Electronic Supplementary Material (ESI) for New Journal of Chemistry.


Supporting Information for

# Aluminium complex–catalysed hydroboration of alkenes and alkynes

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**FS55.** $^{13}C$ NMR spectrum (100 MHz, 25°C, $CDCl_3$ of **3a.**

**FS56.** $^{1}H$ NMR spectrum (400 MHz, 25°C, $CDCl_3$) of **3b.**

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**FS85.** $^{13}C$ NMR spectrum (100 MHz, 25°C, $CDCl_3$ of **3k.**

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**FS87.** $^{13}C$ NMR spectrum (100 MHz, 25°C, $CDCl_3$ of **4a.**

**FS88.** $^{1}H$ NMR spectrum (100 MHz, 25°C, $CDCl_3$ of **4b.**

**FS89.** $^{13}C$ NMR spectrum (100 MHz, 25°C, $CDCl_3$ of **4b.**

**Table TS1.** Crystallographic data and refinement parameters of **1**.

| **Crystal Parameters** | **1 (exp-6202)** |
|---|---|
| CCDC No. | 1909428 |
| Empirical formula | $C_{20}H_{22}N_2PSeAl$ |
| Formula weight | 427.30 |
| $T$ (K) | 150(2) K |
| $\lambda$ (Å) | 1.54184 |
| Crystal system | Monoclinic |
| Space group | *P 21/*n |
| $a$ (Å) | 9.5906(10) |
| $b$ (Å) | 13.1698(2) |
| $c$ (Å) | 16.0726(2) |
| $\alpha$ (°) | 90 |
| $\beta$ (°) | 103.371(2) |
| $\gamma$ (°) | 90 |
| $V$ (Å$^3$) | 1975.04(5) |
| Z | 4 |
| $D_{calc}$ g cm$^{-3}$ | 1.437 |
| $\mu$ (mm$^{-1}$) | 3.799 |
| $F$(000) | 872 |
| Theta range for data collection | 4.389 to 71.407 deg |
| Limiting indices<br>- | -11 ≤ h ≤ 11,<br>-14 ≤ k ≤ 16,<br>-16 ≤ l ≤ 19 |
| Reflections collected / unique | 7011/ 3573<br>[R(int) = 0.0177] |
| Completeness to theta | 99.7 % |
| Absorption correction | Multi-scan |

| | |
|---|---|
| Max. and min. transmission | 1.00000 and 0.54170 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 3753 / 0 / 229 |
| Goodness-of-fit on $F^2$ | 1.037 |
| Final R indices [I>2sigma(I)] | R1 = 0.0311<br>wR2 = 0.0840 |
| R indices (all data) | R1 = 0.0331<br>wR2 = 0.0858 |
| Largest diff. peak and hole | 0.361 and -0.553 e.$Å^{-3}$ |

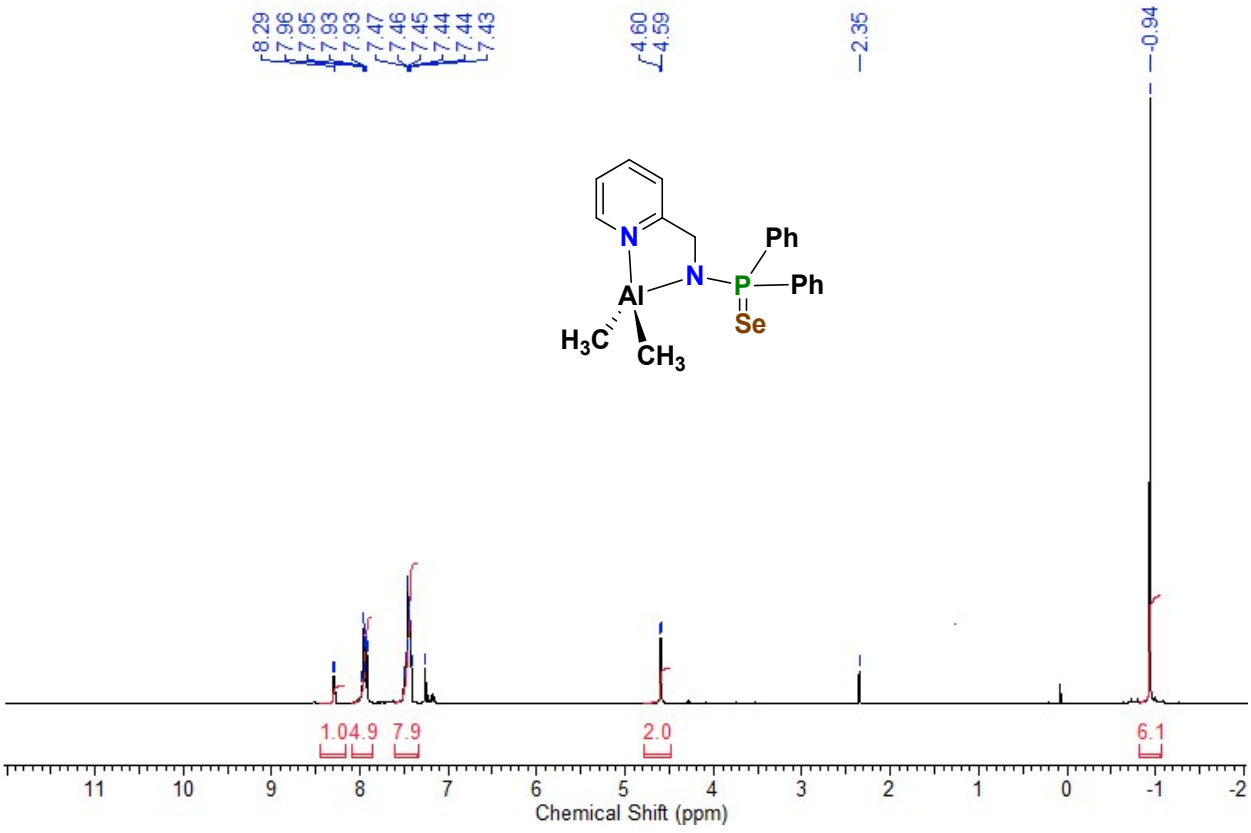



**Figure FS1.** $^1$H NMR spectrum (400 MHz, 25°C, $C_6D_6$) of complex **1.**

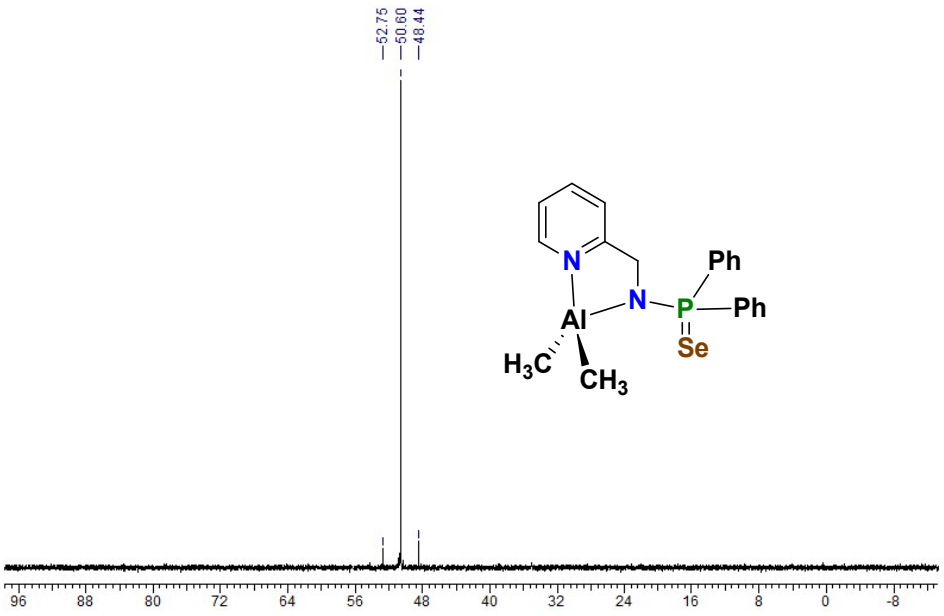



**Figure FS2.** $^{31}P\{^{1}H\}$ NMR spectrum (161.9 MHz, 25°C, $C_6D_6$) of complex **1.**

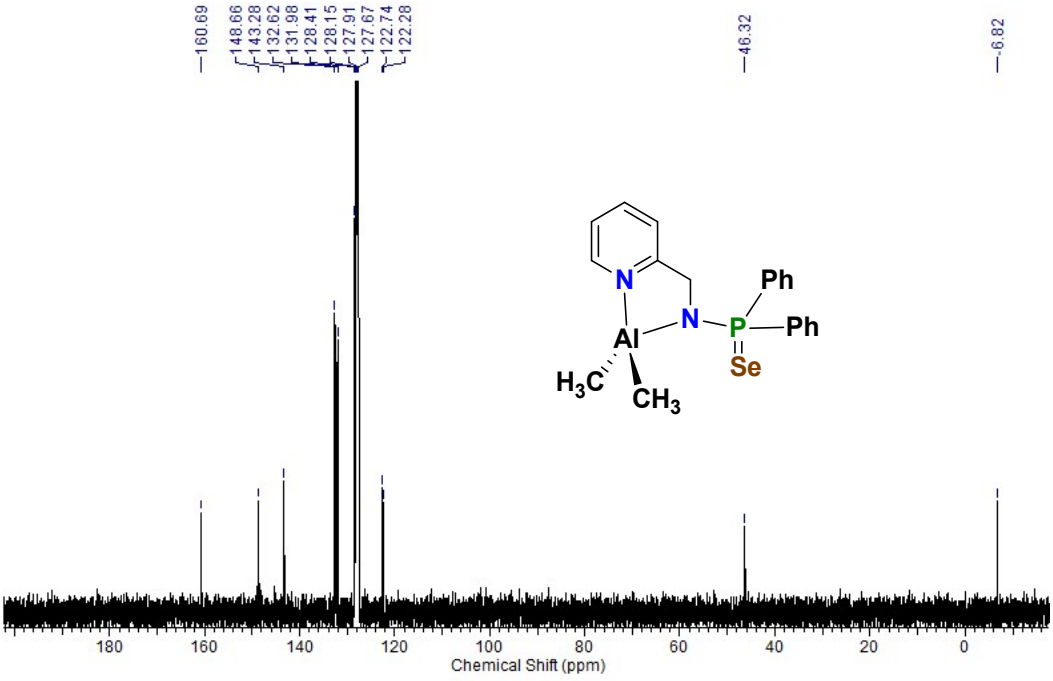


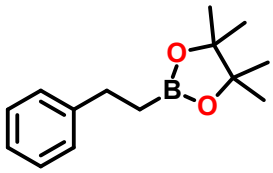

**Figure FS3** $^{13}C\{^{1}H\}$ NMR spectrum (100 MHz, 25°C, $C_6D_6$) of complex **1.**

**General procedure for the synthesis of compounds (2a-2s** and **3a-3m).**

Catalyst **1** (1 mol%), alkenes (**2a-2p)** or alkynes (**3a-3k)** 0.5 mmol and respective boranes such as pinacolborane/ catecholborane (0.6 mmol) were placed in a 25 mL Schlenk flask equipped with a magnetic stir bar inside the glove box. Then the reaction mixture was stirred at 30°C for eight hours The progress of reaction was monitored by $^{1}H$ NMR spectroscopy using hexamethylbenzene (10 mol%) as an internal standard. After the reaction was completed, the resulted boronate ester product was separated by silica-gel column chromatography using hexane as an eluent. Removal of solvent under reduced pressure provided pure products for further analysis. For alkynes hydroboration was carried out in 0.5 ml toluene.

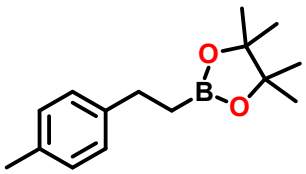
**Characterization Data**

**4,4,5,5-tetramethyl-2-phenethyl-1,3,2-dioxaborolane (2a). [1]**




Yield: [Mass in 232.13 mg (100%), Mass out 227.48 mg (98%)]. $^{1}H$ NMR (400 MHz, $CDCl_3$): $\delta_H$ 7.26 - 7.20 (m, 4H, Ar-*H*), 7.16 - 7.11 (m, 1H, Ar-*H*), 2.74 (t, *J* = 8.0 Hz, 2H, C*H*$_2$), 1.21 (s, 12H, C*H*$_3$), 1.14 (t, *J* = 8.0 Hz, 2H, C*H*$_2$) ppm. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): $\delta_C$ 144.3, 128.1, 127.9, 125.4, 83.0, 29.9, 24.7 ppm. $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$): $\delta_B$ 33.8 ppm

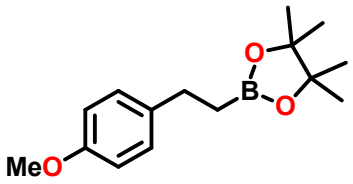
**4,4,5,5-tetramethyl-2-(4-methylphenethyl)-1,3,2-dioxaborolane (2b). [1]**




Yield: [Mass in 246.15 mg (100%), Mass out 221.53 mg (90%)]. $^{1}H$ NMR (400 MHz, $CDCl_3$): $\delta_H$ 7.15 (d, *J* = 8.0 Hz, 2H, Ar-*H*), 7.11 (d, *J* = 8.0 Hz, 2H, Ar-*H*), 2.76 (t, *J* = 8.0 Hz, 2H, C*H*$_2$), 2.35 (s, 3H, C*H*$_3$), 1.27 (s, 12H, C*H*$_3$), 1.17 (t, *J* = 8.0 Hz, 2H, C*H*$_2$) ppm. $^{13}C\{^{1}H\}$ NMR (100

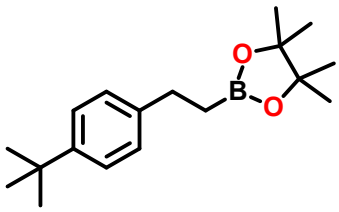
MHz, $CDCl_3$): $\delta_C$ 141.3, 134.7, 128.8, 127.8, 83.0, 29.4, 24.7, 20.9 ppm. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): $\delta_B$ 34.0 ppm

**2-(4-methoxyphenethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2c).** [1]



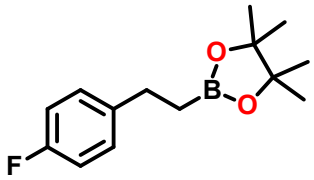

Yield: [Mass in 262.15 mg (100%), Mass out 228.07 mg (87%)]. $^1H$ NMR (400 MHz, $CDCl_3$): **$\delta_H$** 7.15 (d, *J* = 8.0 Hz, 2H, Ar-*H*), 6.82 (d, *J* = 8.0 Hz, 2H, Ar-*H*), 3.78 (s, 3H, C$H_3$), 2.71 (t, *J* = 8.0 Hz, 2H, C$H_2$), 1.23 (s, 12H, C$H_3$), 1.13 (t, *J* = 8.0 Hz, 2H, C$H_2$) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): $\delta_C$ 157.5, 136.4, 128.8, 113.5, 82.9, 55.1, 29.0, 24.7 ppm. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): $\delta_B$ 33.9 ppm.

**2-(4-(tert-butyl)phenethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2d).** [2]



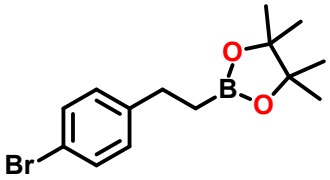

Yield: [Mass in 288.23 mg (100%), Mass out 244.99 mg (85%)]. $^1H$ NMR (400 MHz, $CDCl_3$): $\delta_H$ 7.32 (d, *J* = 8.0 Hz, 2H, Ar-*H*), 7.19 (d, *J* = 8.0 Hz, 2H, Ar-*H*), 2.76 (t, *J* = 8.0 Hz, 2H, C$H_2$), 1.34 (s, 9H, C(C$H_3$)$_3$), 1.26 (s, 12H, C$H_3$), 1.18 (t, *J* = 8.0 Hz, 2H, C$H_2$) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): $\delta_C$ 148.1, 141.3, 127.6, 125.0, 83.0, 34.2, 31.4, 29.3, 24.7 ppm**.** $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): $\delta_B$ 34.1 ppm.

**2-(4-fluorophenethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2d).** [2]



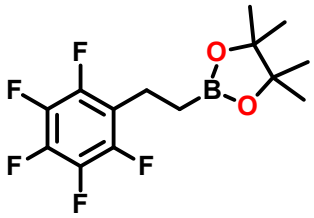
Yield: [Mass in 250.12 mg (100%), Mass out 212.60 mg (85%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 7.18 - 7.15 (m, 2H, Ar-*H*), 6.94 (t, *J* = 8.0 Hz, 2H, Ar-*H*), 2.72 (t, *J* = 8.0 Hz, 2H, C$H_2$), 1.21 (s, 12H, C$H_3$), 1.12 (t, *J* = 8.0 Hz, 2H, C$H_2$) ppm. $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 162.3, 159.8, 139.9, 129.3 (d, *J* = 7.0 Hz), 114.8 (d, *J* = 20.0 Hz), 83.1, 29.1, 24.7 ppm. $^{11}$B{$^{1}$H} NMR (128 MHz, $CDCl_3$): $\delta_B$ 33.5 ppm

**2-(4-Bromophenethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2e).** [3]



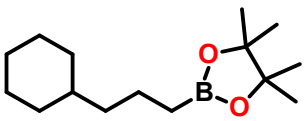

Yield: [Mass in 311.02 mg (100%), Mass out 267.47 mg (86%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 7.37 (d, *J* = 8.0 Hz, 2H, Ar-*H*), 7.09 (d, *J* = 8.0 Hz, 2H, Ar-*H*), 2.70 (t, *J* = 8.0 Hz, 2H, C$H_2$), 1.22 (s, 12H, C$H_3$), 1.11 (t, *J* = 8.0 Hz, 2H, C$H_2$) ppm. $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 143.3, 131.1, 129.8, 119.1, 83.1, 29.3, 24.7 ppm. $^{11}$B{$^{1}$H} NMR (128 MHz, $CDCl_3$): $\delta_B$ 34.0 ppm.

**4,4,5,5-tetramethyl-2-(2-(perfluorophenyl)ethyl)-1,3,2-dioxaborolane (2f).** [1]



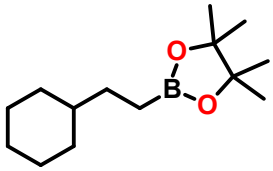

Yield: [Mass in 332.08 mg (100%), Mass out 265.66 mg (80%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 2.80 - 2.75 (m, 2H, C$H_2$), 1.22 (s, 12H, C$H_3$), 1.11 - 1.06 (m, 2H, C$H_2$) ppm. $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 146.2 - 146.0 (m, CF), 143.7-143.6 (m, CF), 140.5 - 140.4 (m, CF), 138.5 - 138.0 (m, CF), 127.3 - 116.9 (m, CF), 83.4, 24.7, 16.9 ppm**.** $^{11}$B{$^{1}$H} NMR (128 MHz, $CDCl_3$): $\delta_B$ 33.2 ppm

**2-(3-cyclohexylpropyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane**



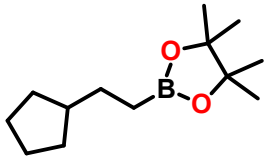
Yield: [Mass in 252.20 mg (100%), Mass out 214.37 mg (85%)] : $\delta_H$ 1.69 - 1.63 (m, 6H, C$H_2$), 1.41 - 1.37 (m, 2H, C$H_2$), 1.22 (s, 12H, C$H_3$), 1.17 - 1.12 (m, 5H, C$H_2$), 0.88 - 0.81 (m, 2H, C$H_2$), 0.72 (t, $J$ = 8.0 Hz, 2H, C$H_2$) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): $\delta_C$ 82.7, 40.4, 37.4, 33.3, 26.7, 26.4, 24.7, 21.2 ppm. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): $\delta_B$ 34.1 ppm.

**2-(2-cyclohexylethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane(2h). [2]**



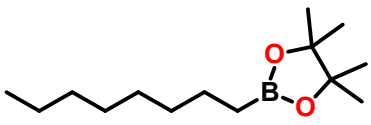
Yield: [Mass in 238.17 mg (100%), Mass out 197.68 mg (83%)]: $\delta_H$ 1.69 - 1.59 (m, 4H, C$H_2$), 1.43 - 1.35 (m, 1H, C$H$), 1.22 (s, 12H, C$H_3$), 1.17 - 1.12 (m, 4H, C$H_2$), 0.88 - 0.81 (m, **2**H, C$H_2$), 0.72 (t, $J$ = 8.0 Hz, 2H, C$H_2$) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): $\delta_C$ 82.7, 40.4, 37.4, 33.4, 26.5 (d, $J$ = 33.0 Hz), 21.2 ppm. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): $\delta_B$ 34.2 ppm.

**2-(2-cyclopentylethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2i).**



Yield: [Mass in 224.15 mg (100%), Mass out 190.52 mg (85%)]. $^1H$ NMR (400 MHz, $CDCl_3$): $\delta_H$ 1.74 - 1.65 (m, 3H, C$H_2$), 1.58 - 1.51 (m, 2H, C$H_2$), 1.49 - 1.44 (m, 1H, C$H$), 1.39 (q, $J$ = 8.0 Hz, 2H, C$H_2$), 1.23 (s, 12H, C$H_3$), 1.10 - 1.03 (m, 2H, C$H_2$), 0.76 (t, $J$ = 8.0 Hz, 2H, C$H_2$) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): $\delta_C$ 82.8, 42.6, 32.3, 30.1, 25.2, 24.7 ppm**.** $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): $\delta_B$ 33.1 ppm

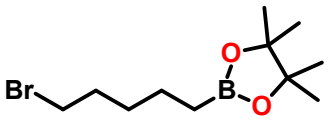
**4,4,5,5-tetramethyl-2-octyl-1,3,2-dioxaborolane (2j).**



Yield: [Mass in 240.19 mg (100%), Mass out 216.17 mg (90%)]. $^1H$ NMR (400 MHz, $CDCl_3$): $\delta_H$ 1.39 - 1.34 (m, 2H, C$H_2$), 1.27 - 1.24 (m, 10H, C$H_2$), 1.22 (s, 12H, C$H_3$), 0.85 (t, $J$ = 8.0 Hz,

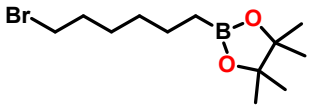
2H, C$H_3$), 0.74 (t, $J$ = 8.0 Hz, 2H, C$H_2$) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): $\delta_C$ 82.7, 32.4, 31.8, 29.3 (d, $J$ = 13.0 Hz), 24.7, 23.9, 22.6, 14.0 ppm. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): $\delta_B$34.1 ppm.

**2-(5-bromopentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2k).**



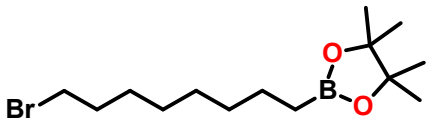

Yield: [Mass in 277.01 mg (100%), Mass out 249.30 mg (90%)]. $^1H$ NMR (400 MHz, $CDCl_3$): $\delta_H$ 3.40 (t, $J$ = 8.0 Hz, 2H, C$H_2$), 1.85 (t, $J$ = 8.0 Hz, 2H, C$H_2$), 1.45 - 1.41 (q, 4H, C$H_2$), 1.24 (s, 12H, C$H_3$), 0.78 (t, $J$ = 8.0 Hz, 2H, C$H_2$) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): $\delta_C$ 83.0 (d, J = 15.0 Hz), 33.9, 32.6, 30.8, 24.7, 23.1 ppm. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): $\delta_B$ 33.3 ppm.

**2-(6-bromohexyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2l)**



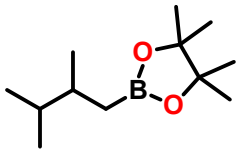

Yield: [Mass in 291.03 mg (100%), Mass out 261.92 mg (90%)]. $^1H$ NMR (400 MHz, $CDCl_3$): $\delta_H$ 3.38 (t, $J$ = 8.0 Hz, 2H, C$H_2$), 1.87 - 1.80 (q, 2H, C$H_2$), 1.45 - 1.37 (m, 4H, C$H_2$), 1.33 - 1.28 (m, 2H, C$H_2$), 1.23 (s, 12H, C$H_3$), 0.76 (t, $J$ = 8.0 Hz, 2H, C$H_2$) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): $\delta_C$ 82.8, 33.9, 32.6, 31.3, 27.8, 24.7, 23.7 ppm. $^{11}B\{^1H\}$ NMR (128 MHz, $CDCl_3$): $\delta_B$ 34.4 ppm.

**2-(8-bromooctyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2m).**



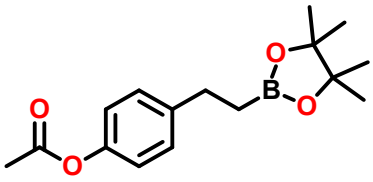

Yield: [Mass in 319.09 mg (100%), Mass out 280.79 mg (88%)]. $^1H$ NMR (400 MHz, $CDCl_3$) $\delta_H$ 3.37 (t, 2H), 1.84 - 1.80 (m, 2H), 1.39 - 1.36 (m, 4H), 1.29 – 1.27 (m, 6H), 1.22 (s, 12H), 0.74 (t, 2H) ppm. $^{13}C(^1H)$ NMR (100 MHz, $CDCl_3$) $\delta_C$ 82.7, 33.9, 32.7, 32.1, 29.0, 28.5, 28.0, 24.7, 23.8 ppm. $^{11}B(^1H)$ NMR (128 MHz, $CDCl_3$) $\delta_B$ 34.0 ppm.

**2-(2,3-dimethylbutyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2n).**



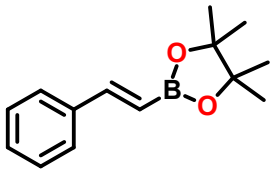
Yield: [Mass in 212.14 mg (100%), Mass out 190.92 mg (90%)]. $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta_H$ 71.58 - 1.53 (m, 1H), 1.47 – 1.42 (m, 1H), 1.22 (s, 12H), 0.83 - 0.78 (m, 10H), 0.61 – 0.55 (m, 1H) ppm. $^{13}$C($^{1}$H) NMR (100 MHz, $CDCl_3$) $\delta_C$ 82.7, 35.0, 34.1, 24.8, 24.6, 19.7, 18.6 ppm. $^{11}$B($^{1}$H) NMR (128 MHz, $CDCl_3$) $\delta_B$ 34.3ppm.

**4-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)ethyl)phenyl acetate (2o). [3]**



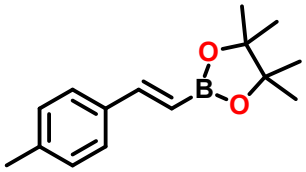
Yield: [Mass in 290.16 mg (100%), Mass out 261.14 mg (90%)]. $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta_H$ 7.21 (d, 2H), 6.96 (d, 2H), 2.74 (t, 2H), 2.27 (s, 3H), 1.21 (s, 12H), 1.13 (t, 2H) ppm. $^{13}$C($^{1}$H) NMR (100 MHz, $CDCl_3$) $\delta_C$ 169.6, 148.4, 141.8, 128.8, 121.0, 83.0, 29.2, 24.7, 21.0 ppm. $^{11}$B($^{1}$H) NMR (128 MHz, $CDCl_3$) $\delta_B$ 34.2 ppm.

**(E)-4,4,5,5-tetramethyl-2-styryl-1,3,2-dioxaborolane (3a). [4]**



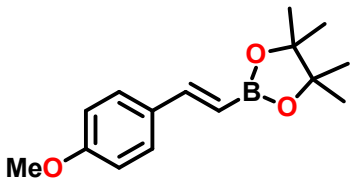
Yield: [Mass in 230.11 mg (100%), Mass out 227.8 mg (99%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 7.42 - 7.40 (m, 2H), 7.36, (d, 1H, $J$ = 16 Hz), 7.25 - 7.20 (m, 3H), 6.10 (d, 1H, $J$ = 16 Hz), 1.24 (s, 12H, C$H_3$, Bpin) ppm. $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 148.0, 133.5, 131.7, 131.6, 128.5, 122.9, 83.4, 24.5 ppm. $^{11}$B{$^{1}$H} NMR (128.3 MHz, $CDCl_3$): $\delta_B$ 30.4 (C-Bpin) ppm.

**(E)-4,4,5,5-tetramethyl-2-(4-methylstyryl)-1,3,2-dioxaborolane (3b). [4]**



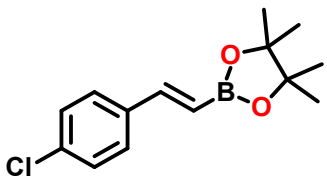
Yield: [Mass in 244.14 mg (100%), Mass out 239.3 mg (98%)]. $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta_H$ 7.42 - 7.37 (m, 3H), 7.15 (d, *J* = 8 Hz, 2H), 6.13 (d, *J* = 20.0 Hz, 1H), 2.35 (s, 3H), 1.32 (s, 12H) ppm. $^{13}C\{^{1}H\}$ NMR (100 MHz, CDCl3) $\delta_C$ 149.5, 138.9, 134.8, 132.0, 129.3,127.0, 83.2, 24.8, 21.3 ppm. $^{11}B\{^{1}H\}$ NMR (128.3 MHz, CDCl3) $\delta_B$ 29.9 ppm.

**(E)-2-(4-methoxystyryl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3c).** [4]



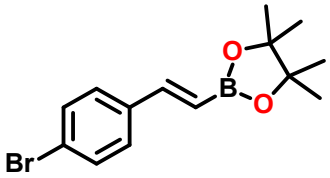
Yield: [Mass in 260.2 mg (100%), Mass out 206 mg (82%)]. $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta_H$ 7.36 (d, *J* = 8 Hz, 2H), 7.33 (d, *J* = 20 Hz, 1H), 6.77 (d, *J* = 8 Hz, 2H), 5.95 (d, *J* = 20 Hz, 1H), 3.68 (s, 3H), 1.22 (s, 12H) ppm. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) $\delta_C$ 160.3, 149.1, 133.5, 129.0, 128.4, 114.0, 83.2, 55.2, 24.8 ppm. $^{11}B\{^{1}H\}$ NMR (128.3 MHz, $CDCl_3$): $\delta_B$ 29.9(C-Bpin) ppm.

**(E)-2-(4-chlorostyryl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3e).** [4]



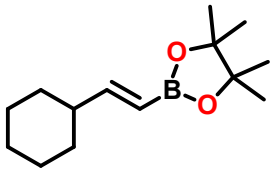
Yield: [Mass in 309.1 mg (100%), Mass out 302.9 mg (98%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 7.48 - 7.44 (m, 2H, ArH), 7.36 (d, 1H, *J* = 20 Hz), 7.04 - 7.00 (m, 2H, ArH), 6.07 (d, 1H, *J* =20 Hz), 1.31 (s, 12H, $CH_3$, Bpin) ppm. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): $\delta_C$ 164.3, 161.9, 148.1, 133.7, 128.7, 115.6, 83.4, 24.8 ppm. $^{11}B\{^{1}H\}$ NMR (128.3 MHz, $CDCl_3$): $\delta_B$ 29.7 (C-Bpin) ppm.

**(E)-2-(4-bromostyryl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3e).** [4]



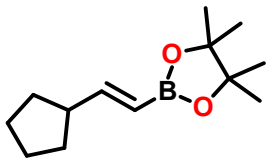
Yield: [Mass in 309.1 mg (100%), Mass out 302.9 mg (98%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 7.46 - 7.44 (m, 2H, ArH), 7.35 - 7.30 (m, 3H, ArH), 6.15 (d, 1H, $J$ = 20 Hz), 1.31 (s, 12H, $CH_3$, Bpin) ppm. $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 160.3, 149.1, 133.5, 129.0, 128.4, 113.9, 83.2, 24.8 ppm. $^{11}$B{$^{1}$H} NMR (128.3 MHz, $CDCl_3$): $\delta_B$ 29.8 (C-Bpin) ppm.

**(E)-2-(2-cyclohexylvinyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3f).** [4]



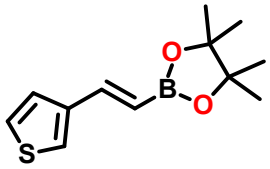
Yield: [Mass in 236.2 mg (100%), Mass out 229.11 mg (97%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 6.59 (dd, 1H, $J_{HH}$ =12 Hz, 8 Hz), 5.38 (d, 1H, $J$ =20 Hz), 2.03 - 2.01 (m, 1H), 1.75 - 1.71 (m, 6H), 1.27 (s, 12H, $CH_3$, Bpin) 1.25 - 1.03 (m, 4H), ppm. $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 159.8, 82.9, 43.2, 31.9, 26.1, 25.9, 24.7, 24.5 ppm. $^{11}$B{$^{1}$H} NMR (128.3 MHz, $CDCl_3$): $\delta_B$ 30.4 (C-Bpin) ppm.

**(E)-2-(2-cyclopentylvinyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3g).** [4]



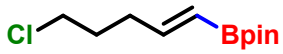
Yield: [Mass in 222.13 mg (100%), Mass out 211.02 mg (95%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 6.63 (dd, 1H, $J_{HH}$ =12 Hz, 8 Hz), 5.42 (d, 1H, $J$ = 20 Hz), 2.56 - 2.54 (m, 1H), 1.81 - 1.65(m, 6H), 1.59 - 1.39 (m, 2H), 1.23 (s, 12H, $CH_3$, Bpin) ppm. $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 158.9, 82.9, 46.1, 32.3, 25.4, 24.7, 24.5 ppm. $^{11}$B{$^{1}$H} NMR (128.3 MHz, $CDCl_3$): $\delta_B$ 30.6 (C-Bpin) ppm.

**(E)-4,4,5,5-tetramethyl-2-(2-(thiophen-3-yl)vinyl)-1,3,2-dioxaborolane (3h).** [4]



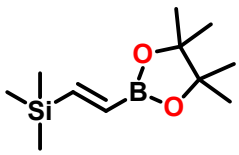
Yield: [Mass in 236.14 mg (100%), Mass out 233.78 mg (99%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 7.305 (d, $J$ =20, 1H), 7.28 – 7.27 (m, 2H, ArH), 7.24 – 7.24 (m, 1H, ArH), 5.93 (d, $J$ = 20 Hz, 1H), 1.28 (s, 12H, $CH_3$, Bpin) ppm. $^{13}$C-{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 143.0, 141.1, 126.0, 124.9 83.2, 24.7ppm. $^{11}$B{$^{1}$H} NMR (128.3 MHz, $CDCl_3$): $\delta_B$ 29.6 (C-Bpin) ppm.

**(E)-2-(5-chloropent-1-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (2j).**



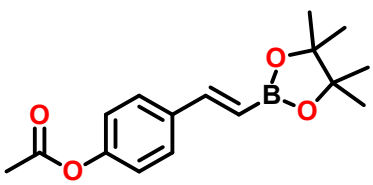
The compound was isolated as a colourless oil. Yield: [Mass in 230.12 mg (100%), Mass out 227.82 mg (99%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 6.61 - 6.53 (m, 1H), 5.48 (d, 1H, $J$ = 20 Hz), 3.52 (t, 2H, $J$ = 8 Hz), 2.34 - 2.27 (m, 2H), 1.92 - 1.85 (m, 2H), 1.25 (s, 12H, $CH_3$, Bpin) ppm. $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 152.0, 83.0, 44.2, 32.6, 30.9, 24.5 ppm. $^{11}$B{$^{1}$H} NMR (128.3 MHz, $CDCl_3$): $\delta_B$ 31.6 (C-Bpin) ppm.

**(E)-trimethyl(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)vinyl)silane (3j). [4]**



Yield: [Mass in 226.20 mg (100%), Mass out 205.9 mg (91%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 6.02 (d, $J$.=8 Hz, 1H), 5.84 (d, 4Hz, 1H), 1.27 (s, 12H, $CH_3$, Bpin), 0.07 (s, 9H, $SiMe_3$) ppm. $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 157.8, 134.4, 83.0, 24.5, -1.88 ppm. $^{11}$B{$^{1}$H} NMR (128.3 MHz, $CDCl_3$): $\delta_B$ 30.9 (C-Bpin) ppm.

**(E)-4-(2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)vinyl)phenyl acetate (3k). [1]**



Yield: [Mass in 288.15 mg (100%), Mass out 273.75 mg (95%)]. $^{1}$H NMR (400 MHz, $CDCl_3$): $\delta_H$ 7.88 - 7.86 (m, 2H), 7.41 - 7.39 (m, 2H), 7.07 (d, $J$ = 20 Hz, 1H), 6.15 (d, $J$ = 16Hz, 1H) 3.77 (s, 3H, $OCH_3$), 1.18 (s, 12H, $CH_3$, Bpin) ppm. $^{13}$C{$^{1}$H} NMR (100 MHz, $CDCl_3$): $\delta_C$ 166.7, 148.1, 141.7, 129.9, 129.0, 126.8, 125.2, 83.5, 52.0, 24.8 ppm. $^{11}$B{$^{1}$H} NMR (128.3 MHz, $CDCl_3$): $\delta_B$ 29.5 (C-Bpin) ppm.

