Supporting Information

Regio- and Stereocontrolled Synthesis of Borylated *E*-Enynes, *Z*-Enediynes and Derivatives from Alkenyl-1,2-Bis-(boronates)

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I. General information.

Unless otherwise noted, all solvents and all commercially available chemicals were used without further purification. Air- and water-sensitive reactions were performed in flame-dried glassware under an argon atmosphere. Anhydrous tetrahydrofuran was obtained after distillation over sodium/benzophenone and anhydrous dichloromethane after distillation over calcium hydride. For oxygen-sensitive reaction, when specified, the solvent was degassed prior to use by slow bubbling of argon. ^{1}H NMR (300, 400 or 500 MHz), ^{13}C NMR (101 or 126 MHz), ^{11}B (128 MHz), and ^{19}F (376 MHz) spectra were recorded on Bruker AC 300, AC 400 and AC 500 spectrometers. Chemical shifts δ are given in ppm, and coupling constants *J* in Hertz. Multiplicities are presented as follows: s = singlet, d = doublet, t = triplet, q = quartet, hex = hexuplet, m = multiplet, br = broad. High-resolution mass spectra (HRMS) were recorded on Q TOF (Agilent) using positive-ion electron-spray ionization techniques (ESI^+). The compounds were purified by column chromatography on 0.060−0.200 mm, 60 Å silica. Analytical thin-layer chromatography was performed on Merck silica gel 60 F254 plates. The ethereal solution of CH_2N_2 was generated from *N*-nitroso-*N*-methylurea according to the literature.[1]

II. Synthesis and characterization of compounds 3 to 10.

(E)-2-(5-(4-(Benzyloxy)-3-methoxyphenyl)-1-cyclohexylpent-2-en-4-yn-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3a). Prepared according to the general procedure **A** as described in main text in 52% (0.126 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R_f = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.28 (m, 5H), 6.99-6.93 (m, 2H), 6.82 (dd, *J* = 13.3, 5.2 Hz, 1H), 6.09 (s, 1H), 5.15 (s, 2H), 3.86 (s, 3H), 2.14 (dd, *J* = 7.0, 0.9 Hz, 2H), 1.73-1.63 (m, 5H), 1.45-1.35 (m, 1H), 1.32 (s, 12H), 1.21-1.11 (m, 3H), 0.92-0.83 (m, 2H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 149.3, 148.5, 137.0, 128.7, 128.0, 127.4, 124.7, 121.0, 117.0, 114.8, 113.7, 91.6, 87.9, 83.7, 71.0, 56.1, 44.8, 38.4, 33.4, 26.7, 26.5, 25.0, the carbon α to boron was not found; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 33.0; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{31}H_{40}{}^{11}BO_4$ 487.3020; Found 487.3009.

[1] Because of its explosiveness and toxicity, diazomethane was directly generated in diethyl ether and used without further purification after simple decantation, see: Ernst Redemann, C.; Rice, F. O.; Roberts, R.; Ward H. P. *Org. Synth*. 1945, **25**, 28.

(E)-3-(5-Cyclohexyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pent-3-en-1-yn-1-yl)benzonitrile (3b). Prepared according to the general procedure **A** as described in main text in 55% (0.110 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (t, *J* = 1.3 Hz, 1H), 7.63-7.61 (m, 1H), 7.56-7.53 (m, 1H), 7.41 (t, *J* = 7.8 Hz, 1H), 6.09 (s, 1H), 2.17 (dd, *J* = 7.0, 0.8 Hz, 2H), 1.71-1.67 (m, 5H), 1.44-1.37 (m, 1H), 1.32 (s, 12H), 1.18-1.11 (m, 3H), 0.93-0.87 (m, 2H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 135.4, 134.8, 131.1, 129.3, 125.9, 120.0, 118.3, 112.8, 91.7, 88.9, 83.9, 44.9, 38.4, 33.4, 26.6, 26.5, 25.0, the carbon α to boron was not found; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 30.9; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{24}H_{31}{}^{11}BNO_2$ 376.2448; Found 376.2448.

(E)-2-(1-Cyclohexyl-5-(3,4,5-trimethoxyphenyl)pent-2-en-4-yn-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3c). Prepared according to the general procedure **A** as described in main text in 55% (0.121 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.68 (s, 2H), 6.08 (s, 1H), 3.85 (s, 3H), 3.83 (s, 6H), 2.15 (dd, *J* = 7.0, 0.9 Hz, 2H), 1.73-1.67 (m, 5H), 1.41-1.35 (m, 1H), 1.32 (s, 12H), 1.22-1.15 (m, 3H), 0.94-0.84 (m, 2H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.1, 138.7, 120.7, 119.2, 108.7, 91.4, 88.2, 83.7, 61.1, 56.2 (2C), 44.8, 38.4, 33.4, 26.6, 26.5, 25.0, the carbon α to boron was not found; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 31.1; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{26}H_{38}{}^{11}BO_5$ 441.2812; Found 441.2820.

(E)-4-(4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)oct-3-en-1-yn-1-yl) benzonitrile (3d). Prepared according to the general procedure **A** as described in main text in 58% (0.097 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H), 6.15 (s, 1H), 2.29-2.26 (m, 2H), 1.44-1.39 (m, 2H), 1.32 (s, 12H), 1.27-1.24 (m, 2H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 132.1, 131.9, 129.2, 119.1, 118.8, 111.1, 93.8, 89.8, 83.9, 36.7, 31.6, 25.0, 22.5, 14.1, the carbon α to boron was not found; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 30.3; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{21}H_{27}{}^{11}BNO_2$ 336.2135; Found

336.2129.

(E)-4,4,5,5-Tetramethyl-2-(1-(3,4,5-trimethoxyphenyl)oct-3-en-1-yn-4-yl)-1,3,2-dioxaborolane (3e). Prepared according to the general procedure **A** as described in main text in 54% (0.121 g) yield, (0.216 g) on a 1 mmol scale. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.68 (s, 2H), 6.13 (s, 1H), 3.85 (s, 3H), 3.83 (s, 6H), 2.27-2.24 (m, 2H), 1.41-1.37 (m, 2H), 1.32 (s, 12H), 1.27-1.24 ((m, 2H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.1, 138.7, 119.8, 119.2, 108.7, 91.6, 88.3, 83.7, 61.1, 56.2 (2C), 36.6, 31.8, 29.8, 25.1, 22.5, 14.1, the carbon α to boron was not found; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 30.4; HRMS (ESI) m/z: $[M+NH4]^+$ Calculated for $C_{23}H_{37}{}^{11}BNO_5$ 418.2765; Found 418.2753.

(E)-Trimethyl(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-4-(3,4,5-trimethoxyphenyl) but-1-en-3-yn-1-yl) silane (3f). Prepared according to the general procedure **A** as described in main text in 62% (0.129 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.67 (s, 2H), 6.51 (s, 1H), 3.85 (s, 3H), 3.82 (s, 6H), 1.33 (s, 12H), 0.14 (s, 9H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 154.3, 140.1, 130.7, 119.9, 110.0, 93.5, 90.4, 84.8, 62.3, 57.3 (2C), 26.3, 0.02; the carbon α to boron was not found; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 32.9; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{22}H_{34}{}^{11}BO_5Si$ 417.2269; Found 417.2273.

(E)-3-(4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-4-(trimethylsilyl)but-3-en-1-yn-1-yl)benzonitrile (3g). Prepared according to the general procedure **A** as described in main text in 56% (0.098 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67-7.56 (m, 3H), 7.42 (t, *J* = 7.8 Hz, 1H), 6.50 (s, 1H), 1.31 (s, 12H), 0.15 (s, 9H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 135.6, 134.9, 131.4, 129.4, 128.8, 125.4, 118.2, 113.0, 92.3, 89.4, 83.8, 25.1, -1.3, the carbon α to boron was not found; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 33.4; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{20}H_{27}{}^{11}BNO_2Si$ 352.1904; Found 352.1898.

(E)-2-(4-Ethyl-6-(3,4,5-trimethoxyphenyl)hex-3-en-5-yn-3-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3h). Prepared according to the general procedure **A** as described in main text in 55% (0.116 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.68 (s, 2H), 3.85 (s, 3H), 3.84 (s, 6H), 2.36-2.23 (m, 4H), 1.31 (s, 12H), 1.16 (t, *J* = 7.5 Hz, 3H), 1.00 (t, *J* = 7.6 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.1, 138.5, 133.7, 119.5, 108.8, 90.8, 90.3, 83.5, 61.1, 56.2 (2C), 25.9, 25.1, 24.1, 14.4, 13.5, the carbon α to boron was not found; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 33.9; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{23}H_{34}{}^{11}BO_5$ 401.2499; Found 401.2510.

(E)-3-(4-Cyclopropyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)but-3-en-1-yn-1-yl) pyridine (3i). Prepared according to the general procedure **A** as described in main text in 57% (0.84 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.64 (dd, *J* = 2.1, 0.8 Hz, 1H), 8.47 (dd, *J* = 4.9, 1.7 Hz, 1H), 7.67 (dt, *J* = 7.9, 1.9 Hz, 1H), 7.22 (ddd, *J* = 7.9, 4.9, 0.9 Hz, 1H), 6.09 (s, 1H), 1.65-1.61 (m, 1H), 1.30 (s, 12H), 0.82-0.77 (m, 2H), 0.71-0.66 (m, 2H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 152.1, 148.1, 138.2, 123.1, 121.4, 115.4, 92.6, 87.8, 84.0, 25.0, 17.8, 8.2, the carbon α to boron was not found; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 32.6; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{18}H_{23}{}^{11}BNO_2$ 296.1822; Found 296.1827.

(E)-3-(4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)non-3-en-1-yn-1-yl) pyridine (3j). Prepared according to the general procedure **A** as described in main text in 55% (0.090 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (d, *J* = 1.4 Hz, 1H), 8.49 (dd, *J* = 4.9, 1.6 Hz, 1H), 7.70 (dt, *J* = 7.9, 1.9 Hz, 1H), 7.23 (ddd, *J* = 7.9, 4.9, 0.8 Hz, 1H), 6.14 (s, 1H), 2.26 (td, *J* = 7.8, 1.3 Hz, 2H), 1.49-1.40 (m, 2H), 1.32 (s, 12H), 1.31-1.23 (m, 4H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 152.2, 148.2, 138.3, 123.1, 121.4, 119.2, 92.5, 88.0, 83.9, 37.0, 31.6, 29.2, 25.0, 22.6, 14.1; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 33.0, the carbon α to boron was not found; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{20}H_{29}{}^{11}BNO_2$ 326.2291; Found 326.2286.

(E)-4,4,5,5-Tetramethyl-2-(1-phenyl-6-(3,4,5-trimethoxyphenyl)hex-3-en-5-yn-3-yl)-1,3,2-dioxaborolane (3k). Prepared according to the general procedure **A** as described in main text in 59% (0.132 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.27 (m, 2H), 7.22-7.18 (m, 3H), 6.69 (s, 2H), 6.15 (s, 1H), 3.86 (s, 3H), 3.84 (s, 6H), 2.78-2.71 (m, 2H), 2.60-2.54 (m, 2H), 1.34 (s, 12H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.1, 141.9, 138.8, 128.6, 128.4, 126.0, 120.9, 119.1, 108.8, 92.2, 88.1, 83.8, 61.1, 56.2 (2C), 38.9, 36.1, 25.1, the carbon α to boron was not found; ^{11}B{^{1}H} NMR (128 MHz, $CDCl_3$) δ 30.2; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{27}H_{34}{}^{11}BO5$ 449.2499; Found 449.2500.

(Z)-5,5'-(3-(Cyclohexylmethyl)hexa-3-en-1,5-diyne-1,6-diyl) bis-(1,2,3-trimethoxybenzene) (4a). Prepared according to the general procedure **B** as described in main text in 63% (0.158 g) yield. It was purified by column chromatography (30% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.74 (s, 2H), 6.71 (s, 2H), 5.85 (s, 1H), 3.84 (s, 3H), 3.84 (s, 3H), 3.78 (s, 6H), 3.76 (s, 6H), 2.20 (d, *J* = 6.5 Hz, 2H), 1.81-1.65 (m, 6H), 1.30-1.18 (m, 3H), 1.02-0.88 (m, 2H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.2, 153.2, 139.3, 139.1, 135.1, 118.7, 118.4, 115.7, 109.8, 109.2, 108.9, 97.1, 95.0, 88.5, 87.6, 61.1, 56.3, 56.2, 44.8, 37.1, 33.1, 26.6, 26.3; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{31}H_{37}O_6$ 505.2590; Found 505.2561.

(Z)-5,5'-(3-Butylhexa-3-en-1,5-diyne-1,6-diyl)bis-(1,2,3-trimethoxybenzene) (4b). Prepared according to the general procedure **B** as described in main text in 64% (0.148 g) yield. It was purified by column chromatography (30% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.74 (s, 2H), 6.71 (s, 2H), 5.90 (t, *J* = 1.1 Hz, 1H), 3.85 (s, 3H), 3.84 (s, 3H), 3.77 (s, 6H), 3.76 (s, 6H), 2.35-2.31 (m, 2H), 1.65-1.60 (m, 2H), 1.43-1.37 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.2, 139.3, 139.1, 136.3, 118.7, 118.4, 116.7, 114.7, 109.8, 109.1, 108.9, 97.2, 95.0, 88.2, 87.5, 61.1, 56.3, 56.2, 36.6, 30.8, 22.2, 14.0; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{28}H_{33}O_6$ 465.2277; Found 465.2269.

(Z)-3,3'-(3-(Trimethylsilyl)hexa-3-en-1,5-diyne-1,6-diyl)dibenzonitrile (4c). Prepared according

to the general procedure **B** as described in main text in 60% (0.105 g) yield. It was purified by column chromatography (10% EtOAc/Hexanes; R*f* = 0.6) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (dd, *J* = 3.1, 1.5 Hz, 2H), 7.68 (ddd, *J* = 7.8, 2.7, 1.3 Hz, 2H), 7.63 -7.58 (m, 2H), 7.51-7.44 (m, 2H), 6.29 (s, 1H), 0.28 (s, 9H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 139.5, 136.6, 135.7, 135.5, 135.0, 134.9, 132.7, 131.9, 131.6, 129.6, 125.3, 124.8, 118.1, 118.0, 113.2, 113.1, 99.7, 94.8, 91.9, 90.6, -1.9; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{23}H_{19}N_2Si$ 351.1390; Found 351.1381.

(Z)-1,2,3-Trimethoxy-5-(3-(4-methoxybenzylidene)hept-1-yn-1-yl) benzene (4d). Prepared according to the general procedure **B** as described in main text in 66 % (0.125 g) yield. It was purified by column chromatography (20% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 1.5 Hz, 1H), 7.28-7.27 (m, 2H), 6.85-6.81 (m, 1H), 6.74 (s, 2H), 6.59 (s, 1H), 3.88 (s, 9H), 3.80 (s, 3H), 2.40 (td, *J* = 7.5, 1.0 Hz, 2H), 1.76-1.63 (m, 2H), 1.43 (hex, *J* = 7.3 Hz, 2H), 0.97 (t, *J* = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.6, 153.3, 138.4, 134.3, 129.2, 122.9, 121.7, 118.7, 113.5, 113.4, 108.8, 96.2, 89.1, 61.1, 56.2 (2C), 55.3, 39.0, 31.1, 22.2, 14.1; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{24}H_{29}O_4$ 381.2066; Found 381.2058.

(Z)-3-(4-((3,4,5-Trimethoxyphenyl)ethynyl)non-3-en-1-yn-1-yl)pyridine (4e). Prepared according to the general procedure **B** as described in main text in 62 % (0.121 g) yield. It was purified by column chromatography (40% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (s, 1H), 8.50 (s, 1H), 7.74 (dt, *J* = 7.9, 1.8 Hz, 1H), 7.23 (dd, *J* = 7.8, 4.9 Hz, 1H), 6.72 (s, 2H), 5.91 (s, 1H), 3.86 (s, 3H), 3.82 (s, 6H), 2.38-2.32 (m, 2H), 1.67-1.60 (m, 2H), 1.39-1.33 (m, 4H), 0.92 (t, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.3, 152.2, 148.6, 139.4, 138.3, 137.6, 123.2, 121.0, 118.1, 113.9, 109.0, 97.9, 91.6, 91.1, 87.8, 61.1, 56.2, 37.1, 31.2, 28.3, 22.6, 14.2; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{25}H_{28}NO_3$ 390.2069; Found 390.2068.

(Z)-4-(3-(3-(Pyridin-3-yl)prop-2-yn-1-ylidene)oct-1-yn-1-yl)benzonitrile (4f). Prepared according to the general procedure **B** as described in main text in 58 % (0.093 g) yield. It was

purified by column chromatography (30% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.70 (s, 1H), 8.53 (d, *J* = 4.0 Hz, 1H), 7.72 (dt, *J* = 7.9, 1.8 Hz, 1H), 7.64-7.60 (m, 2H), 7.58-7.54 (m, 2H), 7.29-7.24 (m, 1H), 5.99 (t, *J* = 1.2 Hz, 1H), 2.35 (td, *J* = 7.0, 1.3 Hz, 2H), 1.68-1.59 (m, 2H), 1.38-1.32 (m, 4H), 0.91 (t, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 152.2, 148.8, 138.3, 136.3, 132.3, 132.2, 128.0, 123.3, 120.6, 118.5, 115.7, 112.0, 95.4, 92.6, 92.0, 90.9, 36.9, 31.2, 28.2, 22.5, 14.1; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{23}H_{21}N_2$ 325.1705; Found 325.1699.

(Z)-3-(4-((2-(Trifluoromethyl)phenyl)ethynyl)non-3-en-1-yn-1-yl) pyridine (4g). Prepared according to the general procedure **B** as described in main text in 60 % (0.110 g) yield. It was purified by column chromatography (30% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (d, *J* = 1.4 Hz, 1H), 8.51 (dd, *J* = 4.9, 1.6 Hz, 1H), 7.75 (dt, *J* = 7.9, 1.9 Hz, 1H), 7.67 (d, *J* = 7.8 Hz, 1H), 7.63 (d, *J* = 7.7 Hz, 1H), 7.51 (t, *J* = 7.3 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 7.25 (ddd, *J* = 7.9, 4.9, 0.7 Hz, 1H), 5.97 (t, *J* = 1.1 Hz, 1H), 2.38 (td, *J* = 7.7, 1.1 Hz, 2H), 1.72-1.61 (m, 2H), 1.38-1.32 (m, 4H), 0.91 (t, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 152.3, 148.6, 138.4, 136.7, 134.3, 131.6, 131.5 (q, *J* = 27.6 Hz), 128.5, 126.1 (q, *J* = 5.0 Hz), 123.6 (q, *J* = 273.4 Hz), 123.2, 121.3, 120.8, 114.9, 93.7, 93.2, 91.5, 90.9, 37.3, 31.2, 28.0, 22.6, 14.1; ^{19}F{^{1}H} NMR (377 MHz, $CDCl_3$) δ -62.0; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{23}H_{21}{}^{19}F_3N$ 368.1626; Found 368.1619.

(Z)-3-(4-Cyclopropyl-6-(3,4,5-trimethoxyphenyl)hexa-3-en-1,5-diyn-1-yl)pyridine (4h). Prepared according to the general procedure **B** as described in main text in 62 % (0.111 g) yield. It was purified by column chromatography (40% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (brs, 1H), 8.50 (brs, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 6.68 (s, 3H), 6.03 (s, 1H), 3.86 (s, 3H), 3.81 (s, 6H), 1.78-1.68 (m, 1H), 0.98-0.84 (m, 4H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.3 (2C), 140.2, 139.5, 138.2, 117.8, 111.6, 109.0 (2C), 108.6, 97.9, 91.8, 91.1, 84.2, 61.1, 56.3 (2C), 17.0, 7.6; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{23}H_{22}NO_3$ 360.1600; Found 360.1597.

(Z)-2,2'-(3-Pentylhexa-3-en-1,5-diyne-1,6-diyl)bis-((trifluoromethyl)benzene) (4i). Prepared

according to the general procedure **C** as described in main text in 65 % (0.141 g) yield. It was purified by column chromatography (5% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72-7.62 (m, 4 H), 7.54-7.46 (m, 2H), 7.44-7.34 (m, 2H), 6.01 (s, 1H), 2.37 (td, *J* = 7.7, 1.2 Hz, 2H), 1.70-1.62 (m, 2H), 1.40-1.32 (m, 4H), 0.92 (t, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 136.5, 134.2, 134.1, 131.6, 131.5 (q, *J* = 30.6 Hz), 131.3 (q, *J* = 30.6 Hz), 131.0, 128.2, 127.9, 126.1 (q, *J* = 4.8 Hz), 123.7 (q, *J* = 273.5 Hz), 121.8, 121.4, 114.8, 93.6, 93.0 (2C), 90.7, 37.4, 31.2, 27.9, 22.46, 14.0; ^{19}F{^{1}H} NMR (377 MHz, $CDCl_3$) δ -66.9, -66.9; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{25}H_{21}{}^{19}F_6$ 435.1547; Found 435.1551.

(Z)-4,4'-(3-Propylhexa-3-en-1,5-diyne-1,6-diyl)dibenzonitrile (4j). Prepared according to the general procedure **C** as described in main text in 59% (0.101 g) yield. It was purified by column chromatography (10% EtOAc/Hexanes; R*f* = 0.6) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68-7.58 (m, 4H), 7.58-7.51 (m, 4H), 6.00 (s, 1H), 2.37 (td, *J* = 7.6, 1.1 Hz, 2H), 1.71-1.64 (hex, *J* = 7.4 Hz, 2H), 0.99 (t, *J* = 7.4 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 137.0, 132.3, 132.3, 132.2, 132.0, 128.3, 128.0, 118.5, 118.5, 115.6, 112.2, 111.8, 95.7, 93.7, 92.5, 92.0, 39.1, 21.8, 13.6; HRMS (ESI) m/z: $[M+Na]^+$ Calculated for $C_{23}H_{16}N_2Na$ 343.1211; Found 343.1216.

(Z)-5,5'-(3-Benzylhexa-3-en-1,5-diyne-1,6-diyl)bis-(1,2,3-trimethoxybenzene (4k). Prepared according to the general procedure **C** as described in main text in 62% (0.154 g) yield. It was purified by column chromatography (30% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.25 (m, 5H), 6.70 (s, 2H), 6.65 (s, 2H), 5.89 (t, *J* = 1.2 Hz, 1H), 3.84 (s, 3H), 3.83 (s, 3H), 3.76 (s, 12H), 3.66 (s, 2H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.1 (2C), 139.2, 139.0, 137.9, 134.6, 129.1, 128.5, 126.7, 118.4, 118.1, 115.5, 109.0, 108.8, 97.7, 95.7, 88.1, 87.2, 60.9 (2C), 56.1 (2C), 56.1 (2C), 42.8; HRMS (ESI) m/z: $[M+Na]^+$ Calculated for $C_{31}H_{30}NaO_6$ 521.1940; Found 521.1949.

(Z)-4-(4-Methoxyphenyl)-1-(1-(3,4,5-trimethoxyphenyl)oct-3-en-1-yn-4-yl)-1H-1,2,3-triazole (5). To a stirred solution of boronic ester **3e** (200 mg, 0.5 mmol) in MeOH (2 mL), sodium azide

(78 mg, 1.2 mmol) and copper sulfate (80 mg, 0.5 mmol) were added at room temperature and stirred for 6 h. To this reaction mixture, sodium ascorbate (59 mg, 0.3 mmol) and 4-methoxyphenylacetylene (66 µL, 0.5 mmol) were added, and the reaction mixture was stirred for 12 h. The solvent was evaporated under reduced pressure, the residue diluted with water and extracted with CH_2Cl_2 (2 x 15 mL). The combined organic extracts were dried over $MgSO_4$, filtered and evaporated under reduced pressure. The residue was purified by column chromatography (30% EtOAc/Hexanes; R*f* = 0.5) to give 68% (0.150 g) yield of compound **5** as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (s, 1H), 7.82 (dt, *J* = 9.6, 2.8 Hz, 2H), 6.95 (dt, *J* = 9.6, 2.7 Hz, 2H), 6.56 (s, 2H), 5.68 (s, 1H), 3.84 (s, 3H), 3.84 (s, 3H), 3.72 (s, 6H), 2.95 (t, *J* = 7.5 Hz, 2H), 1.61-1.53 (m, 2H), 1.45-1.35 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 159.9, 153.4, 146.5, 146.0, 139.5, 127.2, 123.1, 119.4, 117.6, 114.5, 108.6, 98.9, 96.6, 84.1, 61.1, 56.1 (2C), 55.5, 34.4, 29.9, 22.3, 13.9; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{26}H_{30}N_3O_4$ 448.2236; Found 448.2227.

(Z)-4-(4-Methoxyphenyl)-1-(2-((3,4,5-trimethoxyphenyl)ethynyl)hex-1-en-1-yl)-1H-1,2,3-triazole (7). To a stirred solution of boronic ester **1b** (168 mg, 0.5 mmol) in MeOH (2 mL), tetramethylsilylazide (79 µL, 0.6 mmol) and copper sulfate (80 mg, 0.5 mmol) were added at room temperature and stirred for 12 h. To this reaction mixture, sodium ascorbate (59 mg, 0.3 mmol) and 4-methoxyphenylacetylene (66 µL, 0.5 mmol) were added, and the reaction mixture was stirred for another 12 h. The solvent was evaporated under reduced pressure, diluted with water and extracted with CH_2Cl_2 (2 x 15 mL). The combined organic extracts were dried over $MgSO_4$, filtered and evaporated under reduced pressure. The residue was purified by column chromatography (30% EtOAc/Hexanes; R*f* = 0.5) to give 72% (138 mg) yield of compound[2] **6** as a colorless oil. This compound **6**, was then engaged in a Suzuki coupling reaction with 5-(bromoethynyl)-1,2,3-trimethoxybenzene (135 mg, 0.5 mmol) following the general procedure **A** as described in main text to give 65% (0.150 g) yield of compound **7**. It was purified by column chromatography (30% EtOAc/Hexanes; R*f* = 0.5) to afford a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.95 (s, 1H), 7.81-7.76 (m, 2H), 7.52 (s, 1H), 6.97-6.92 (m, 2H), 6.70 (s, 2H), 3.89 (s, 3H), 3.84 (s, 3H), 3.83 (s, 6H), 2.43 (t, *J* = 7.1 Hz, 2H), 1.76-1.67 (m, 2H), 1.51-1.43 (m, 2H),

[2] Mali, M.; Jayaram, V.; Sharma, G. V. M.; Ghosh, S.; Berrée, F.; Dorcet, V.; Carboni, B. *J. Org. Chem.,* 2020, **85**, 15104–15115.

0.99 (t, *J* = 7.3 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 159.9, 153.5, 147.0, 139.8, 127.2, 127.1, 123.1, 117.3, 117.1, 114.5, 114.4, 108.8, 99.3, 85.4, 61.2, 56.3, 55.5, 35.3, 30.7, 22.1, 14.0; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{26}H_{30}N_3O_4$ 448.2236; Found 448.2224.

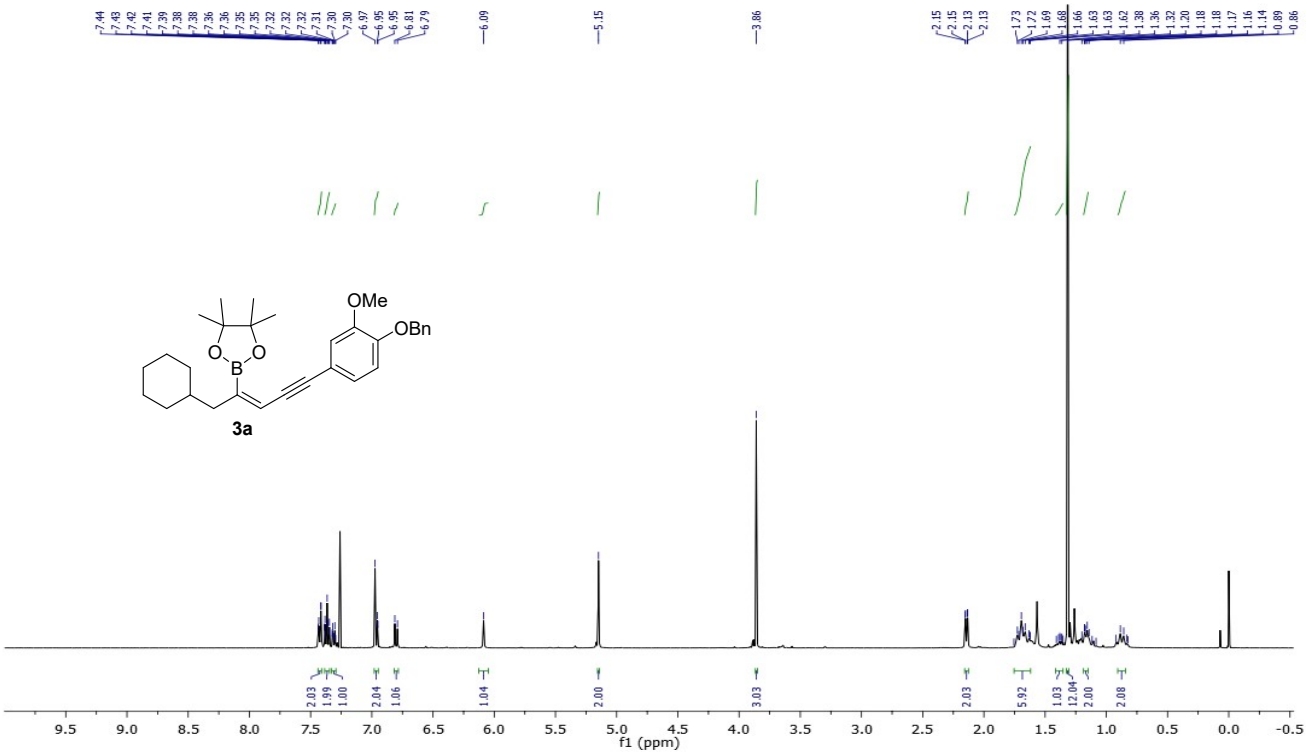
Methyl (Z)-2-morpholino-3-(3-(3,4,5-trimethoxyphenyl) prop-2-yn-1-ylidene) heptanoate (8). Glyoxylic acid monohydrate (51 mg, 0.5 mmol) and morpholine (44 µL, 0.5 mmol) were added to a stirred solution of boronic ester **3e** (200 mg, 0.5 mmol) in 1,1,1,3,3,3-hexafluoropropan-2-ol (1 mL) under argon atmosphere at room temperature. The reaction mixture was stirred for 72 h. The solvent was removed under reduced pressure to give a residue which was directly used for further esterification reaction. To a solution of the crude acid in diethyl ether (5 mL) at 0 °C, a solution of CH_2N_2 in diethylether[1] was added until the persistence of yellow color. After 2 hours at room temperature, the solvent was evaporated and residue was purified by column chromatography (40% EtOAc/Hexanes; R*f* = 0.5) to give 56% (0.120 g) yield of compound **8** as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.67 (s, 2H), 5.81 (s, 1H), 4.34 (s, 1H), 3.85 (s, 6H), 3.84 (s, 3H), 3.80-3.71 (m, 4H), 3.71 (s, 3H), 2.67-2.55 (m, 2H), 2.53-2.37 (m, 2H), 2.28-2.15 (m, 2H), 1.51-1.39 (m, 2H), 1.39-1.29 (m, 2H), 0.90 (t, *J* = 7.2 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ 170.7, 153.2, 148.4, 139.1, 118.5, 111.1, 108.7, 95.4, 85.2, 71.8, 66.9 (2C), 61.1, 56.3 (2C), 52.2, 51.7 (2C), 31.2, 30.0, 22.6, 14.1; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{24}H_{34}NO_6$ 432.2386; Found 432.2377.

