

Supporting Information

Pd-Catalyzed Homo-Coupling of Benzofurans: One-Pot Synthesis of Diverse 3,3'-Bisbenzofurans

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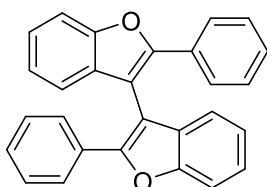
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1. General methods: Unless stated otherwise, solvents and chemicals were obtained from commercial sources and were used without further purification. Reactions were monitored by thin layer chromatography (TLC) on silica gel plates (60 F254), visualizing with ultraviolet light or iodine spray. Flash chromatography was performed on silica gel (100-200 mesh) using hexane and ethyl acetate. Melting points were recorded on a DBK digital melting point apparatus and were uncorrected. ^1H NMR and ^{13}C NMR spectra were recorded on a 500 or 400 or 125 or 100 Bruker Biospin A III FT-NMR spectrometer. ^1H and ^{13}C NMR spectra were determined in CDCl_3 solution by using 500 or 400 or 125 or 100 MHz spectrometers, respectively. Proton chemical shifts (δ) are relative to tetramethylsilane (TMS, $\delta = 0.00$) as internal standard and expressed in ppm. Spin multiplicities are given as s (singlet), d (doublet), dd (doublet of doublet), t (triplet) and m (multiplet). Coupling constants (J) are given in hertz. High-resolution mass spectra were recorded on a Bruker maxis-TOF mass spectrometer. All starting materials substituted 2-phenylbenzofuran were prepared according to the known literature procedure.¹

2. General procedure and characterisation of substituted 2,2'-diphenyl-3,3'-bibenzofuran (2)

An oven dried schlenk tube was charged with magnetic stirring bar, 2-substituted benzofuran (1) (0.50 mmol), in DMSO (2 mL), $\text{Pd}(\text{OAc})_2$ (10 mol %), $\text{Cu}(\text{OAc})_2$ (1.5 eq) were added and schlenk tube was closed with oxygen balloon. The resulting reaction mixture was heated at 90 °C for 24 h. After completion of the reaction, the mixture was cooled to room temperature, diluted with water (10 mL) and extracted with ethyl acetate (2×20 mL). The organic layers were collected, washed with brine solution and dried over Na_2SO_4 , filtered and concentrated under vacuum to give the crude, which was further purified by flash column chromatography, was performed on silica gel (100-200 mesh) using hexane and ethyl acetate as eluent to afford the desired product (2).

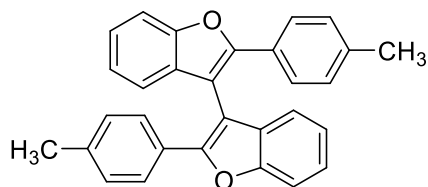
2,2'-Diphenyl-3,3'-bibenzofuran (2a)



White solid; Yield: 72 %; mp: 148-150°C (Lit²); $R_f = 0.4$ (100% *n*-Hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.77 – 7.74 (m, 3H), 7.62 (d, $J = 8.0$ Hz, 2H), 7.34-7.30 (m, 2H), 7.26 – 7.19 (m, 7H), 7.14 – 7.08 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.4 (2C), 151.9 (2C), 130.4

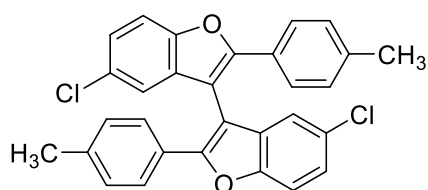
(2C), 129.5 (2C), 128.6 (4C), 128.5 (2C), 126.2 (4C), 124.9 (2C), 123.0 (2C), 120.7 (2C), 111.2 (2C), 107.6 (2C).

2,2'-Di-p-tolyl-3,3'-bibenzofuran (2b)



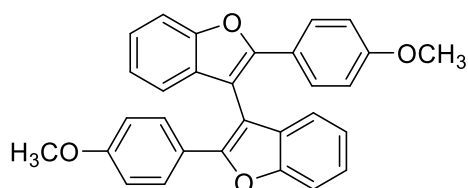
White solid; Yield: 70%; mp: 189 – 191 °C (Lit³ 190 – 192 °C); $R_f = 0.4$ (100% *n*-Hexane); **¹H NMR** (400 MHz, CDCl₃) δ 7.66 (d, $J = 8.0$ Hz, 4H), 7.60 (d, $J = 8.4$ Hz, 2H), 7.32 – 7.28 (m, 2H), 7.12 – 7.03 (m, 8H), 2.27 (s, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 154.2 (2C), 152.2 (2C), 138.5 (2C), 129.7 (4C), 129.3 (2C), 127.79 (2C), 126.1(4C), 124.6 (2C), 122.9 (2C), 120.6 (2C), 111.1 (2C), 107.0 (2C), 21.3 (2C).

5,5'-Dichloro-2,2'-di-p-tolyl-3,3'-bibenzofuran (2c)



White solid; Yield: 68%; mp: 185 - 187 °C $R_f = 0.4$ (100% *n*-Hexane); **¹H NMR** (400 MHz, CDCl₃) δ 7.61 (d, $J = 8.0$ Hz, 4H), 7.53 (d, $J = 8.4$ Hz, 2H), 7.27 - 7.26 (m, 2H), 7.07 (d, $J = 8.4$ Hz, 6H), 2.29 (s, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 158.9 (2C), 153.4 (2C), 152.6 (2C), 139.3 (2C), 131.0 (2C), 129.5 (4C), 128.8 (2C), 127.0 (2C), 126.1 (4C), 125.0 (2C), 119.7 (2C), 112.2 (2C), 21.3 (2C); **HR-MS** (ESI+) m/z calculated for [C₃₀H₂₁Cl₂O₂]⁺ = [M + H]⁺ 483.0913, found 483.0913.

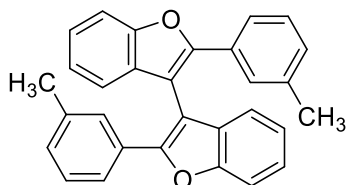
2,2'-Bis(4-methoxyphenyl)-3,3'-bibenzofuran (2d)



White solid; Yield: 68%; mp: 177 - 179 °C (Lit³ 178 – 180 °C); $R_f = 0.4$ (5% EtOAc/*n*-Hexane); **¹H NMR** (500 MHz, CDCl₃) δ 7.70 (d, $J = 8.0$ Hz, 4H), 7.59 (d, $J = 8.0$ Hz, 2H),

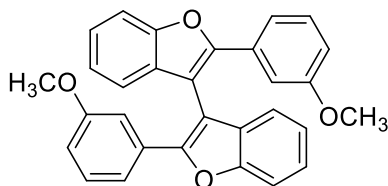
7.29 – 7.24 (m, 2H), 7.09 (d, $J = 8.0$ Hz, 4H), 6.77 (d, $J = 8.0$ Hz, 4H), 3.74 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.8 (2C), 154.2 (2C), 152.1 (2C), 129.8 (2C), 127.7 (4C), 124.4 (2C), 123.3 (2C), 122.9 (2C), 120.5 (2C), 114.1 (4C), 111.0 (2C), 106.1 (2C), 55.2 (2C).

2,2'-Di-*m*-tolyl-3,3'-bibenzofuran (2e)



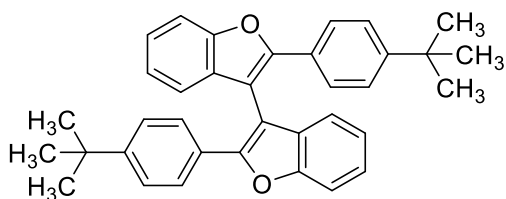
White solid; Yield: 69%; mp: 182 - 184 °C (Lit⁴); $R_f = 0.4$ (100% *n*-Hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.65 (s, 2H), 7.62 (d, $J = 8.4$ Hz, 2H), 7.48 (d, $J = 7.6$ Hz, 2H), 7.33 – 7.29 (m, 2H), 7.14 – 7.02 (m, 8H), 2.24 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.3 (2C), 152.1 (2C), 138.1 (2C), 130.4 (2C), 129.5 (2C), 129.3 (2C), 128.5 (2C), 126.8 (2C), 124.8 (2C), 123.6 (2C), 122.9 (2C), 120.8 (2C), 111.1 (2C), 107.7 (2C), 21.4 (2C).

2,2'-Bis(3-methoxyphenyl)-3,3'-bibenzofuran (2f)



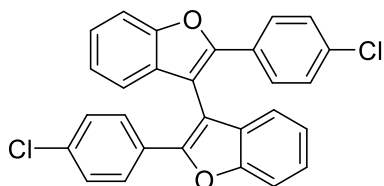
White solid; Yield: 68%; mp: 104 - 106 °C; $R_f = 0.4$ (5% EtOAc/*n*-Hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.62 (d, $J = 8.0$ Hz, 2H), 7.37 – 7.25 (m, 6H), 7.17 – 7.10 (m, 6H), 6.78 (dd, $J = 8.4, 2.0$ Hz, 2H), 3.52 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.5 (2C), 154.3 (2C), 151.8 (2C), 131.5 (2C), 129.6 (2C), 129.4 (2C), 125.1 (2C), 123.1 (2C), 120.8 (2C), 118.8 (2C), 115.2 (2C), 111.2 (2C), 110.8 (2C), 107.9 (2C), 54.9 (2C); HR-MS (ESI+) m/z calculated for $[\text{C}_{30}\text{H}_{23}\text{O}_4]^+ = [\text{M} + \text{H}]^+ 447.1591$, found 447.1591.

2,2'-Bis(4-(*tert*-butyl)phenyl)-3,3'-bibenzofuran (2g)



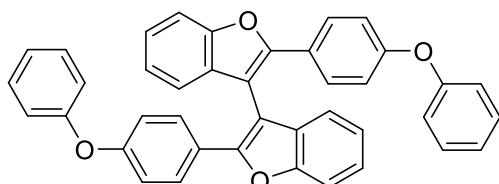
White solid; Yield: 65%; mp: 212 - 214 °C (Lit⁴); $R_f = 0.4$ (100% *n*-Hexane); ¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, $J = 8.8$ Hz, 4H), 7.62 (d, $J = 8.4$ Hz, 2H), 7.34 – 7.30 (m, 2H), 7.26 – 7.24 (m, 4H), 7.17 – 7.09 (m, 4H), 1.24 (s, 18H); ¹³C NMR (100 MHz, CDCl₃) δ 154.2 (2C), 152.2 (2C), 151.5 (2C), 129.9 (2C), 127.6 (2C), 125.9 (4C), 125.5 (4C), 124.6 (2C), 122.9 (2C), 120.5 (2C), 111.1 (2C), 107.0 (2C), 34.6 (2C), 31.1 (6C).

2,2'-Bis(4-chlorophenyl)-3,3'-bibenzofuran (2h)



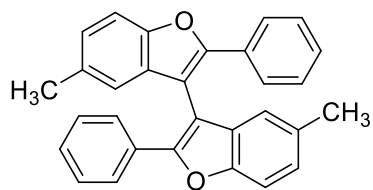
White solid; Yield: 68%; mp: 240 - 242 °C (Lit⁴); $R_f = 0.4$ (100% *n*-Hexane); ¹H NMR (400 MHz, CDCl₃) δ 7.67 – 7.61 (m, 6H), 7.37 – 7.33 (m, 2H), 7.21 (d, $J = 8.4$ Hz, 4H), 7.13 (d, $J = 2.4$ Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 154.4 (2C), 150.9 (2C), 134.5 (2C), 129.1 (2C), 128.9 (4C), 128.8 (2C), 127.4 (4C), 125.4 (2C), 123.3 (2C), 120.7 (2C), 111.4 (2C), 107.8 (2C).