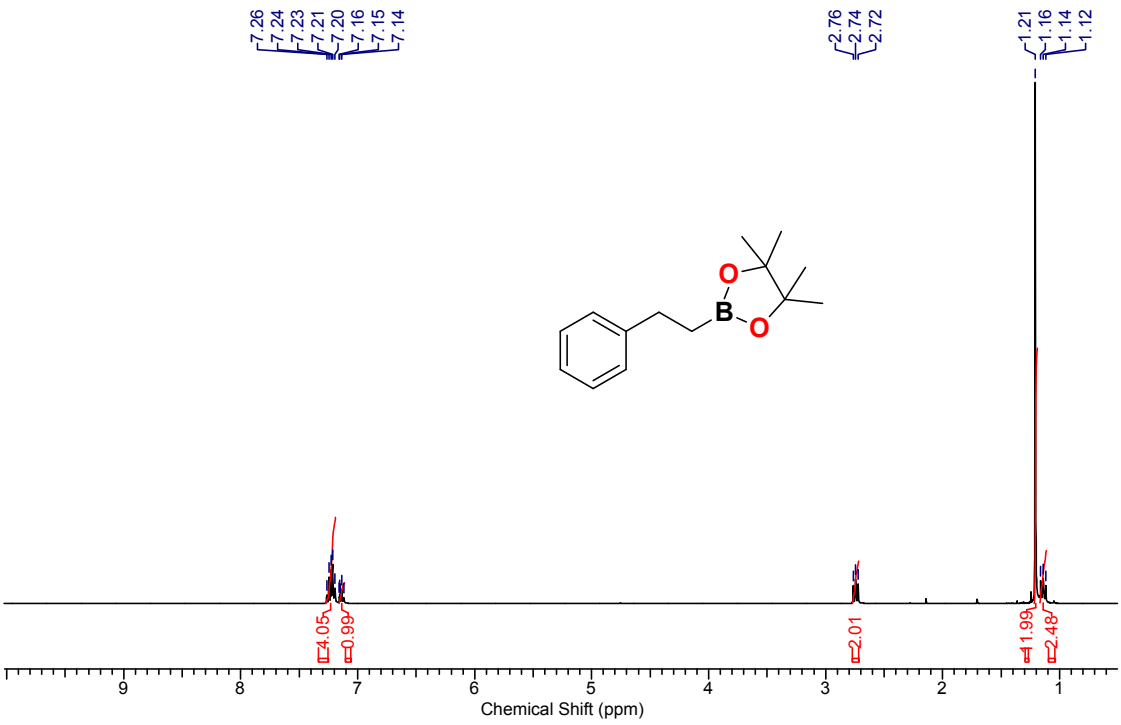



**Figure FS4.** $^{1}$H NMR (400 MHz, $CDCl_3$) spectrum of **2a**.

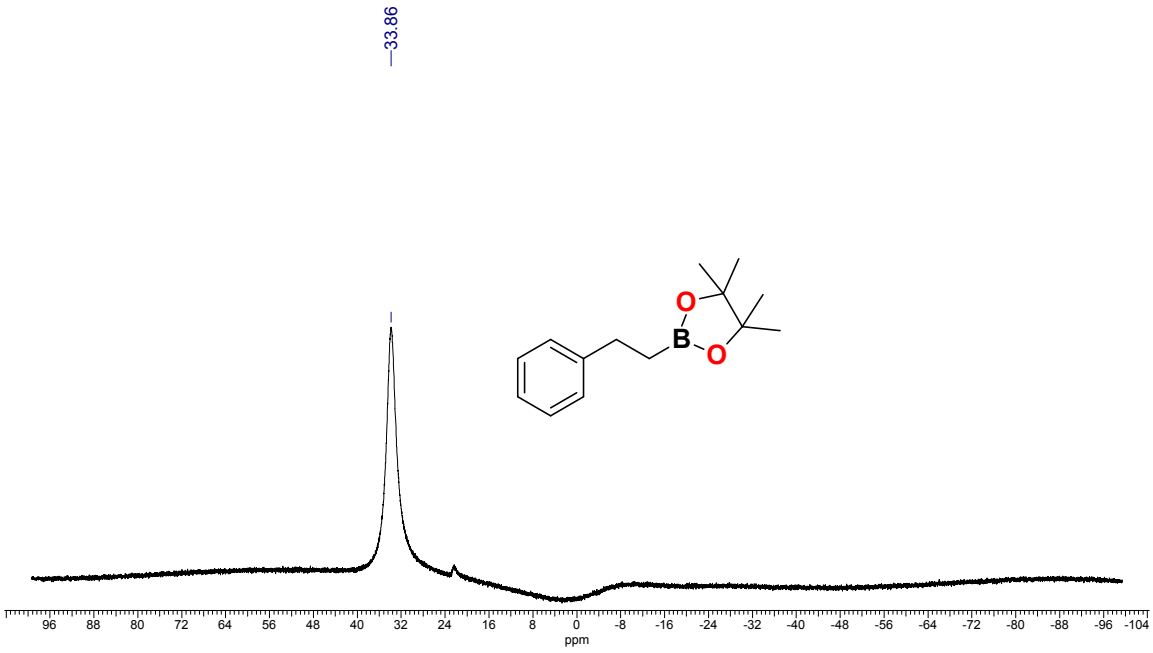



**Figure FS5.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**a**

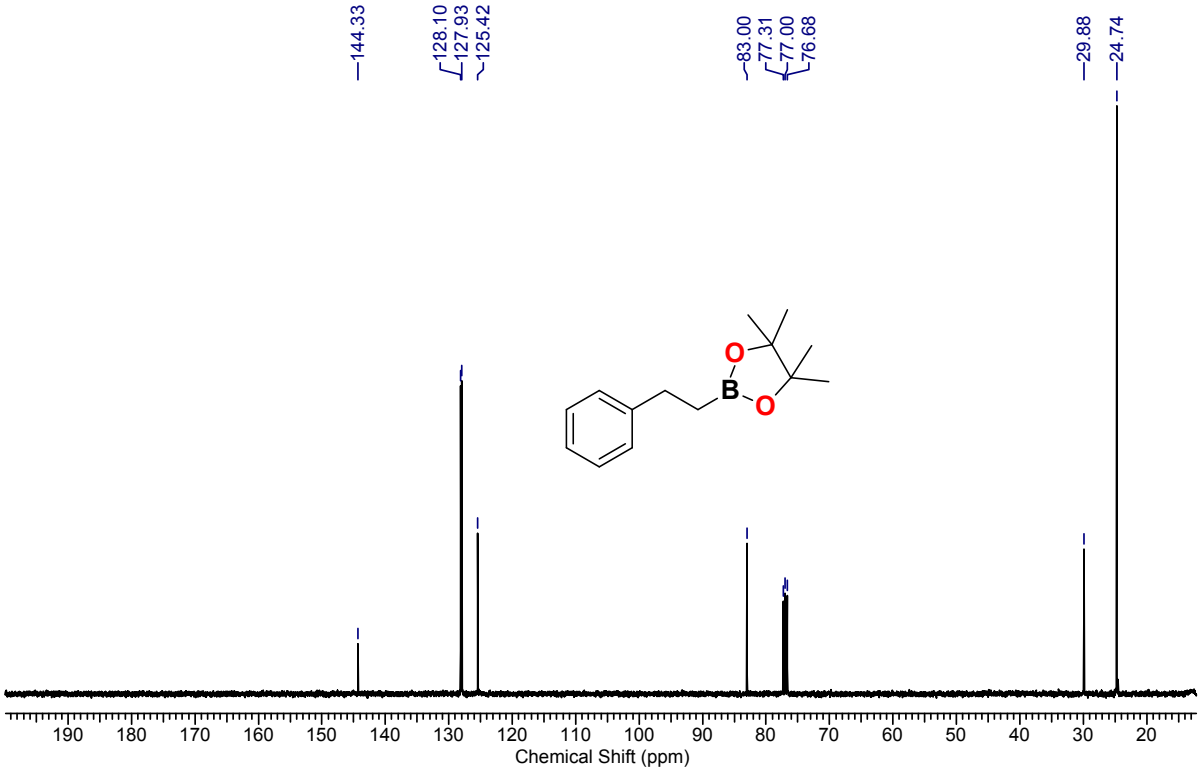



**Figure FS6.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2a.**

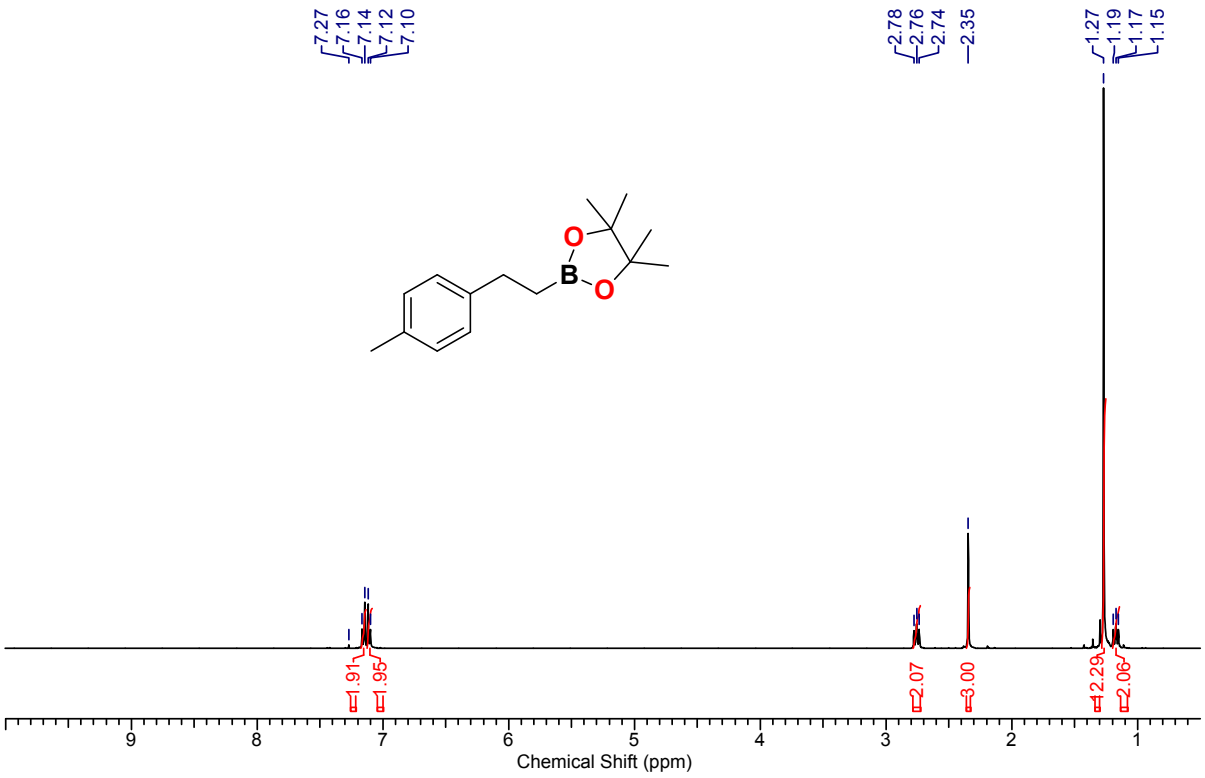



**Figure FS7.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2b.**

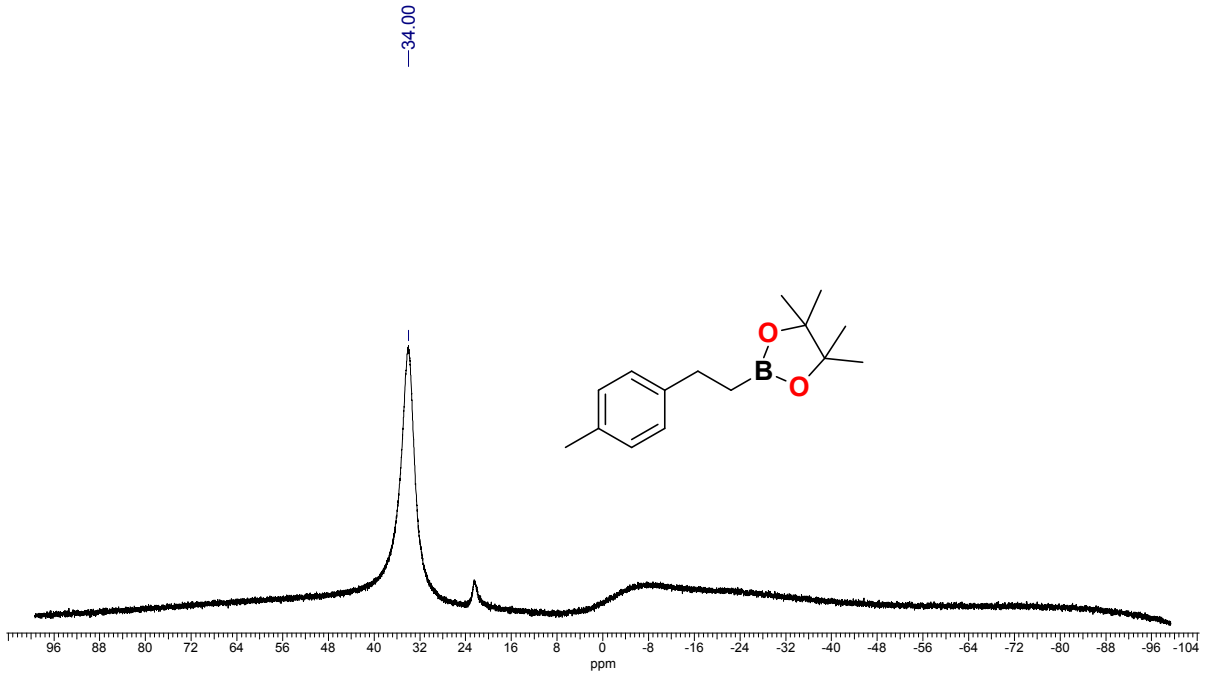



**Figure FS8.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **2b.**

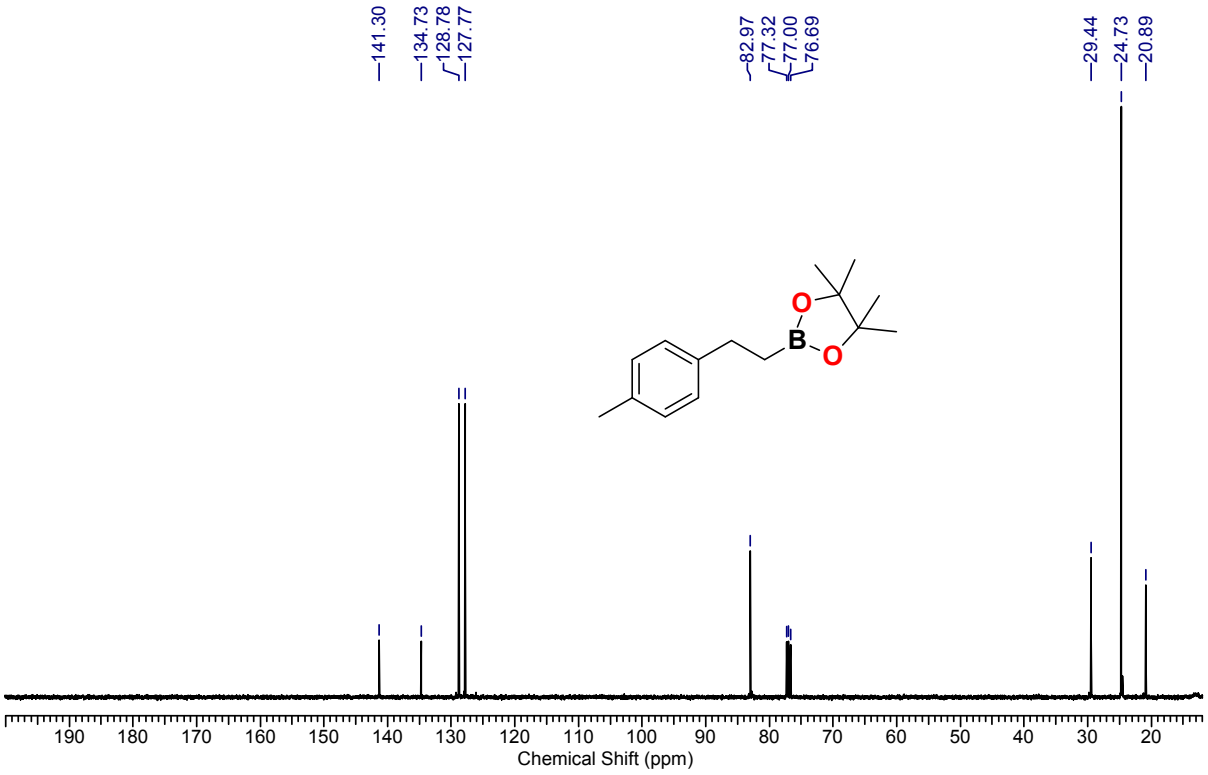



**Figure FS9.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2b.**

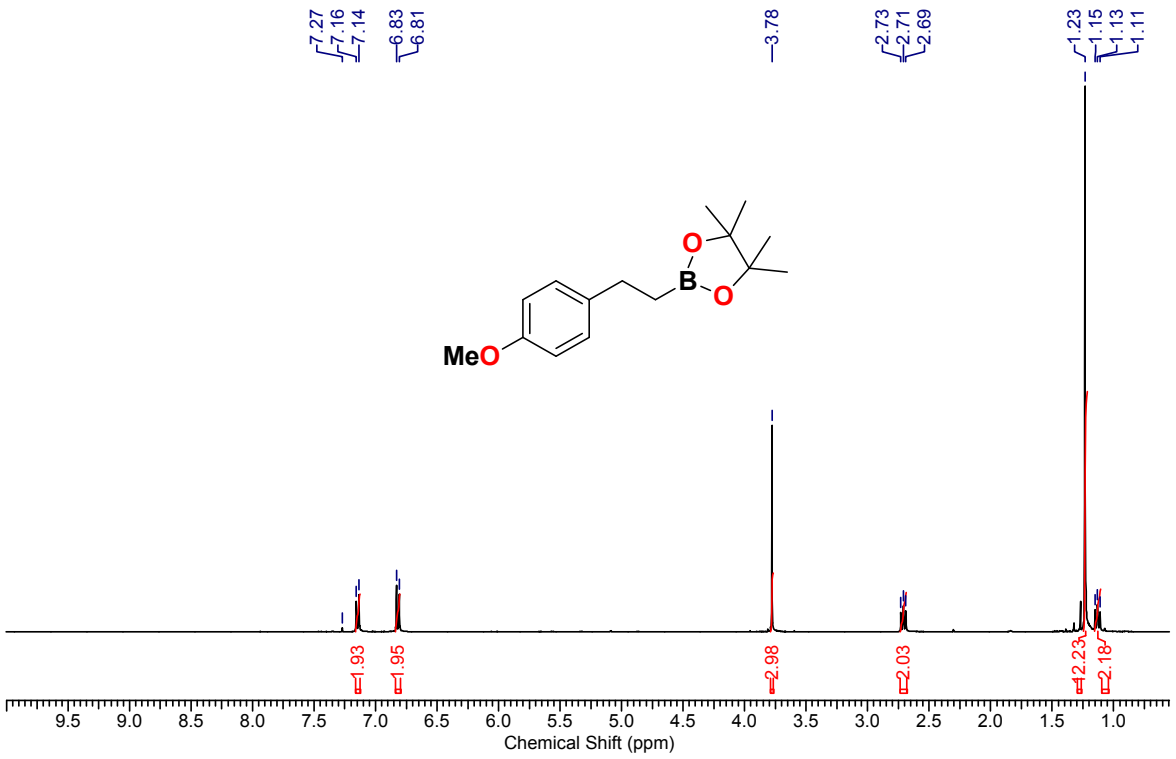



**Figure FS10.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2c.**

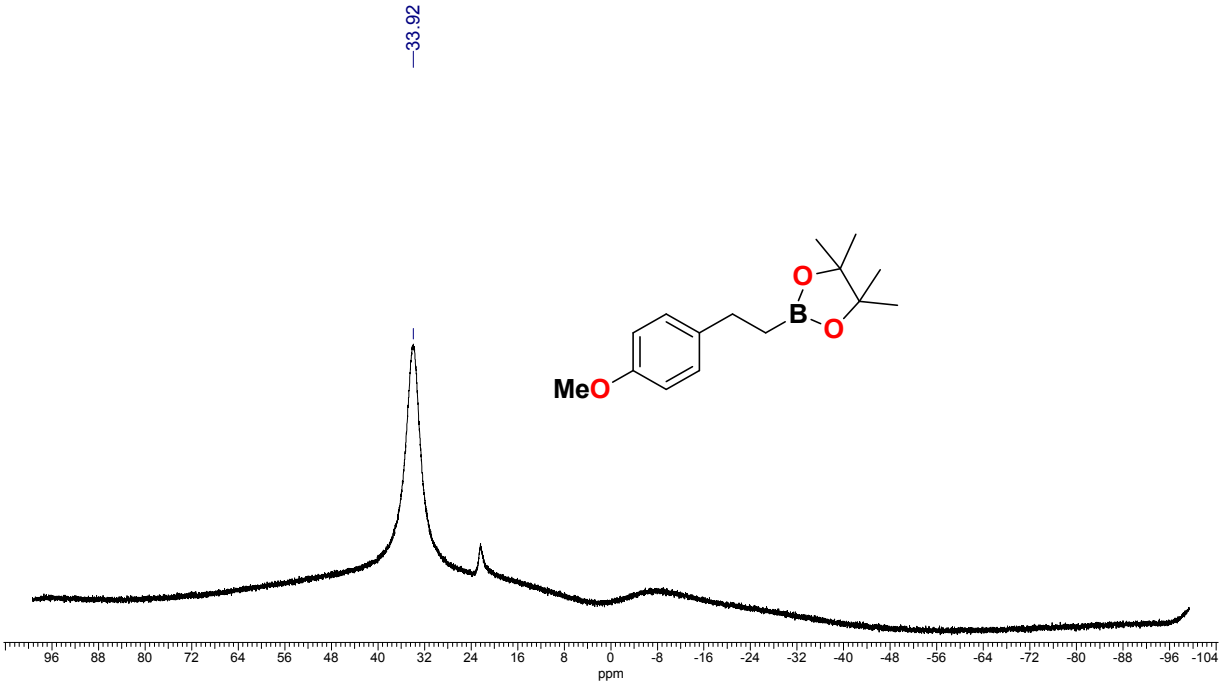



**Figure FS11.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**c.**

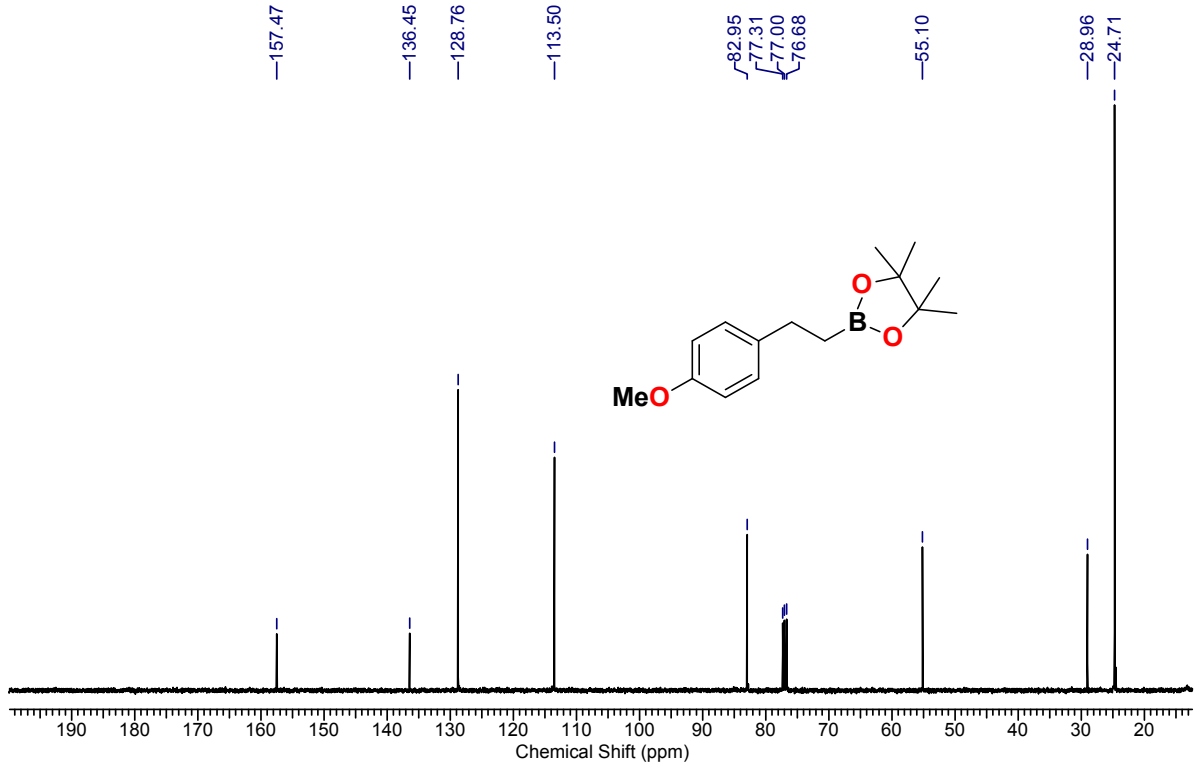



**Figure FS12.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2c.**

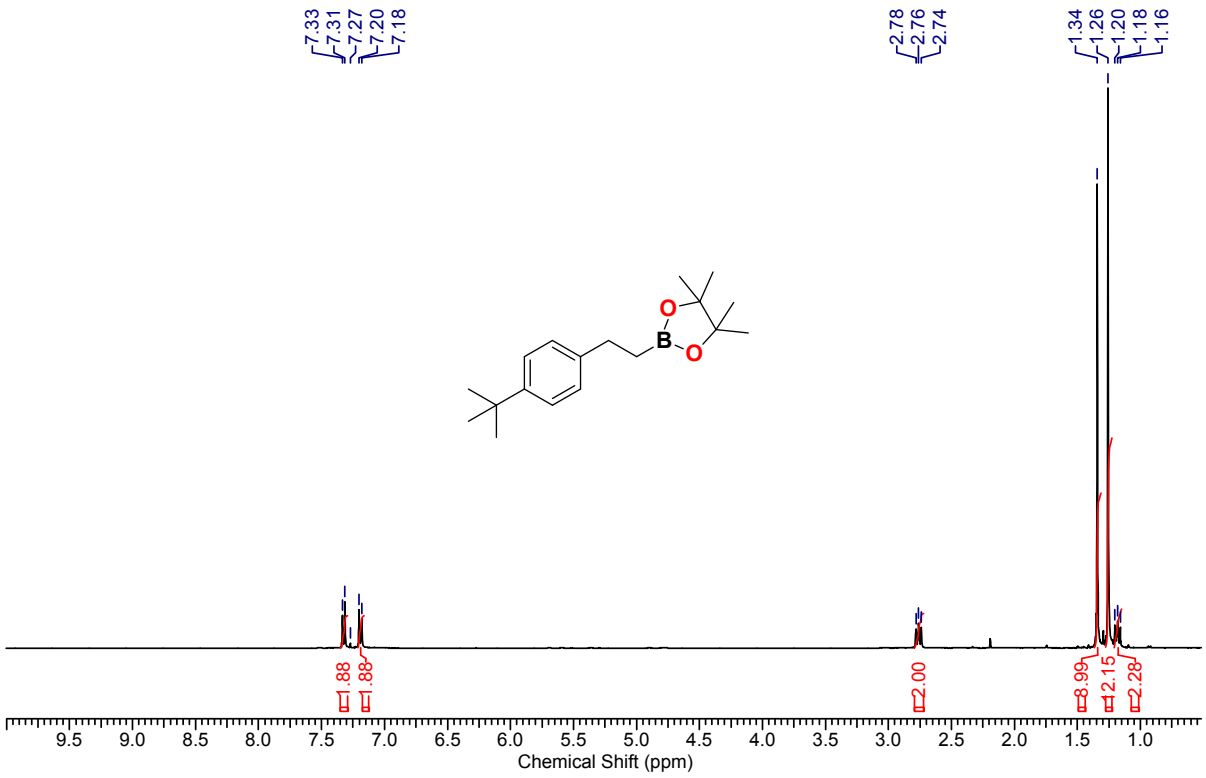



**Figure FS13.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2d.**

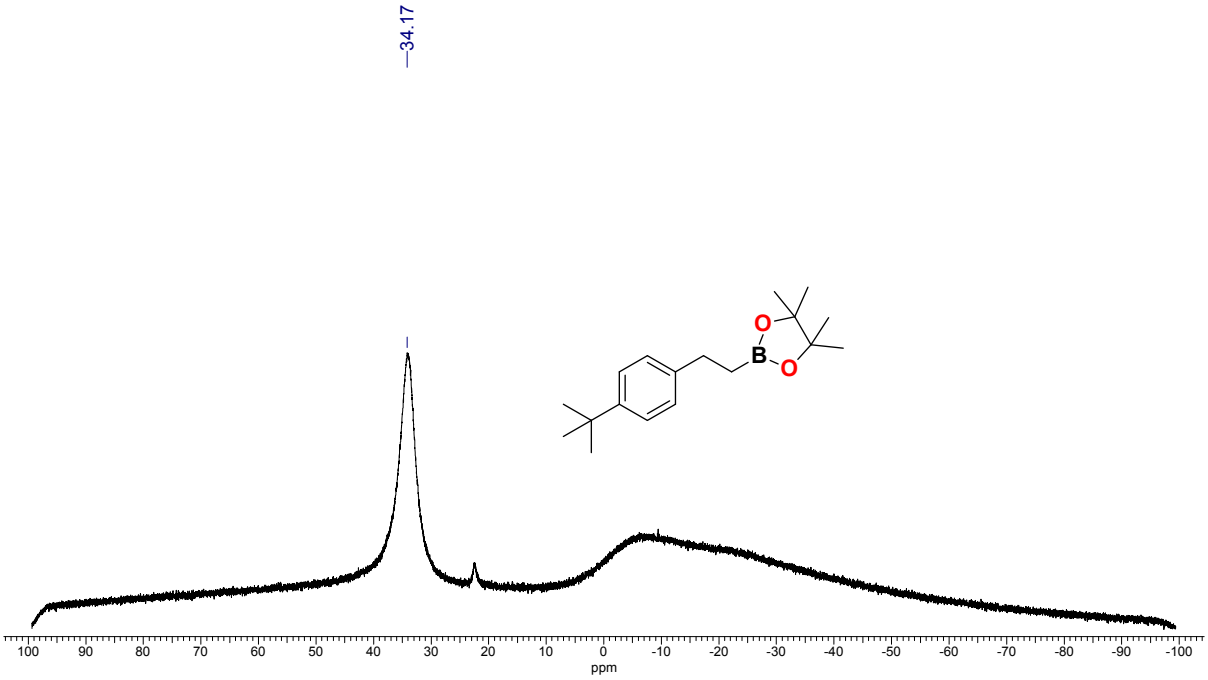



**Figure FS14.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**d.**

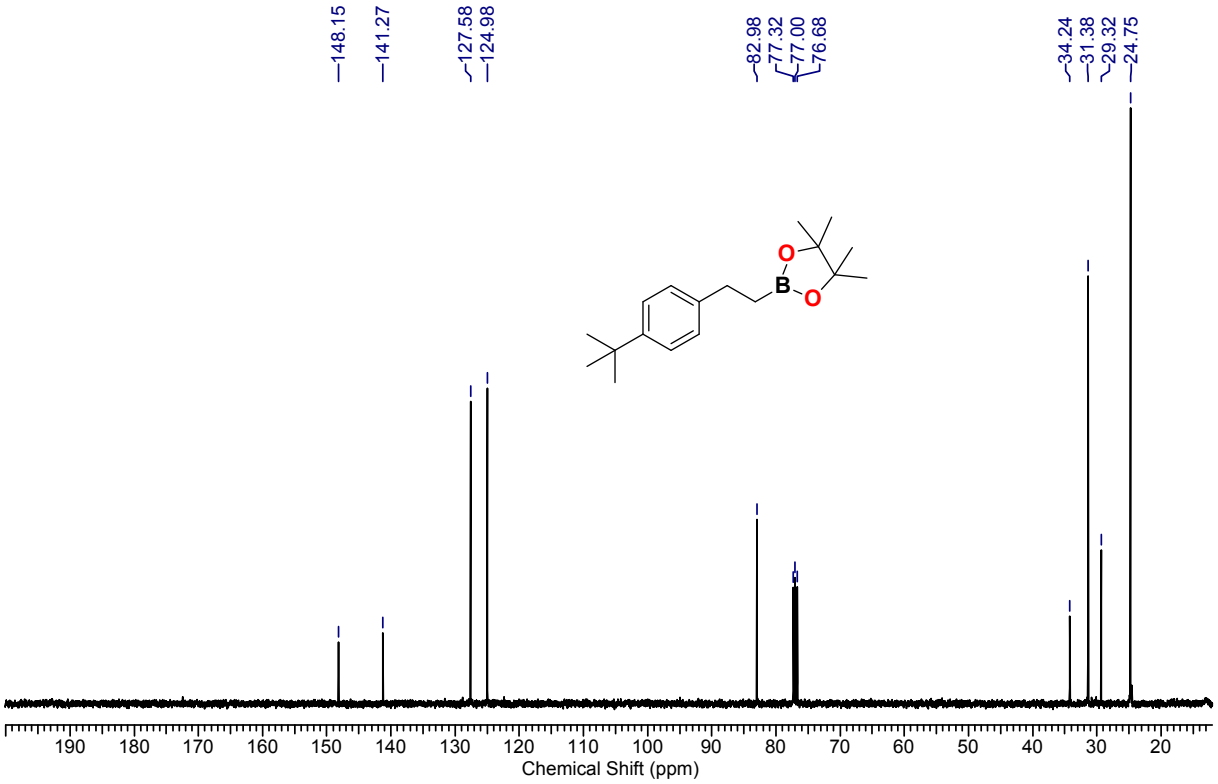



**Figure FS15.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2d.**

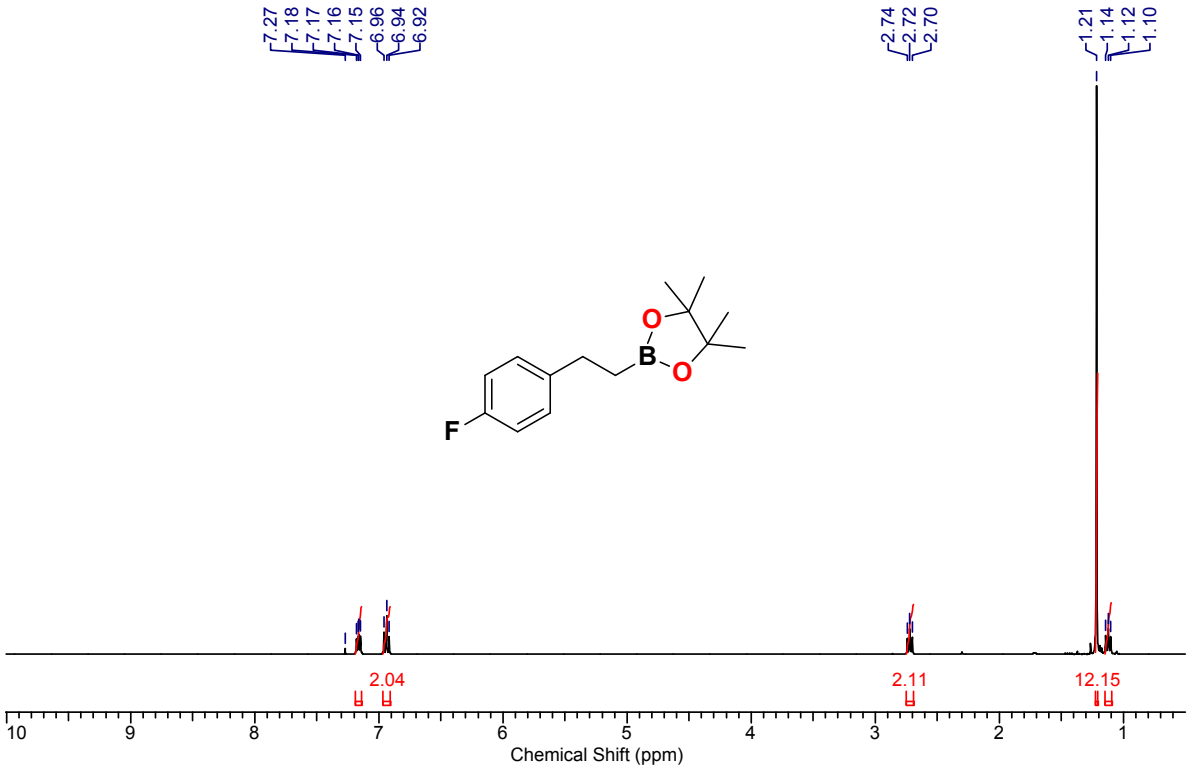



**Figure FS16.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2e.**

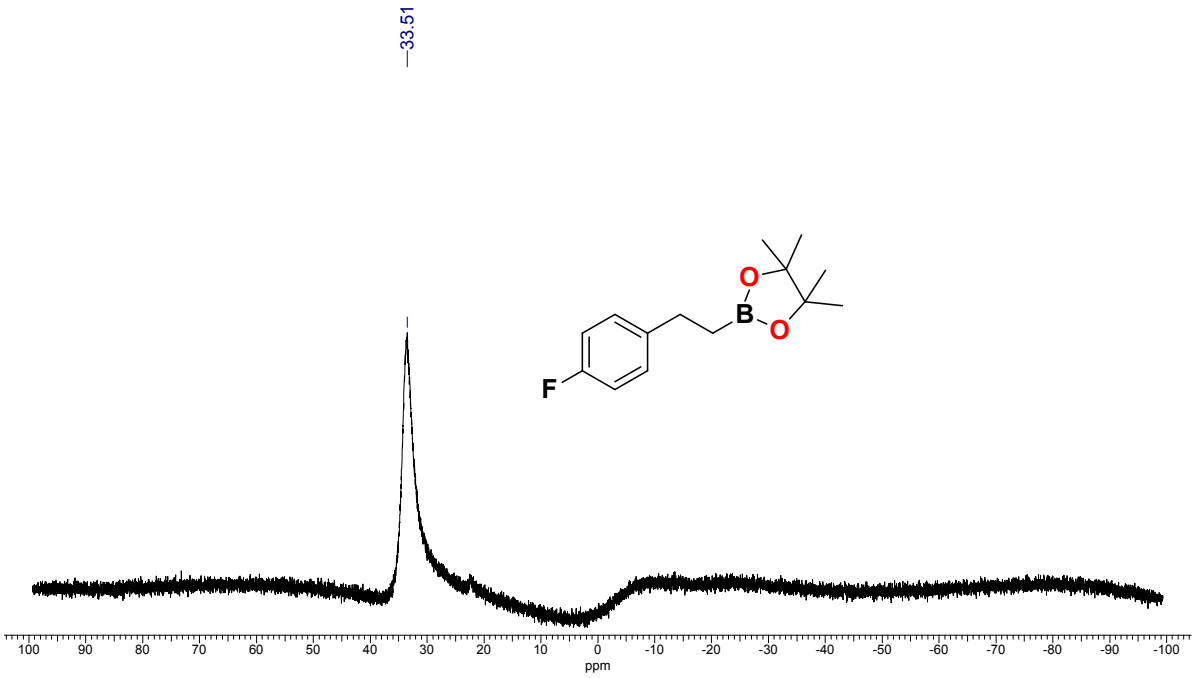



