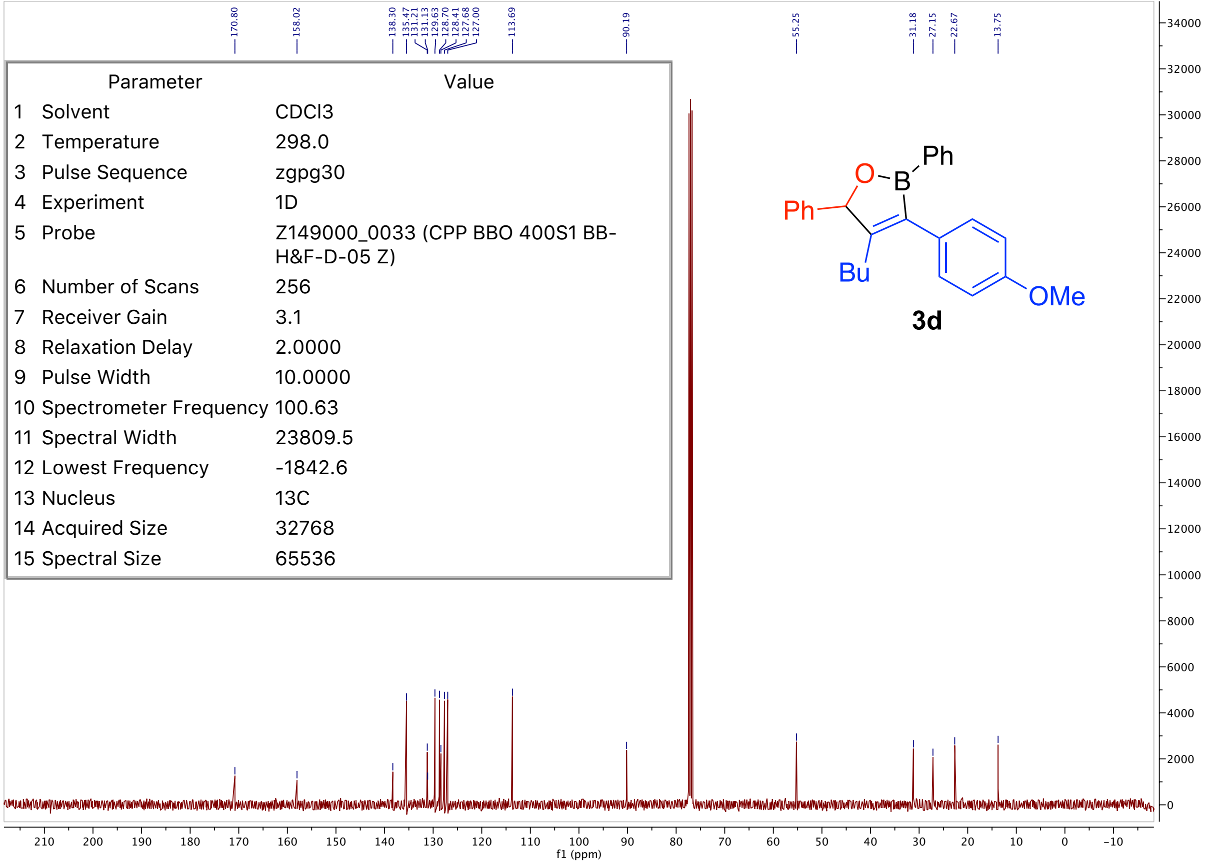
15	Spectral Size	65536

3d



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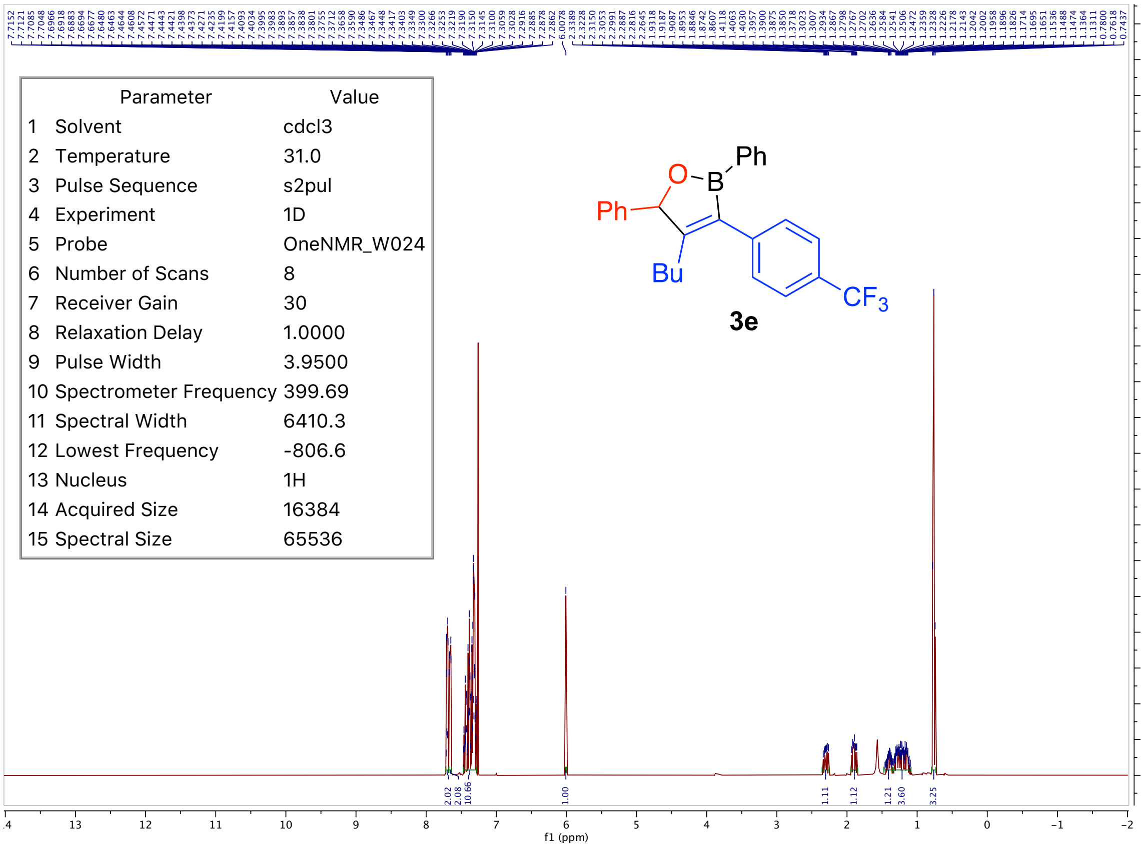
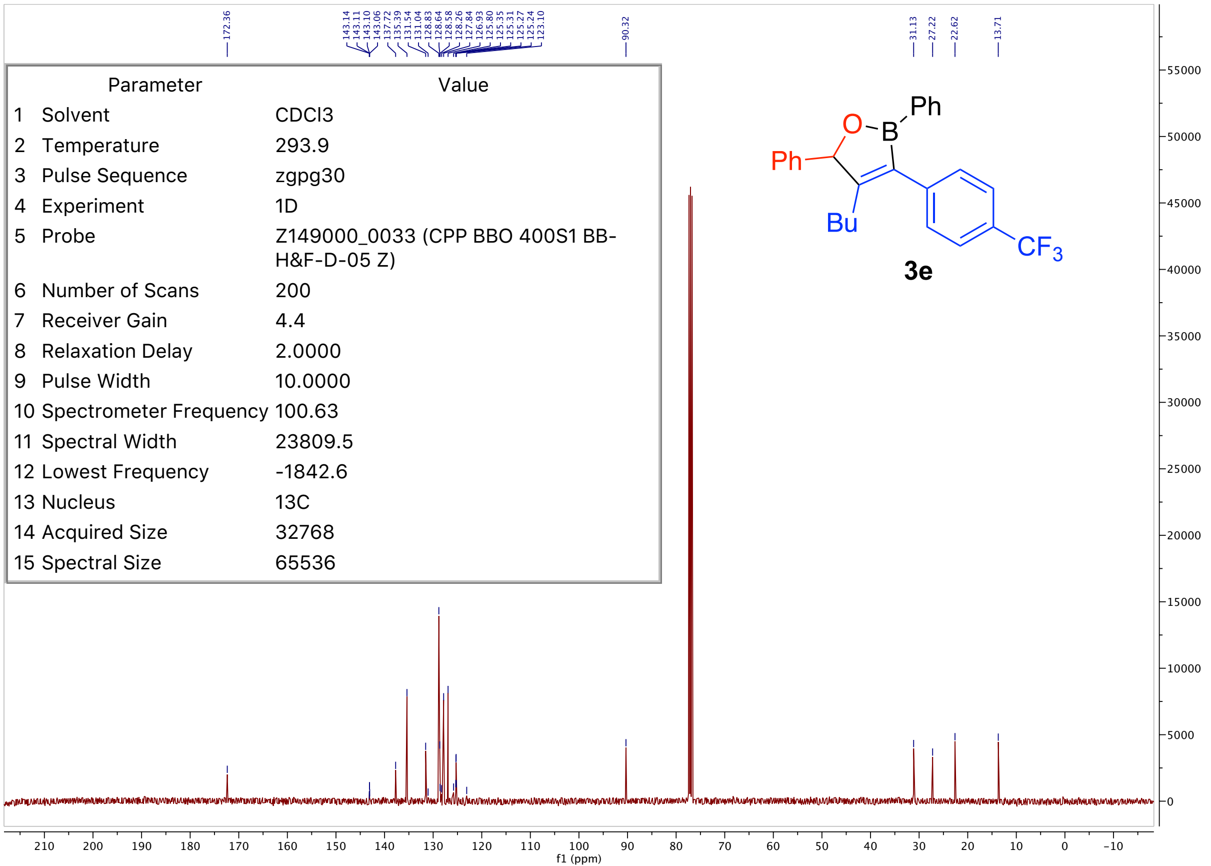
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536

3d



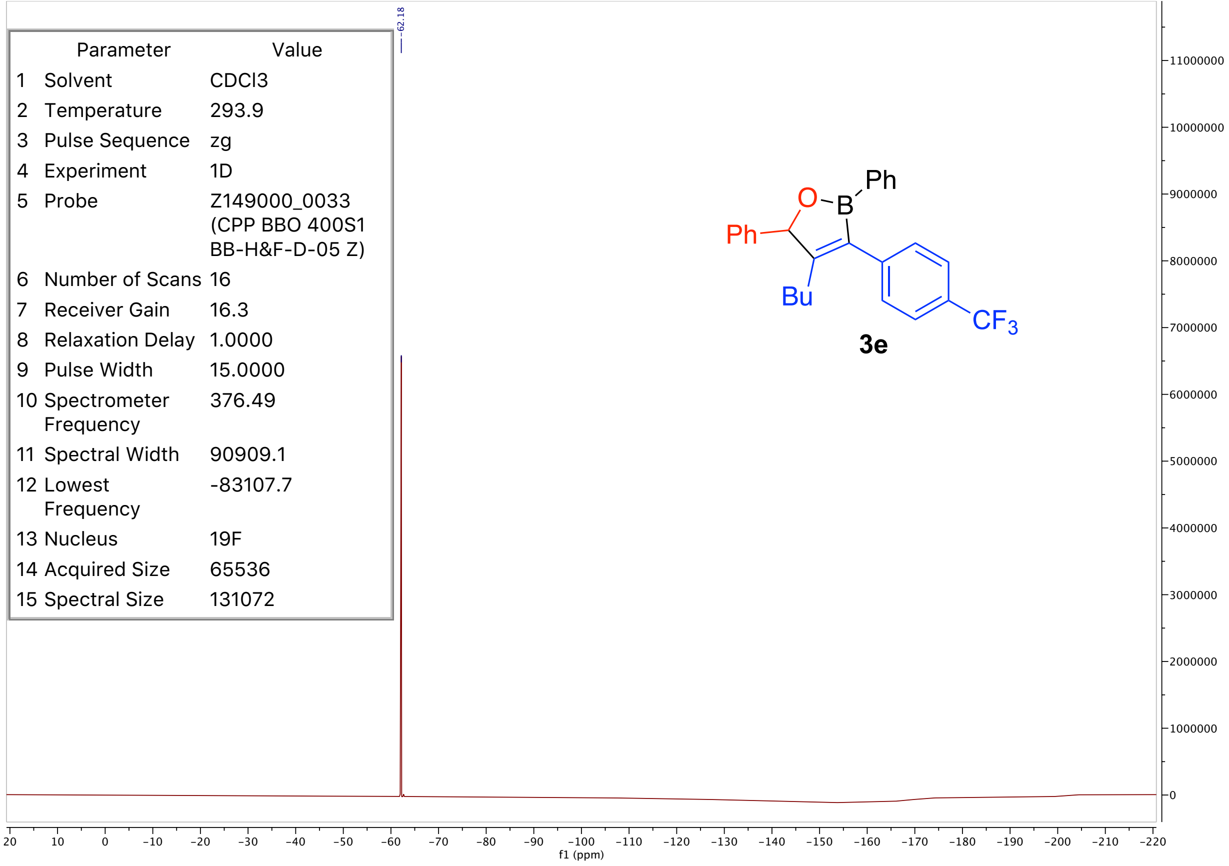
3e

	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	30
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.6
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



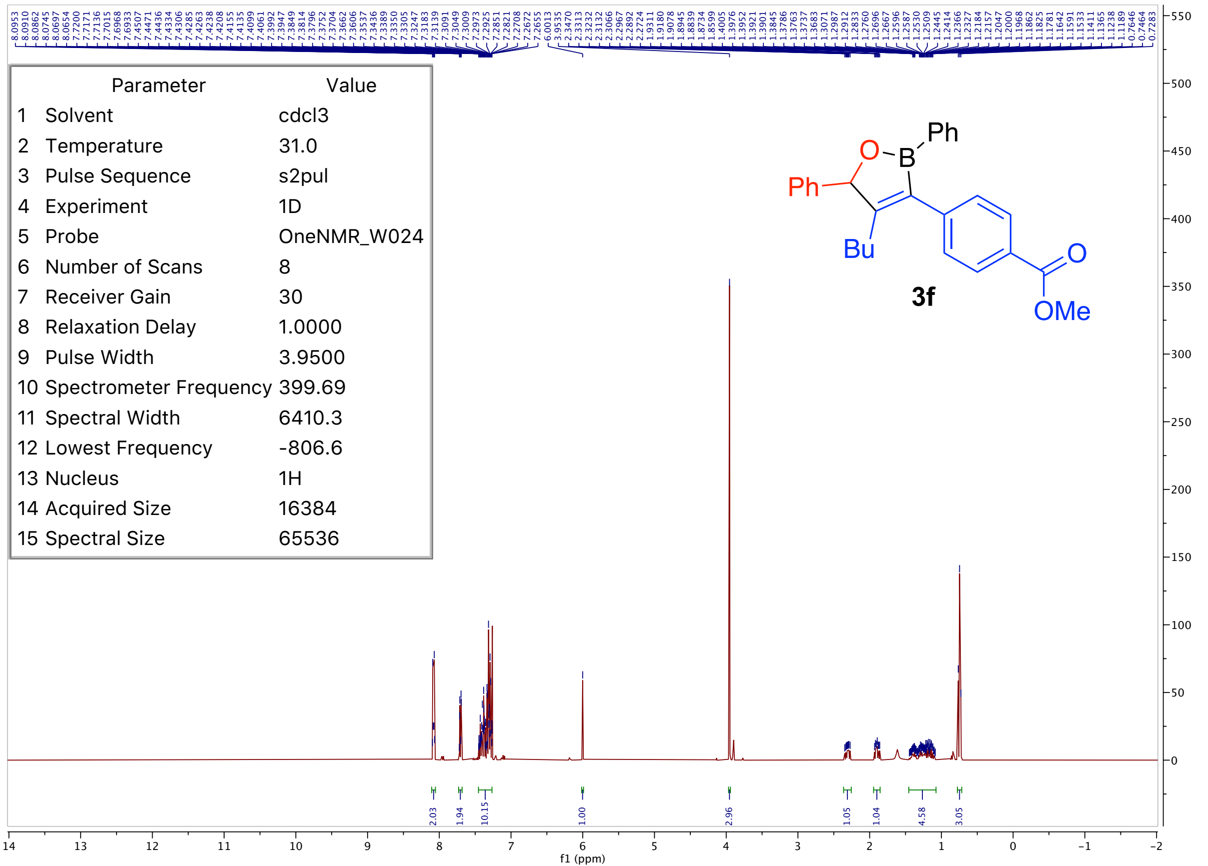
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	293.9
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	200
7	Receiver Gain	4.4
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536

3e

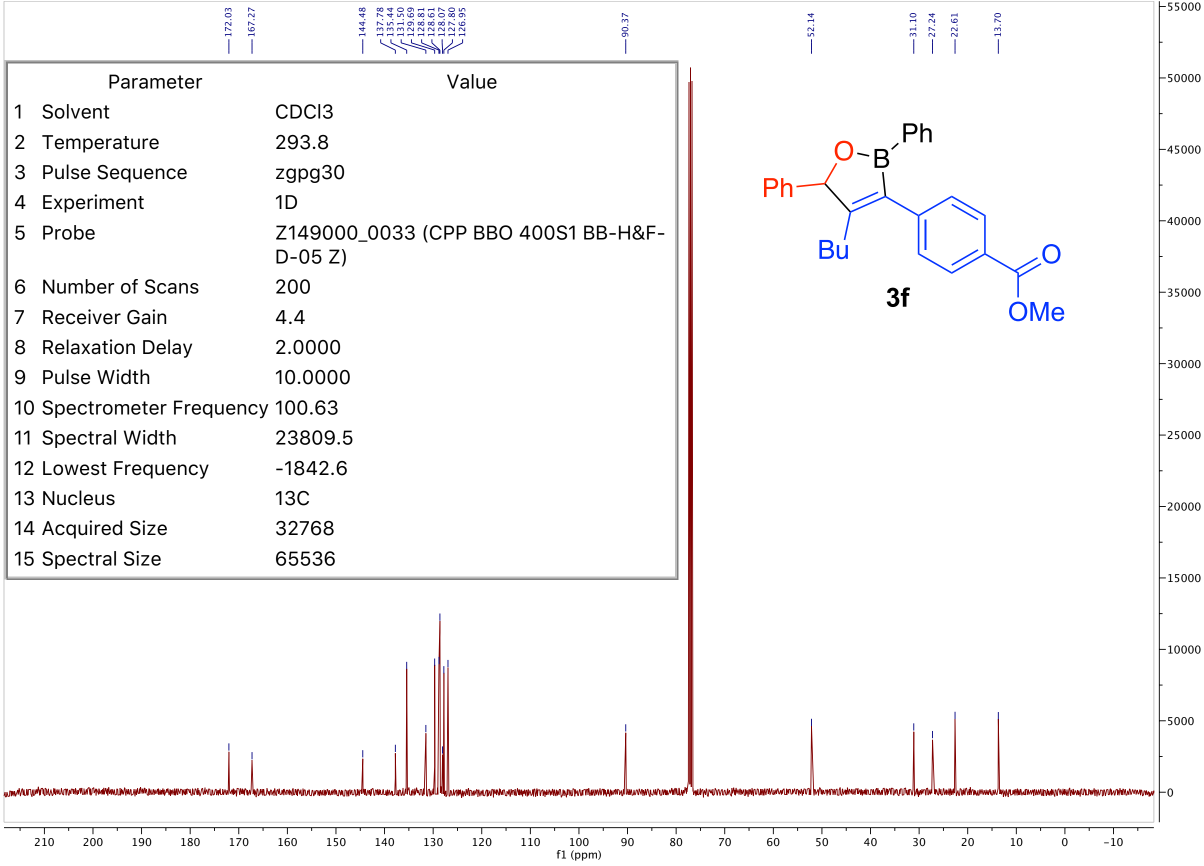


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	293.9
3	Pulse Sequence	zg
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	16
7	Receiver Gain	16.3
8	Relaxation Delay	1.0000
9	Pulse Width	15.0000
10	Spectrometer Frequency	376.49
11	Spectral Width	90909.1
12	Lowest Frequency	-83107.7
13	Nucleus	19F
14	Acquired Size	65536
15	Spectral Size	131072

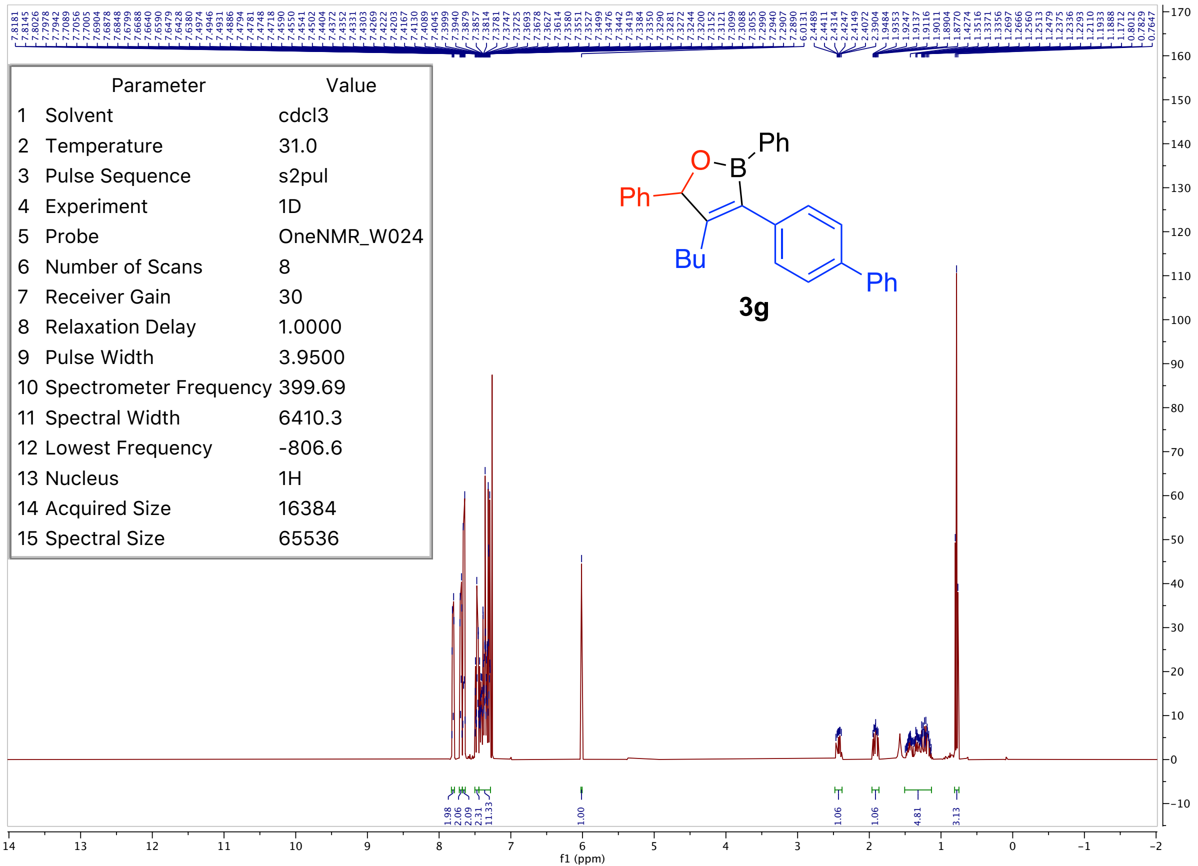
3e



	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	30
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.6
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

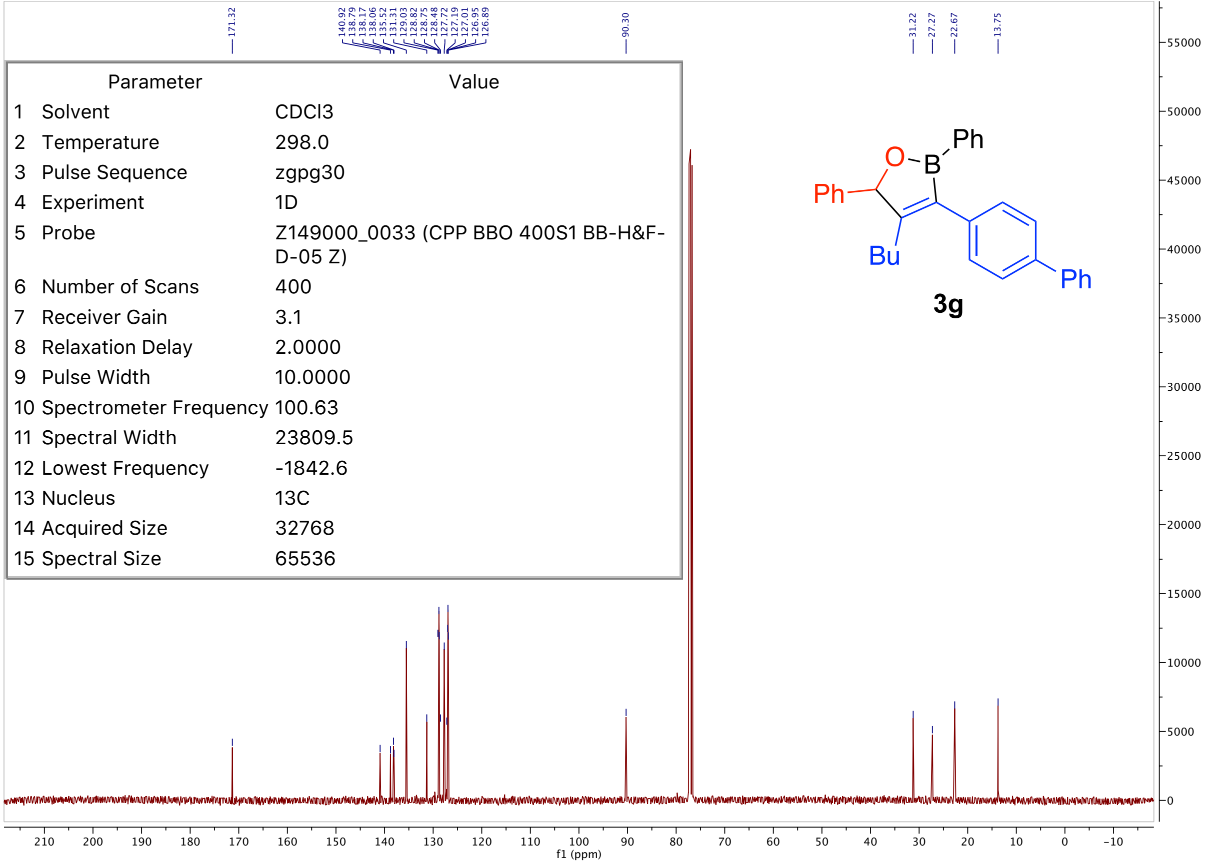


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	293.8
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	200
7	Receiver Gain	4.4
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



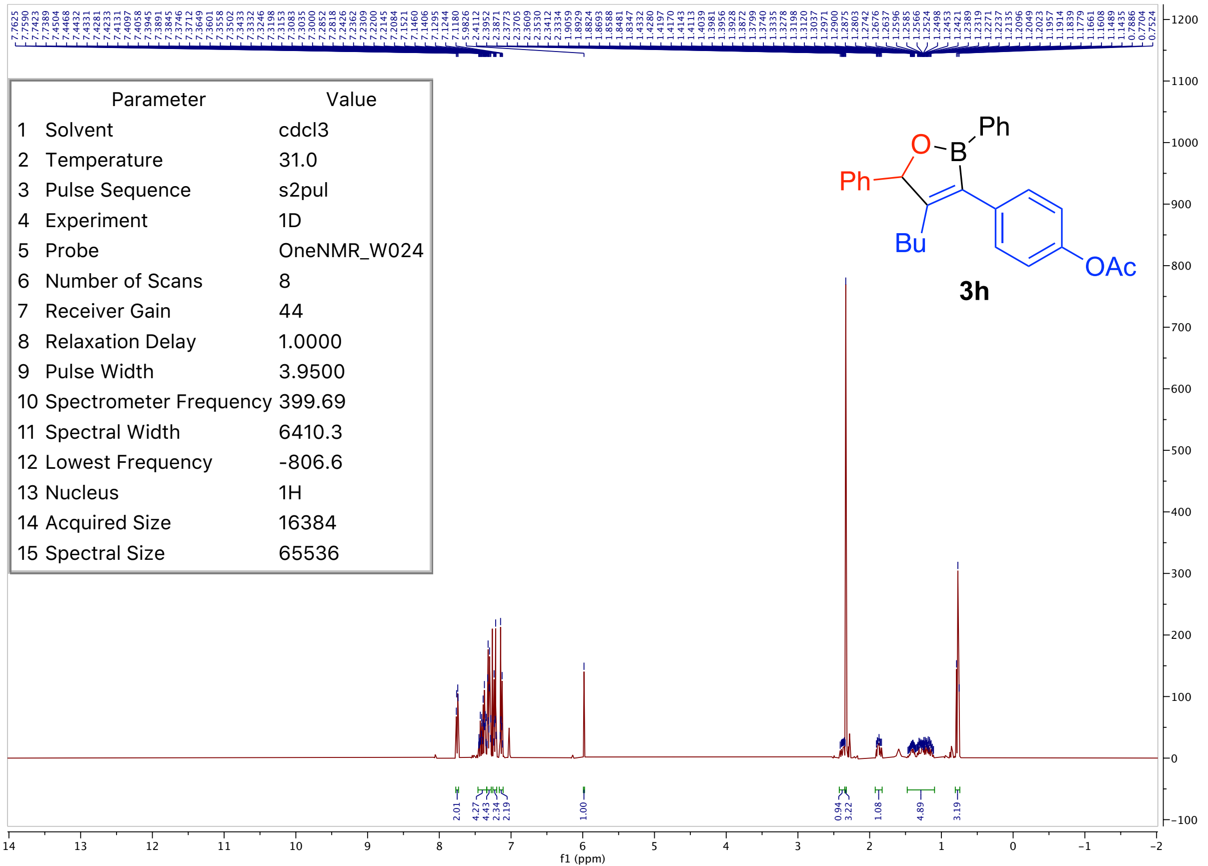
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	30
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.6
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