2,2'-Bis(4-phenoxyphenyl)-3,3'-bibenzofuran (2i)



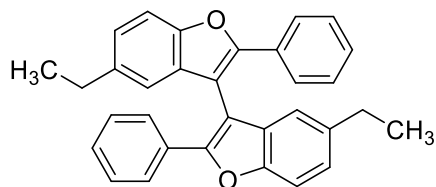
White solid; Yield: 68%; mp: 152 - 153 °C; $R_f = 0.4$ (100% *n*-Hexane); ¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, $J = 8.8$ Hz, 4H), 7.59 (d, $J = 8.0$ Hz, 2H), 7.33 – 7.28 (m, 6H), 7.18 – 7.07 (m, 6H), 6.96 (d, $J = 7.6$ Hz, 4H), 6.85 (d, $J = 8.8$ Hz, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 157.8 (2C), 156.4 (2C), 154.3 (2C), 151.6 (2C), 129.8 (4C), 129.6 (2C), 127.8 (4C), 125.4 (2C), 124.7 (2C), 123.8 (2C), 123.0 (2C), 120.6 (2C), 119.4 (4C), 118.4 (4C), 111.2 (2C), 106.7 (2C); HR-MS (ESI+) m/z calculated for [C₄₀H₂₇O₄]⁺ = [M + H]⁺ 571.1904, found 571.1902.

5,5'-Dimethyl-2,2'-diphenyl-3,3'-bibenzofuran (2j)



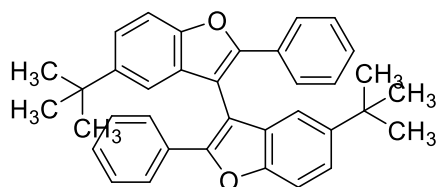
White solid; Yield: 70%; mp: 156 - 158 °C; $R_f = 0.4$ (100% *n*-Hexane); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.74 (dd, $J = 8.0, 1.6$ Hz, 4H), 7.50 (d, $J = 8.0$ Hz, 2H), 7.25 – 7.19 (m, 6H), 7.14 (dd, $J = 8.0, 0.4$ Hz, 2H), 6.95 (s, 2H), 2.29 (s, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 152.7 (2C), 152.0 (2C), 132.6 (2C), 130.6 (2C), 130.0 (2C), 128.6 (4C), 128.3 (2C), 126.3 (2C), 126.0 (4C), 120.3 (2C), 110.7 (2C), 107.6 (2C), 21.3 (2C); **HR-MS** (ESI+) m/z calculated for $[\text{C}_{30}\text{H}_{23}\text{O}_2]^+ = [\text{M} + \text{H}]^+ 415.1693$, found 415.1691.

5,5'-Diethyl-2,2'-diphenyl-3,3'-bibenzofuran (2k)



Off white solid; Yield: 68%; mp: 128 - 130 °C; $R_f = 0.4$ (100% *n*-Hexane); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.75 – 7.72 (m, 4H), 7.52 (d, $J = 8.0$ Hz, 2H), 7.24 – 7.14 (m, 8H), 6.94 (d, $J = 0.8$ Hz, 2H), 2.57 (q, $J = 7.6$ Hz, 4H), 1.09 (t, $J = 7.6$ Hz, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 152.9 (2C), 152.0 (2C), 139.1 (2C), 130.7 (2C), 129.6 (2C), 128.5 (4C), 128.3 (2C), 126.2 (4C), 125.1 (2C), 119.4 (2C), 110.7 (2C), 107.7 (2C), 28.8 (2C), 16.2 (2C); **HR-MS** (ESI+) m/z calculated for $[\text{C}_{32}\text{H}_{27}\text{O}_2]^+ = [\text{M} + \text{H}]^+ 443.2006$, found 443.2005.

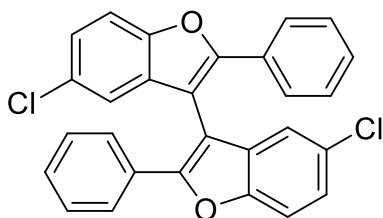
5,5'-Di-tert-butyl-2,2'-diphenyl-3,3'-bibenzofuran (2l)



Yellow solid; Yield: 62%; mp: 153 - 155 °C (Lit³ 155 - 157 °C); $R_f = 0.4$ (100% *n*-Hexane); $^1\text{H NMR}$ (400 MHz, $\text{CDCl}_3 + \text{DMSO-}d_6$) δ 7.67 (dd, $J = 8.4, 1.6$ Hz, 4H), 7.45 (d, $J = 8.8$ Hz, 2H), 7.27 (dd, $J = 8.8, 1.6$ Hz, 2H), 7.18 – 7.15 (m, 6H), 6.93 (d, $J = 2.0$ Hz, 2H), 1.05 (s, 18H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 152.7 (2C), 152.2 (2C), 145.8 (2C), 130.9 (2C), 128.4

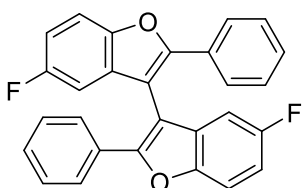
(4C), 128.3 (2C), 128.2 (2C), 126.7 (4C), 122.5 (2C), 117.7 (2C), 110.3 (2C), 108.1 (2C), 34.5 (2C), 31.6 (6C).

5,5'-Dichloro-2,2'-diphenyl-3,3'-bibenzofuran (2m)



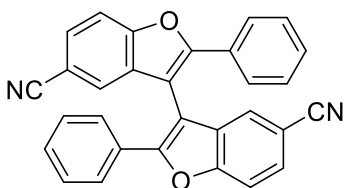
White solid; Yield: 67%; mp: 225 - 226 °C; $R_f = 0.4$ (100% *n*-Hexane); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.72 (s, 4H), 7.54 (d, $J = 8.4$ Hz, 2H), 7.29 – 7.26 (m, 8H), 7.09 (s, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 153.7 (2C), 152.7 (2C), 130.8 (2C), 129.7 (2C), 129.2 (2C), 128.9 (2C), 128.8 (4C), 126.2 (4C), 125.4 (2C), 119.84 (2C), 112.4 (2C), 106.5 (2C); **HR-MS** (ESI+) m/z calculated for $[\text{C}_{28}\text{H}_{17}\text{Cl}_2\text{O}_2]^+ = [\text{M} + \text{H}]^+ 455.0600$, found 455.0602.

5,5'-Difluoro-2,2'-diphenyl-3,3'-bibenzofuran (2n)



White solid; Yield: 68%; mp: 198 - 200 °C (Lit⁴); $R_f = 0.4$ (100% *n*-Hexane); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.74 – 7.71 (m, 4H), 7.55 (dd, $J = 8.8, 4.0$ Hz, 2H), 7.27 – 7.25 (m, 6H), 7.06 – 7.01 (m, 2H), 6.76 (dd, $J = 8.4, 2.4$ Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 154.1 (2C), 151.8 (2C), 147.9 (2C), 147.8 (2C), 129.6 (2C), 124.6 (2C), 123.0 (2C), 120.8 (2C), 120.4 (2C), 111.1 (2C), 108.6 (2C), 106.6 (2C), 106.4 (2C), 101.2 (2C).

2,2'-Diphenyl-[3,3'-bibenzofuran]-5,5'-dicarbonitrile (2o)



Off white solid; Yield: 58%; mp: 282 - 284 °C; $R_f = 0.4$ (10% EtOAc/*n*-Hexane); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.74 – 7.71 (m, 6H), 7.63 (dd, $J = 8.4, 1.6$ Hz, 2H), 7.37 – 7.28 (m, 8H);

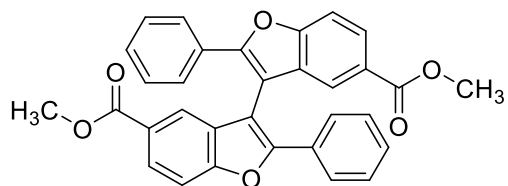
^{13}C NMR (100 MHz, CDCl_3) δ 156.0 (2C), 154.9 (2C), 130.0 (2C), 129.6 (2C), 129.0 (4C), 128.9 (2C), 128.8 (2C), 126.5 (4C), 125.1 (2C), 119.0 (2C), 112.8 (2C), 107.3 (2C), 105.9 (2C); **HR-MS** (ESI+) m/z calculated for $[\text{C}_{30}\text{H}_{16}\text{N}_2\text{O}_2\text{Na}]^+ = [\text{M} + \text{Na}]^+$ 459.1104, found 459.1103.

4,4'-([3,3'-Bibenzofuran]-2,2'-diyl)dibenzaldehyde (2q)

c

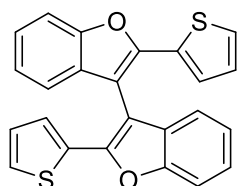
Yellow solid; Yield: 62%; mp: 218 - 220 °C; $R_f = 0.4$ (10% EtOAc/*n*-Hexane); ^1H NMR (400 MHz, CDCl_3) δ 9.92 (s, 2H), 7.90 (d, $J = 8.4$ Hz, 4H), 7.75 (d, $J = 8.4$ Hz, 4H), 7.68 (d, $J = 8.0$ Hz, 2H), 7.44 – 7.39 (m, 2H), 7.17 (d, $J = 4.0$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 191.4 (2C), 154.8 (2C), 150.4 (2C), 135.7 (2C), 135.6 (2C), 130.0 (4C), 128.8 (2C), 126.4 (4C), 126.3 (2C), 123.6 (2C), 120.9 (2C), 111.7 (2C), 110.1 (2C); **HR-MS** (ESI+) m/z calculated for $[\text{C}_{30}\text{H}_{18}\text{O}_4\text{Na}]^+ = [\text{M} + \text{Na}]^+$ 465.1097, found 465.1210.

Bimethyl 2,2'-diphenyl-[3,3'-bibenzofuran]-5,5'-dicarboxylate (2r)



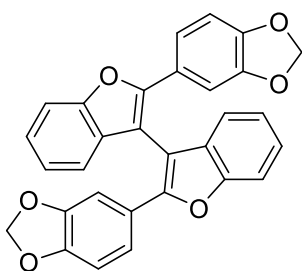
White solid; Yield: 60%; mp: 175 - 177 °C; $R_f = 0.5$ (10% EtOAc/*n*-Hexane); ^1H NMR (500 MHz, CDCl_3) δ 8.09 (dd, $J = 8.5, 1.5$ Hz, 2H), 7.84 (d, $J = 1.1$ Hz, 2H), 7.74 – 7.72 (m, 4H), 7.68 (d, $J = 8.5$ Hz, 2H), 7.25 – 7.24 (m, 6H), 3.81 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.9 (2C), 156.9 (2C), 153.7 (2C), 129.7 (2C), 129.7 (2C), 129.2 (2C), 128.8 (4C), 126.9 (2C), 126.2 (4C), 125.7 (2C), 122.6 (2C), 111.3 (2C), 107.3 (2C), 52.0 (2C); **HR-MS** (ESI+) m/z calculated for $[\text{C}_{32}\text{H}_{22}\text{O}_6\text{Na}]^+ = [\text{M} + \text{Na}]^+$ 525.1314, found 525.1292.

2,2'-Di(thiophen-2-yl)-3,3'-bibenzofuran (2s)



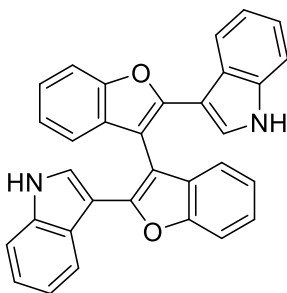
Yellow solid; Yield: 65%; mp: 108 -110 °C; $R_f = 0.4$ (100% *n*-Hexane); $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.62 (d, $J = 7.5$ Hz, 1H), 7.56-7.49 (m, 5H), 7.35 - 7.32 (m, 2H), 7.29 – 7.23 (m, 4H), 7.06 (t, $J = 4.25$ Hz, 1H), 6.91 (s, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 154.6, 153.7, 150.9, 148.0, 134.3, 132.9, 131.9, 129.9, 129.1 (2C), 127.6, 127.0, 126.8, 125.2, 125.1, 124.5, 123.5, 123.2, 120.8, 120.0, 111.1 (2C), 109.5, 101.4; **HR-MS** (ESI+) m/z calculated for $[\text{C}_{24}\text{H}_{15}\text{O}_2\text{S}_2]^+ = [\text{M} + \text{H}]^+ 399.0508$, found 399.0516.