**Figure FS17.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**e.**

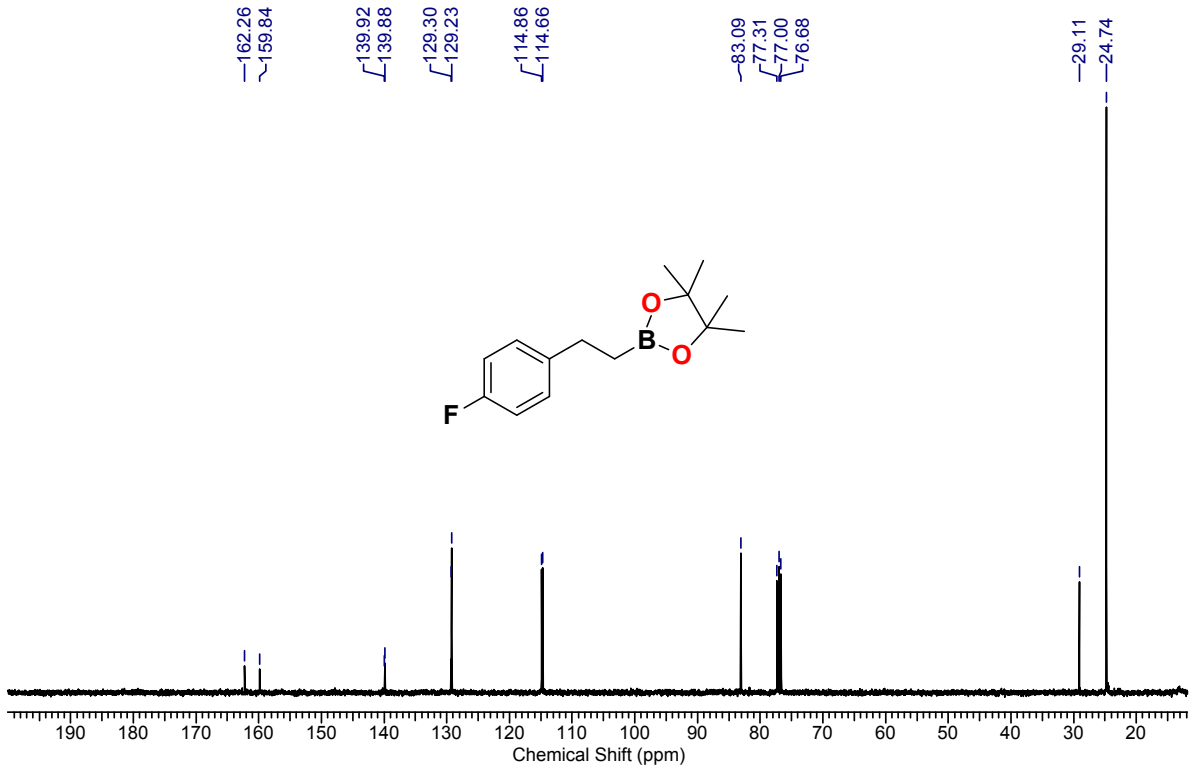



**Figure FS18.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2e**.

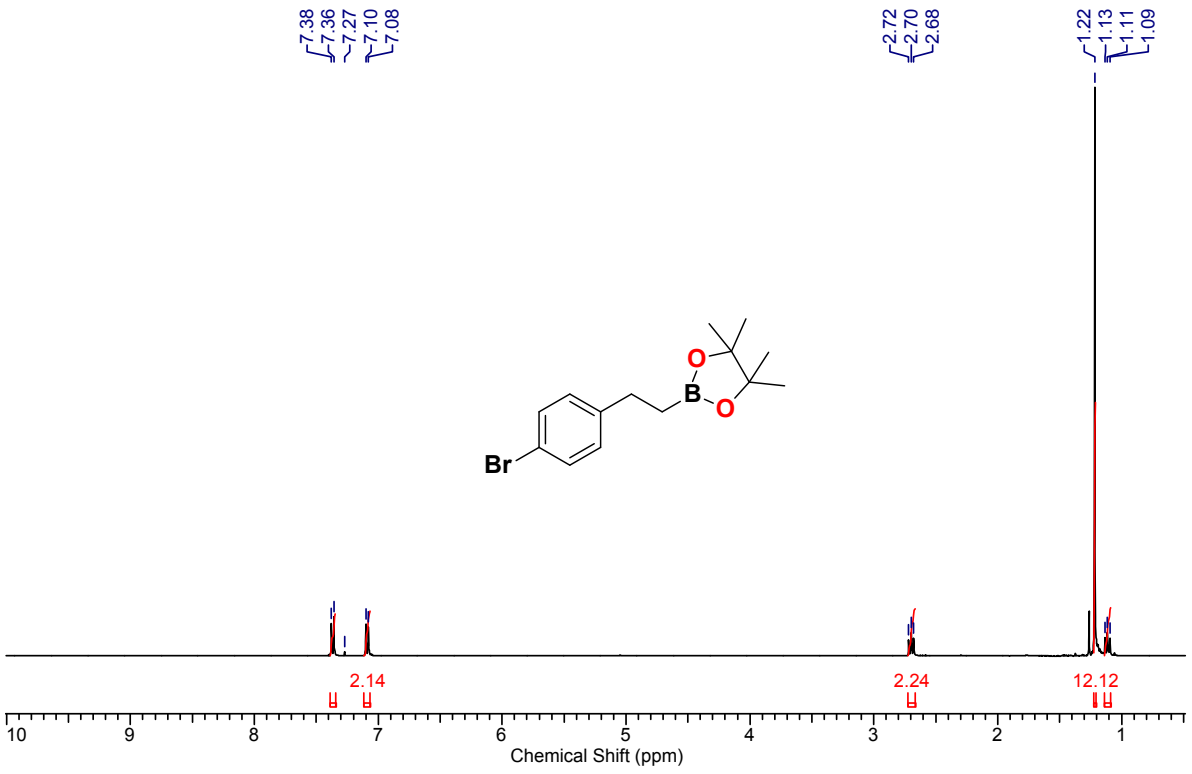



**Figure FS19.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2f.**

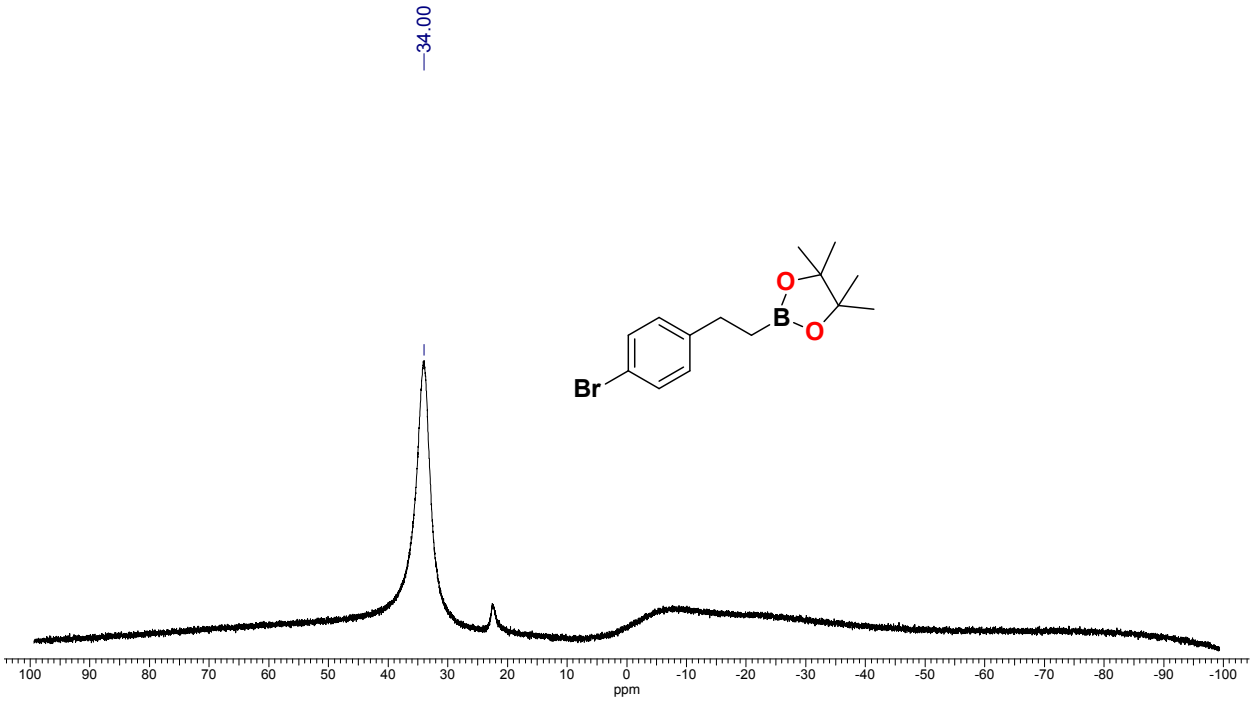



**Figure FS20.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**f.**

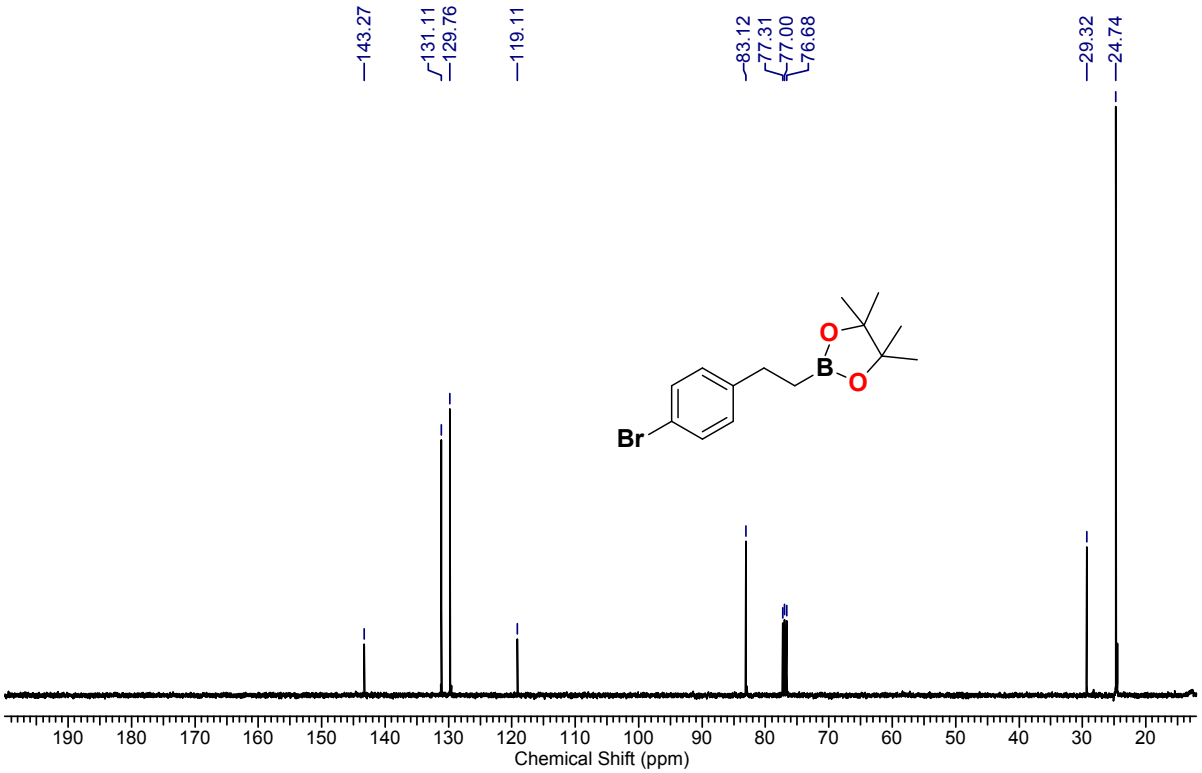



**Figure FS21.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2f**.

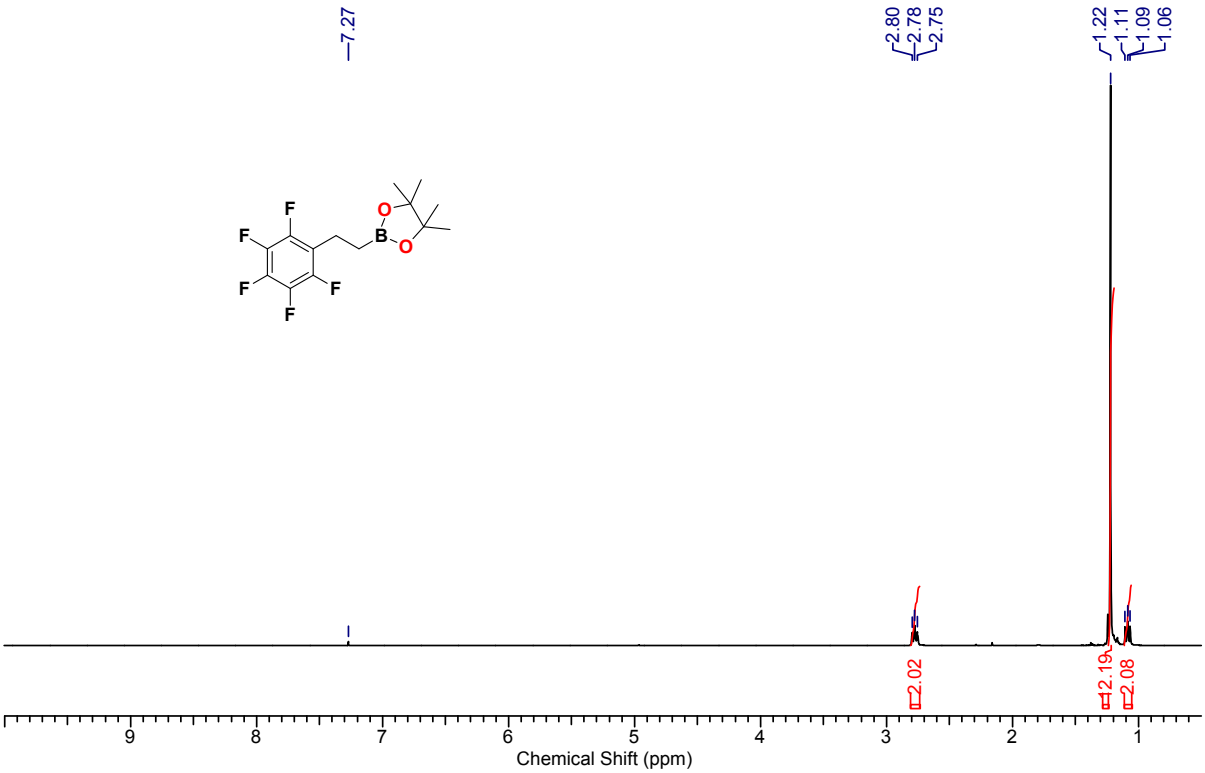



**Figure FS22.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2g.**

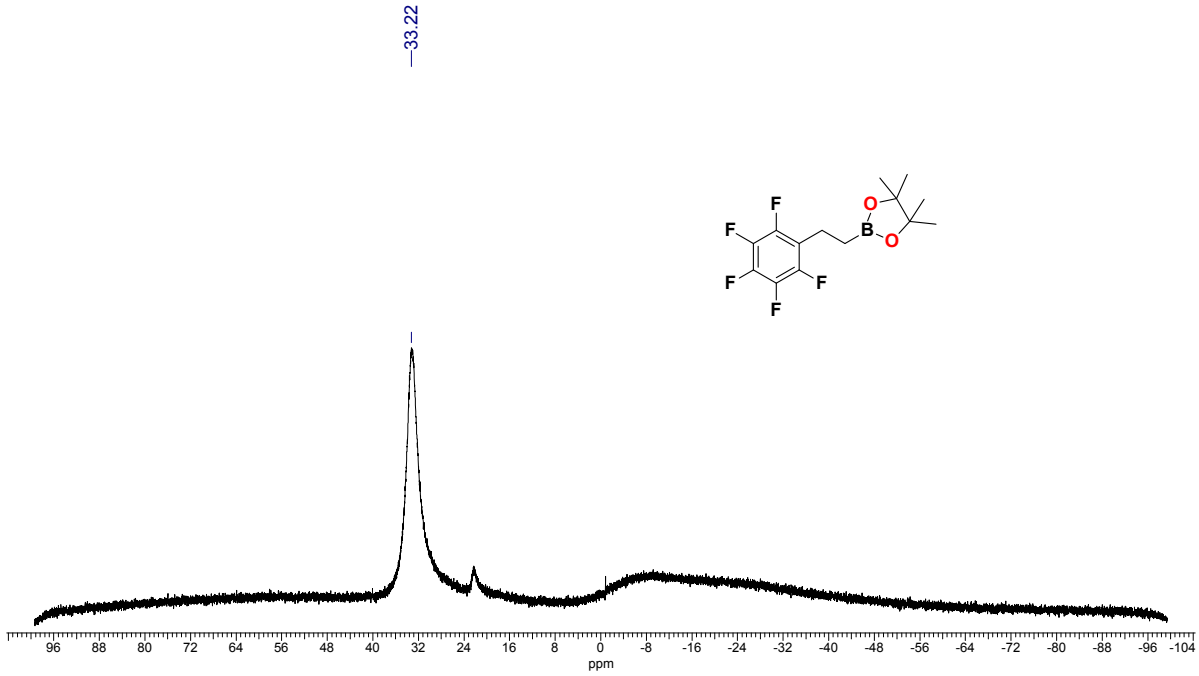



**Figure FS23.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**g.**

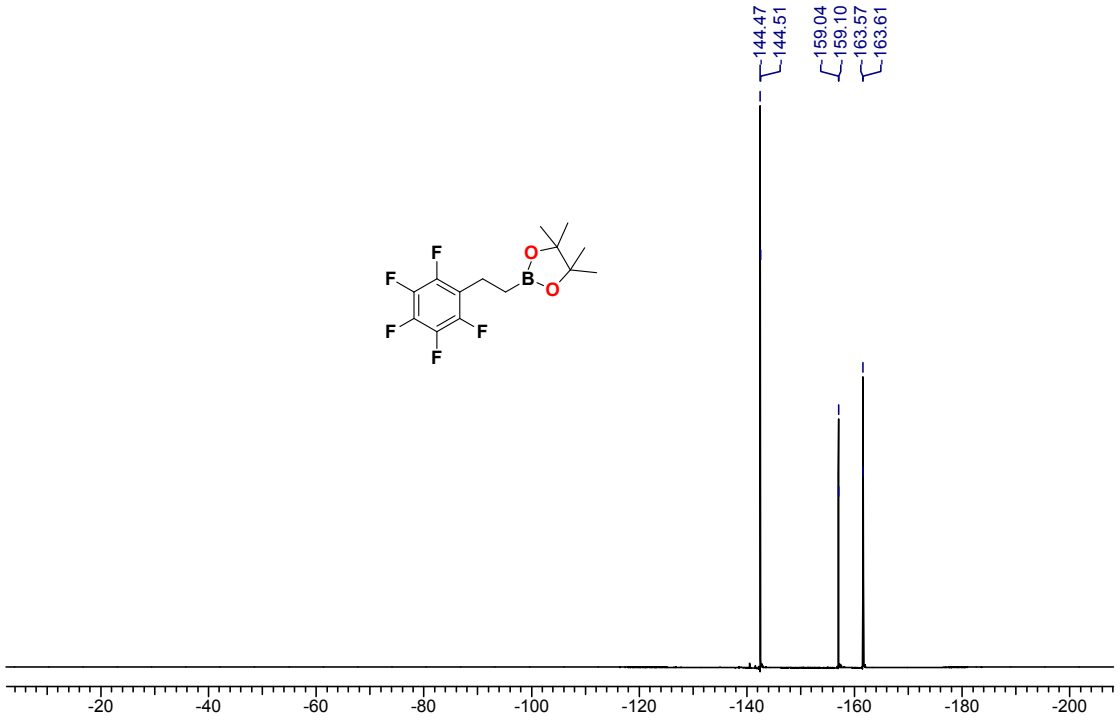



**Figure FS24.** $^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$) spectrum of 2**g.**

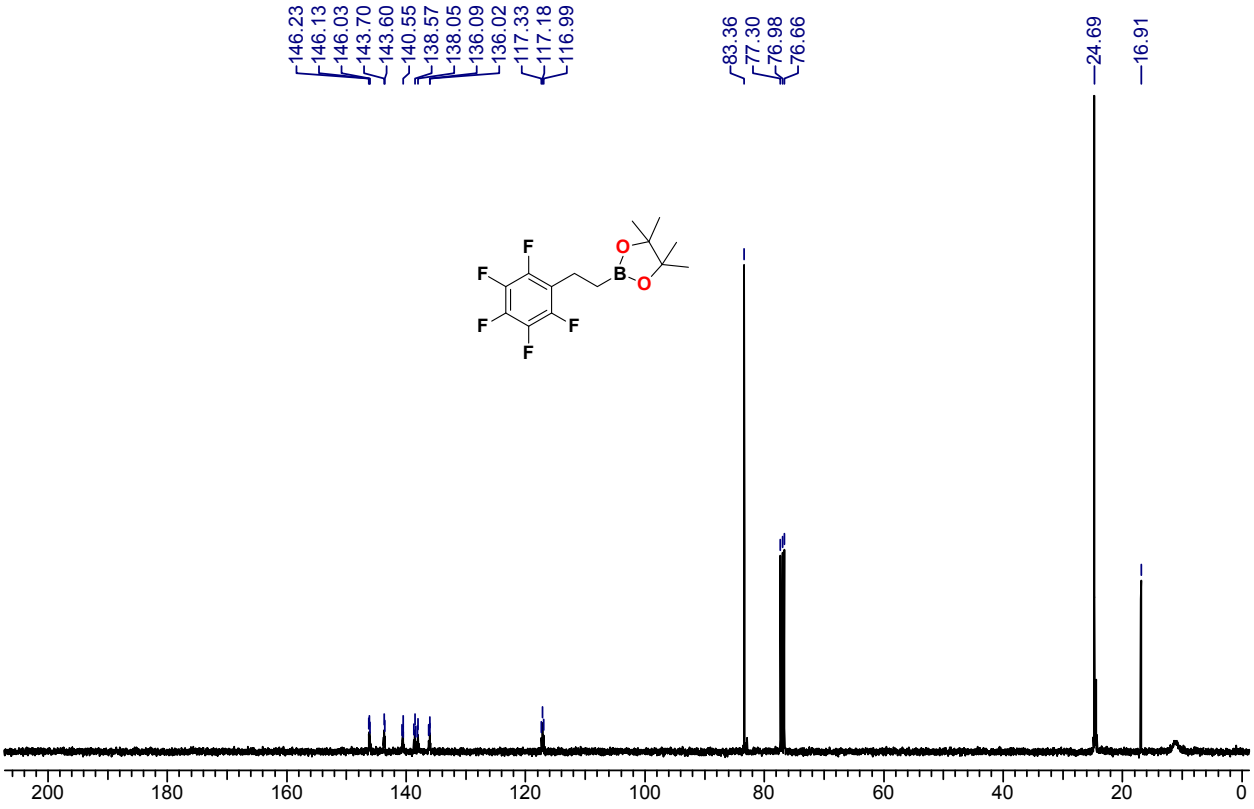



**Figure FS25.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2g.**

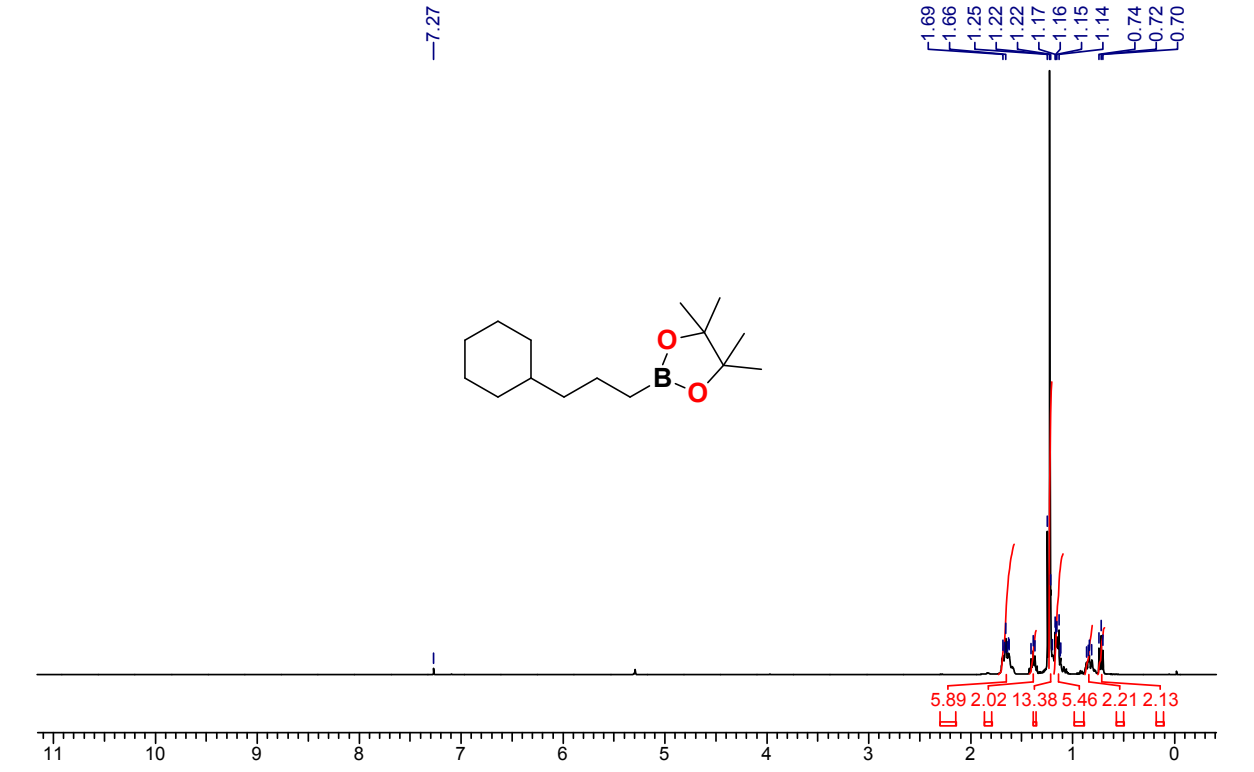



**Figure FS26.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2h.**

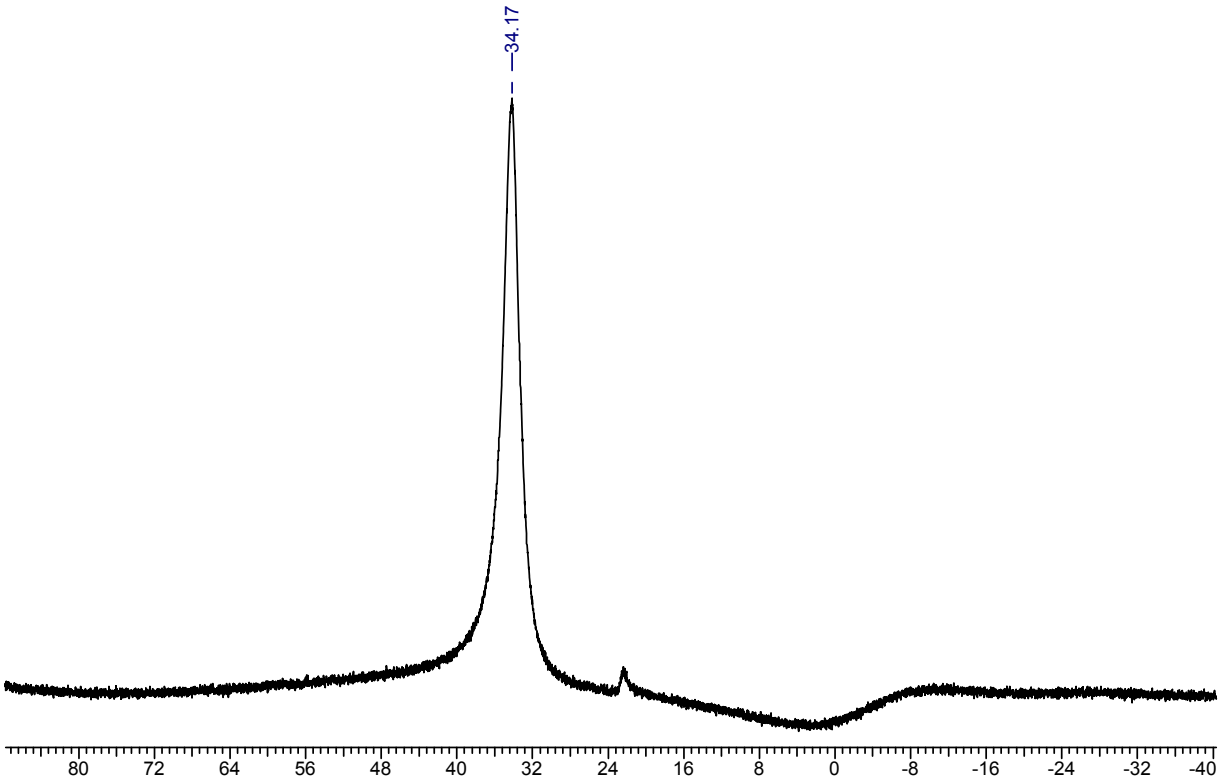



**Figure FS27.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**h.**

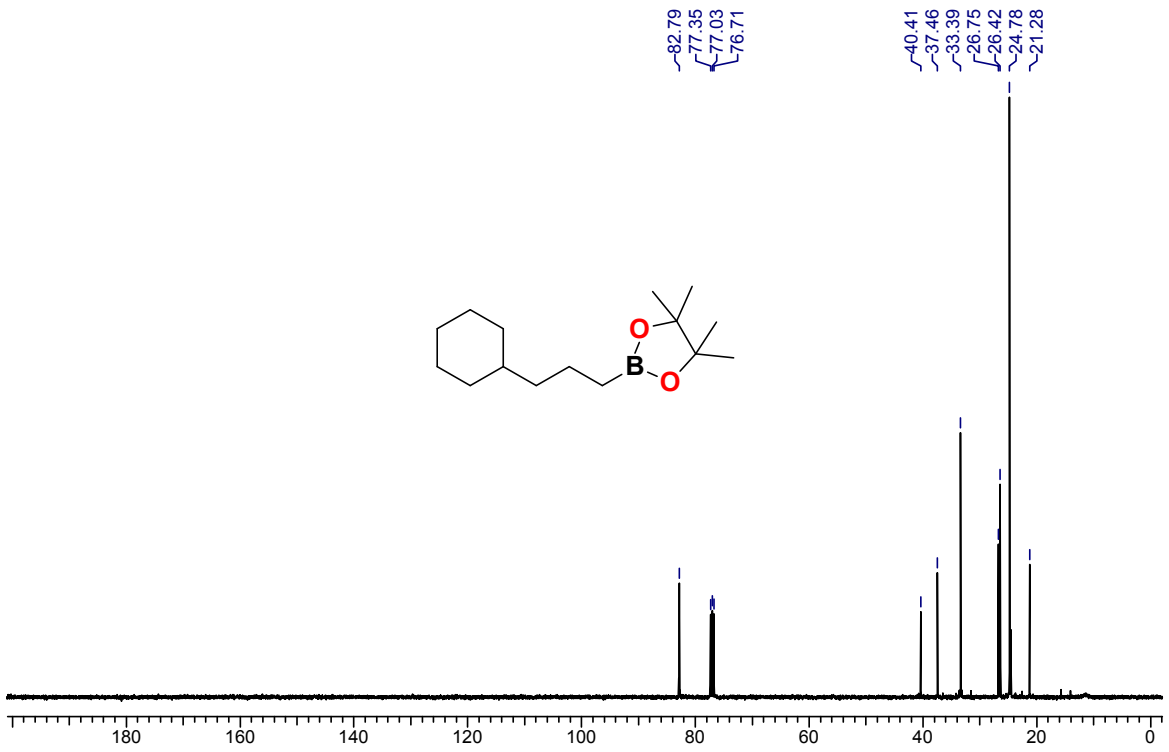



**Figure FS28.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2h.**

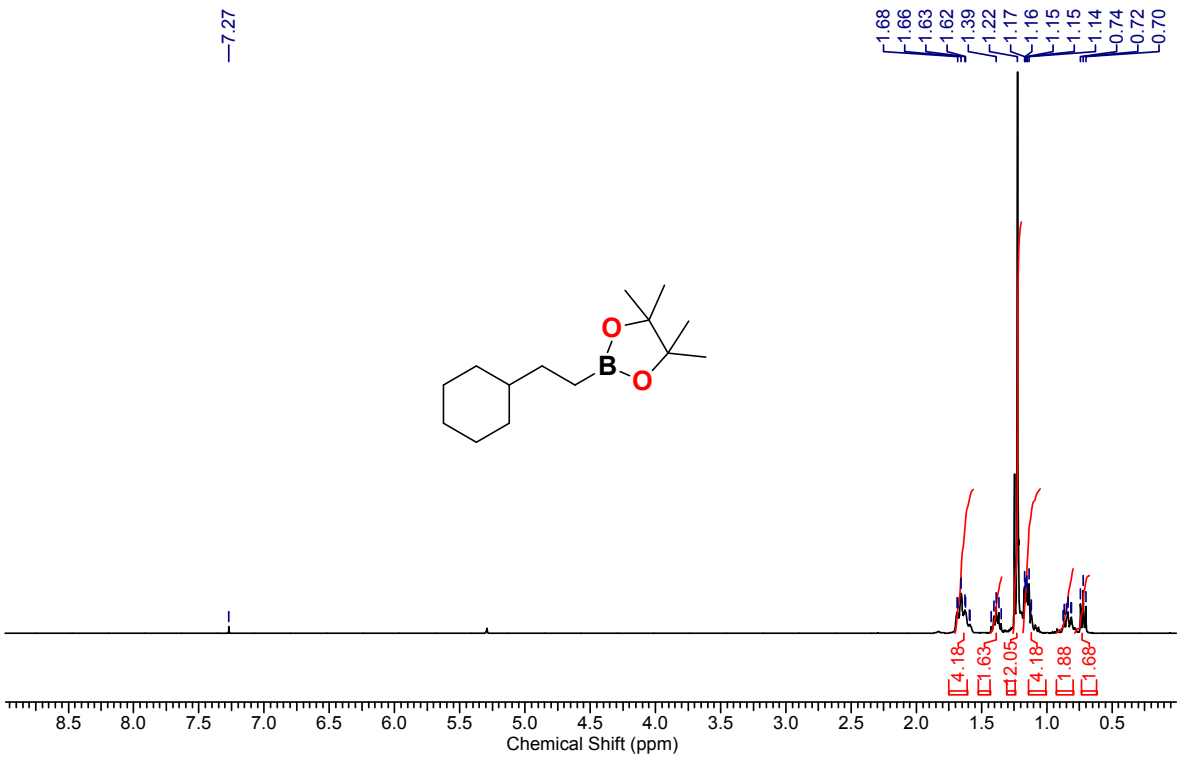