3g

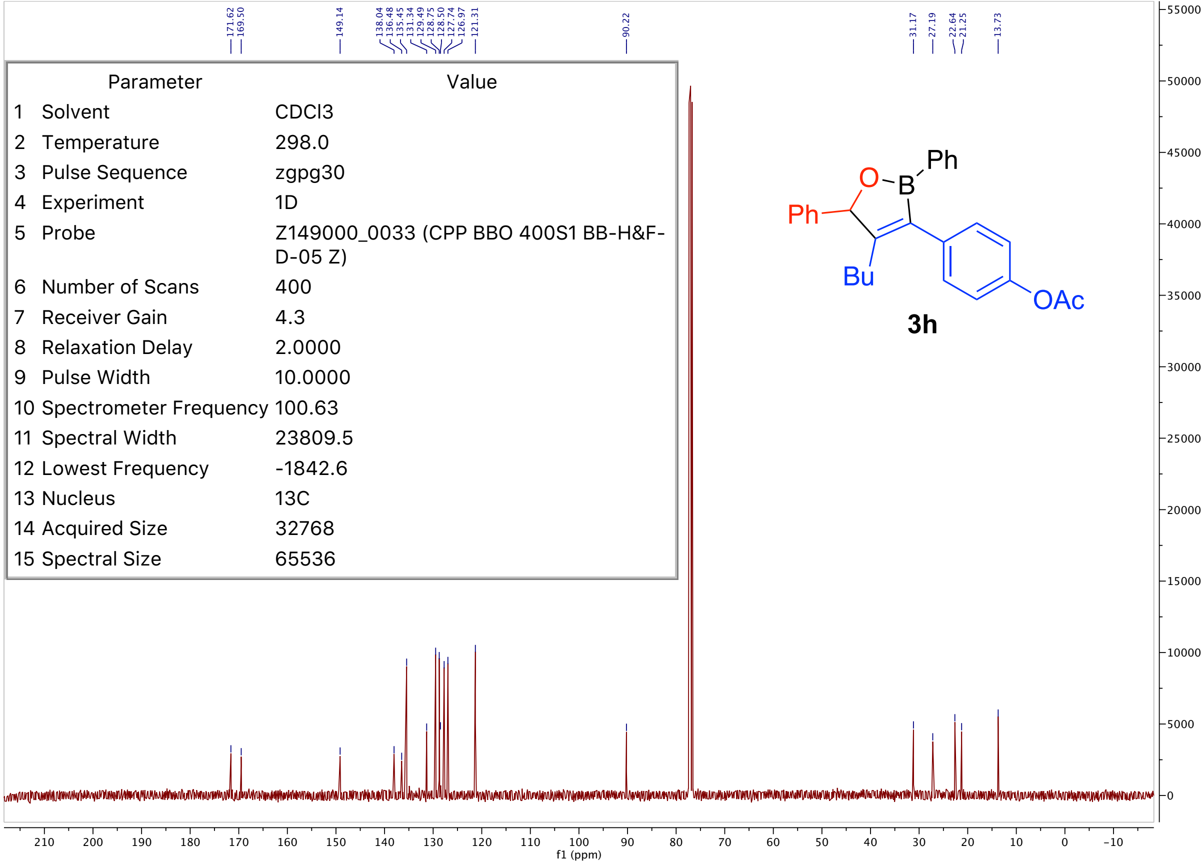


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	400
7	Receiver Gain	3.1
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536

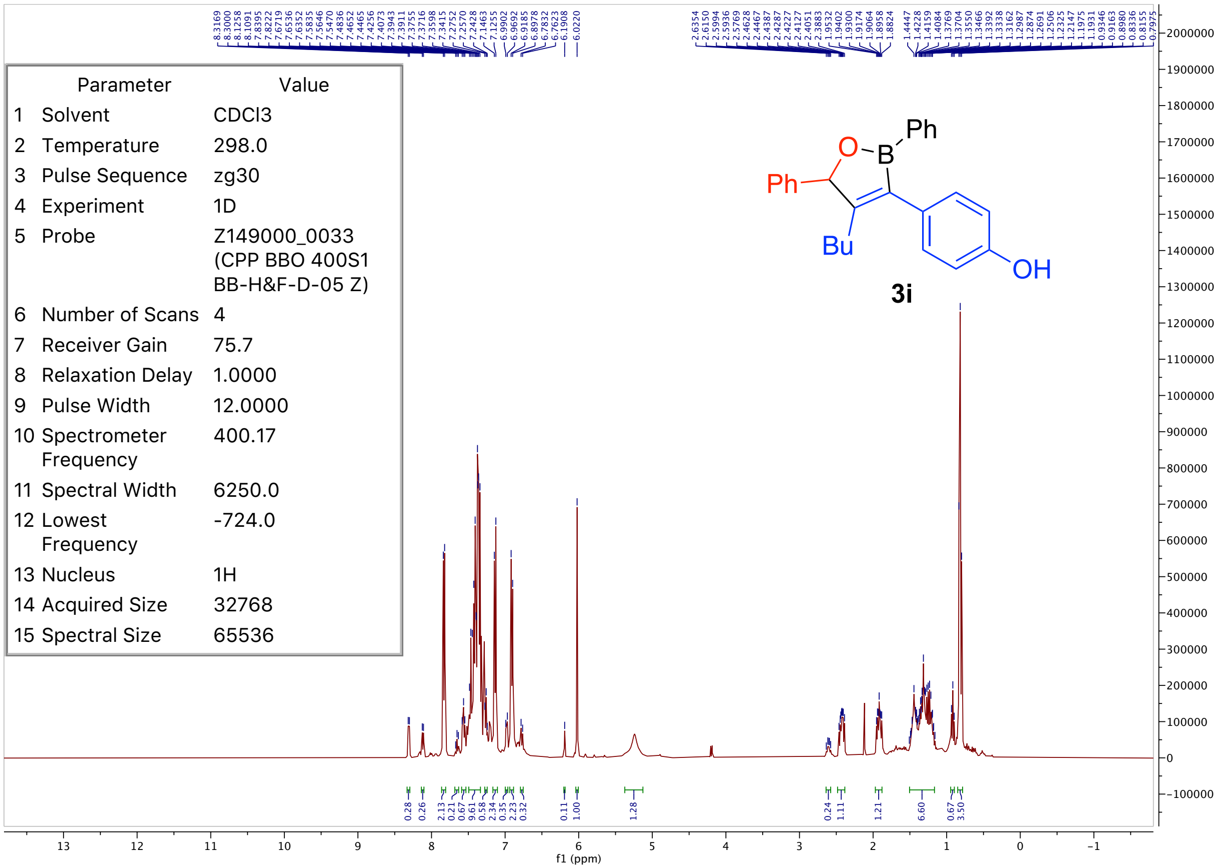
3g



	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	44
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.6
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

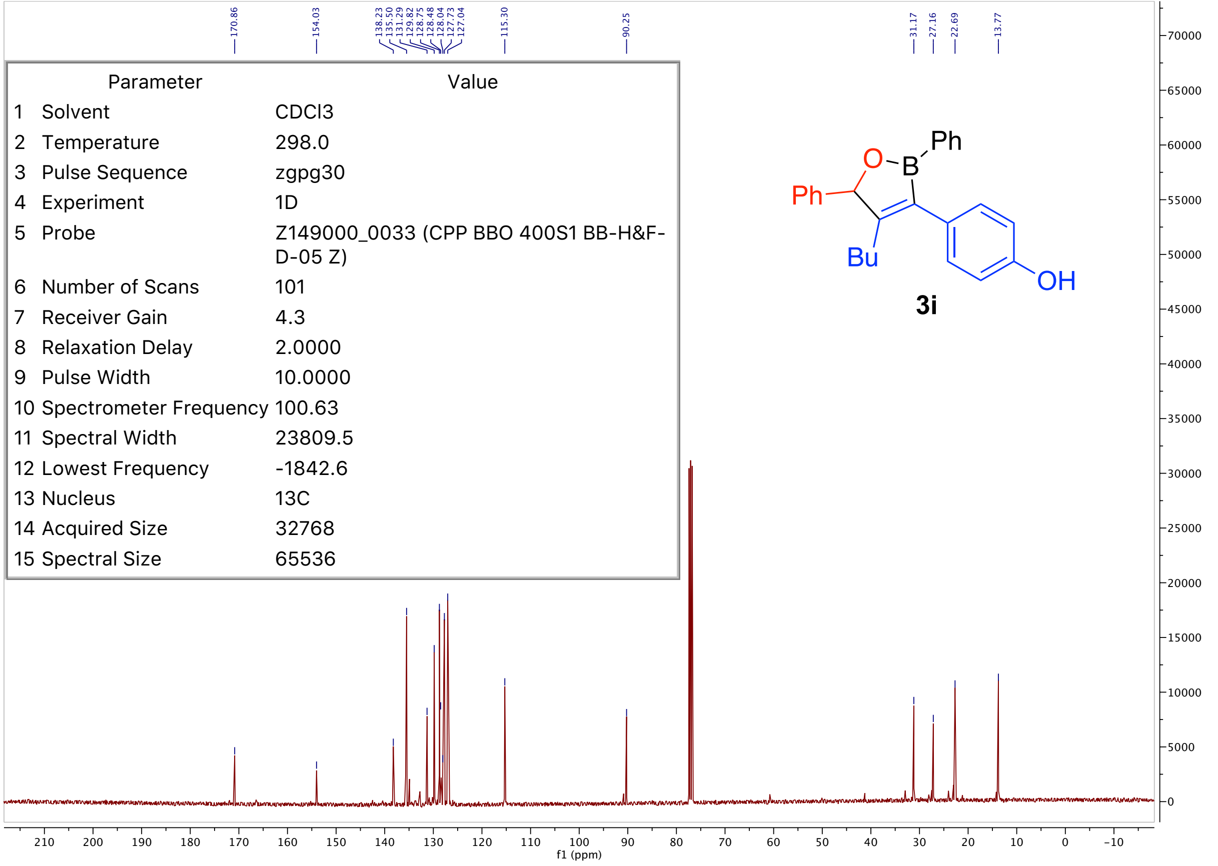


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	400
7	Receiver Gain	4.3
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



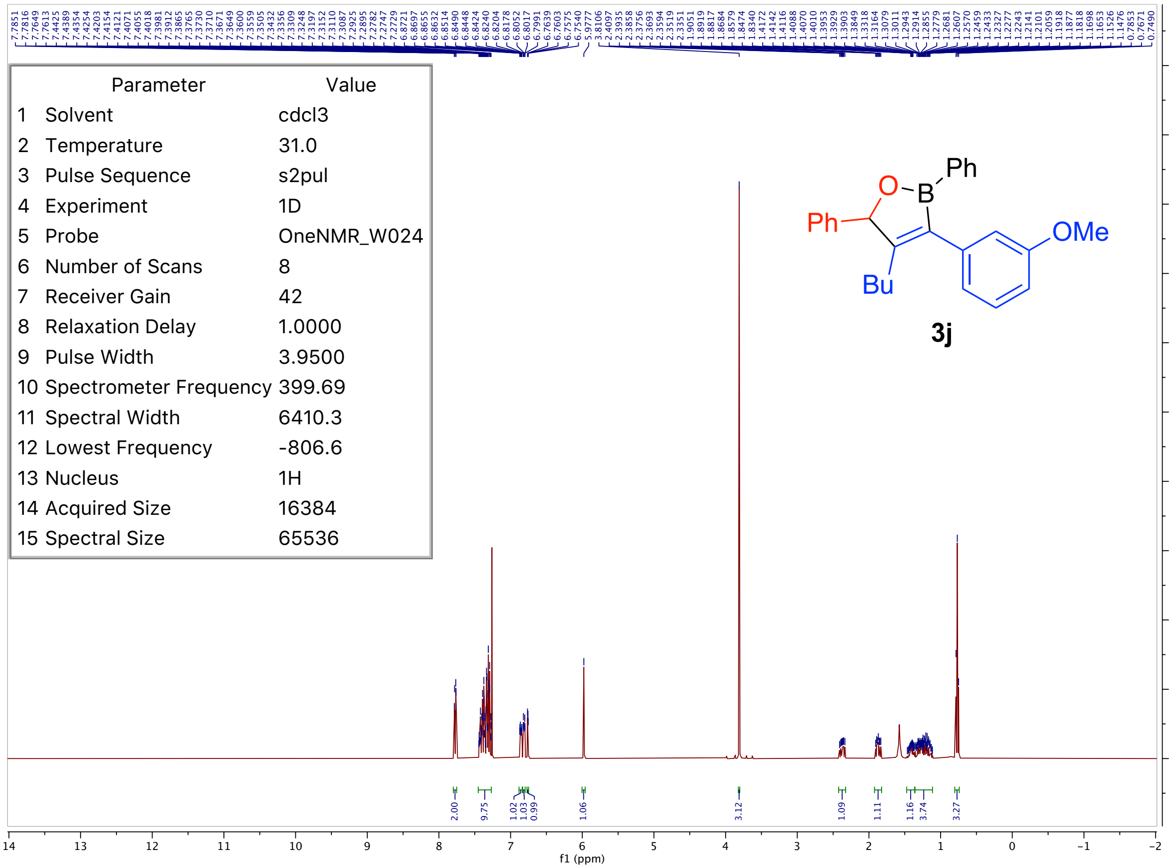
3i

	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	4
7	Receiver Gain	75.7
8	Relaxation Delay	1.0000
9	Pulse Width	12.0000
10	Spectrometer Frequency	400.17
11	Spectral Width	6250.0
12	Lowest Frequency	-724.0
13	Nucleus	1H
14	Acquired Size	32768
15	Spectral Size	65536

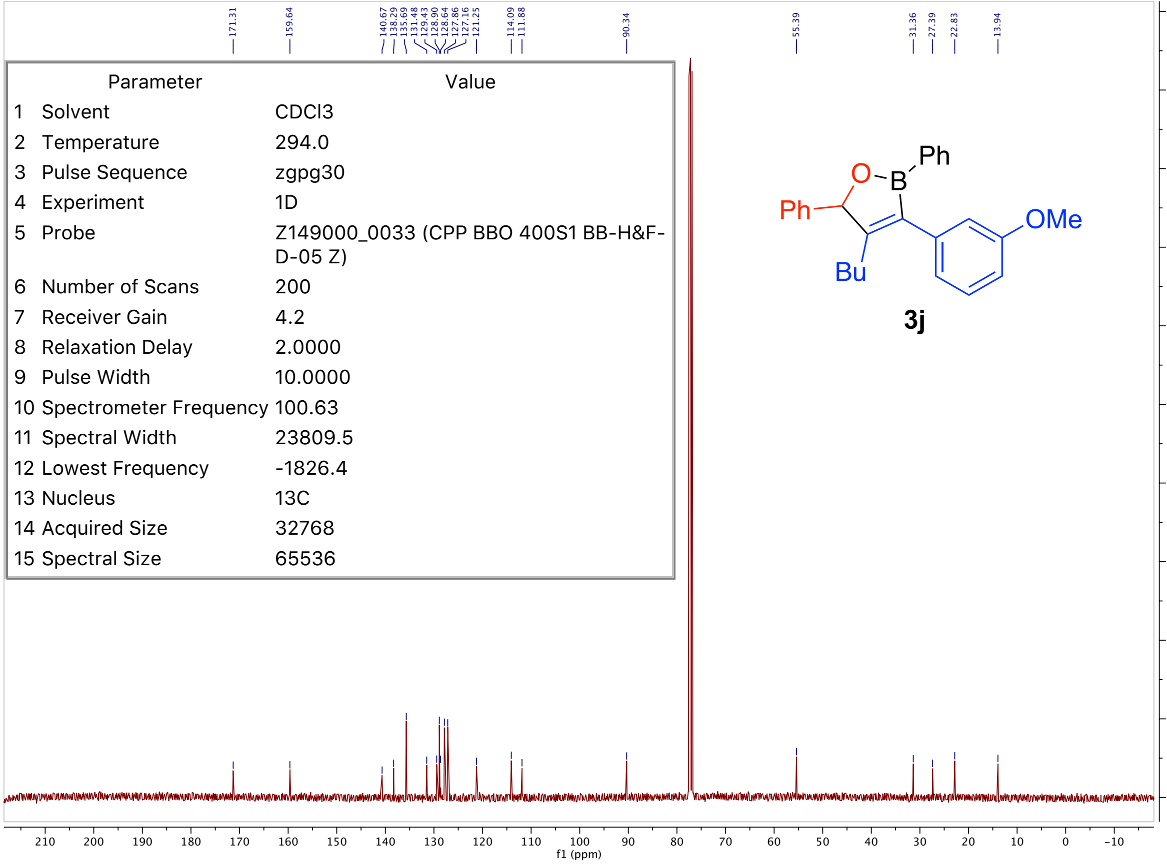


3i

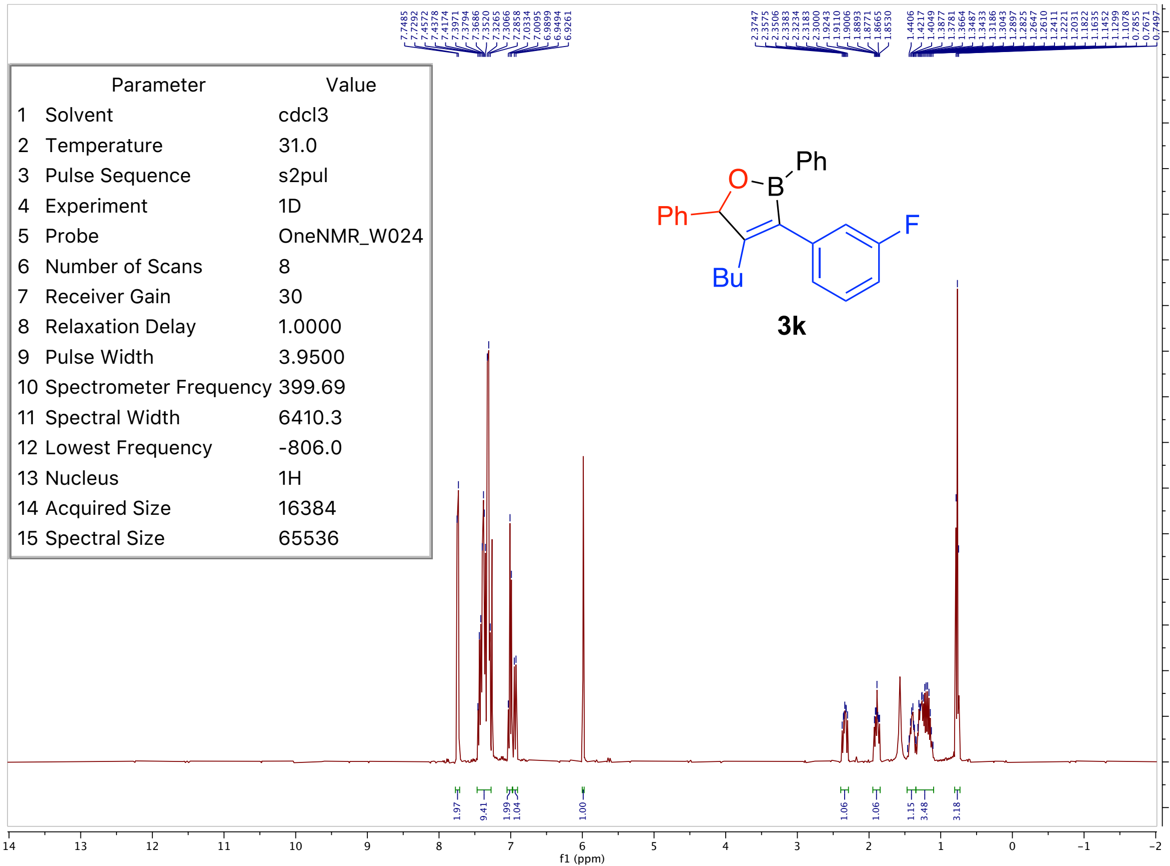
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	101
7	Receiver Gain	4.3
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	42
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.6
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

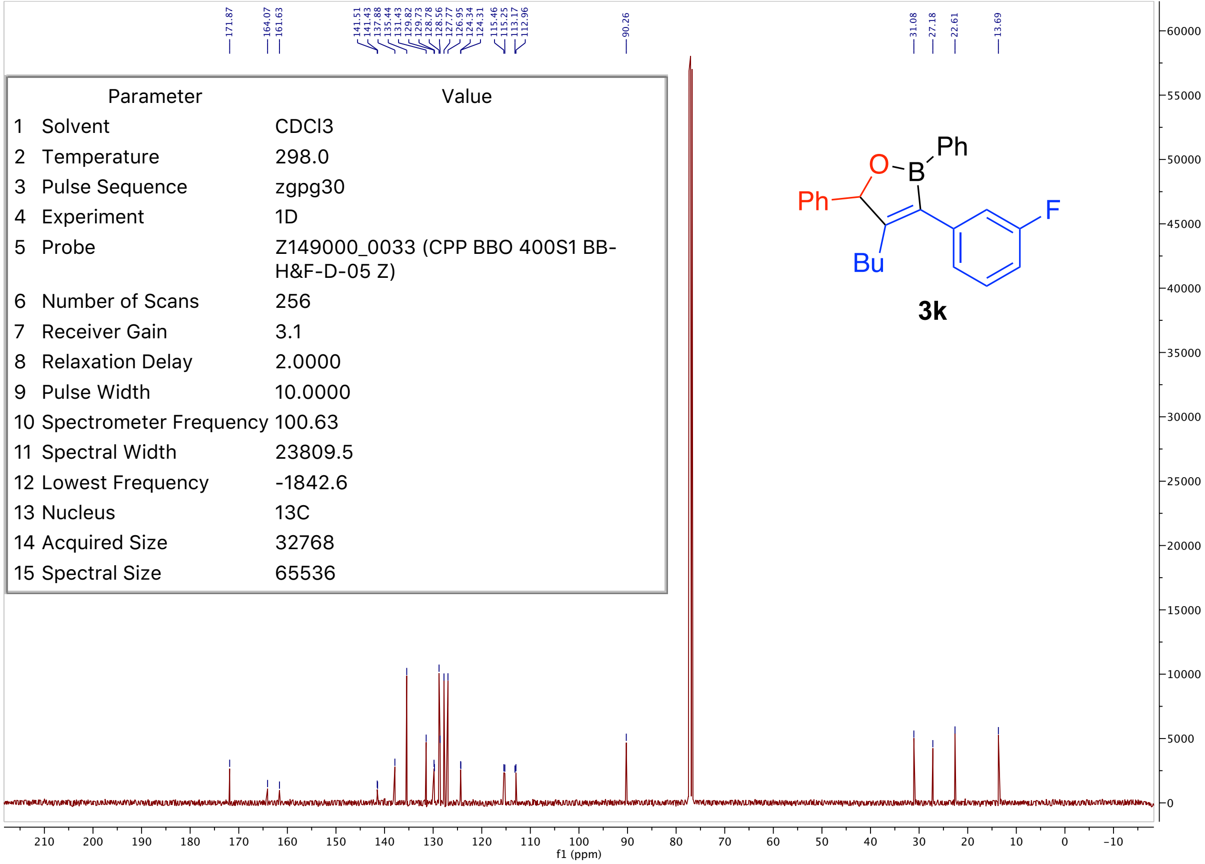


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	294.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	200
7	Receiver Gain	4.2
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1826.4
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



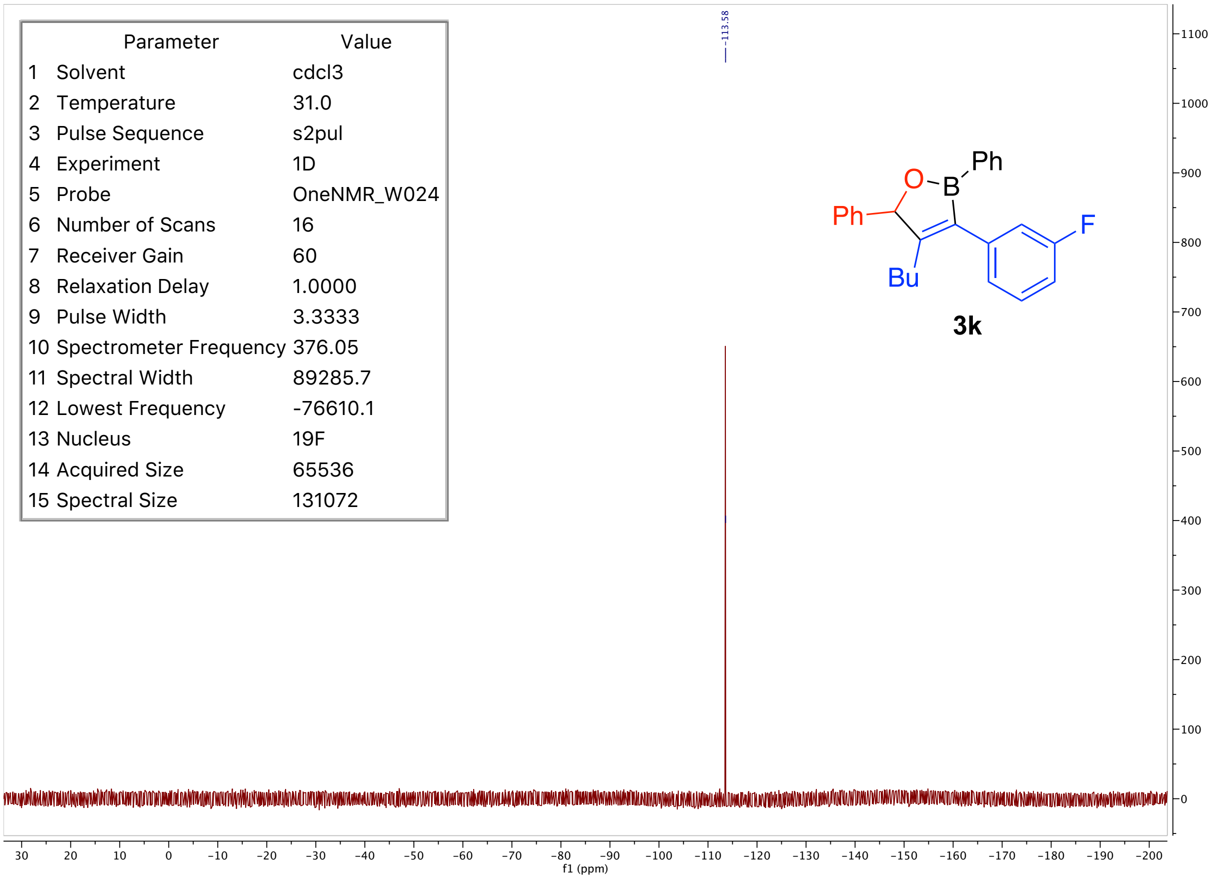
Parameter	Value
1 Solvent	cdcl3
2 Temperature	31.0
3 Pulse Sequence	s2pul
4 Experiment	1D
5 Probe	OneNMR_W024
6 Number of Scans	8
7 Receiver Gain	30
8 Relaxation Delay	1.0000
9 Pulse Width	3.9500
10 Spectrometer Frequency	399.69
11 Spectral Width	6410.3
12 Lowest Frequency	-806.0
13 Nucleus	1H
14 Acquired Size	16384
15 Spectral Size	65536

3k



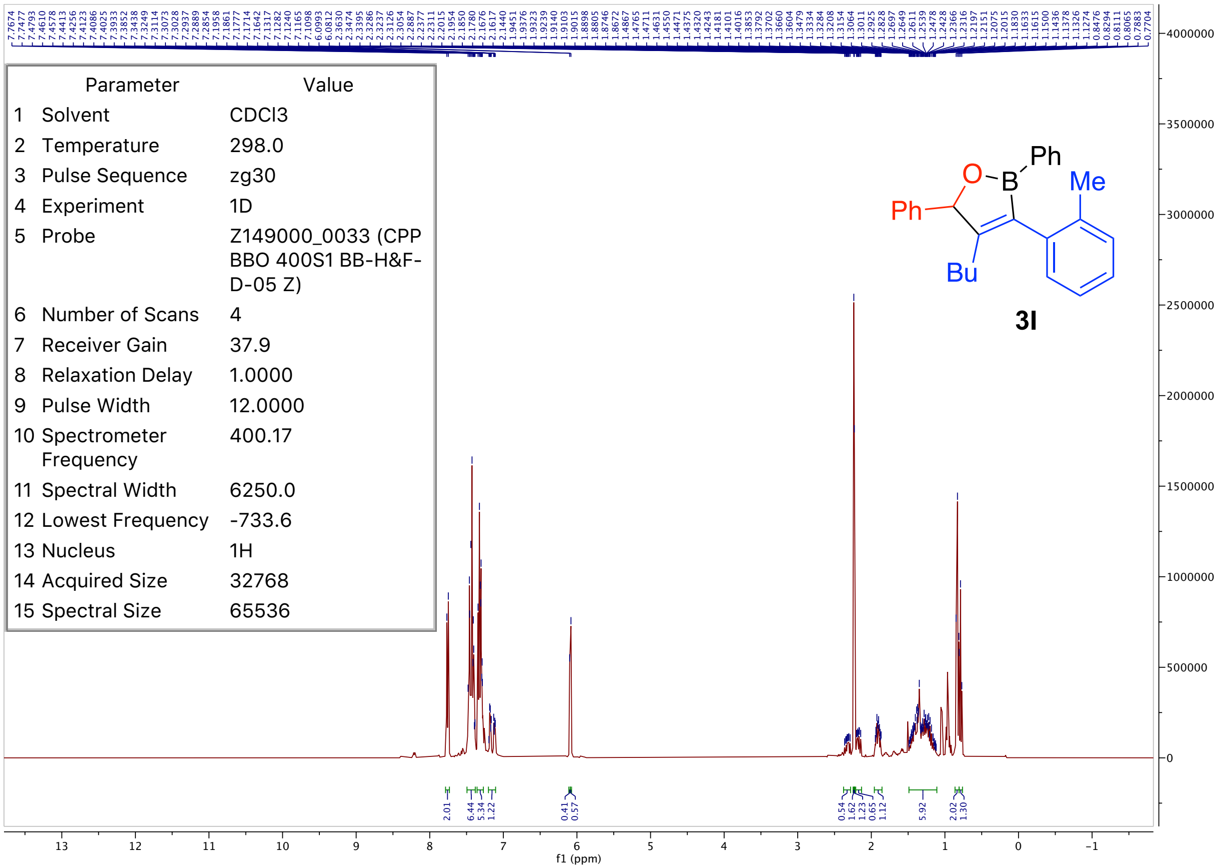
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Pulse Sequence	zgpg30
4 Experiment	1D
5 Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans	256
7 Receiver Gain	3.1
8 Relaxation Delay	2.0000
9 Pulse Width	10.0000
10 Spectrometer Frequency	100.63
11 Spectral Width	23809.5
12 Lowest Frequency	-1842.6
13 Nucleus	13C
14 Acquired Size	32768
15 Spectral Size	65536