2-(1-Butyl-2-((3,4,5-trimethoxyphenyl)ethynyl)cyclopropyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (10). To freshly distilled dichloromethane (2 mL) was added Et_2Zn (1 mL, 1 M in Hexane, 1 mmol) at 0 °C under argon. To this solution was added very slowly trifluoroacetic acid (114 mg, 76 µL, 1 mmol). Once the addition was complete (5 min), the reaction mixture was stirred for 30 min at 0 °C. Then, diiodomethane (267 mg, 80 µL, 1 mmol) was added in 5 min, and the resulting reaction mixture was stirred for an additional 20 min at 0 °C. To this solution was added the bis-boronate **1b** (168 mg, 0.5 mmol) in CH_2Cl_2 (1 mL). After 18 h at rt, the reaction was terminated by addition of saturated aqueous NH_4Cl (0.5 mL) and diluted with EtOAc (10 mL). Extraction of the aqueous phase was carried out with EtOAc (3 × 10 mL). The combined organic layers were dried on magnesium sulfate and filtered. Concentration in vacuo

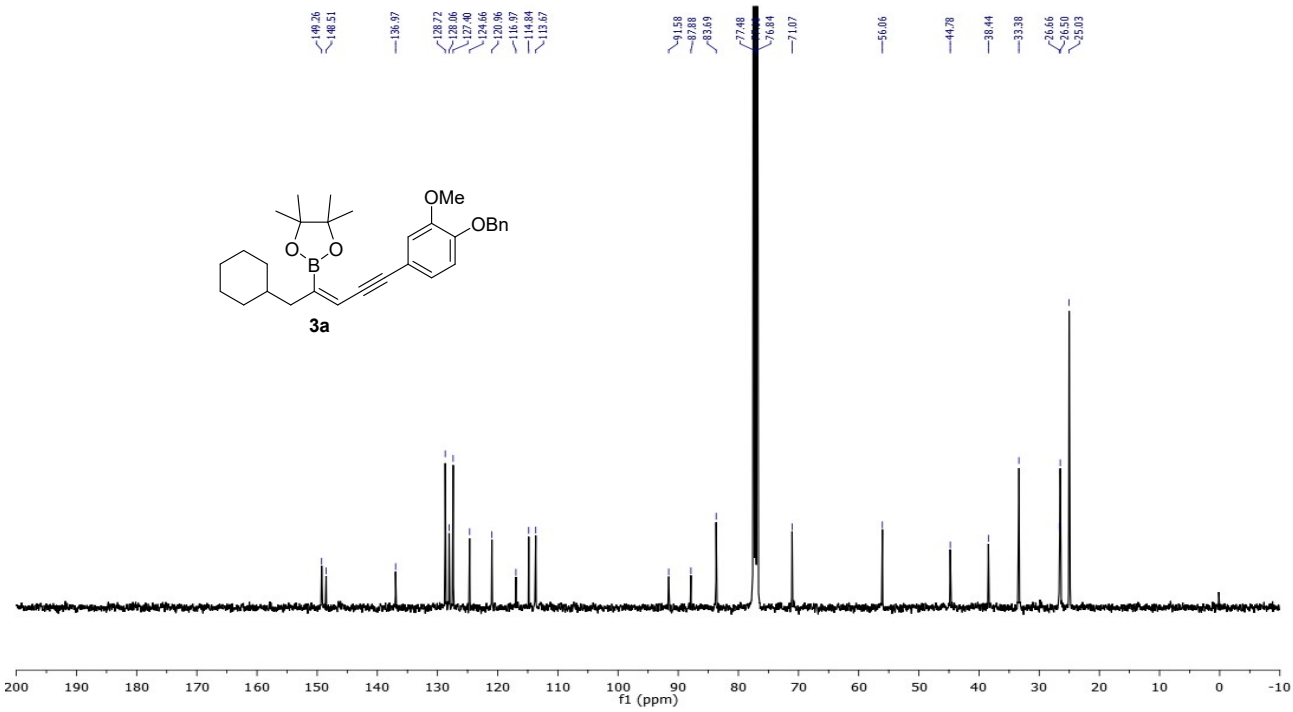
gave oil that was purified by column chromatography on silica gel to give the corresponding cyclopropane derivative **9**[3] (67 %). This compound was then engaged in a Suzuki reaction with 5-(bromoethynyl)-1,2,3-trimethoxybenzene following the general procedure **A** as described in main text. Purification by flash chromatography (20% EtOAc/Hexanes; R*f* = 0.5) afforded 114 mg (55 %) of compound **10** as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.59 (s, 2H), 3.82 (s, 3H), 3.80 (s, 6H), 1.97-1.80 (m, 1H), 1.44-1.34 (m, 2H), 1.33-1.26 (m, 2H), 1.25 (s, 6H), 1.22 (s, 6H), 1.22-1.16 (m, 2H), 0.88 (t, *J* = 7.1 Hz, 3H), 0.84-0.76 (m, 1H), 0.79-0.70 (m, 1H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.0 (2C), 138.1, 119.5, 108.9, 91.1, 83.6, 61.1, 56.2 (2C), 36.9, 31.3, 25.5, 24.8, 23.0, 20.2, 14.3, 13.3, the carbon α to boron was not found; HRMS (ESI) m/z: $[M+H]^+$ Calculated for $C_{24}H_{36}{}^{11}BO_5$ 415.2656; Found 415.2663.

[3] Mali, M.; Sharma, G. V. M.; Ghosh, S.; Roisnel, T.; Carboni, B.; Berrée, F. Simmons-Smith Cyclopropanation of Alkenyl 1,2-Bis-(Boronates): Stereoselective Access to Functionalized Cyclopropyl Derivatives. *J. Org. Chem*., 2022, **87**, 7649–7657.

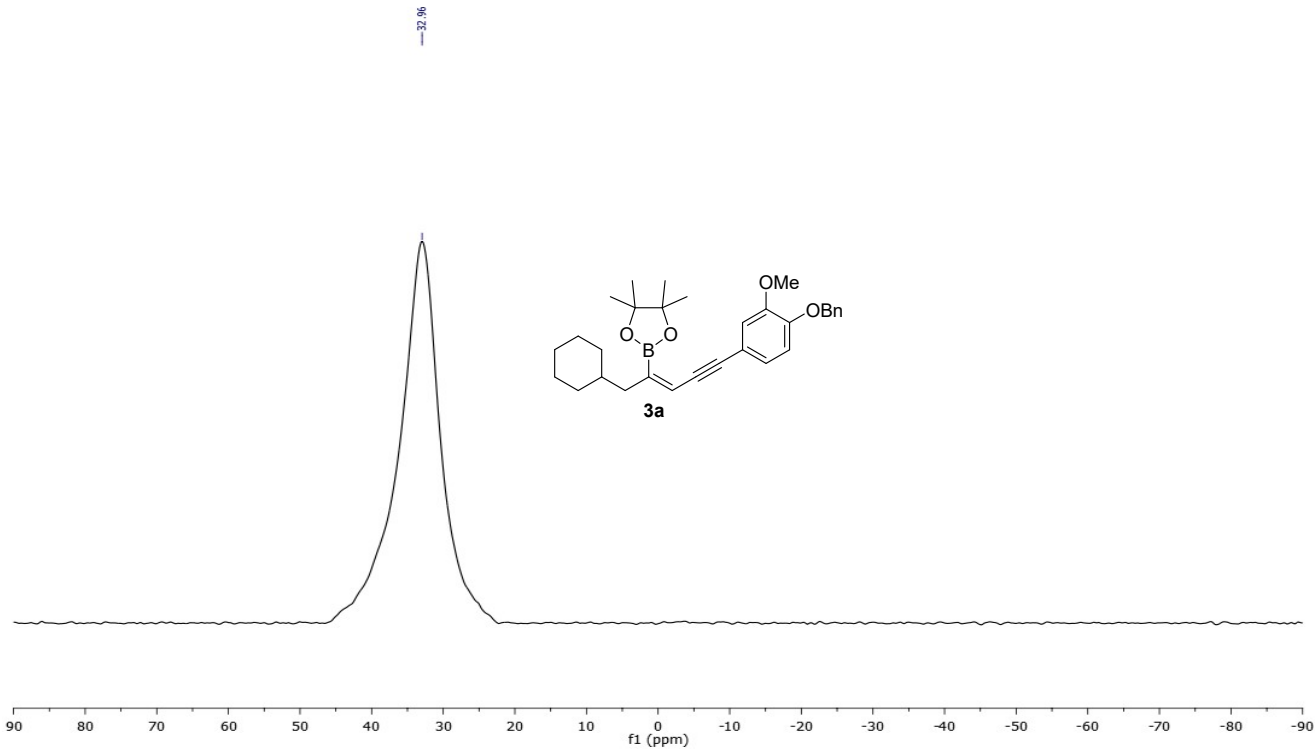
III. NMR spectra of new compounds



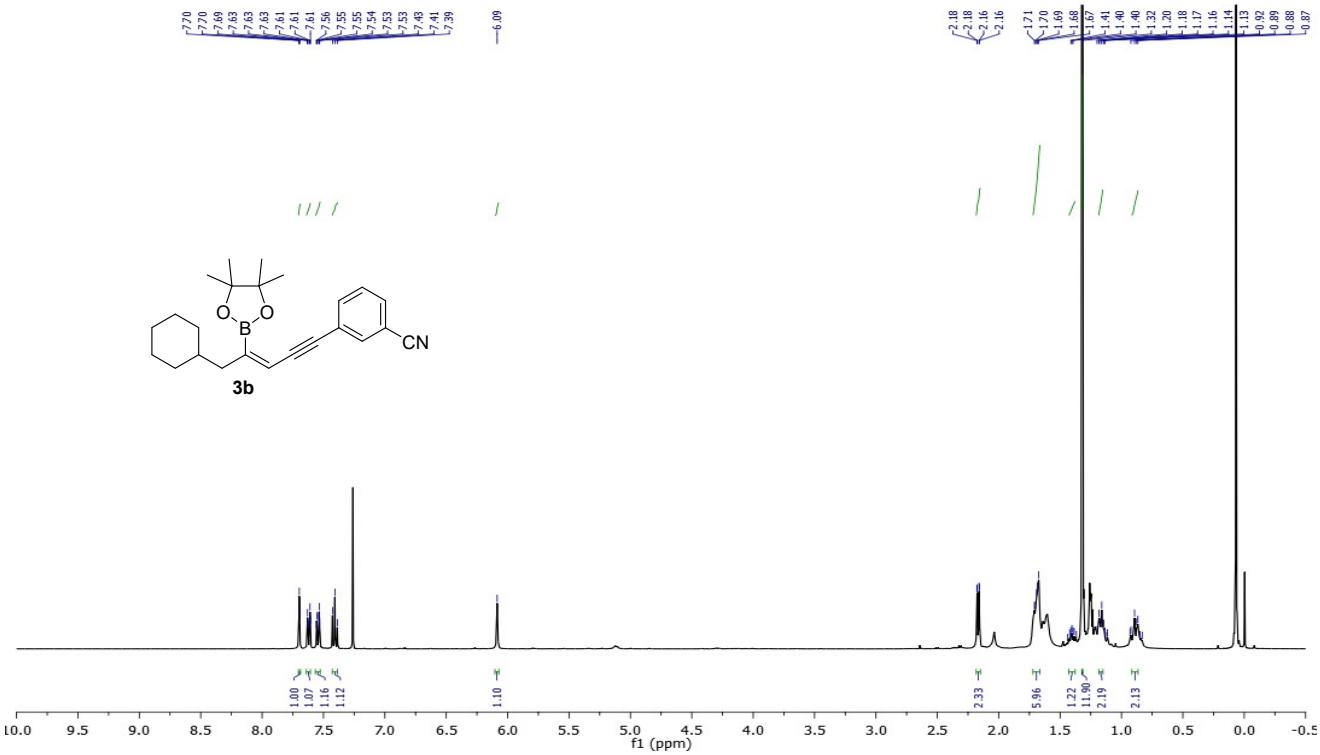
^{1}H NMR Spectrum of compound 3a (400 MHz, $CDCl_3$)



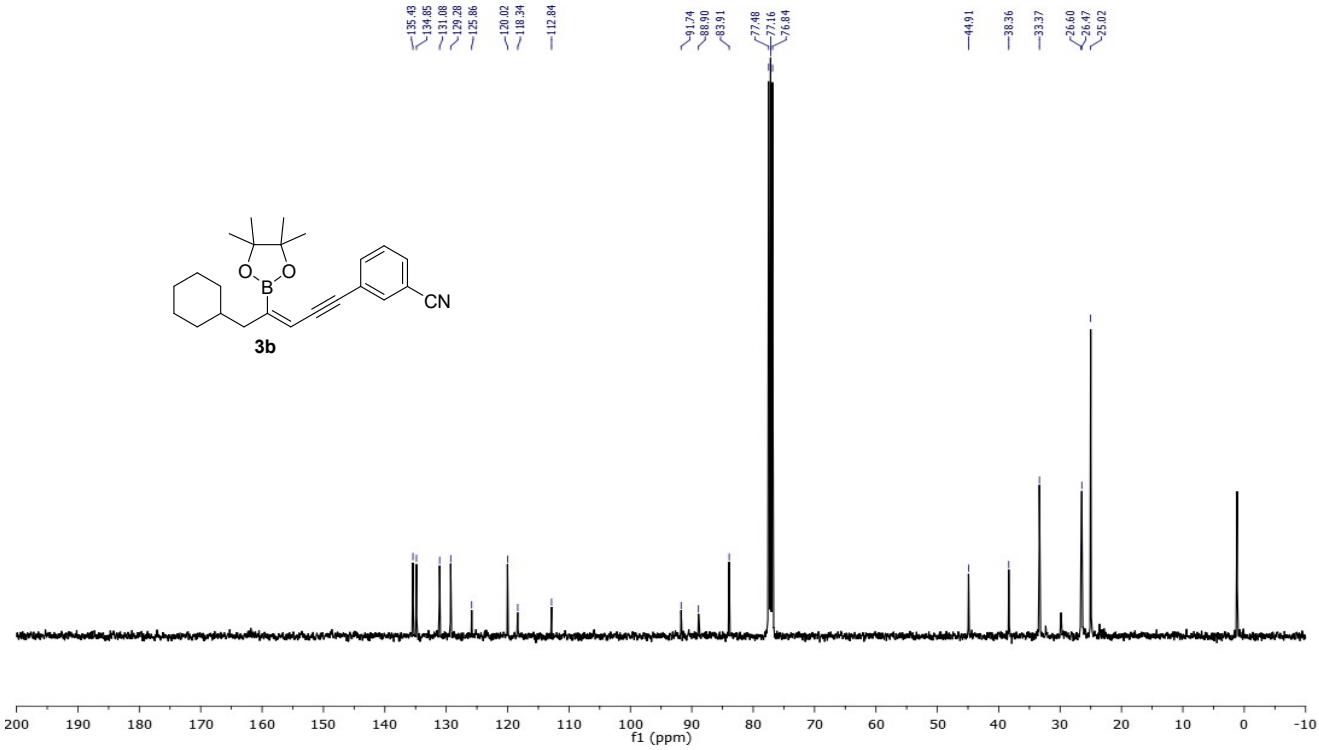
^{13}C{^{1}H} NMR Spectrum of compound 3a (101 MHz, $CDCl_3$)



$^{11}B\{^{1}H\}$ NMR Spectrum of compound 3a (128 MHz, $CDCl_3$)



^{1}H NMR Spectrum of compound 3b (400 MHz, $CDCl_3$)



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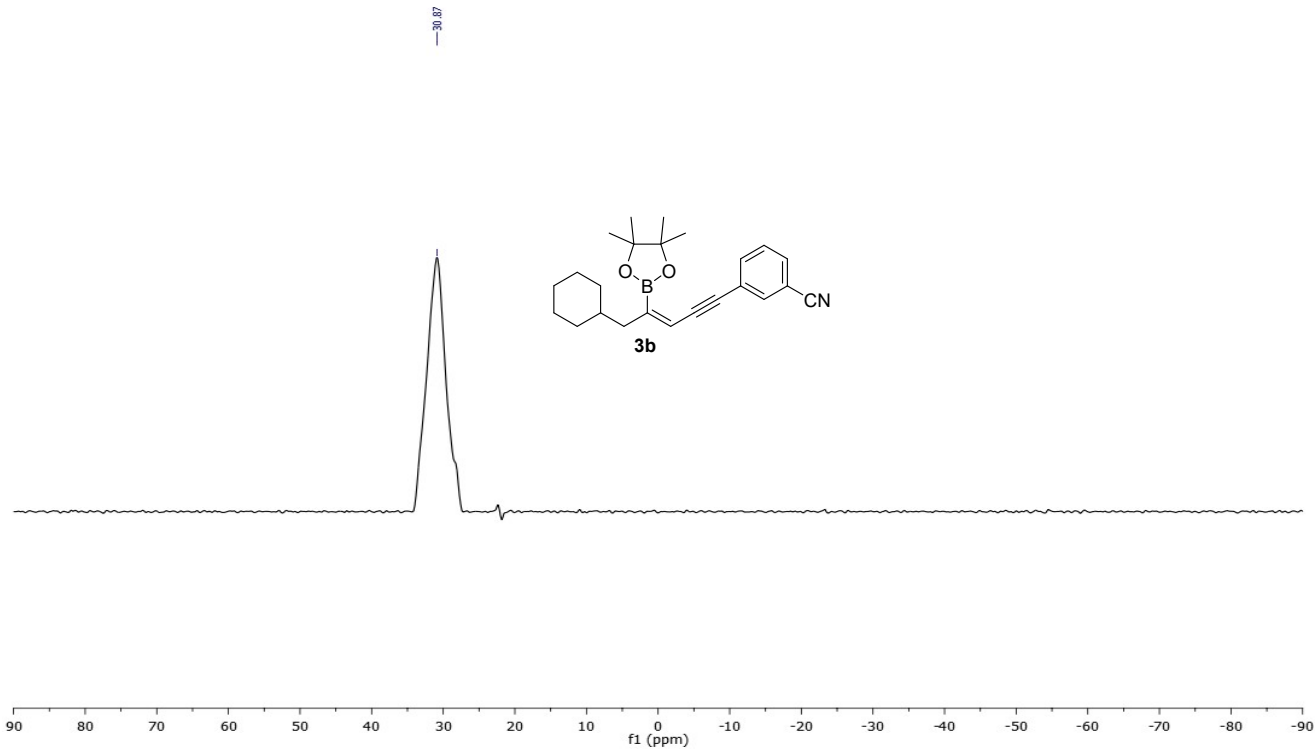

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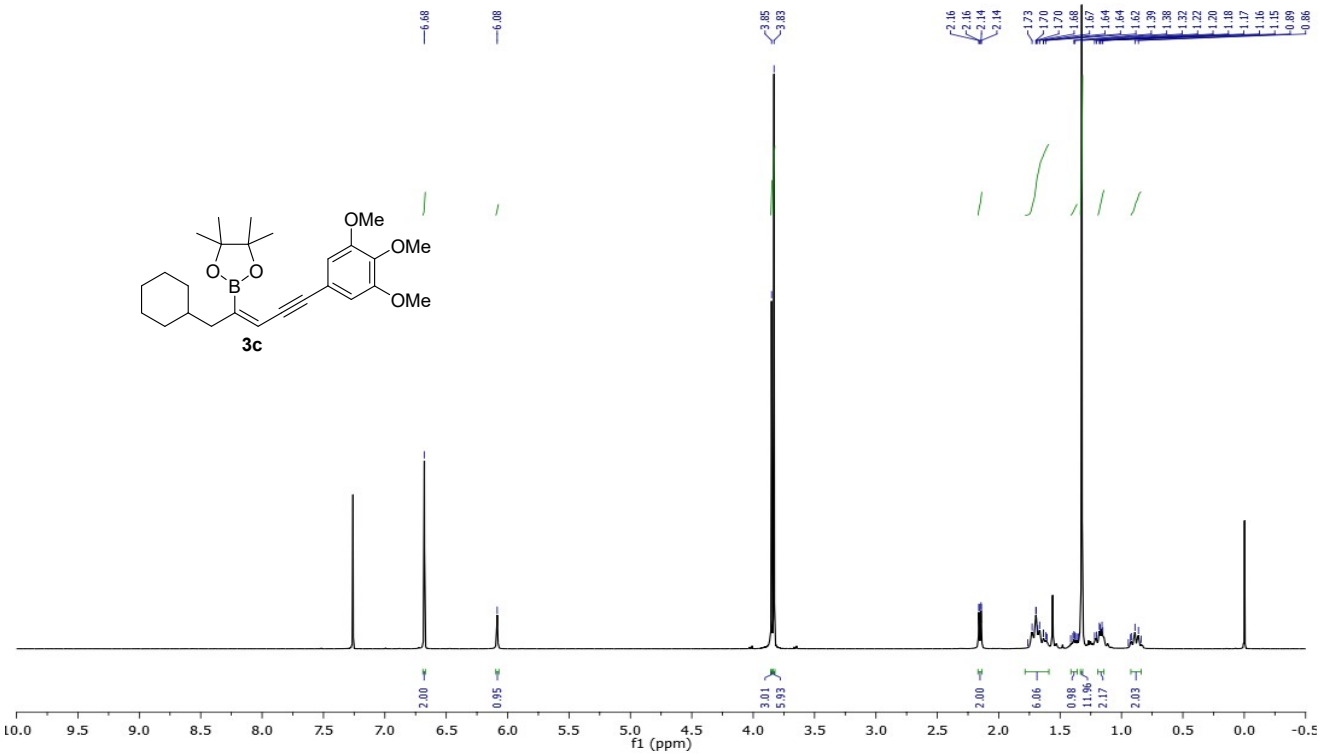
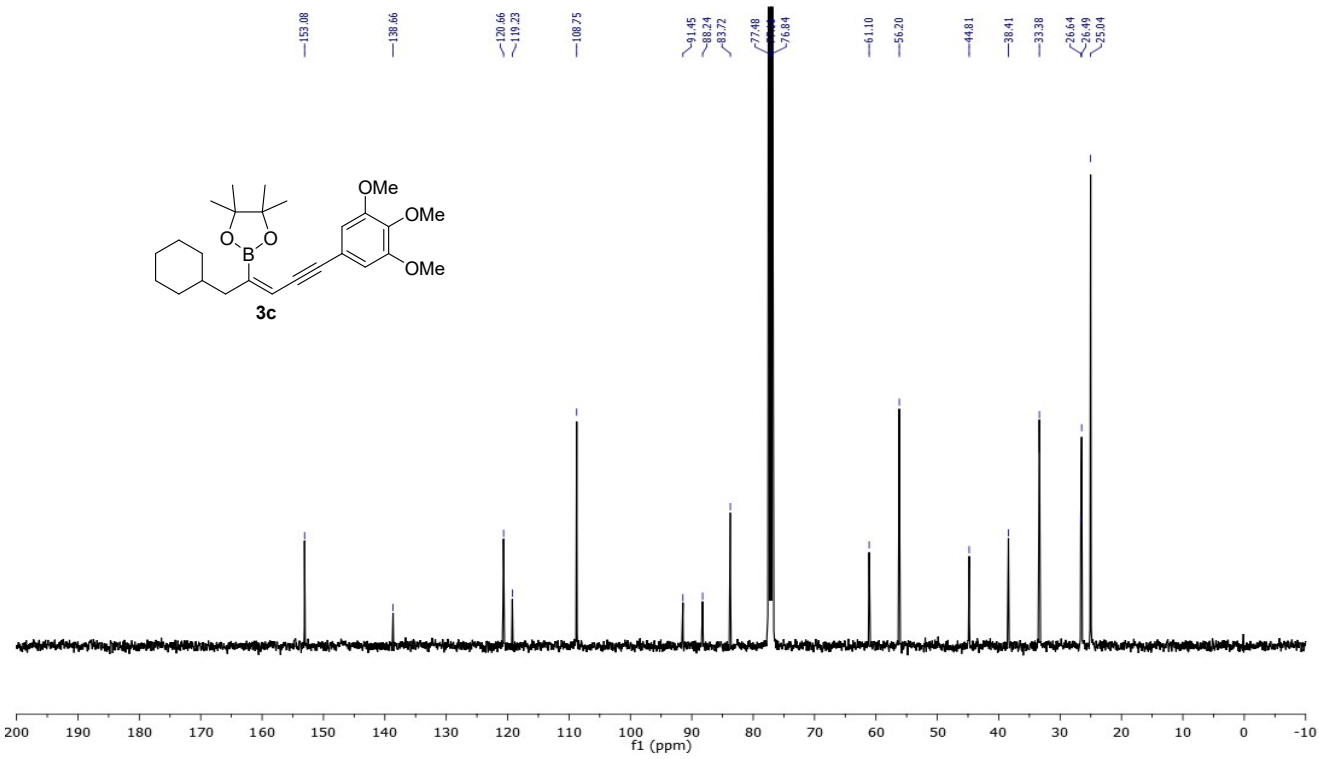


^{1}H NMR Spectrum of compound 3c (400 MHz, $CDCl_3$)



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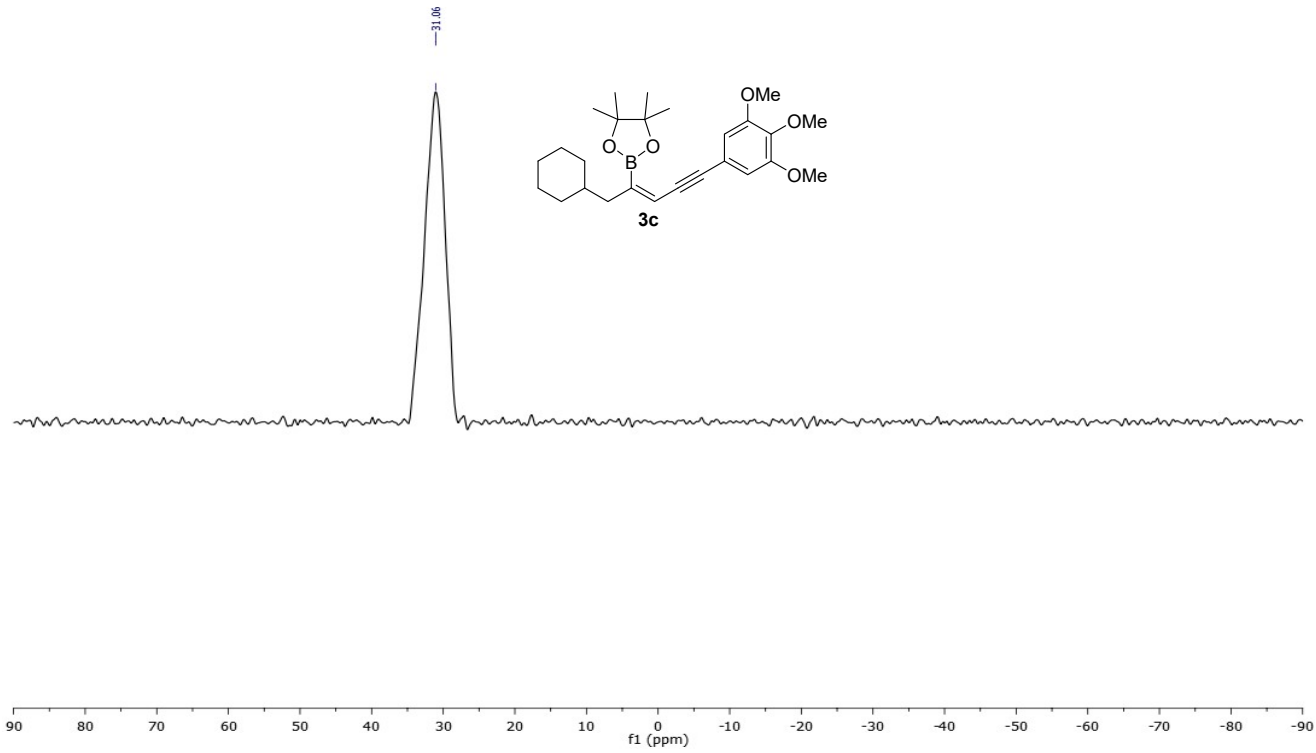