**Figure FS29.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2i.**

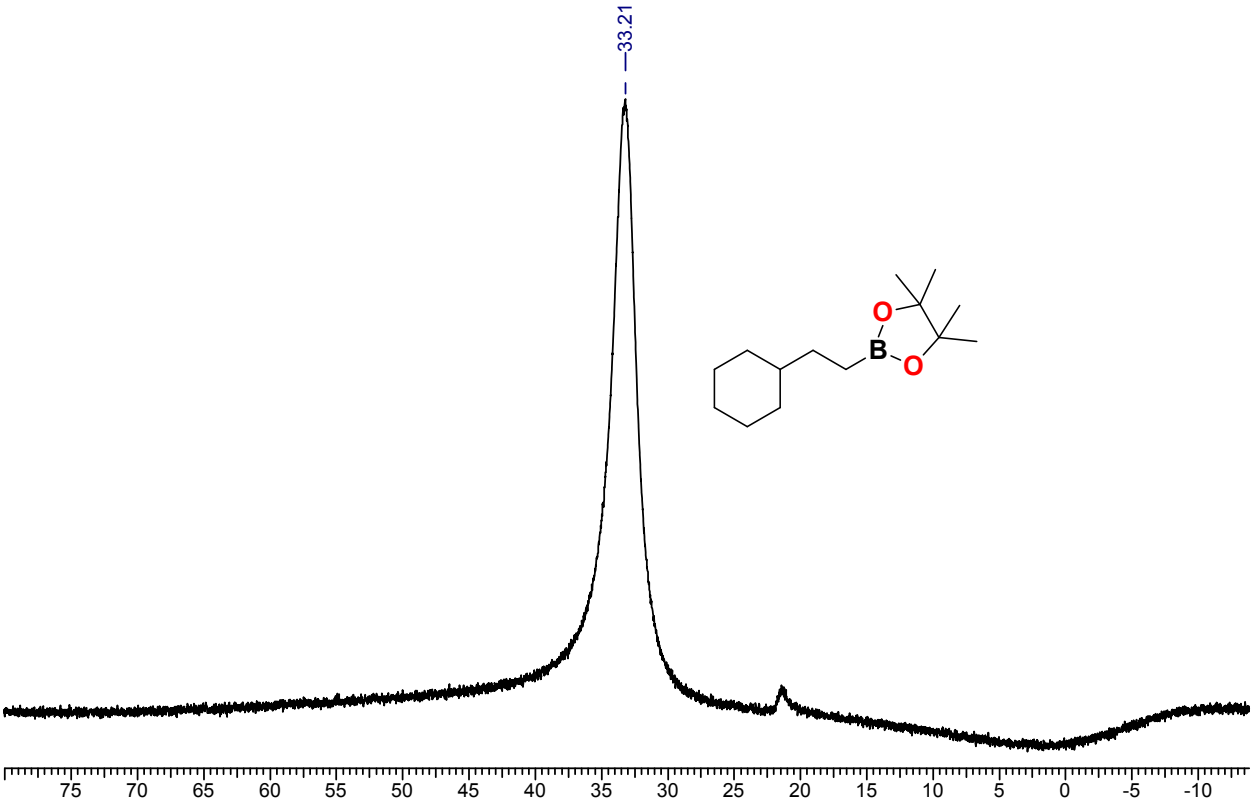



**Figure FS30.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**i.**

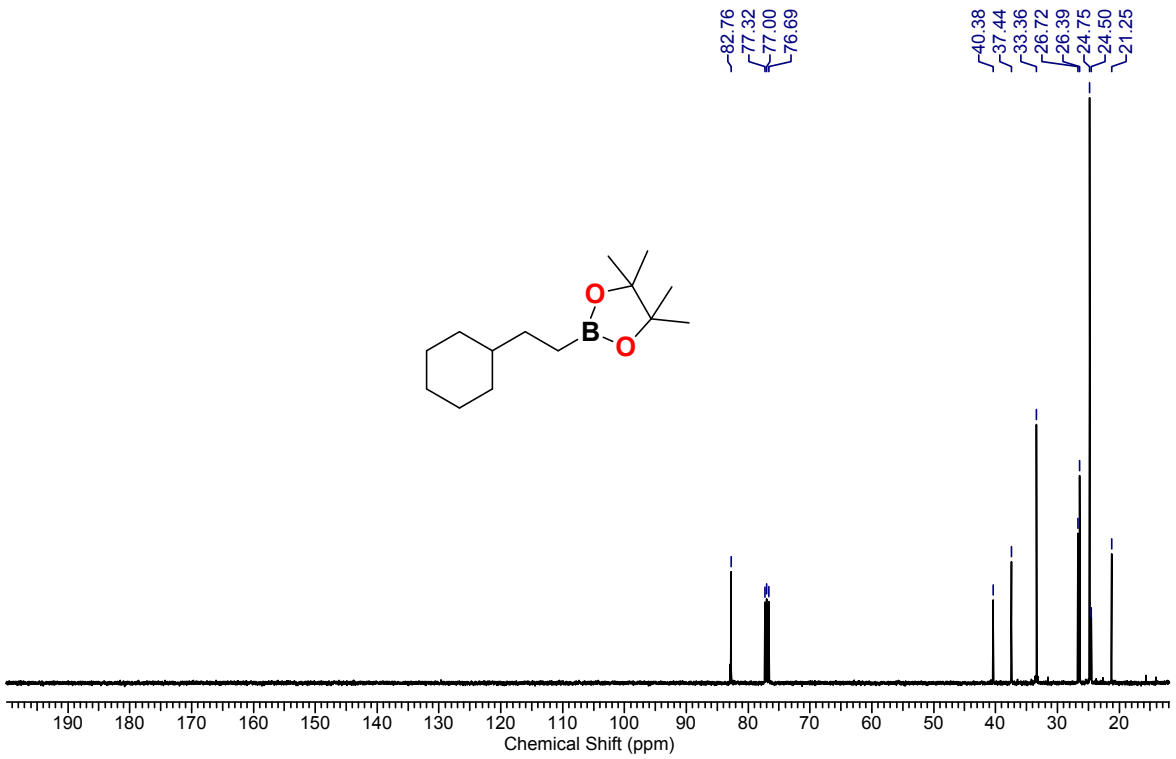



**Figure FS31.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2i.**

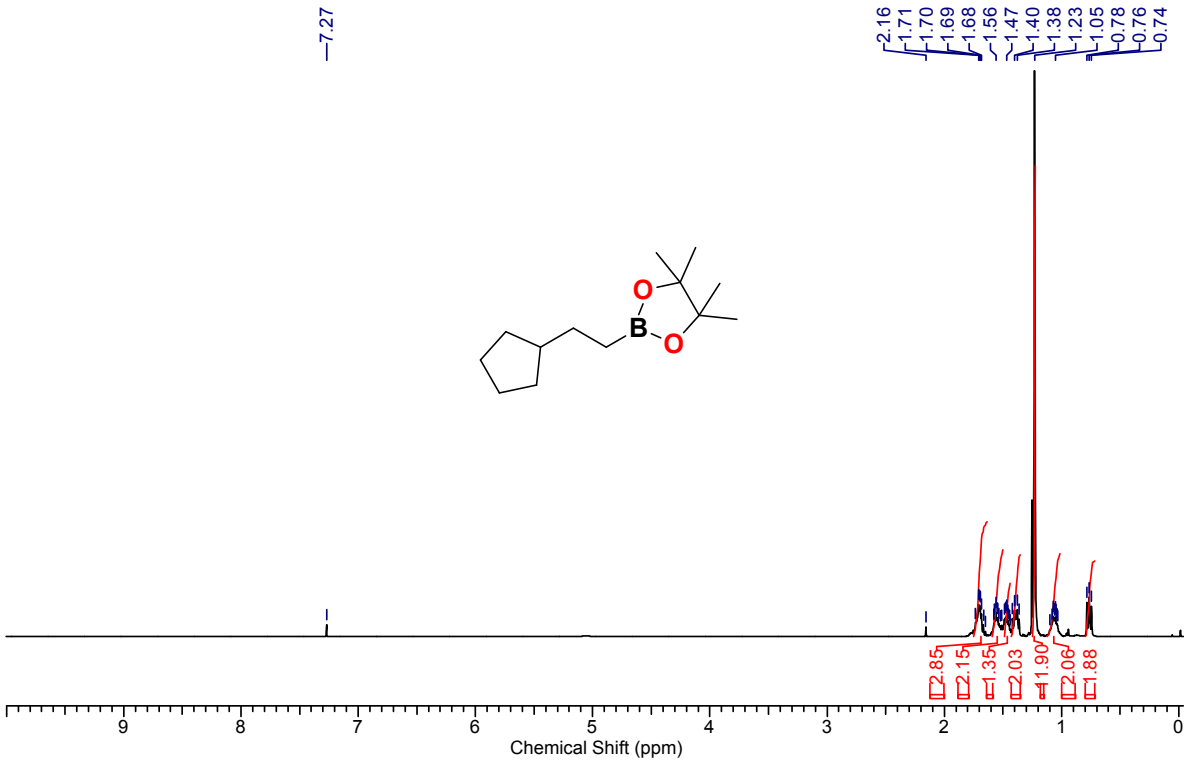



**Figure FS32.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2j.**

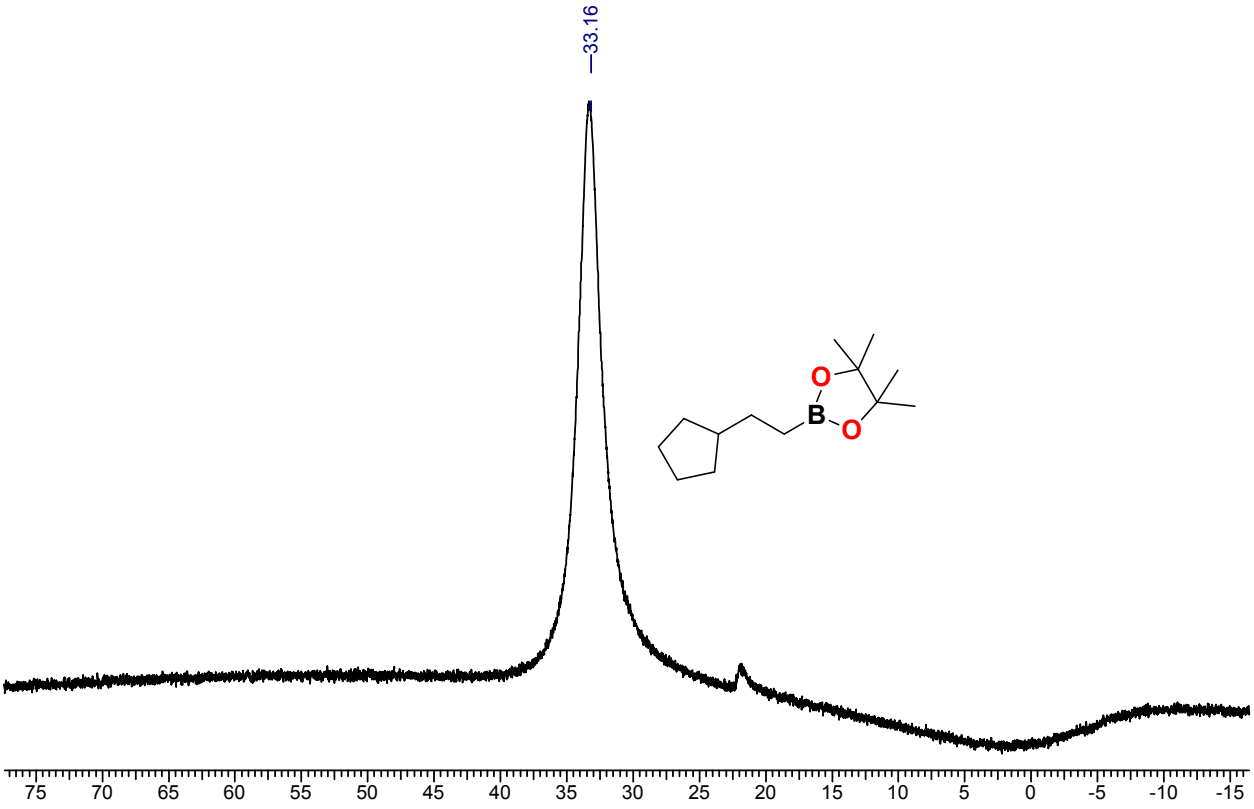



**Figure FS33.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**j.**

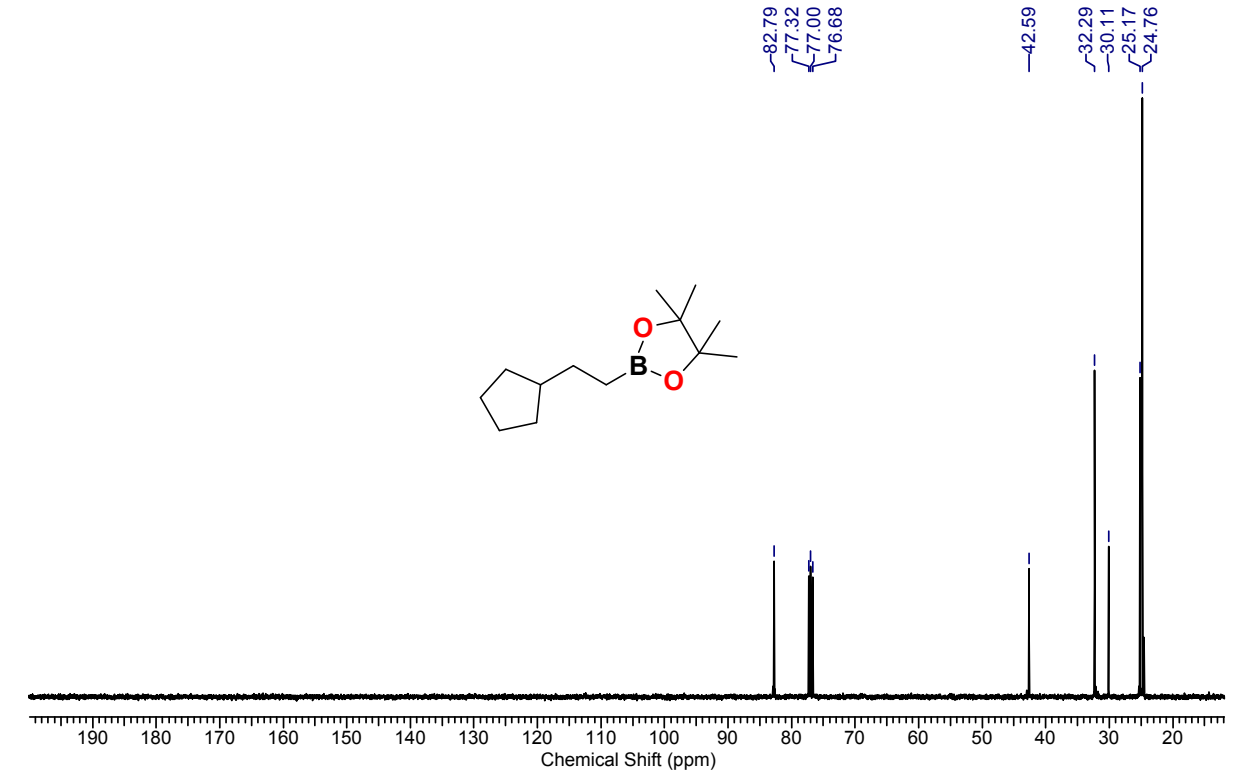



**Figure FS34.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2j.**

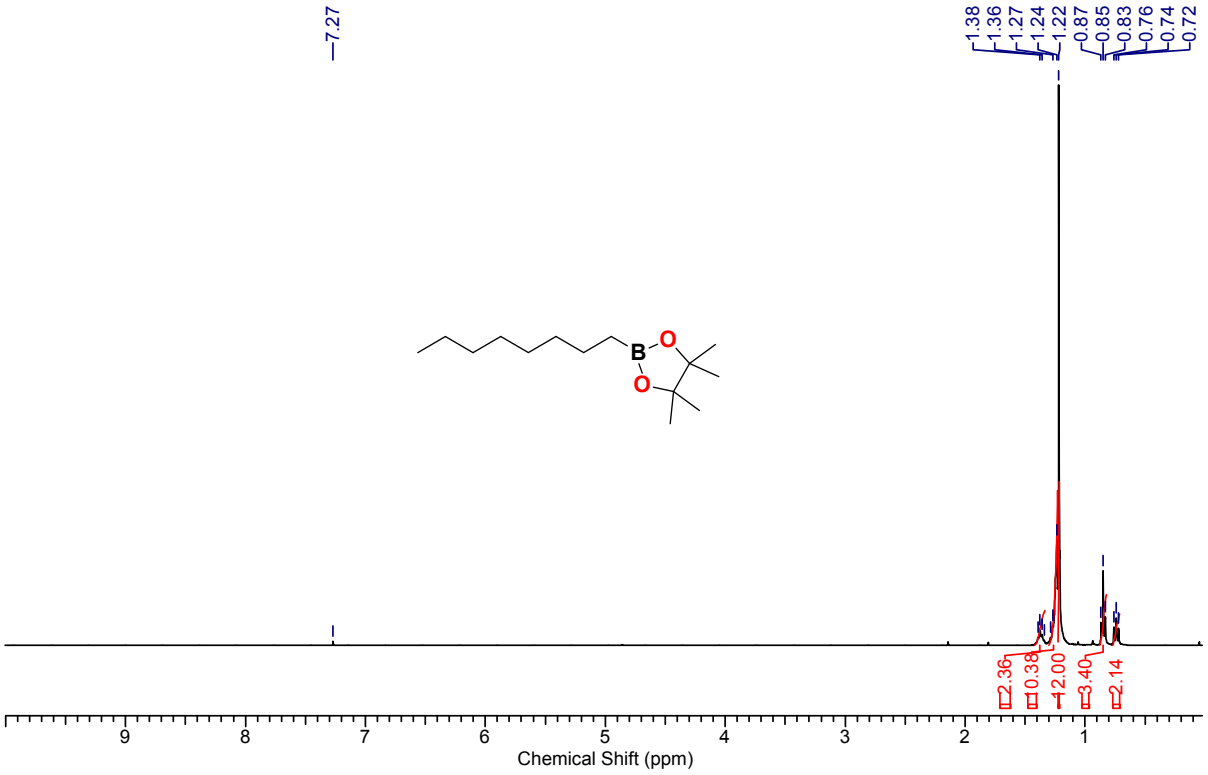



**Figure FS35.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2k.**

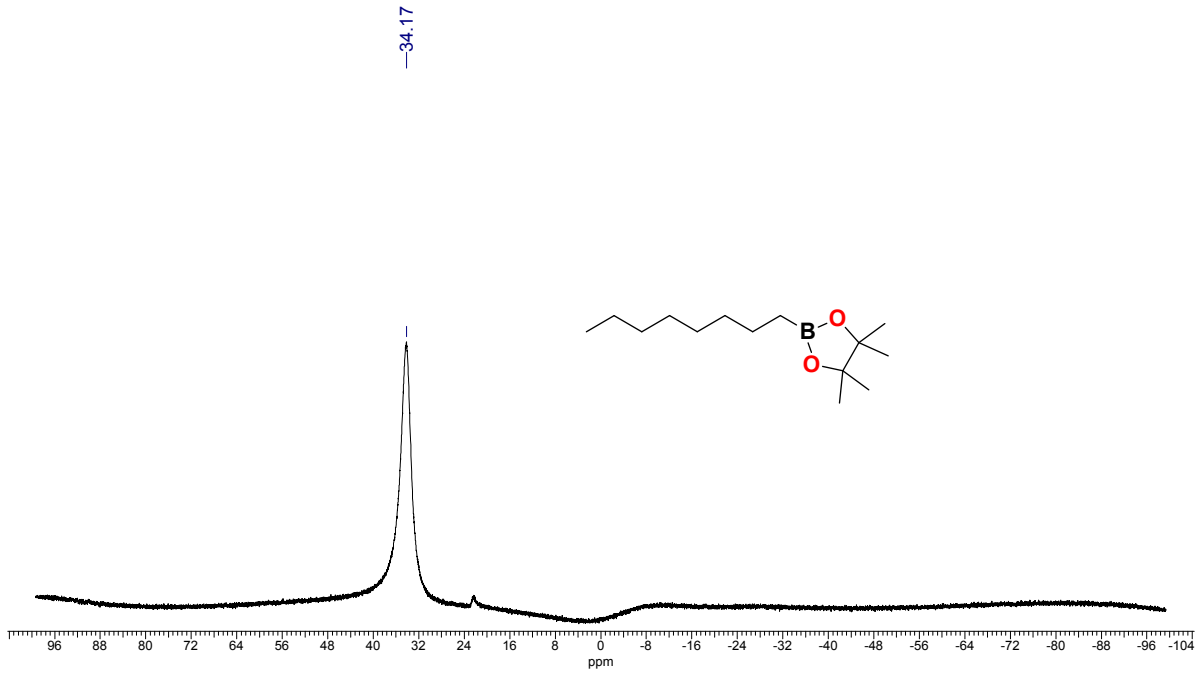



**Figure FS36.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**k.**

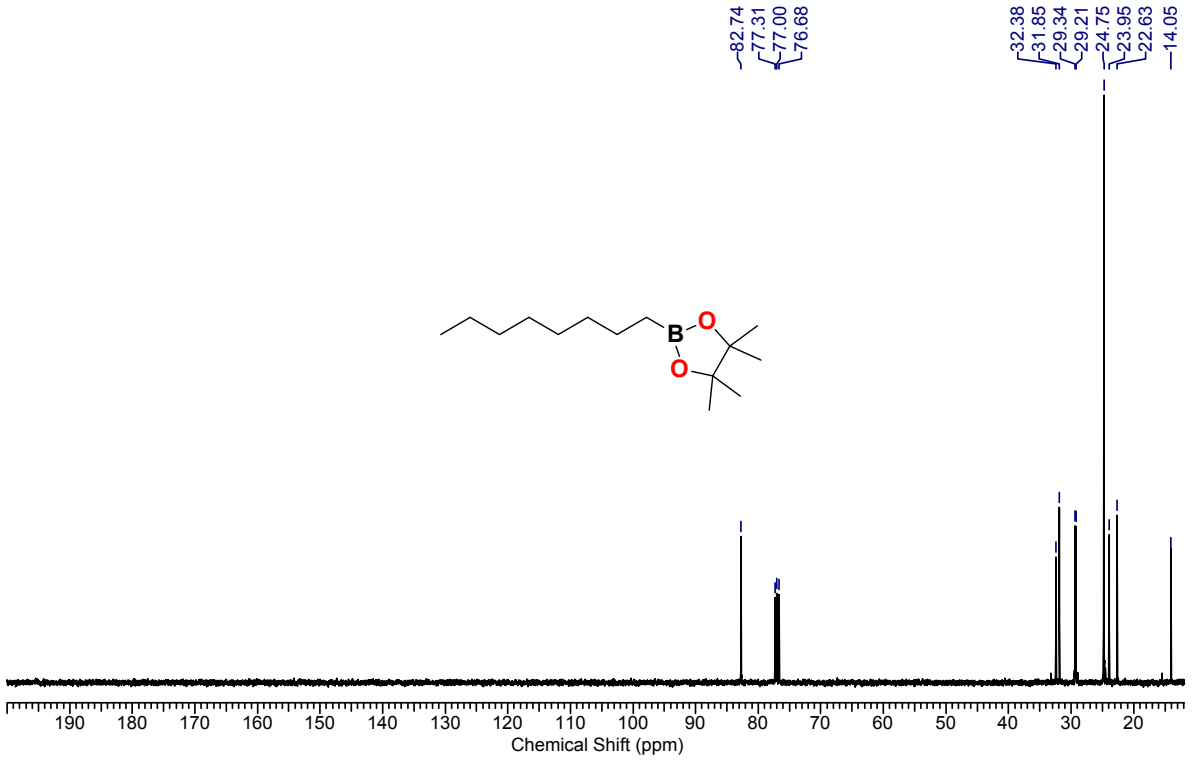



**Figure FS37.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2k**.

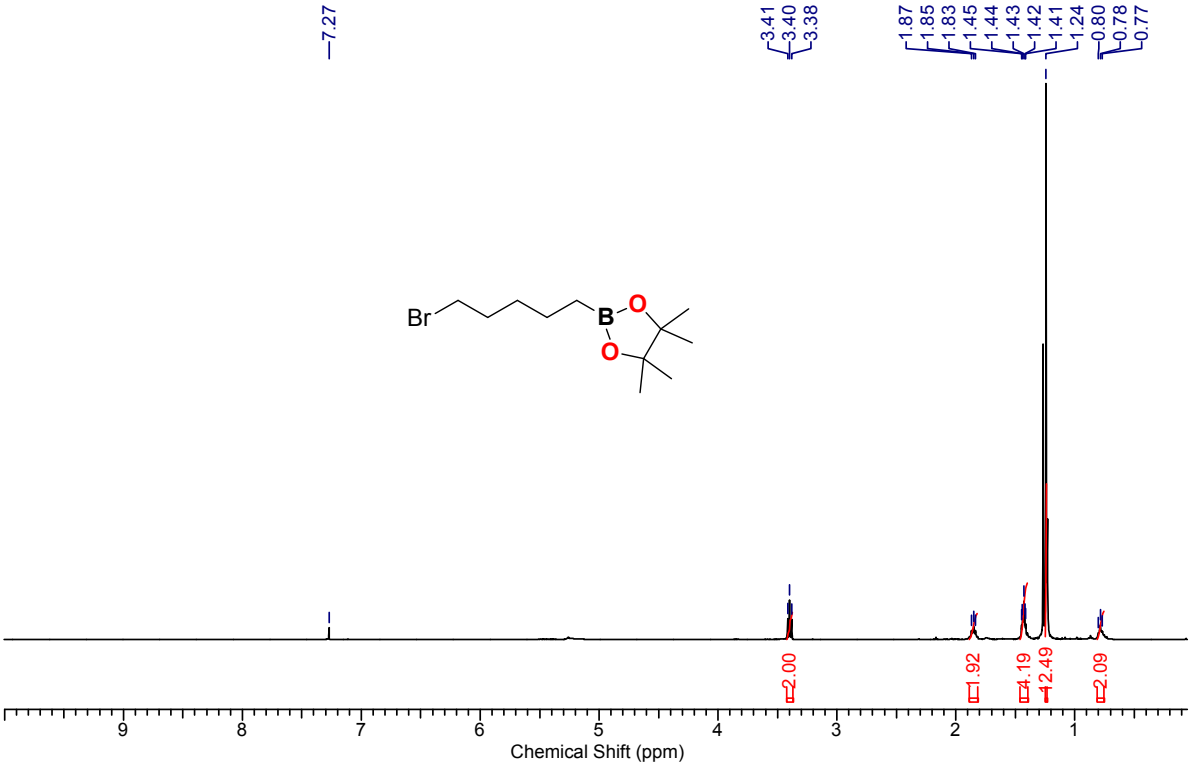



**Figure FS38.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2l.**

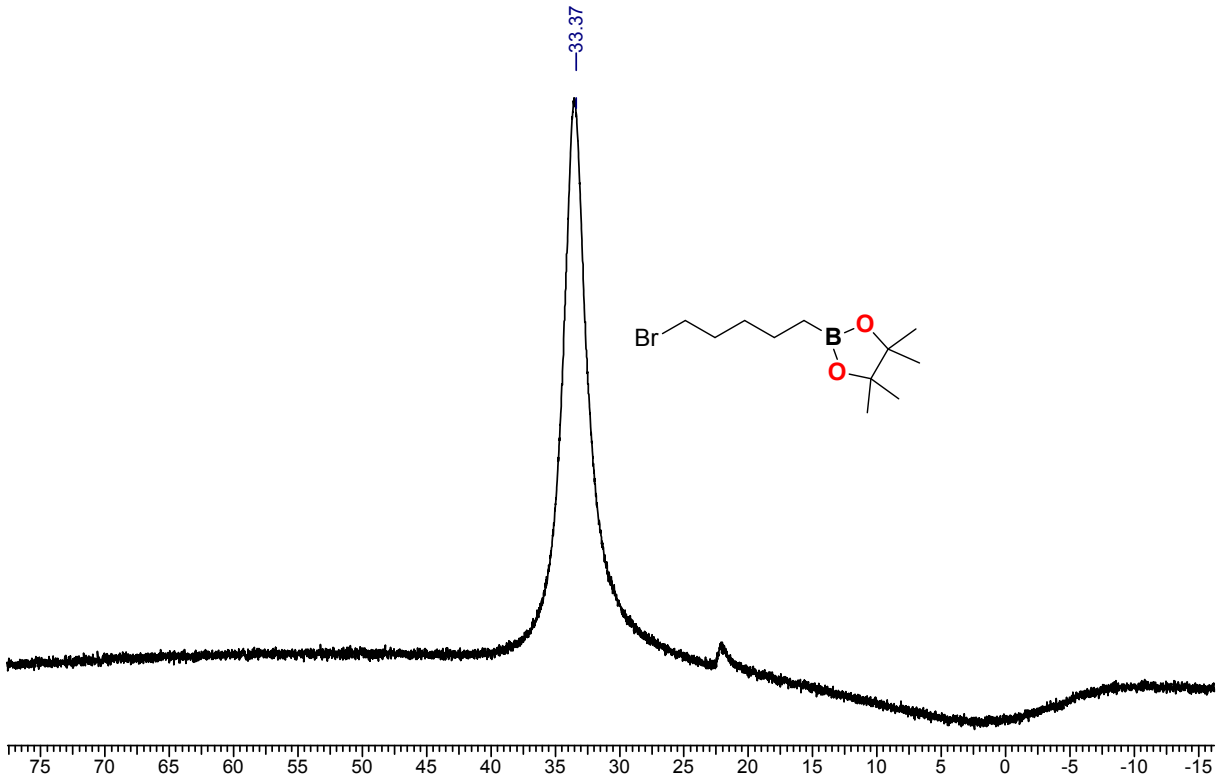



**Figure FS39.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **2l.**

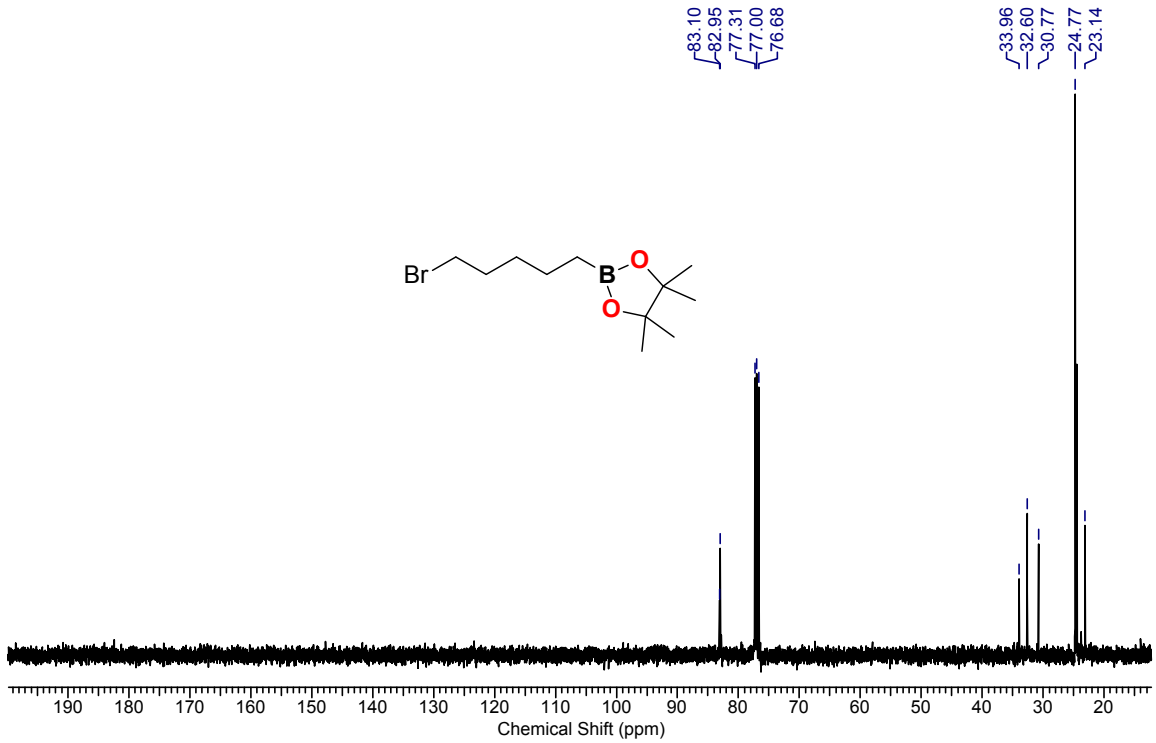



**Figure FS40.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2l.**

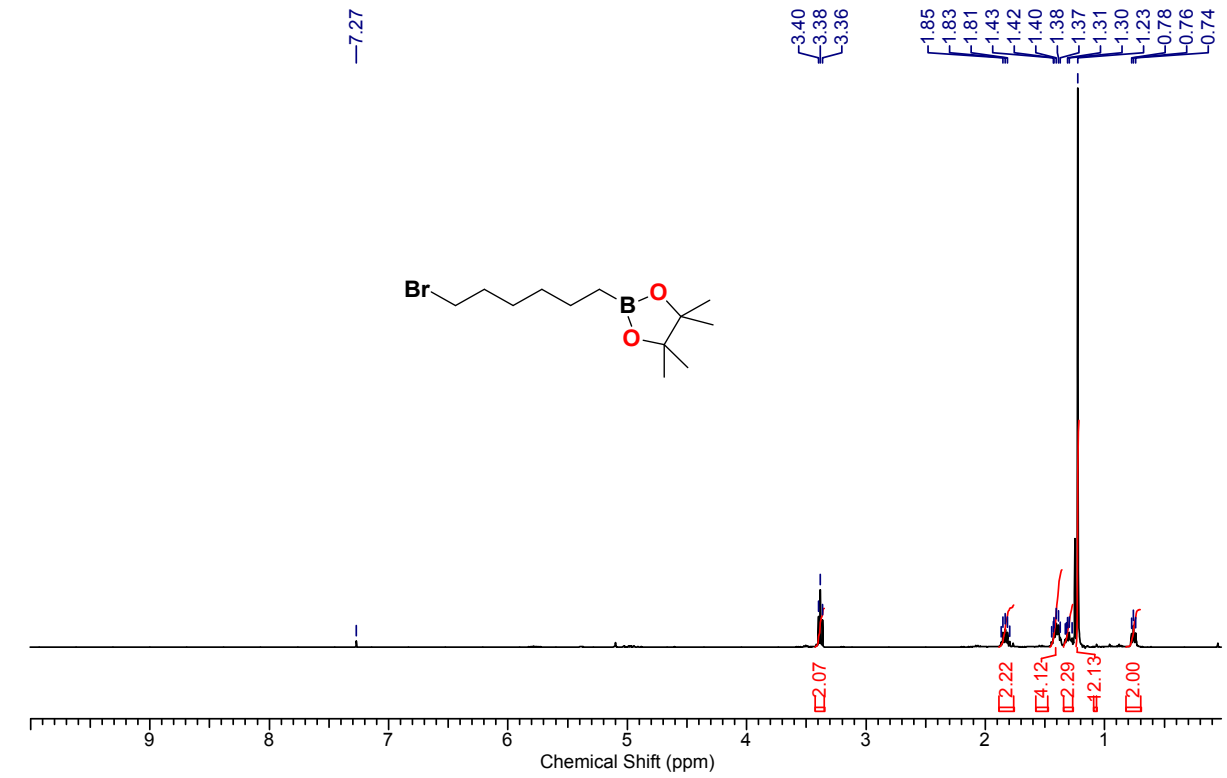



**Figure FS41.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2m.**

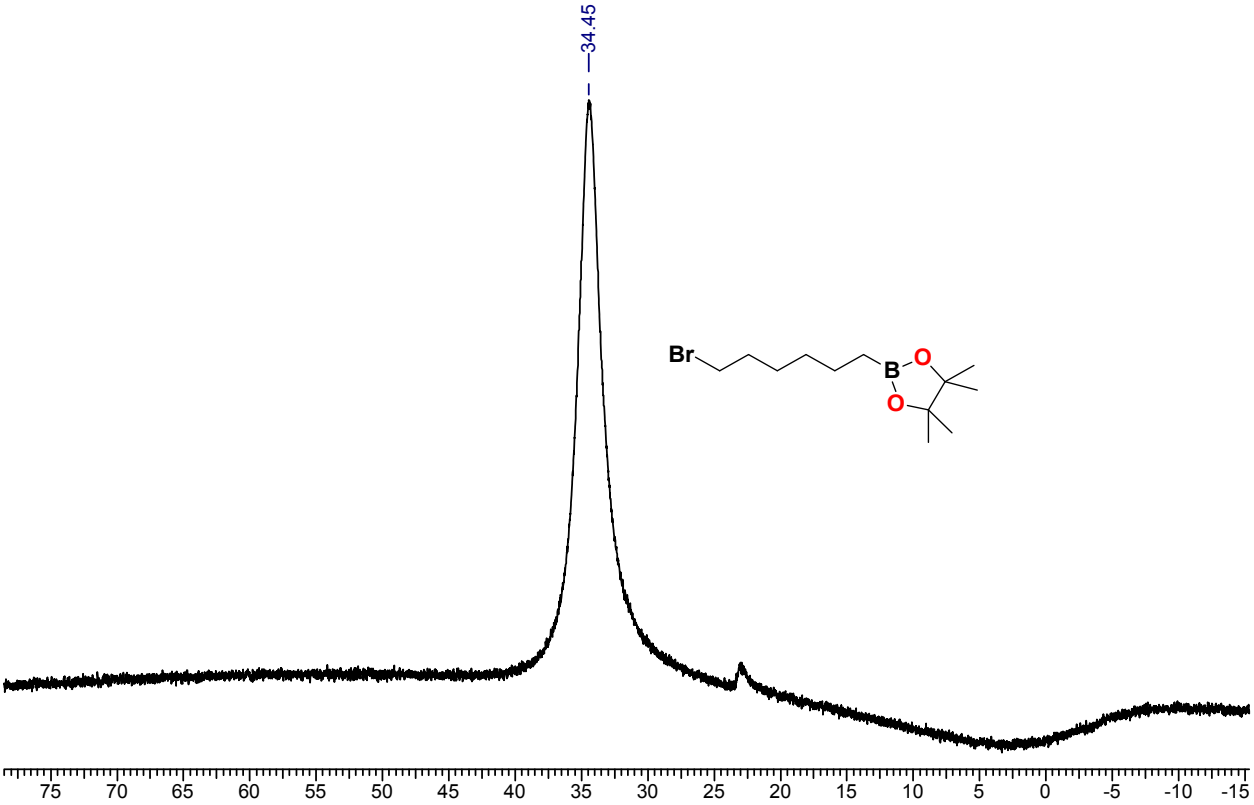