3k

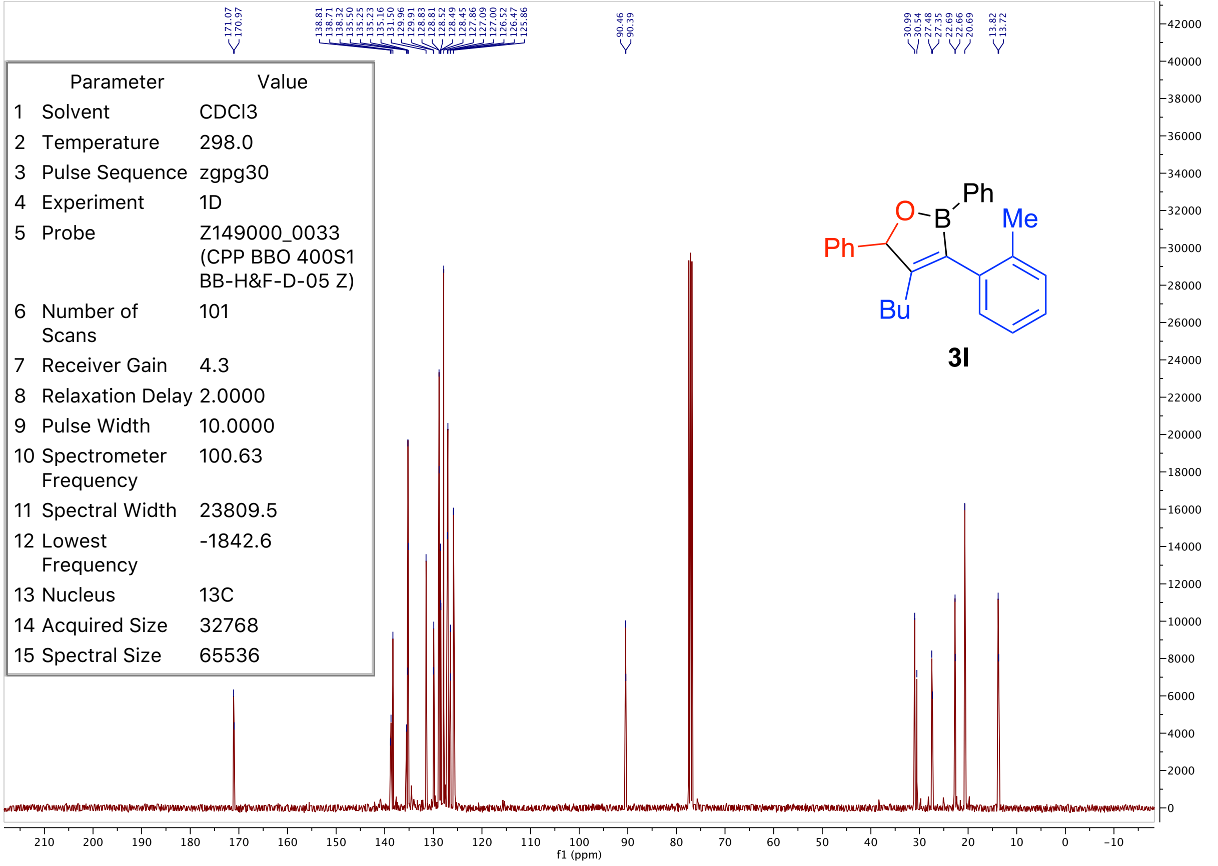


	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	16
7	Receiver Gain	60
8	Relaxation Delay	1.0000
9	Pulse Width	3.3333
10	Spectrometer Frequency	376.05
11	Spectral Width	89285.7
12	Lowest Frequency	-76610.1
13	Nucleus	19F
14	Acquired Size	65536
15	Spectral Size	131072

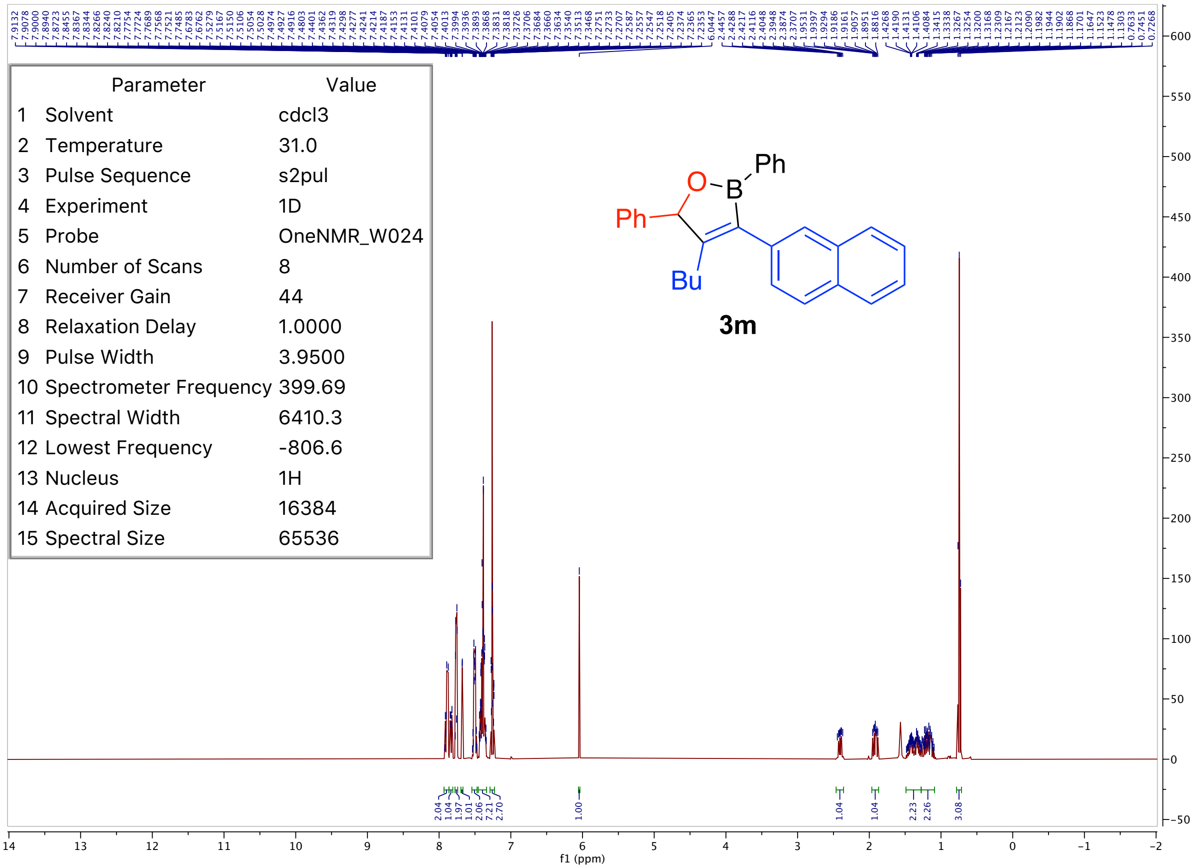
3k



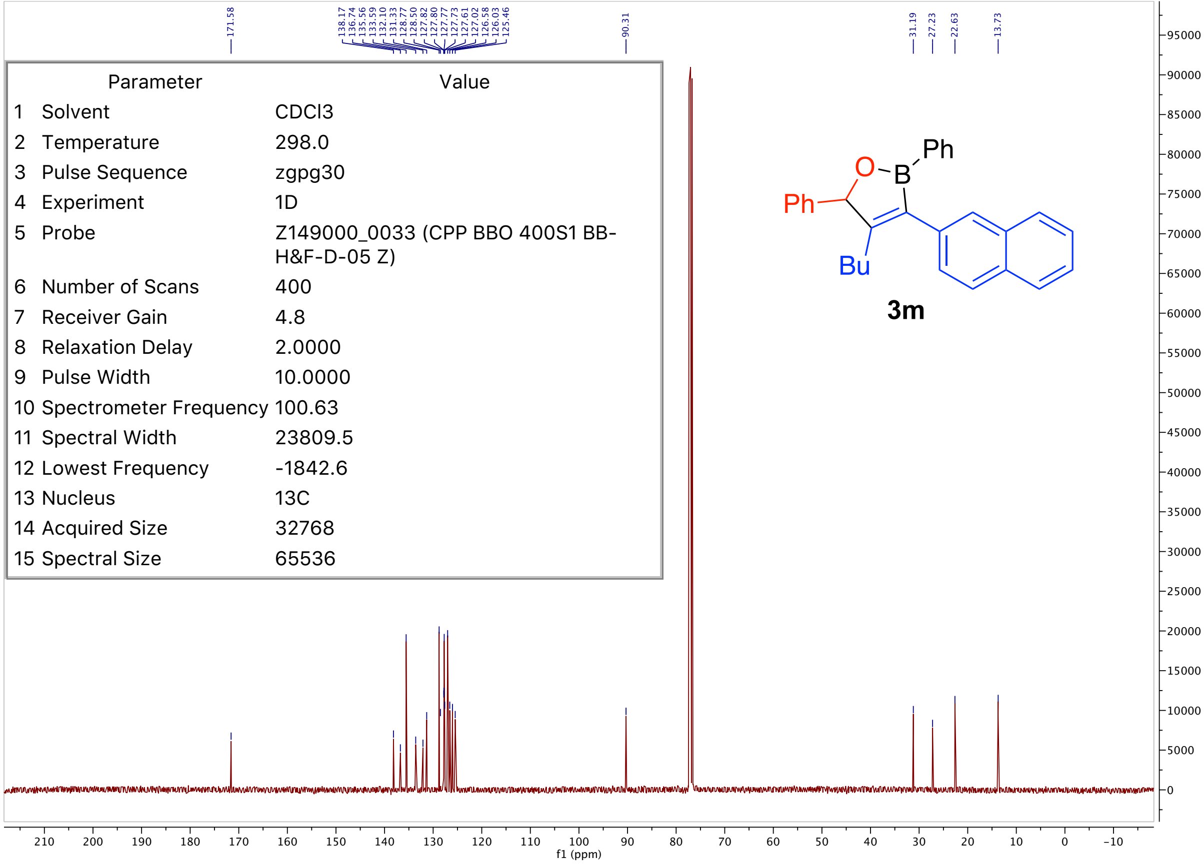
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	4
7	Receiver Gain	37.9
8	Relaxation Delay	1.0000
9	Pulse Width	12.0000
10	Spectrometer Frequency	400.17
11	Spectral Width	6250.0
12	Lowest Frequency	-733.6
13	Nucleus	1H
14	Acquired Size	32768
15	Spectral Size	65536



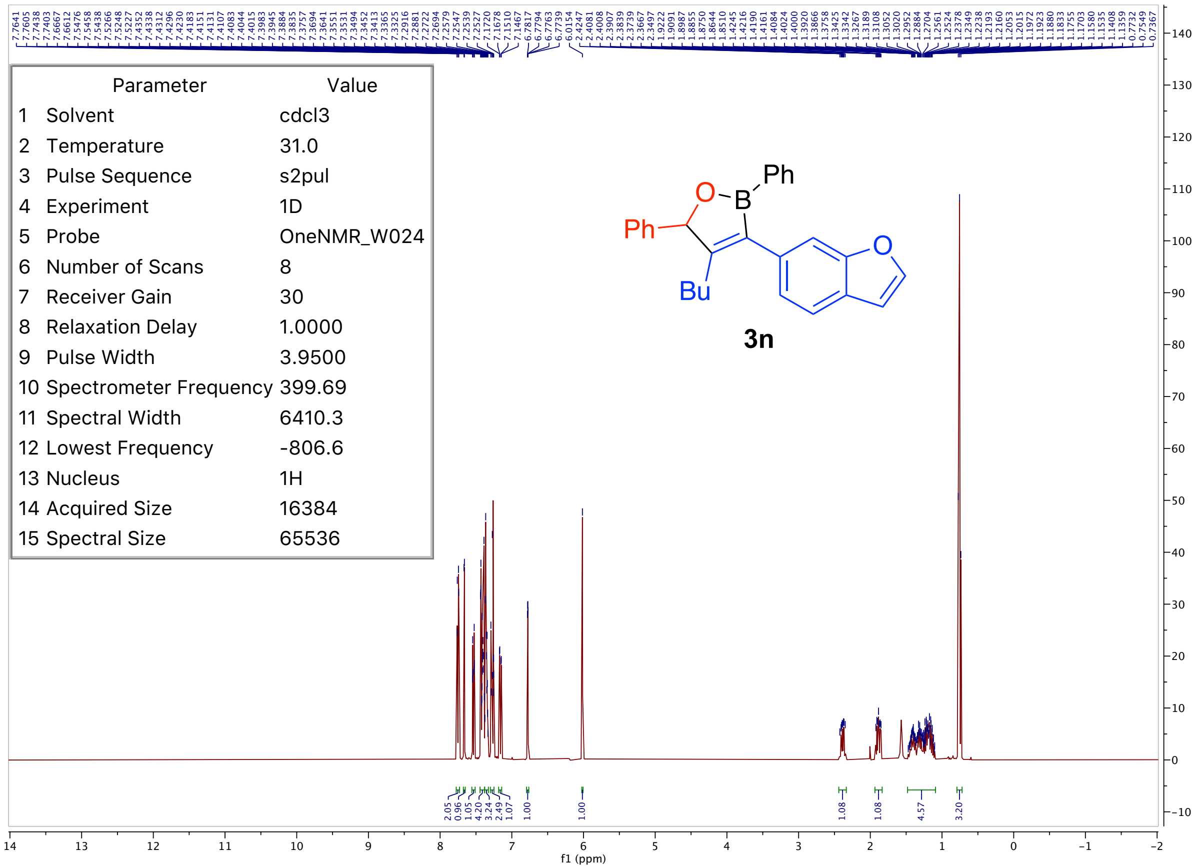
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	101
7	Receiver Gain	4.3
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



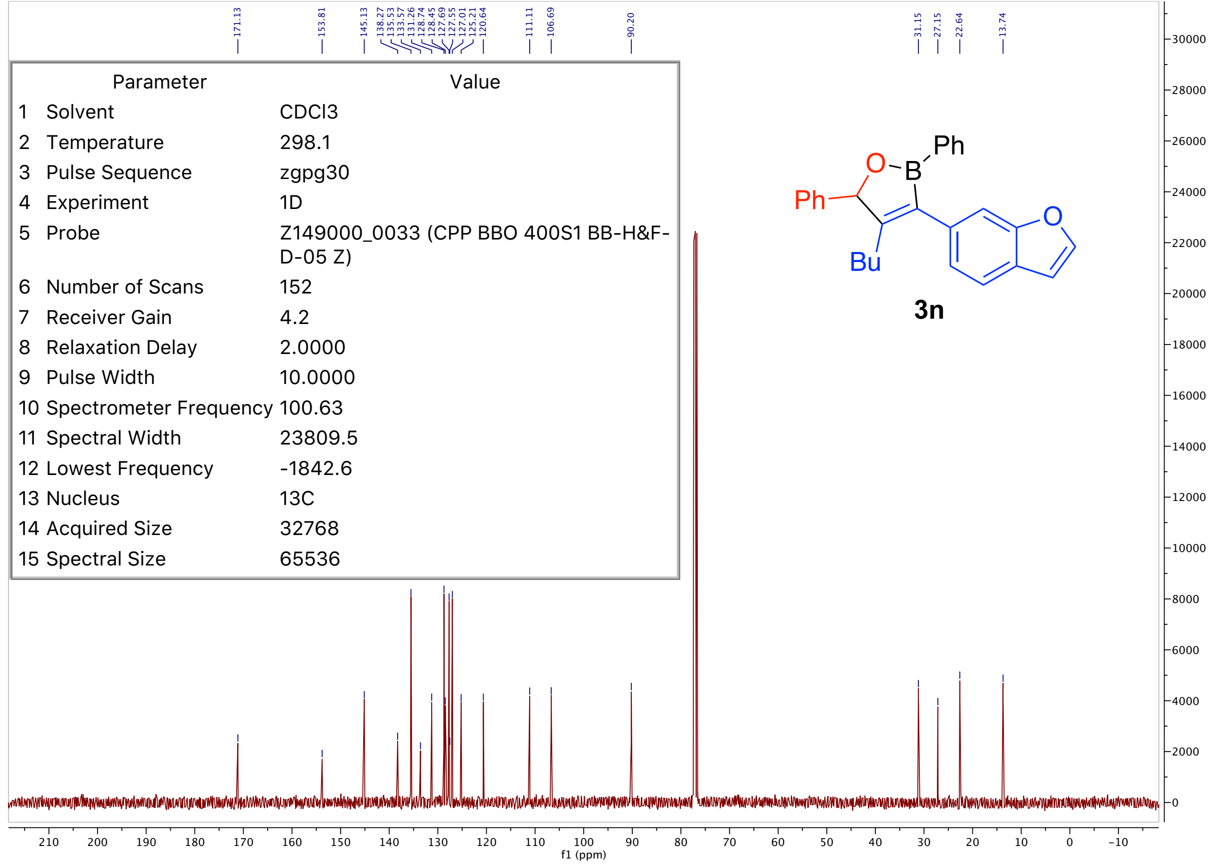
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	44
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.6
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



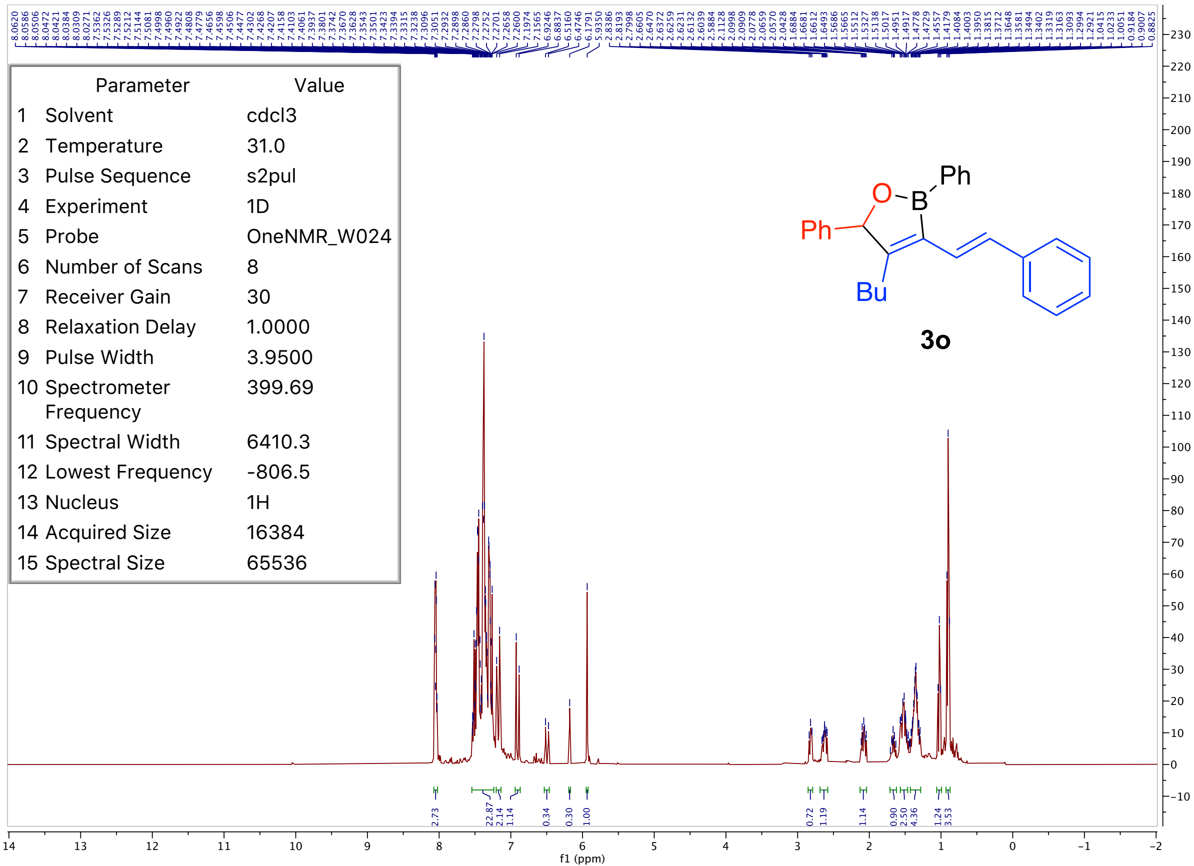
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	400
7	Receiver Gain	4.8
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



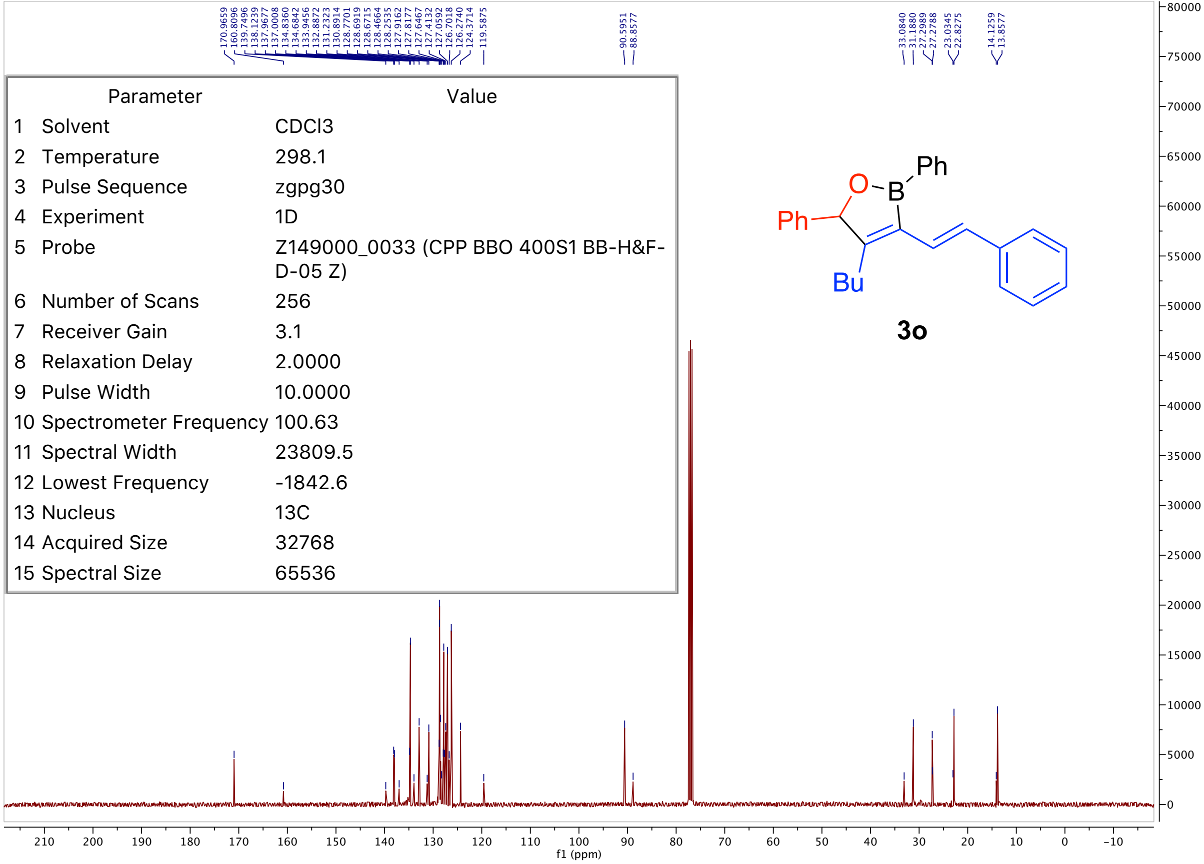
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	30
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.6
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



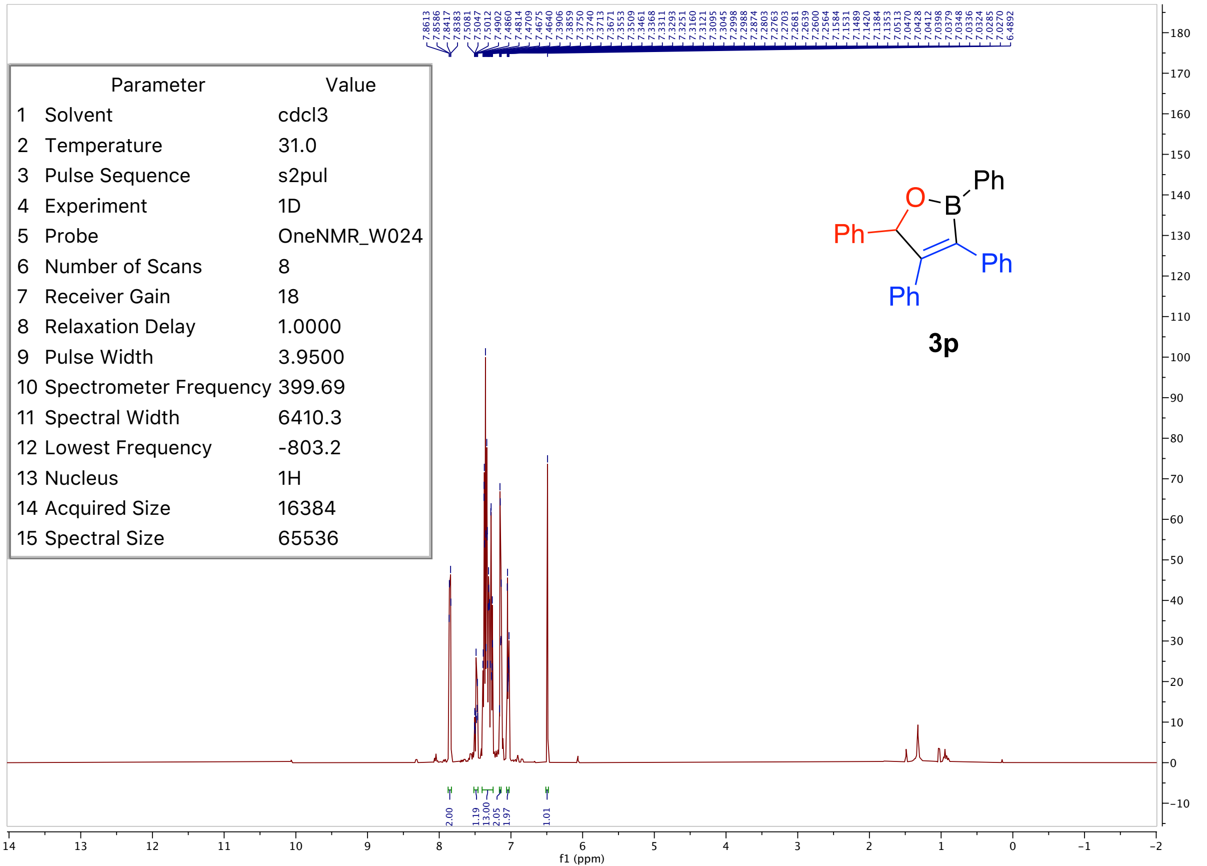
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.1
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	152
7	Receiver Gain	4.2
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	30
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.5
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

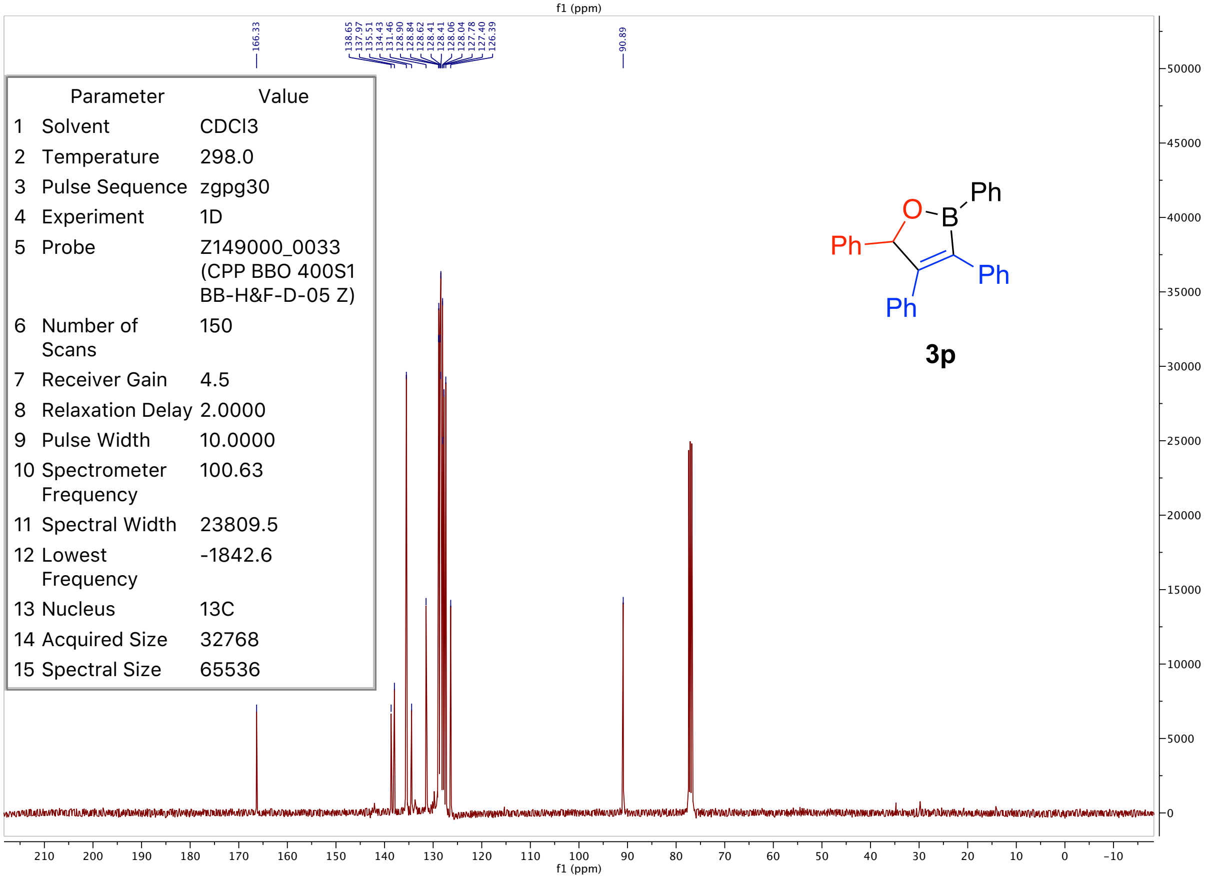


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.1
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	256
7	Receiver Gain	3.1
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



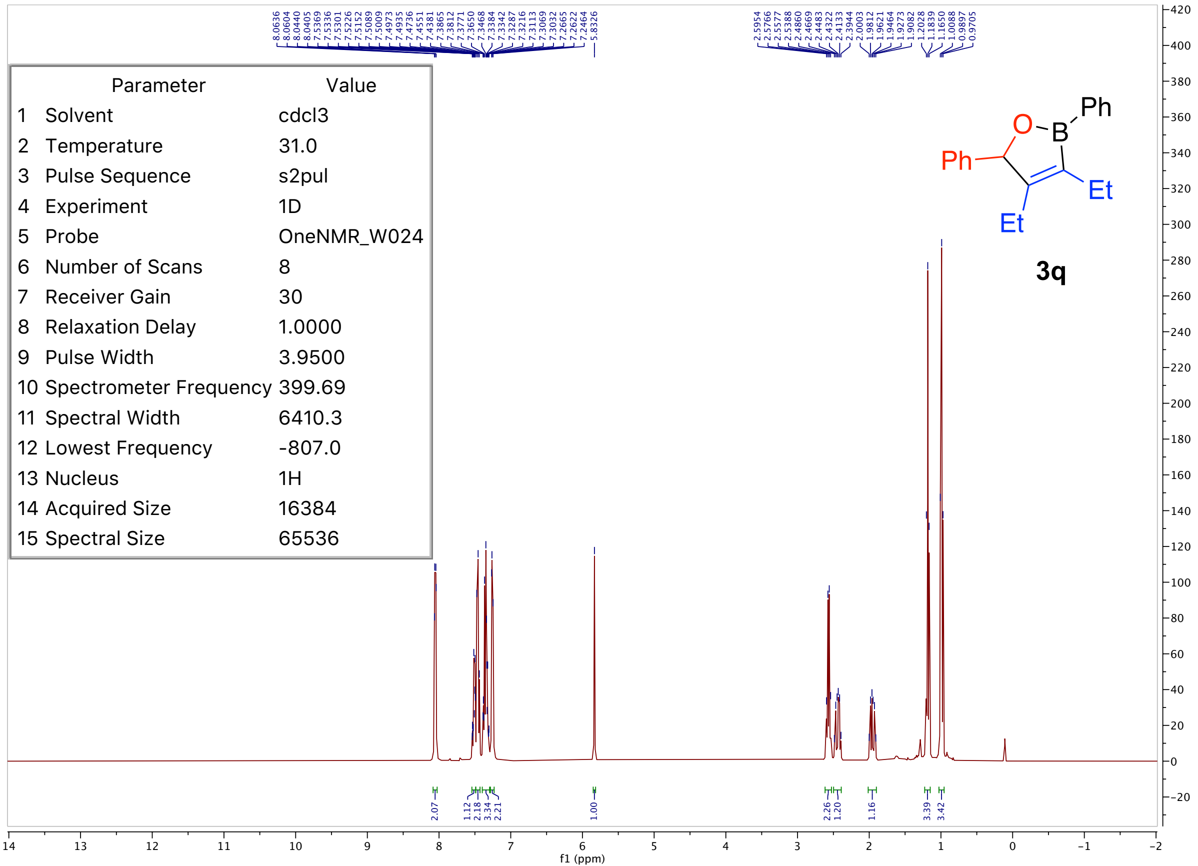
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-803.2
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