2,2'-Bis(benzo[d][1,3]dioxol-5-yl)-3,3'-bibenzofuran (2t)



white solid; Yield: 63%; mp: 210 - 212 °C; $R_f = 0.3$ (5% EtOAc/*n*-Hexane); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.58 (d, $J = 8.0$ Hz, 2H), 7.31 – 7.25 (m, 6H), 7.23 (s, 2H), 7.12 (s, 2H), 6.68 (d, $J = 8.0$ Hz, 2H), 5.91 (s, 4H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 158.5 (2C), 153.9 (2C), 150.5 (2C), 129.9 (2C), 129.0 (2C), 128.7 (4C), 126.2 (4C), 112.9 (2C), 112.7 (2C), 112.1 (2C), 112.0 (2C), 106.1 (2C), 105.9 (2C); **HR-MS** (ESI+) m/z calculated for $[\text{C}_{30}\text{H}_{19}\text{O}_6]^+ = [\text{M} + \text{H}]^+ 475.1176$, found 475.1176.

2,2'-Di(1H-indol-3-yl)-3,3'-bibenzofuran (2u)



Brown solid; Yield: 66%; mp: 204 - 206 °C; $R_f = 0.4$ (20% EtOAc/*n*-Hexane); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.50 (d, $J = 6.8$ Hz, 2H), 7.86 (s, 2H), 7.69 (d, $J = 7.6$ Hz, 2H), 7.28 (s, 2H), 7.26 – 7.20 (m, 6H), 7.13 – 7.09 (m, 2H), 7.05 (s, 2H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 154.2 (2C), 151.3 (2C), 135.5 (2C), 129.3 (2C), 125.1 (2C), 124.7 (2C), 123.4 (2C), 122.9 (2C), 122.8 (2C), 121.9 (2C), 121.0 (2C), 120.1 (2C), 111.3 (2C), 111.0 (2C), 107.5 (2C), 104.3

(2C); **HR-MS** (ESI+) m/z calculated for $[C_{32}H_{21}N_2O_2]^+ = [M + H]^+$ 465.1598, found 465.1594.

3. X-ray Data: The quality single crystals suitable for SC-XRD experiments of all the three compounds were obtained from $CHCl_3:MeOH$ (50:50) solvent by the slow evaporation method. The single-crystal X-ray diffraction measurements were performed to determine the crystal structure of three compounds using APEX3 (Bruker, 2016; Bruker D8 Venture photon 100 CMOS detector) diffractometer having graphite-monochromatized ($MoK\alpha = 0.71073 \text{ \AA}$). The X-ray generator was operated at 50 kV and 30 mA. A preliminary set of unit cell parameters and an orientation matrix were calculated from 36 frames, and the cell refinement was performed by SAINT-Plus (Bruker, 2016). An optimized strategy used for data collection consisted of different sets of φ and ω scans with 0.5° steps φ/ω . The data were collected with a time frame of 10 sec for the three components by setting the sample to detector distance fixed at 40 cm. The data points were corrected for Lorentzian, polarization, and absorption effects using SAINT-Plus and SADABS programs (Bruker, 2016). SHELXS-97 (Sheldrick, 2018) was used for structure solution and full-matrix least-squares refinement on F^2 .⁵ The program(s) used to refine the molecular structures of **2i** are SHELXL 2018/3 (Sheldrick, 2018). All non-hydrogen atoms were refined by the anisotropic method and hydrogen atoms were either refined or placed in calculated positions. The molecular graphics of ORTEP diagrams were performed by XP software.

Single crystals suitable for X-ray diffraction of **2i** ethylacetate: hexene (1:1). Molecular formula = $C_{40}H_{26}O_4$, Molecular weight = 570.61, space group = Pna21, $a = 12.3643(4) \text{ \AA}$, $b = 7.5268(4) \text{ \AA}$, $c = 31.4217(12) \text{ \AA}$, $T = 297 \text{ K}$, $R(\text{reflections}) = 0.1349(2598)$ $wR2(\text{reflections}) = 0.4051(6198)$, $S = 1.154$. Crystallographic data for compound deposited with the Cambridge Crystallographic Data Centre as supplementary publication no CCDC 2240724.

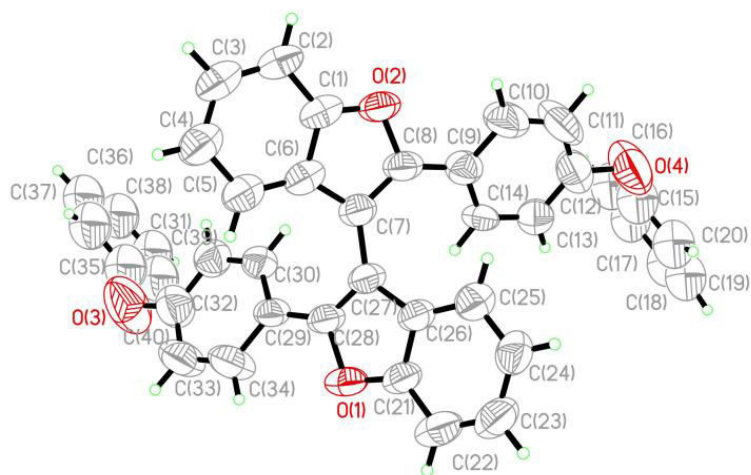
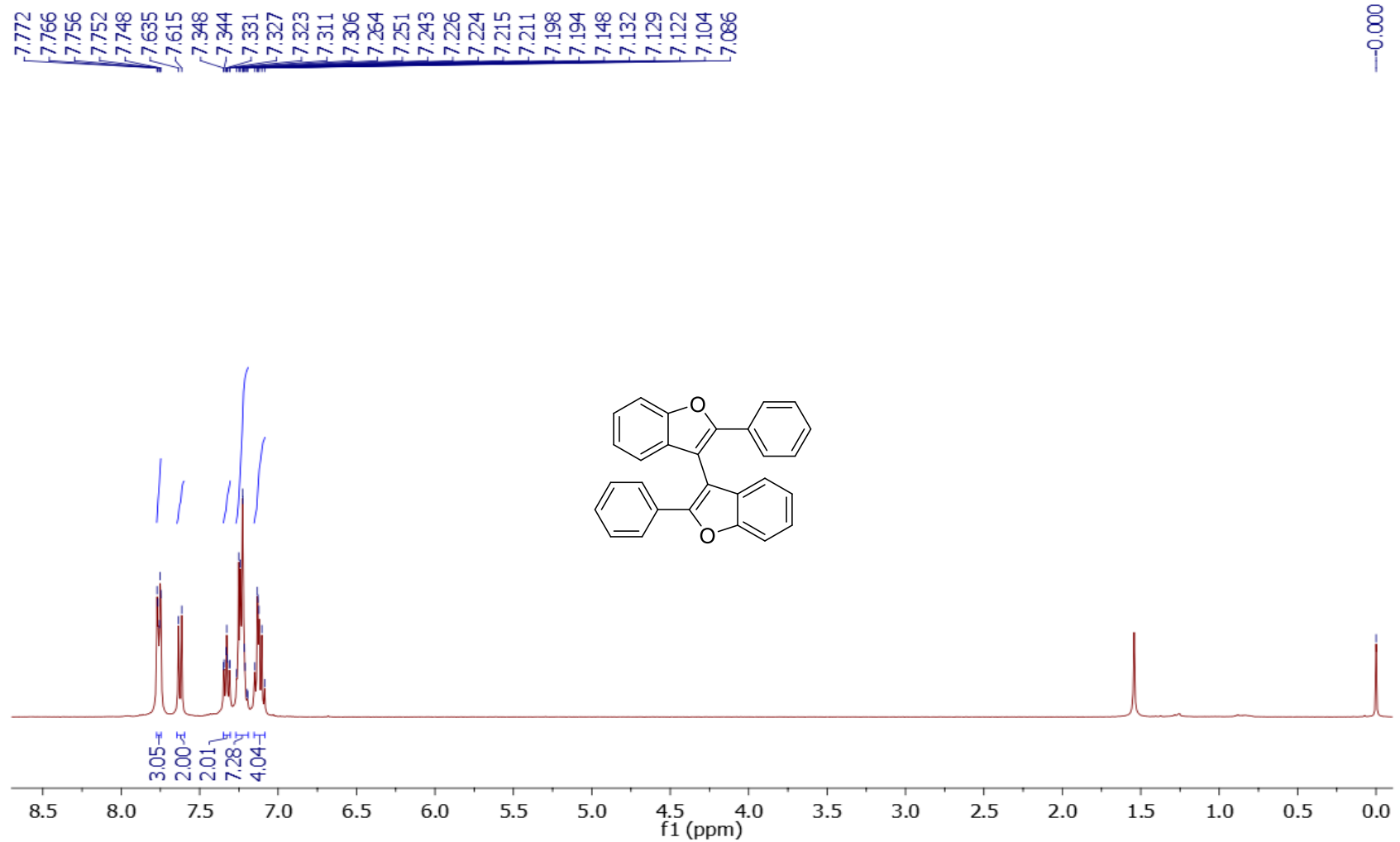


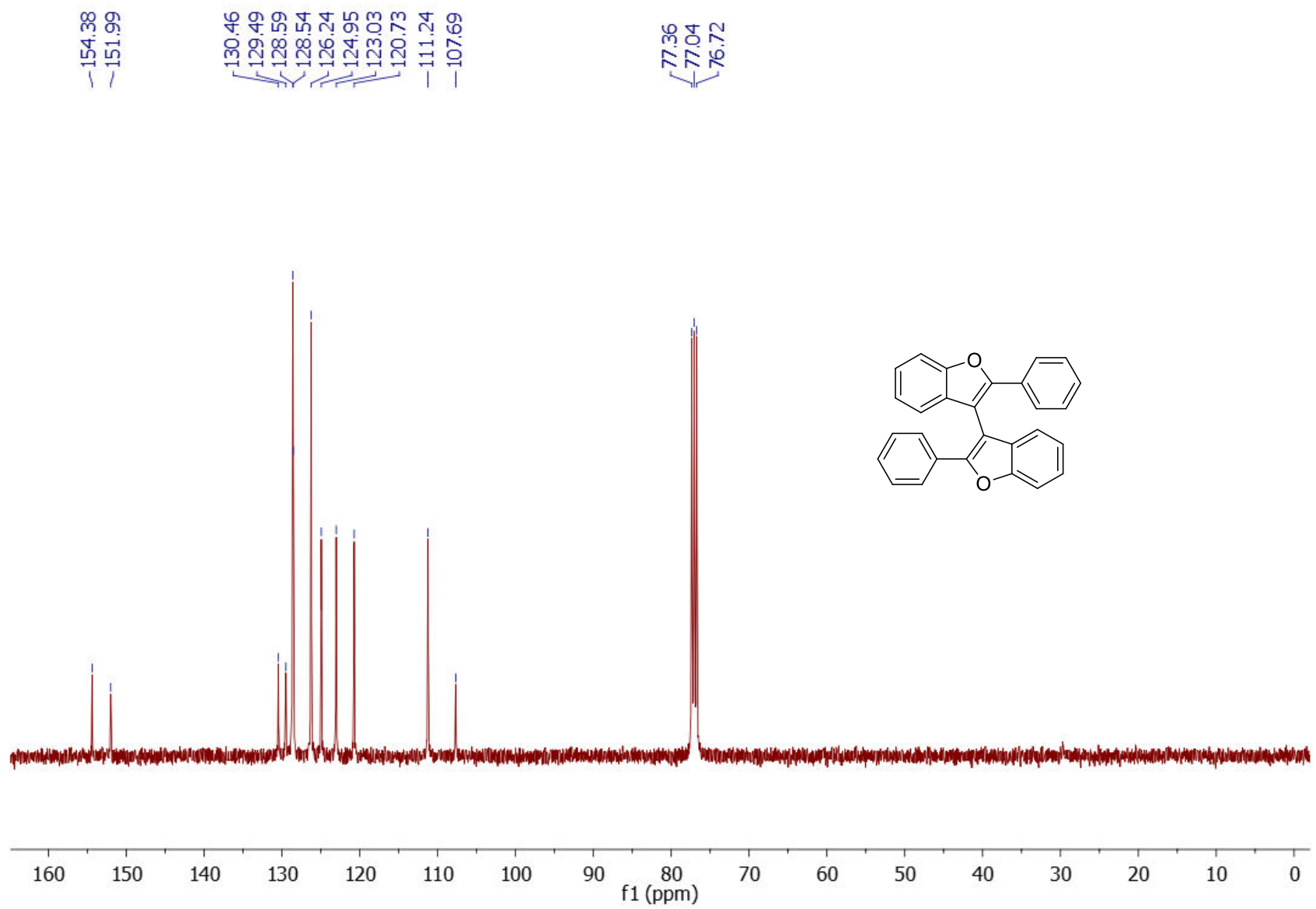
Figure S1. X-ray crystal structure of **2i** (ORTEP diagram). Thermal ellipsoids are drawn at the 50% probability level.