**Figure FS42.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **2m.**

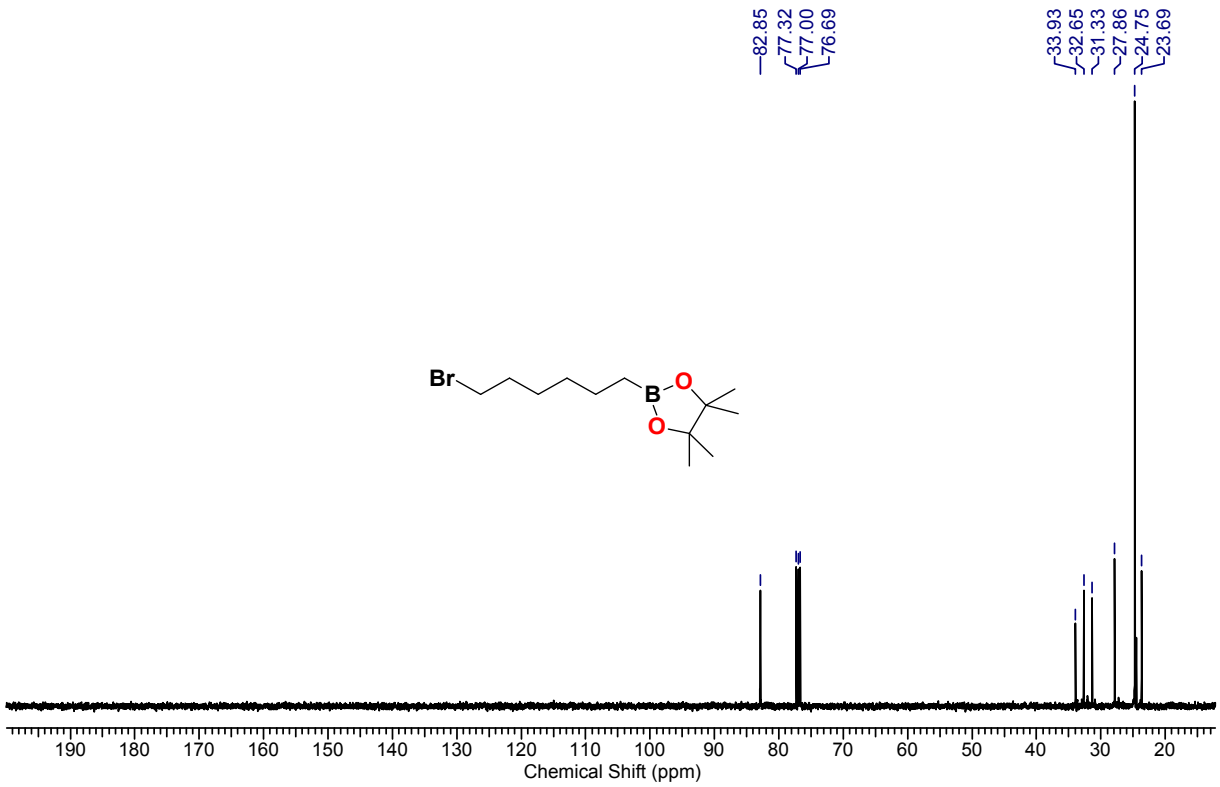



**Figure FS43.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2m.**

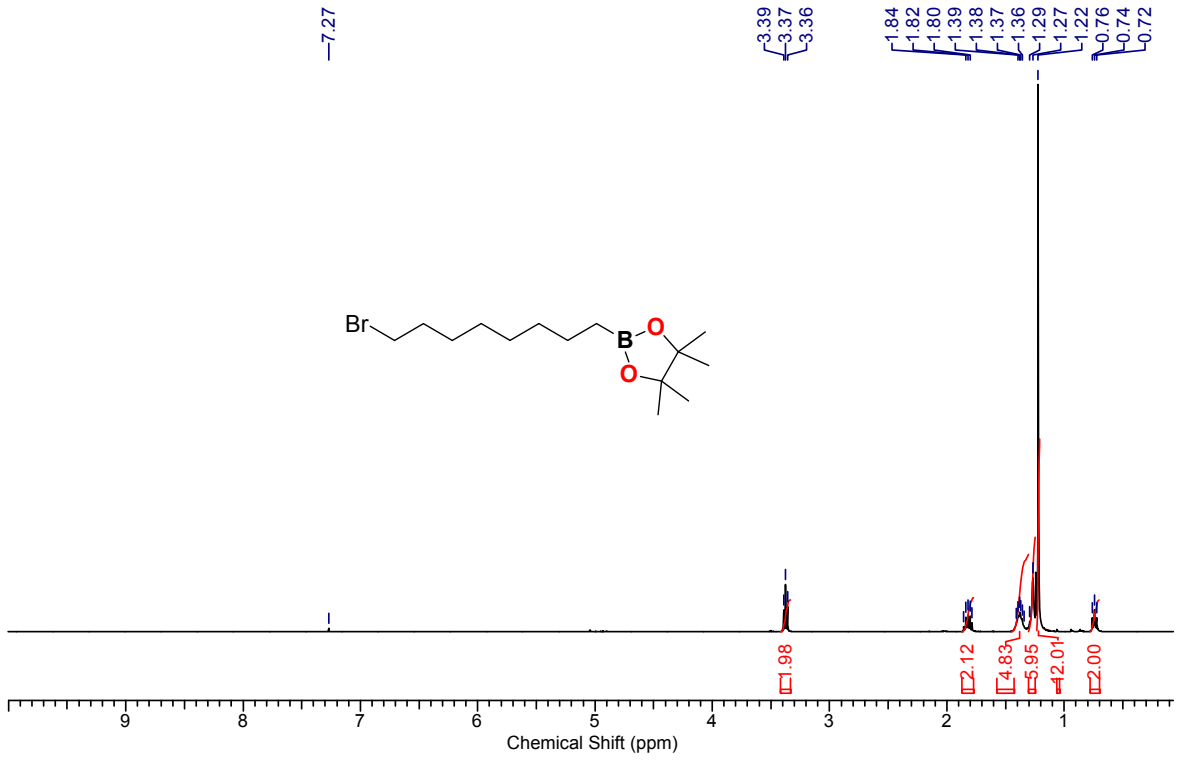



**Figure FS44.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2n.**

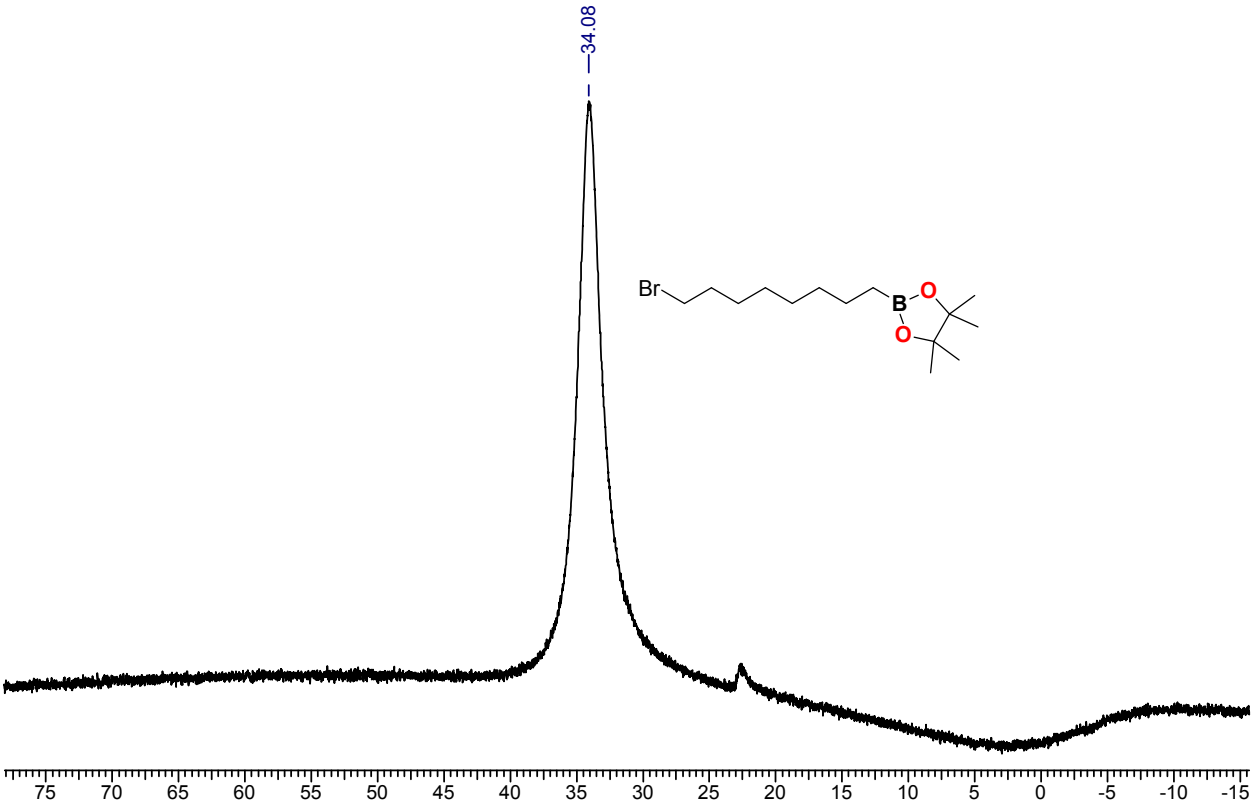



**Figure FS45.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**n.**

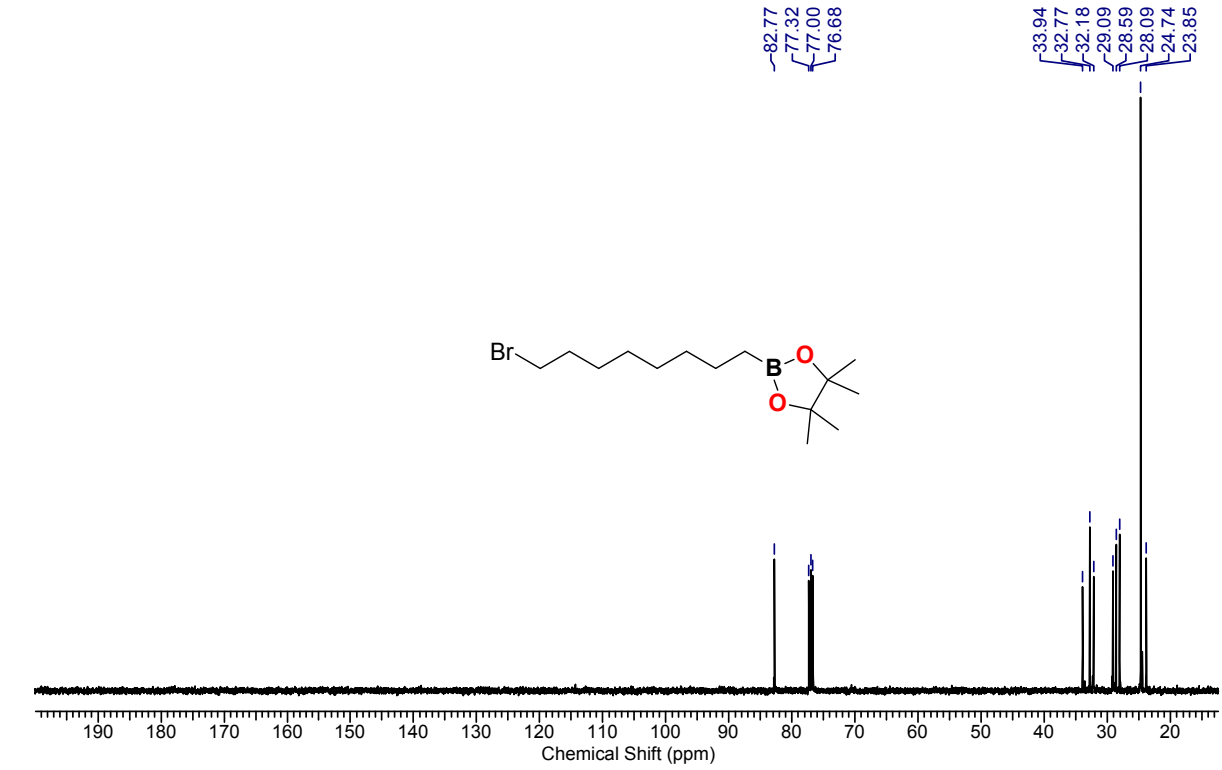



**Figure FS46.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2n.**

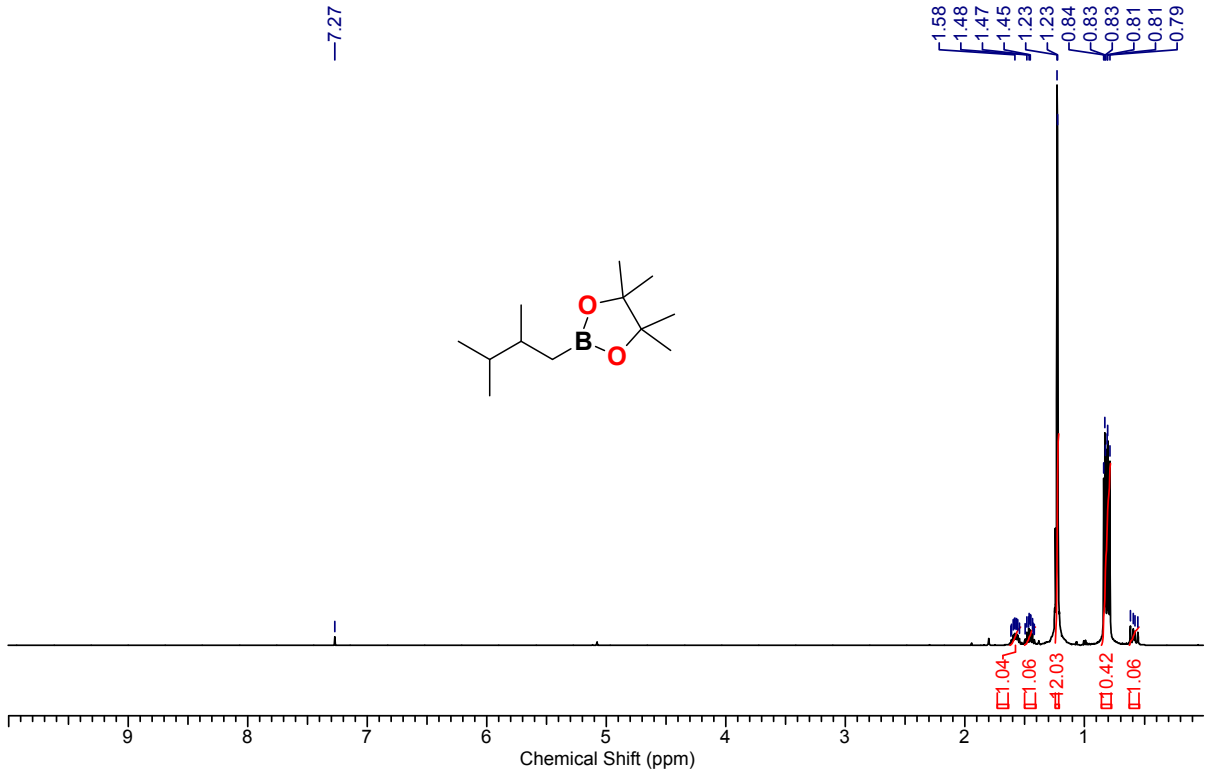



**Figure FS47.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2o.**

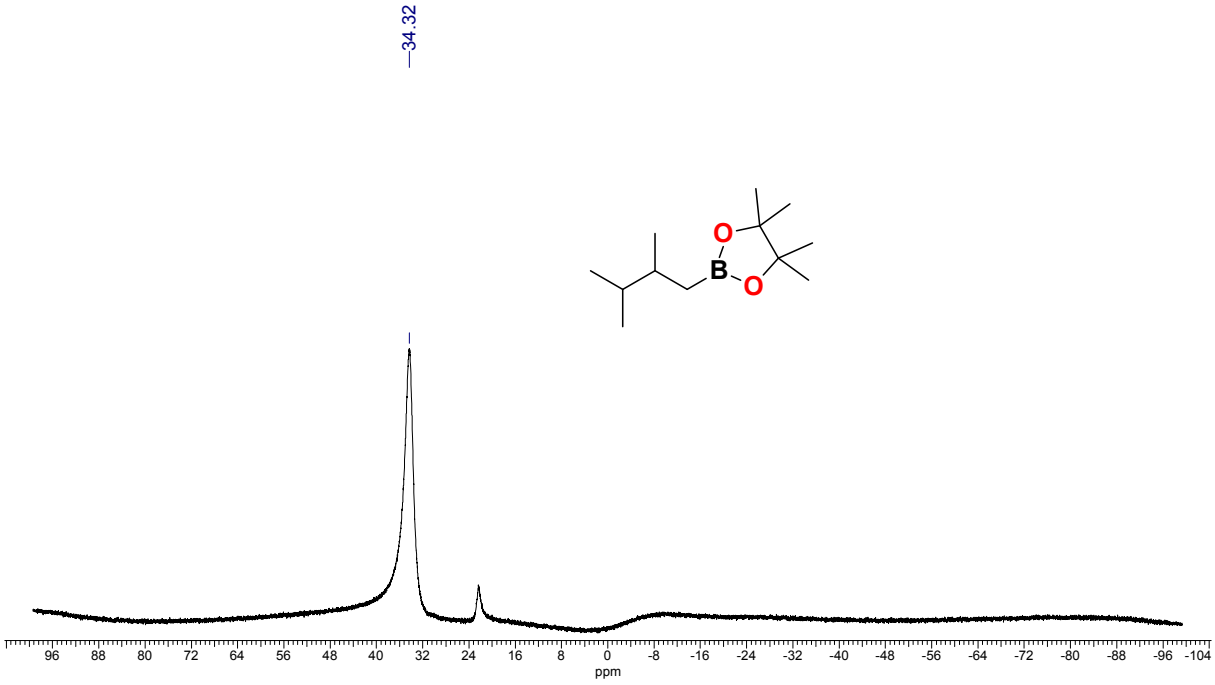



**Figure FS48.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**o.**

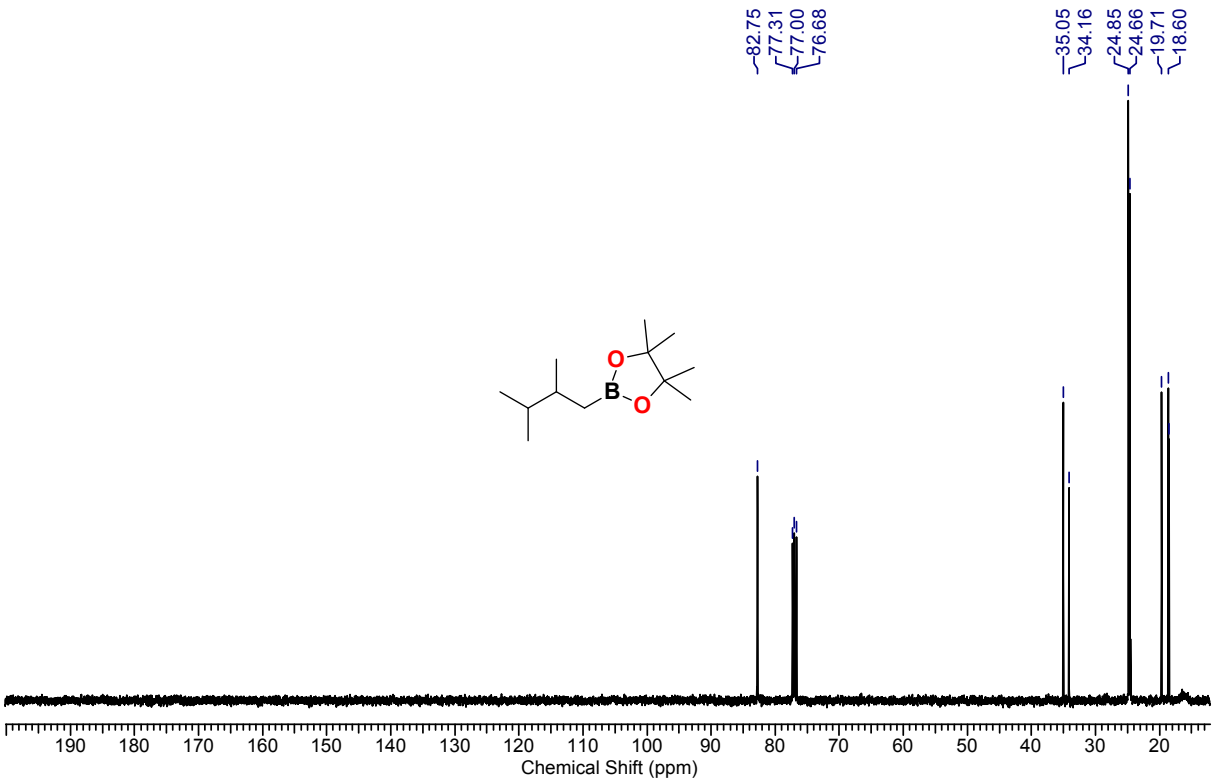



**Figure FS49.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2o.**

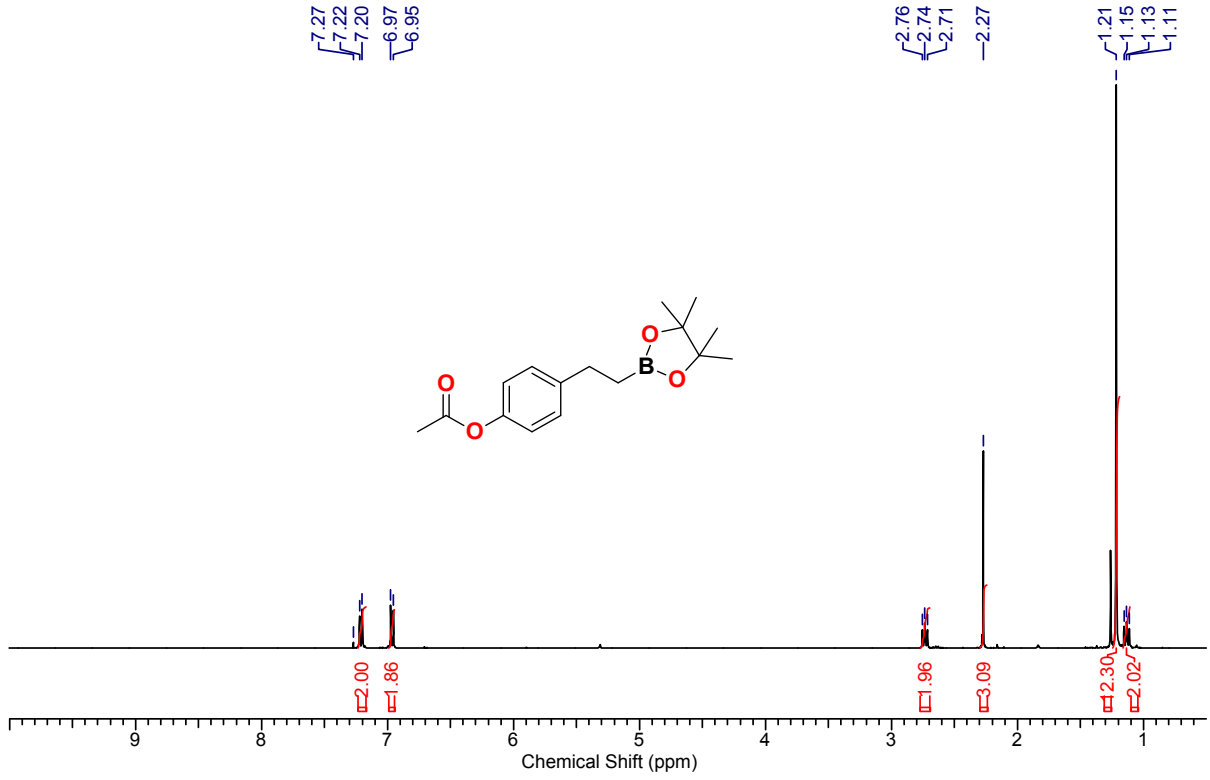



**Figure FS50.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **2p.**

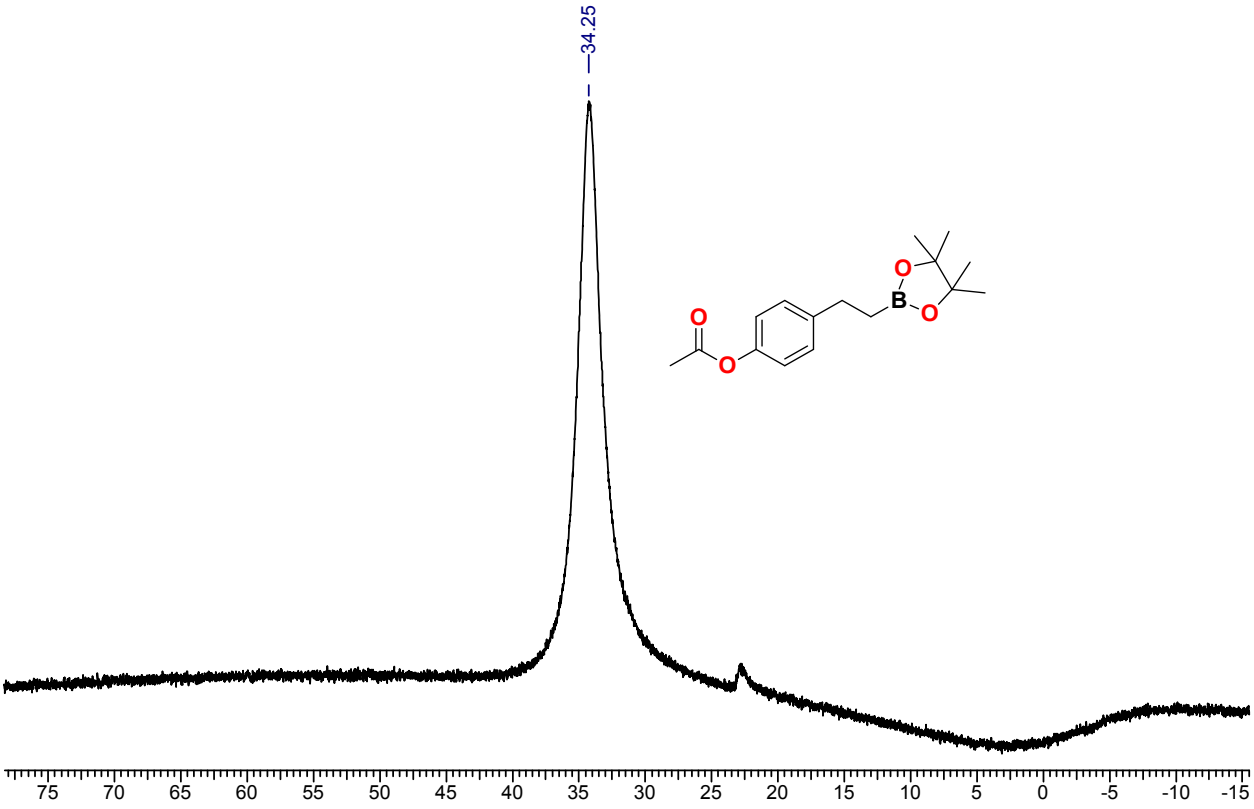



**Figure FS51.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of 2**p.**

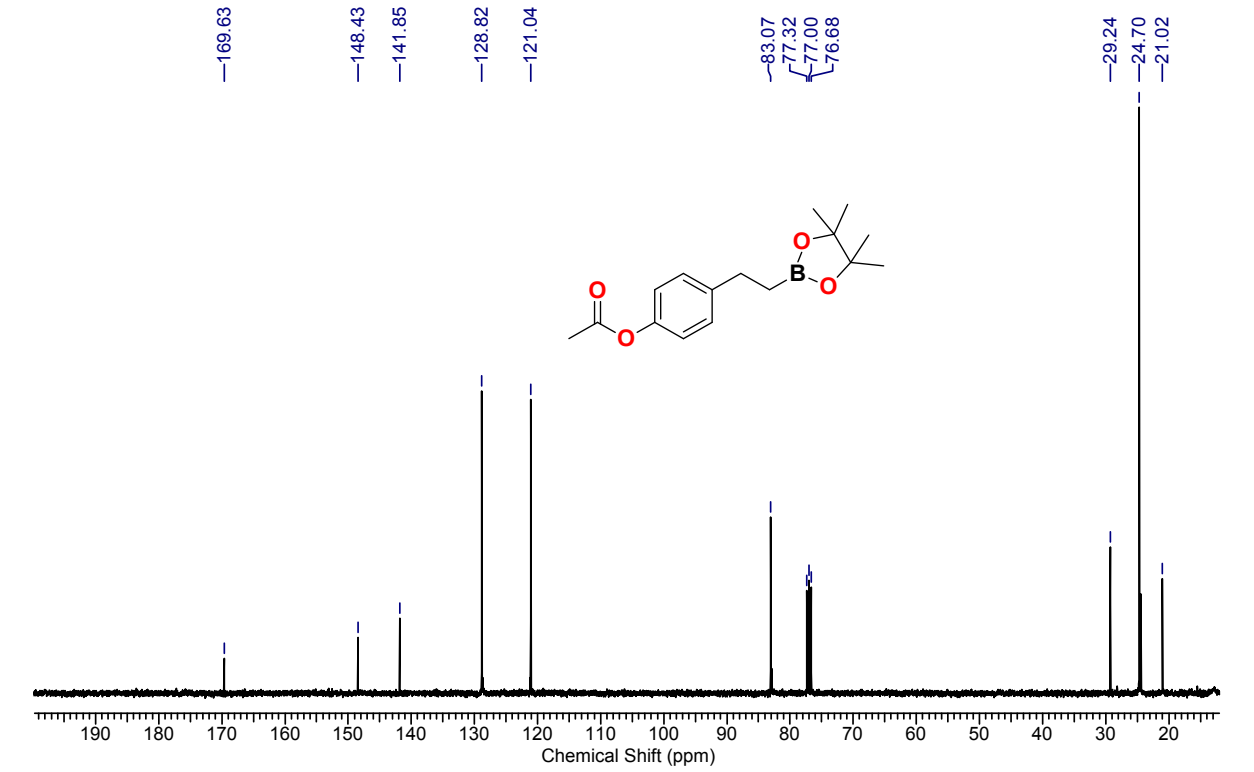



**Figure FS52.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **2p.**

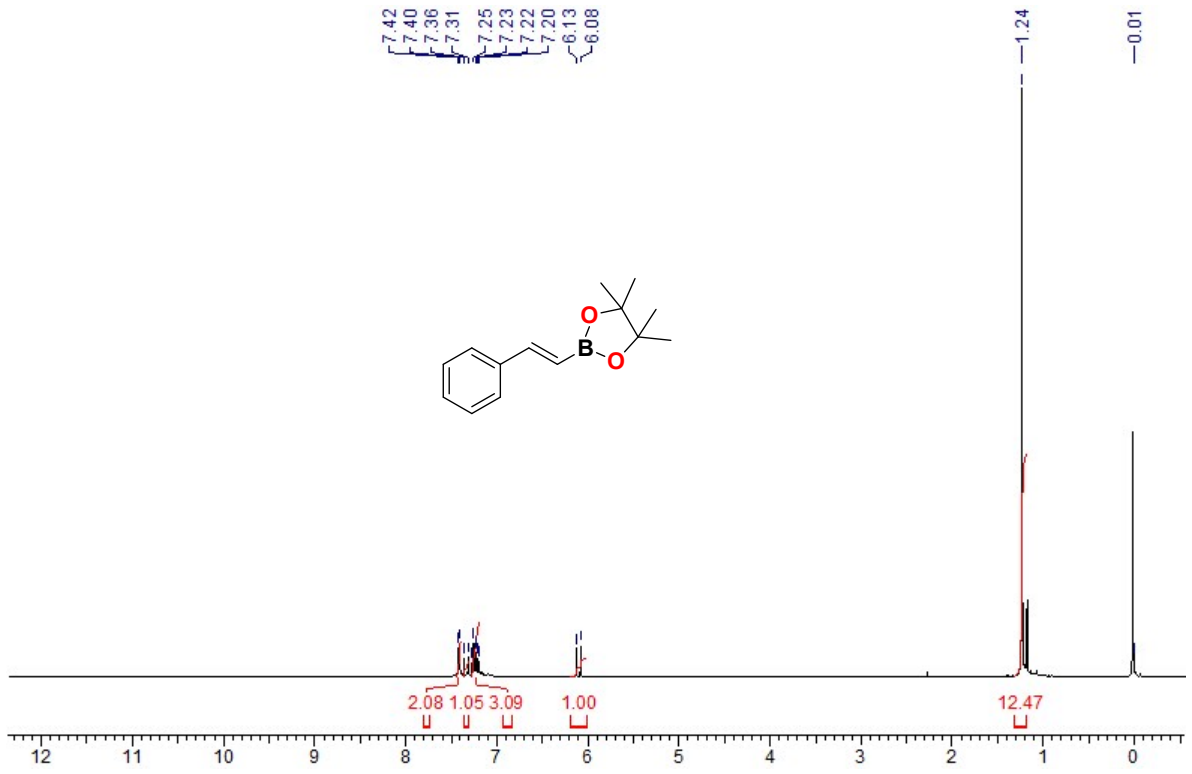



**Figure FS53.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3a.**

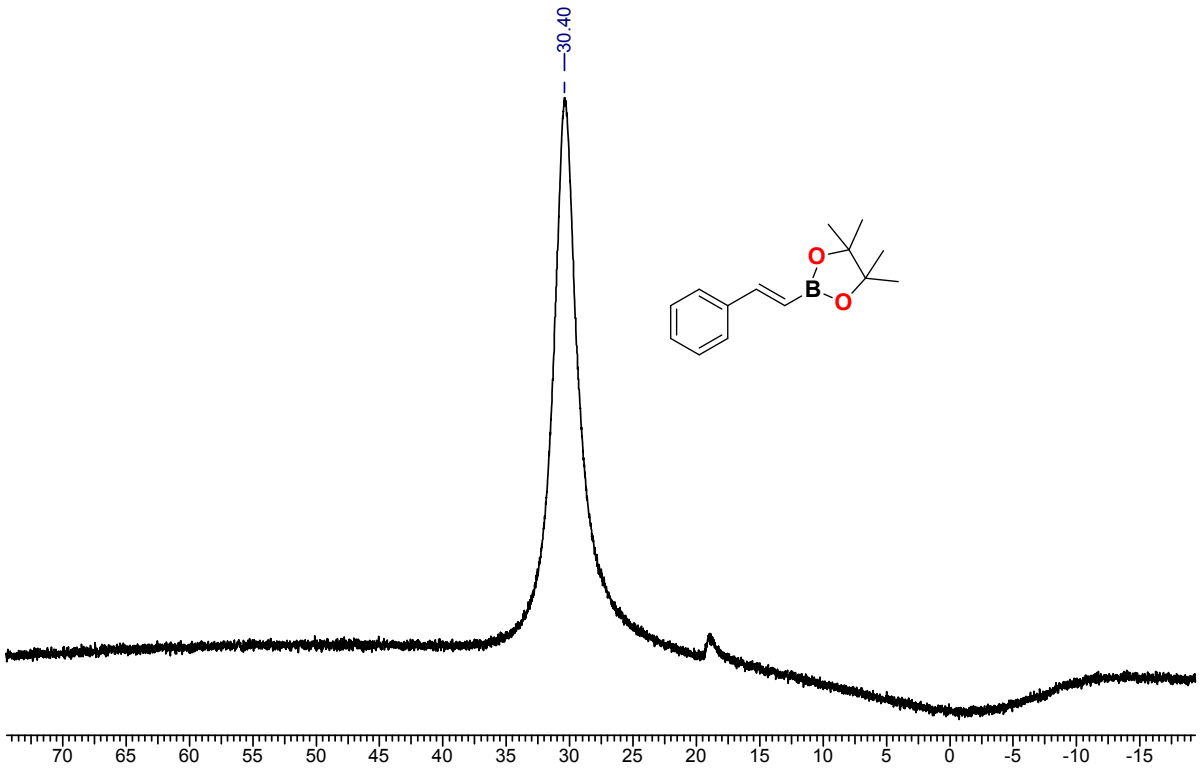



**Figure FS54.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3a**.

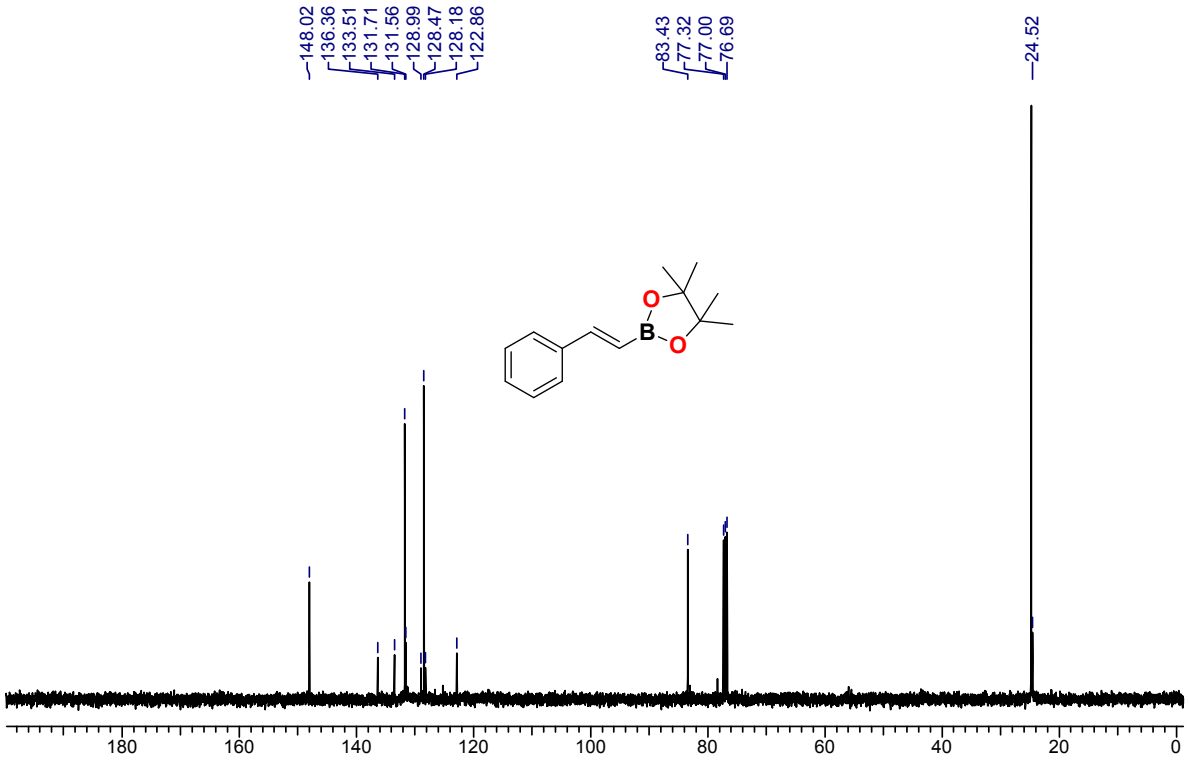