3p

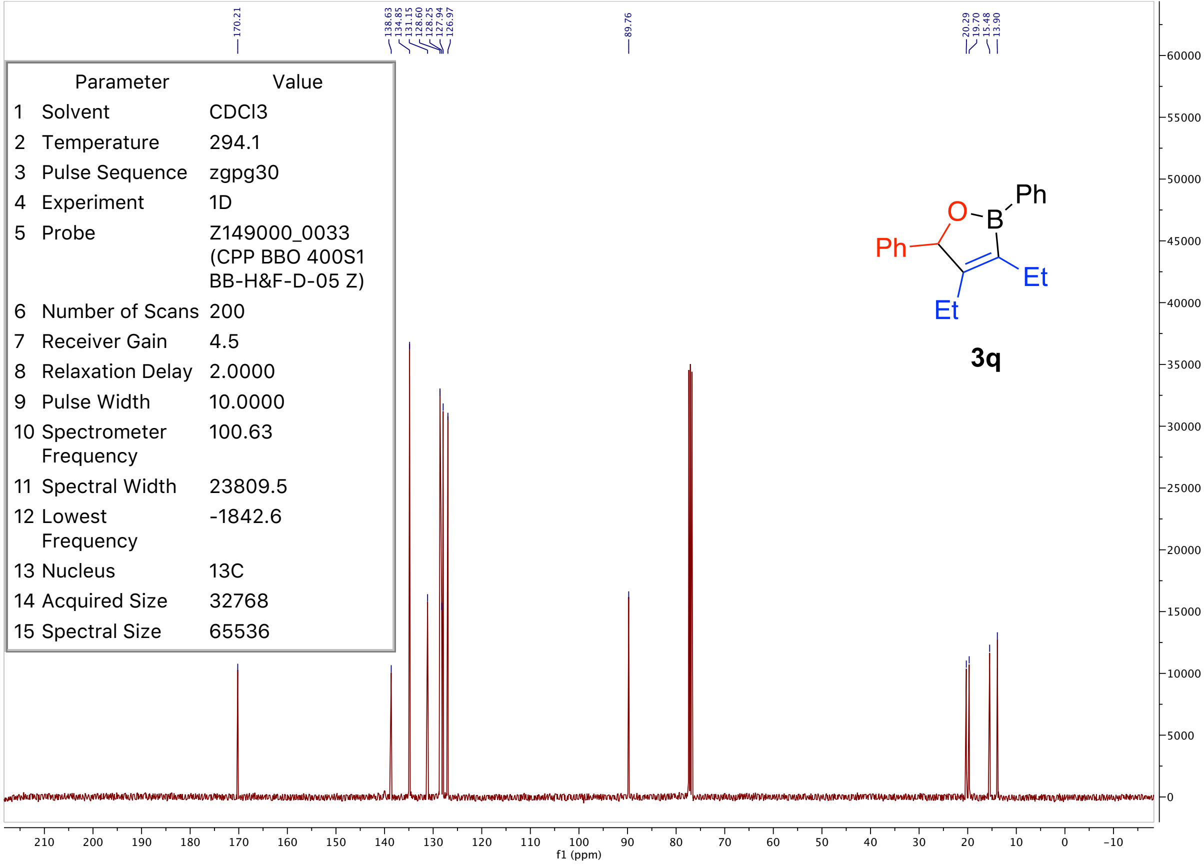


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	150
7	Receiver Gain	4.5
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536

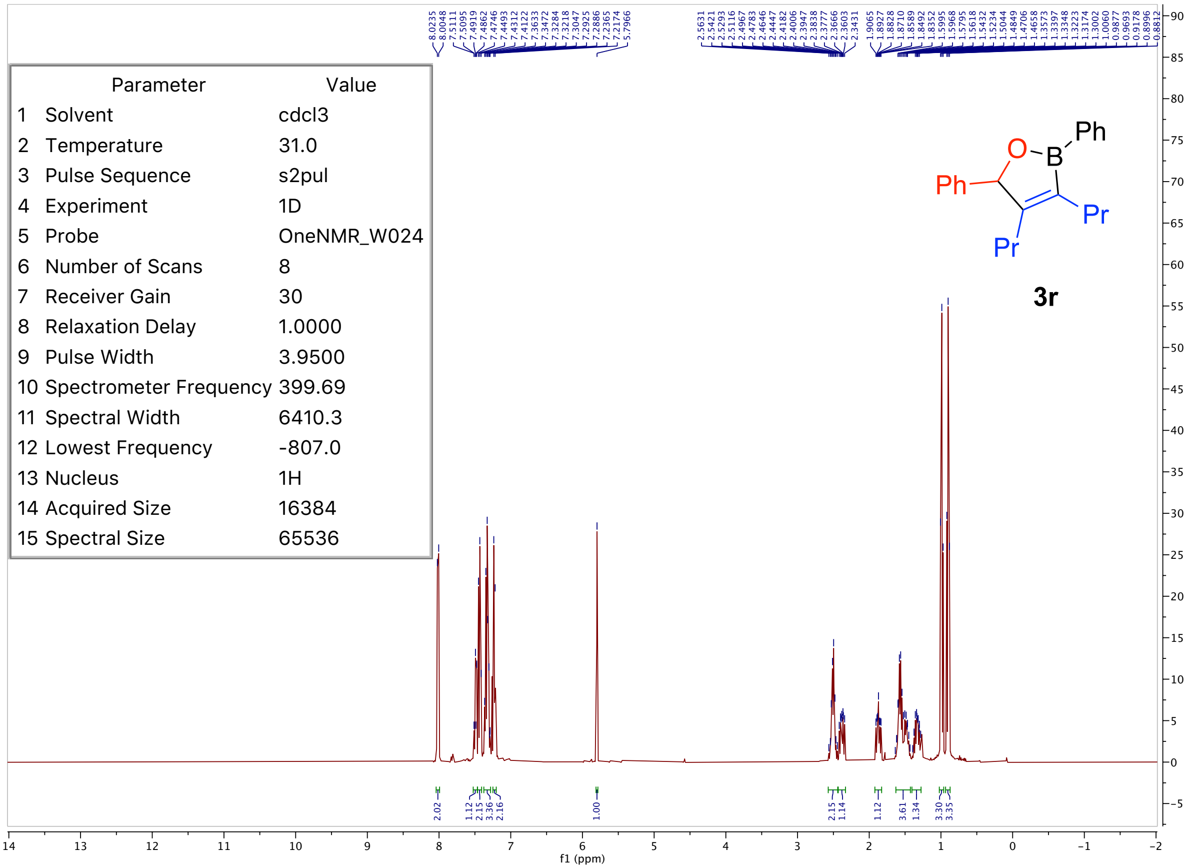
3p



	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	30
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-807.0
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

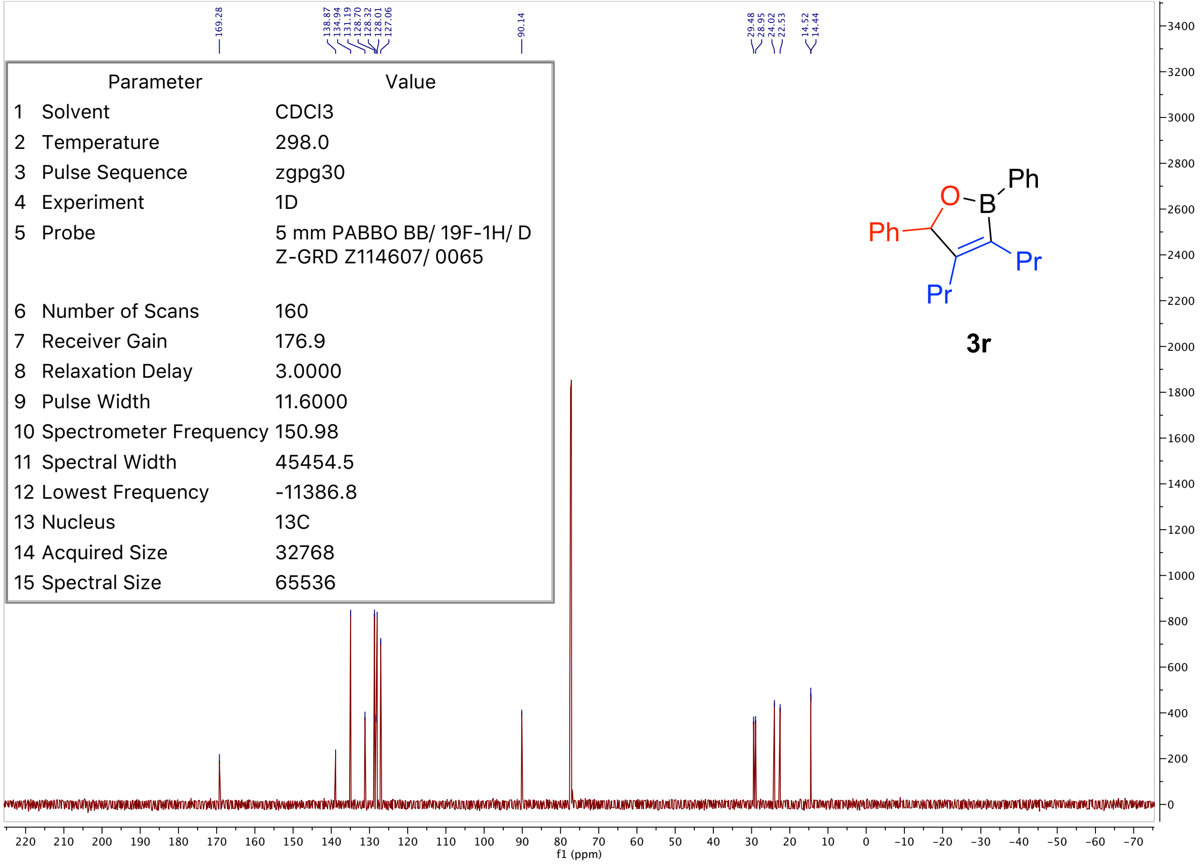


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	294.1
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	200
7	Receiver Gain	4.5
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



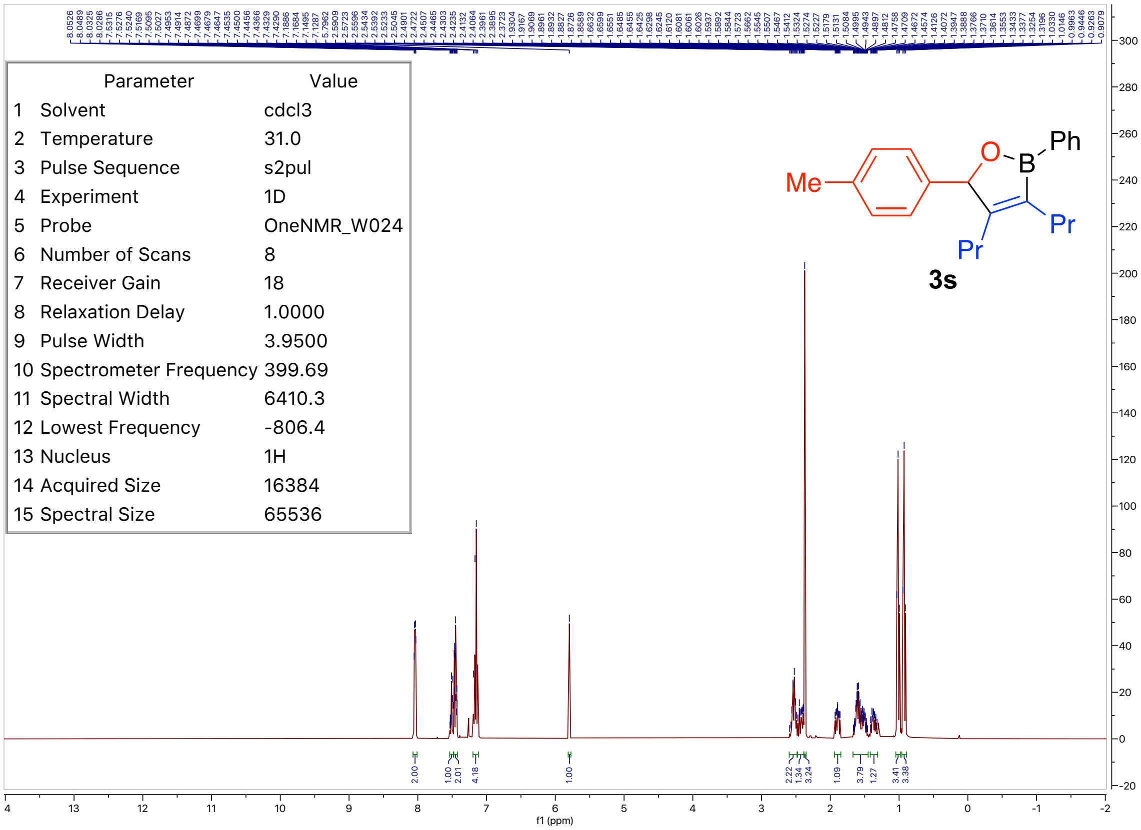
3r

Parameter	Value
1 Solvent	cdcl3
2 Temperature	31.0
3 Pulse Sequence	s2pul
4 Experiment	1D
5 Probe	OneNMR_W024
6 Number of Scans	8
7 Receiver Gain	30
8 Relaxation Delay	1.0000
9 Pulse Width	3.9500
10 Spectrometer Frequency	399.69
11 Spectral Width	6410.3
12 Lowest Frequency	-807.0
13 Nucleus	1H
14 Acquired Size	16384
15 Spectral Size	65536



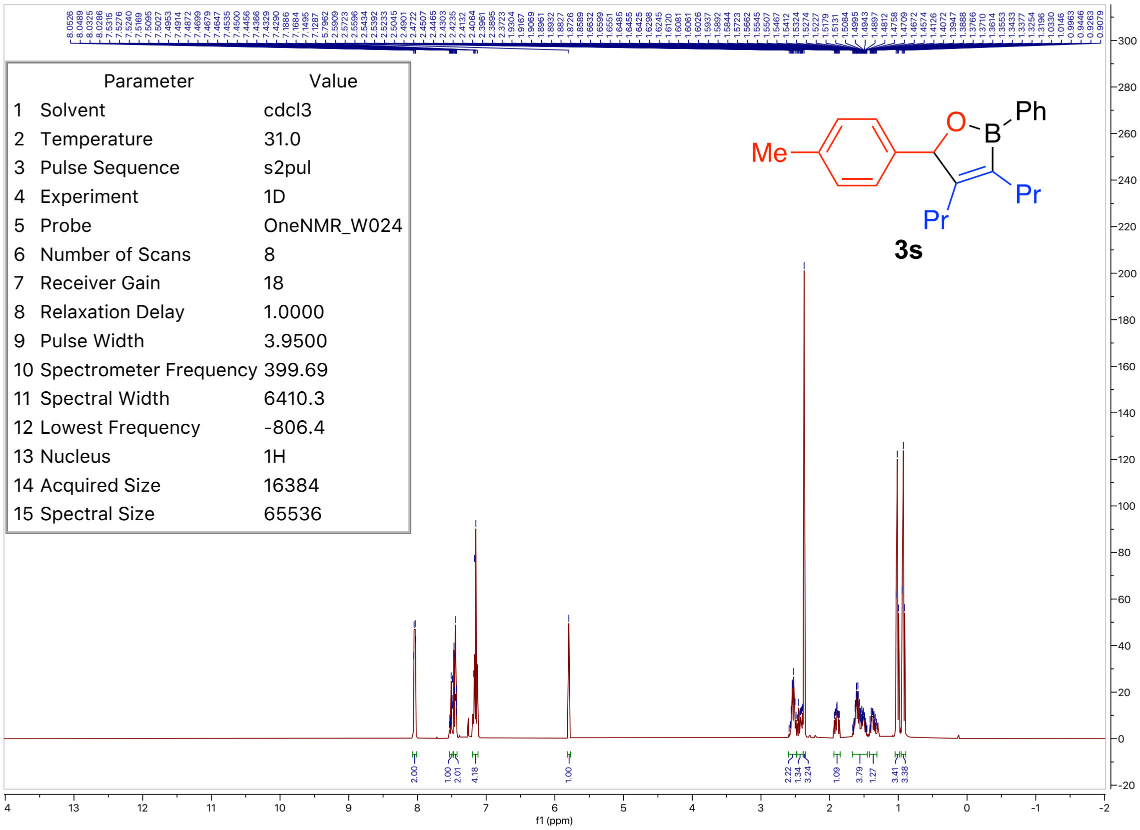
3r

Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Pulse Sequence	zgpg30
4 Experiment	1D
5 Probe	5 mm PABBO BB/ 19F-1H/ D Z-GRD Z114607/ 0065
6 Number of Scans	160
7 Receiver Gain	176.9
8 Relaxation Delay	3.0000
9 Pulse Width	11.6000
10 Spectrometer Frequency	150.98
11 Spectral Width	45454.5
12 Lowest Frequency	-11386.8
13 Nucleus	13C
14 Acquired Size	32768
15 Spectral Size	65536



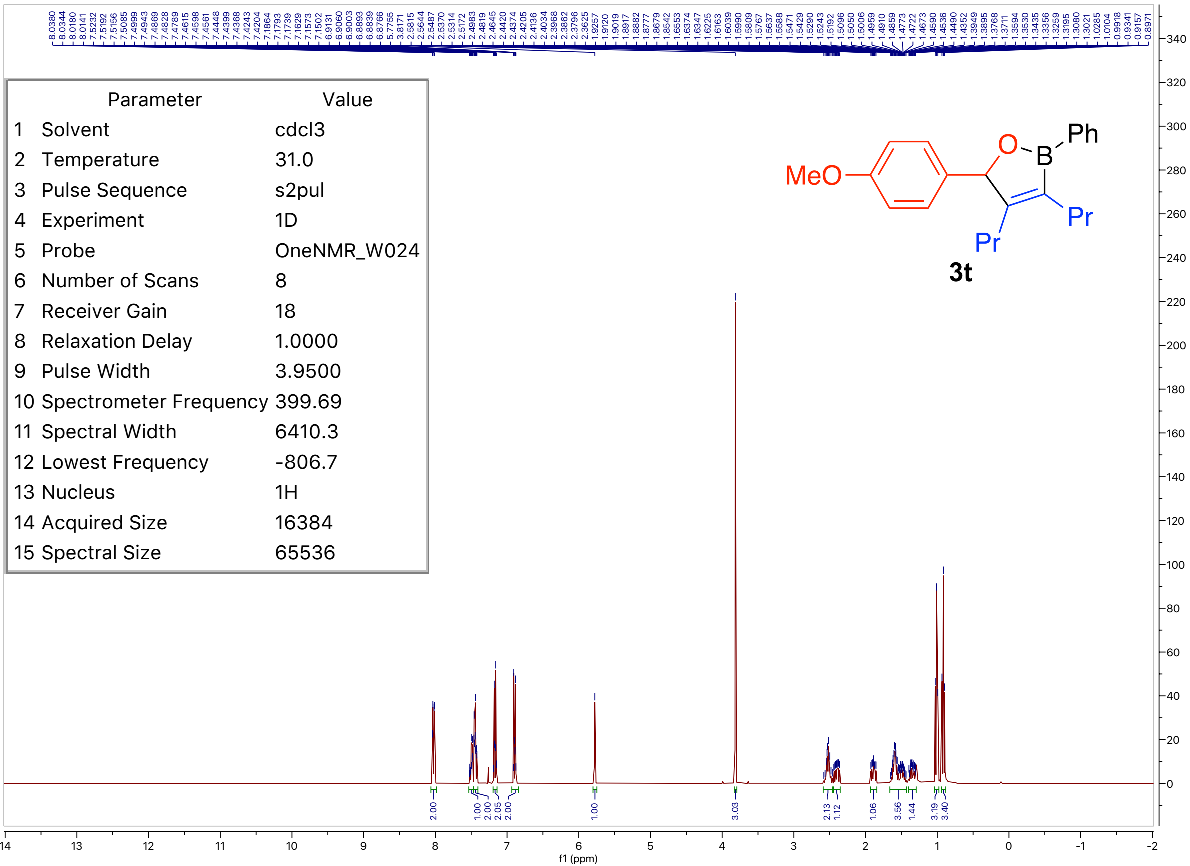
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.4
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

3s

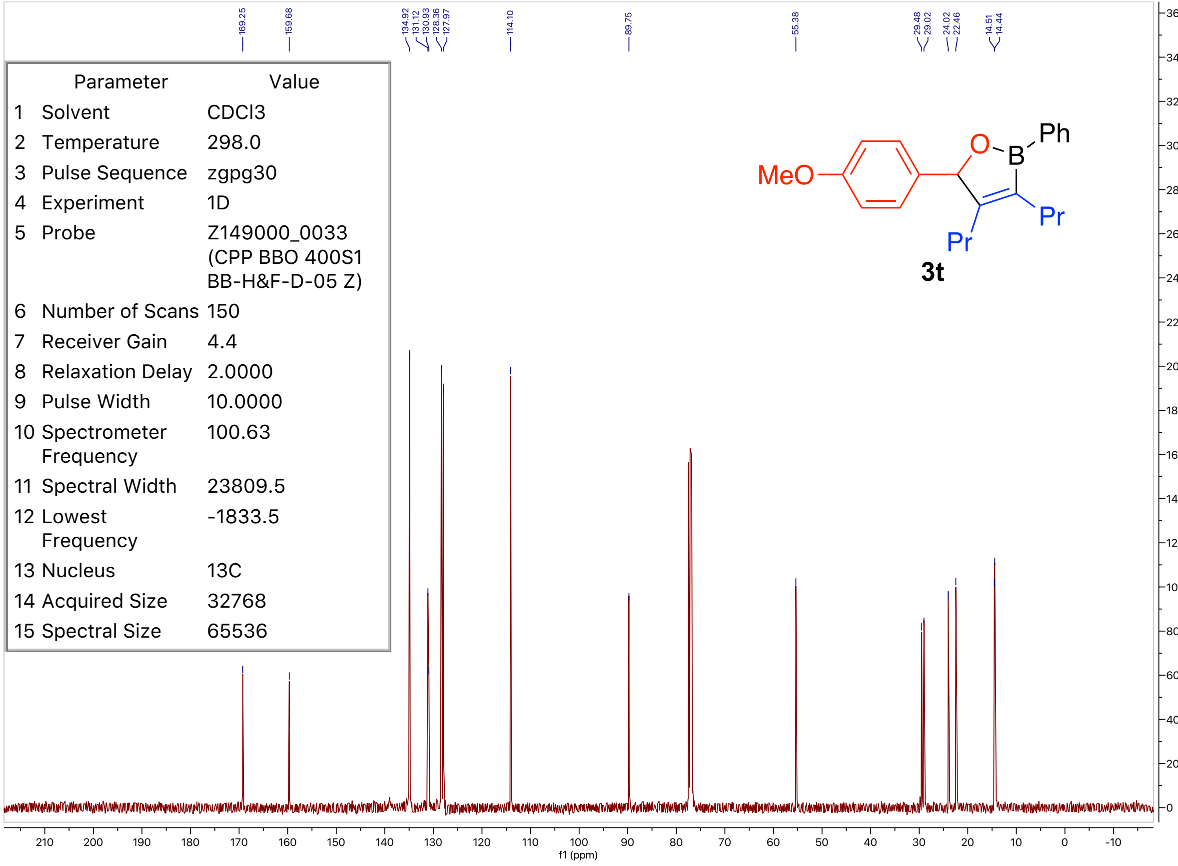


	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.4
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

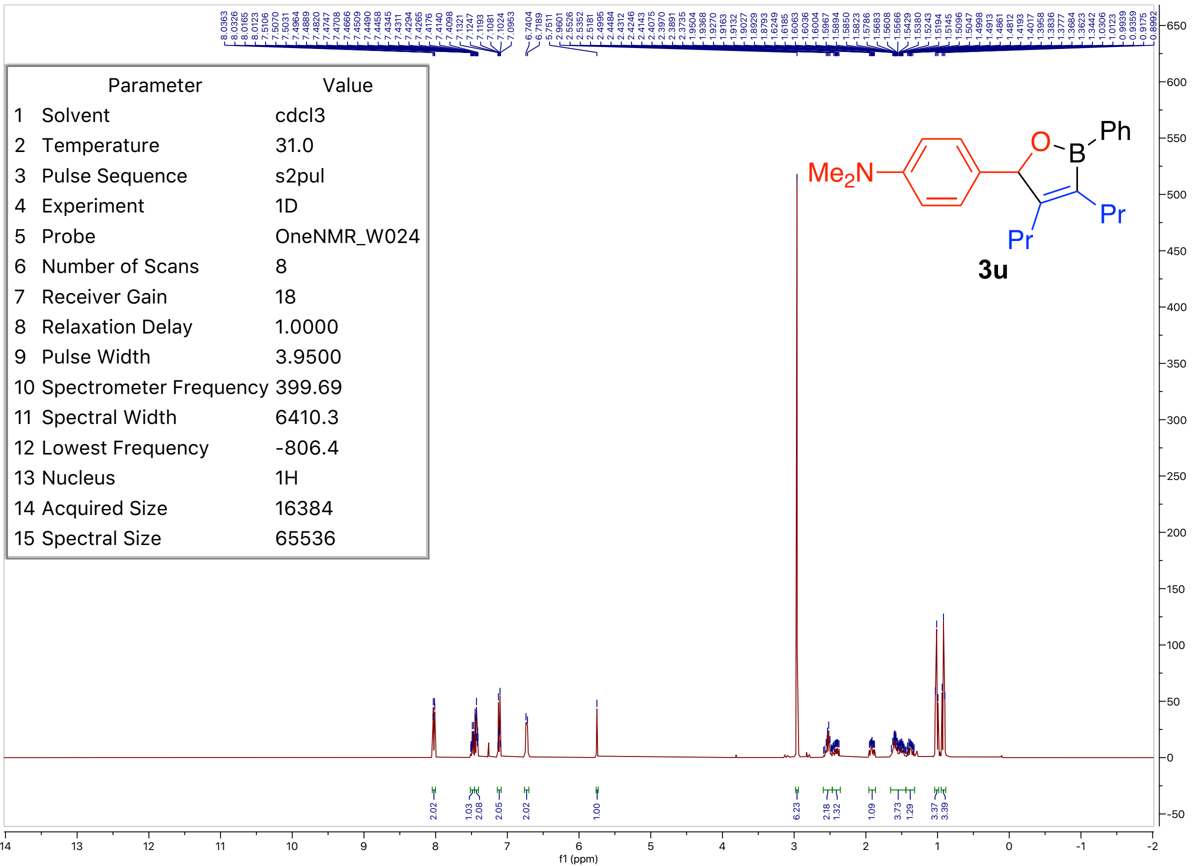
3s



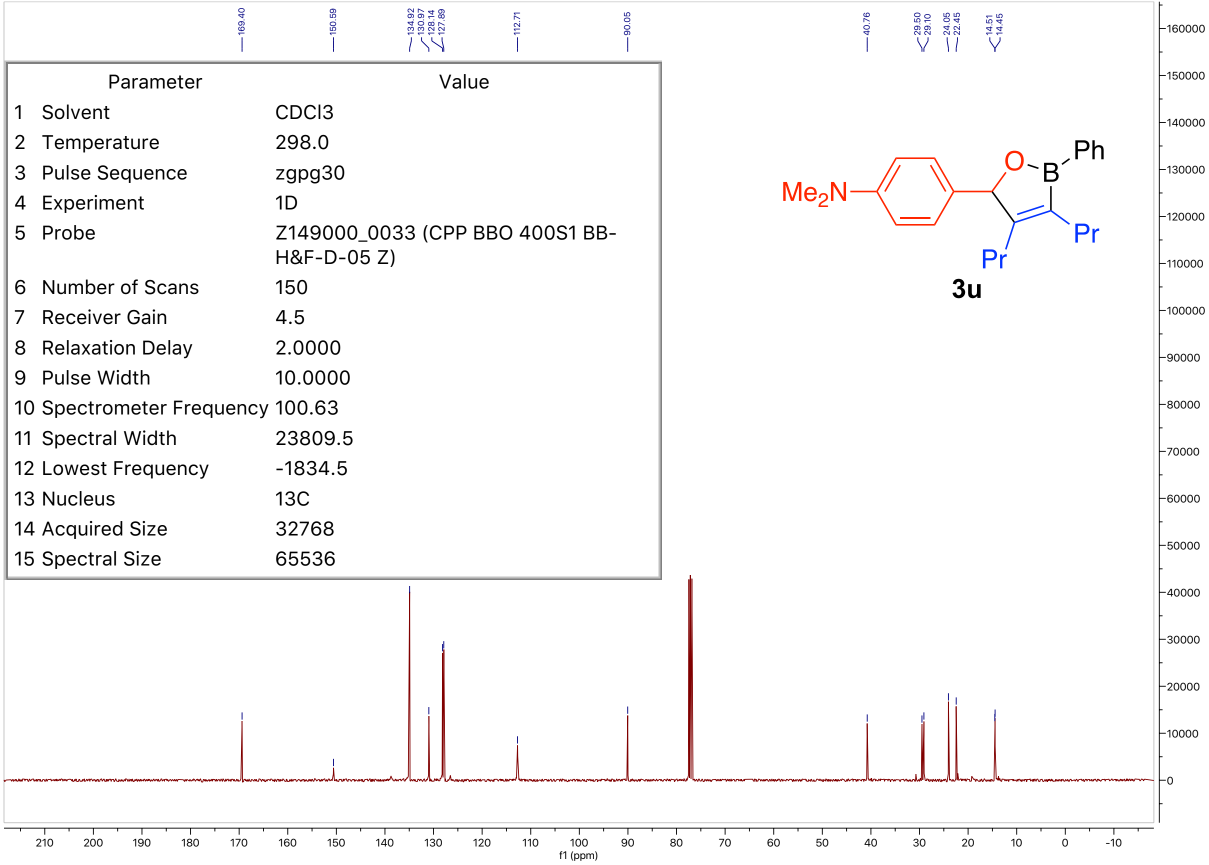
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.7
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