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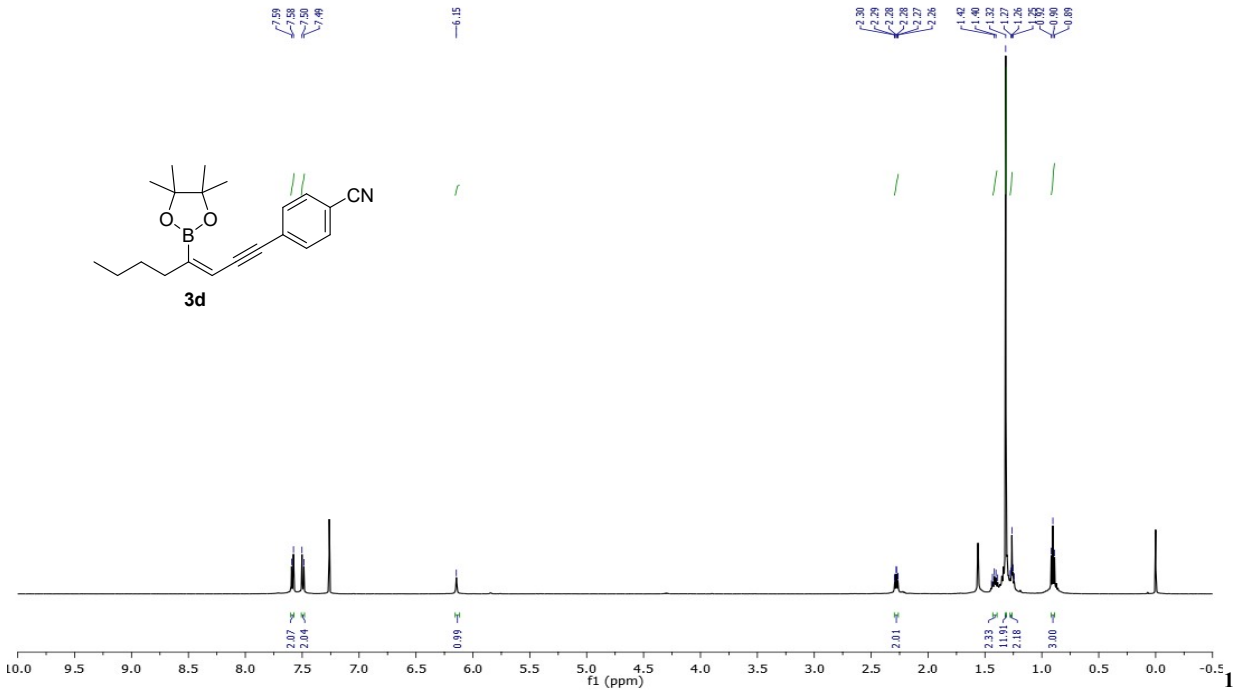
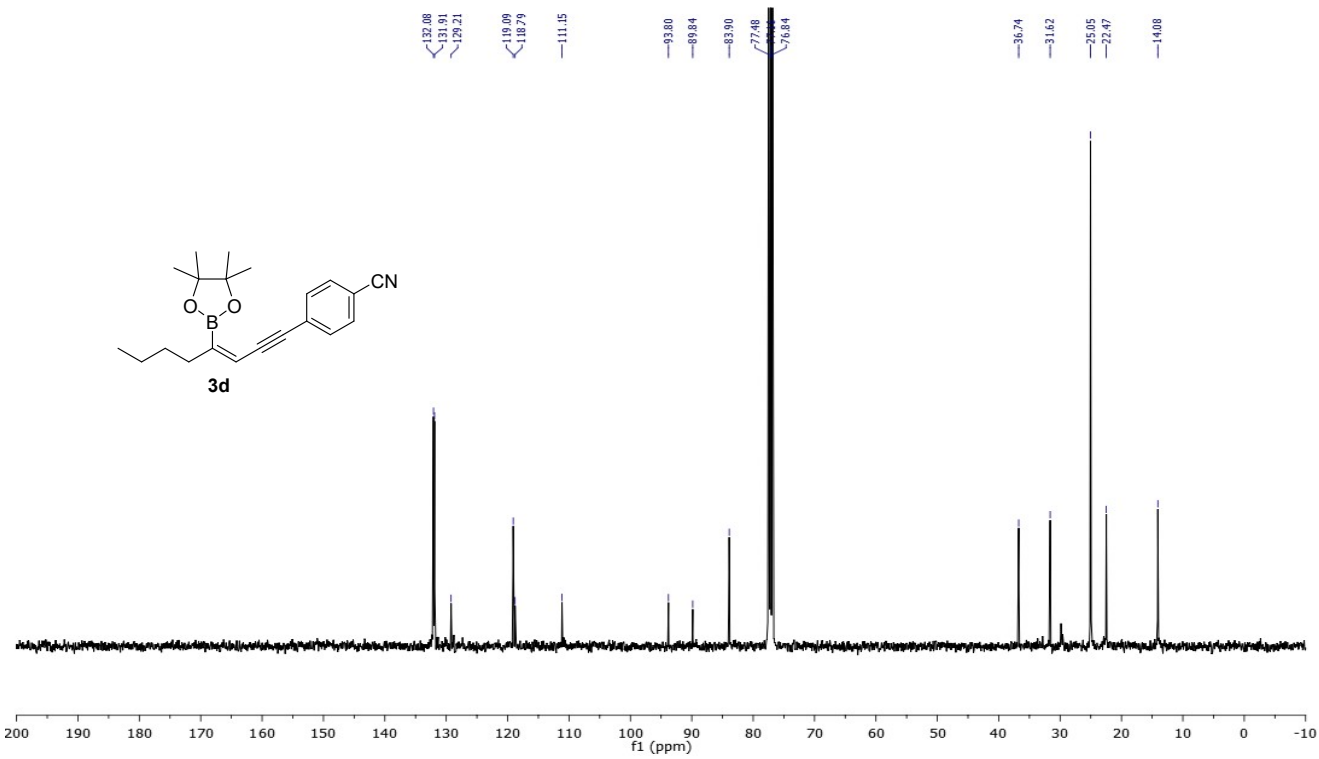


^{1}H NMR Spectrum of compound 3d (500 MHz, $CDCl_3$)



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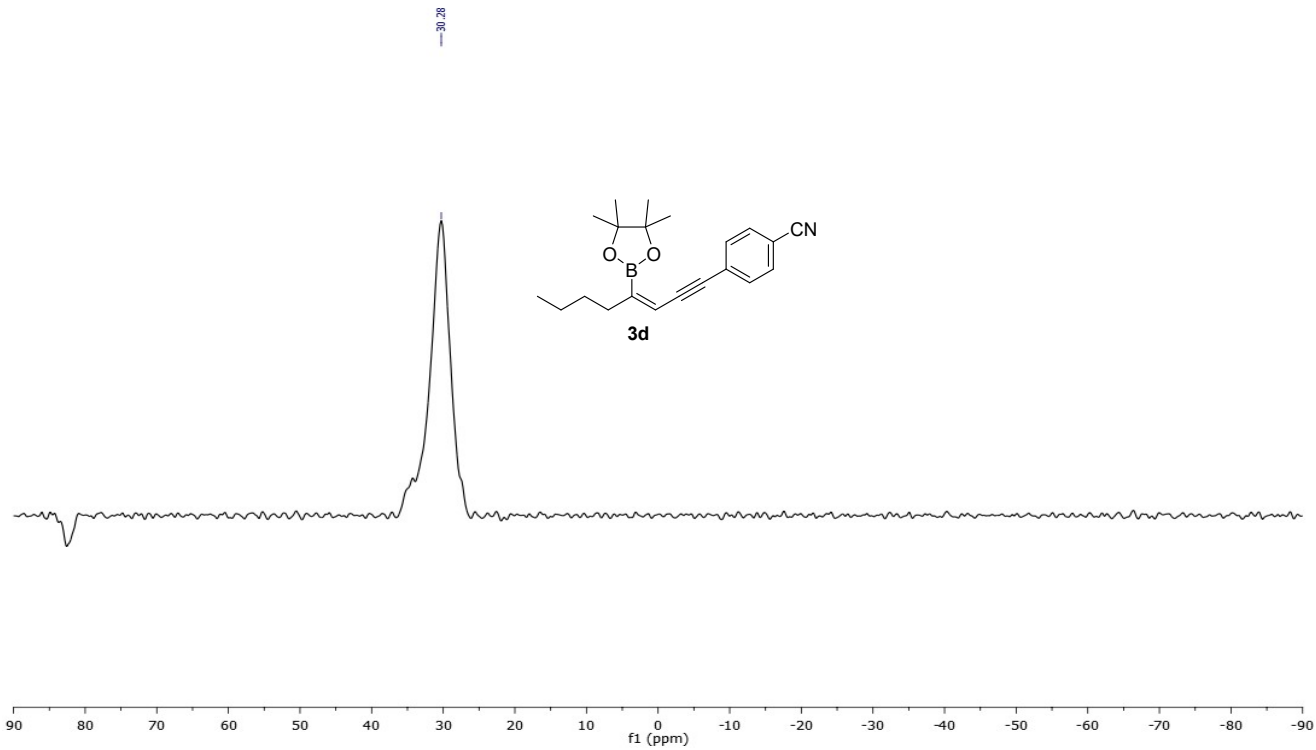

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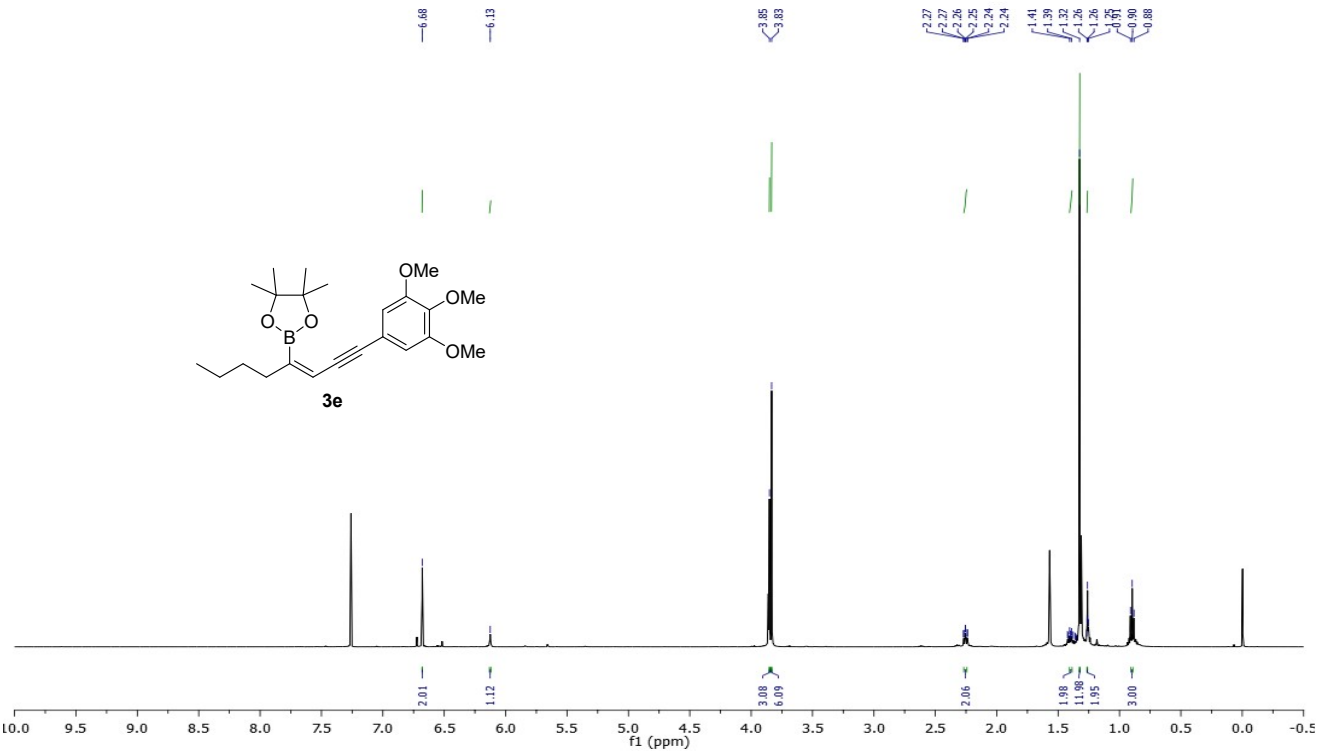

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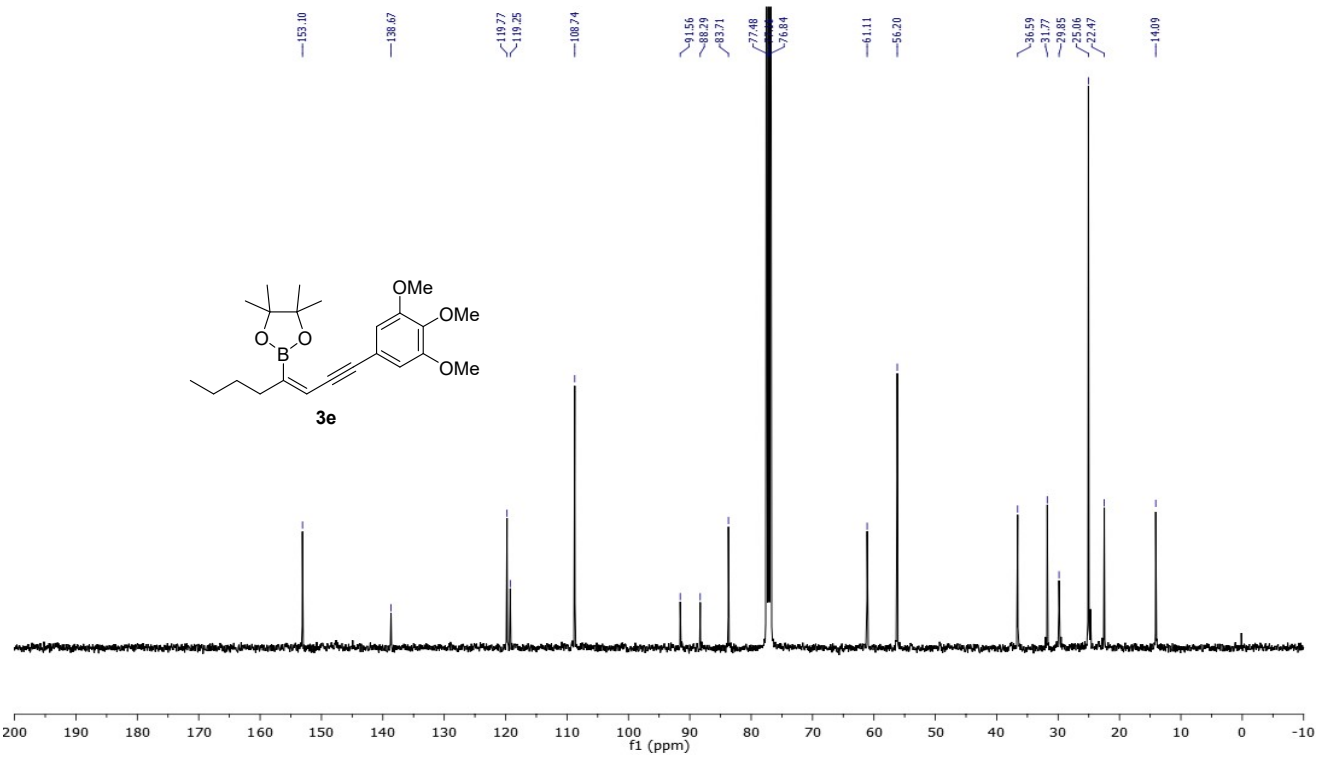

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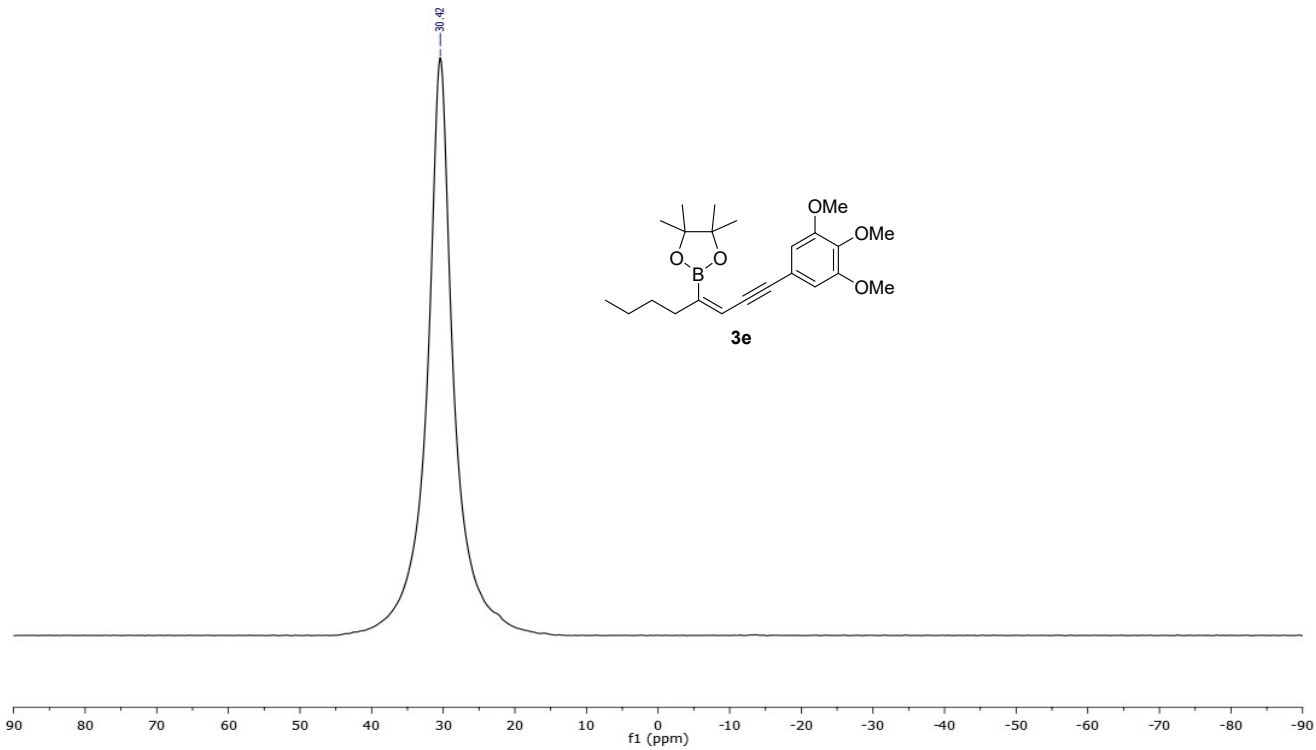
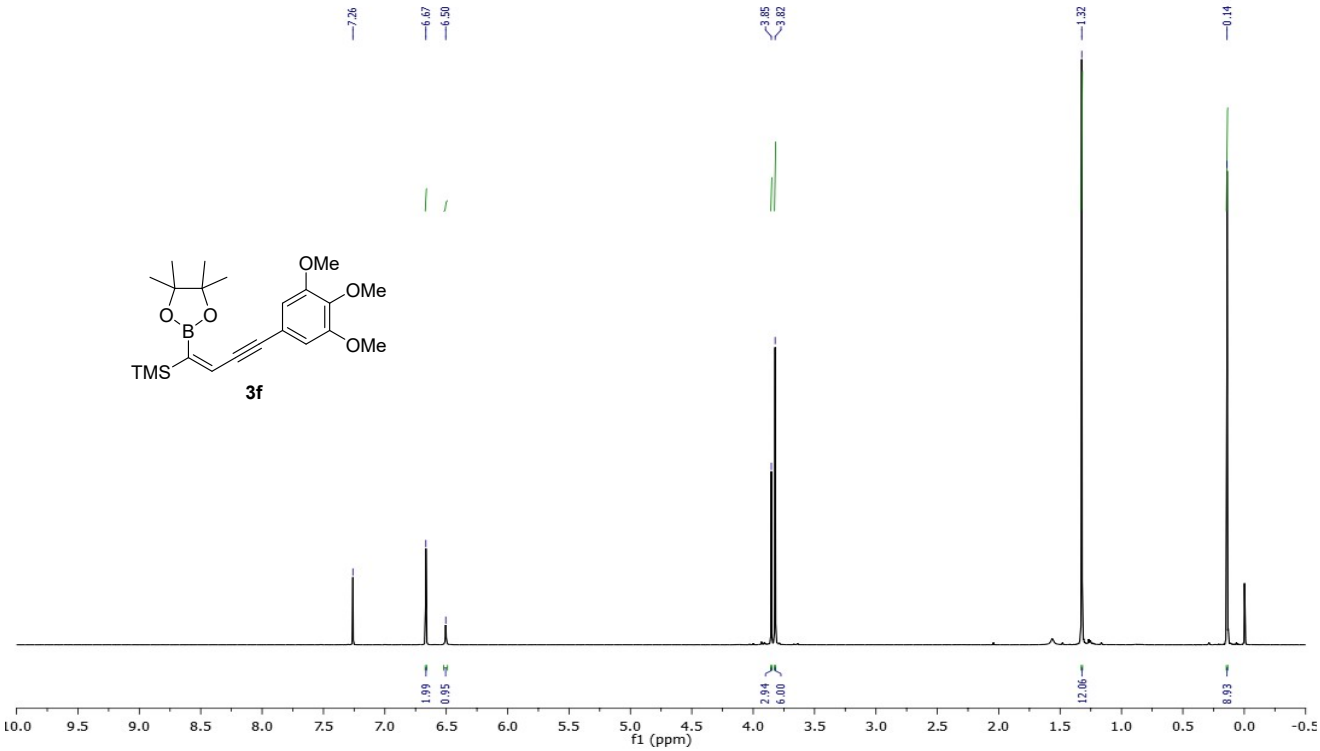


$^{11}B\{^{1}H\}$ NMR Spectrum of compound 3e (128 MHz, $CDCl_3$)



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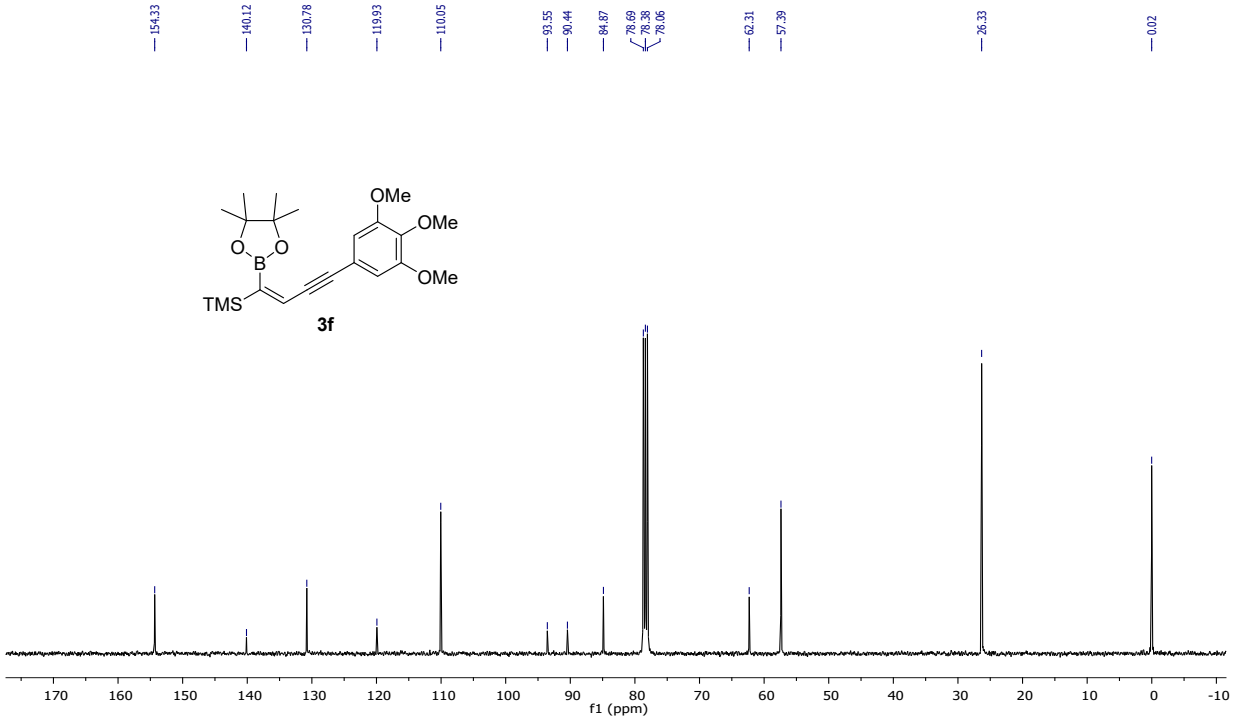
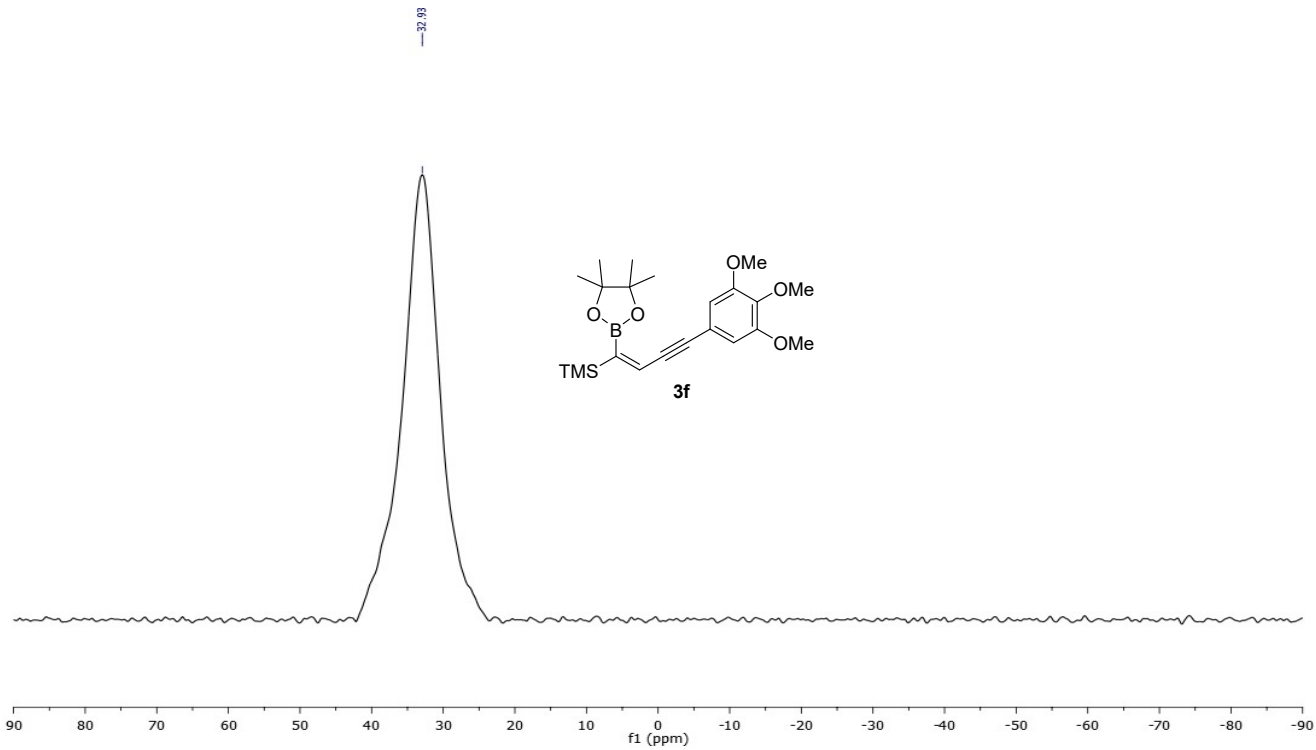
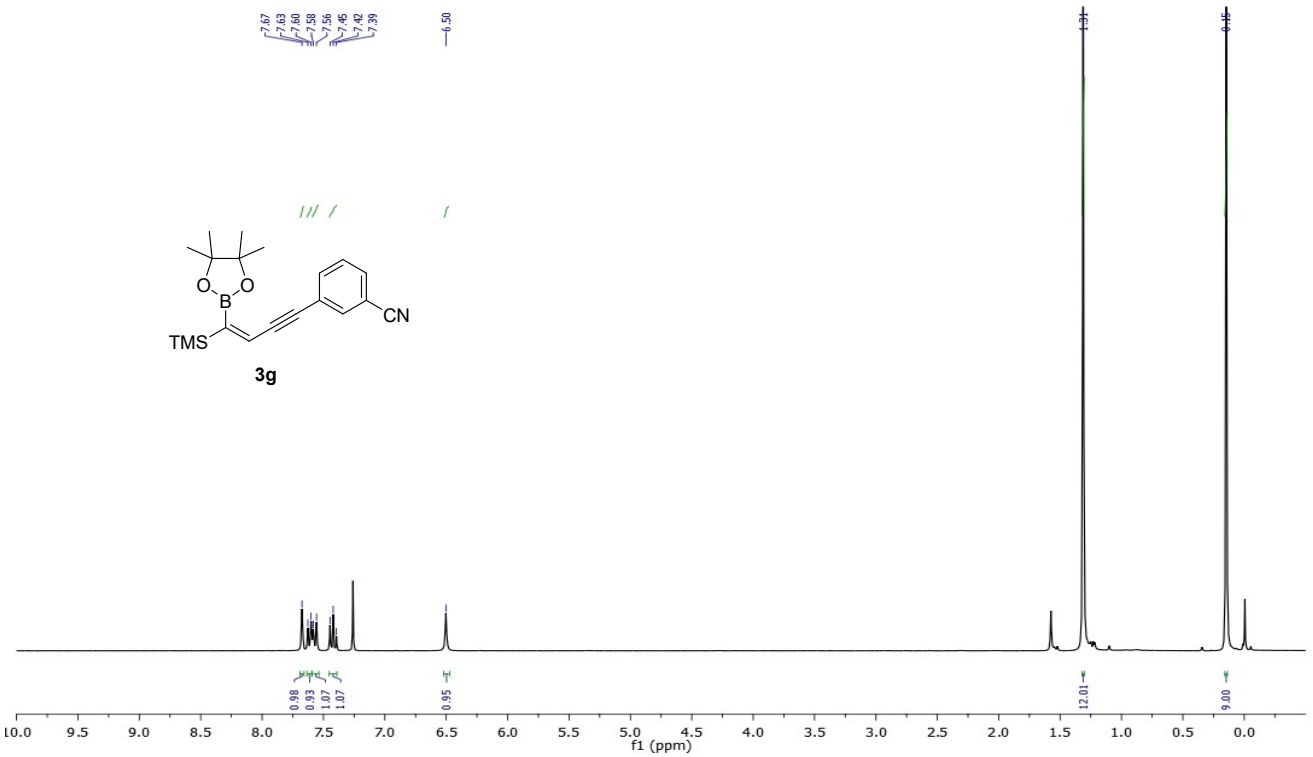