**Figure FS55.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3a.**

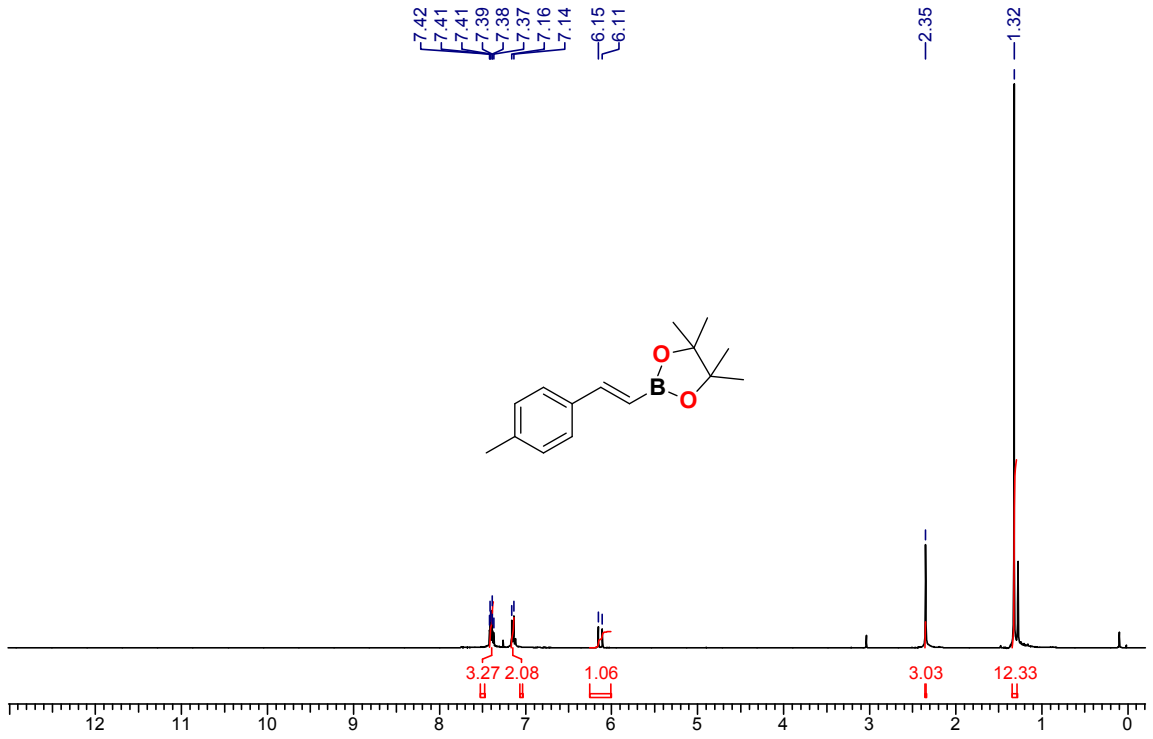



**Figure FS56.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3b.**

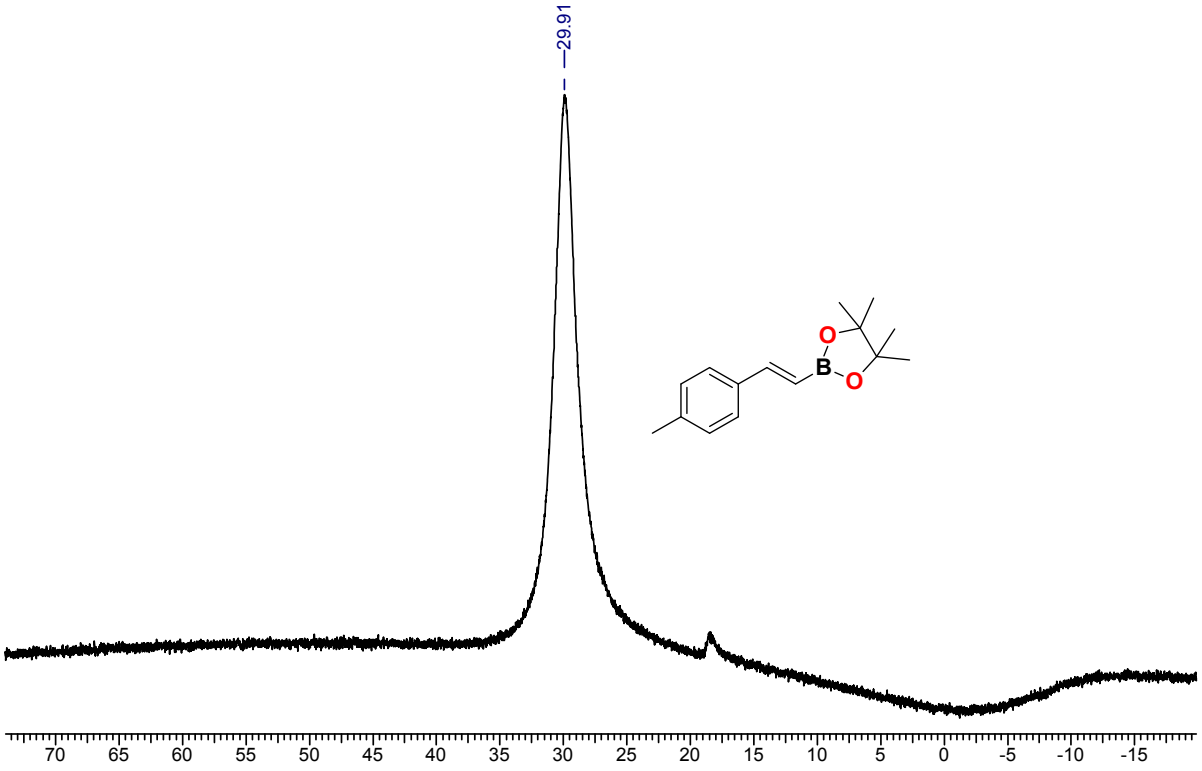



**Figure FS57.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3b**.

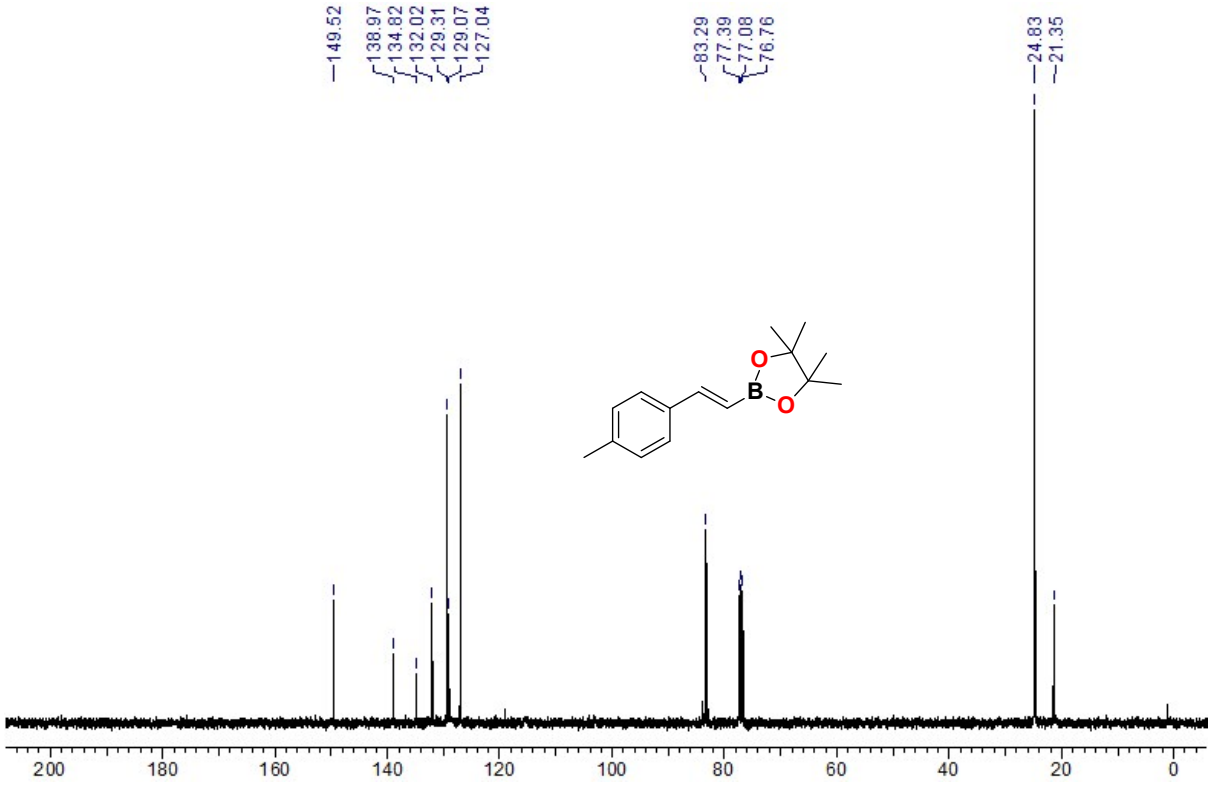



**Figure FS58.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3b.**

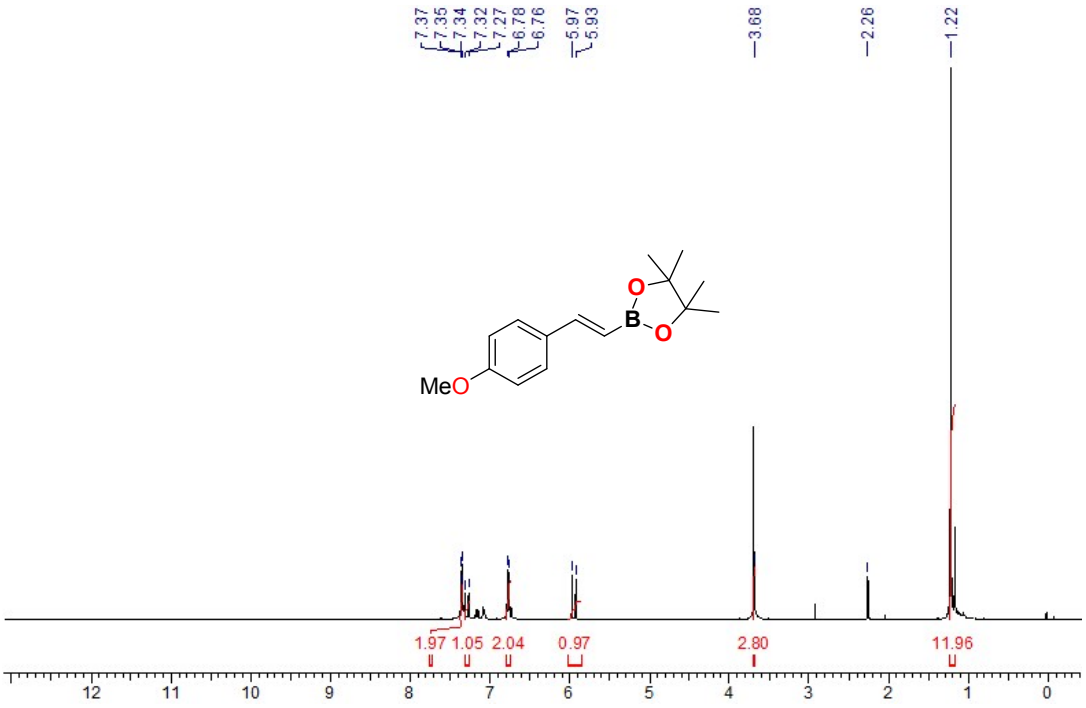



**Figure FS59.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3c.**

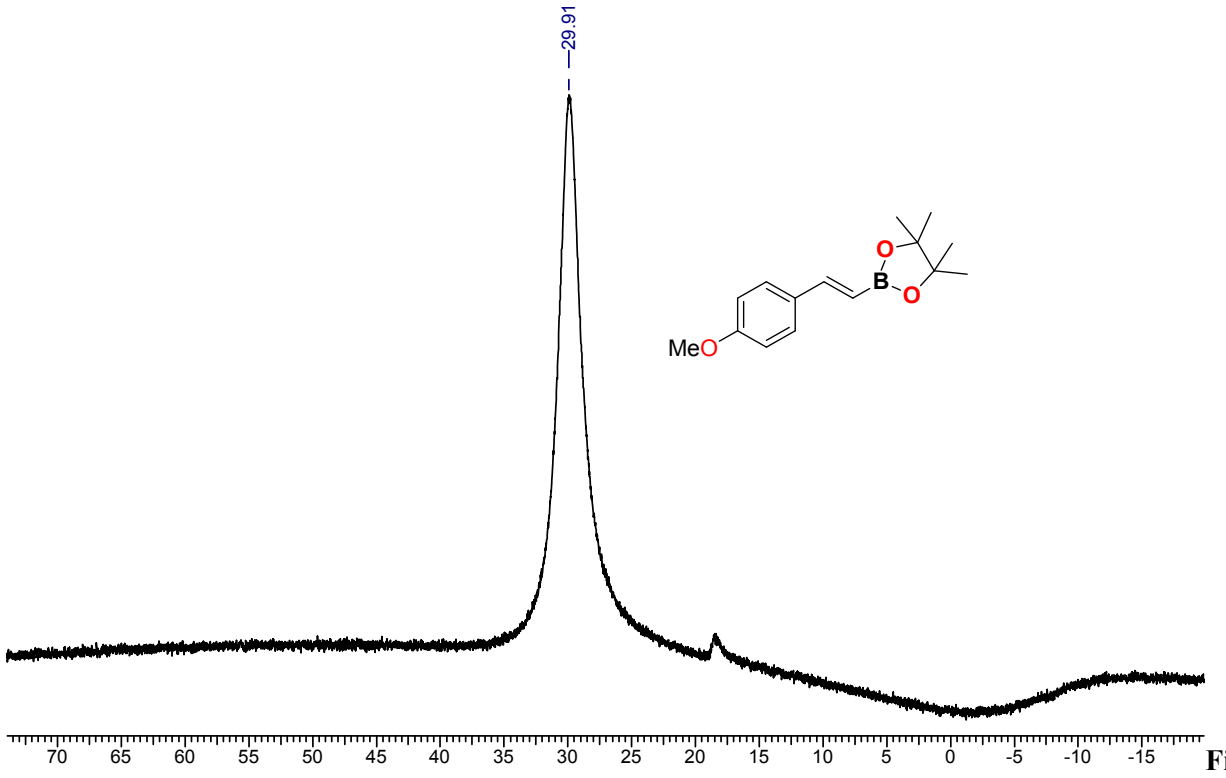



**Figure FS60.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3c**.

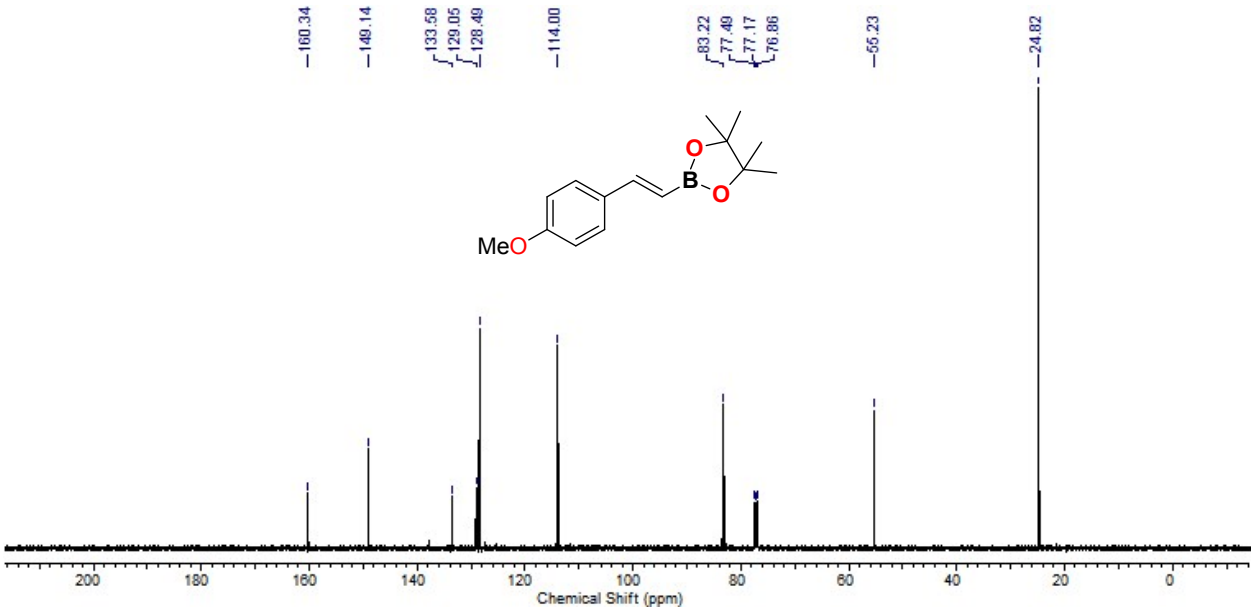



**Figure FS61.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3c.**

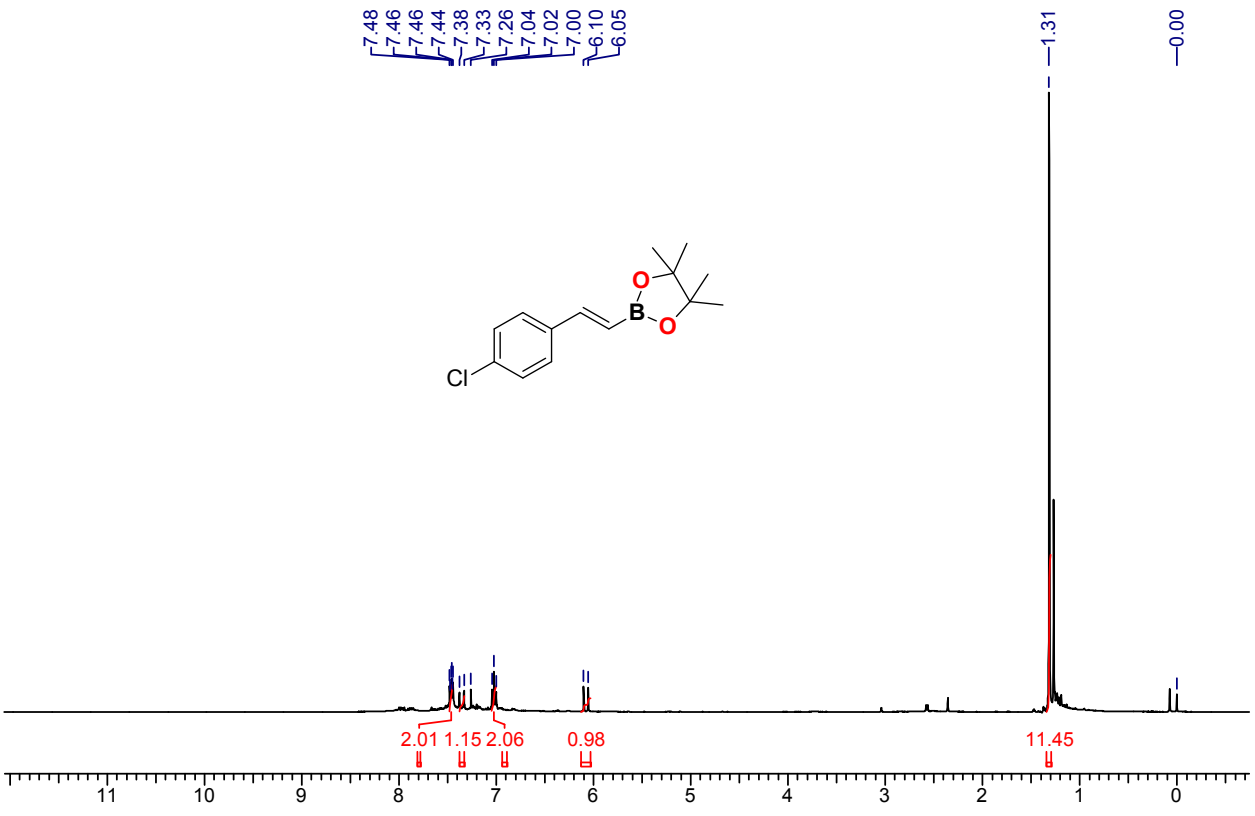



**Figure FS62.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3d.**

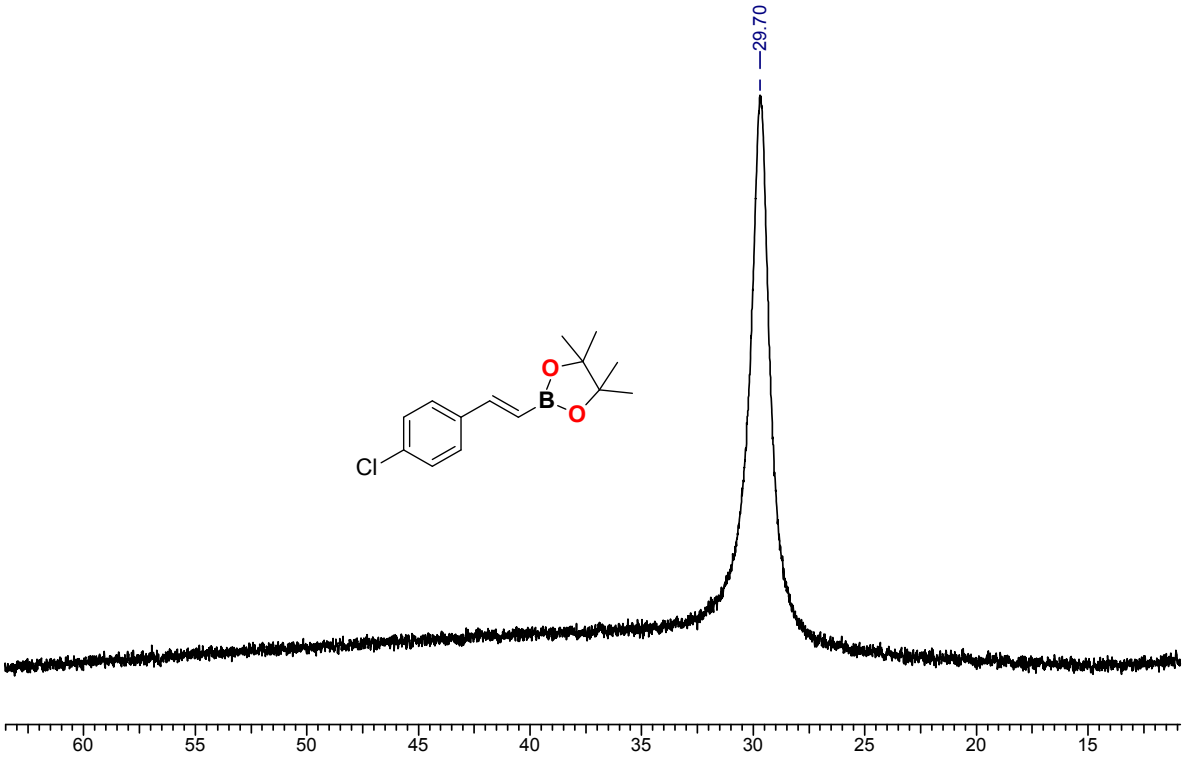



**Figure FS63.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3d**.

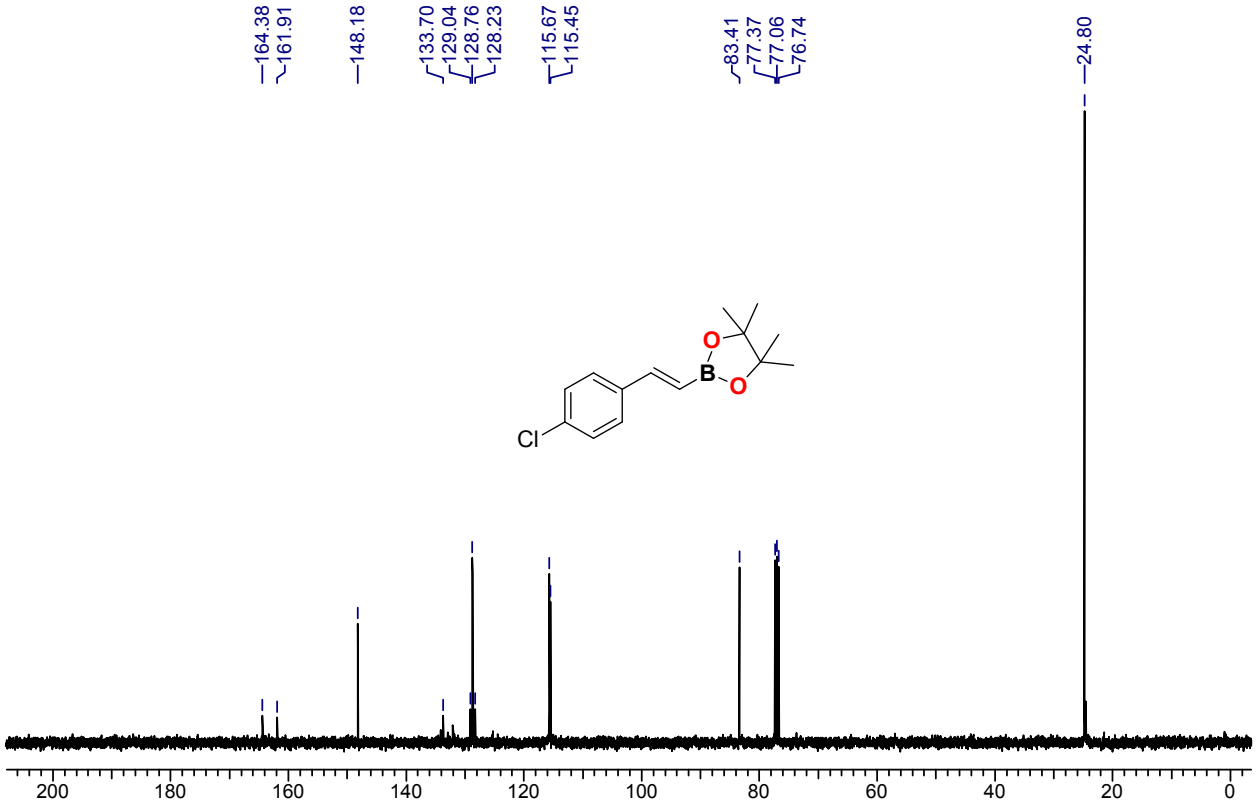



**Figure FS64.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3d.**

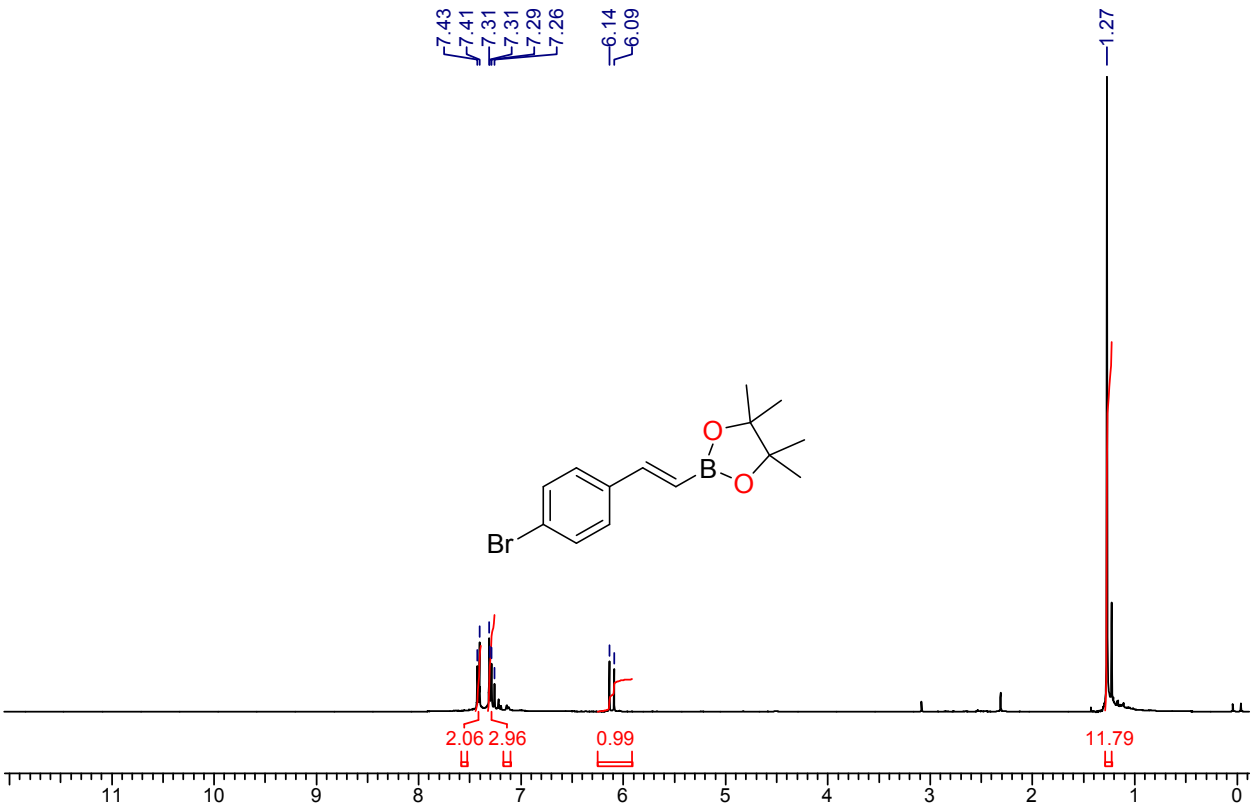



**Figure FS65.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3e.**

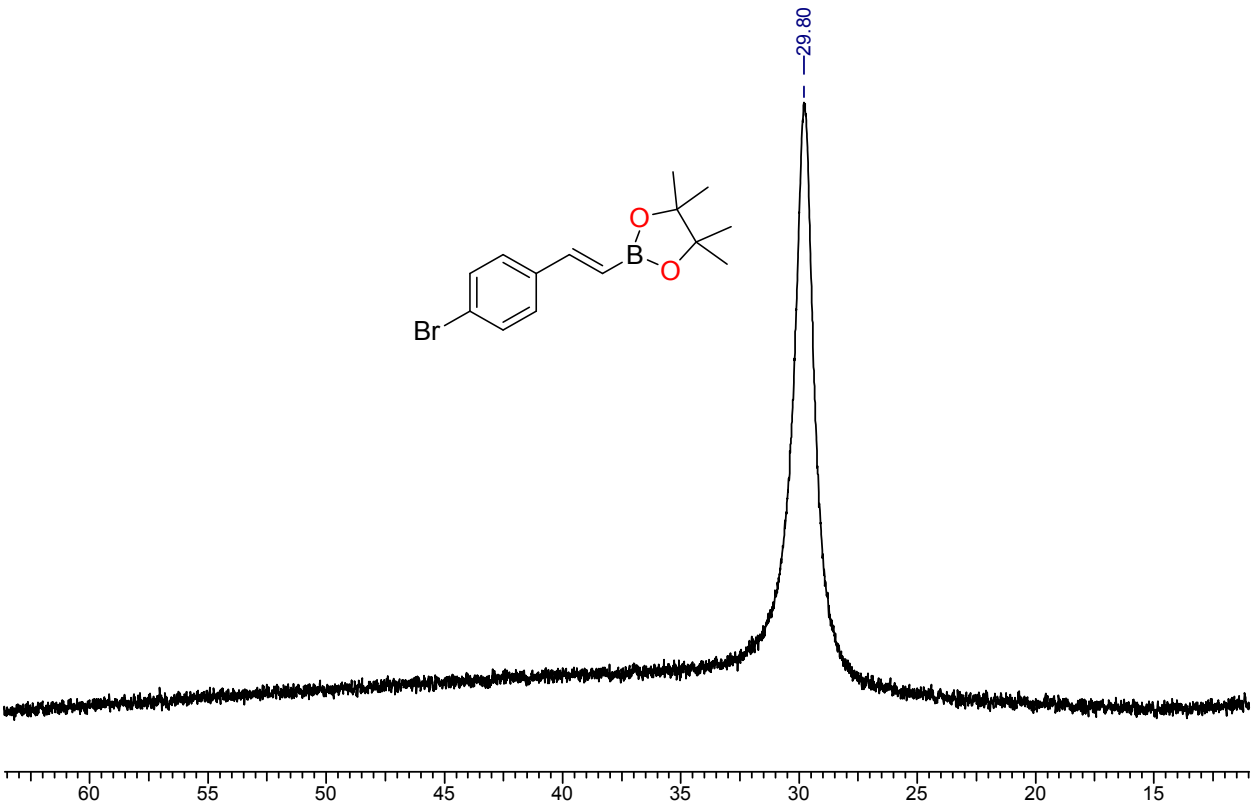



**Figure FS66.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3e**.

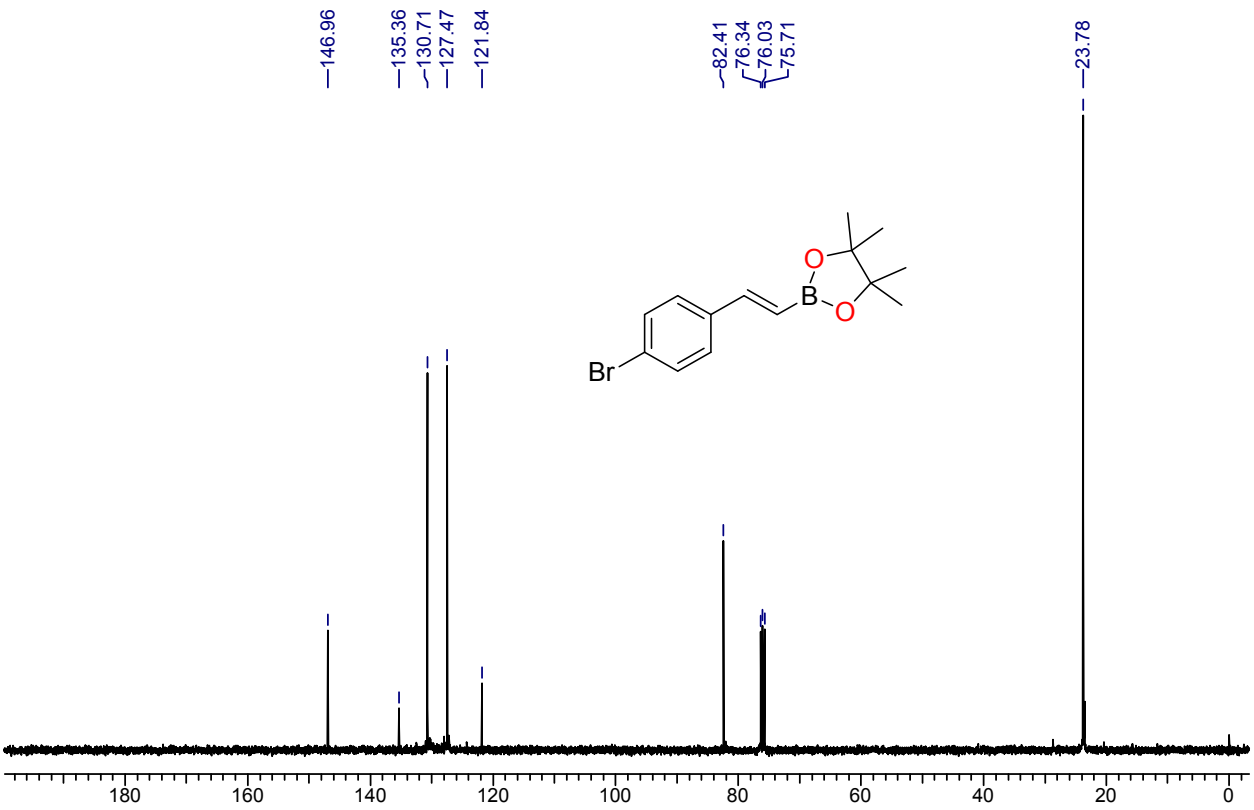



**Figure FS67.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3e.**

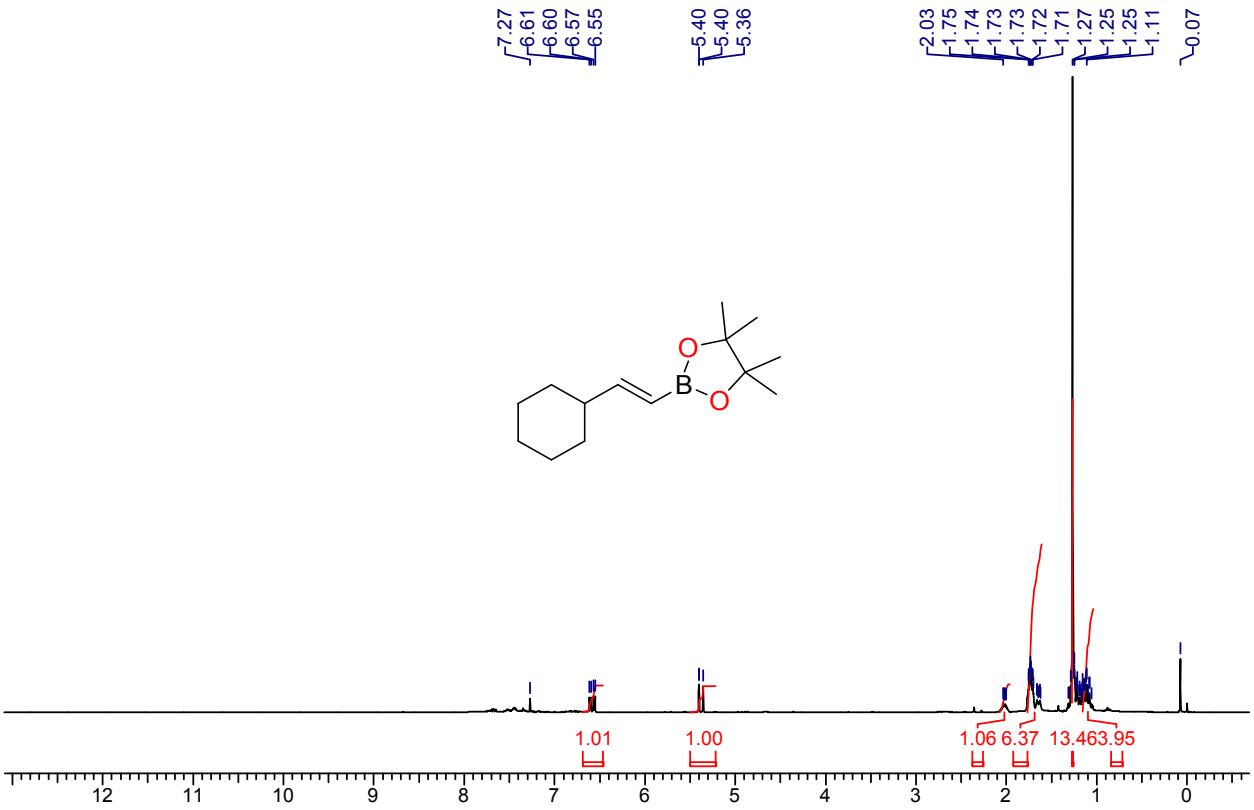