References

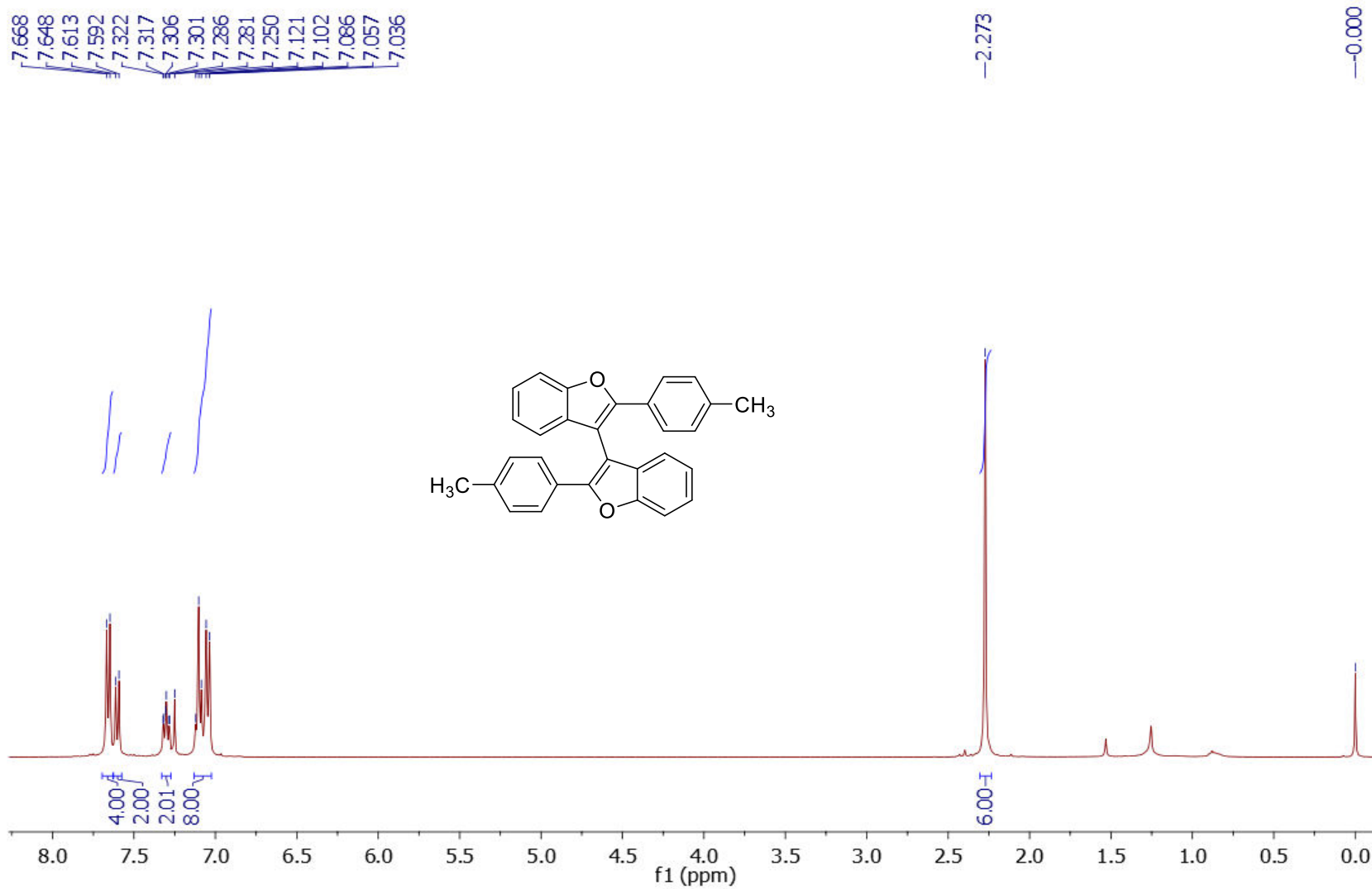
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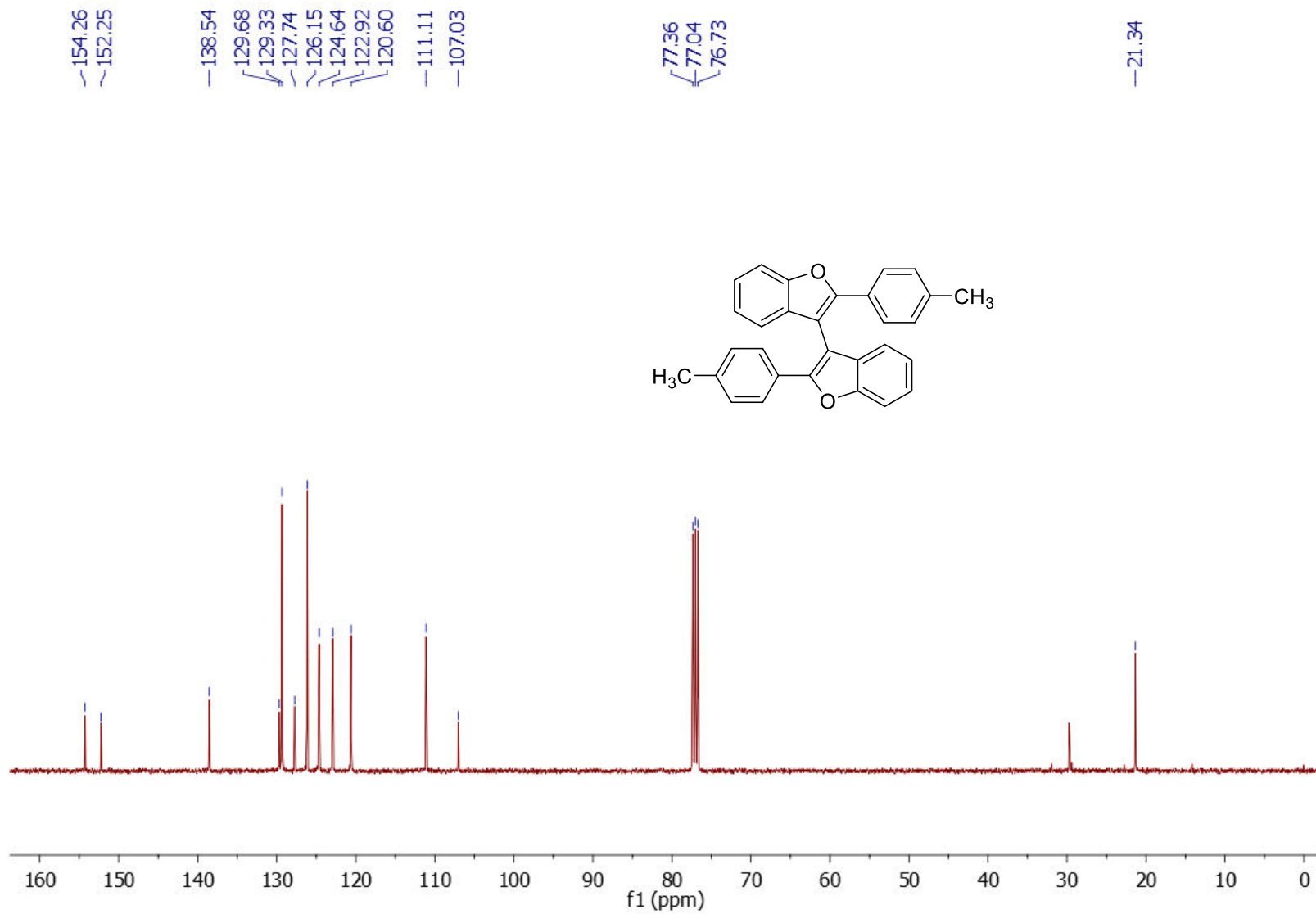
Copies of ^1H and ^{13}C NMR spectra of products
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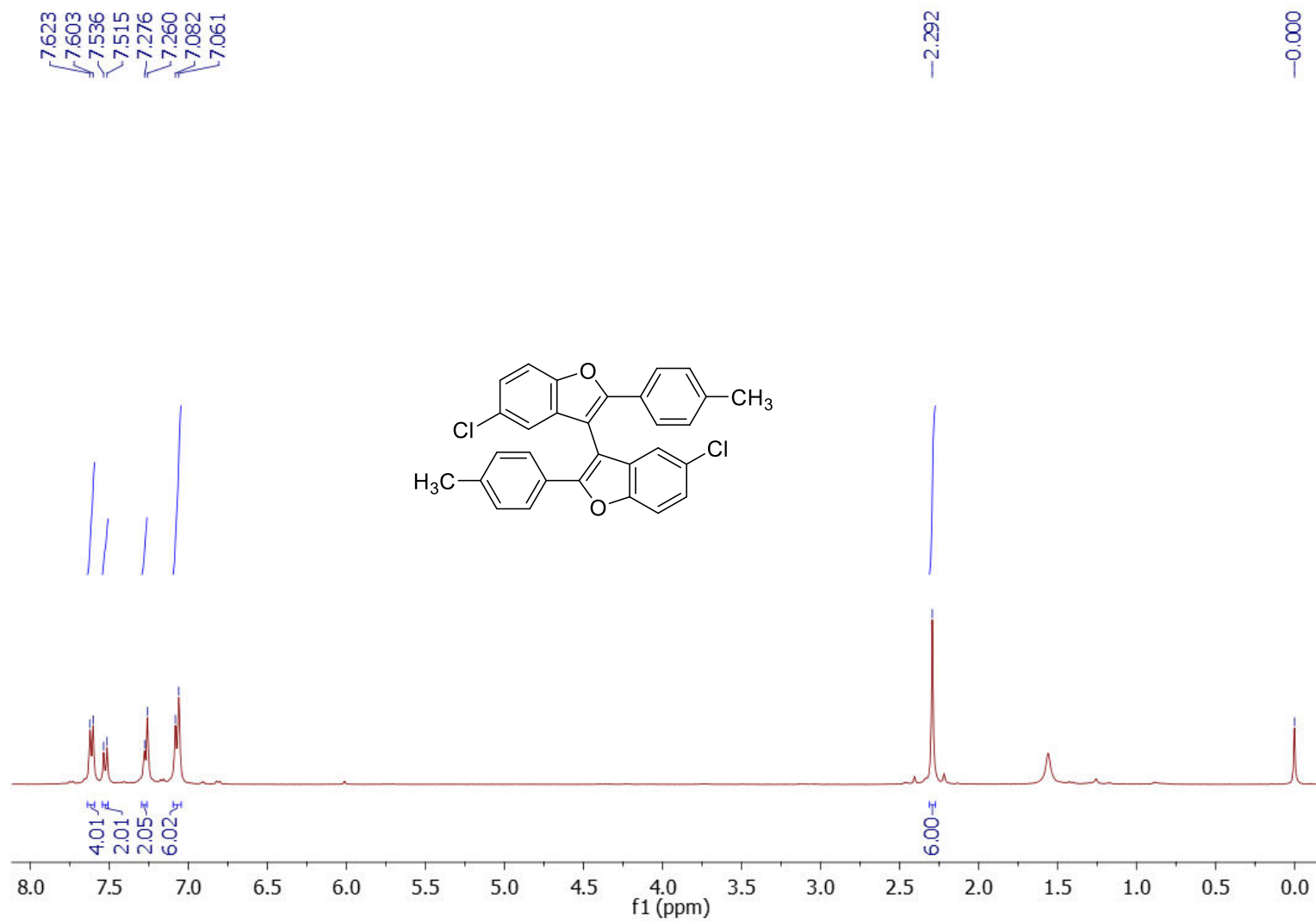