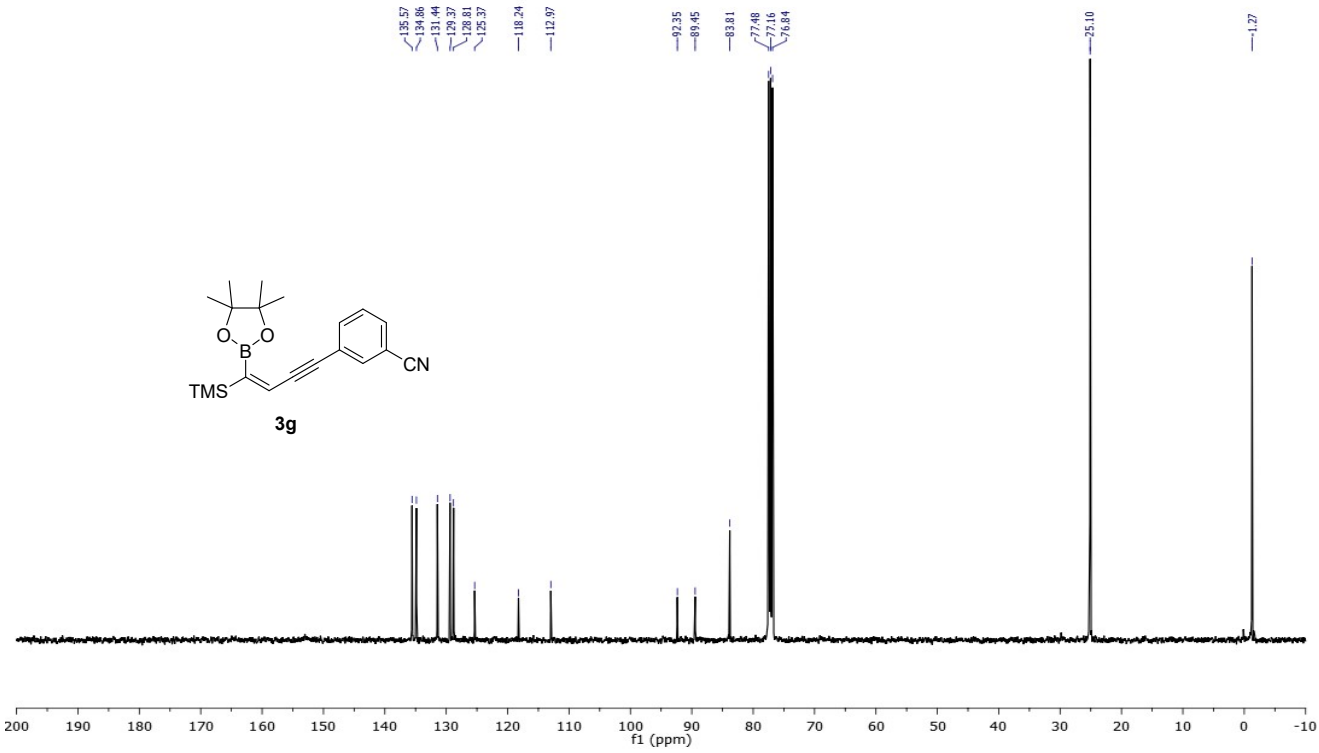
^{13}C{^{1}H} NMR Spectrum of compound 3f (101 MHz, $CDCl_3$)



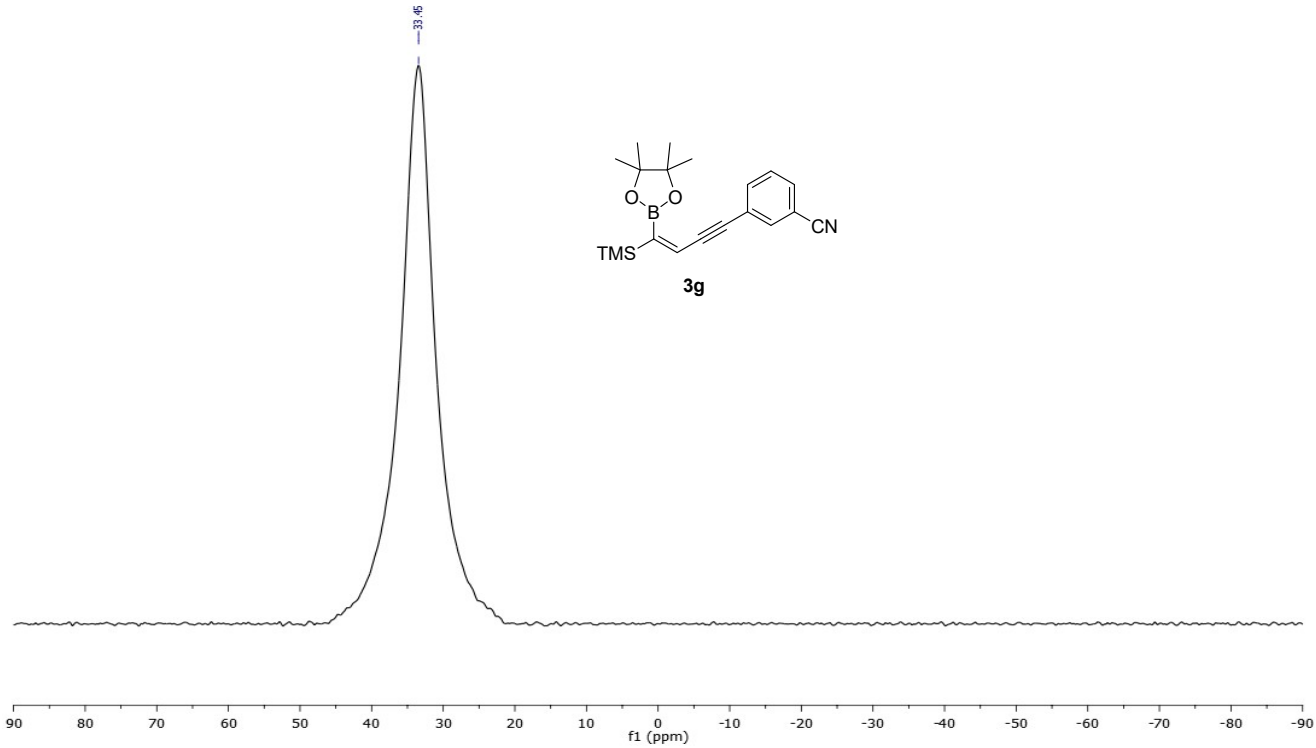
$^{11}B\{^{1}H\}$ NMR Spectrum of compound 3f (128 MHz, $CDCl_3$)



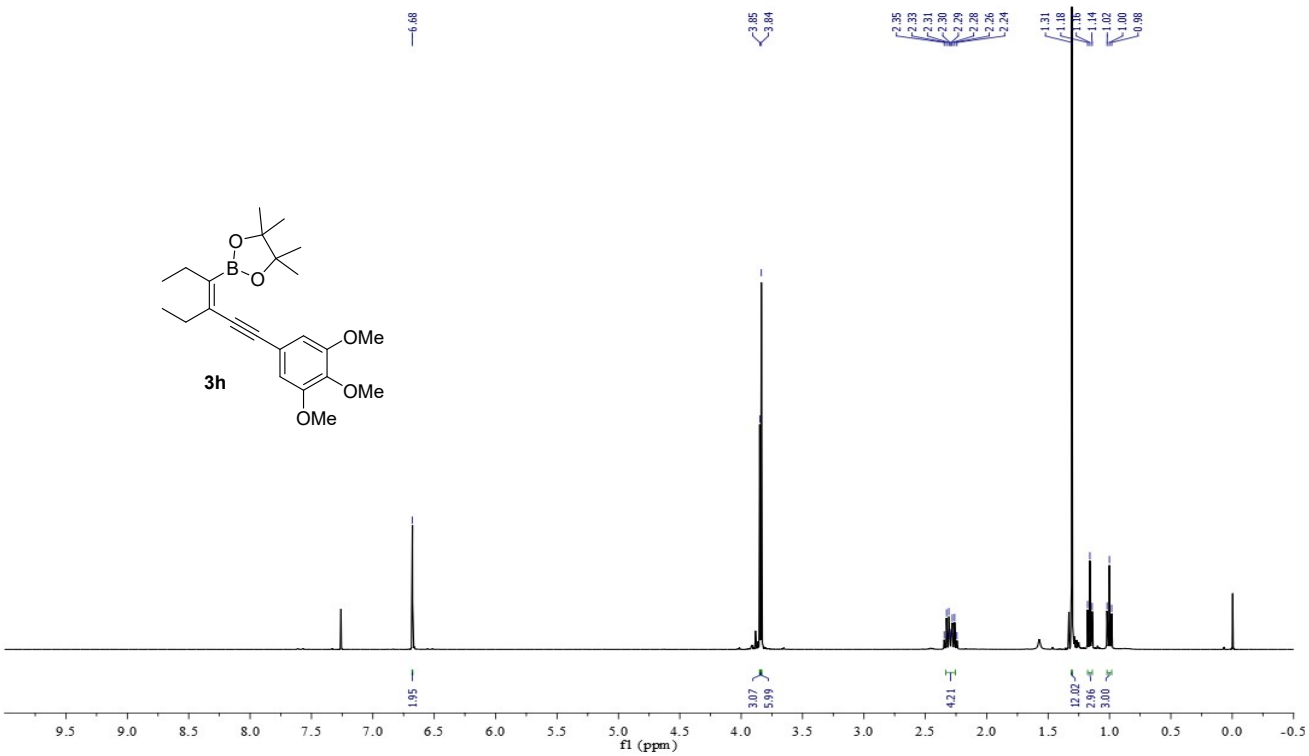
^{1}H NMR Spectrum of compound 3g (300 MHz, $CDCl_3$)



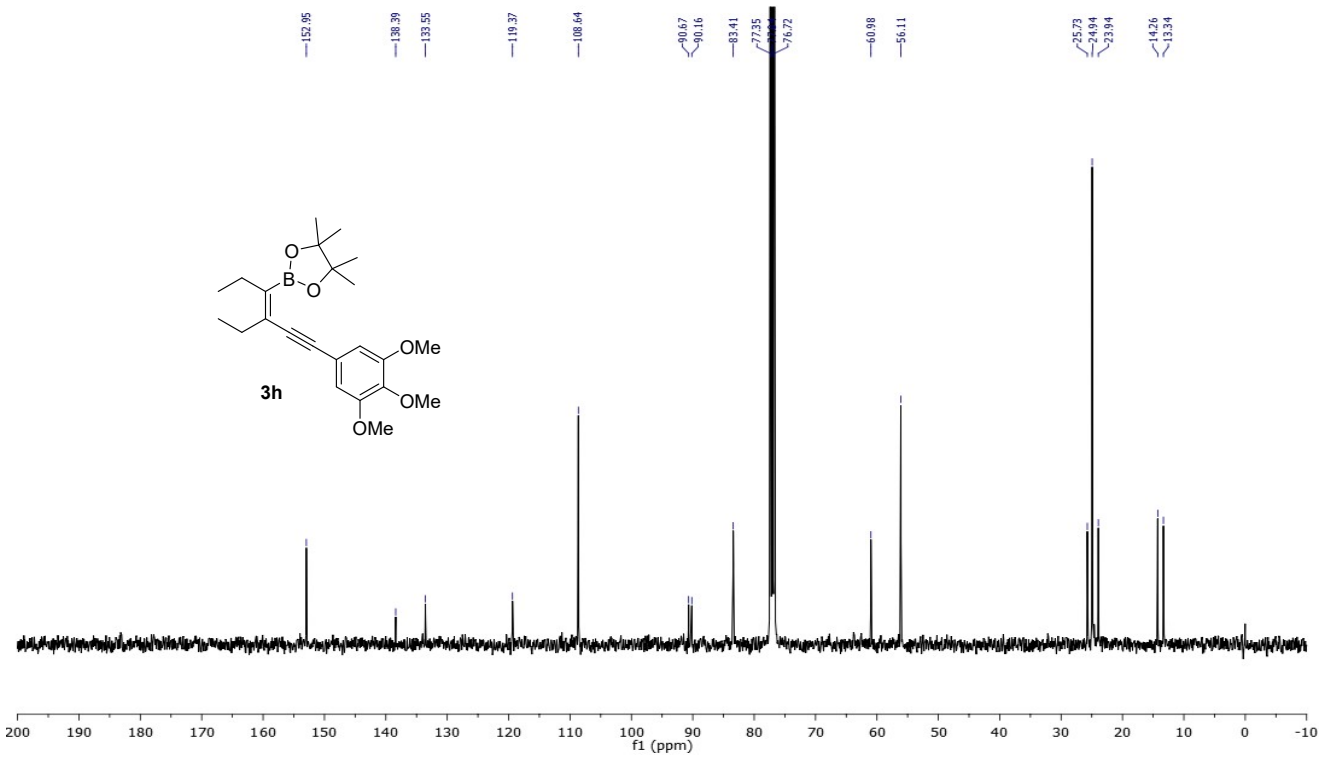
^{13}C{^{1}H} NMR Spectrum of compound 3g (101 MHz, $CDCl_3$)



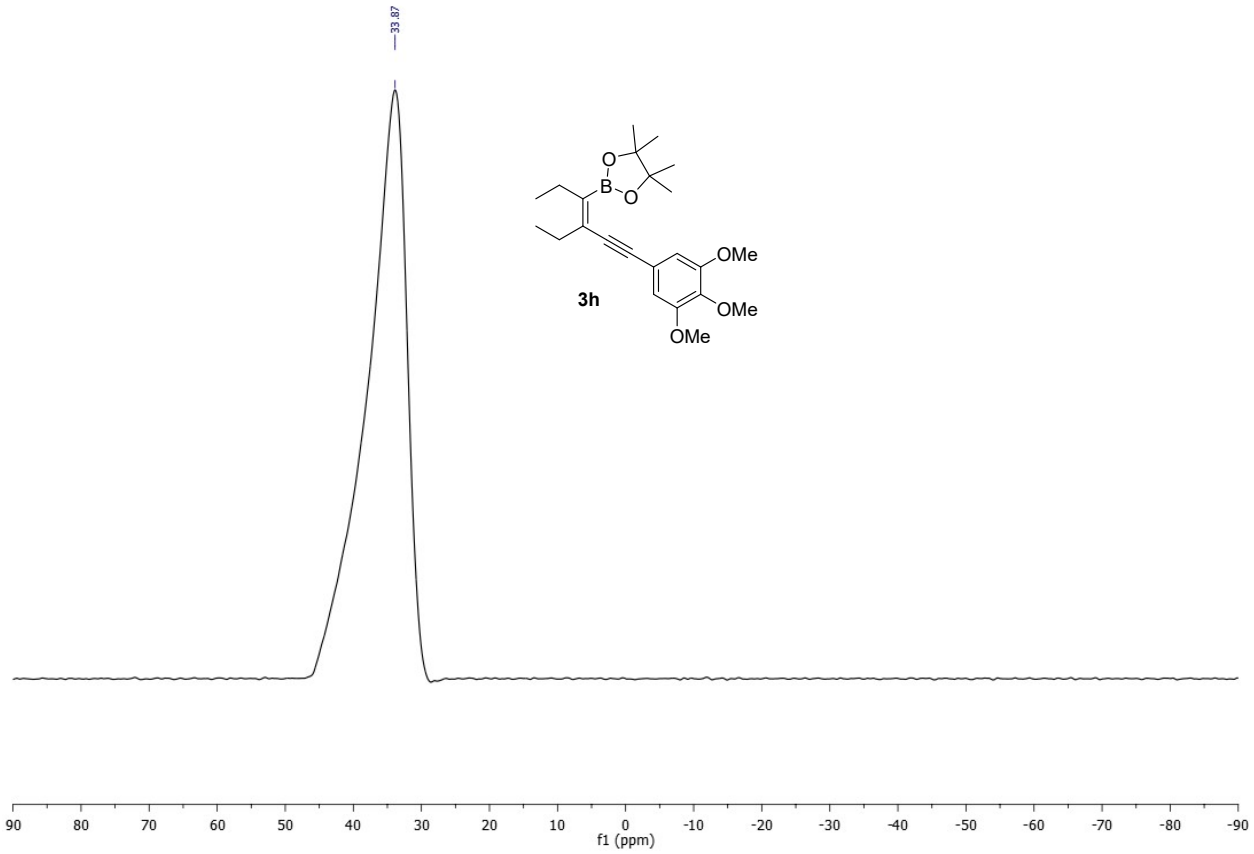
$^{11}B\{^{1}H\}$ NMR Spectrum of compound 3g (128 MHz, $CDCl_3$)



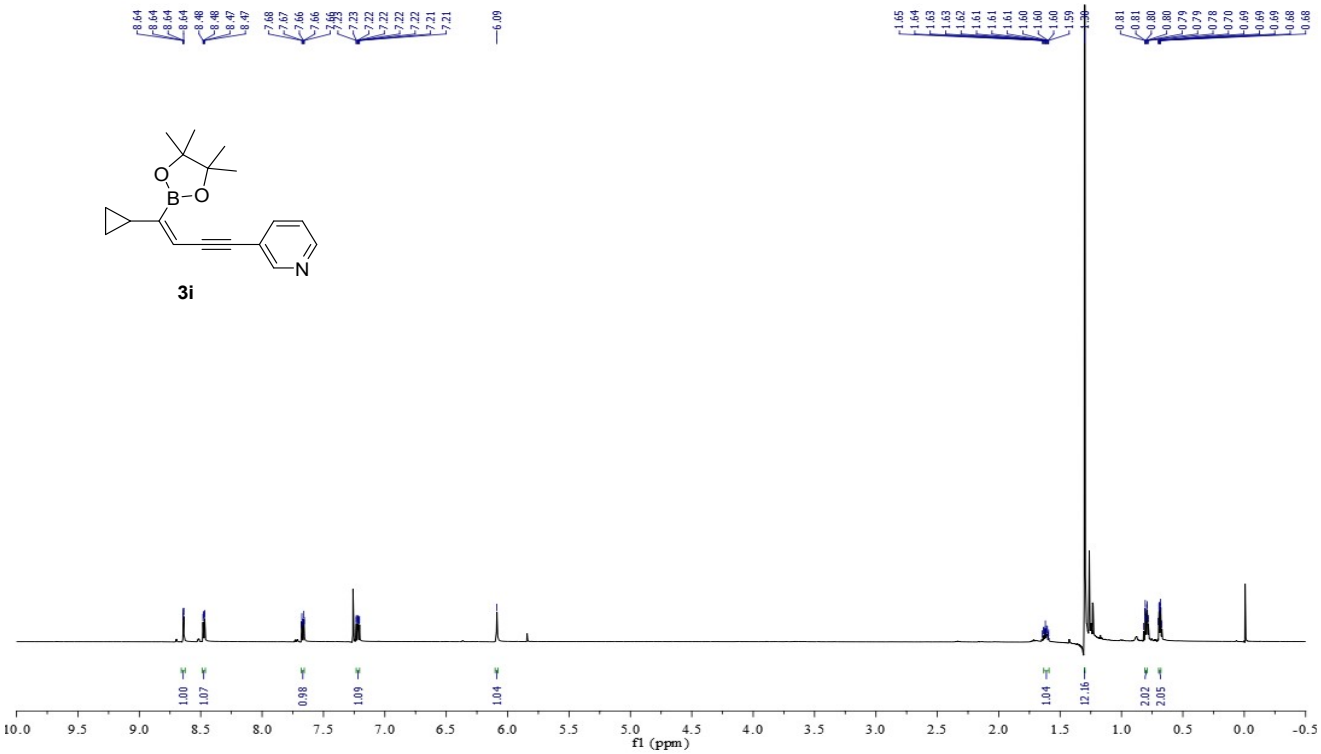
^{1}H NMR Spectrum of compound 3h (400 MHz, $CDCl_3$)



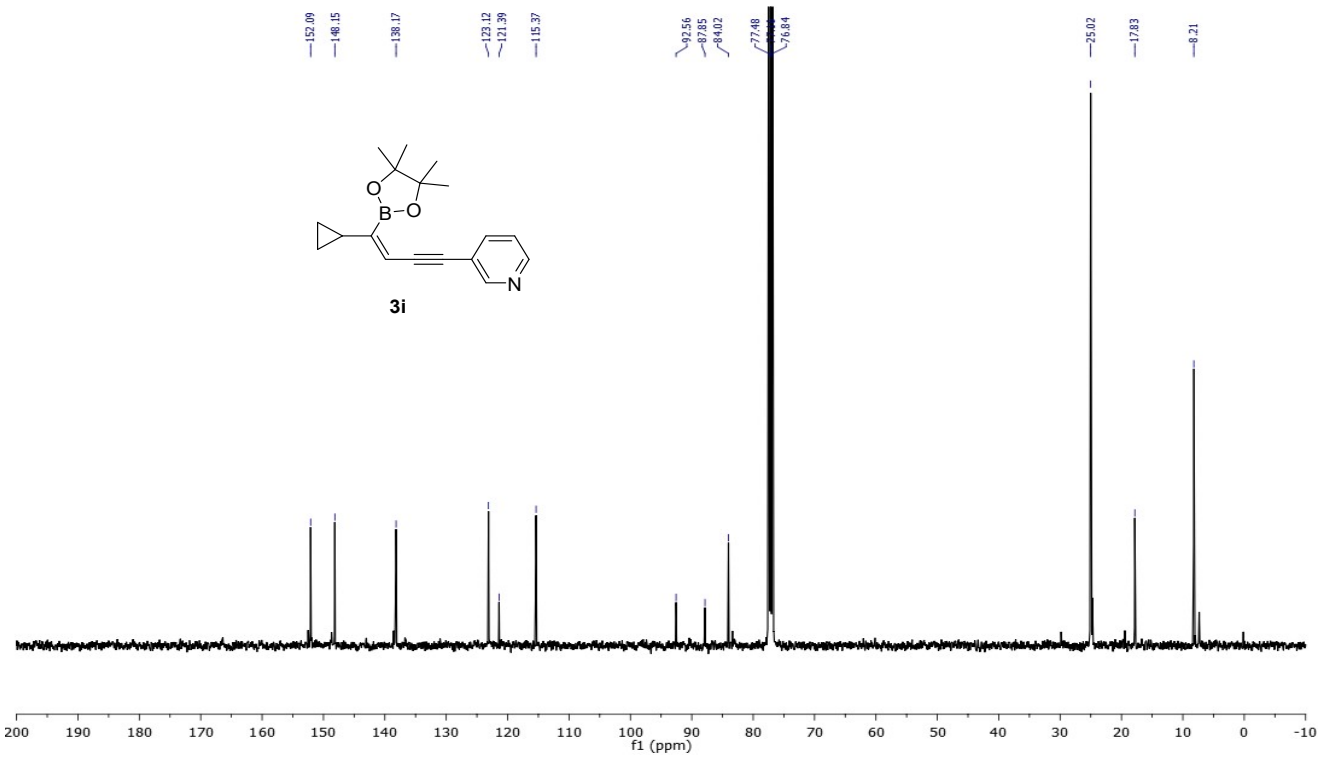
^{13}C{^{1}H} NMR Spectrum of compound 3h (101 MHz, $CDCl_3$)



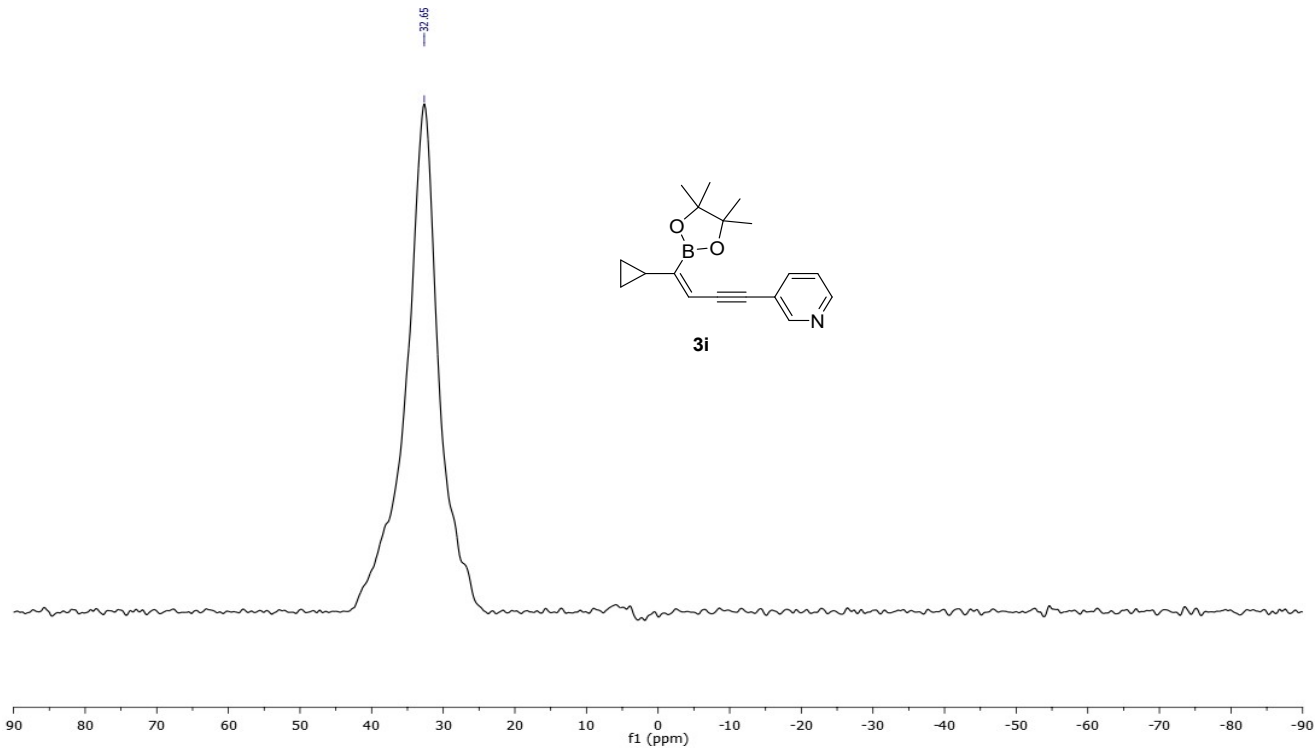
$^{11}B\{^{1}H\}$ NMR Spectrum of compound 3h (128 MHz, $CDCl_3$)



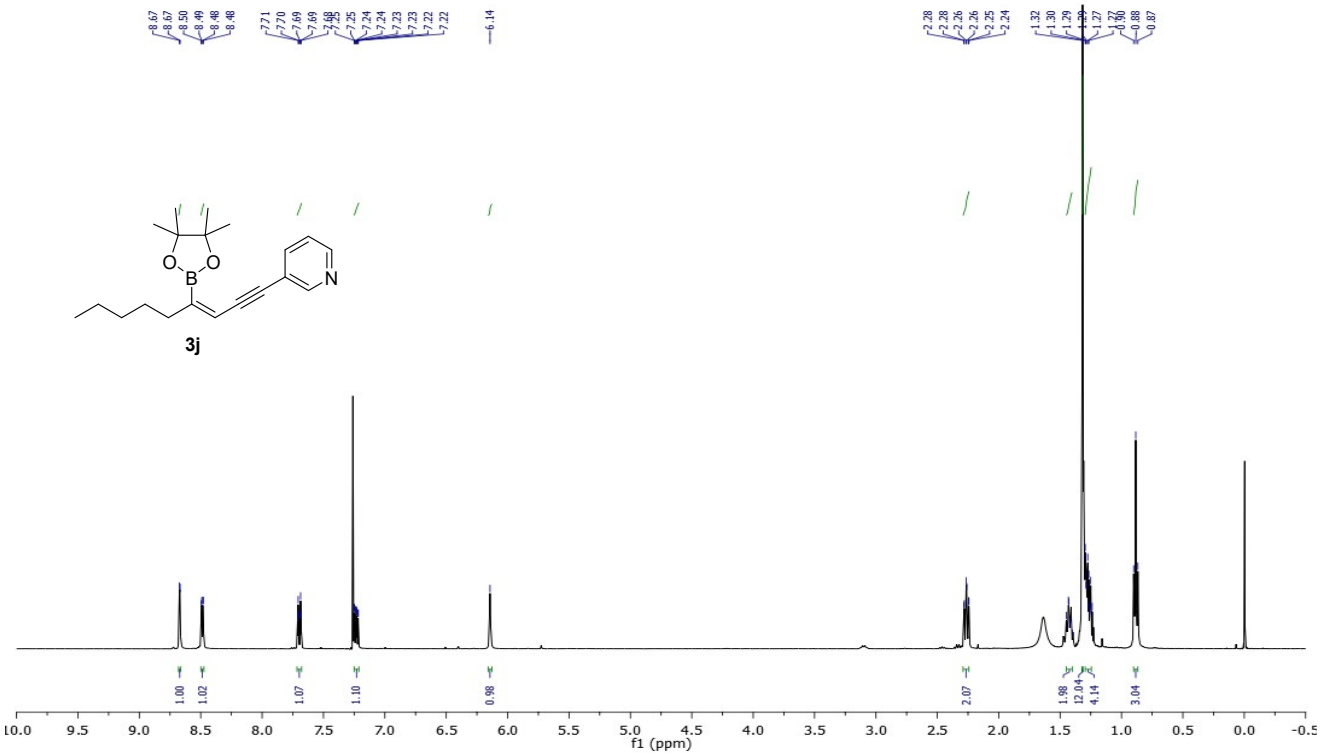
^{1}H NMR Spectrum of compound 3i (500 MHz, $CDCl_3$)