**Figure FS68.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3f.**

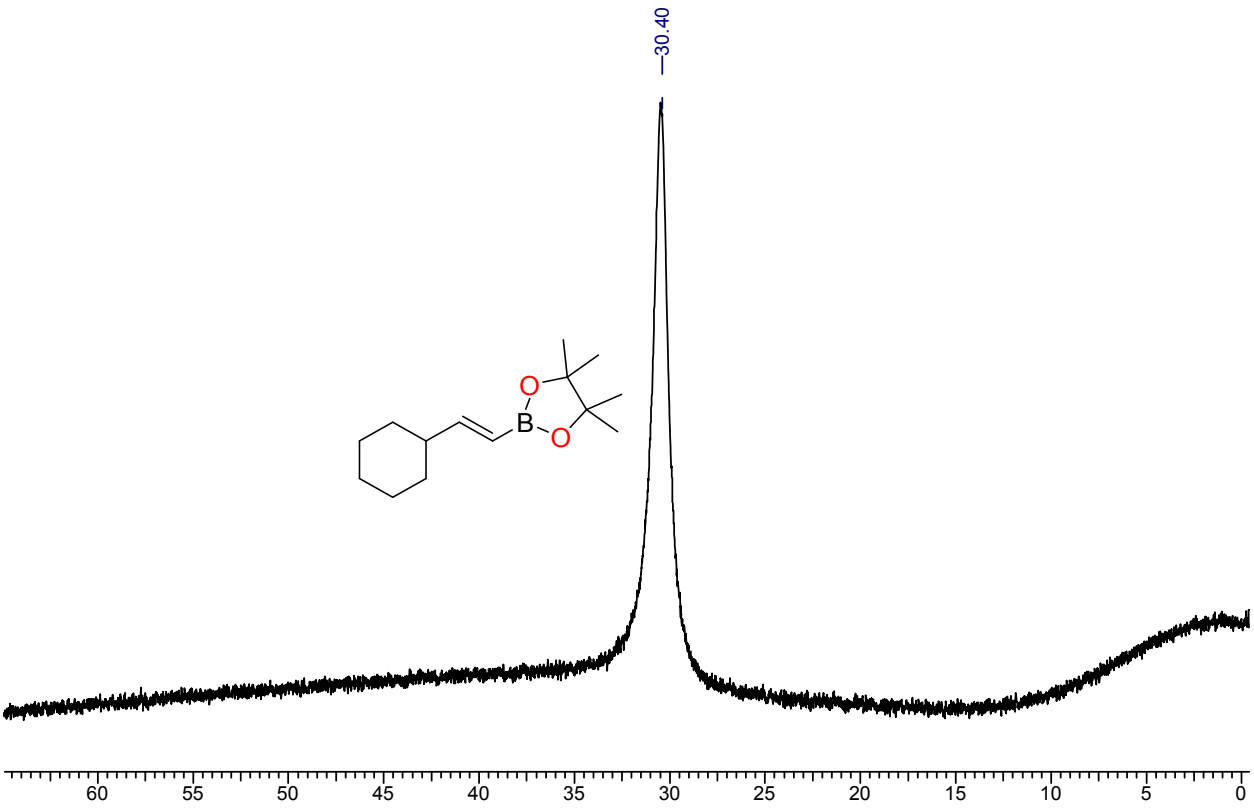



**Figure FS69.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3f**.

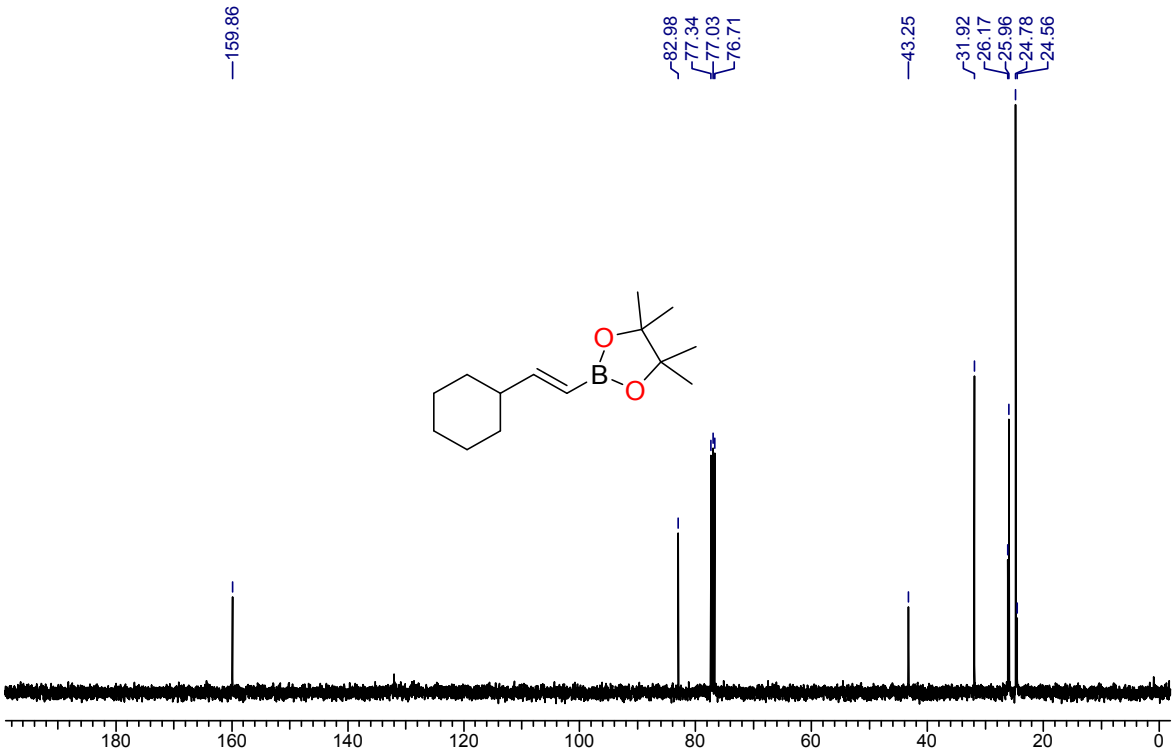



**Figure FS70.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3f.**

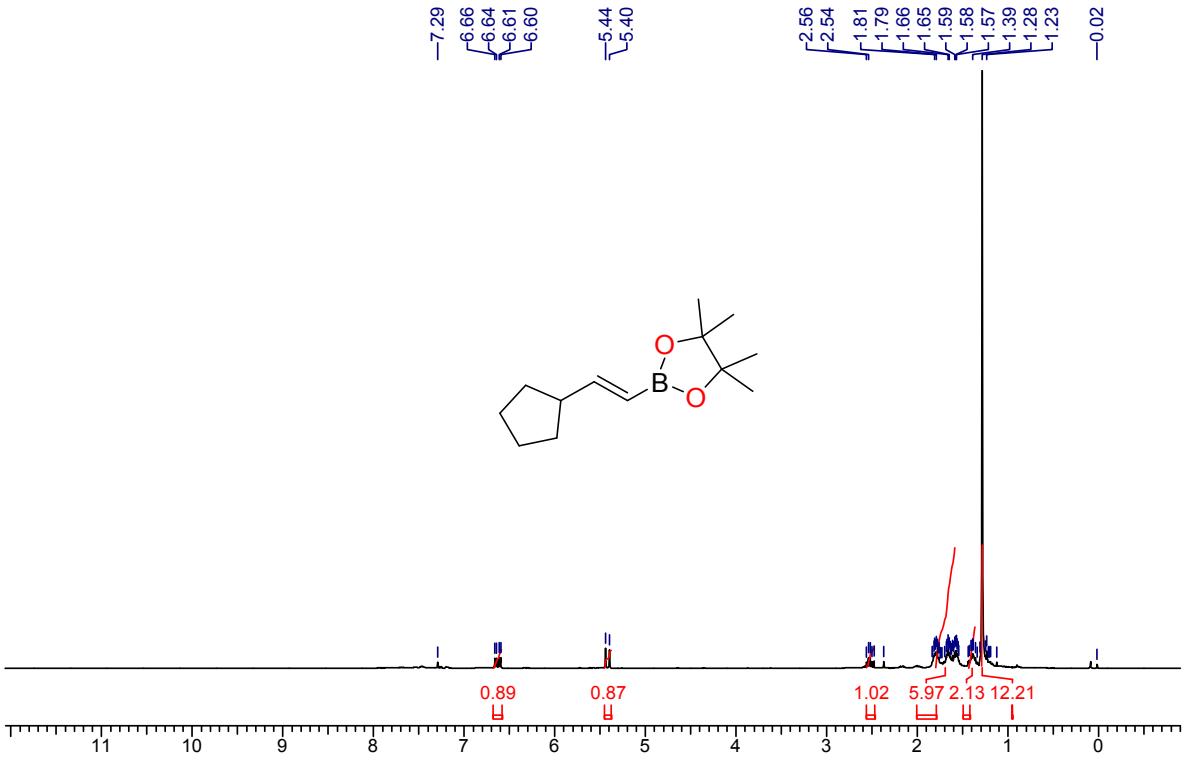



**Figure FS71.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3g.**

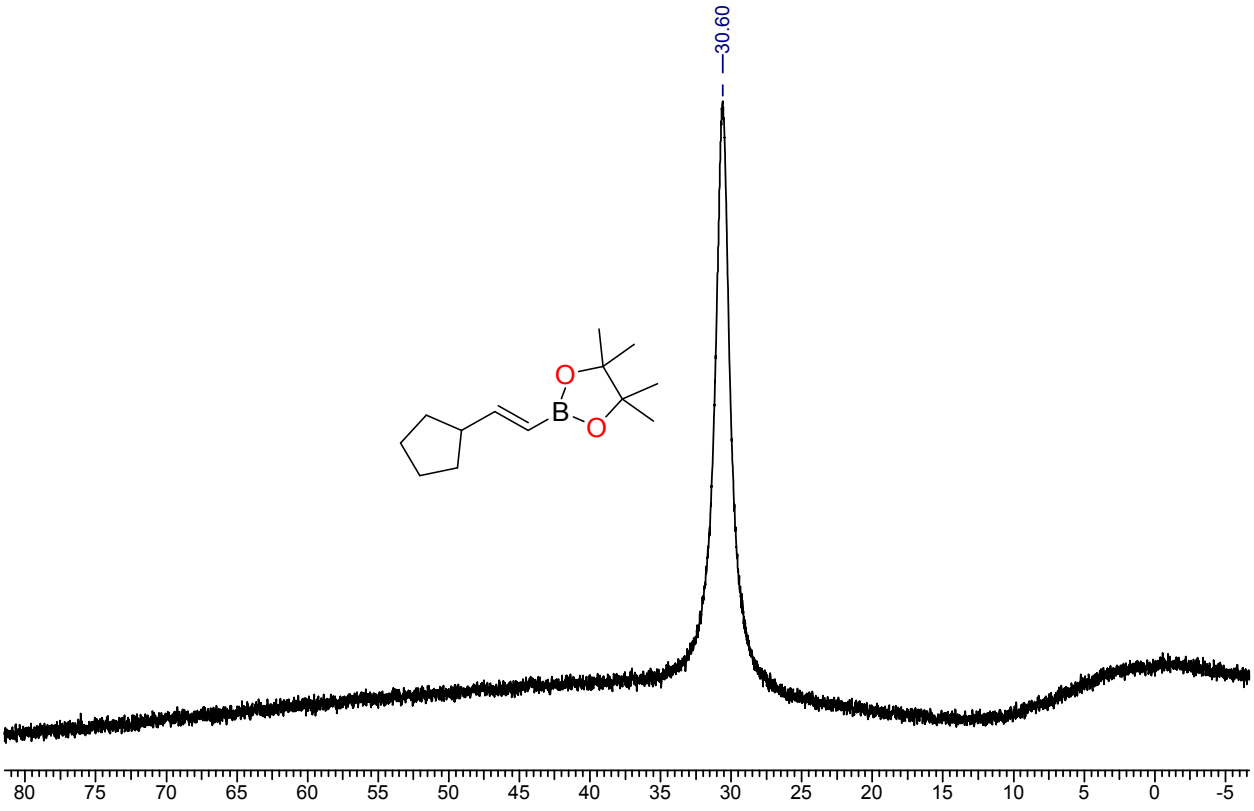



**Figure FS72.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3g**.

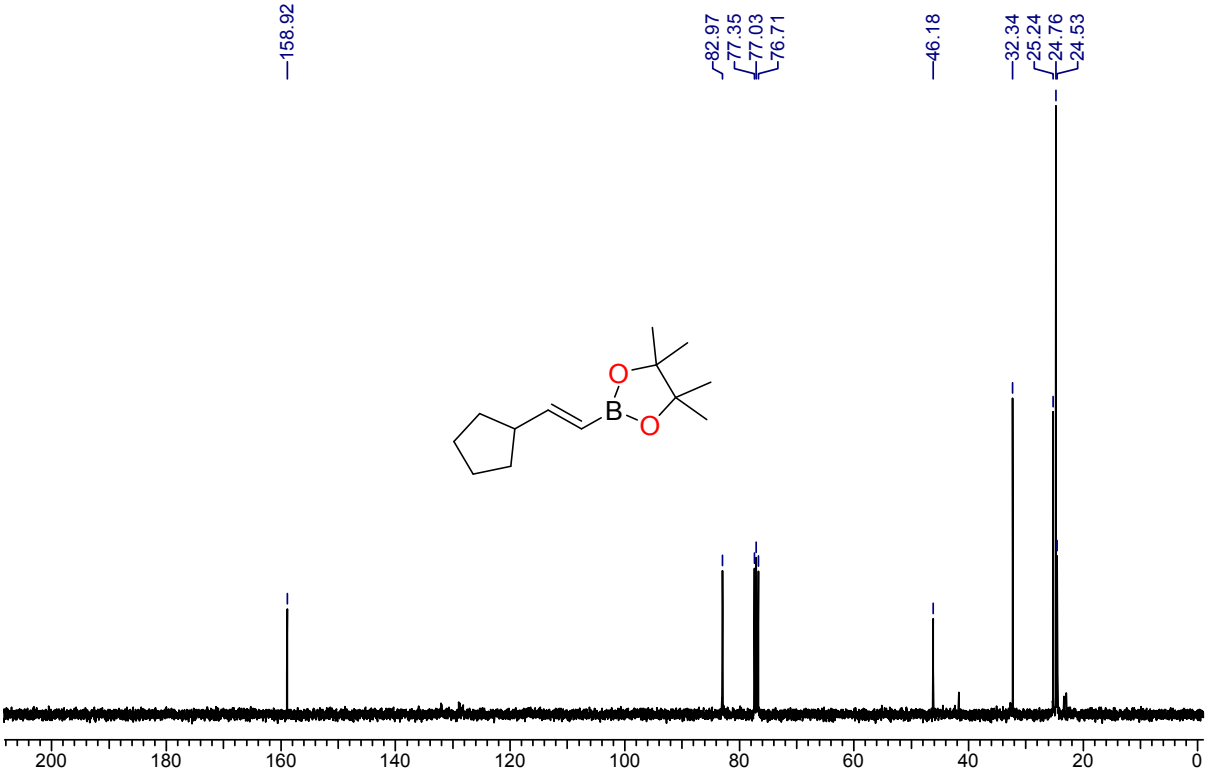



**Figure FS73.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3g.**

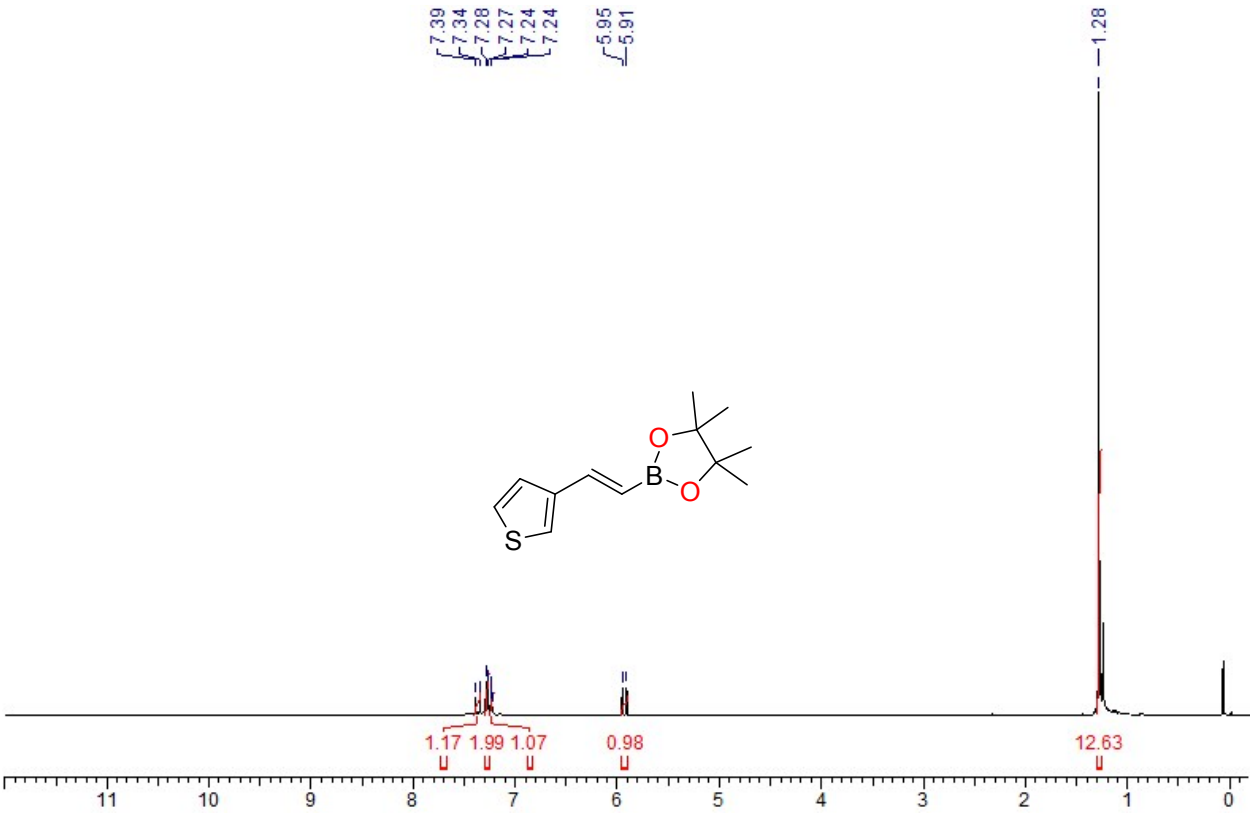



**Figure FS74.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3h.**

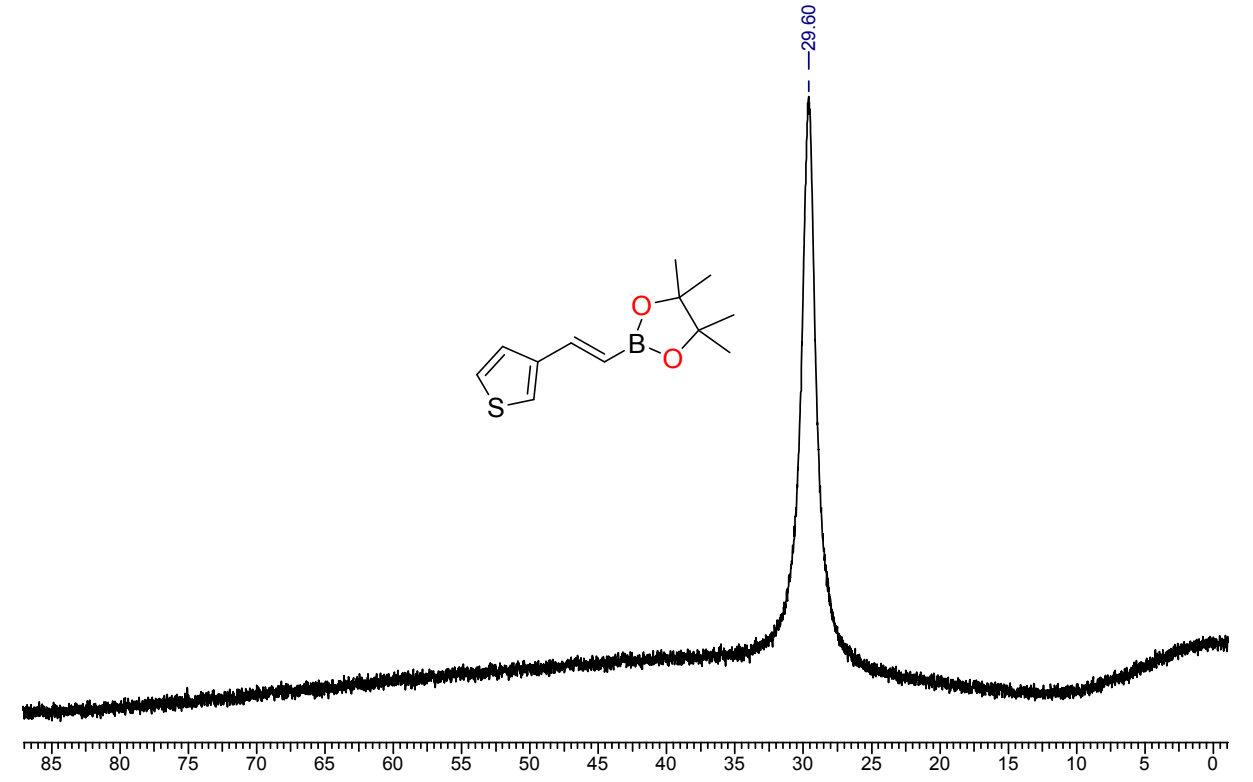



**Figure FS75.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3h**.

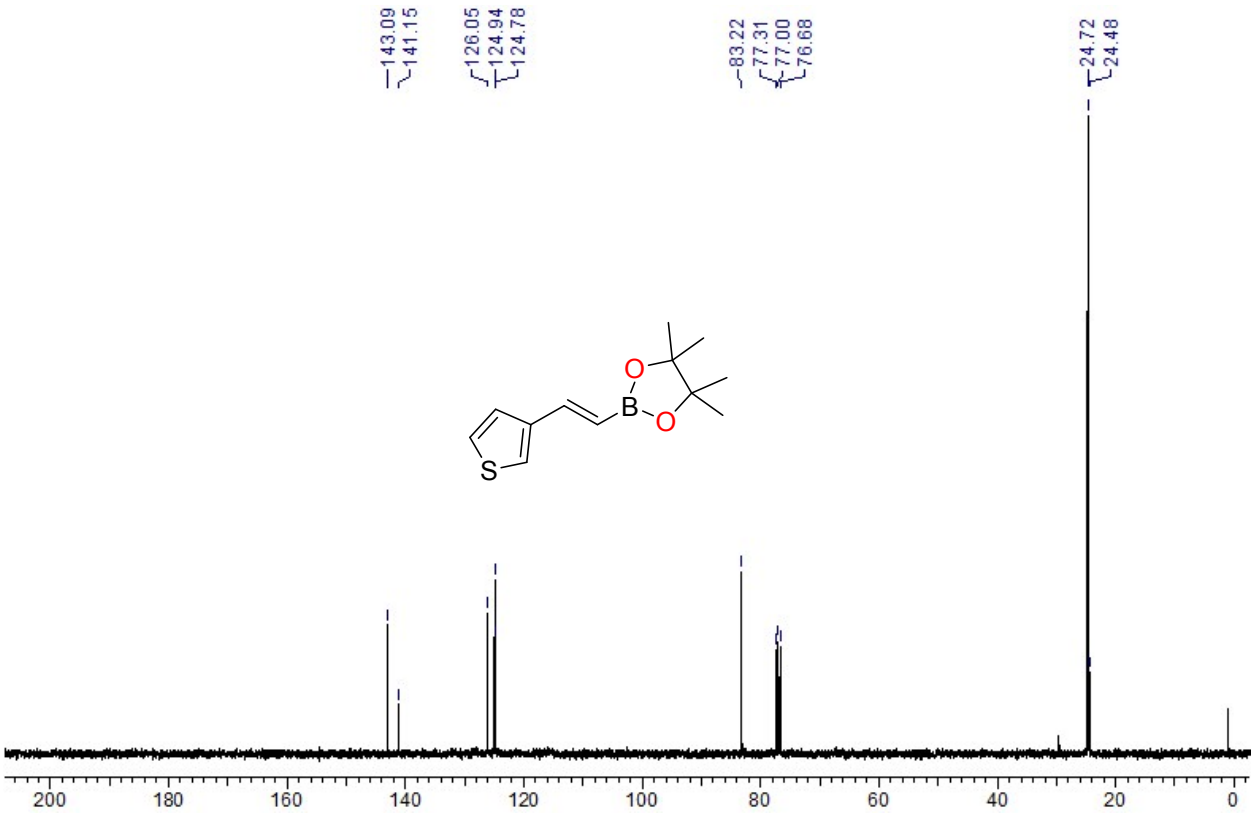



**Figure FS76.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3h.**

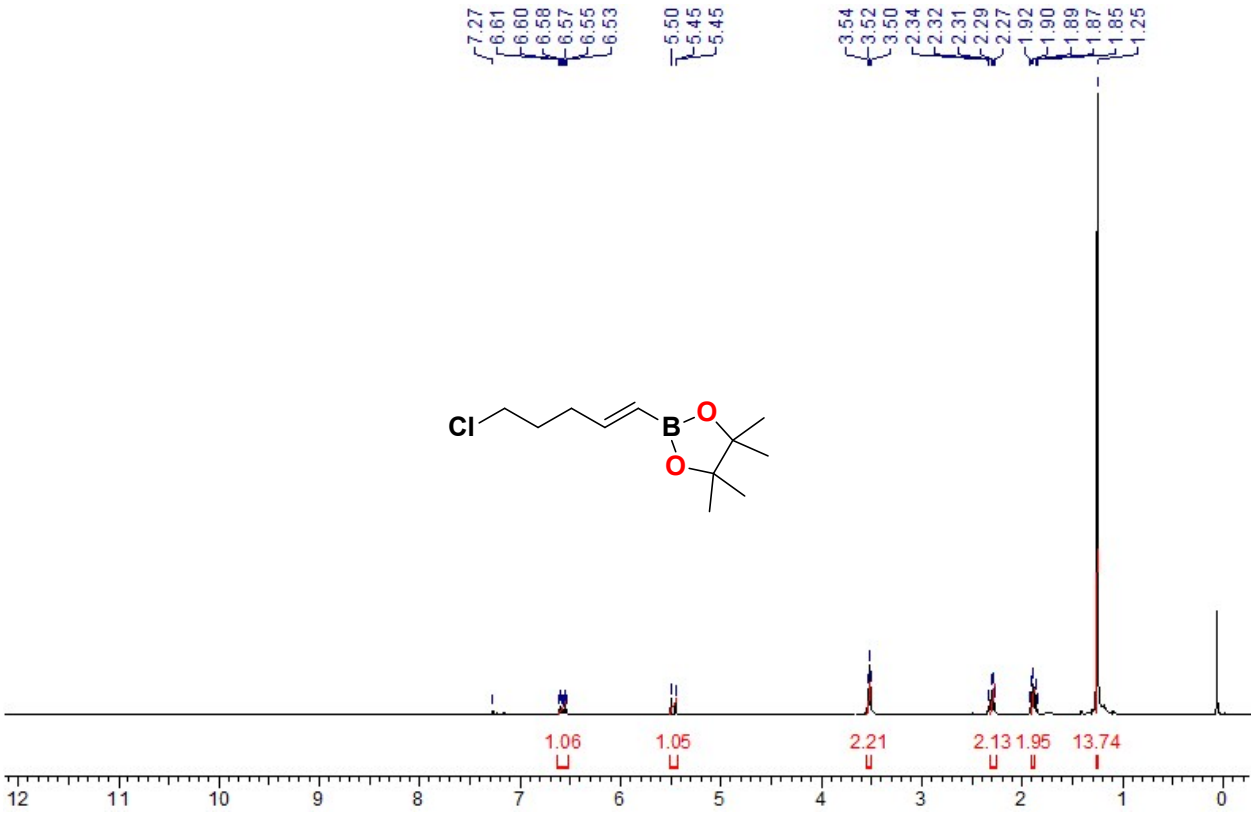


**Figure FS77.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3i.**

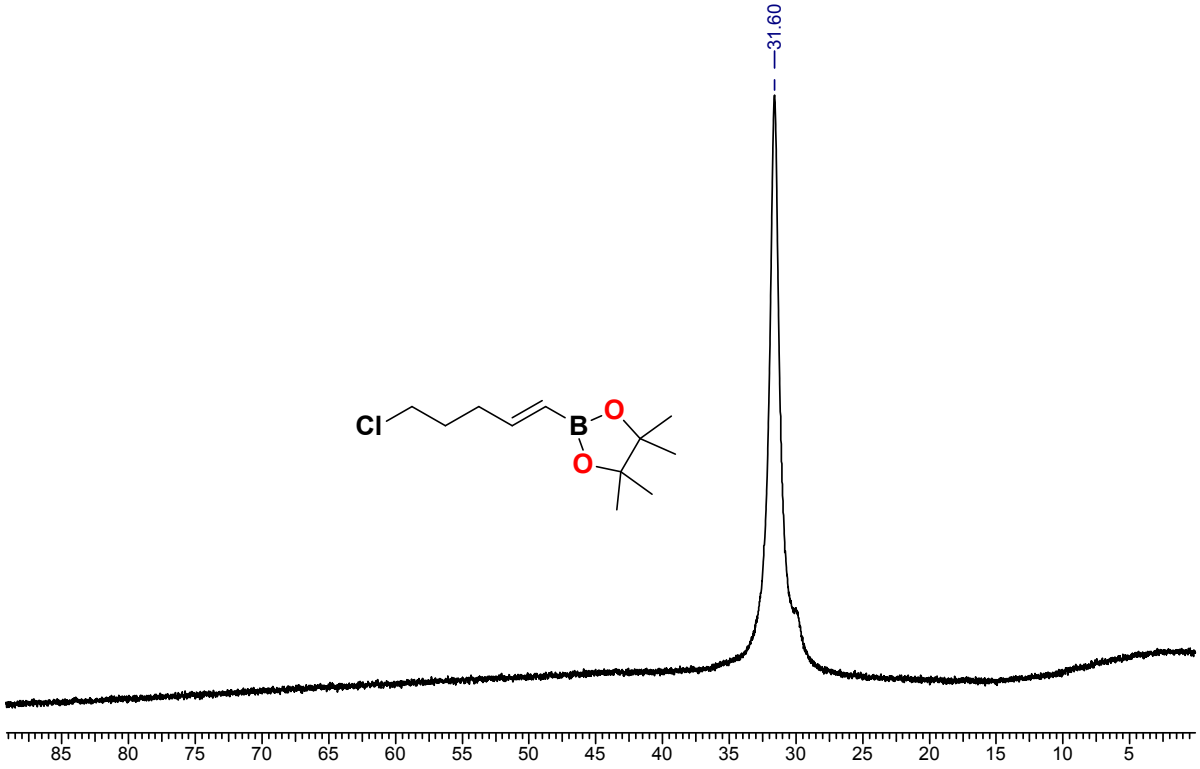



**Figure FS78.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3i**.

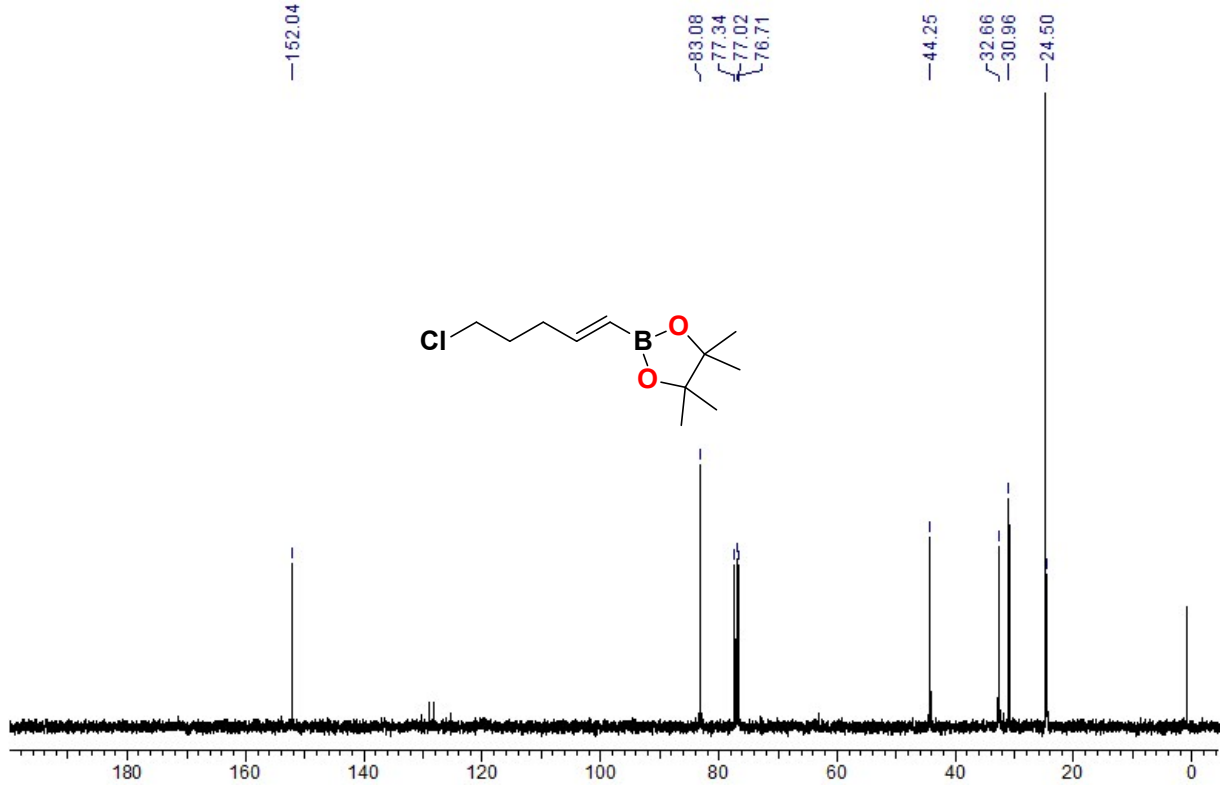


**Figure FS79.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3i.**

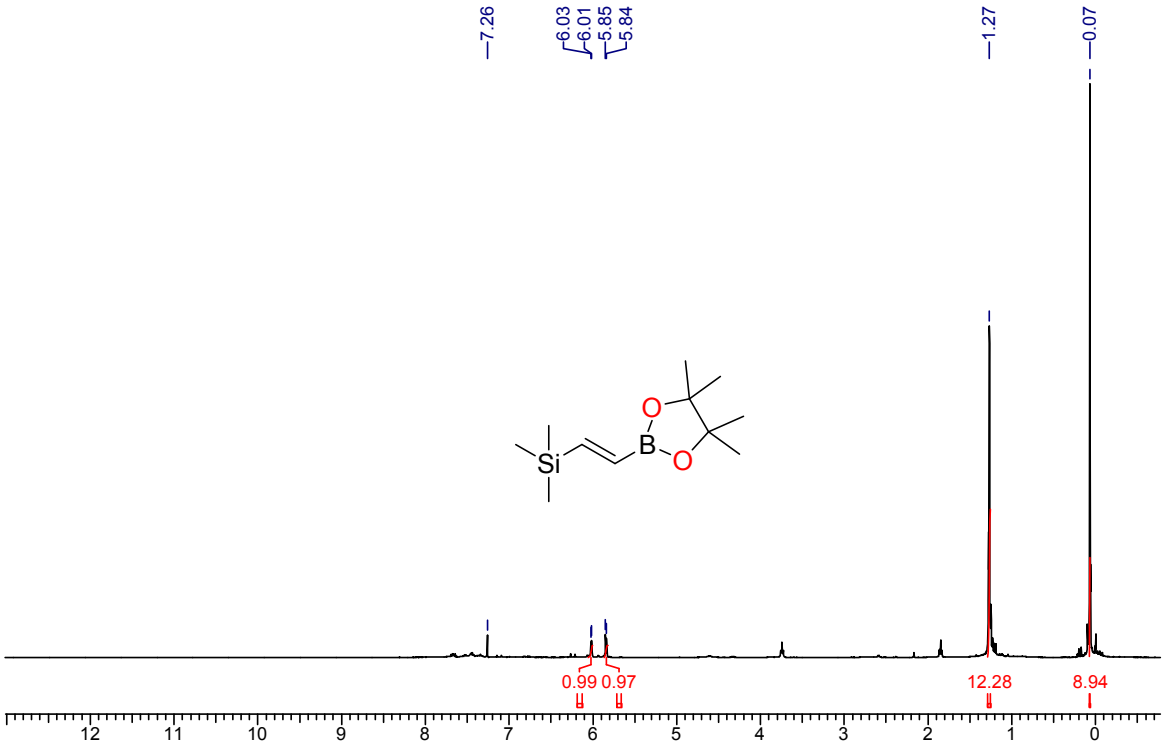



**Figure FS80.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3j.**

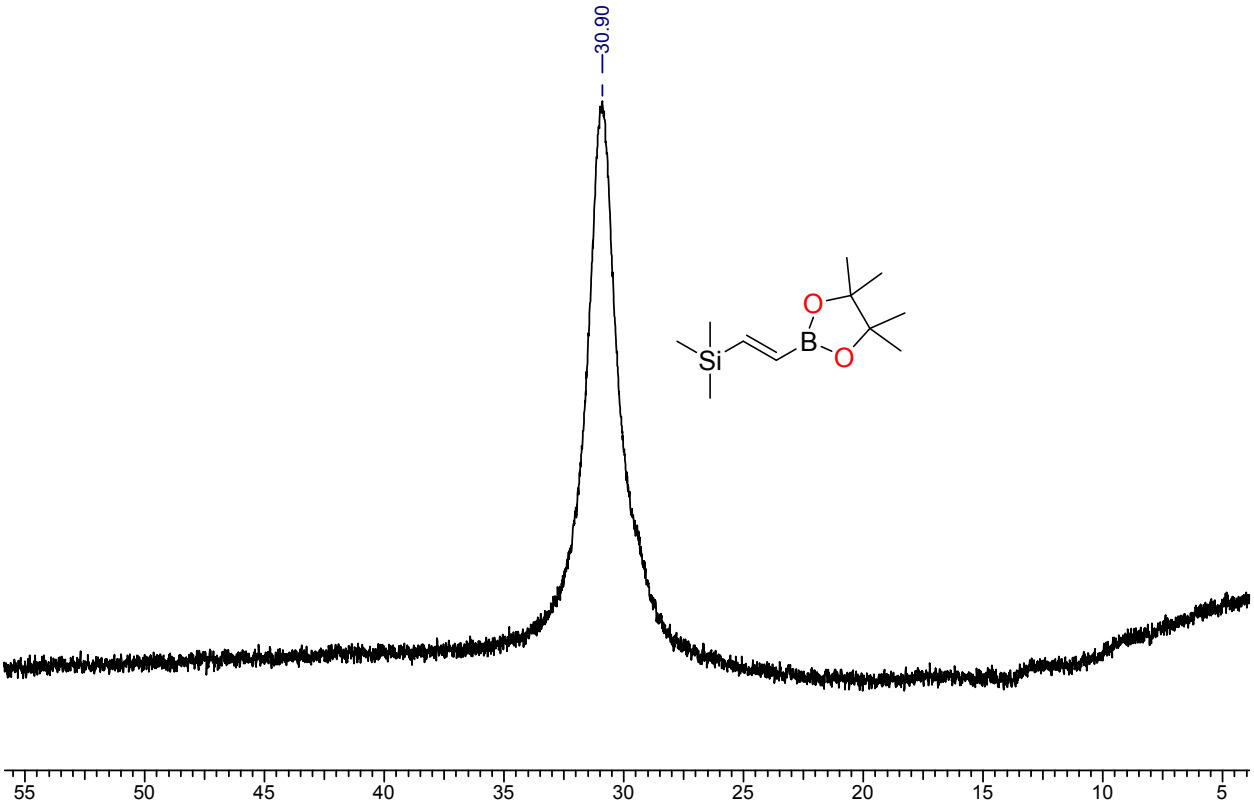