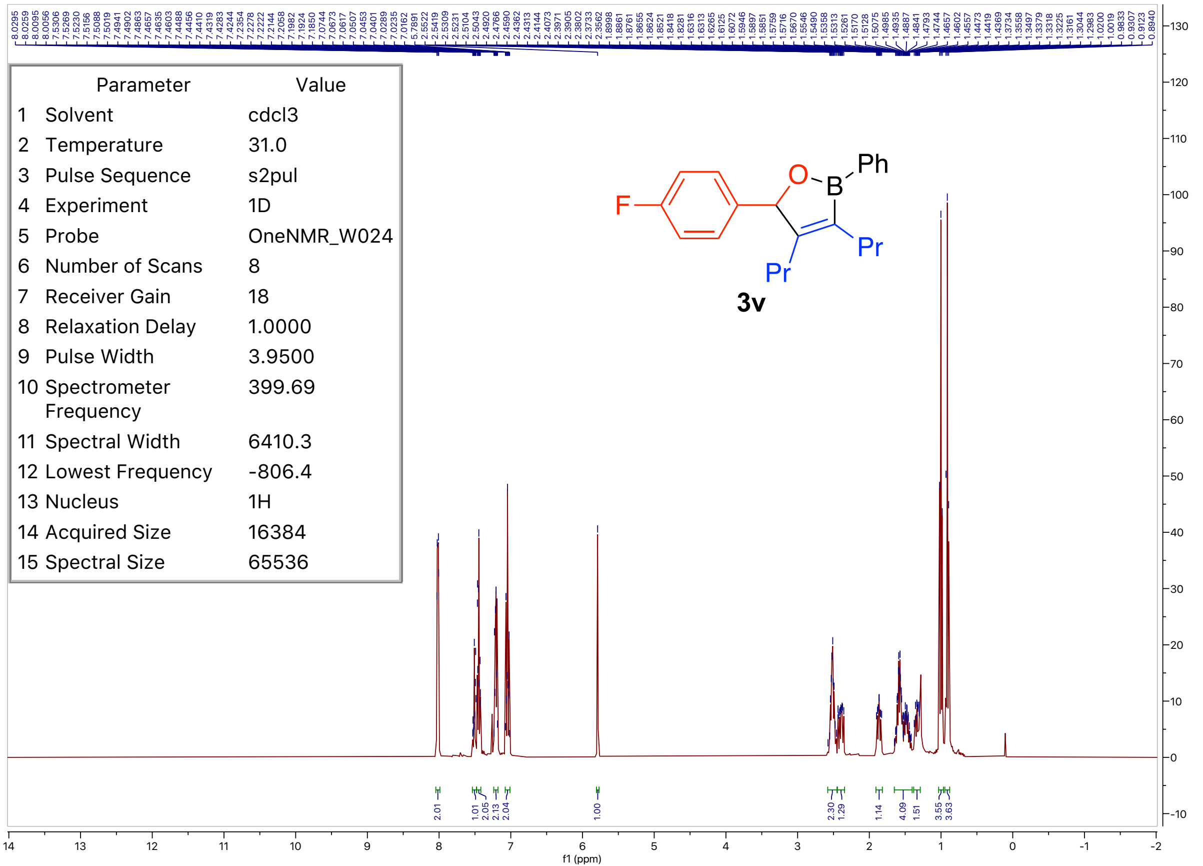
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	150
7	Receiver Gain	4.4
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1833.5
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



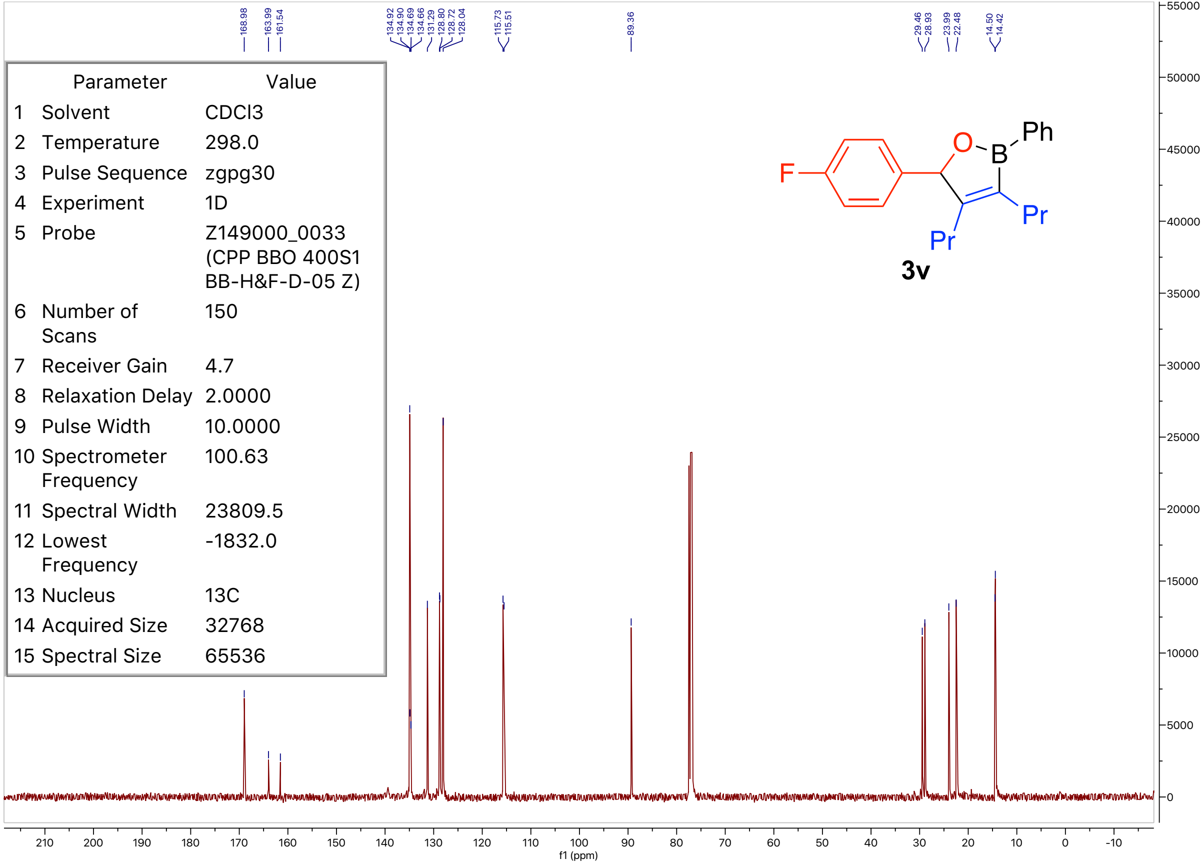
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.4
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



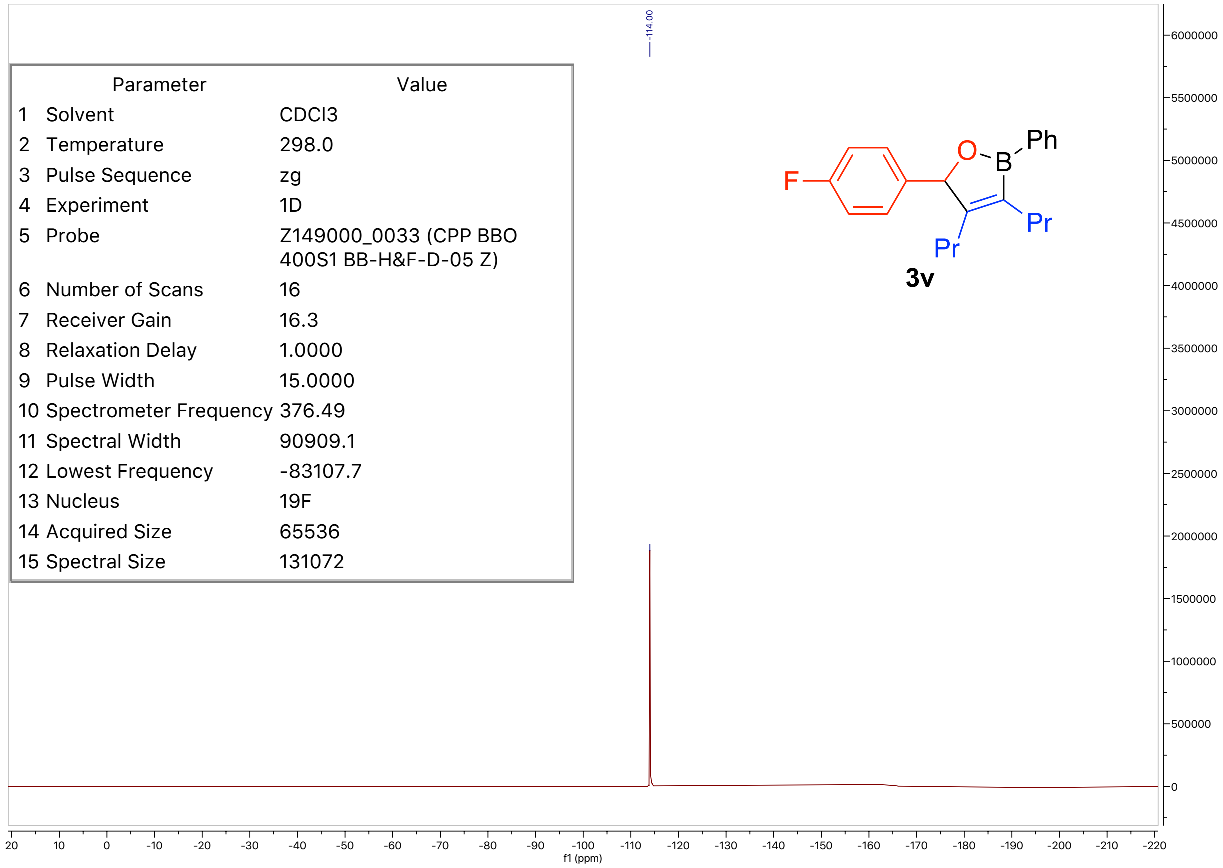
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	150
7	Receiver Gain	4.5
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1834.5
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.4
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

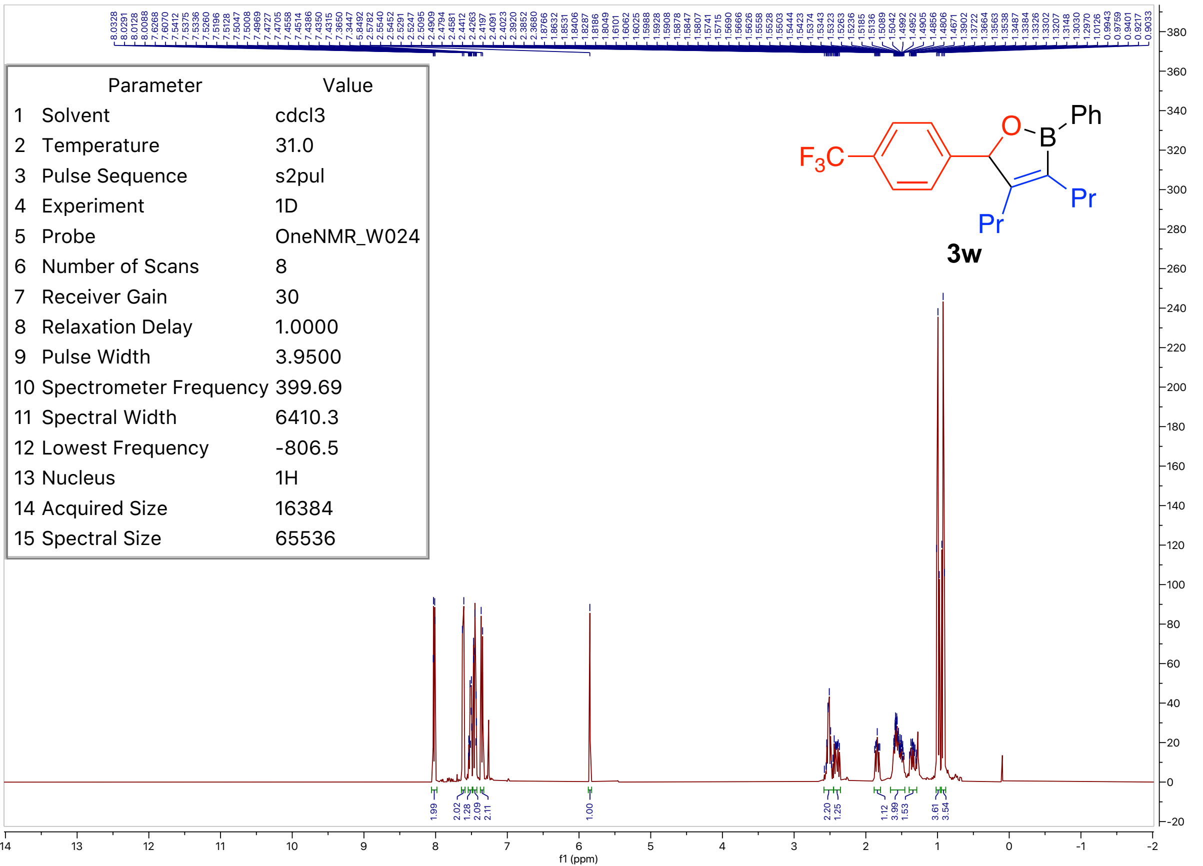


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	150
7	Receiver Gain	4.7
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1832.0
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536

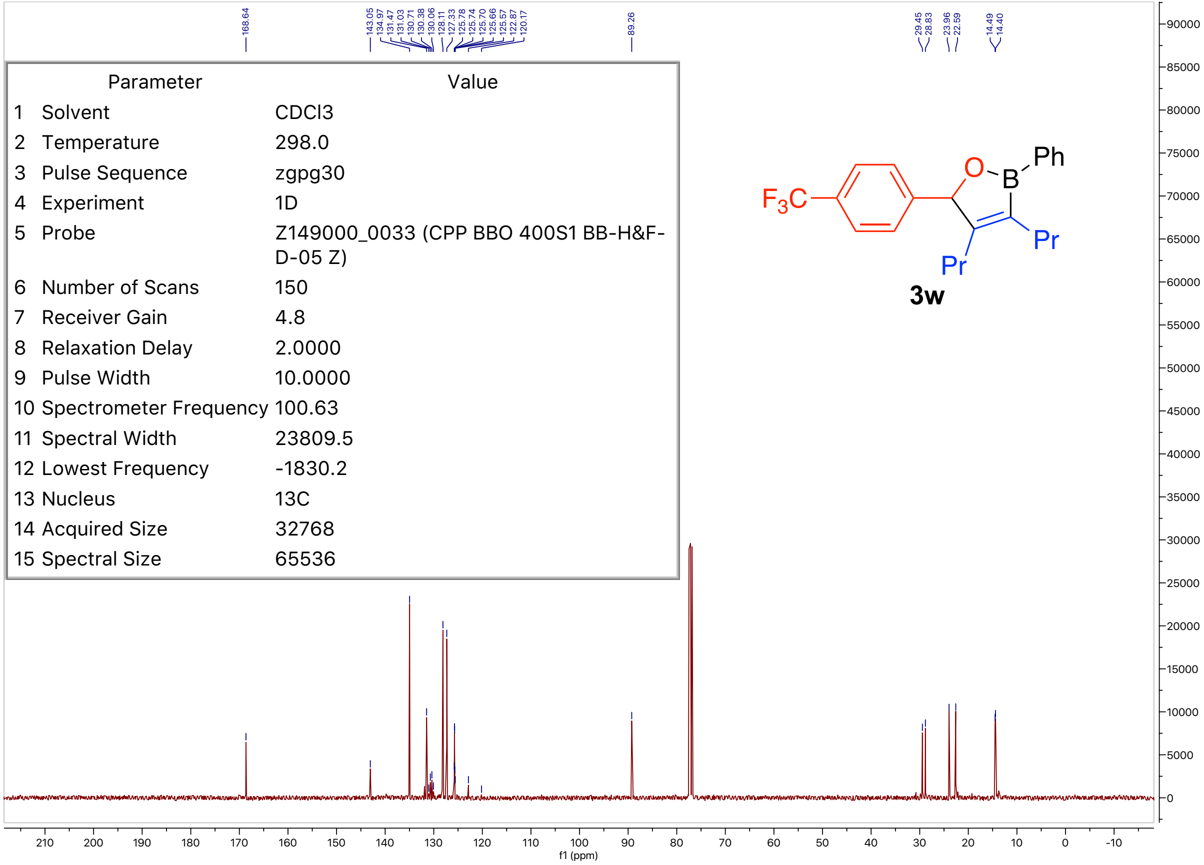


3v

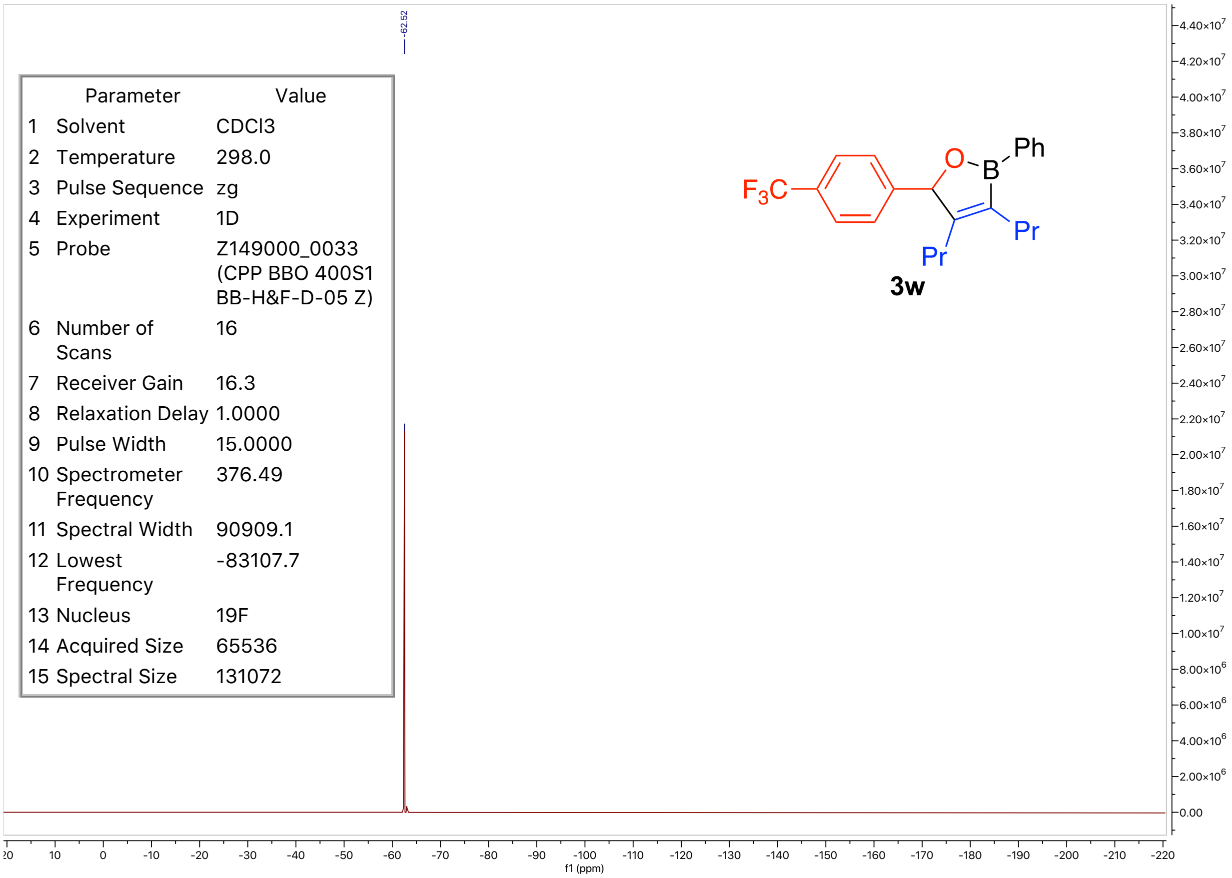
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	16
7	Receiver Gain	16.3
8	Relaxation Delay	1.0000
9	Pulse Width	15.0000
10	Spectrometer Frequency	376.49
11	Spectral Width	90909.1
12	Lowest Frequency	-83107.7
13	Nucleus	19F
14	Acquired Size	65536
15	Spectral Size	131072



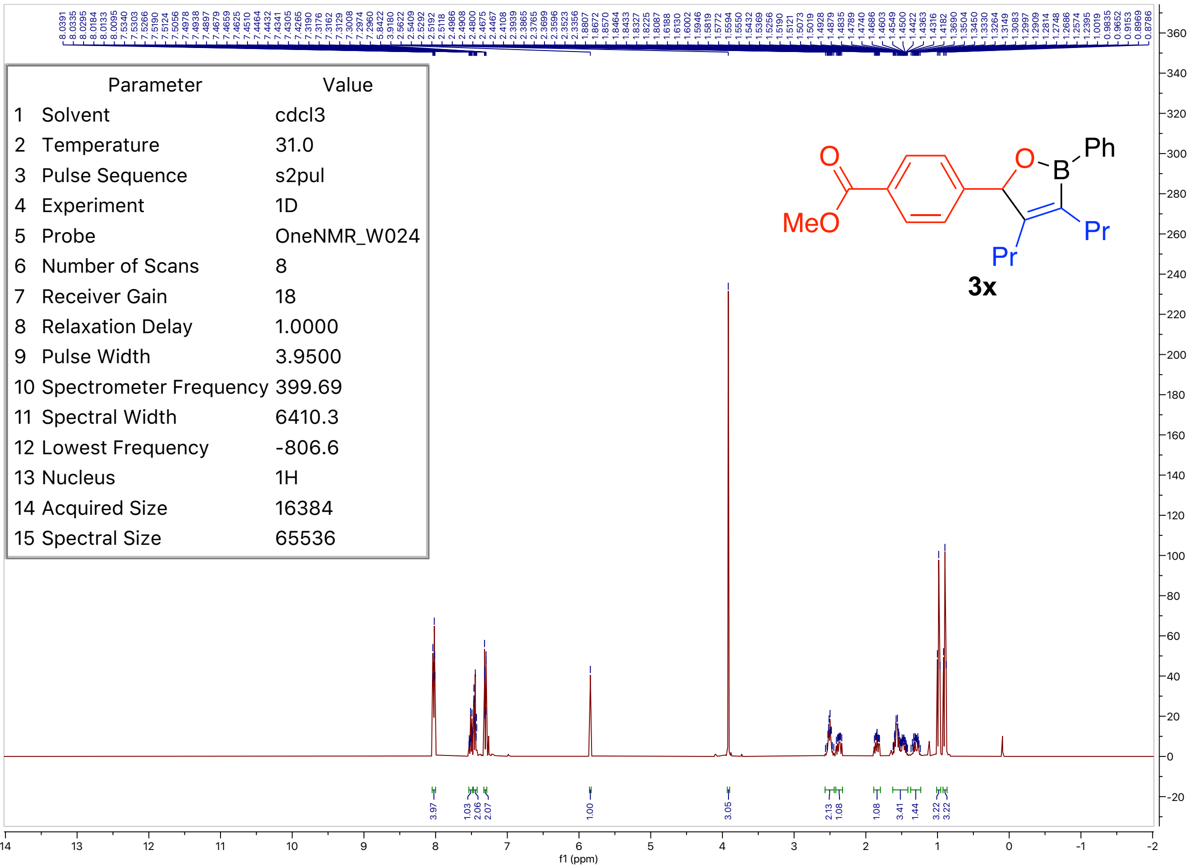
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	30
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.5
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



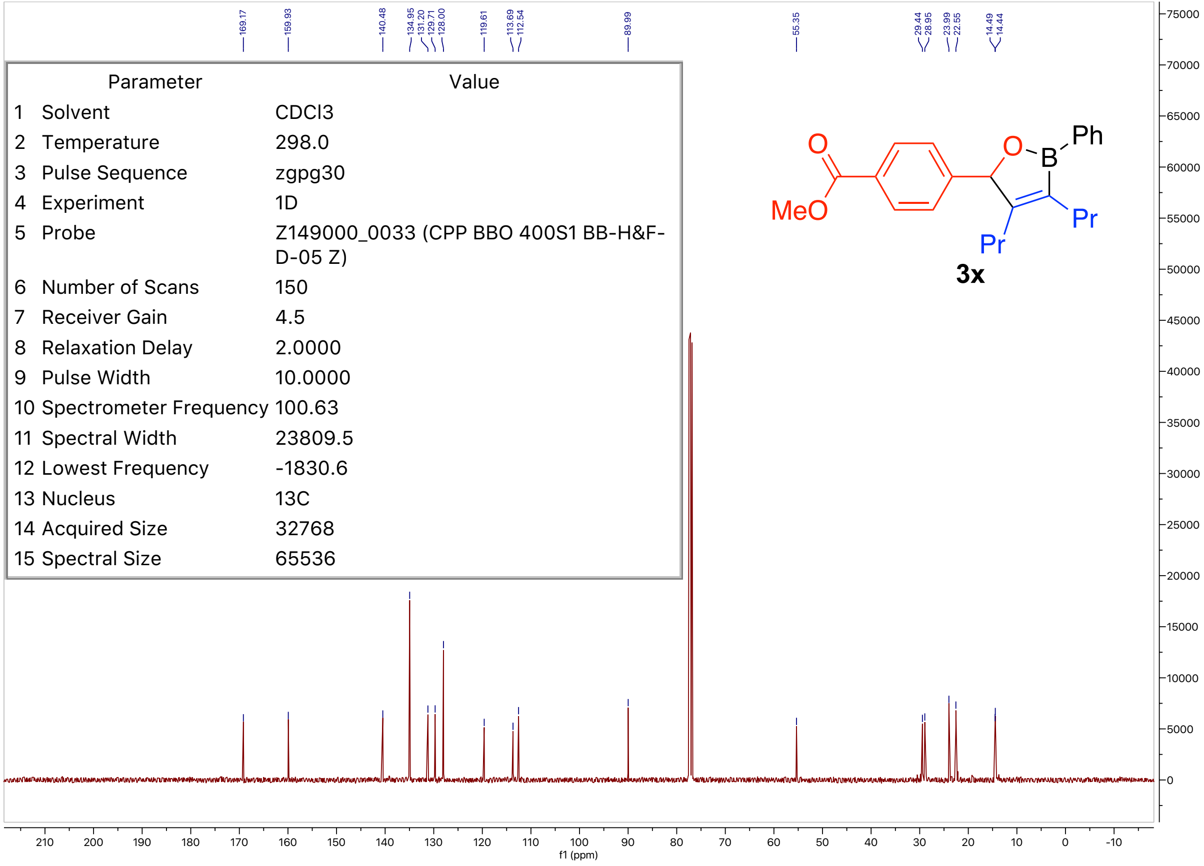
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	150
7	Receiver Gain	4.8
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1830.2
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



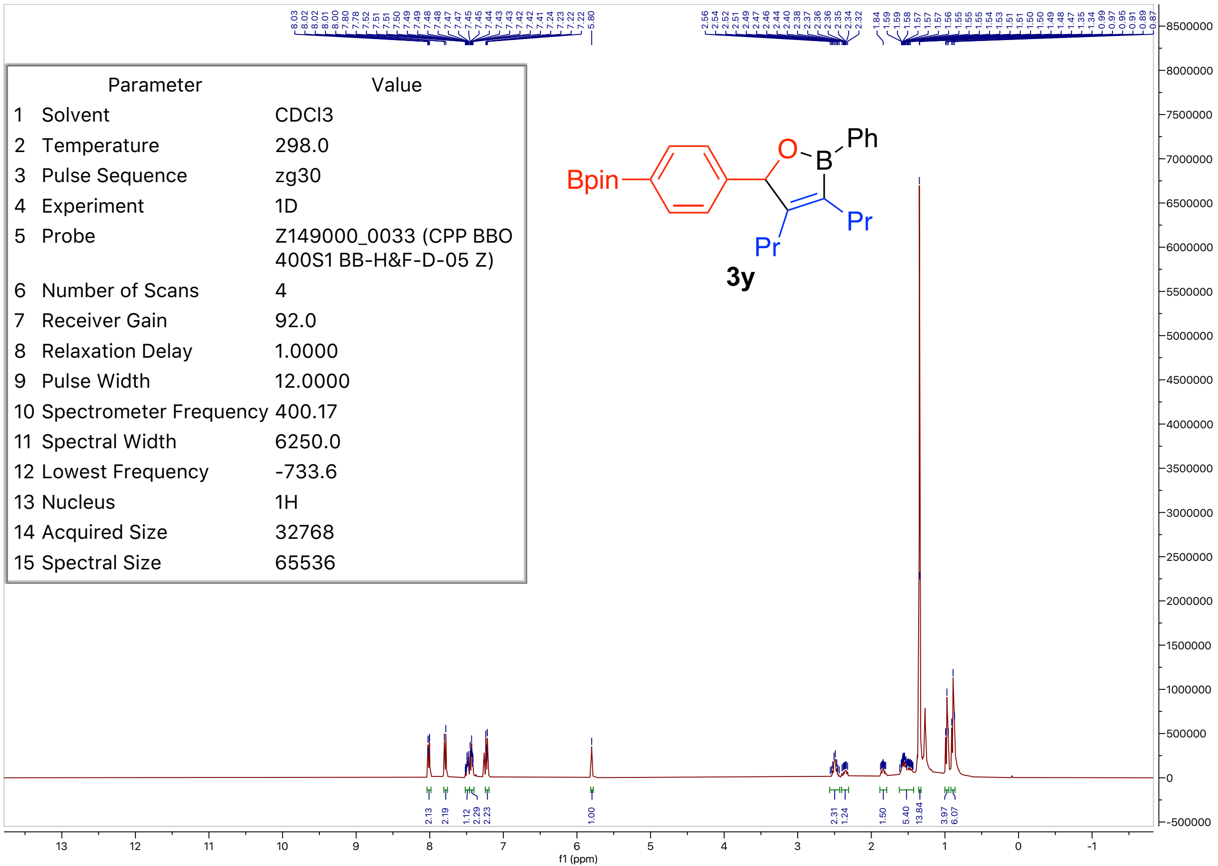
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	16
7	Receiver Gain	16.3
8	Relaxation Delay	1.0000
9	Pulse Width	15.0000
10	Spectrometer Frequency	376.49
11	Spectral Width	90909.1
12	Lowest Frequency	-83107.7
13	Nucleus	19F
14	Acquired Size	65536
15	Spectral Size	131072