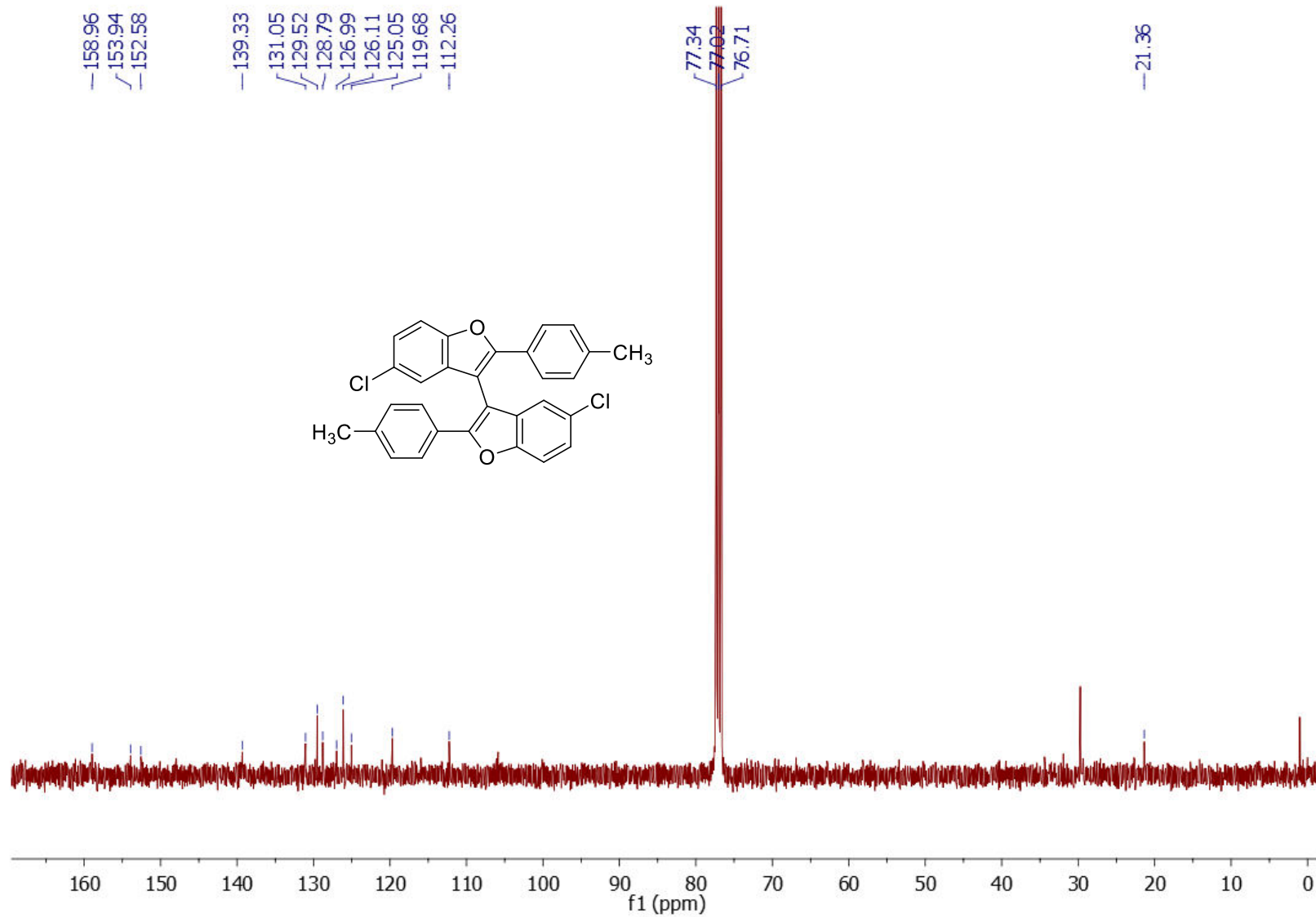
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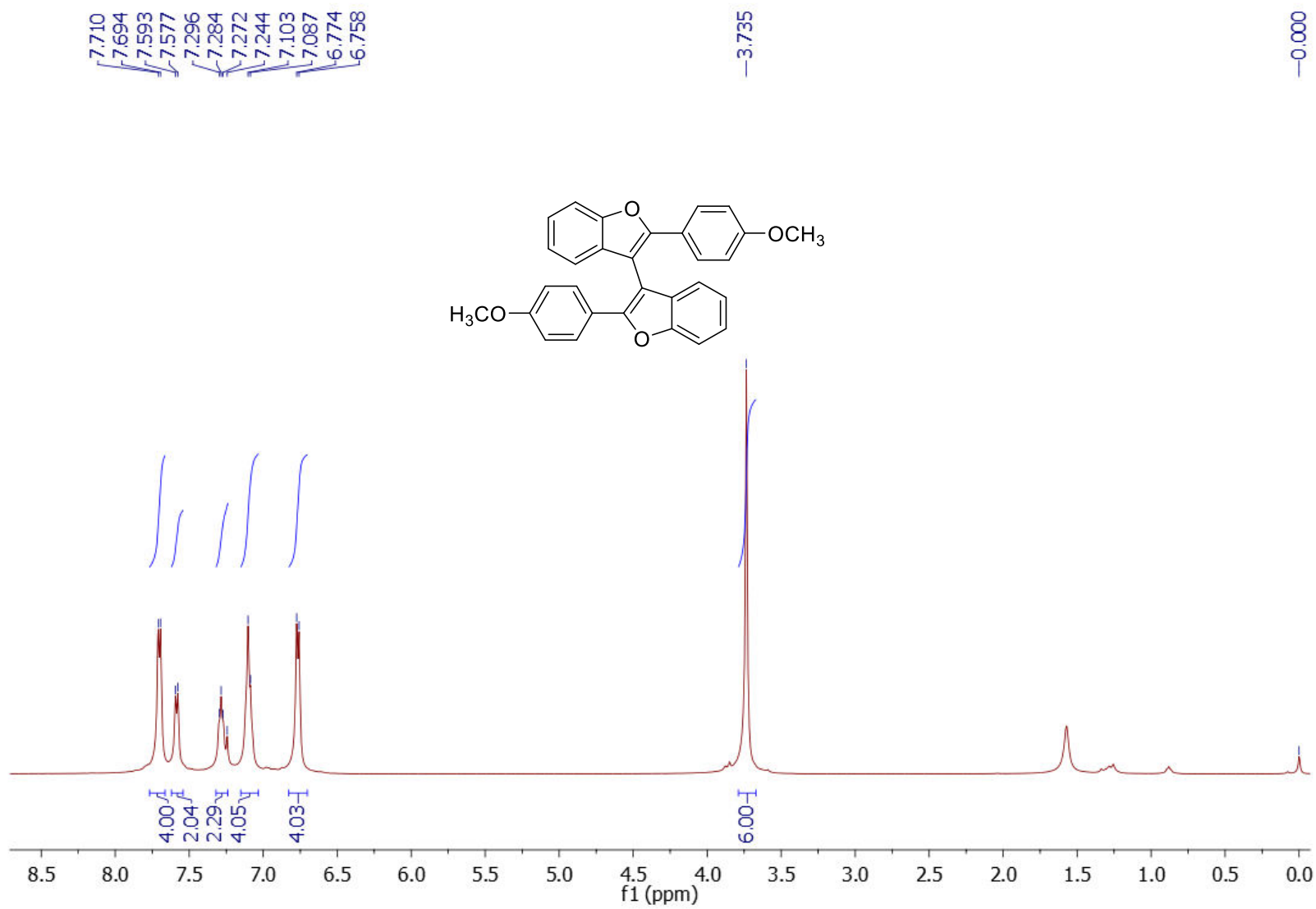