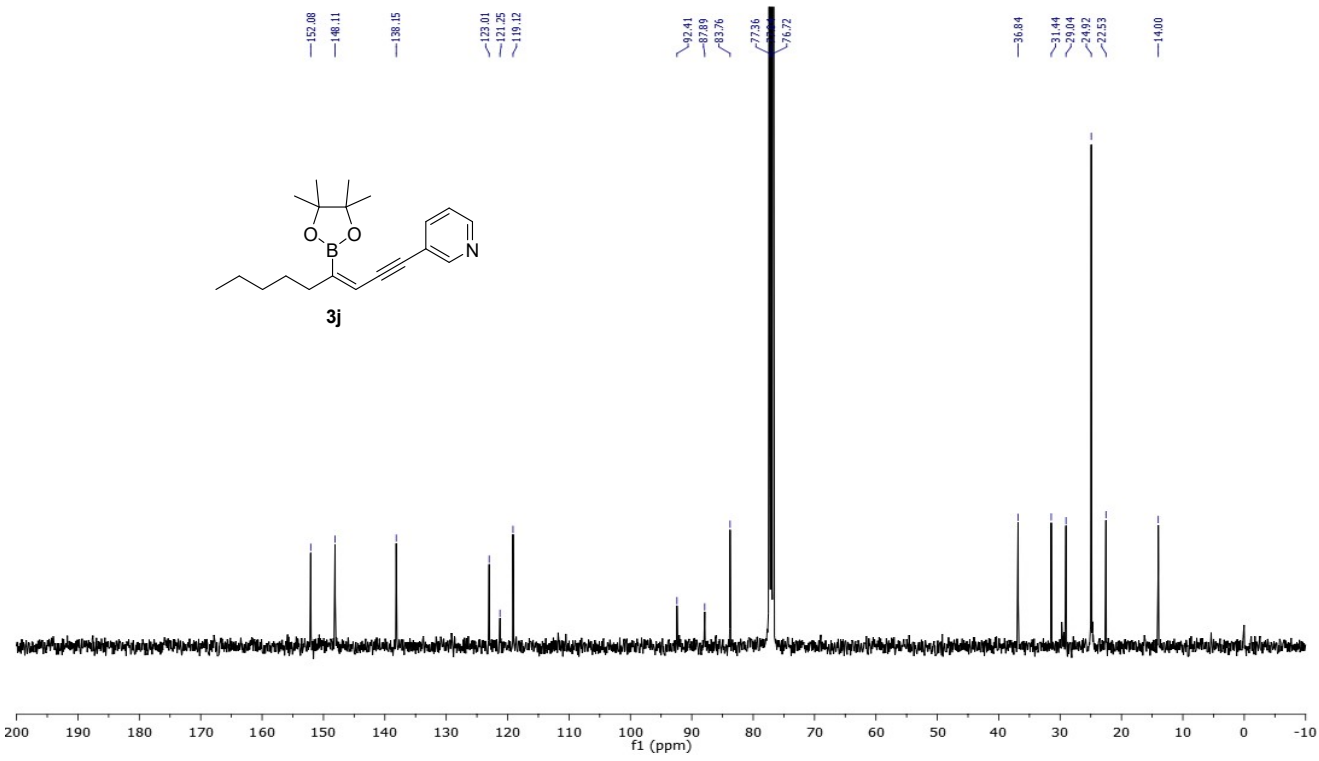
^{13}C{^{1}H} NMR Spectrum of compound 3i (101 MHz, $CDCl_3$)



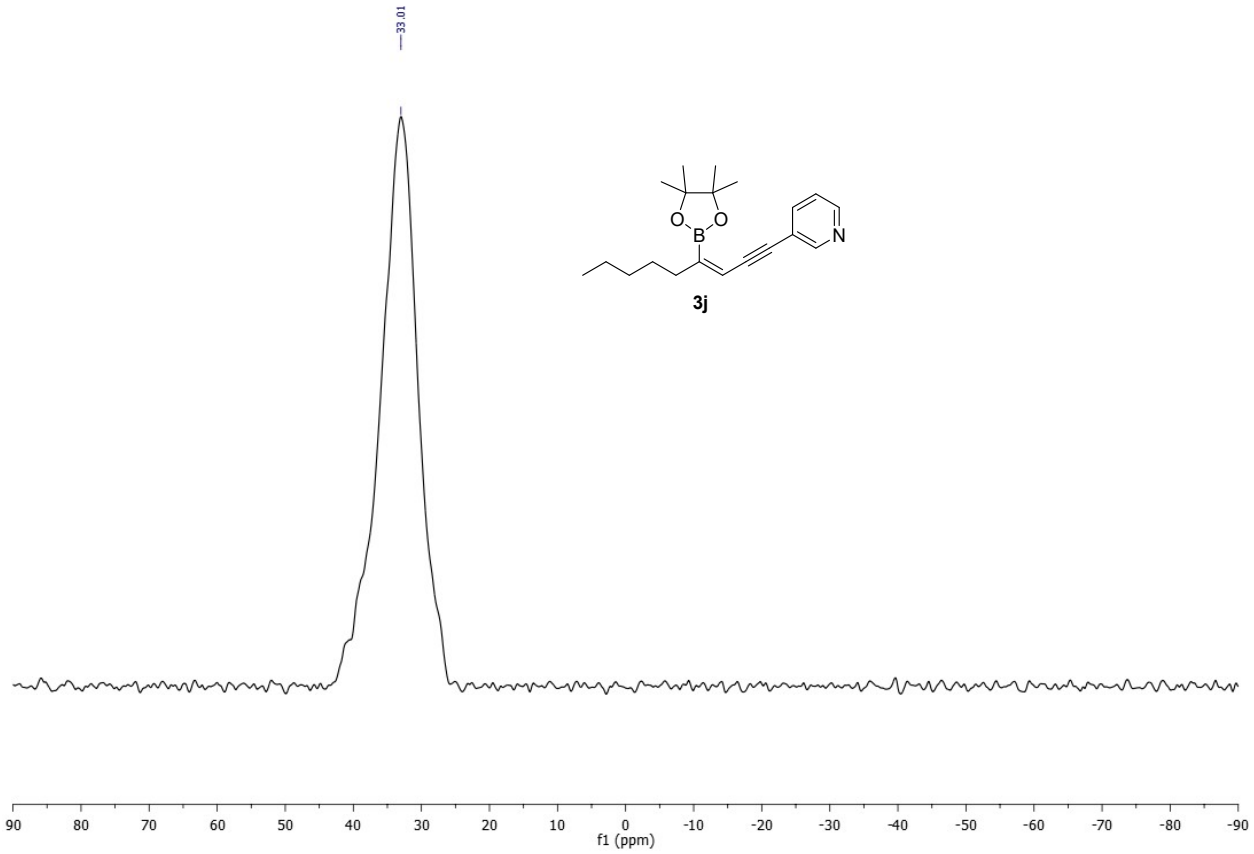
$^{11}B\{^{1}H\}$ NMR Spectrum of compound 3i (128 MHz, $CDCl_3$)



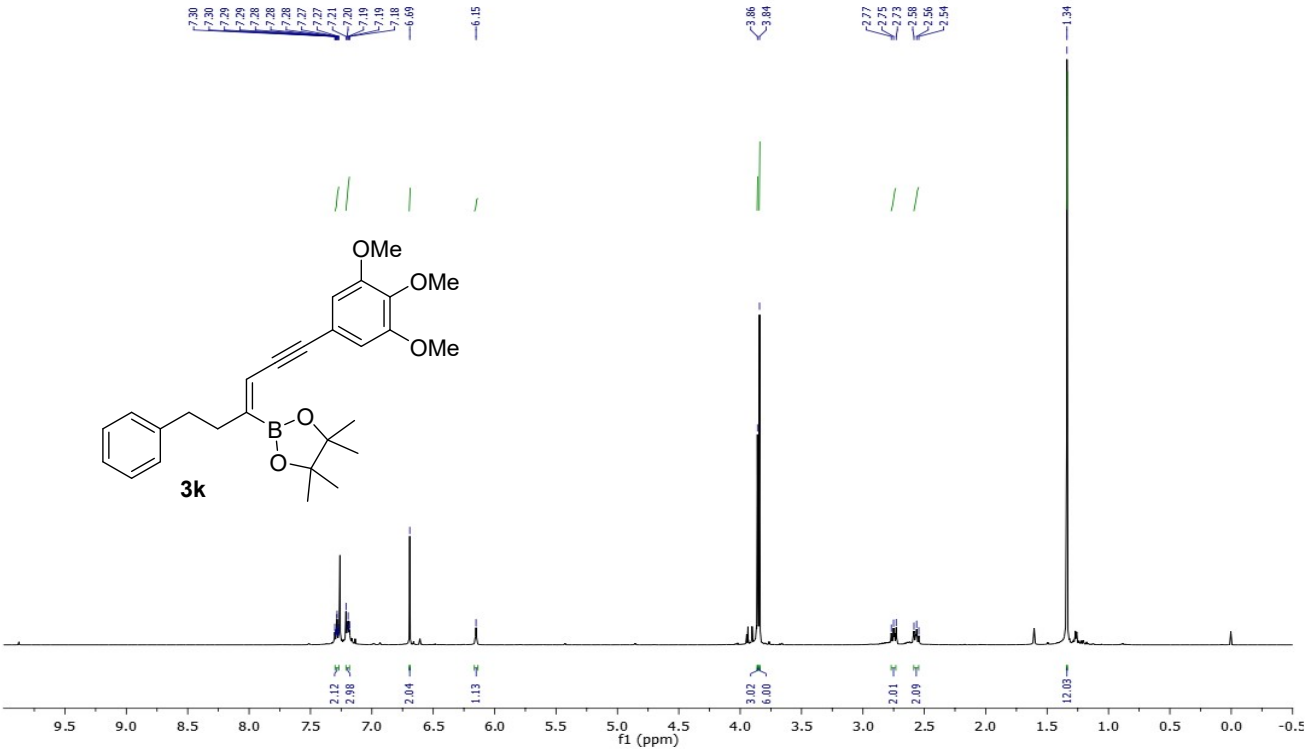
^{1}H NMR Spectrum of compound 3j (400 MHz, $CDCl_3$)



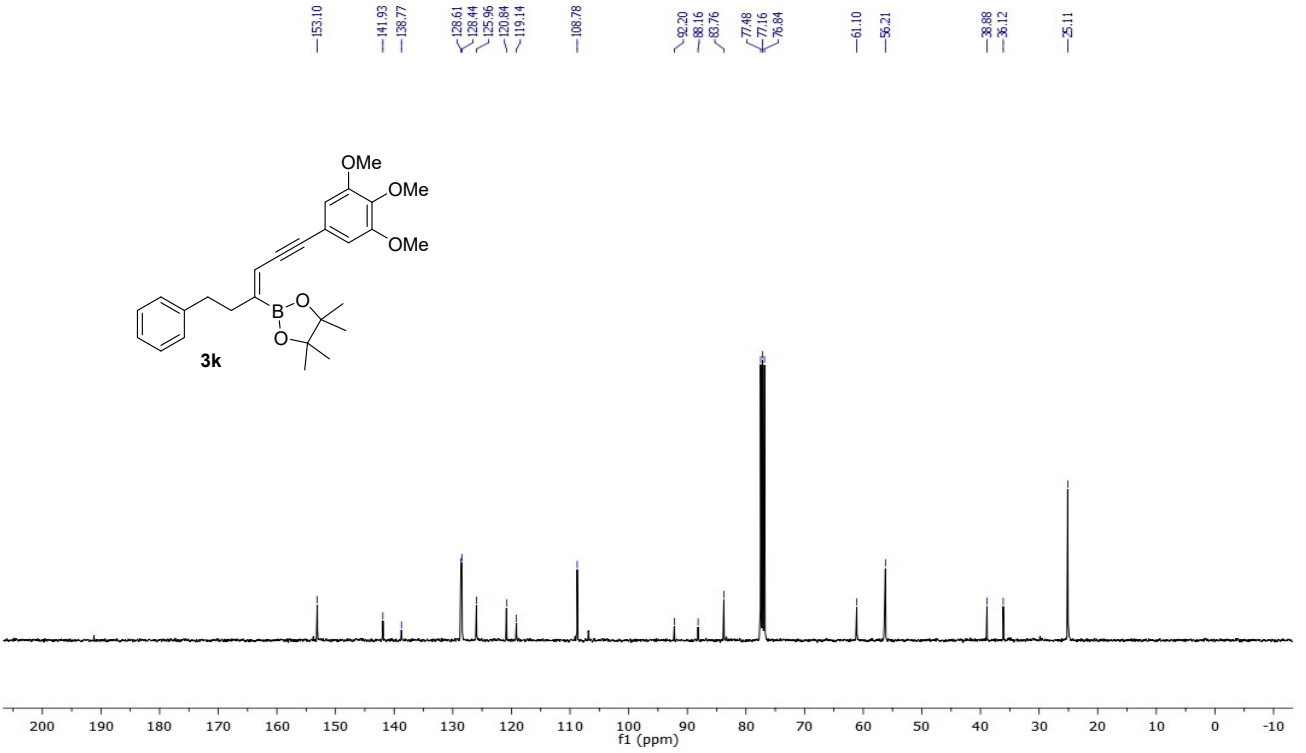
^{13}C{^{1}H} NMR Spectrum of compound 3j (101 MHz, $CDCl_3$)



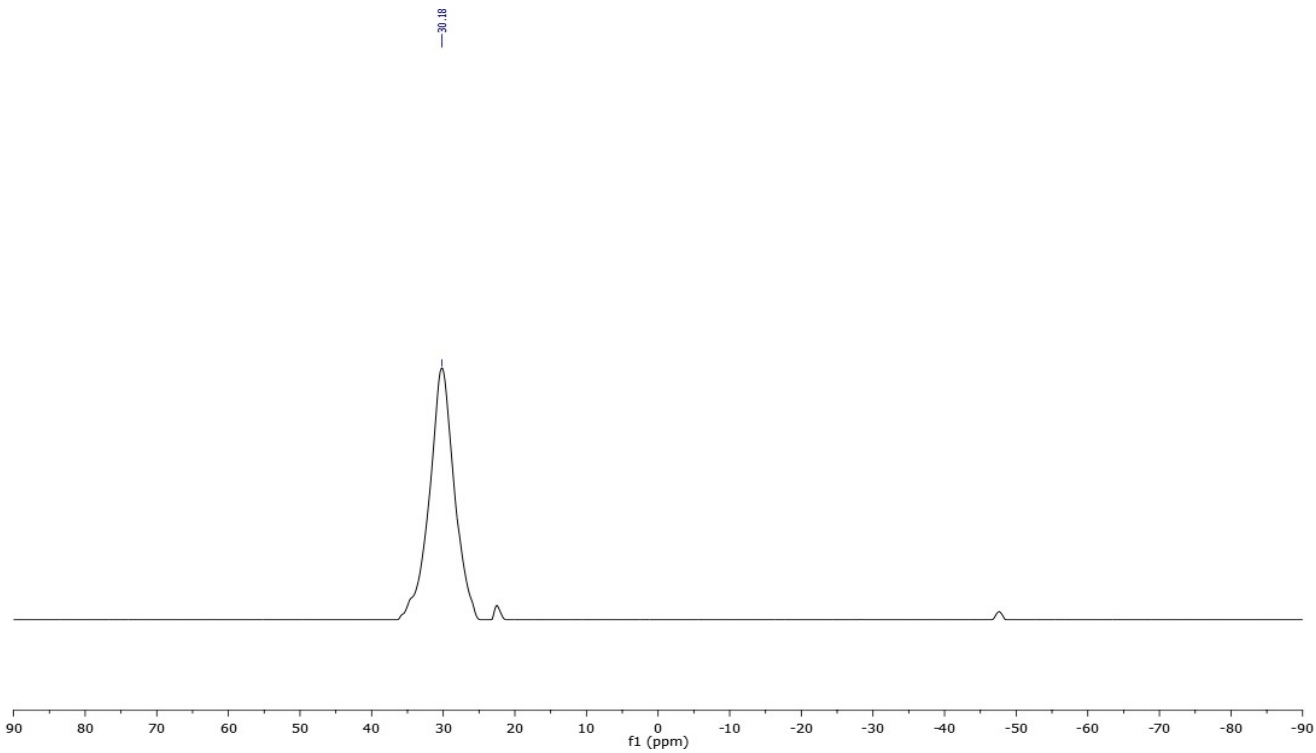
$^{11}B\{^{1}H\}$ NMR Spectrum of compound 3j (128 MHz, $CDCl_3$)



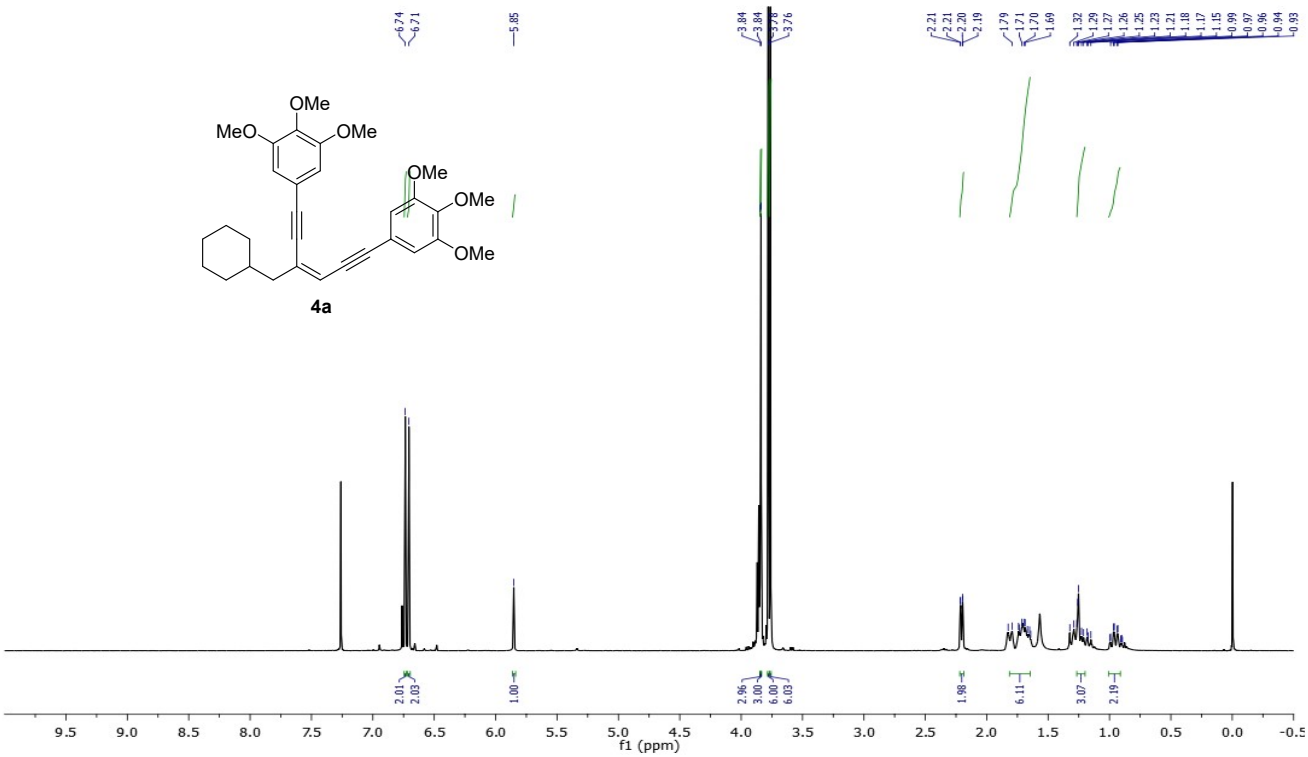
^{1}H NMR Spectrum of compound 3k (400 MHz, $CDCl_3$)



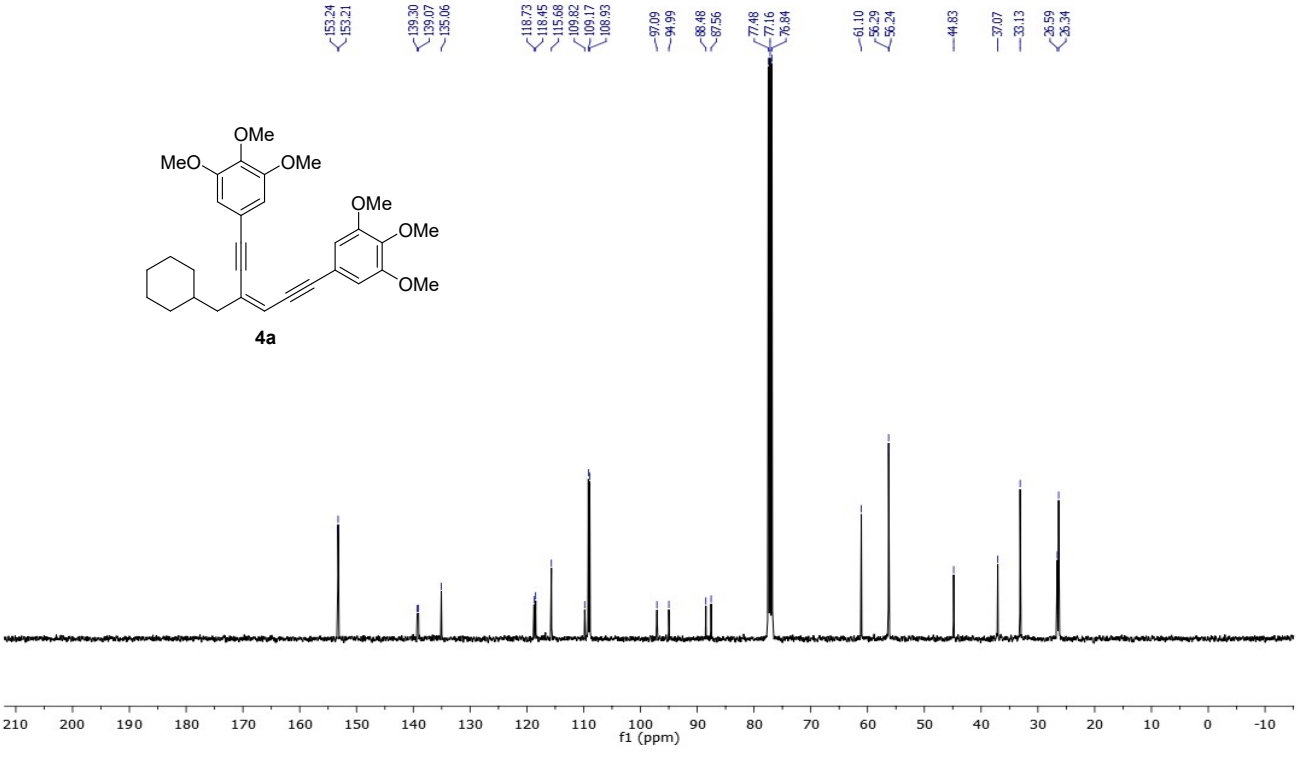
^{13}C{^{1}H} NMR Spectrum of compound 3k (101 MHz, $CDCl_3$)



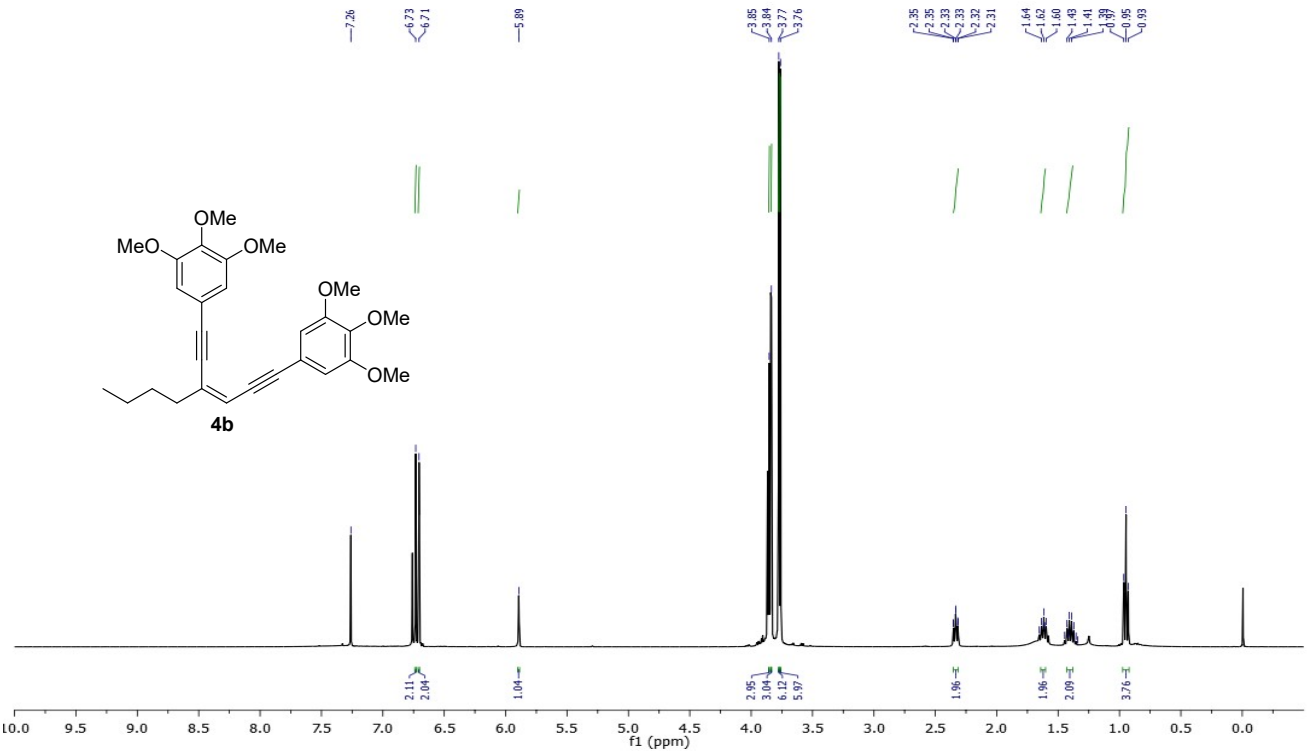
$^{11}B\{^{1}H\}$ NMR Spectrum of compound 3k (128 MHz, $CDCl_3$)



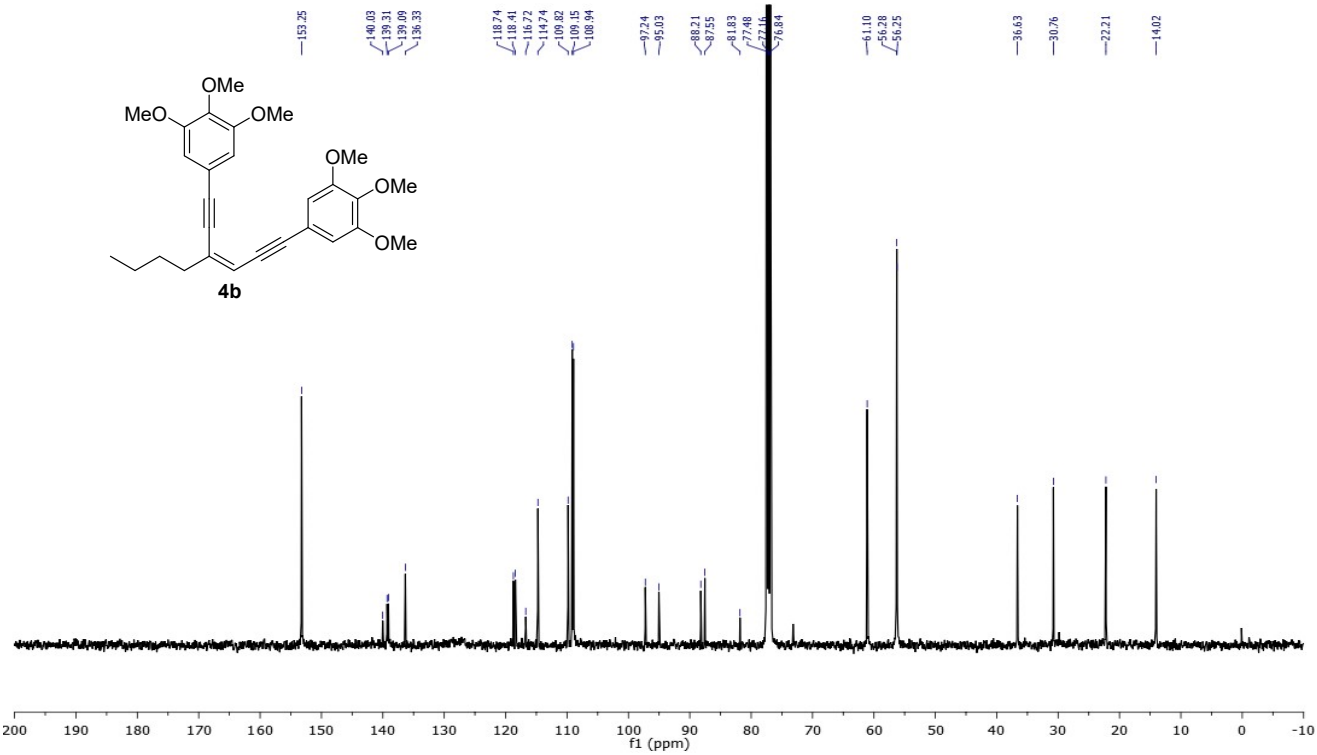
^{1}H NMR Spectrum of compound 4a (400 MHz, $CDCl_3$)