**Figure FS81.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3j**.

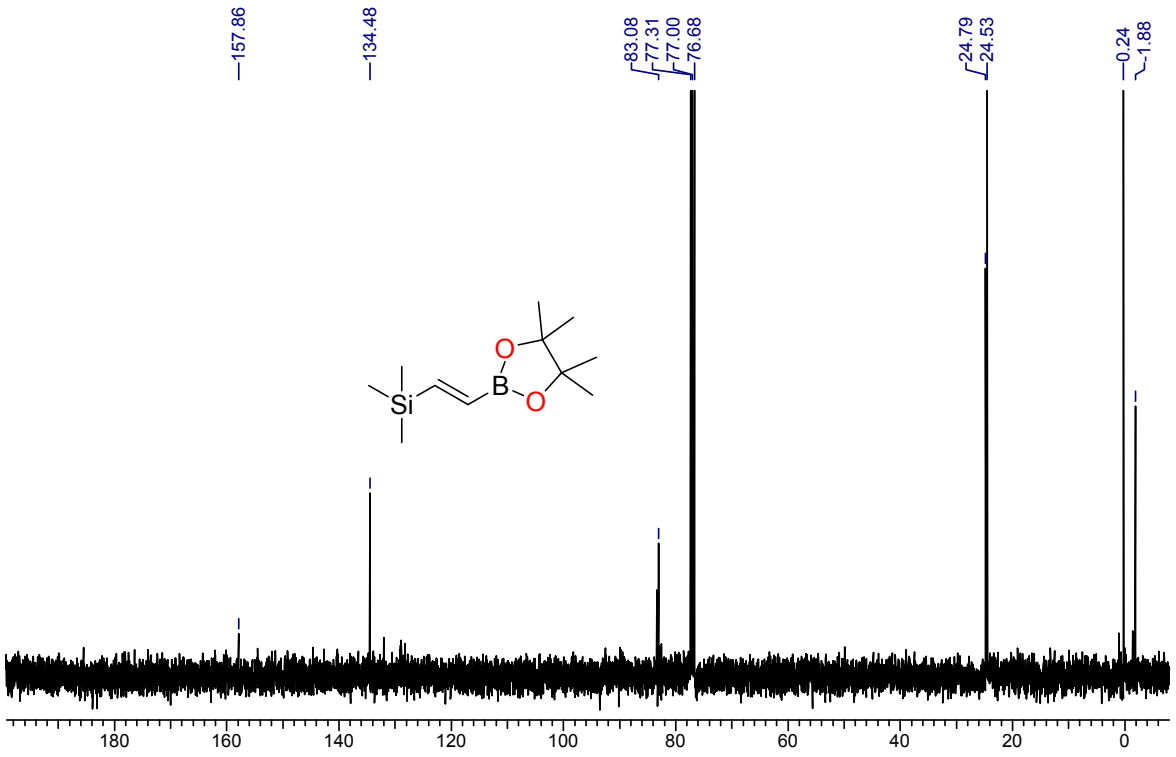



**Figure FS82.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3j.**

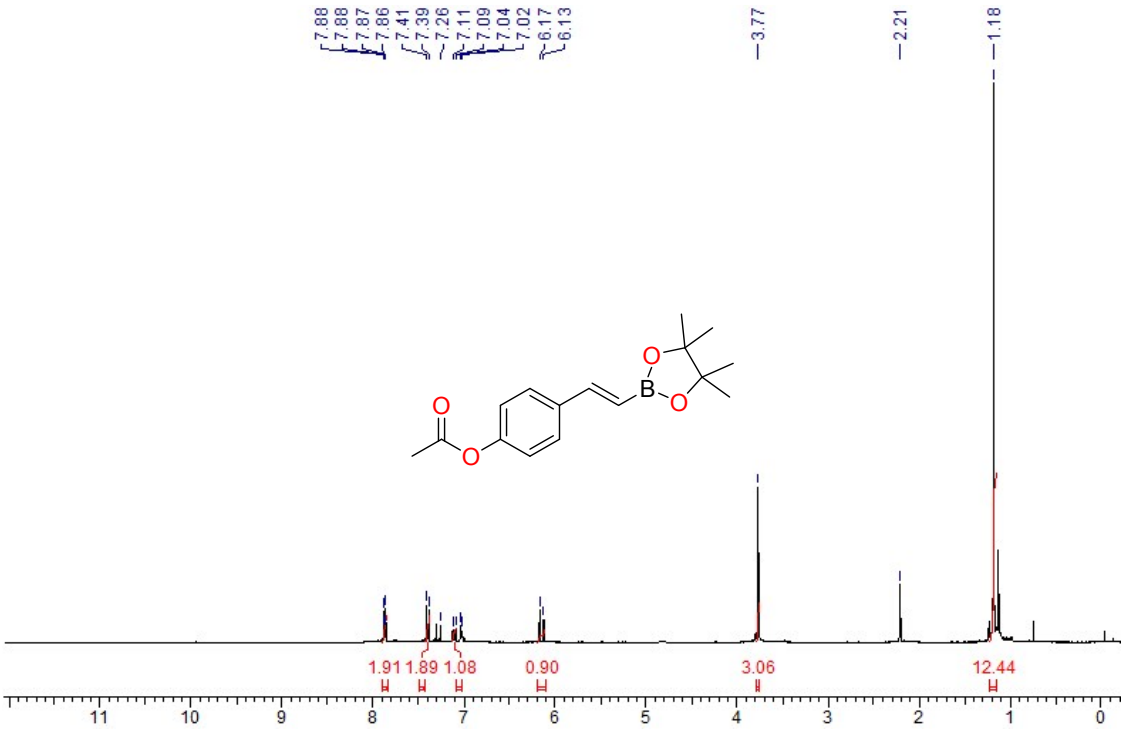



**Figure FS83.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **3k.**

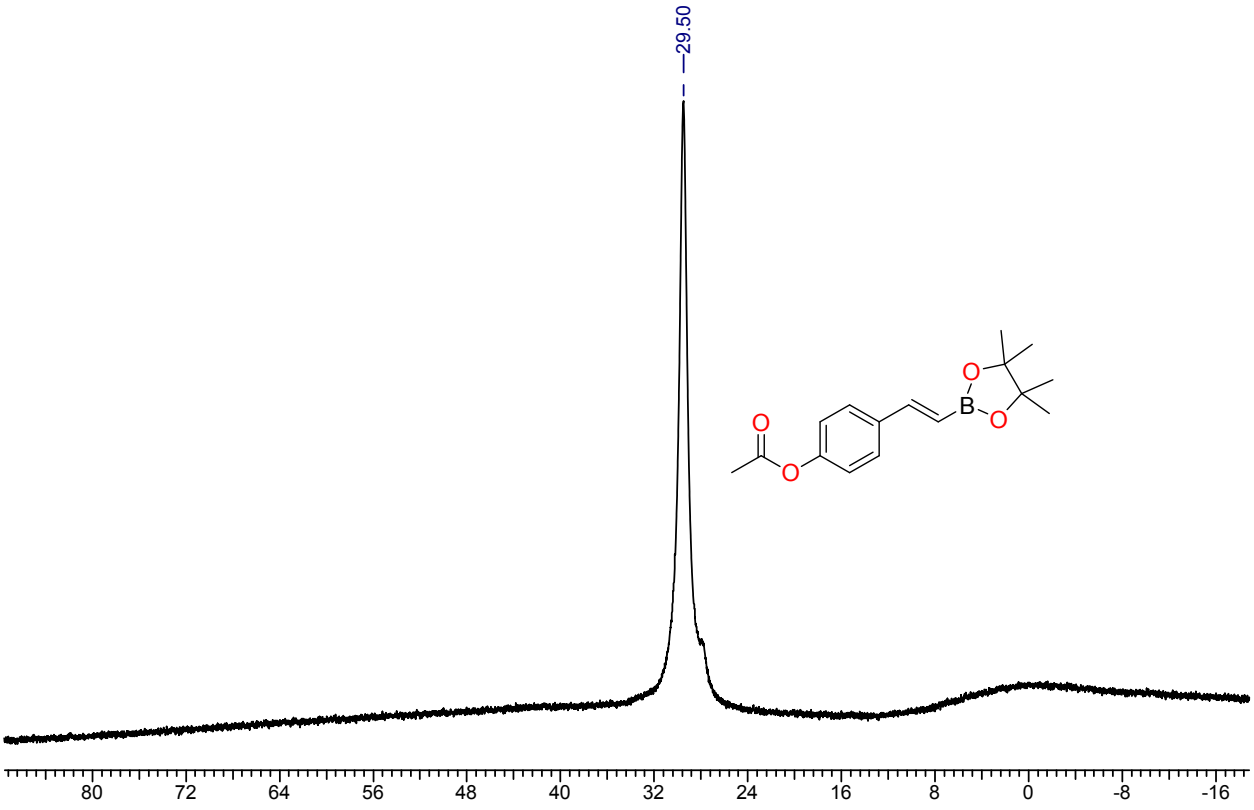



**Figure FS84.** $^{11}B\{^{1}H\}$ NMR (128 MHz, $CDCl_3$) spectrum of **3k**.

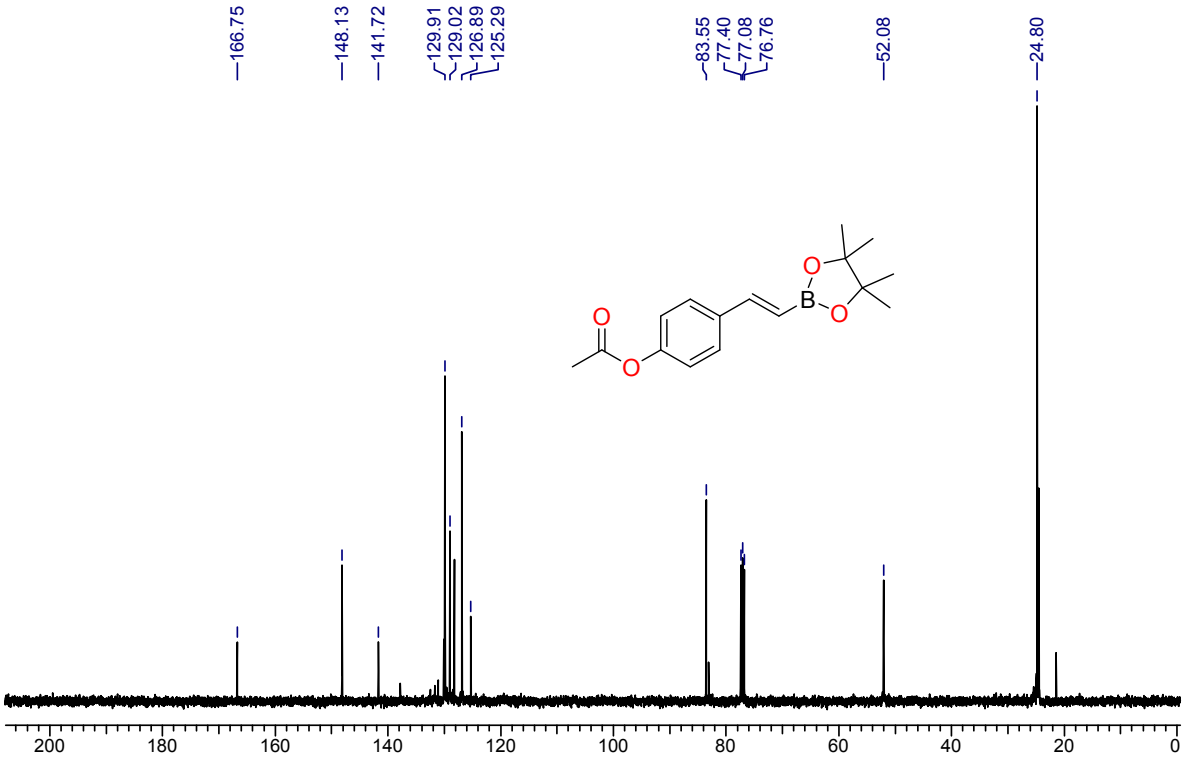



**Figure FS85.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **3k.**

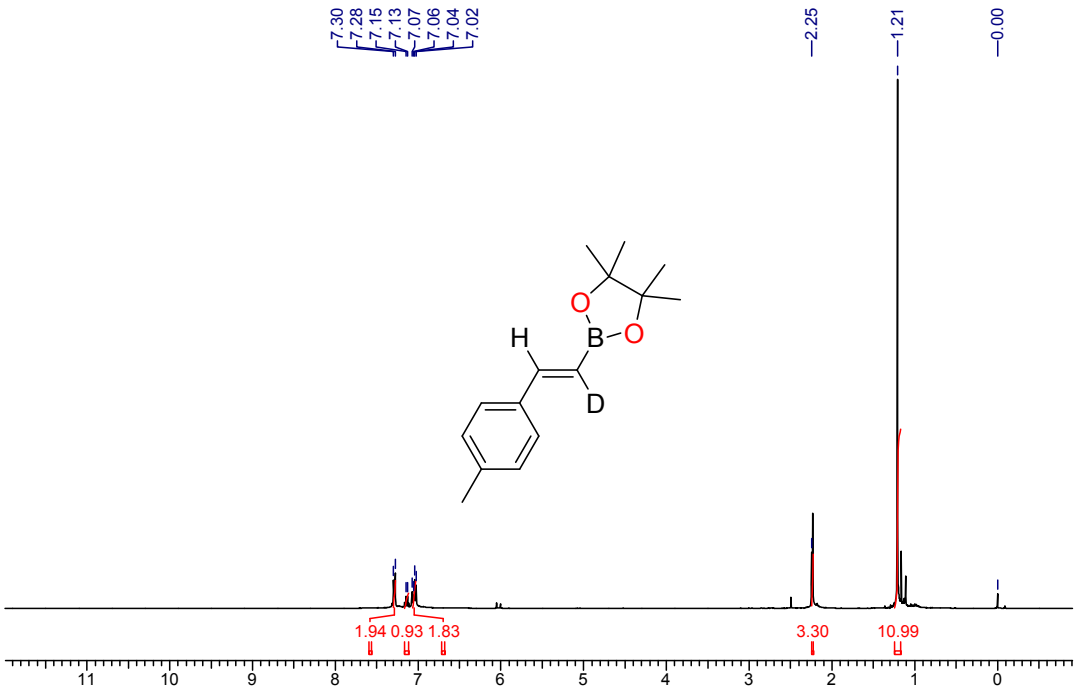



**Figure FS86.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **4a.**

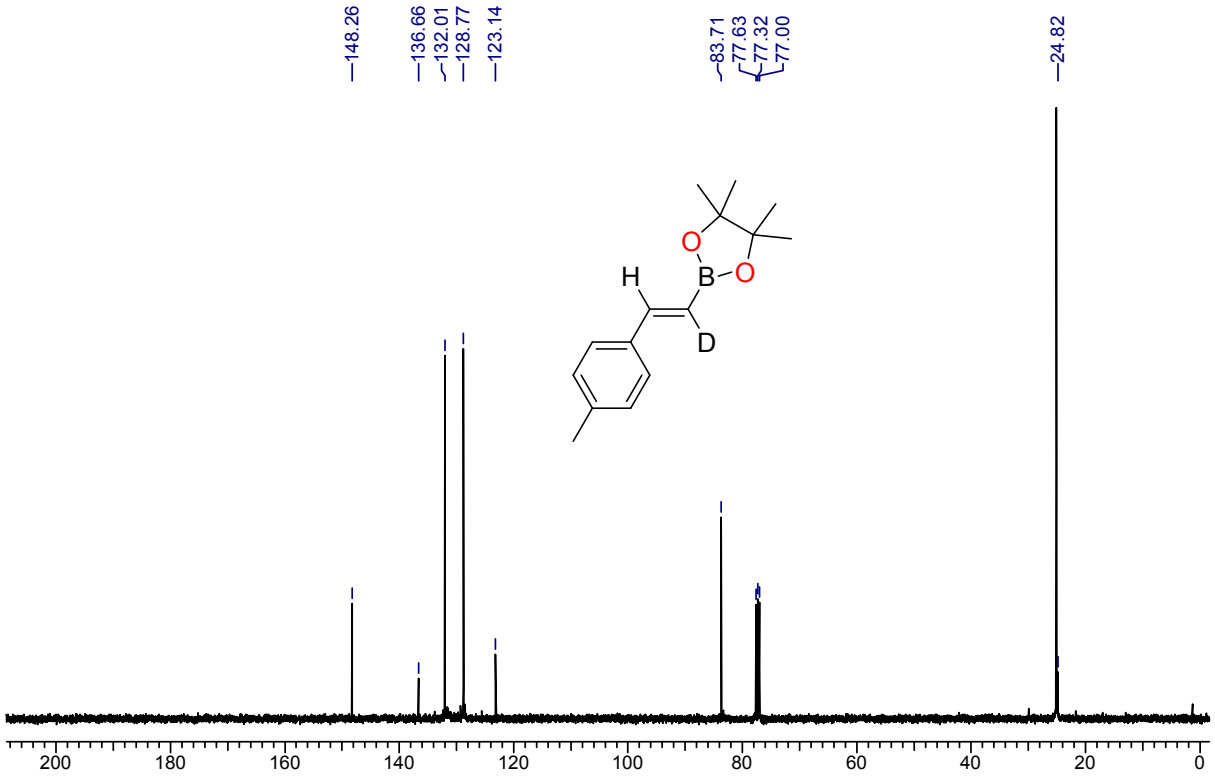



**Figure FS87.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **4a.**

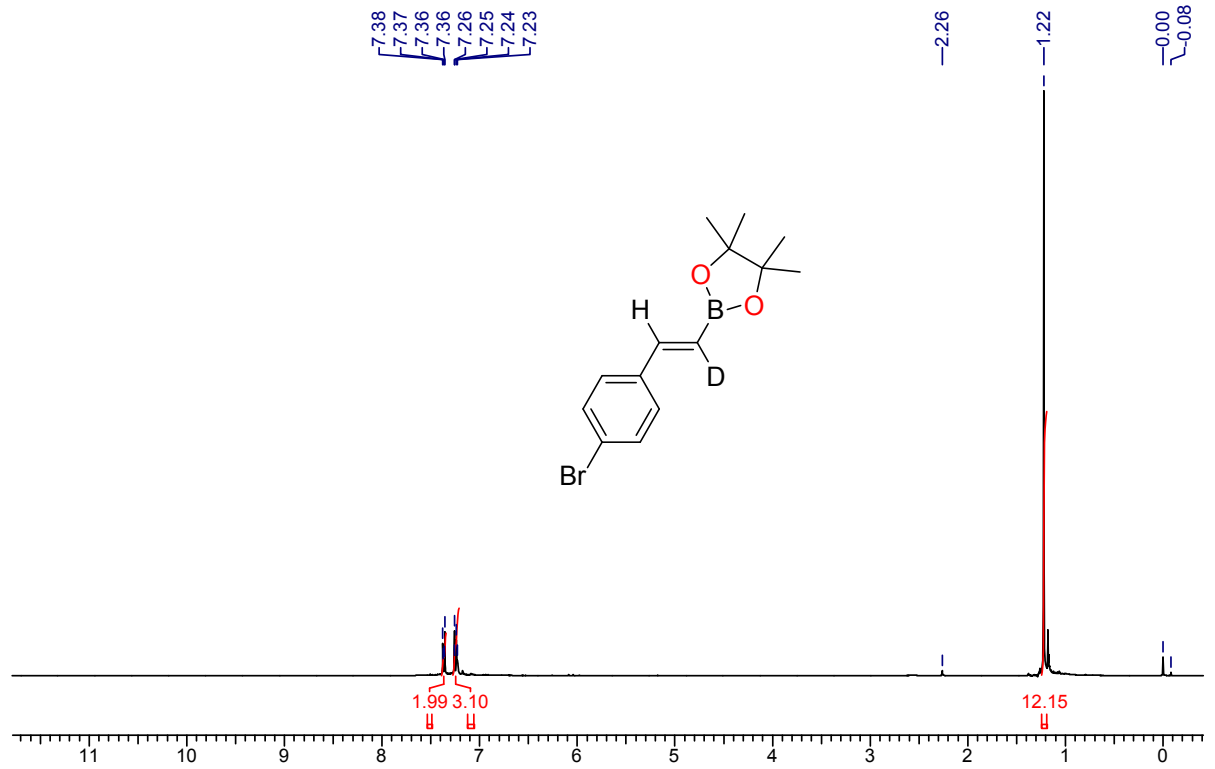



**Figure FS88.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of **4b.**

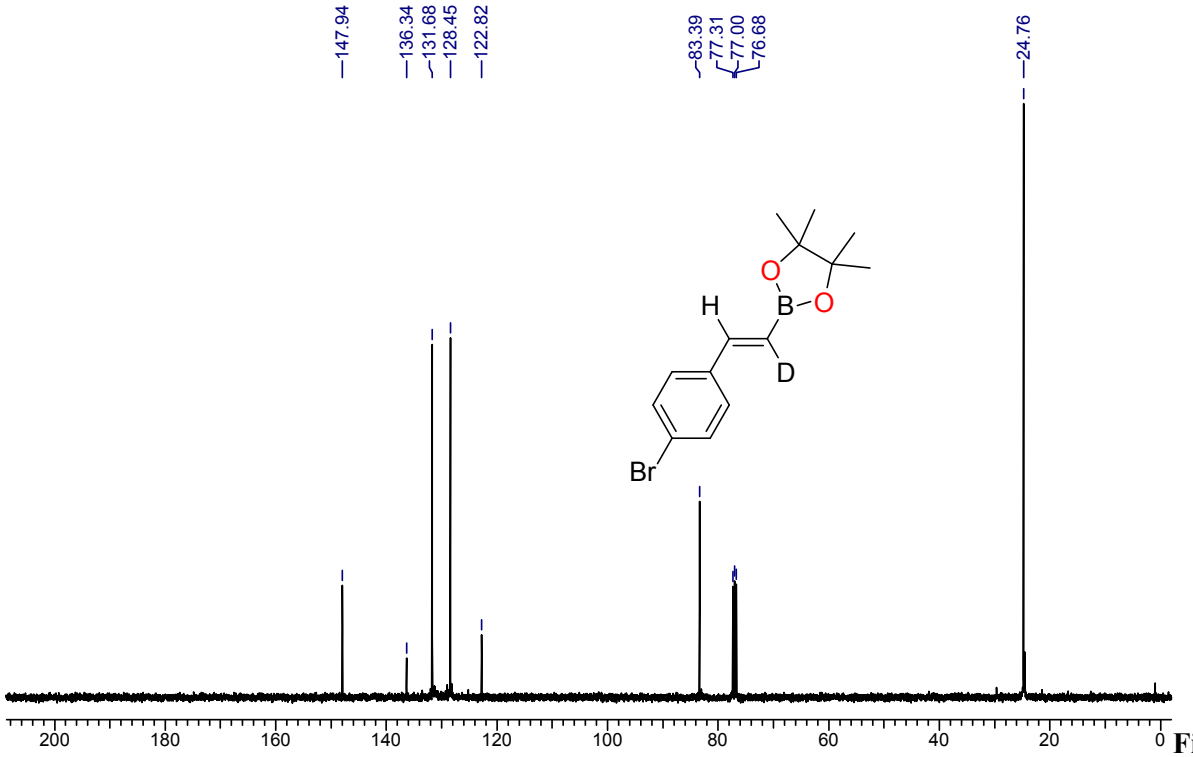



**Figure FS89.** $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) spectrum of **4b.**

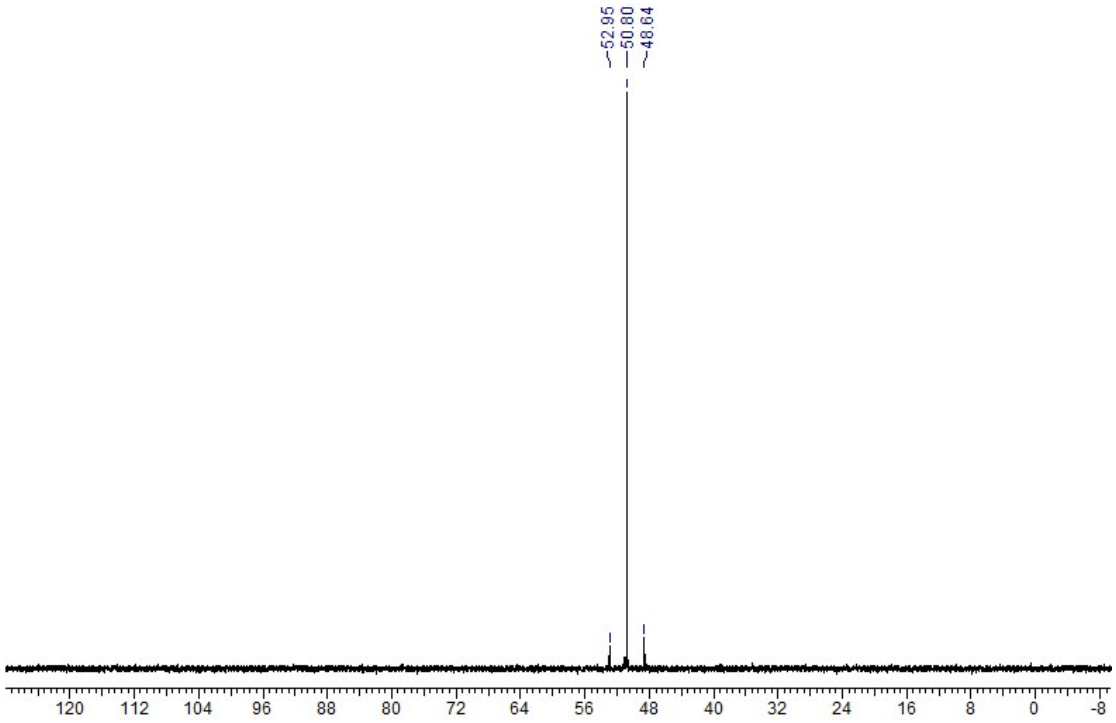



**Figure FS90.** $^{31}P\{^{1}H\}$ NMR spectrum (161.9 MHz, 25°C, $C_6D_6$) reaction mixture

**Reference:**


1. S. Kisan, V. Krishnakumar and C. Gunanathan, *ACS Catal.* 2017, **7**, 5950-5954.
2. A. Bismuto, M. J. Cowley and S. P. Thomas, *ACS Catal.* 2018, **8**, 2001-2005.
3. G. Zhang, H. Zeng, J. Wu, Z. Yin, S. Zheng, and J. C. Fettinger. *Angew. Chem. Int. Ed.* 2016, **55**, 14369 –14372.
4. S. Mandal, P. K. Verma and K. Geetharani, *Chem. Commun.,* 2018, **54**, 13690-13693.