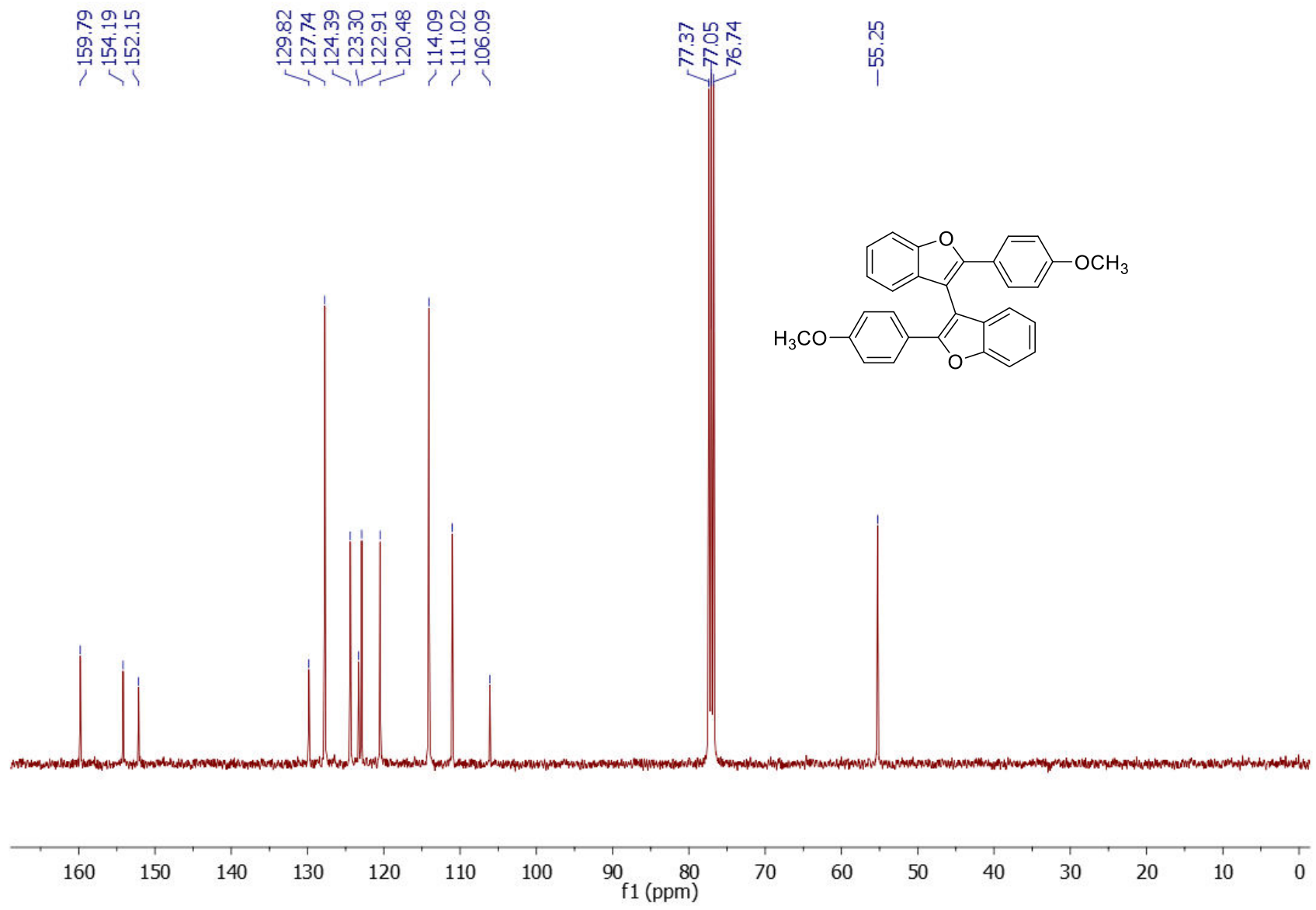
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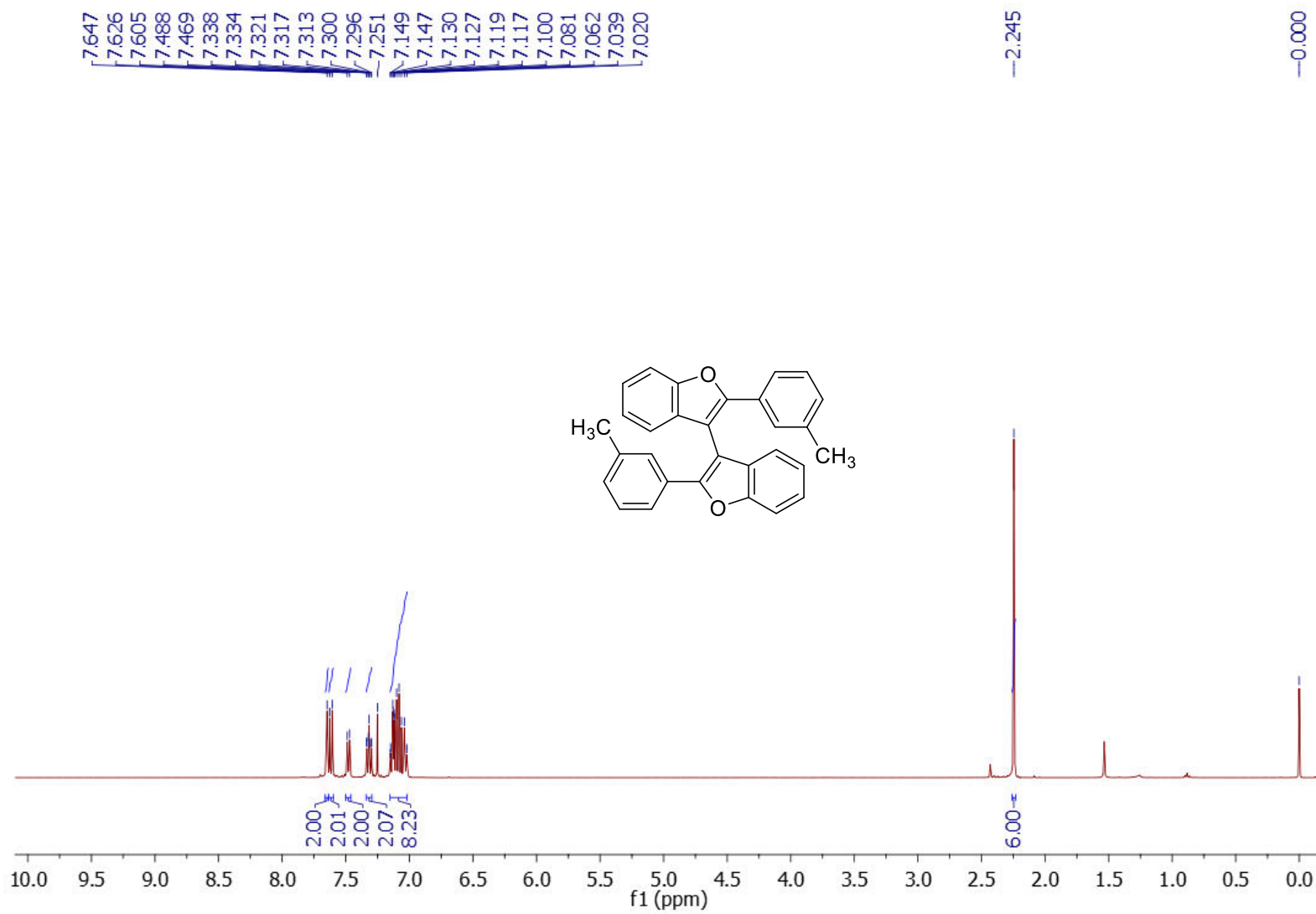


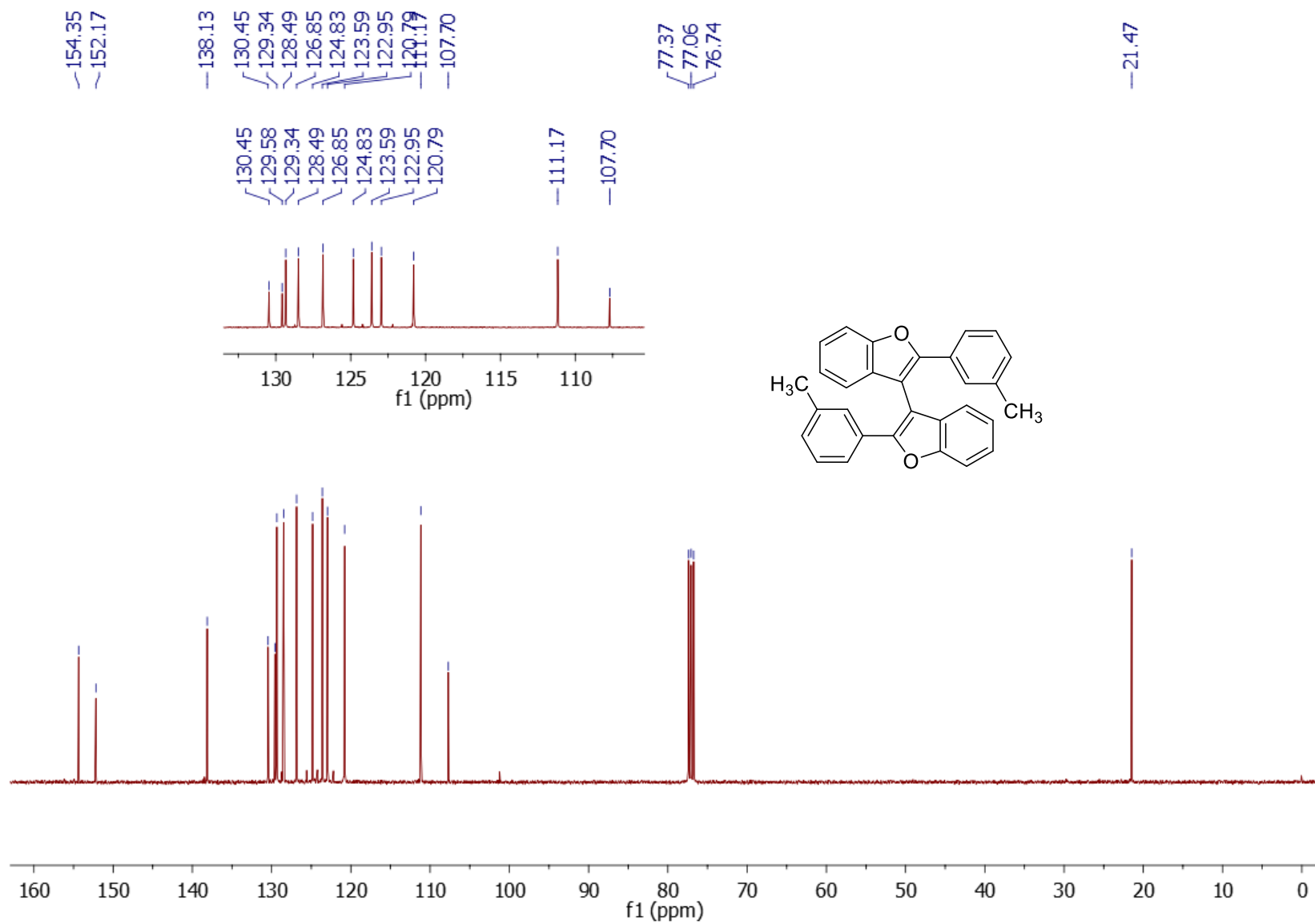
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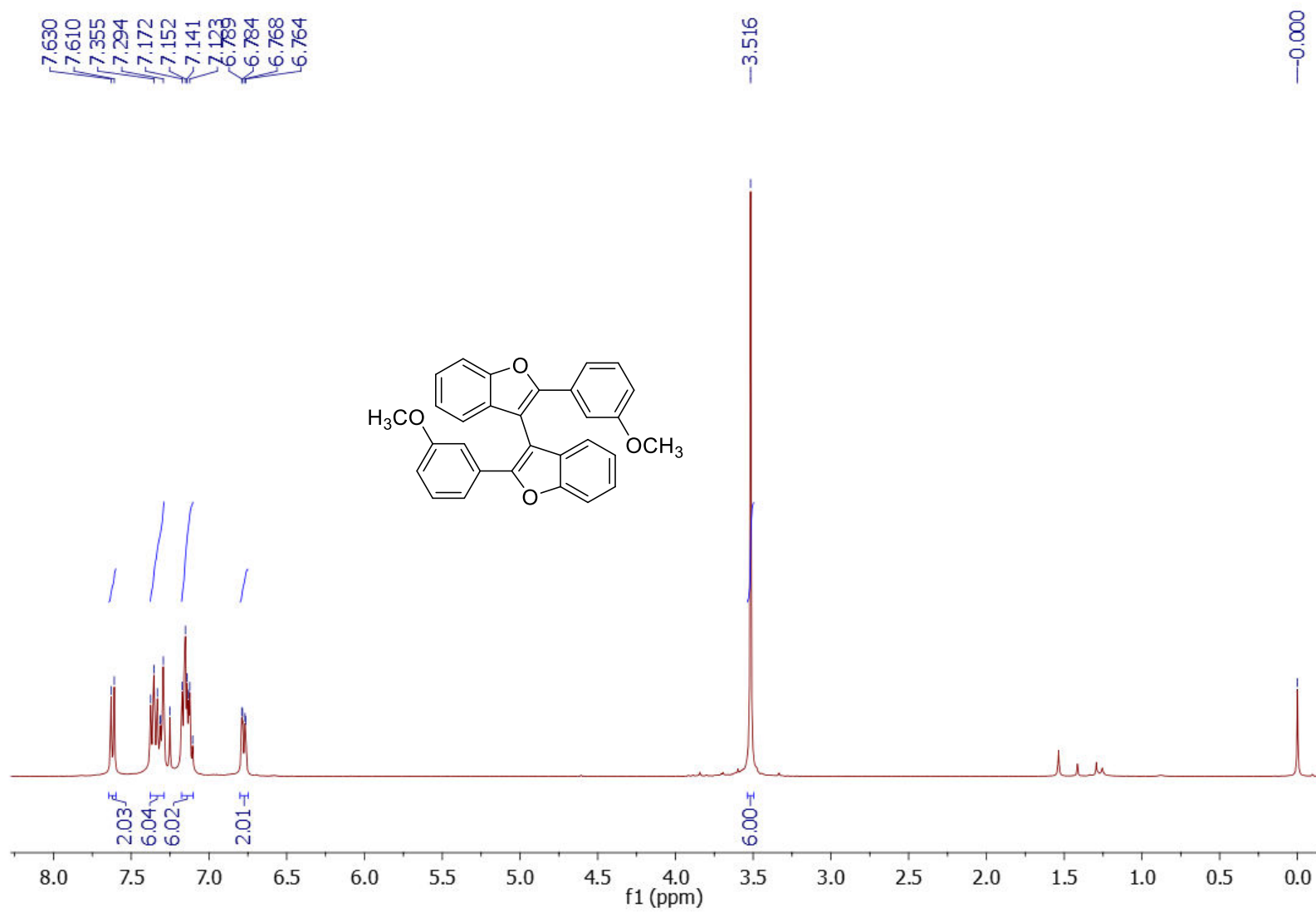