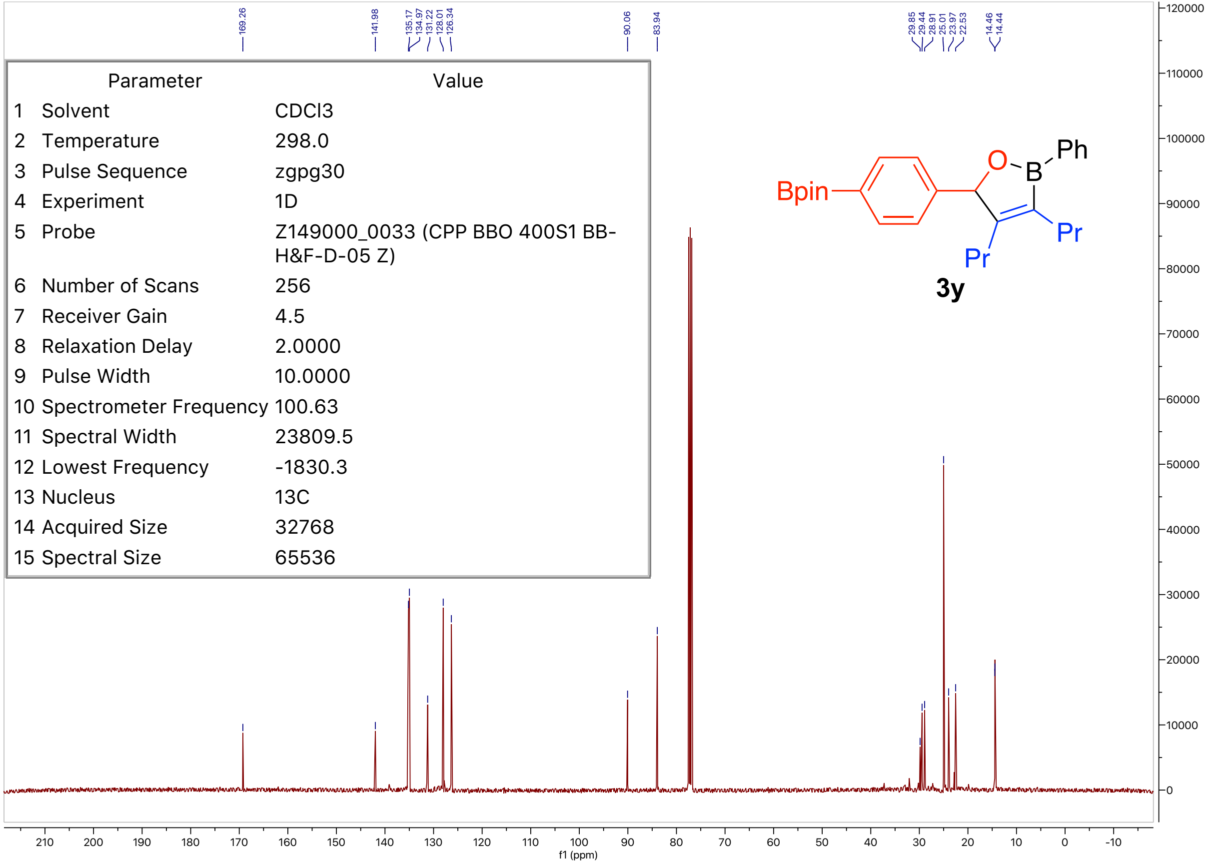
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.6
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



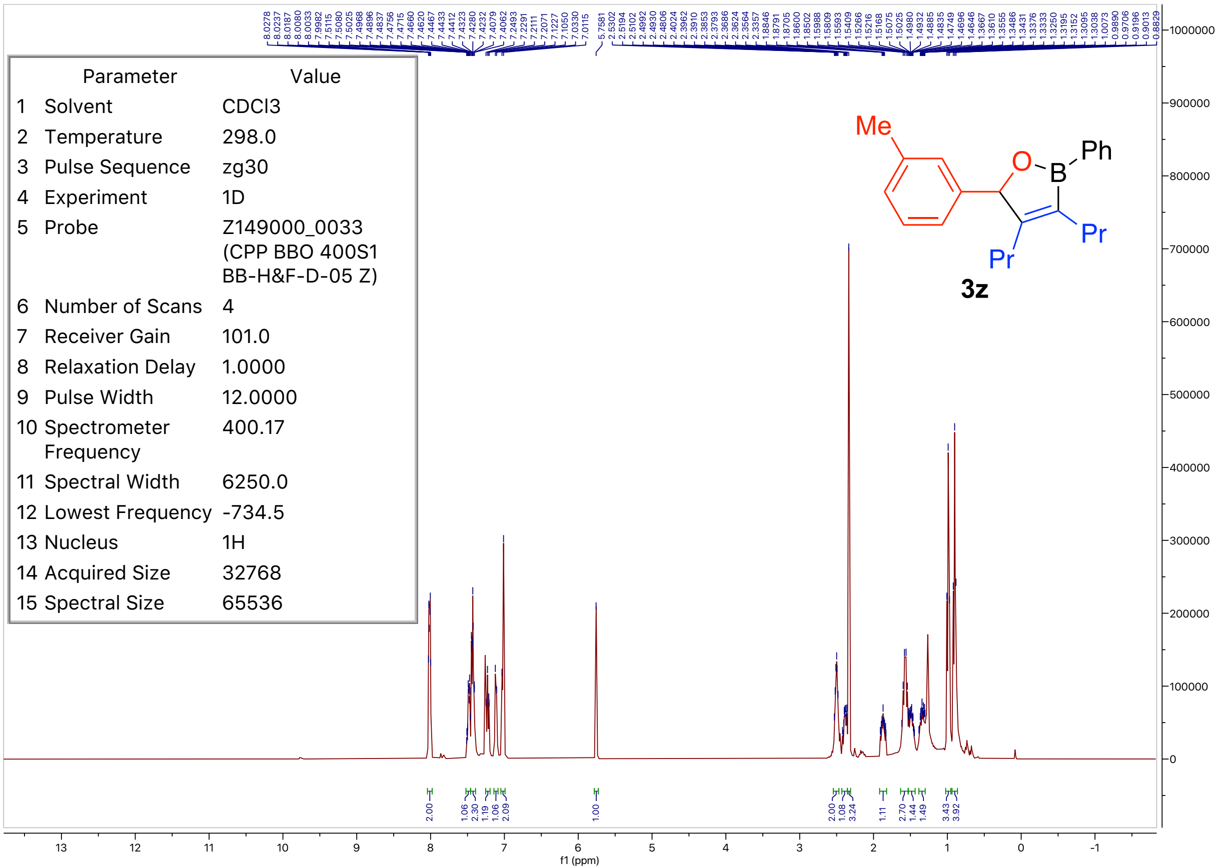
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	150
7	Receiver Gain	4.5
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1830.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



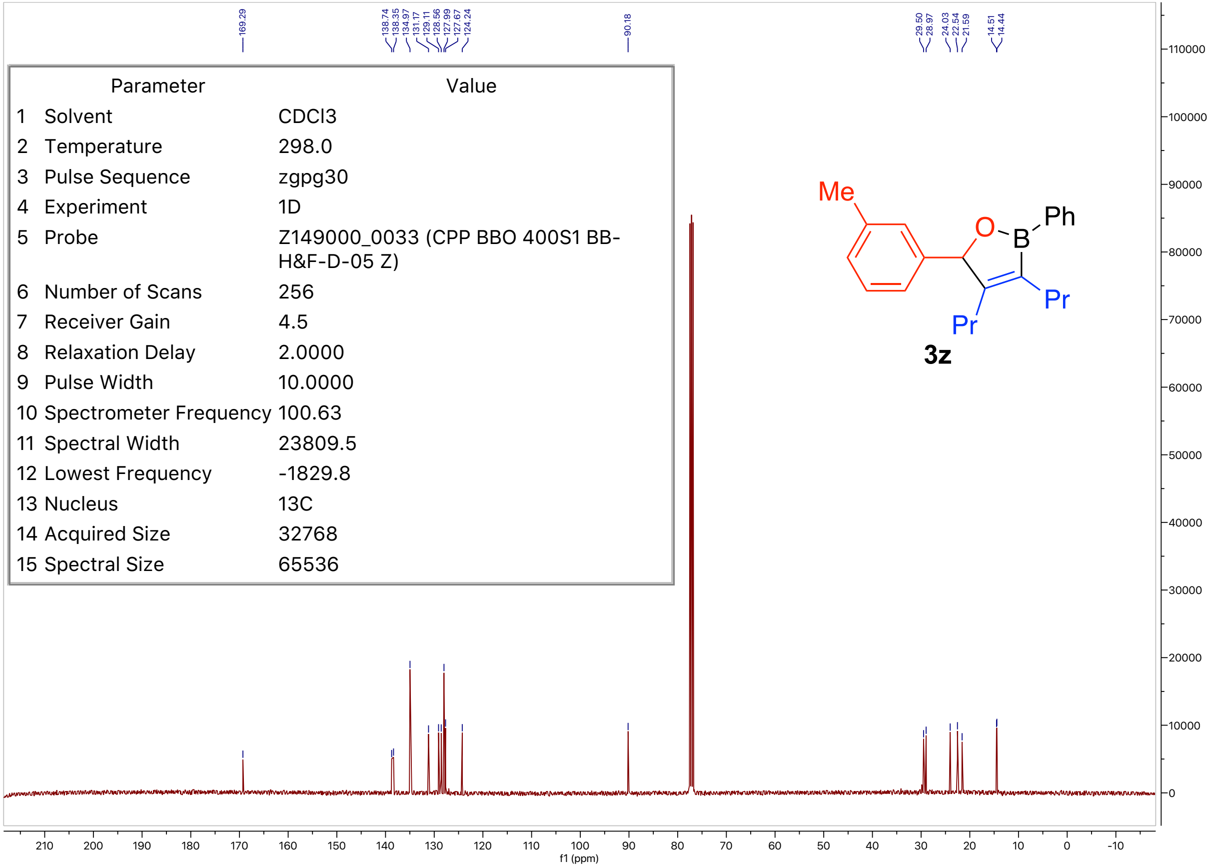
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	4
7	Receiver Gain	92.0
8	Relaxation Delay	1.0000
9	Pulse Width	12.0000
10	Spectrometer Frequency	400.17
11	Spectral Width	6250.0
12	Lowest Frequency	-733.6
13	Nucleus	1H
14	Acquired Size	32768
15	Spectral Size	65536



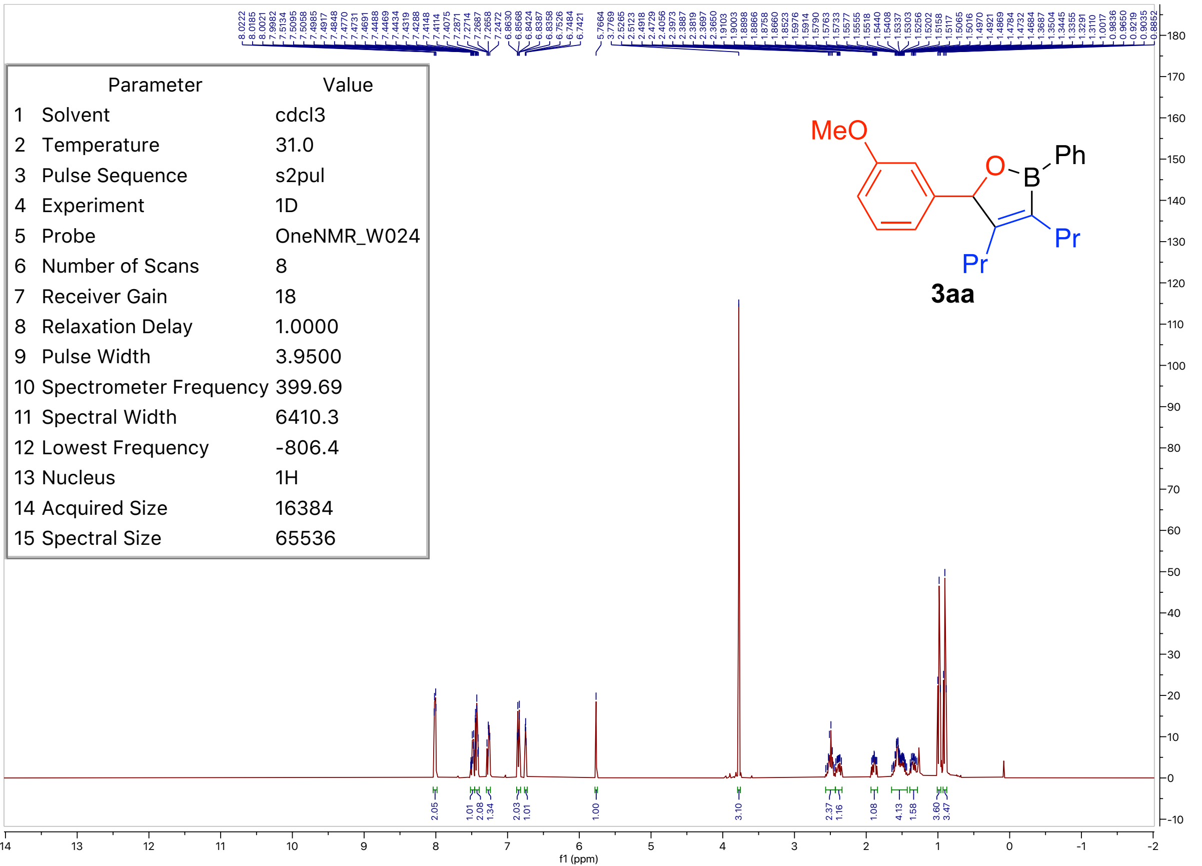
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	256
7	Receiver Gain	4.5
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1830.3
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	4
7	Receiver Gain	101.0
8	Relaxation Delay	1.0000
9	Pulse Width	12.0000
10	Spectrometer Frequency	400.17
11	Spectral Width	6250.0
12	Lowest Frequency	-734.5
13	Nucleus	1H
14	Acquired Size	32768
15	Spectral Size	65536

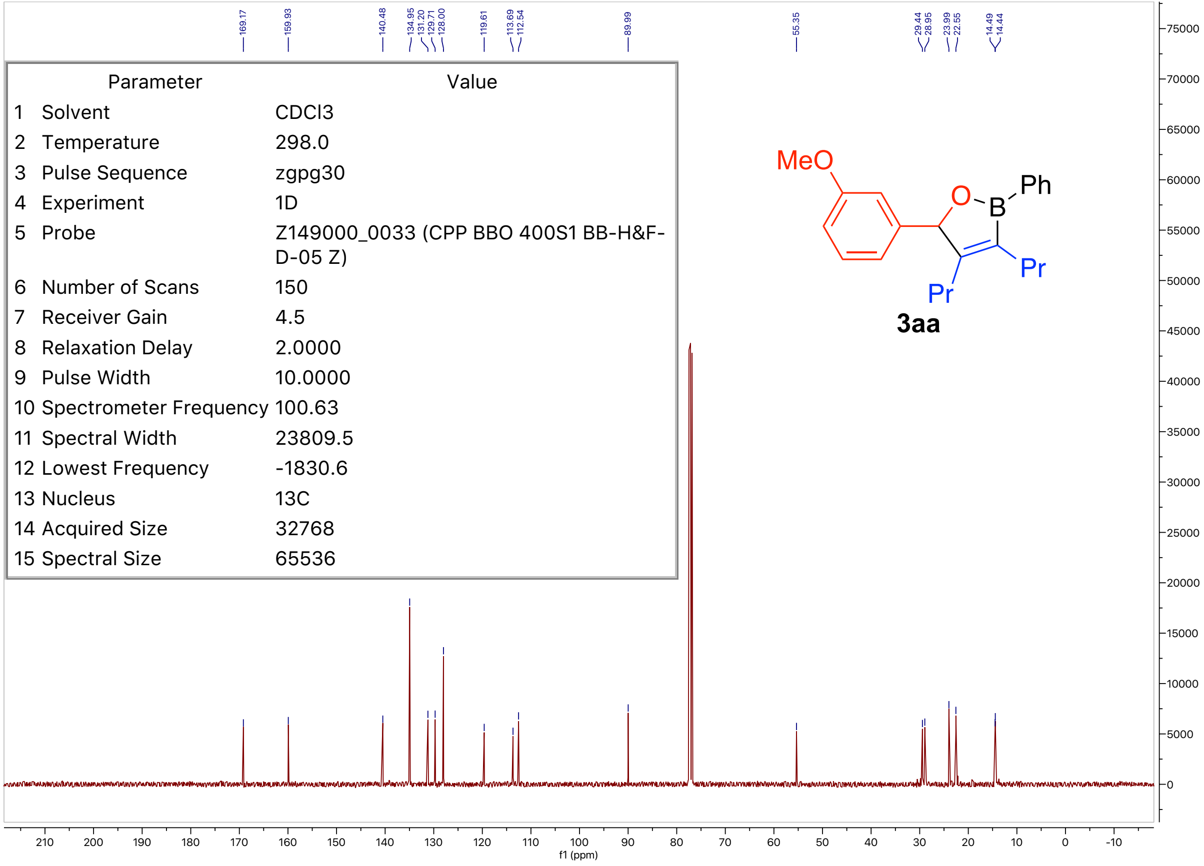


	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	256
7	Receiver Gain	4.5
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1829.8
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



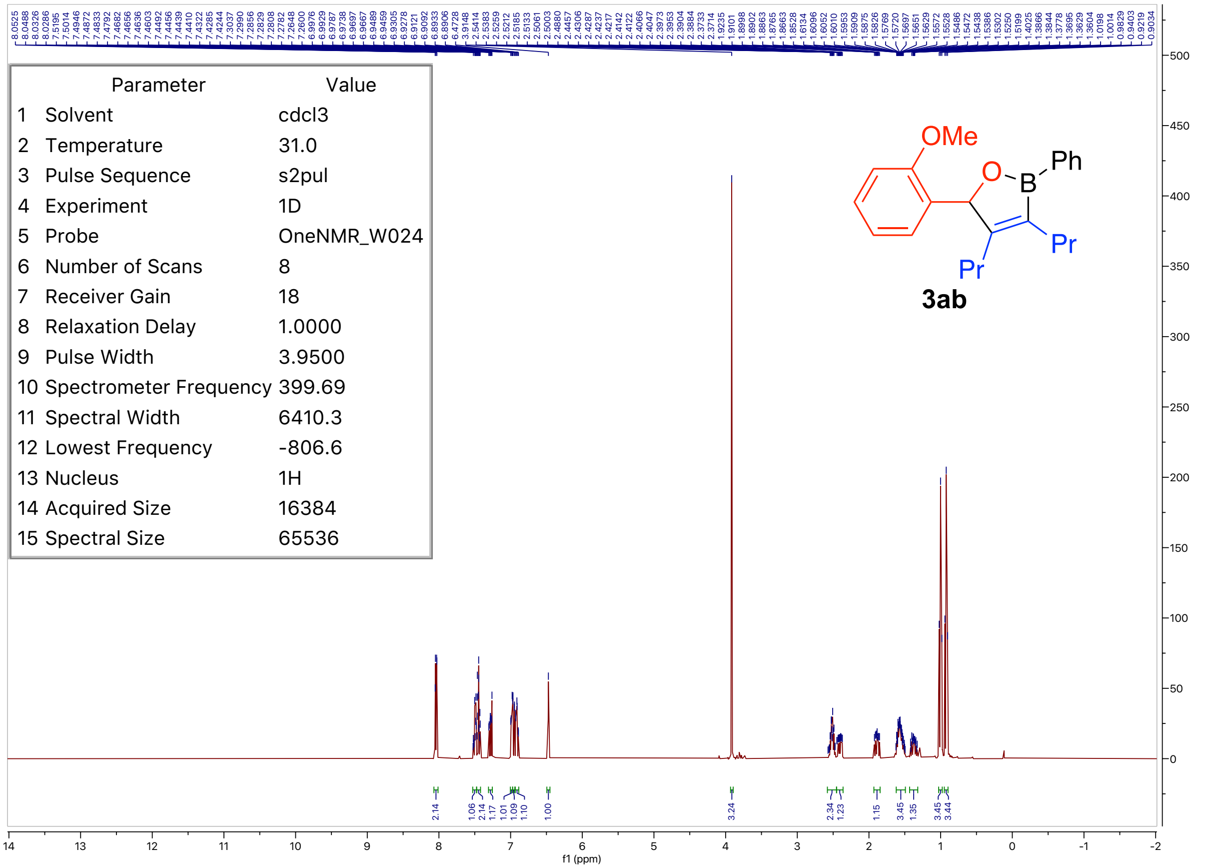
3aa

	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.4
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536

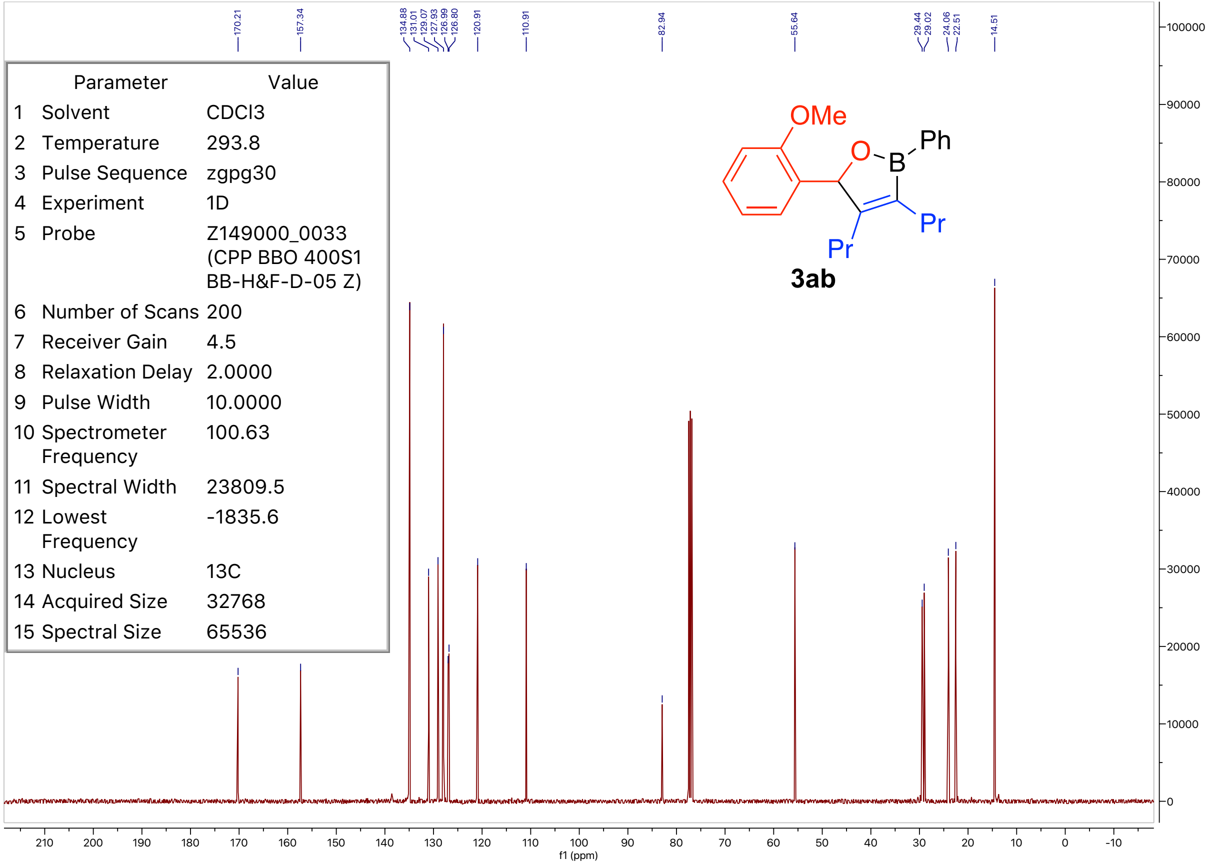


3aa

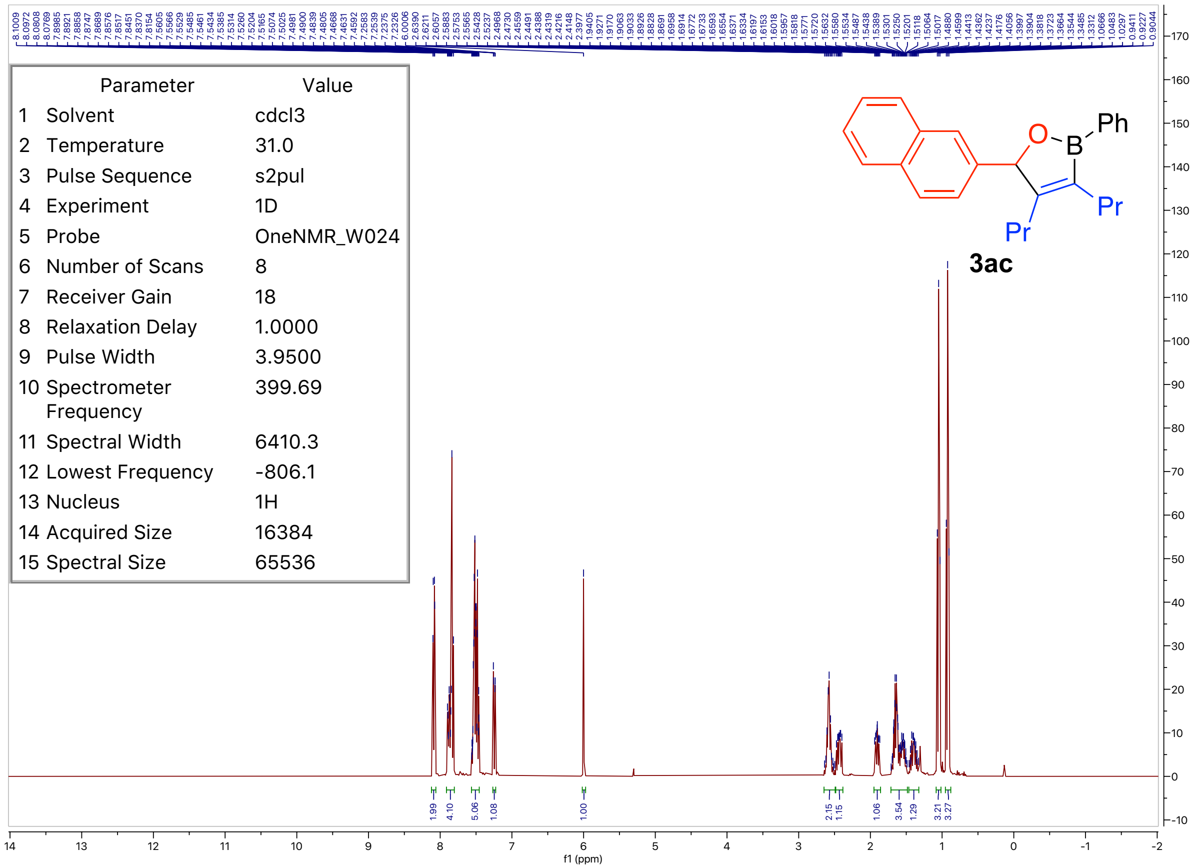
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	150
7	Receiver Gain	4.5
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1830.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



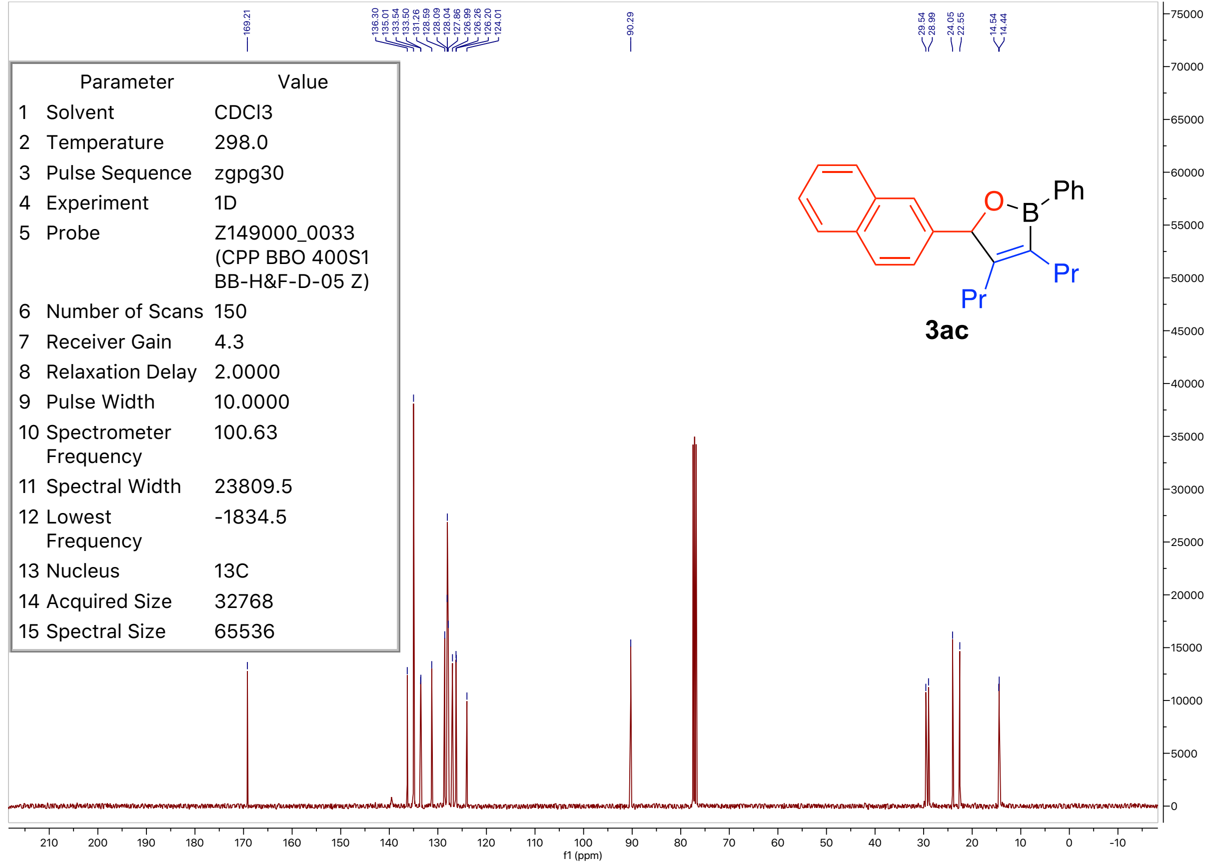
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.6
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