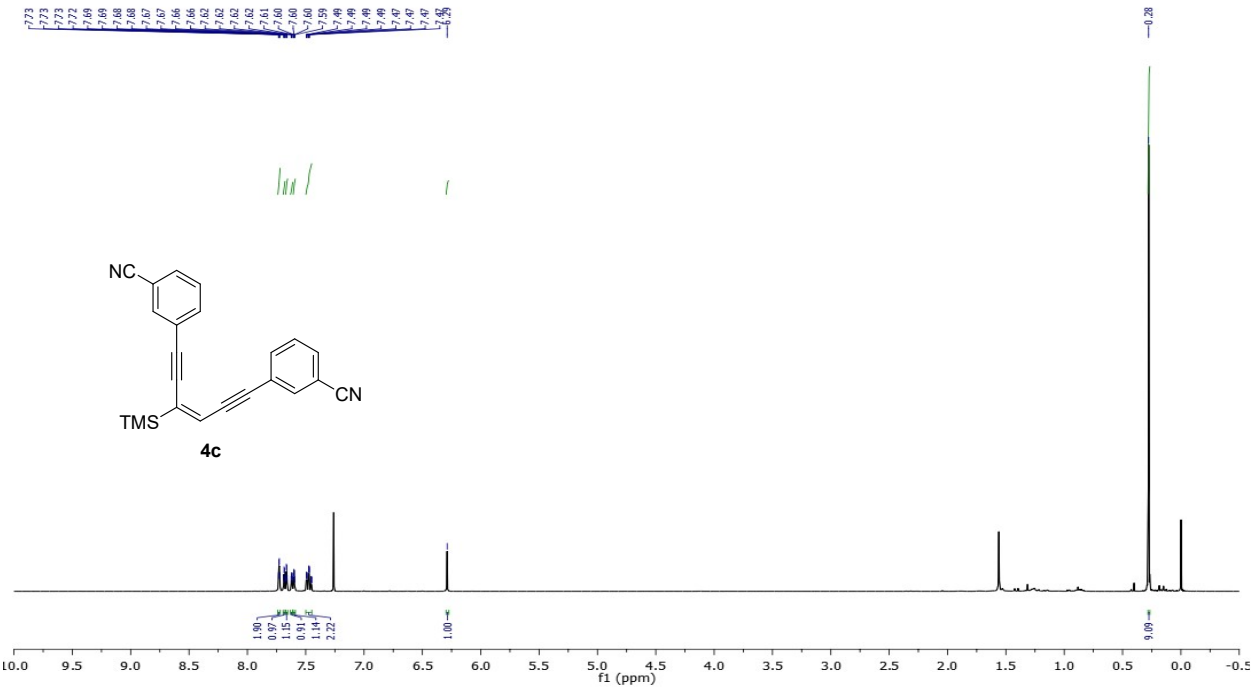
^{13}C{^{1}H} NMR Spectrum of compound 4a (101 MHz, $CDCl_3$)



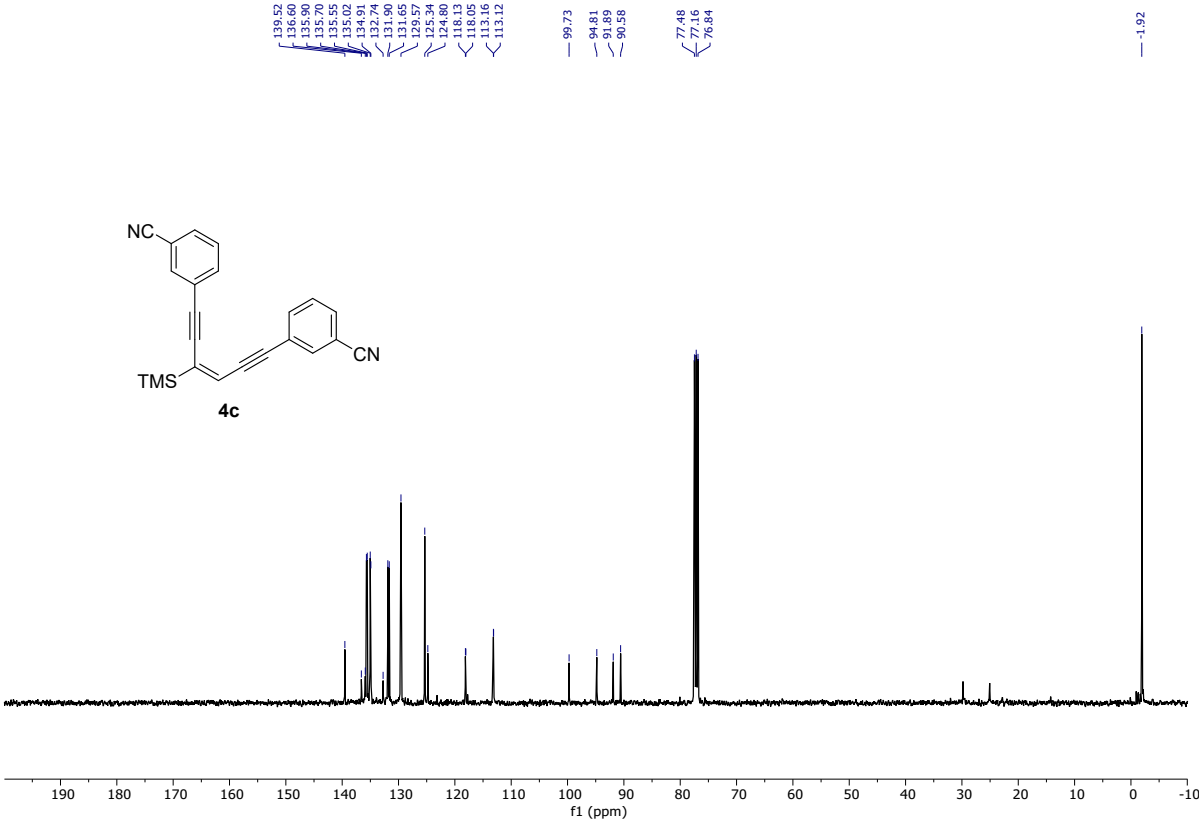
^{1}H NMR Spectrum of compound 4b (400 MHz, $CDCl_3$)



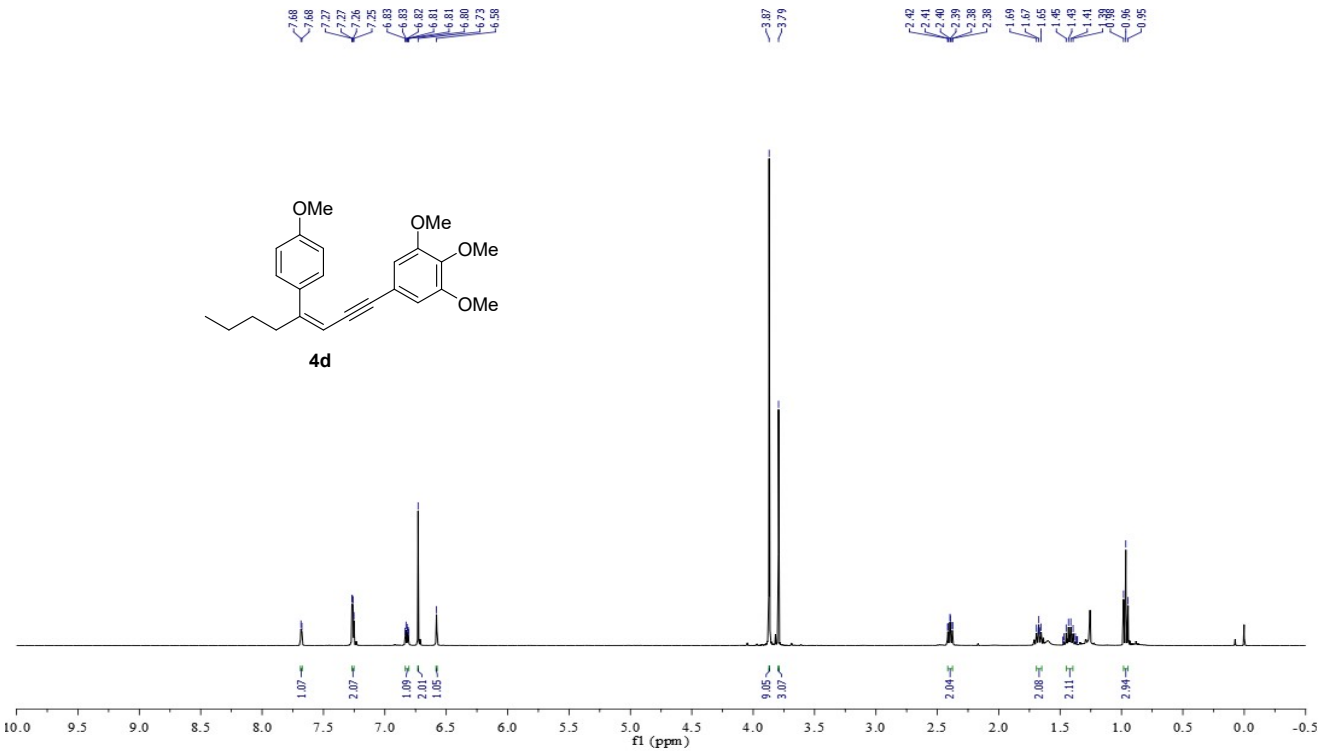
^{13}C{^{1}H} NMR Spectrum of compound 4b (101 MHz, $CDCl_3$)



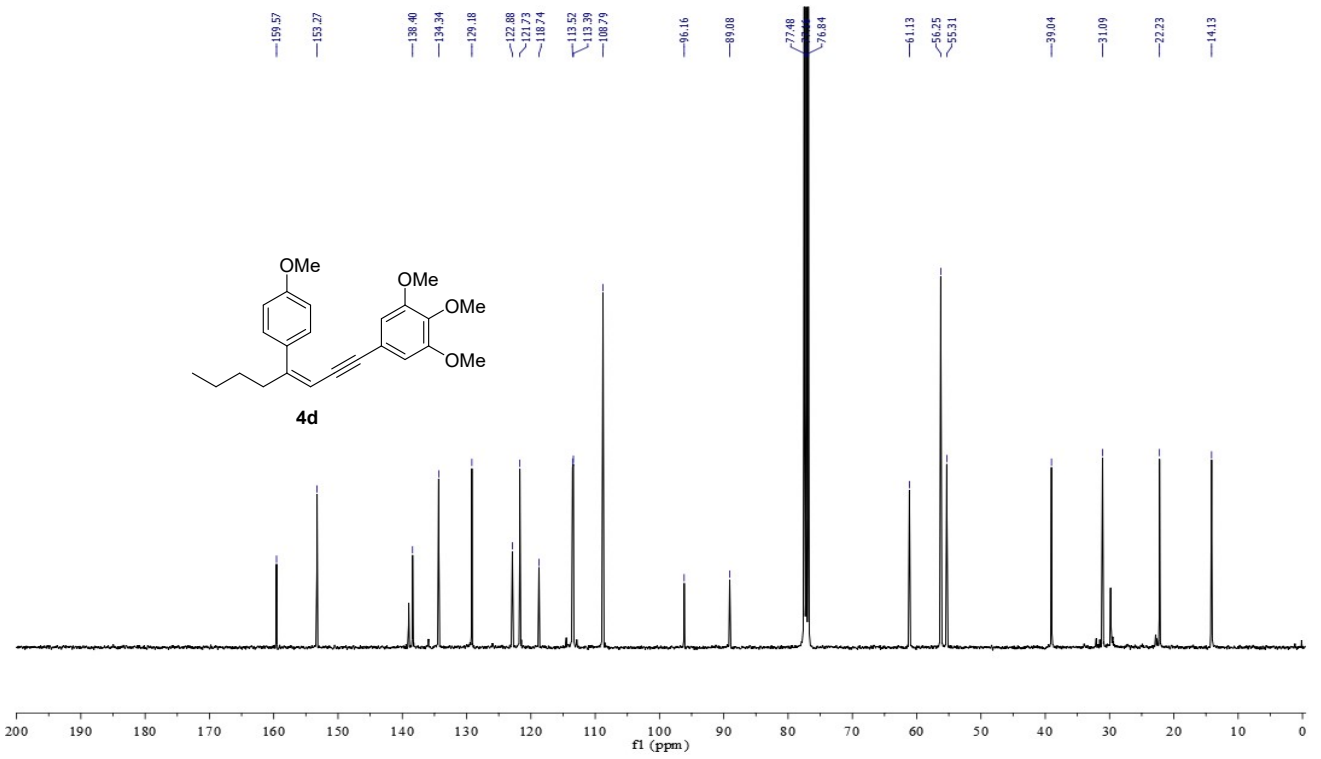
^{1}H NMR Spectrum of compound 4c (400 MHz, $CDCl_3$)



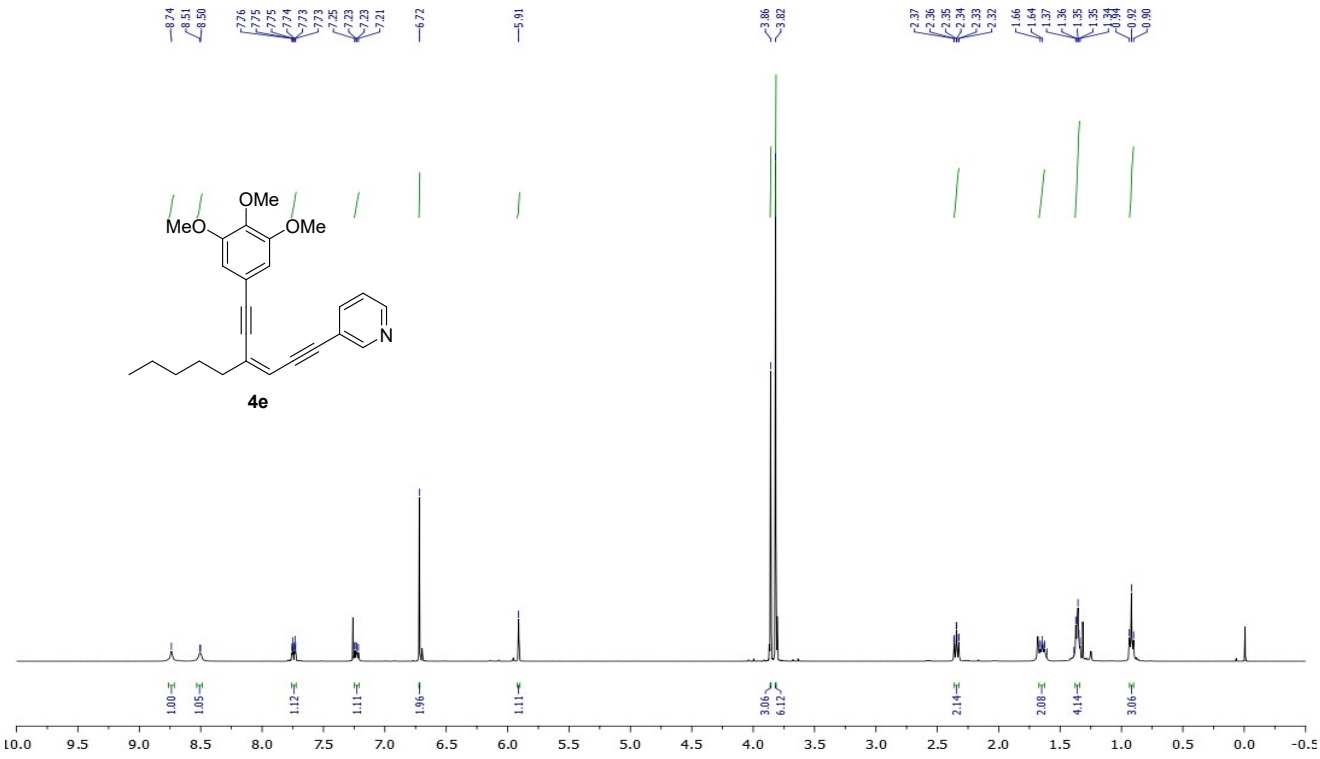
^{13}C{^{1}H} NMR Spectrum of compound 4c (101 MHz, $CDCl_3$)



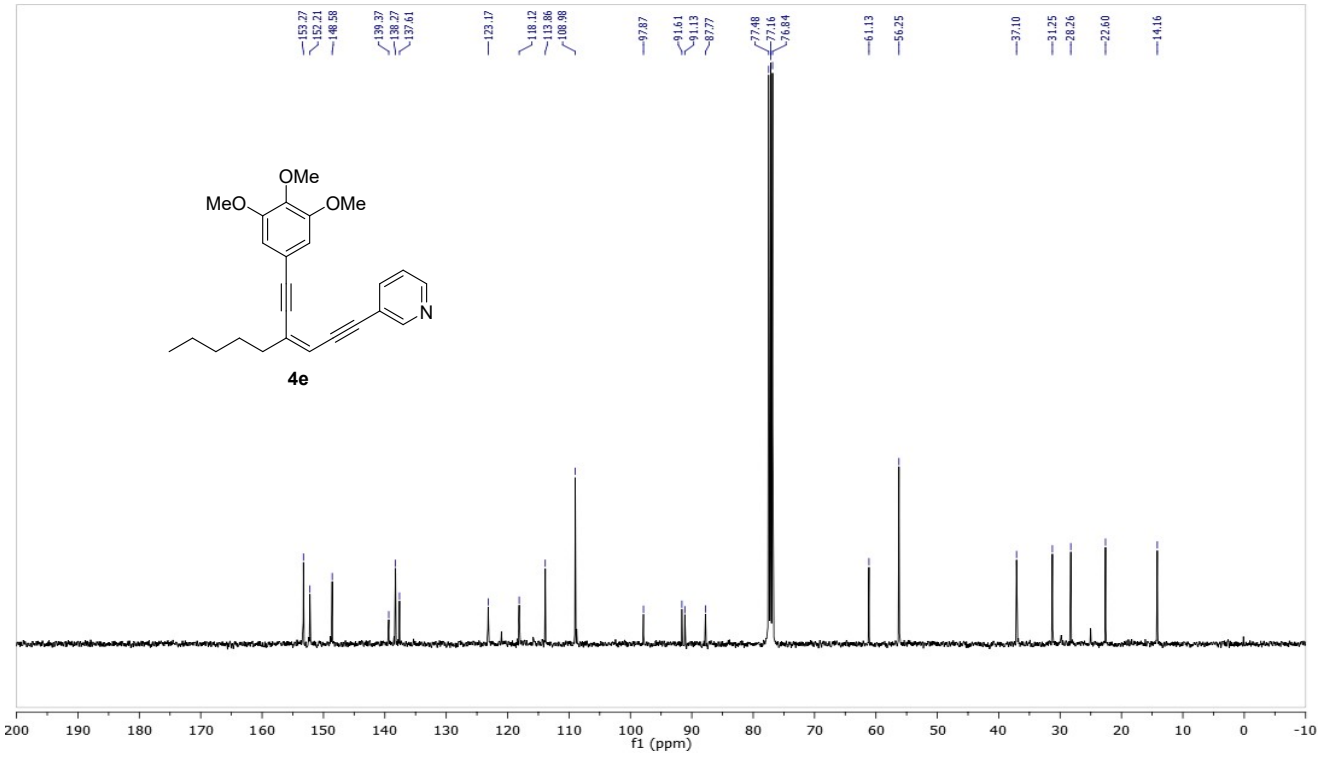
^{1}H NMR Spectrum of compound 4d (400 MHz, $CDCl_3$)



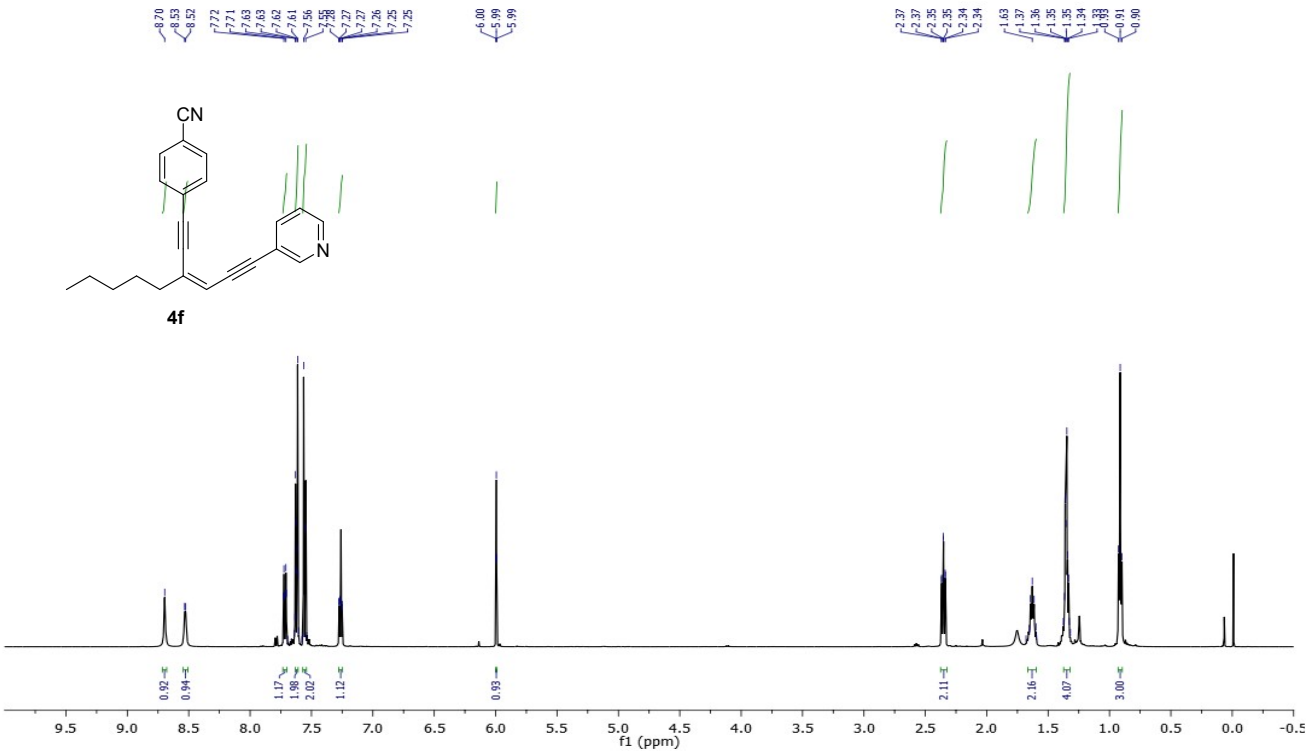
^{13}C{^{1}H} NMR Spectrum of compound 4d (101 MHz, $CDCl_3$)



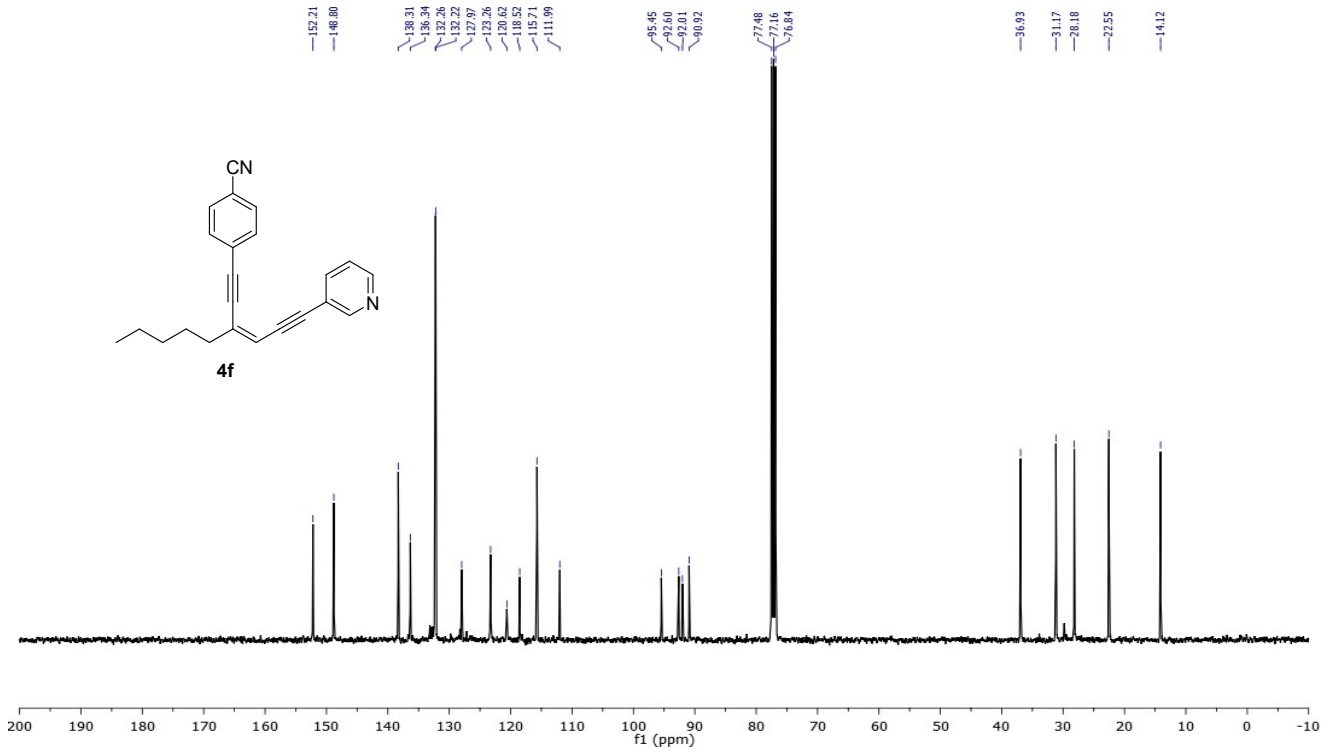
^{1}H NMR Spectrum of compound 4e (400 MHz, $CDCl_3$)



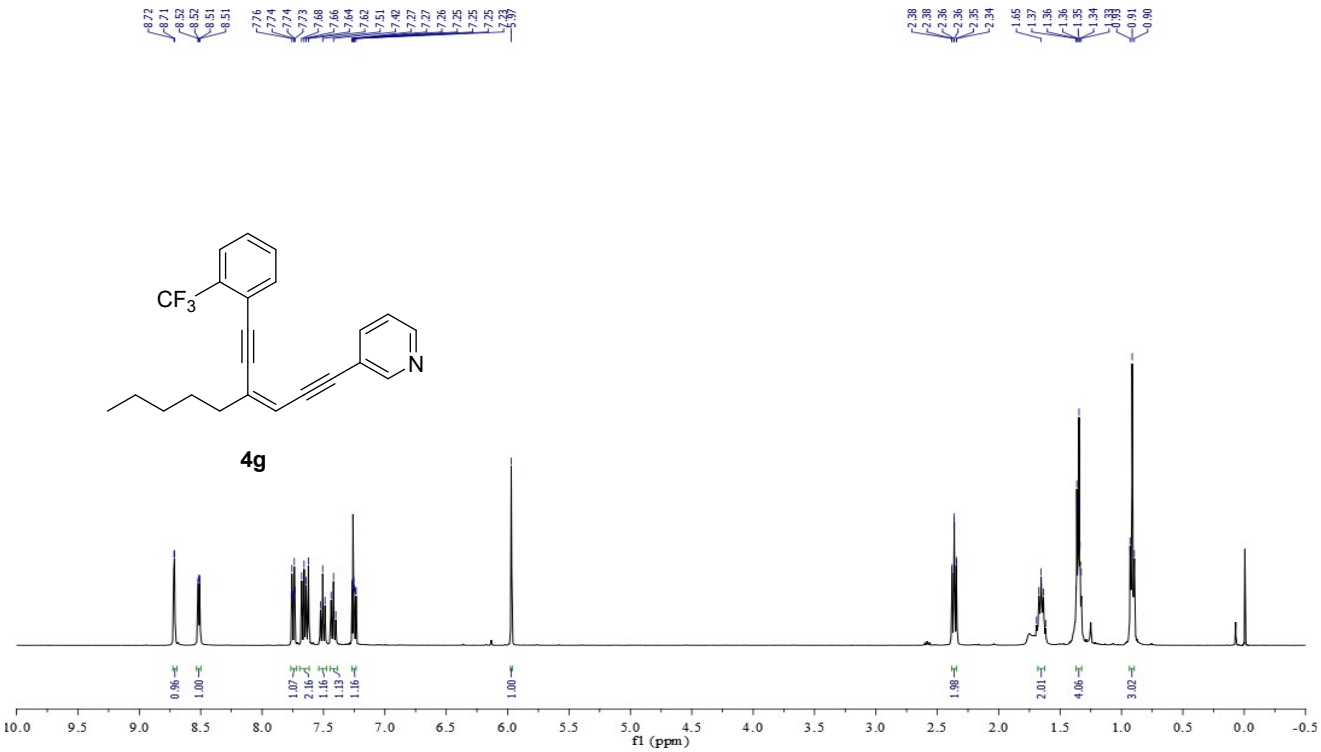
^{13}C{^{1}H} NMR Spectrum of compound 4e (101 MHz, $CDCl_3$)



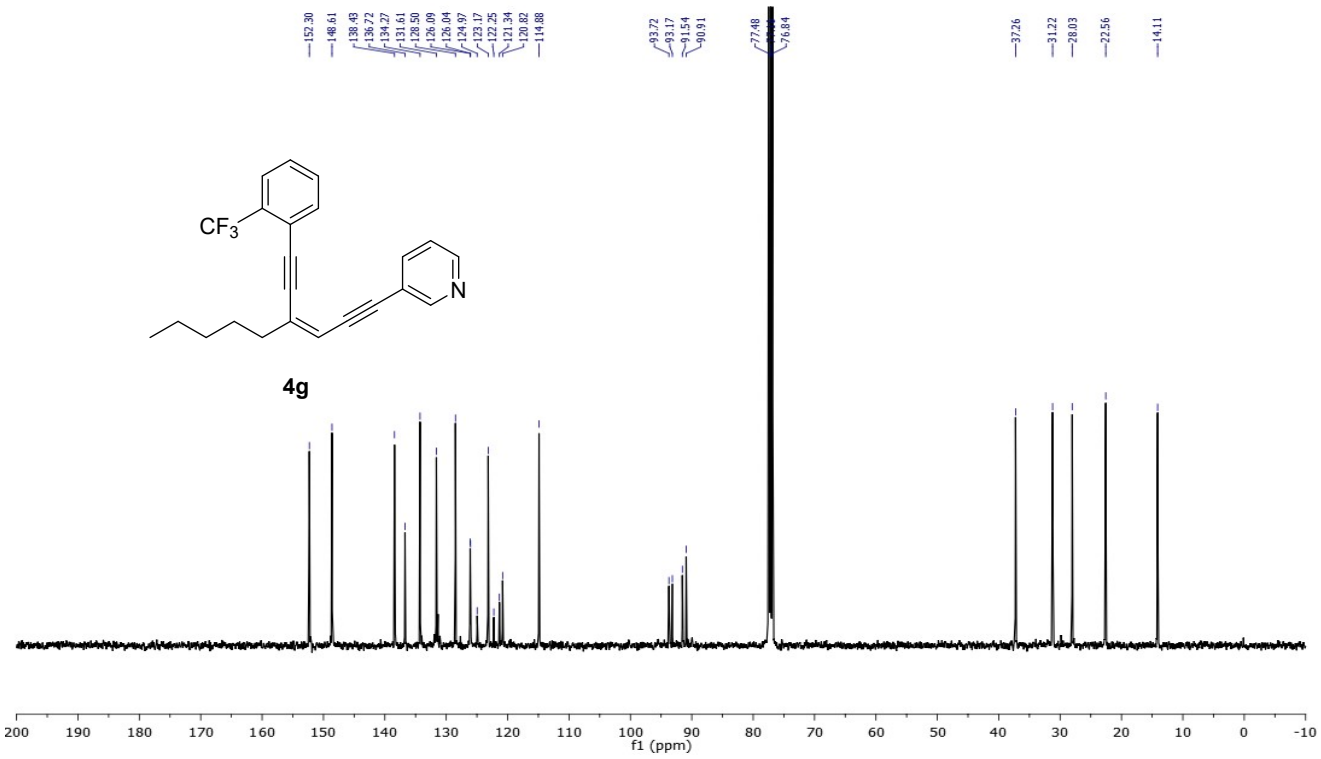
^{1}H NMR Spectrum of compound 4f (500 MHz, $CDCl_3$)