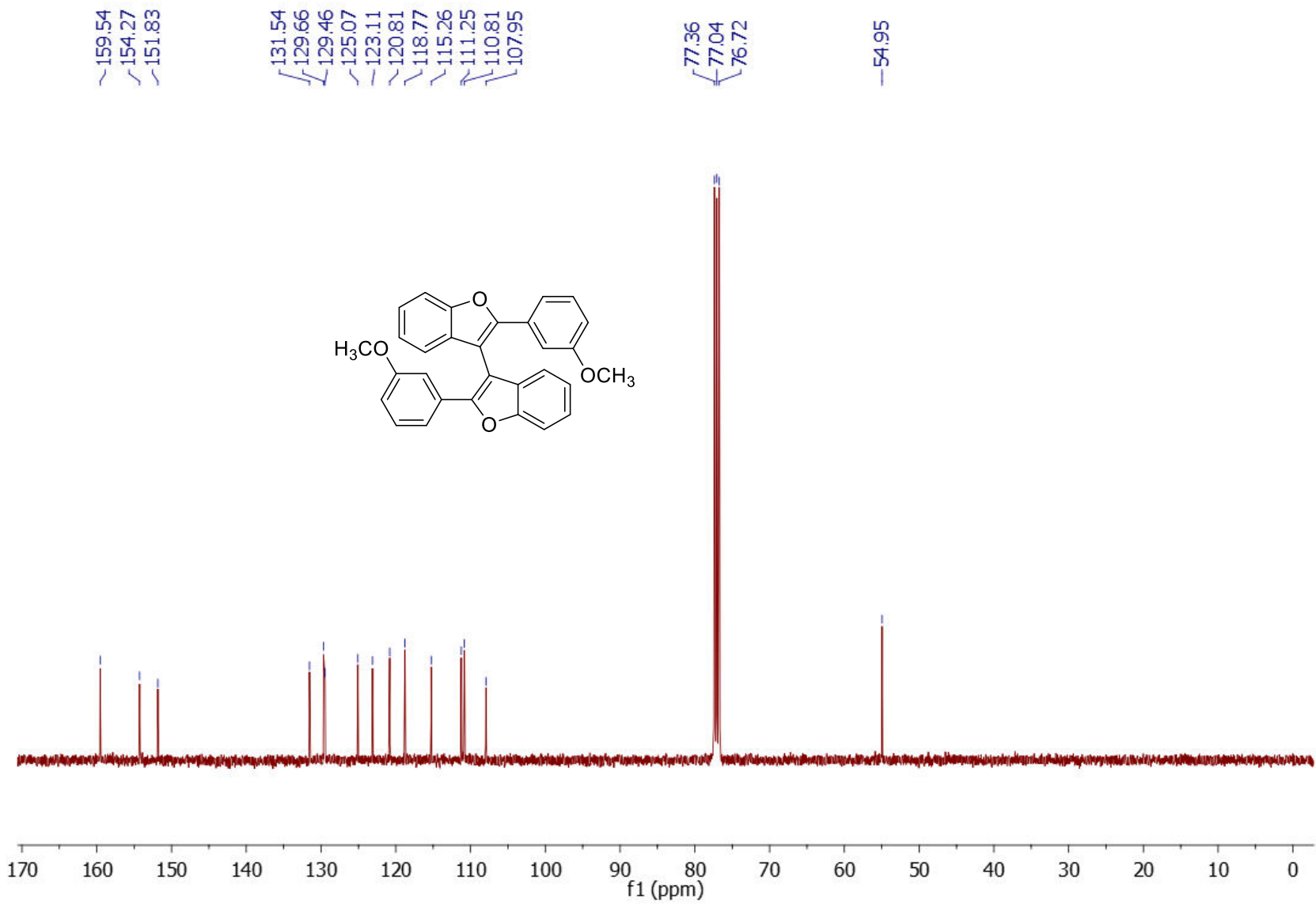
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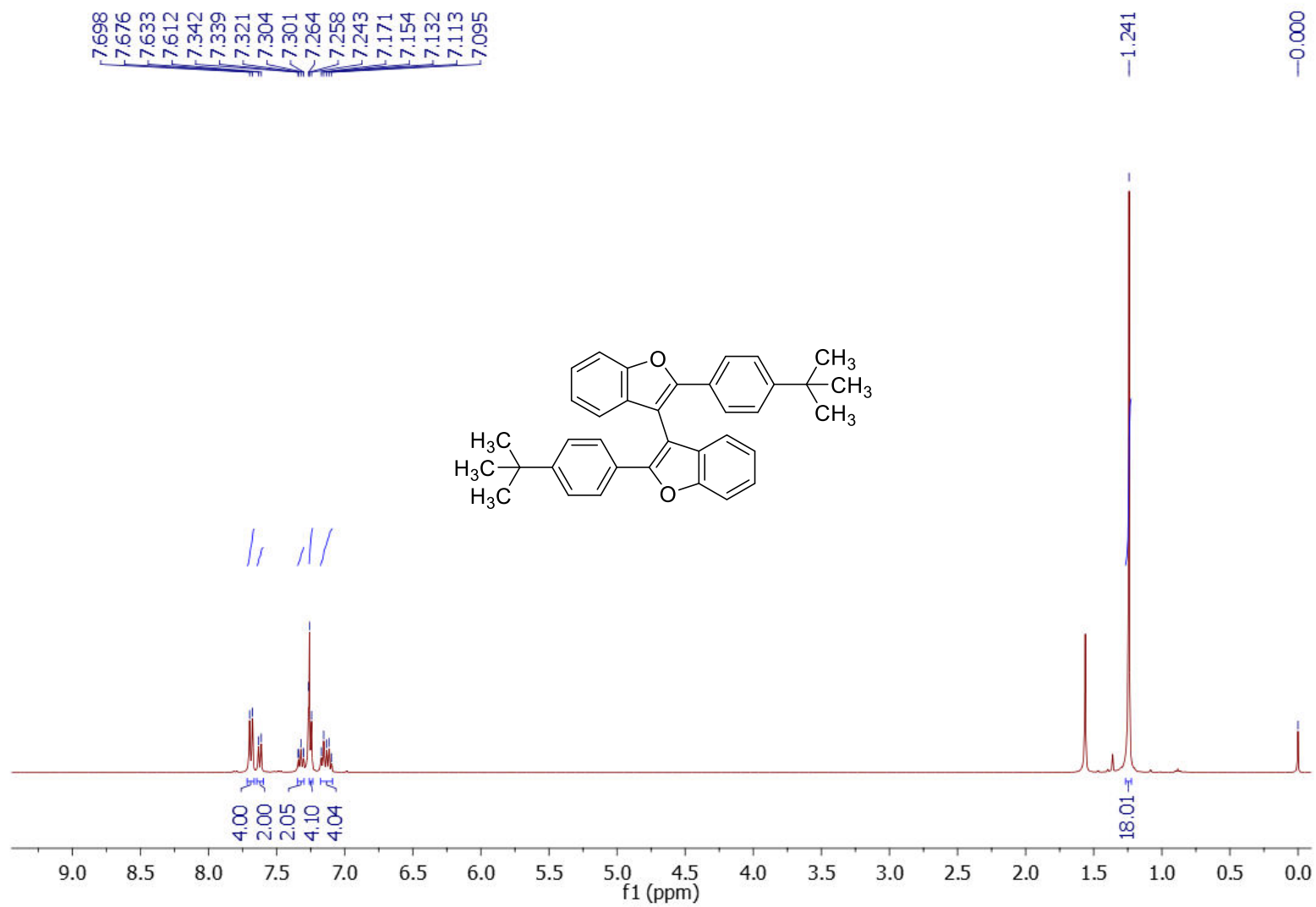