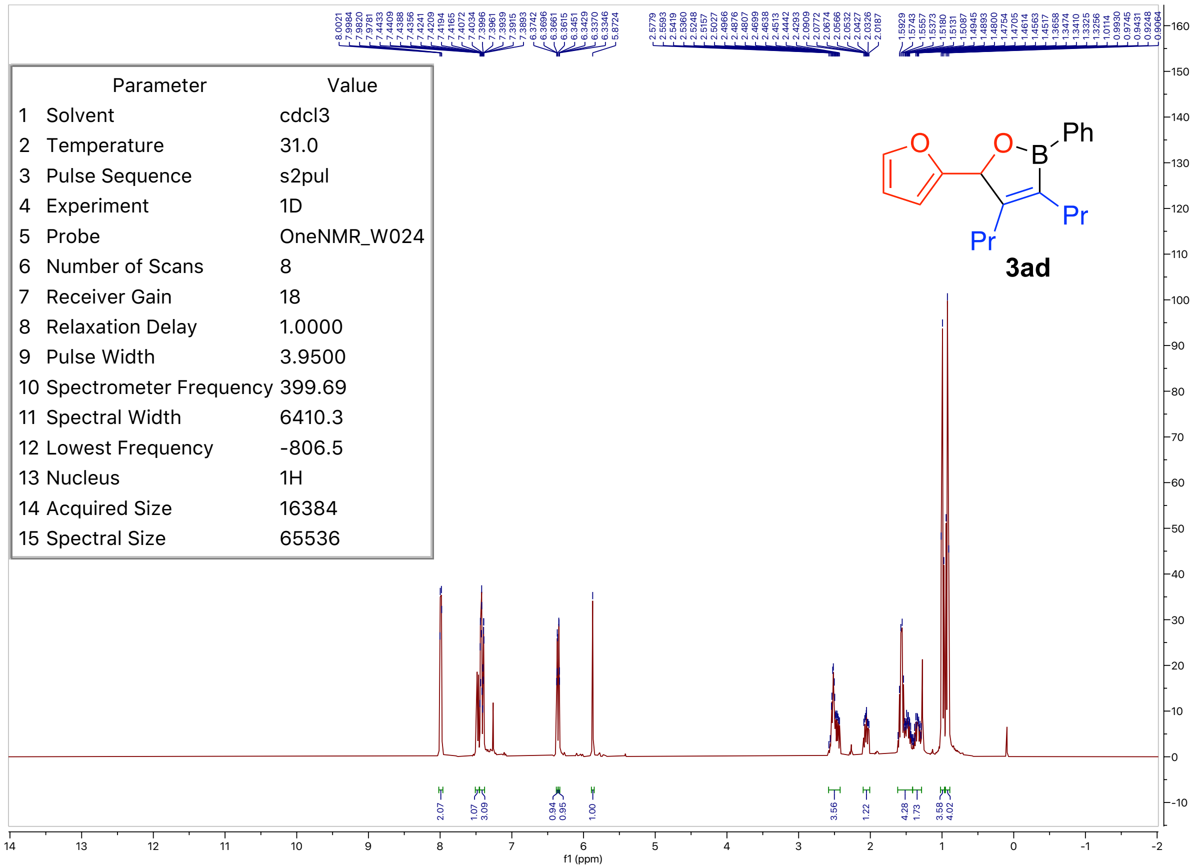
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	293.8
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	200
7	Receiver Gain	4.5
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1835.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



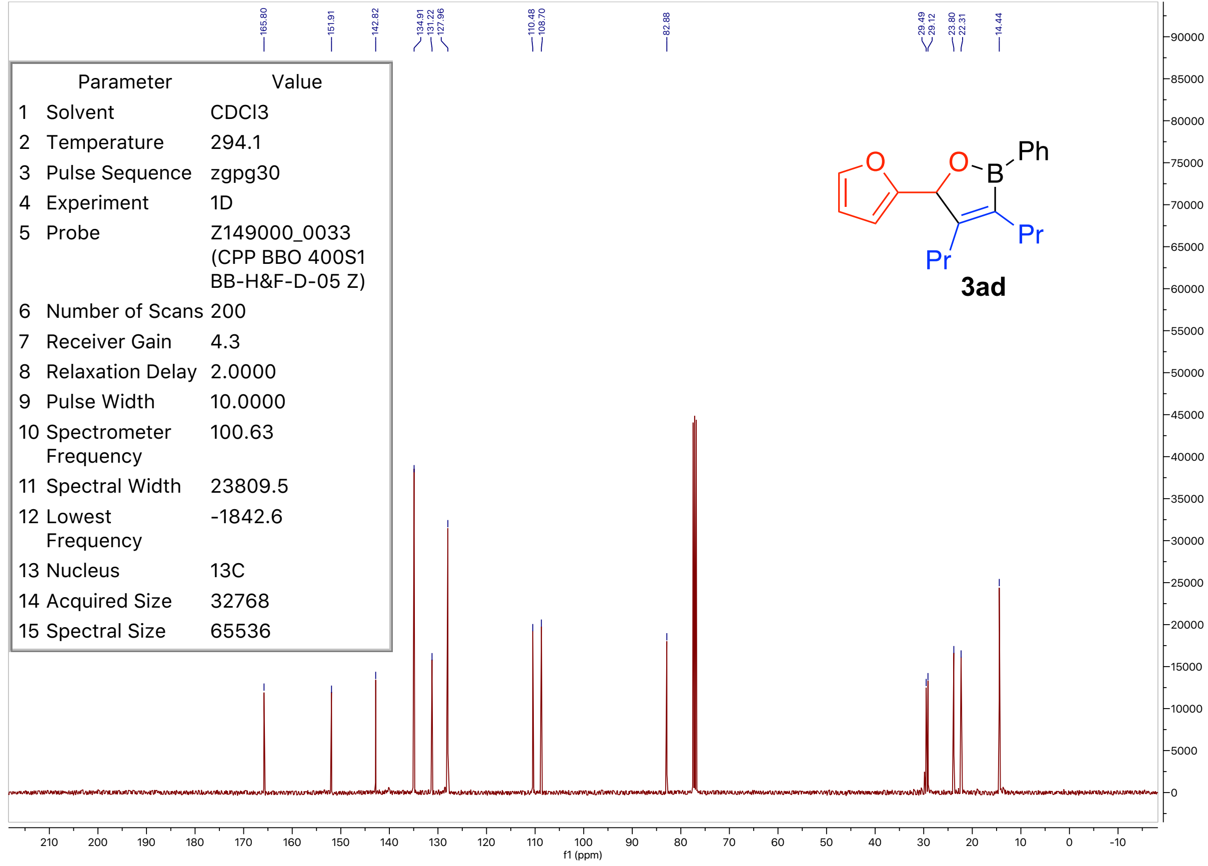
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.1
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



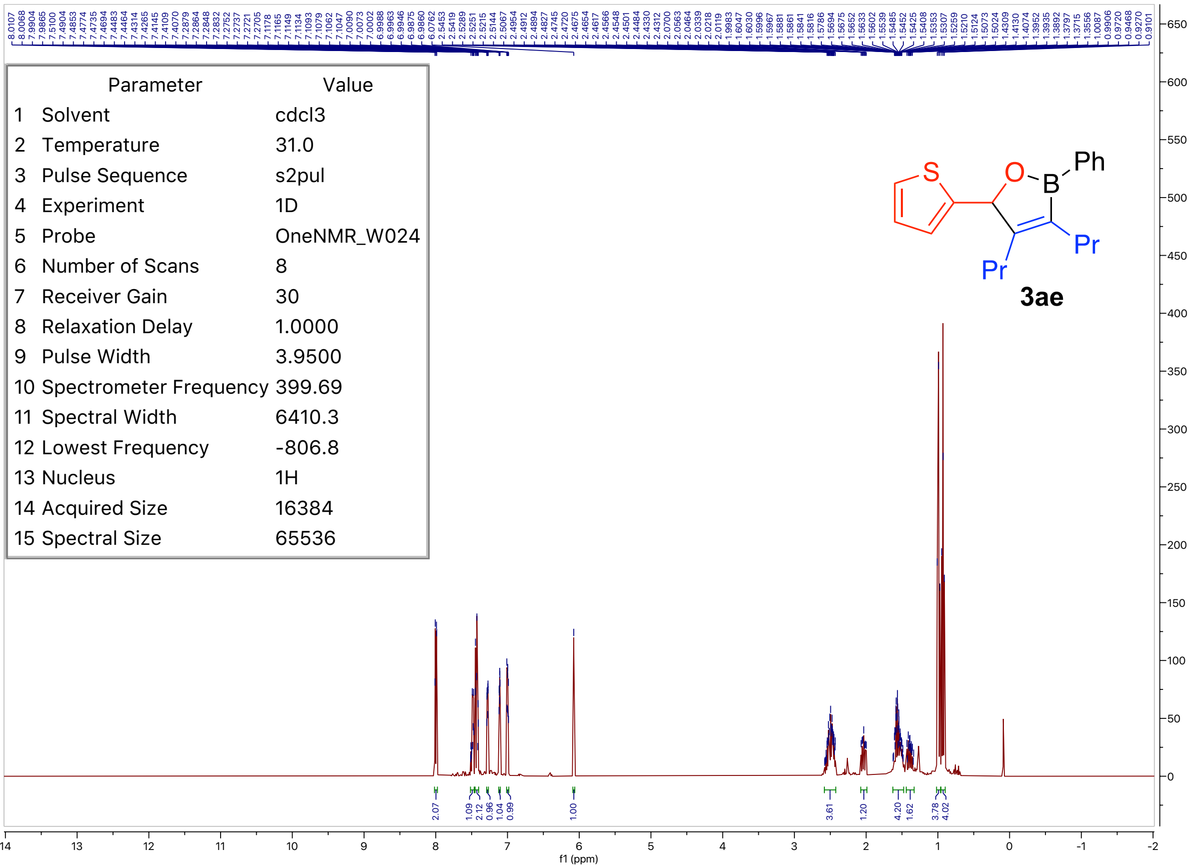
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	150
7	Receiver Gain	4.3
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1834.5
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



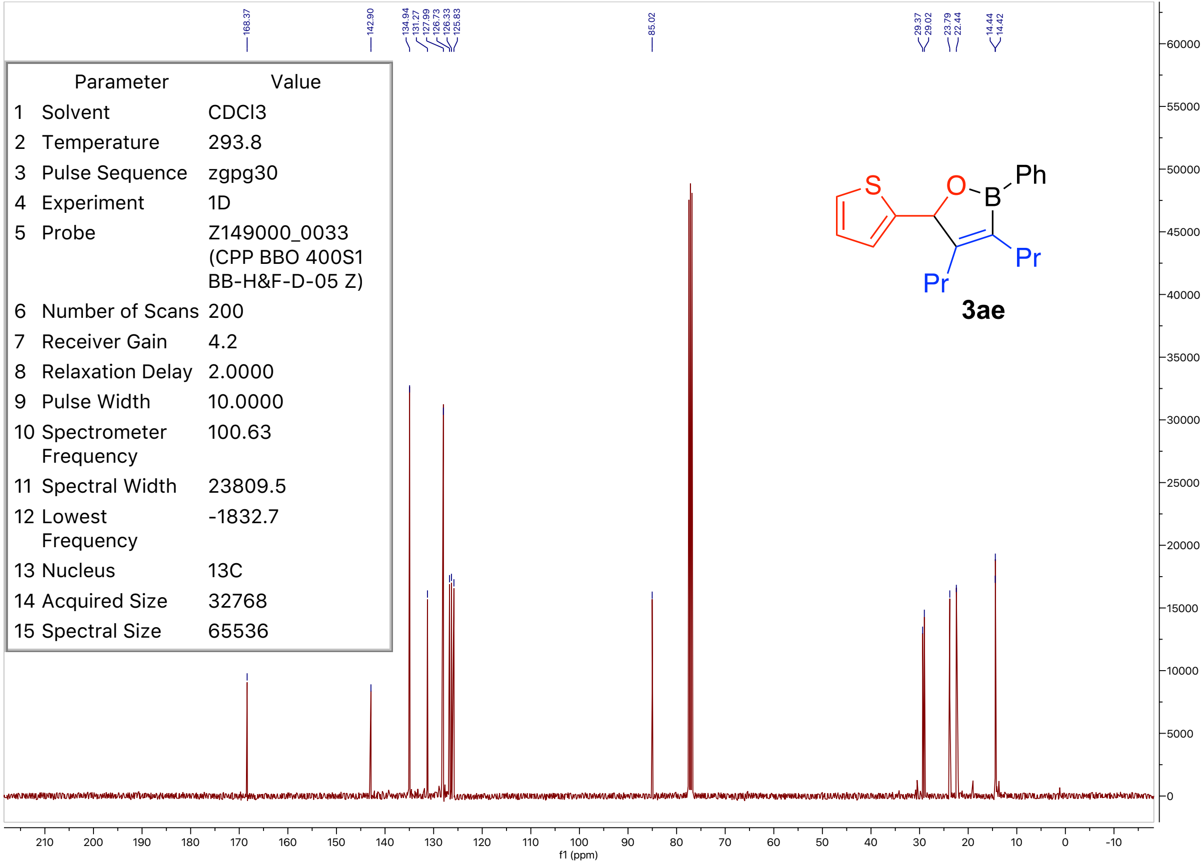
Parameter	Value
1 Solvent	cdcl3
2 Temperature	31.0
3 Pulse Sequence	s2pul
4 Experiment	1D
5 Probe	OneNMR_W024
6 Number of Scans	8
7 Receiver Gain	18
8 Relaxation Delay	1.0000
9 Pulse Width	3.9500
10 Spectrometer Frequency	399.69
11 Spectral Width	6410.3
12 Lowest Frequency	-806.5
13 Nucleus	1H
14 Acquired Size	16384
15 Spectral Size	65536



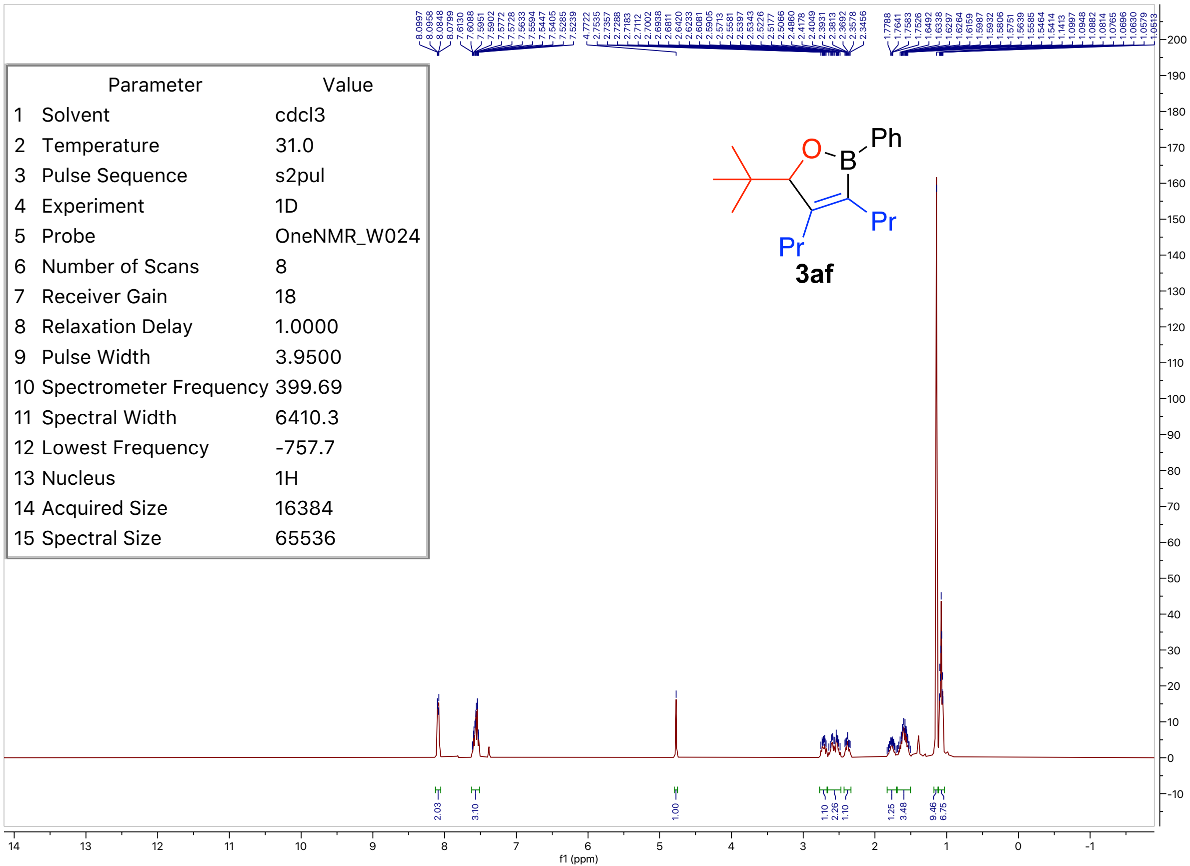
Parameter	Value
1 Solvent	CDCl3
2 Temperature	294.1
3 Pulse Sequence	zgpg30
4 Experiment	1D
5 Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6 Number of Scans	200
7 Receiver Gain	4.3
8 Relaxation Delay	2.0000
9 Pulse Width	10.0000
10 Spectrometer Frequency	100.63
11 Spectral Width	23809.5
12 Lowest Frequency	-1842.6
13 Nucleus	13C
14 Acquired Size	32768
15 Spectral Size	65536



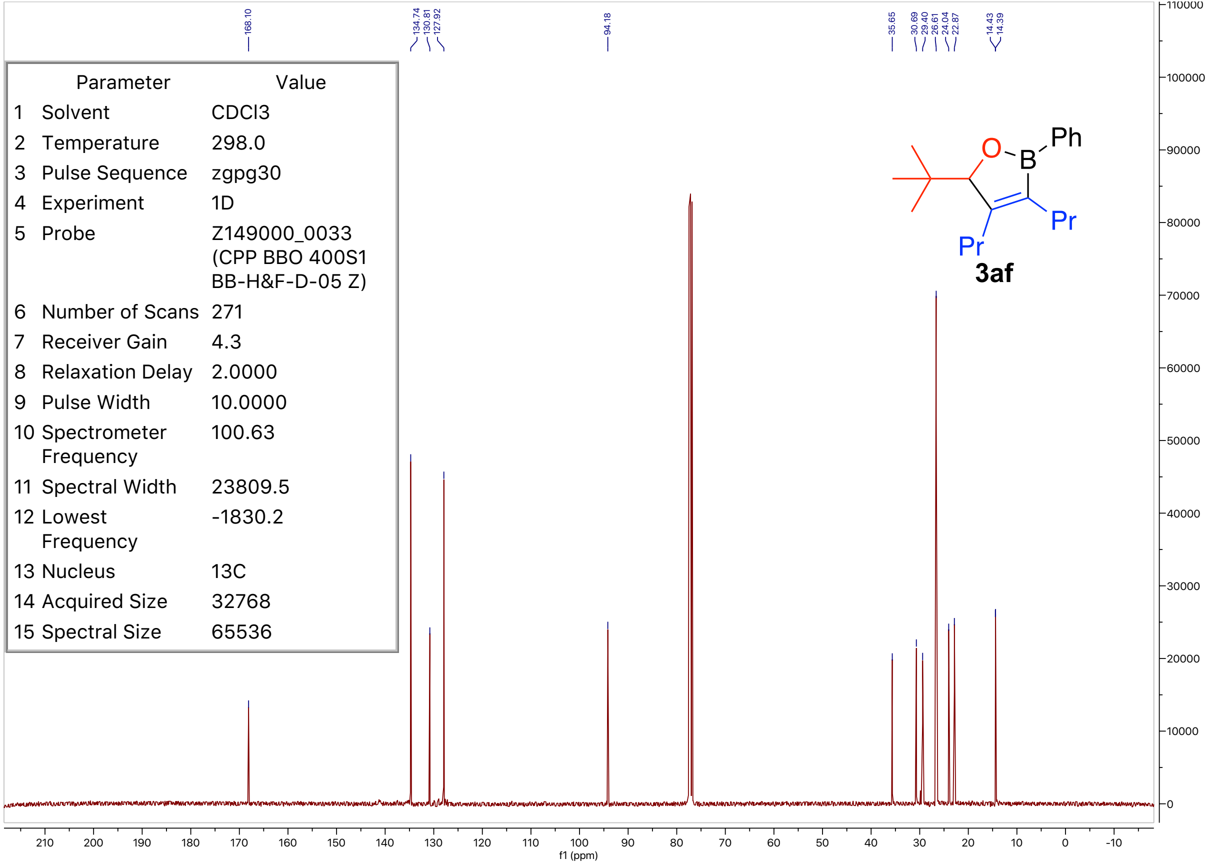
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	30
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.8
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



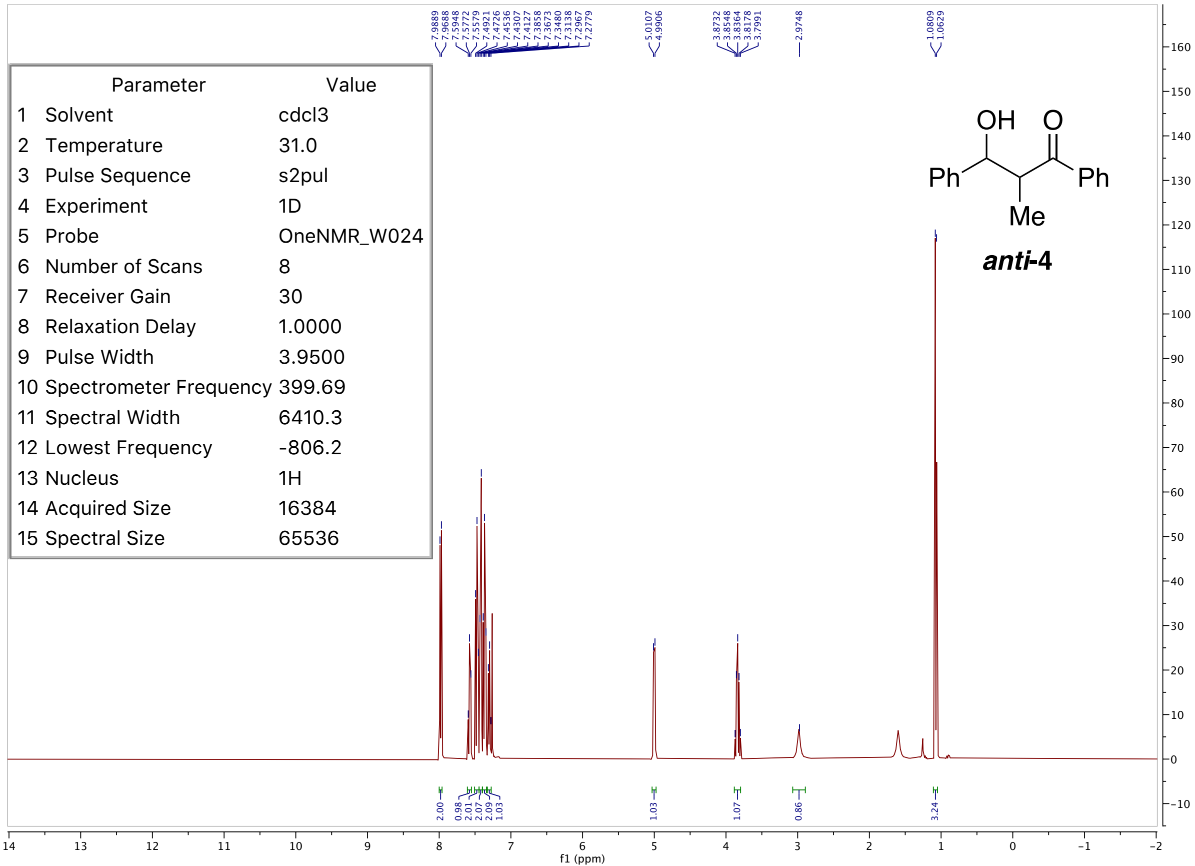
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	293.8
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	200
7	Receiver Gain	4.2
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1832.7
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



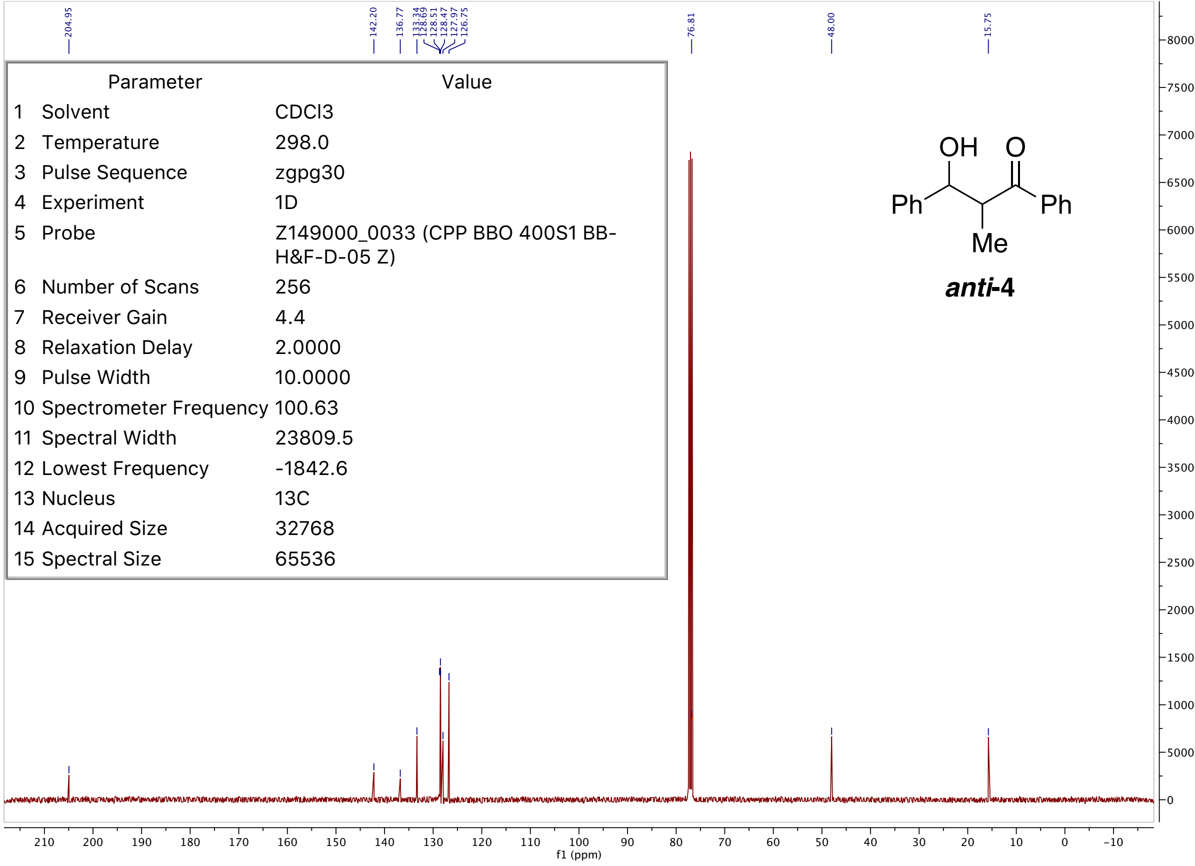
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-757.7
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



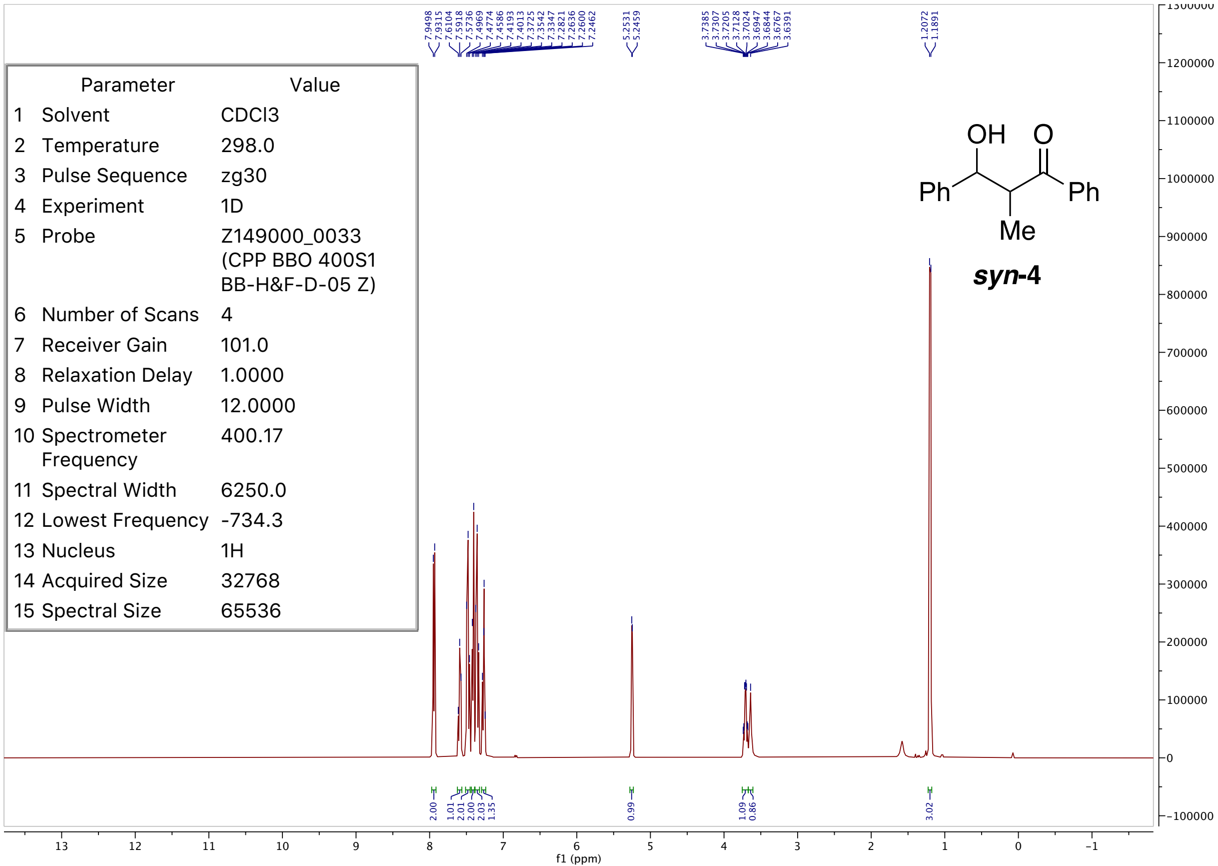
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	271
7	Receiver Gain	4.3
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1830.2
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