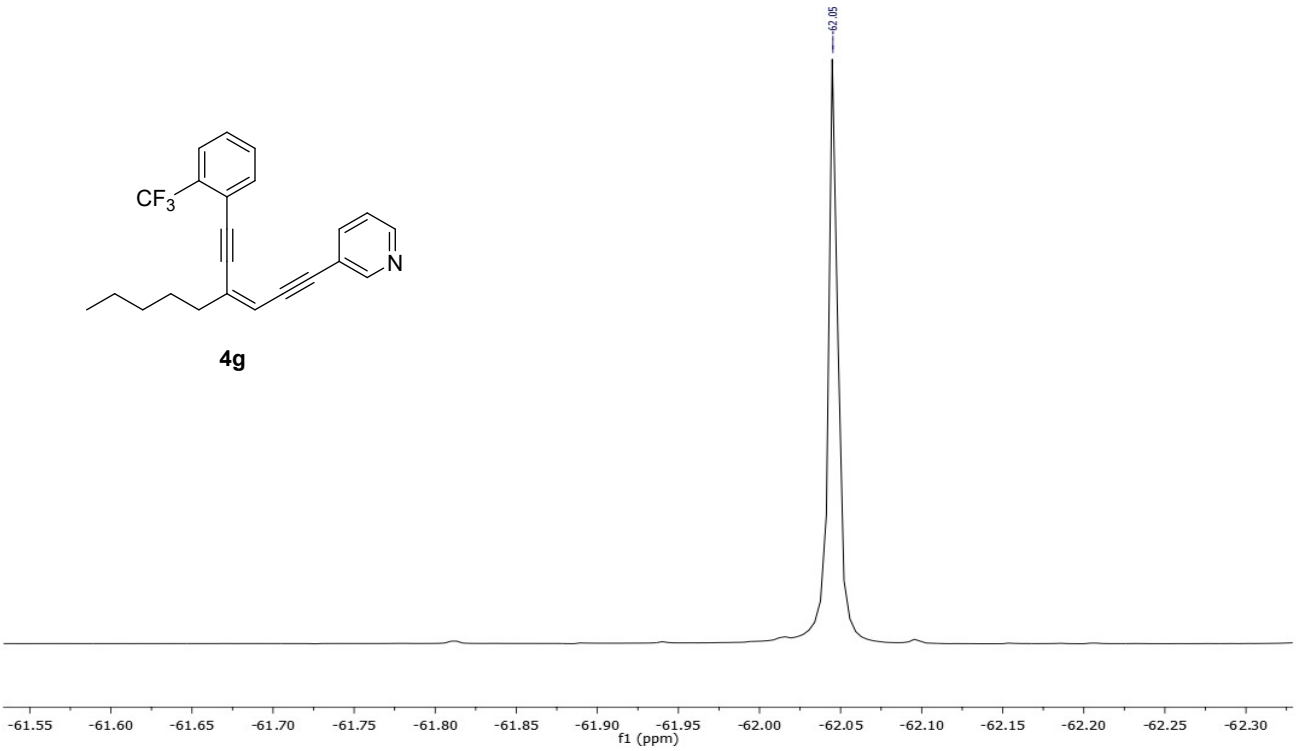
^{13}C{^{1}H} NMR Spectrum of compound 4f (101 MHz, $CDCl_3$)



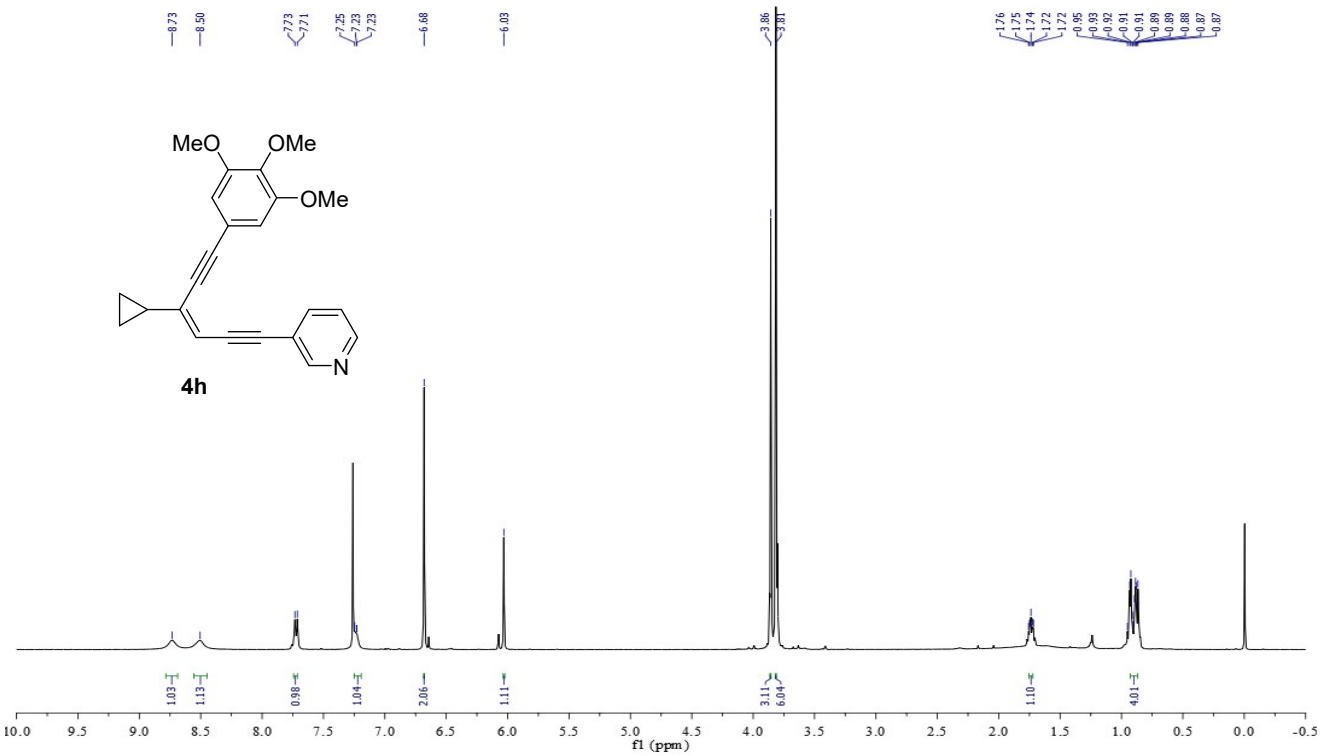
^{1}H NMR Spectrum of compound 4g (400 MHz, $CDCl_3$)



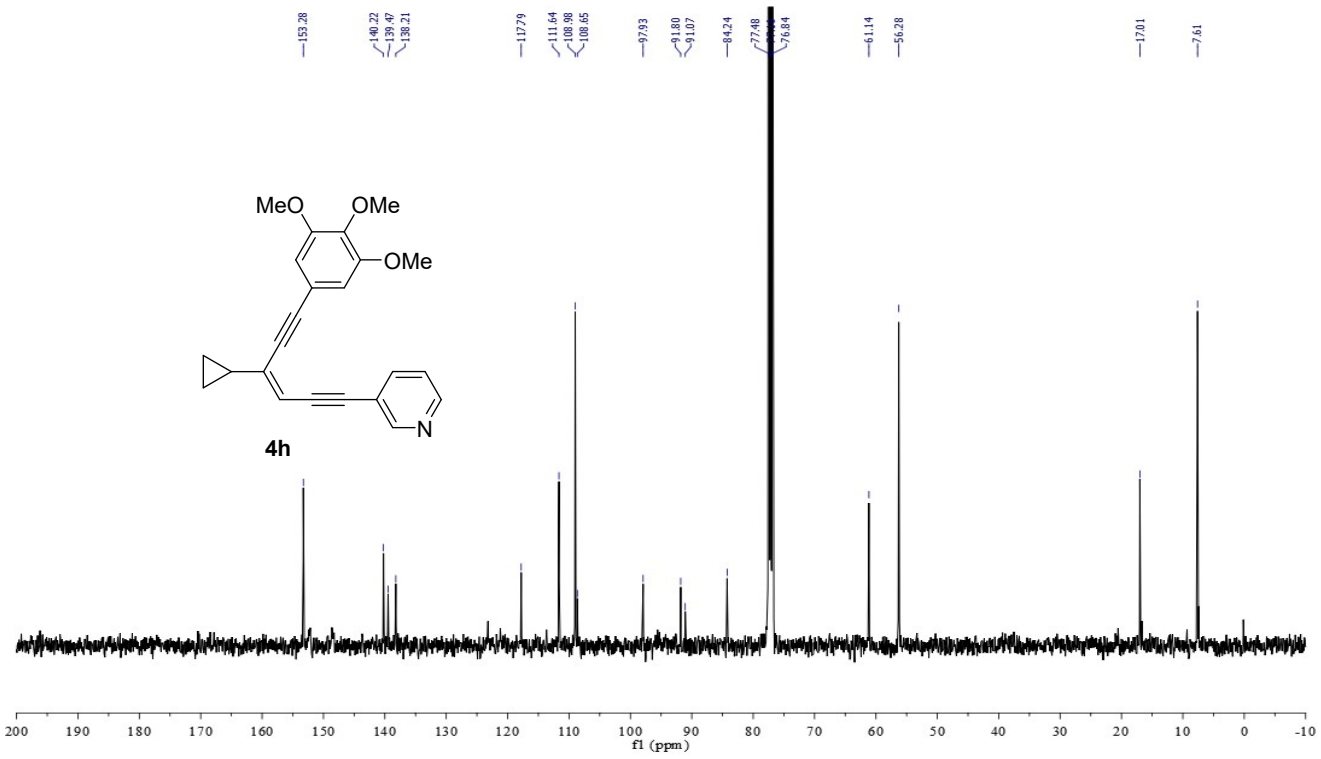
^{13}C{^{1}H} NMR Spectrum of compound 4g (101 MHz, $CDCl_3$)



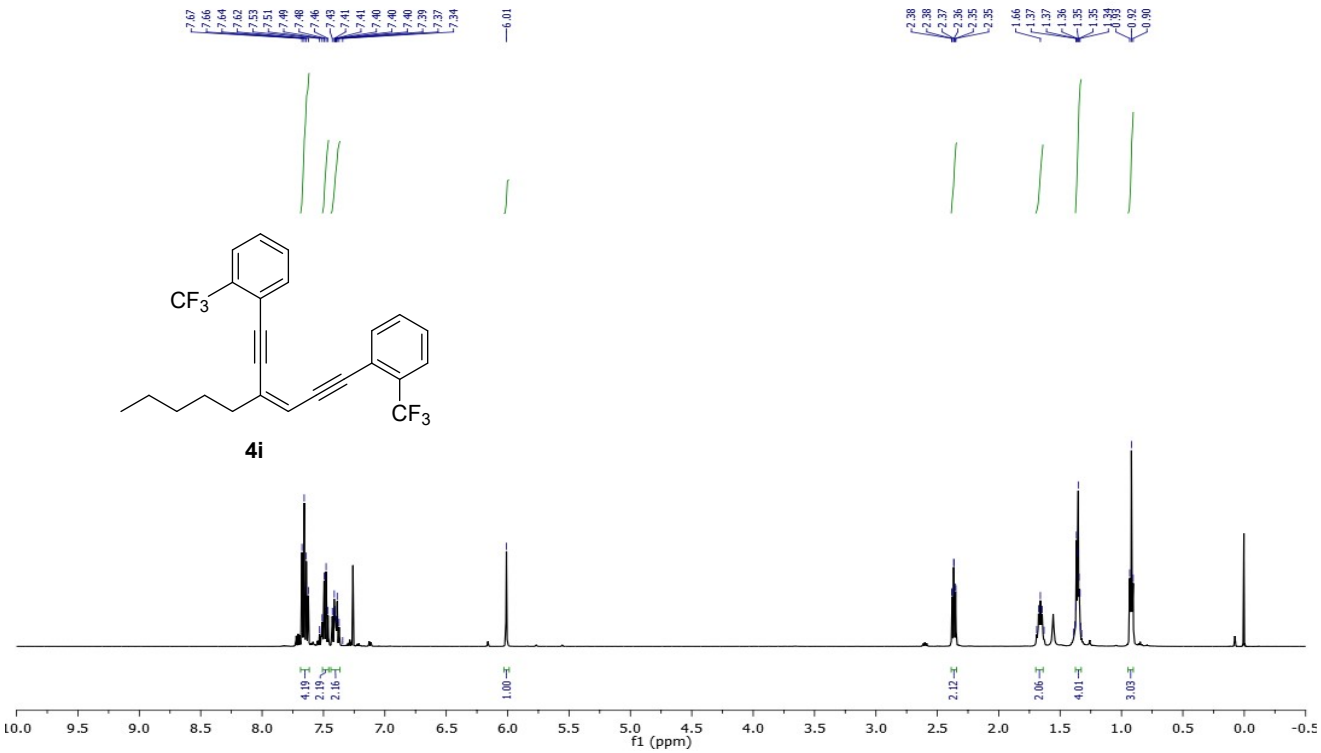
$^{19}F\{^{1}H\}$ NMR Spectrum of compound 4g (377 MHz, $CDCl_3$)



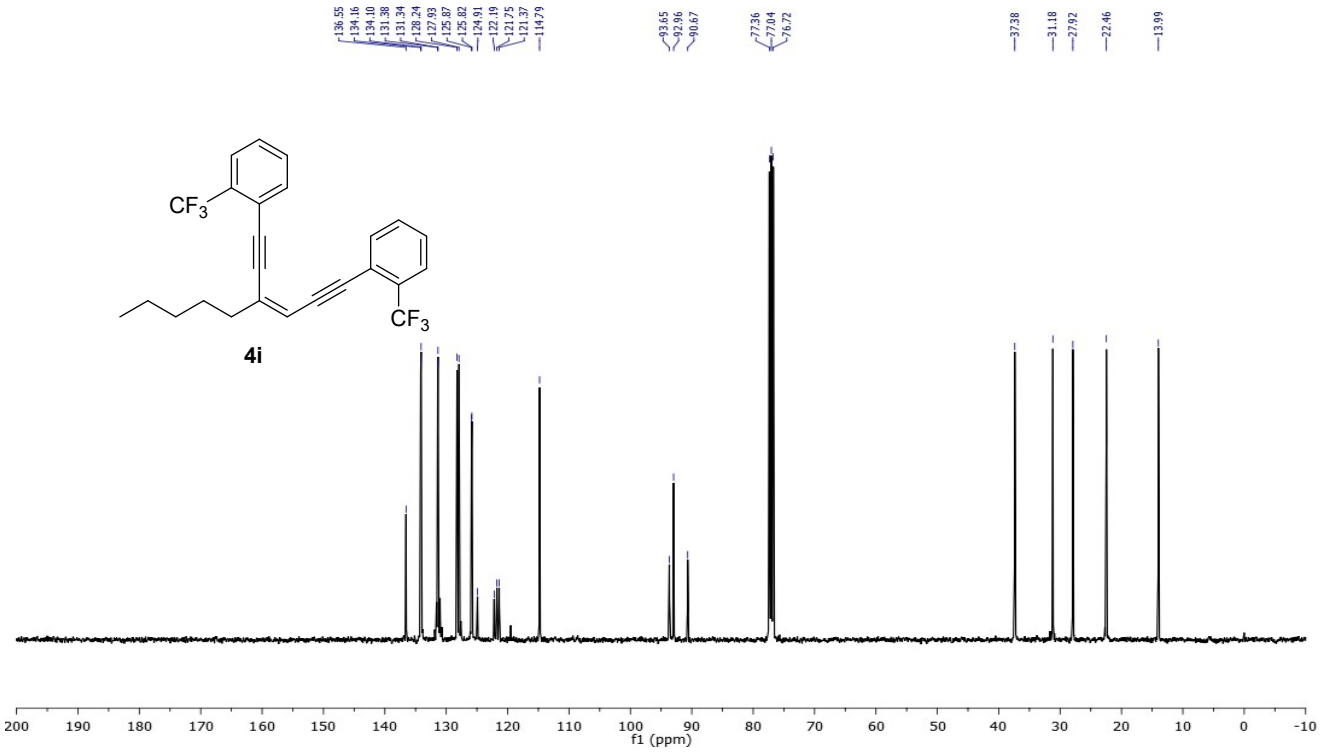
^{1}H NMR Spectrum of compound 4h (400 MHz, $CDCl_3$)



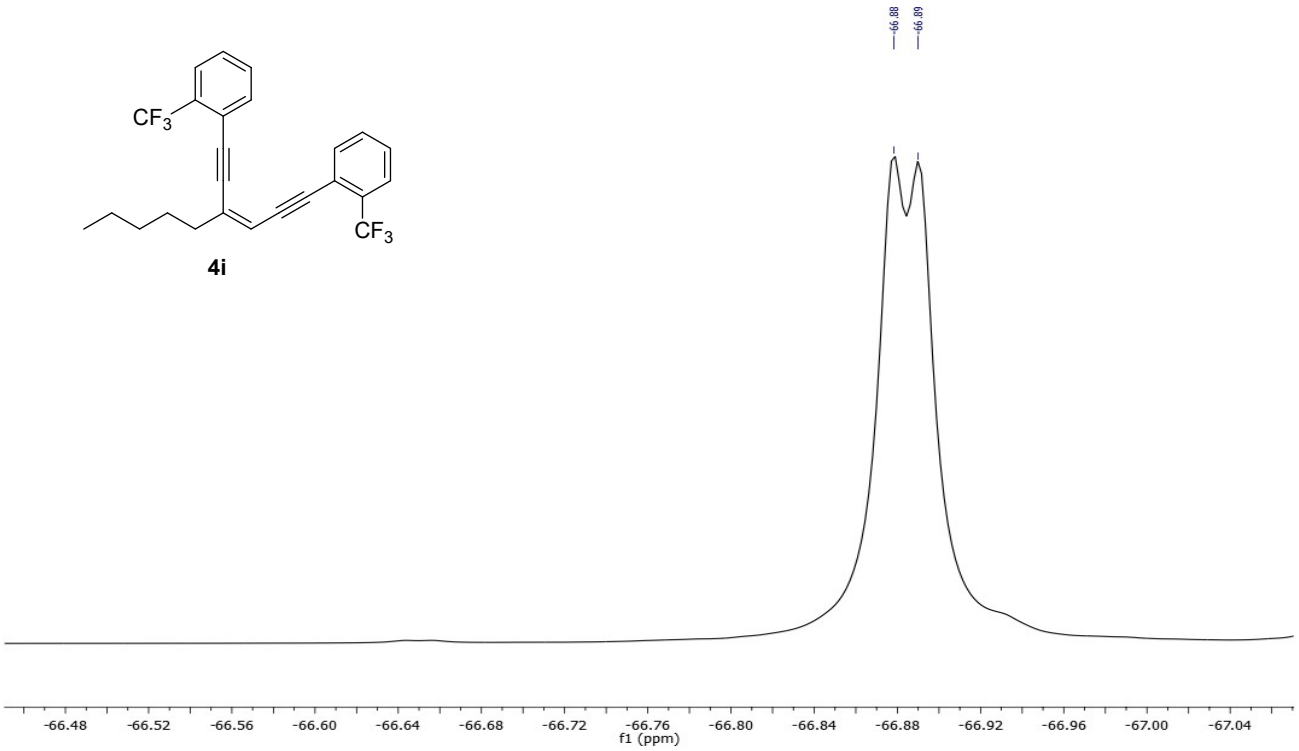
^{13}C{^{1}H} NMR Spectrum of compound 4h (101 MHz, $CDCl_3$)



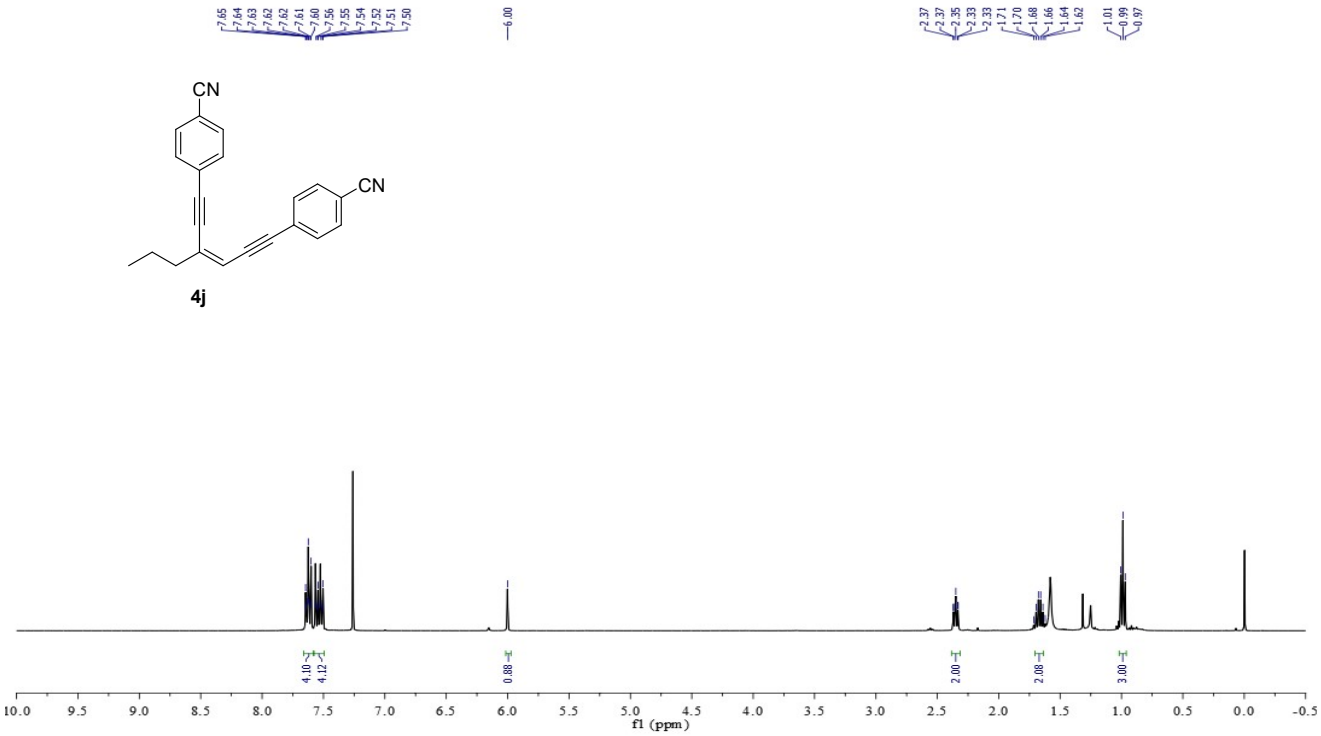
^{1}H NMR Spectrum of compound 4i (500 MHz, $CDCl_3$)



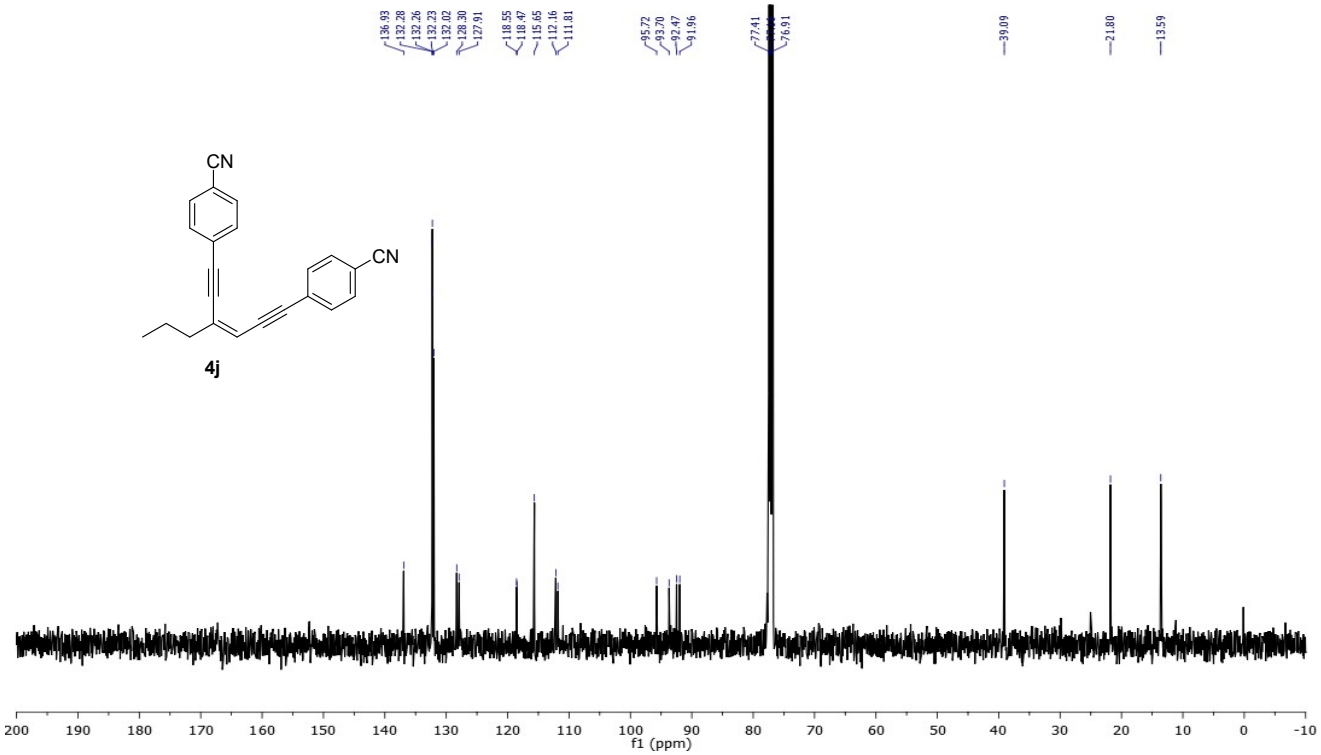
^{13}C{^{1}H} NMR Spectrum of compound 4i (101 MHz, $CDCl_3$)



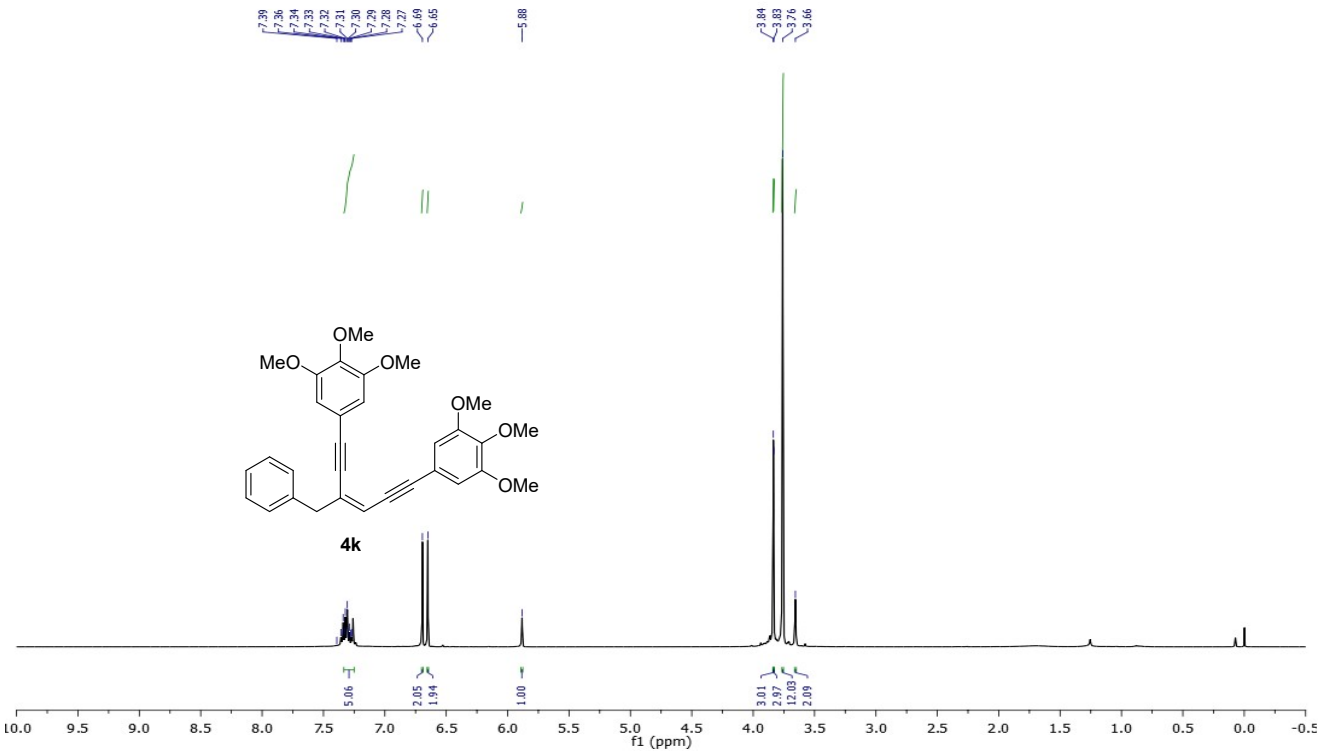
$^{19}F\{^{1}H\}$ NMR Spectrum of compound 4i (377 MHz, $CDCl_3$)



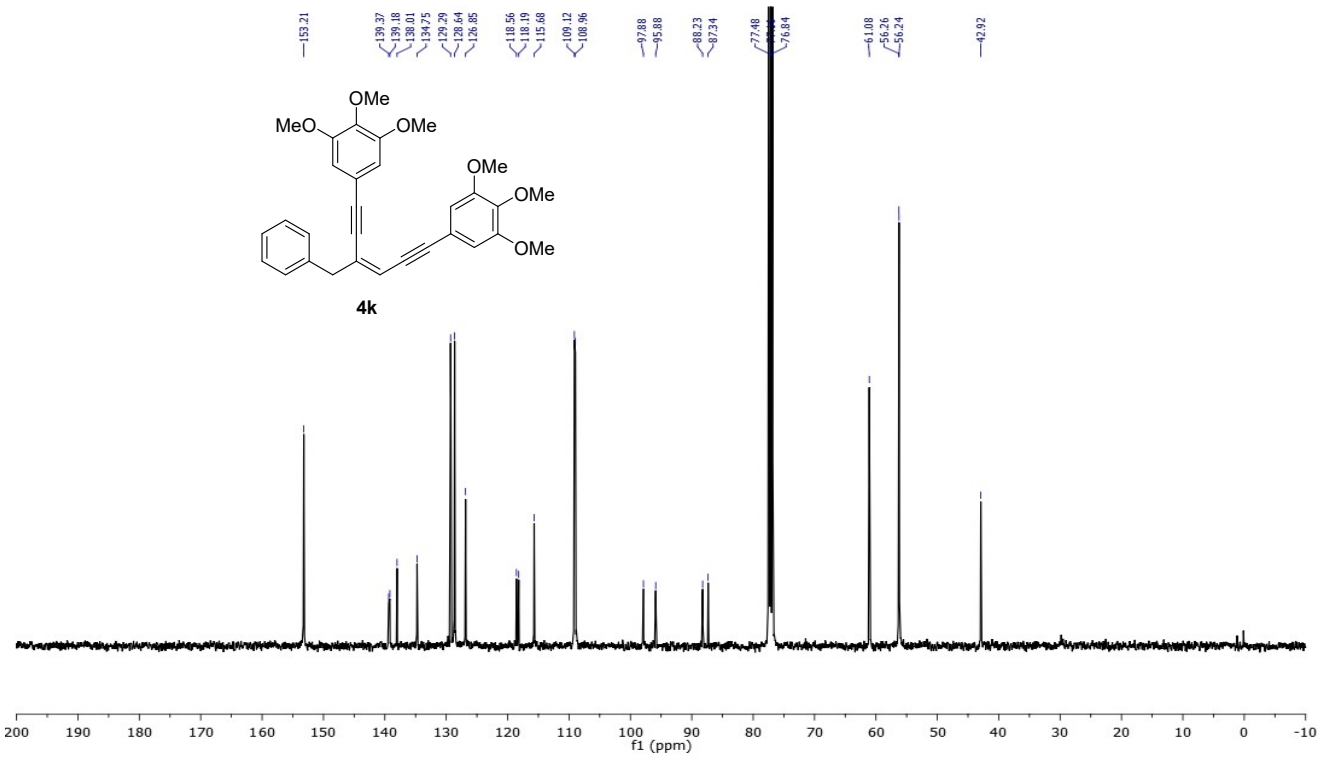
^{1}H NMR Spectrum of compound 4j (400 MHz, $CDCl_3$)



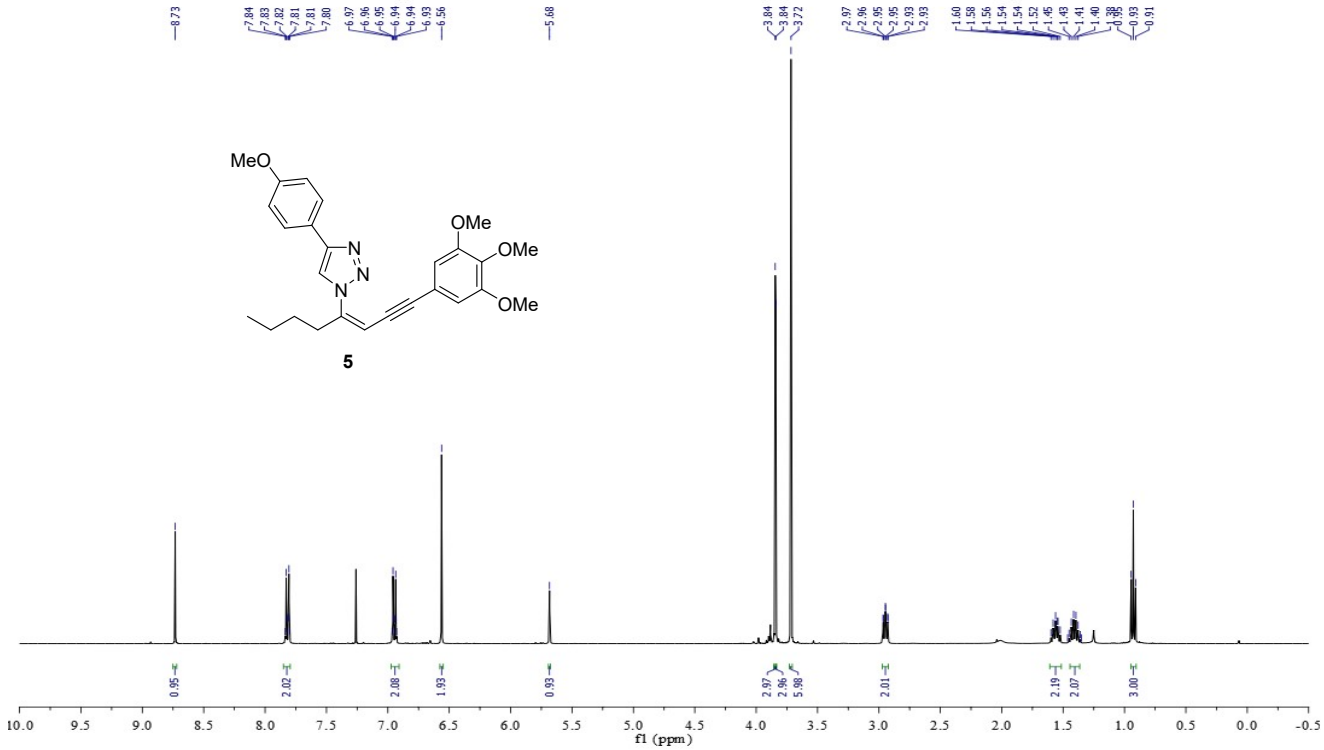
^{13}C{^{1}H} NMR Spectrum of compound 4j (126 MHz, $CDCl_3$)



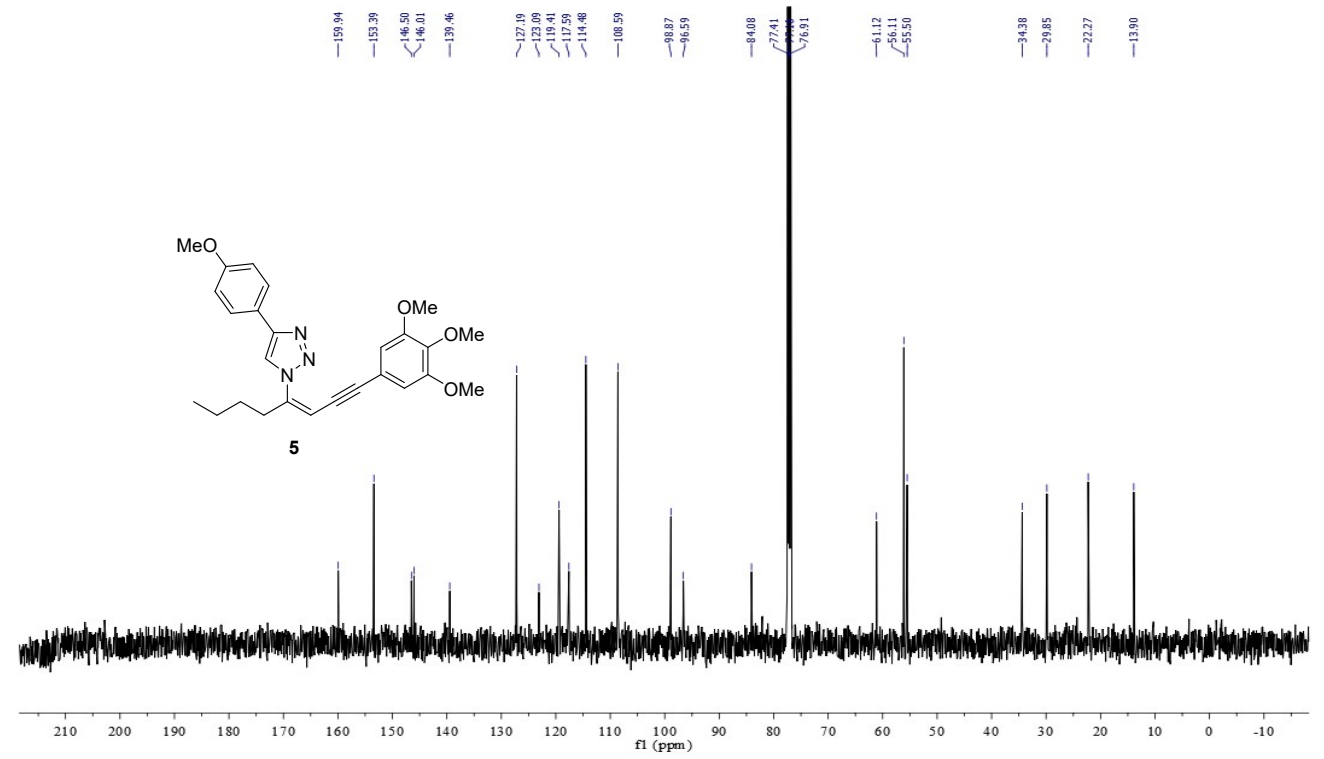
^{1}H NMR Spectrum of compound 4k (400 MHz, $CDCl_3$)



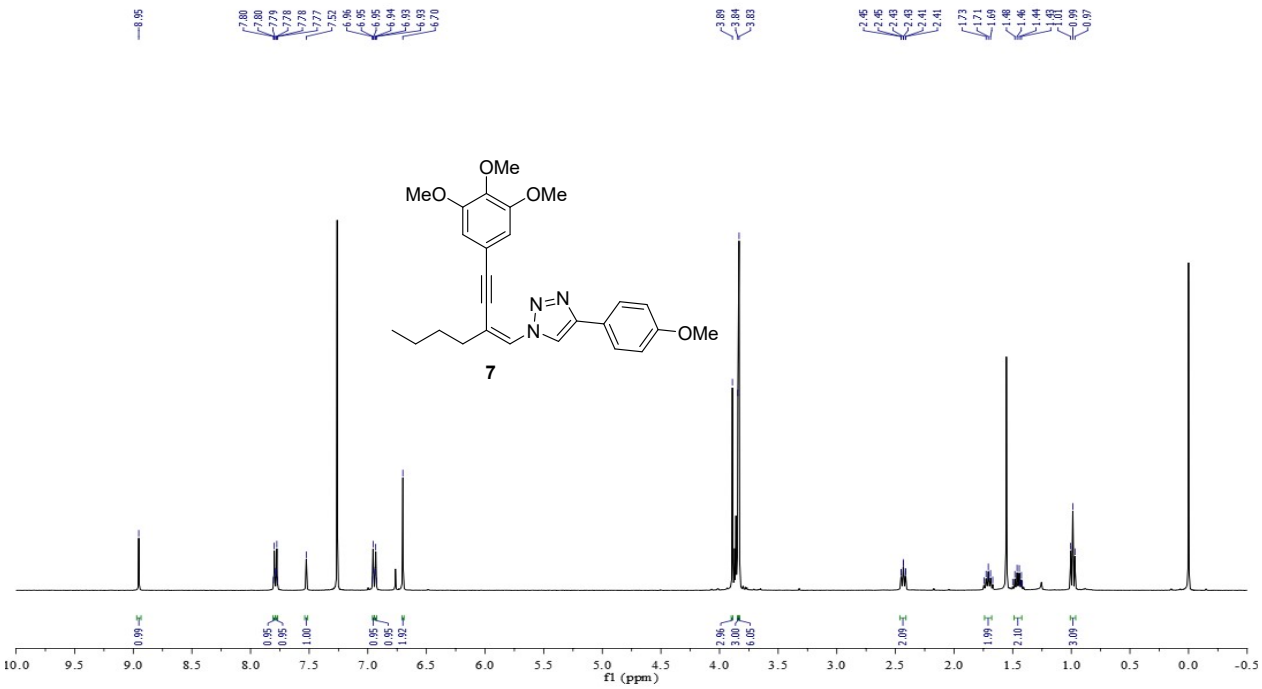
^{13}C{^{1}H} NMR Spectrum of compound 4k (101 MHz, $CDCl_3$)



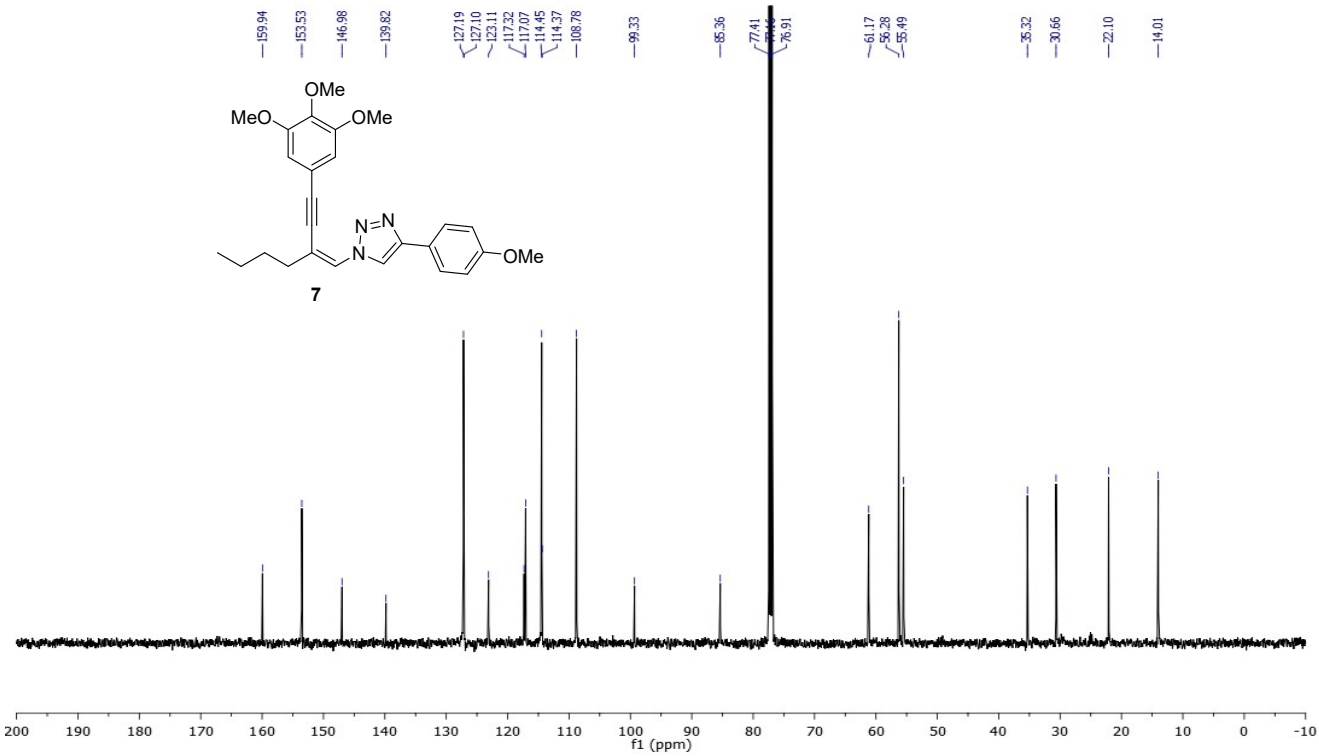
^{1}H NMR Spectrum of compound 5 (400 MHz, $CDCl_3$)



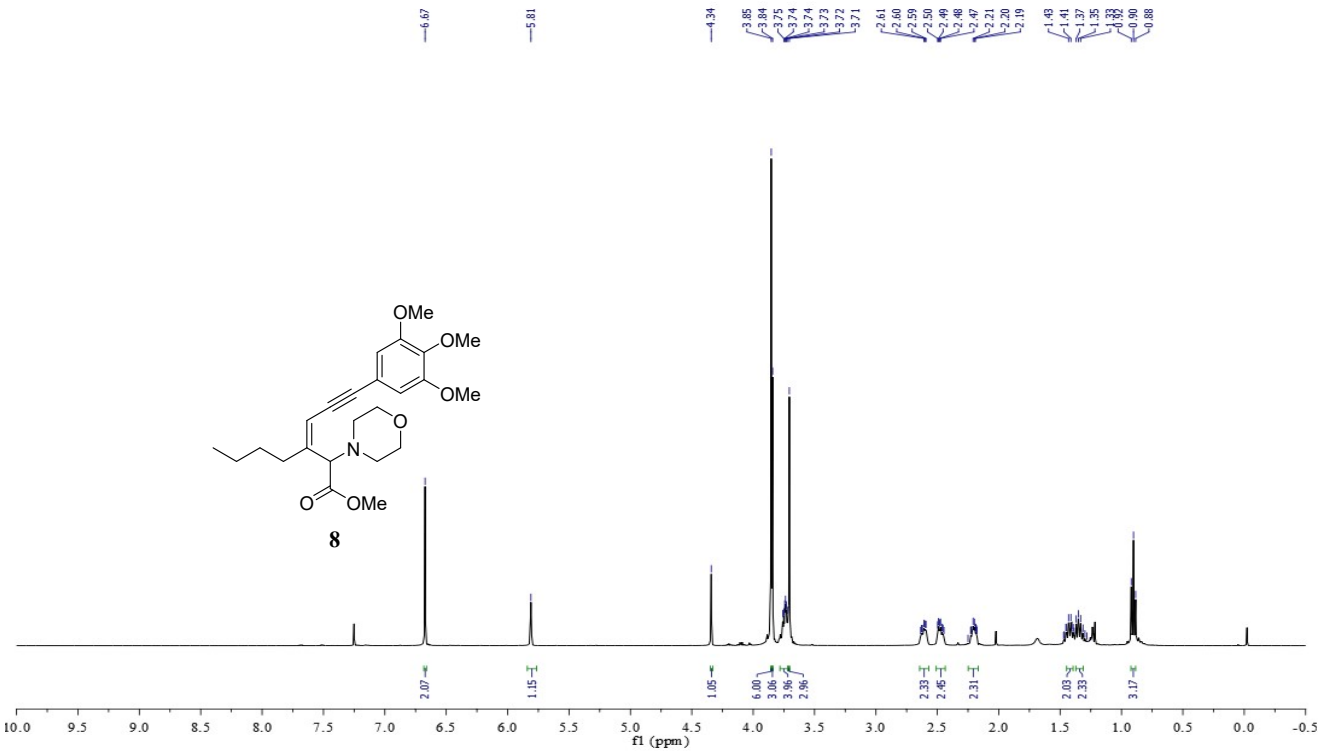
^{13}C{^{1}H} NMR Spectrum of compound 5 (126 MHz, $CDCl_3$)



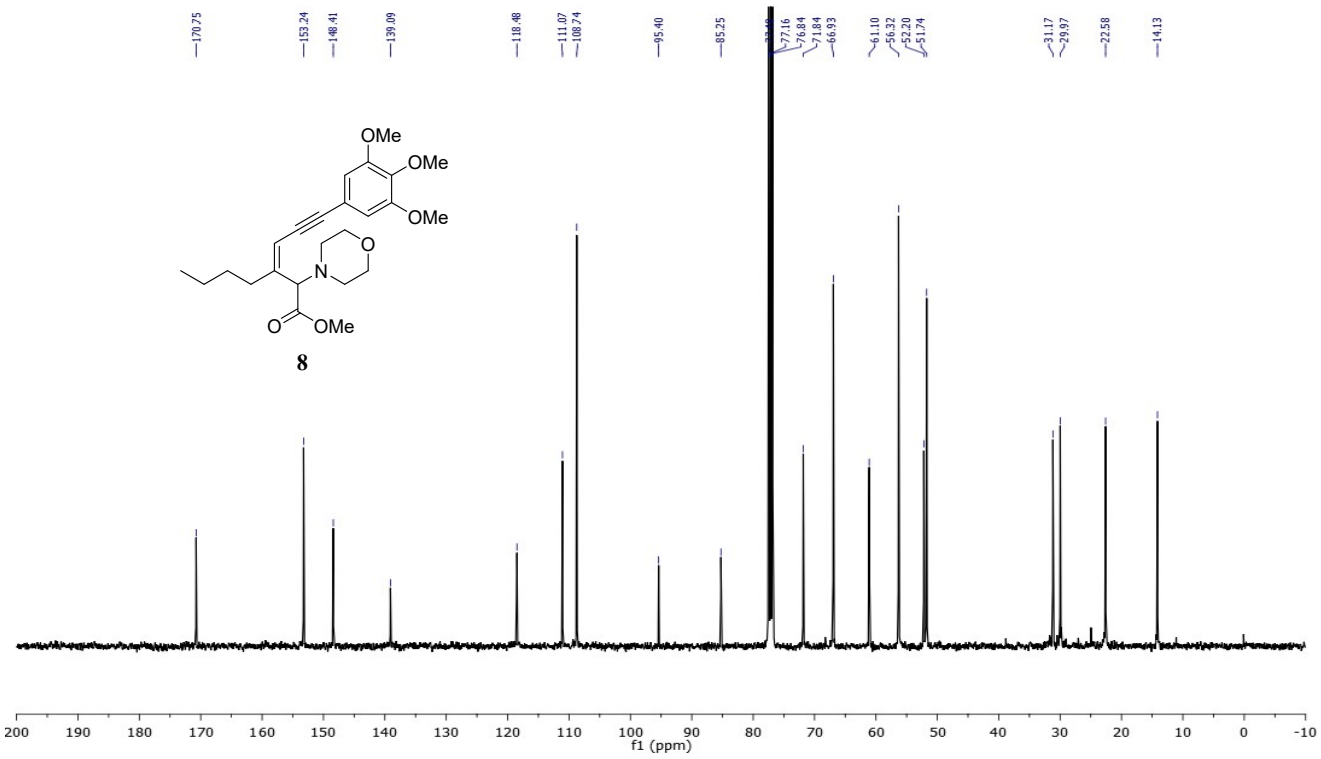
^{1}H NMR Spectrum of compound 7 (400 MHz, $CDCl_3$)



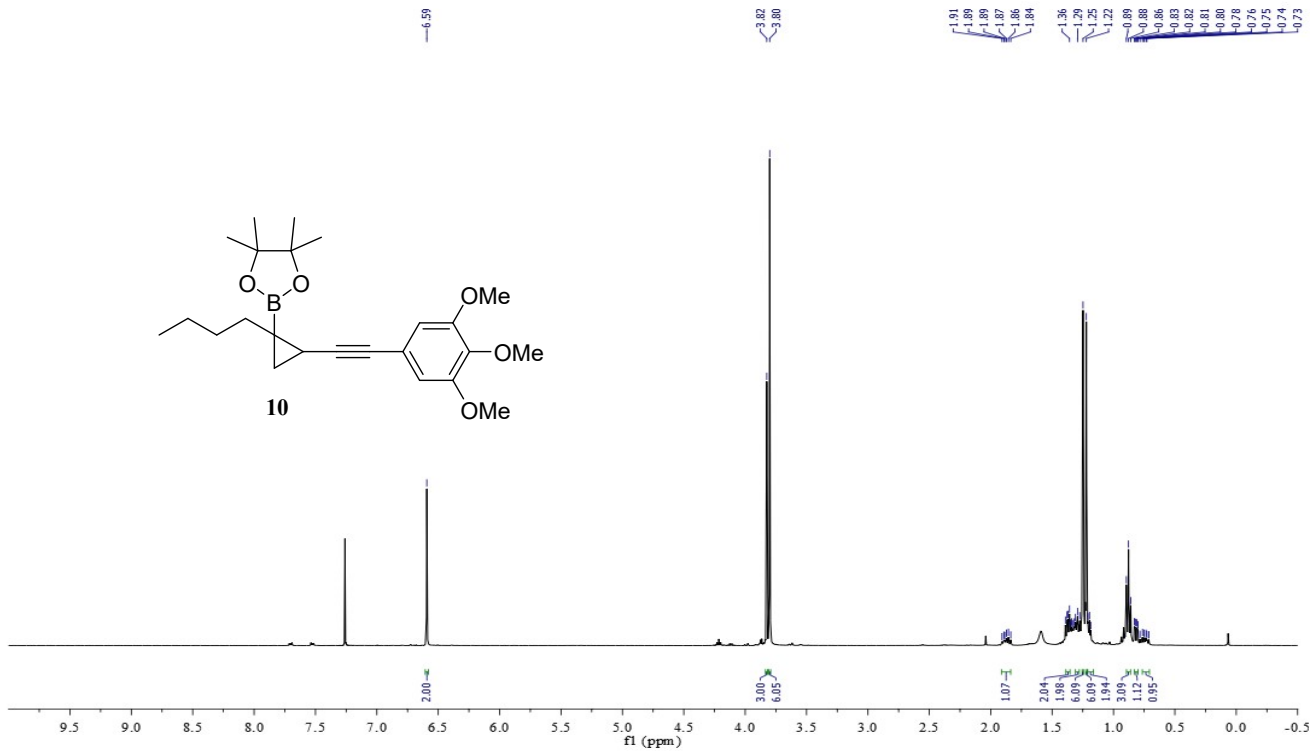
^{13}C{^{1}H} NMR Spectrum of compound 7 (126 MHz, $CDCl_3$)



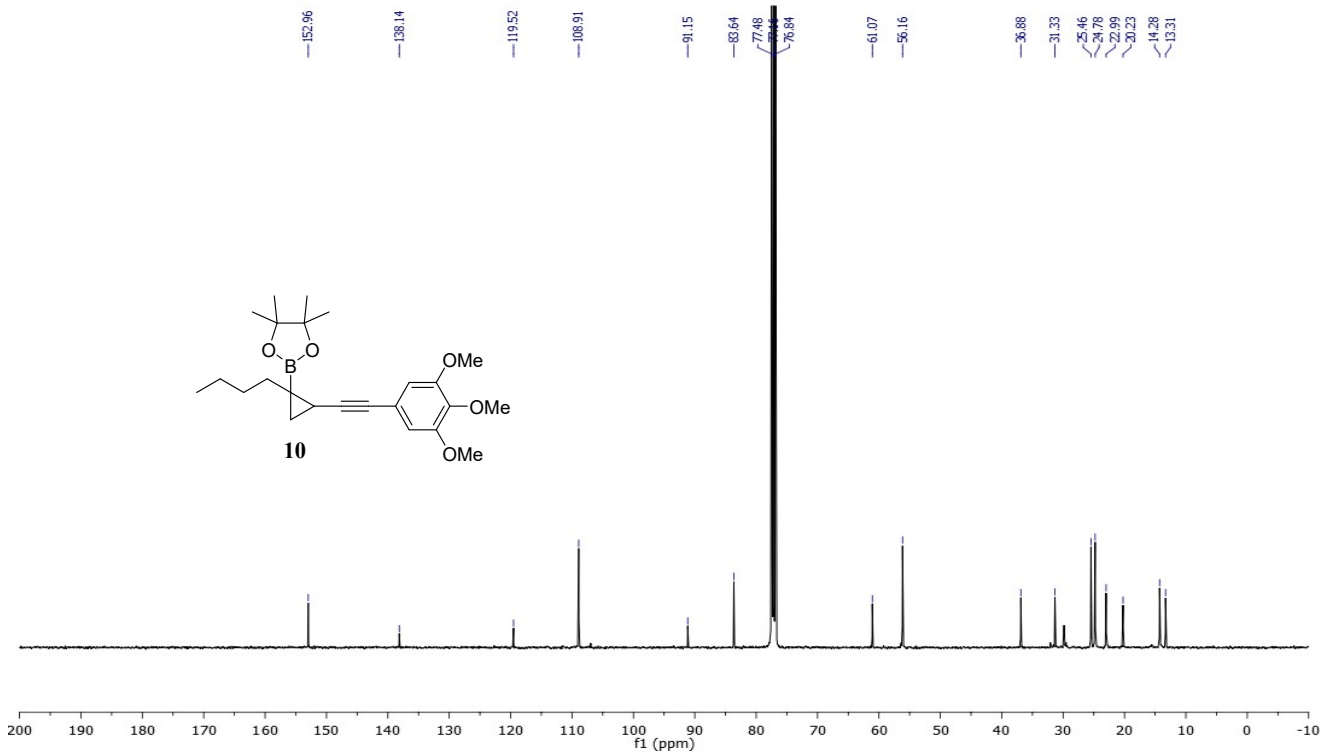
^{1}H NMR Spectrum of compound 8 (400 MHz, $CDCl_3$)



^{13}C{^{1}H} NMR Spectrum of compound 8 (101 MHz, $CDCl_3$)



^{1}H NMR Spectrum of compound 10 (400 MHz, $CDCl_3$)



^{13}C{^{1}H} NMR Spectrum of compound 10 (101 MHz, $CDCl_3$)