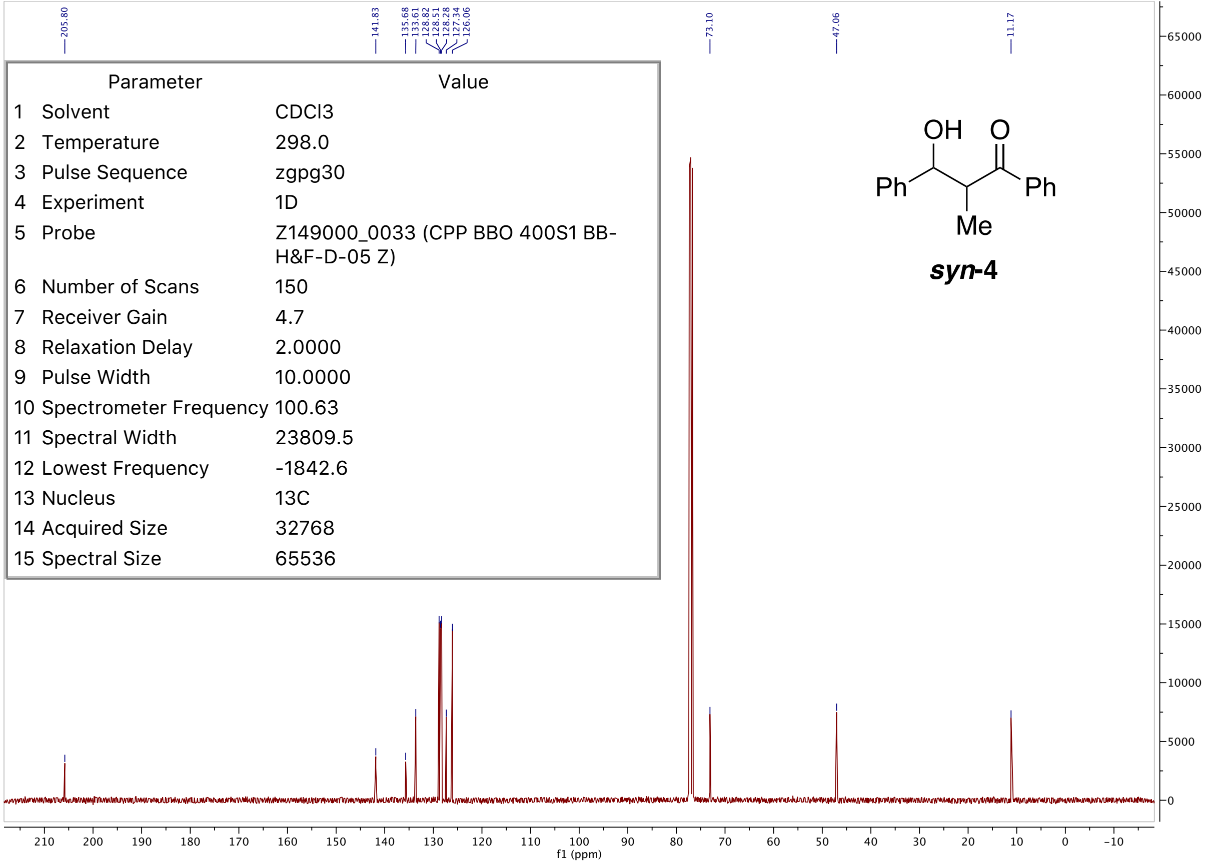
	Parameter	Value
1	Solvent	cdcl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	30
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-806.2
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



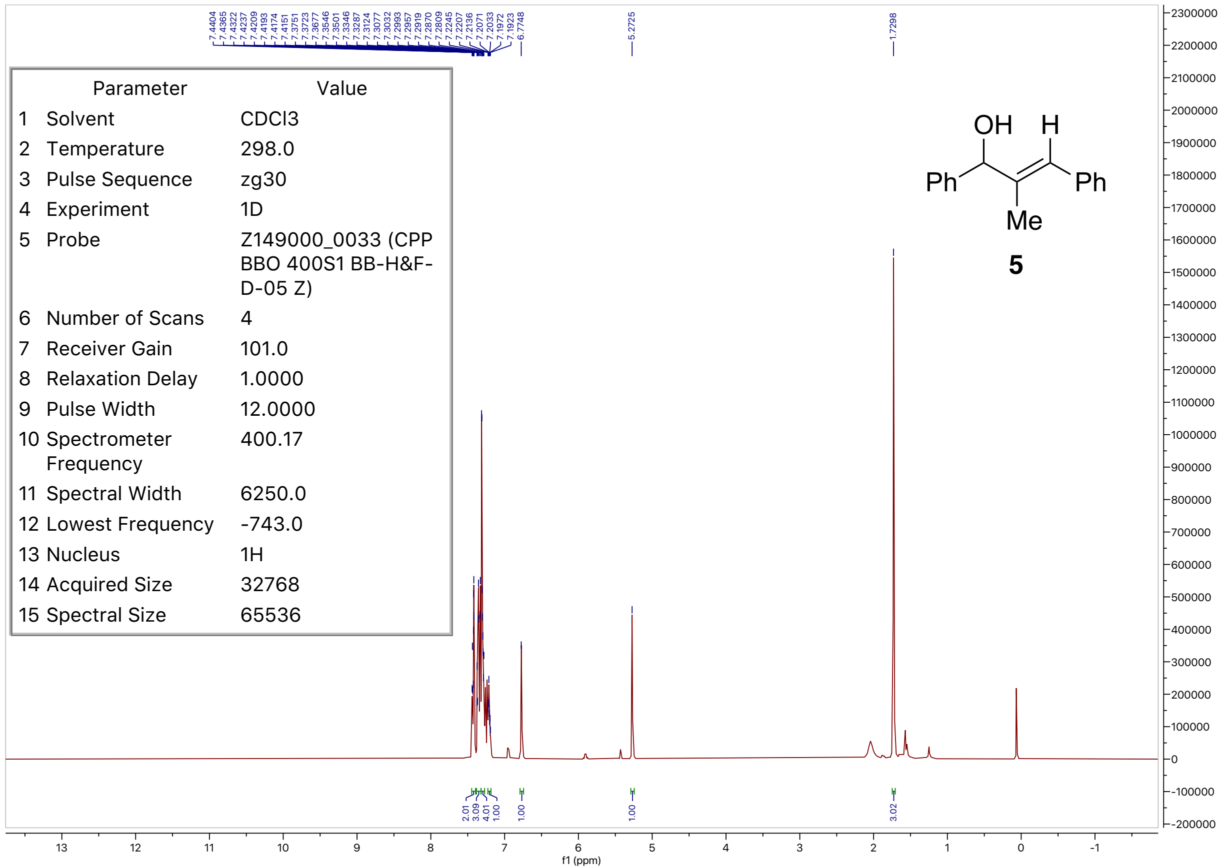
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	256
7	Receiver Gain	4.4
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



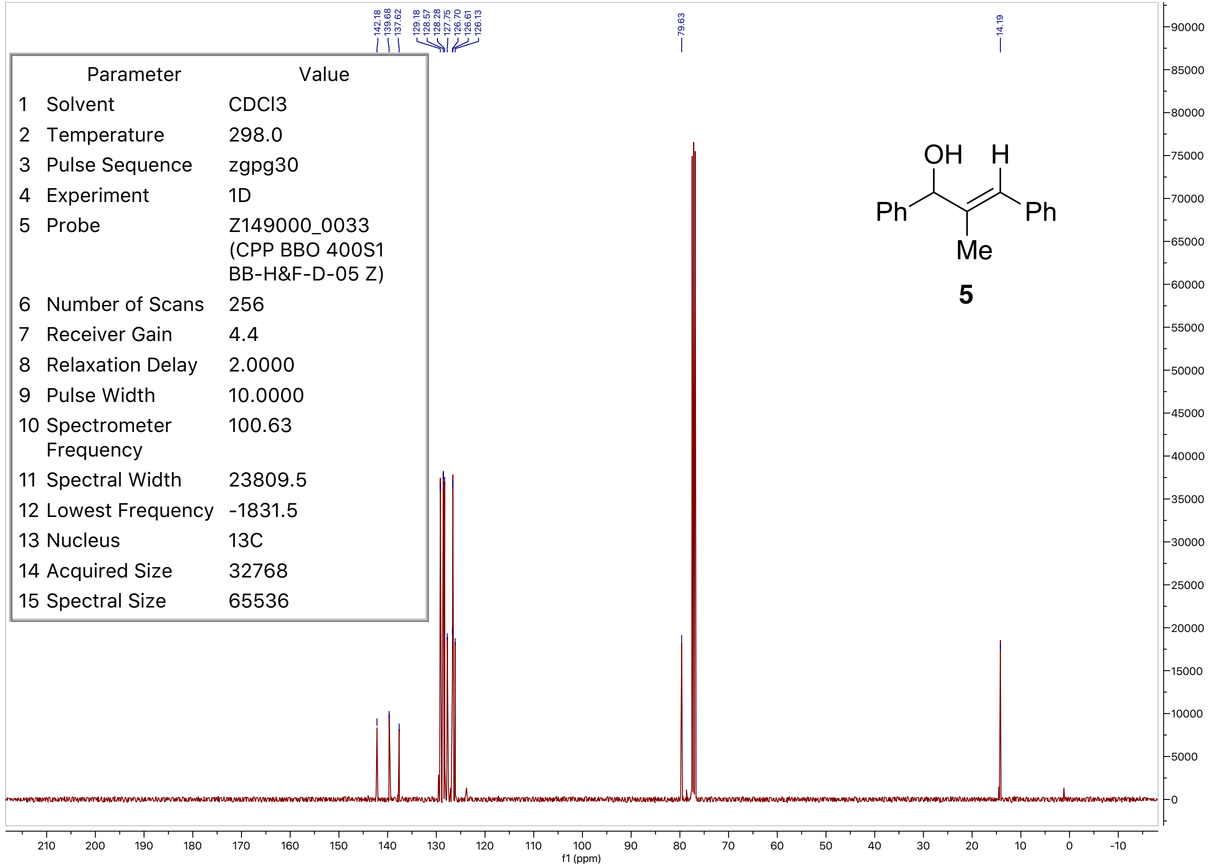
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	4
7	Receiver Gain	101.0
8	Relaxation Delay	1.0000
9	Pulse Width	12.0000
10	Spectrometer Frequency	400.17
11	Spectral Width	6250.0
12	Lowest Frequency	-734.3
13	Nucleus	1H
14	Acquired Size	32768
15	Spectral Size	65536



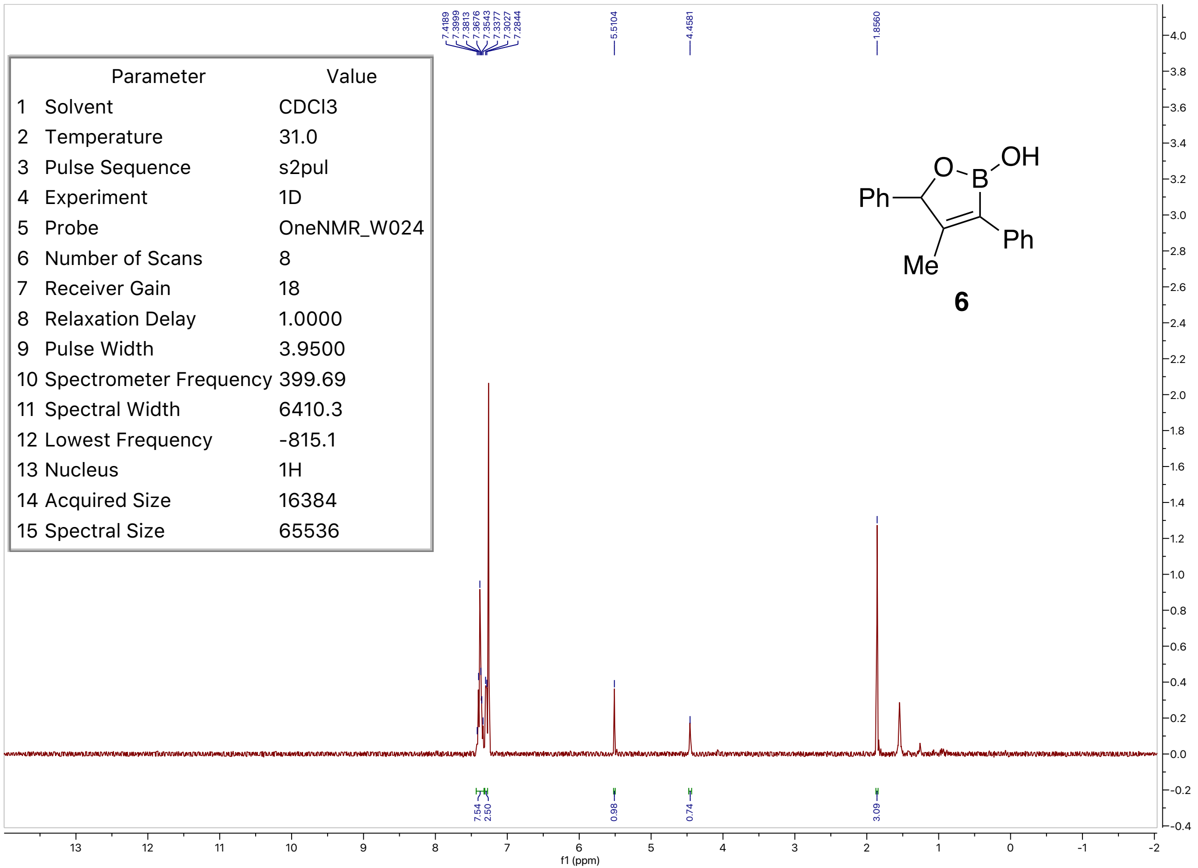
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	150
7	Receiver Gain	4.7
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



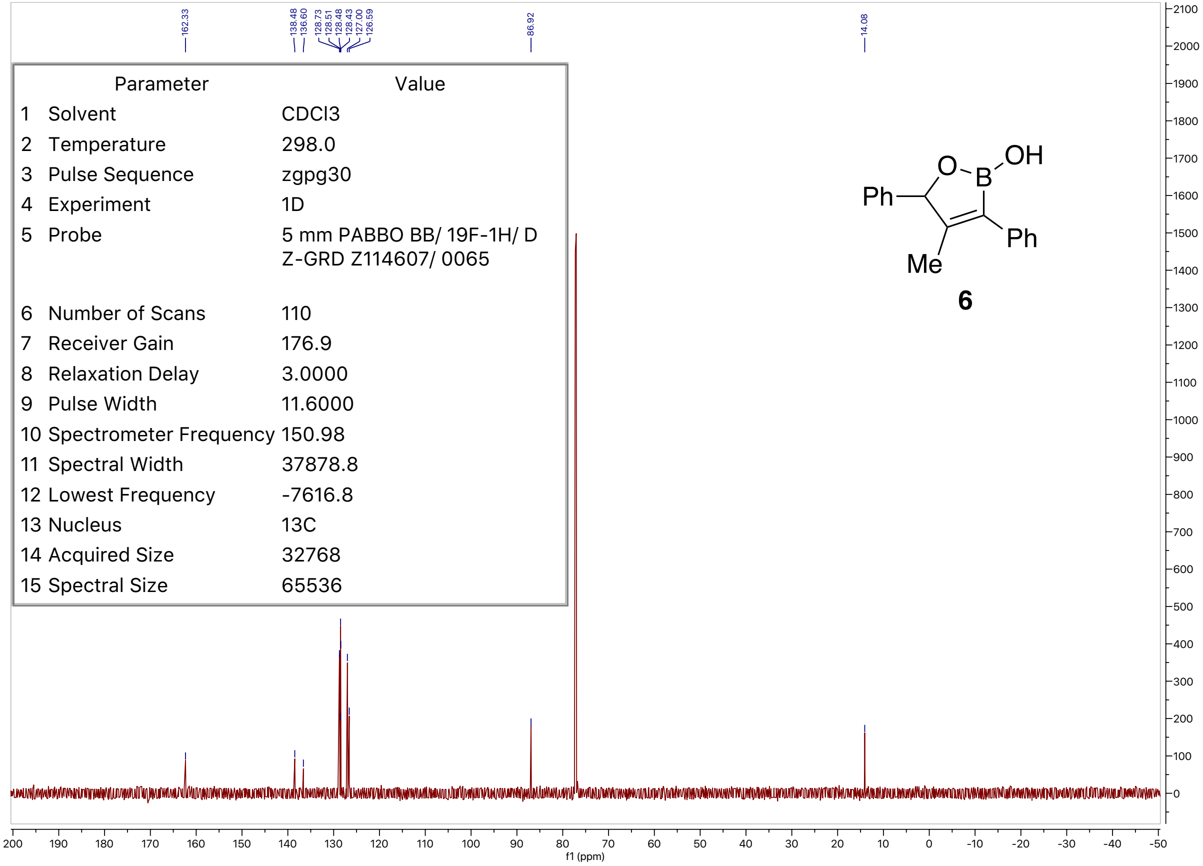
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	4
7	Receiver Gain	101.0
8	Relaxation Delay	1.0000
9	Pulse Width	12.0000
10	Spectrometer Frequency	400.17
11	Spectral Width	6250.0
12	Lowest Frequency	-743.0
13	Nucleus	1H
14	Acquired Size	32768
15	Spectral Size	65536



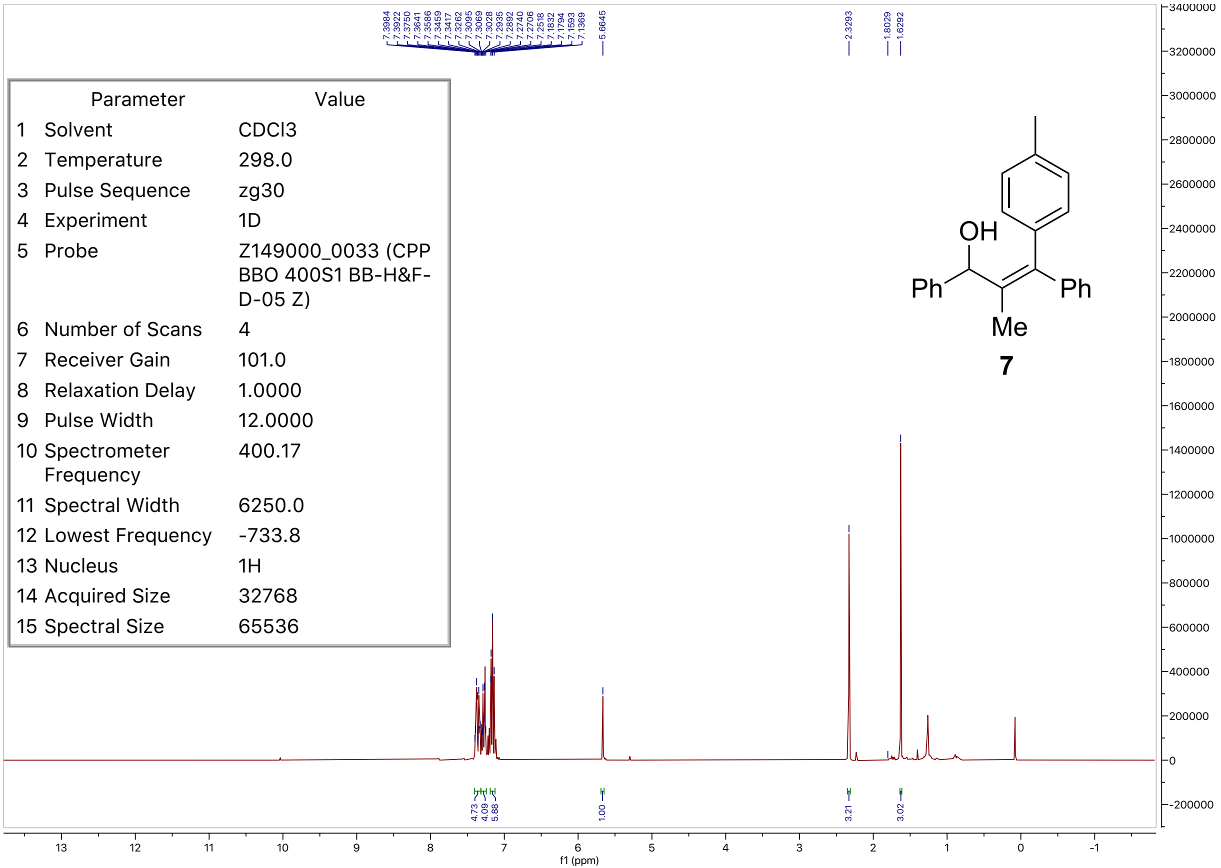
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	256
7	Receiver Gain	4.4
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1831.5
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



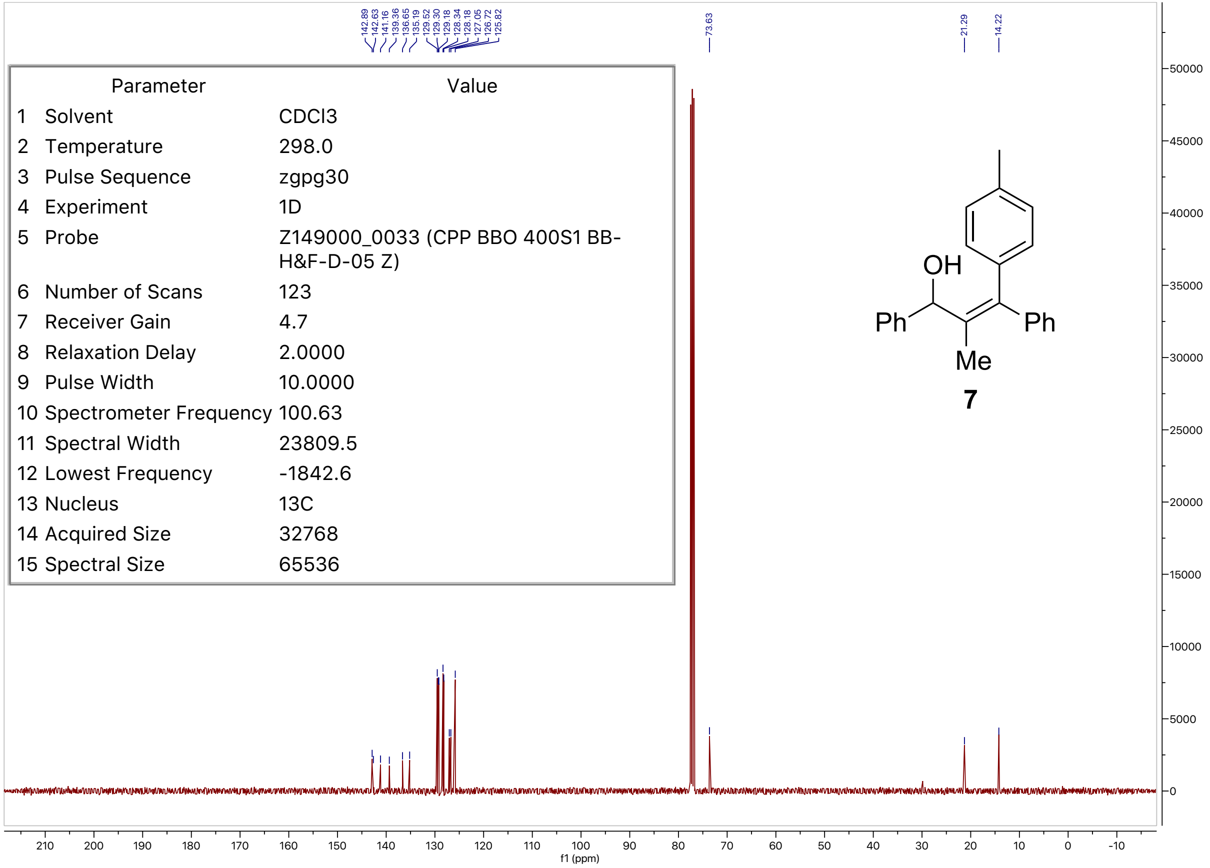
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	31.0
3	Pulse Sequence	s2pul
4	Experiment	1D
5	Probe	OneNMR_W024
6	Number of Scans	8
7	Receiver Gain	18
8	Relaxation Delay	1.0000
9	Pulse Width	3.9500
10	Spectrometer Frequency	399.69
11	Spectral Width	6410.3
12	Lowest Frequency	-815.1
13	Nucleus	1H
14	Acquired Size	16384
15	Spectral Size	65536



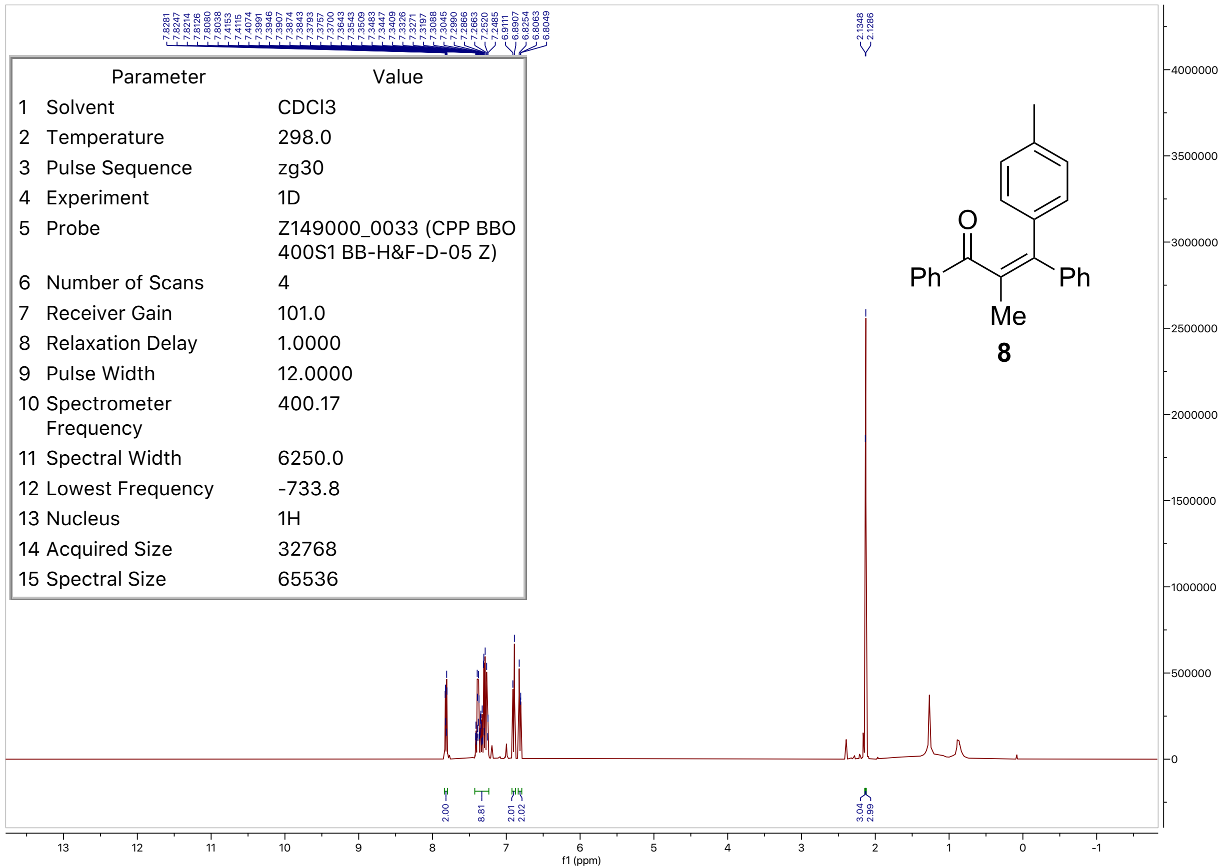
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	5 mm PABBO BB/ 19F-1H/ D Z-GRD Z114607/ 0065
6	Number of Scans	110
7	Receiver Gain	176.9
8	Relaxation Delay	3.0000
9	Pulse Width	11.6000
10	Spectrometer Frequency	150.98
11	Spectral Width	37878.8
12	Lowest Frequency	-7616.8
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



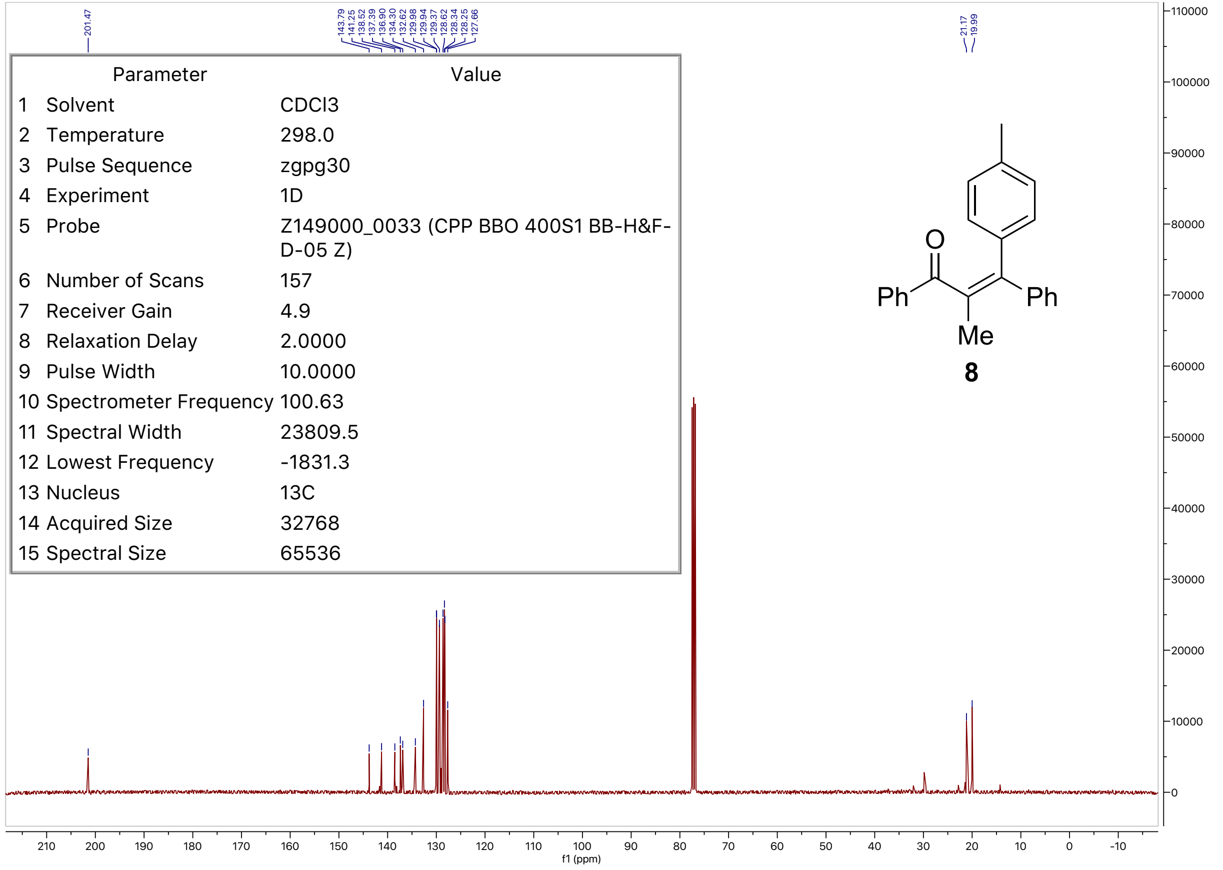
	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	4
7	Receiver Gain	101.0
8	Relaxation Delay	1.0000
9	Pulse Width	12.0000
10	Spectrometer Frequency	400.17
11	Spectral Width	6250.0
12	Lowest Frequency	-733.8
13	Nucleus	1H
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	123
7	Receiver Gain	4.7
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1842.6
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	4
7	Receiver Gain	101.0
8	Relaxation Delay	1.0000
9	Pulse Width	12.0000
10	Spectrometer Frequency	400.17
11	Spectral Width	6250.0
12	Lowest Frequency	-733.8
13	Nucleus	1H
14	Acquired Size	32768
15	Spectral Size	65536



	Parameter	Value
1	Solvent	CDCl3
2	Temperature	298.0
3	Pulse Sequence	zgpg30
4	Experiment	1D
5	Probe	Z149000_0033 (CPP BBO 400S1 BB-H&F-D-05 Z)
6	Number of Scans	157
7	Receiver Gain	4.9
8	Relaxation Delay	2.0000
9	Pulse Width	10.0000
10	Spectrometer Frequency	100.63
11	Spectral Width	23809.5
12	Lowest Frequency	-1831.3
13	Nucleus	13C
14	Acquired Size	32768
15	Spectral Size	65536