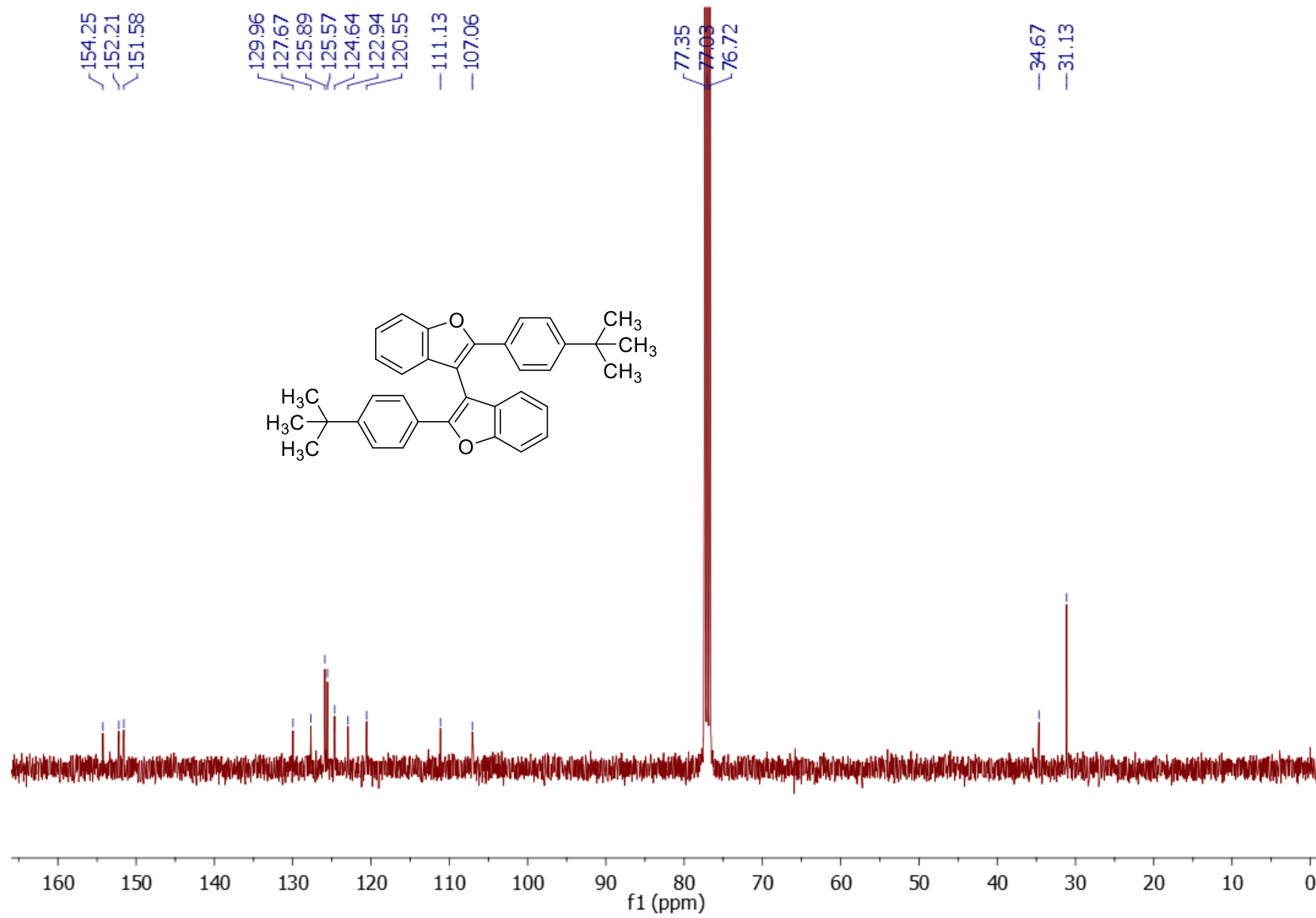
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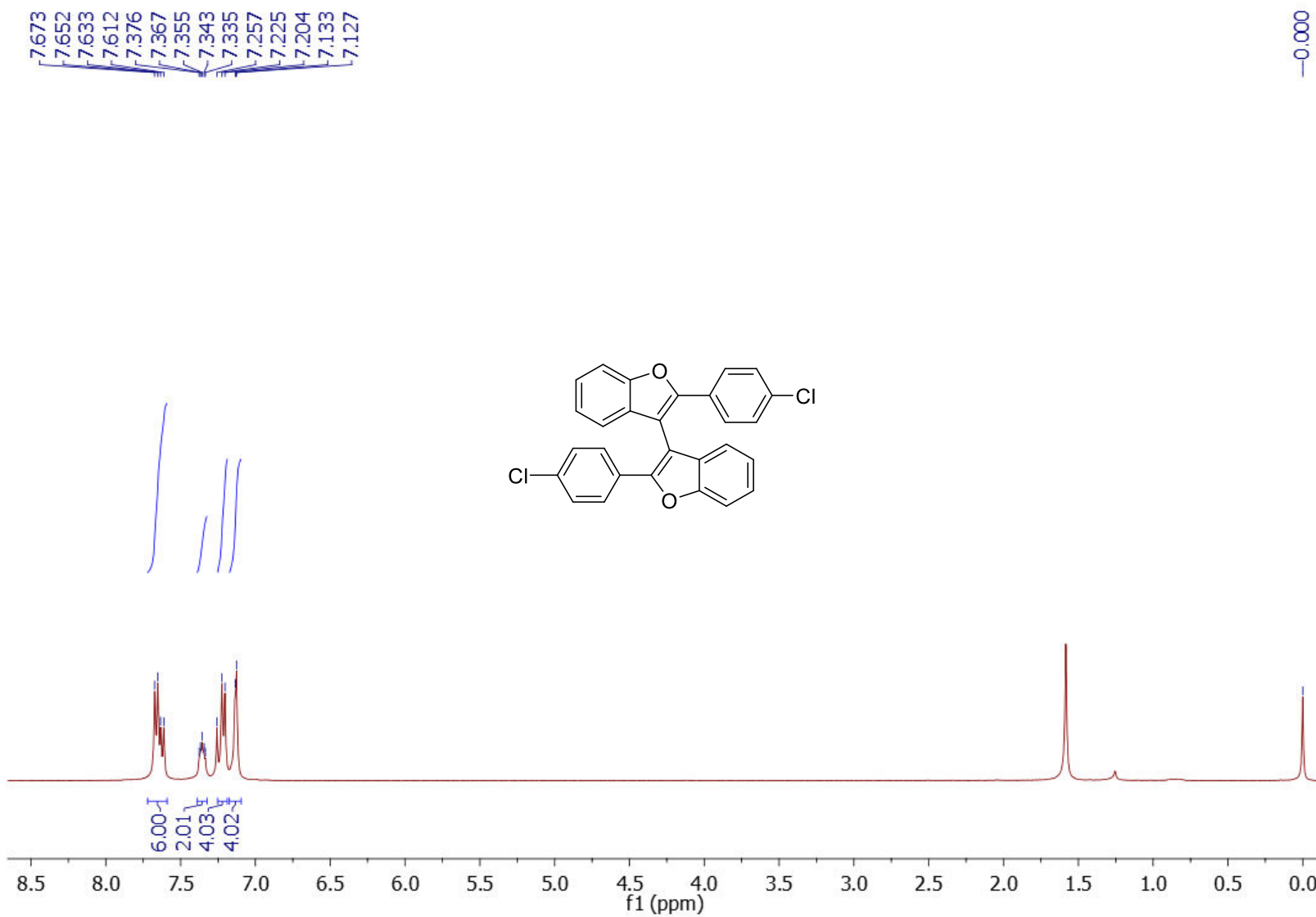


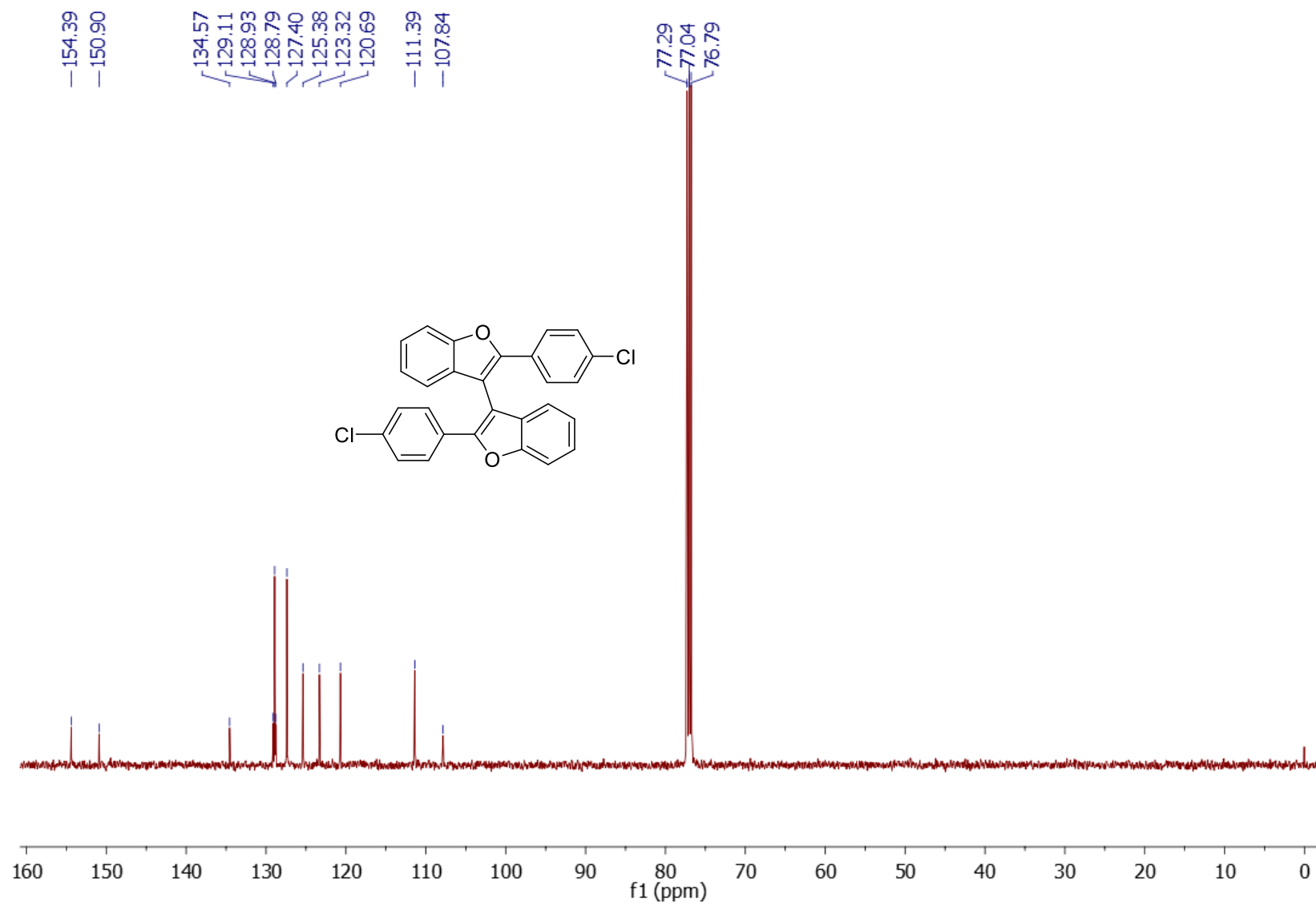
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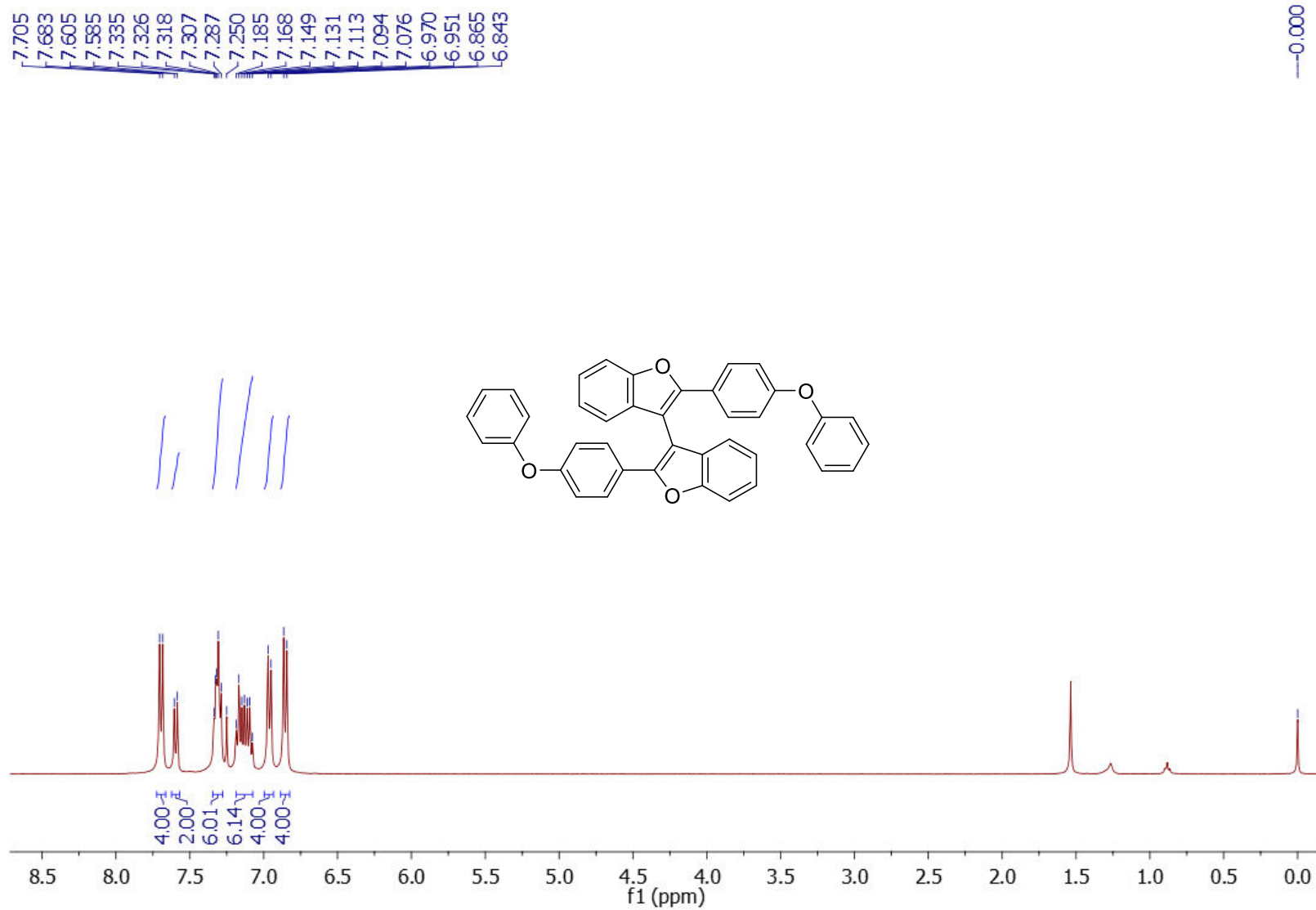


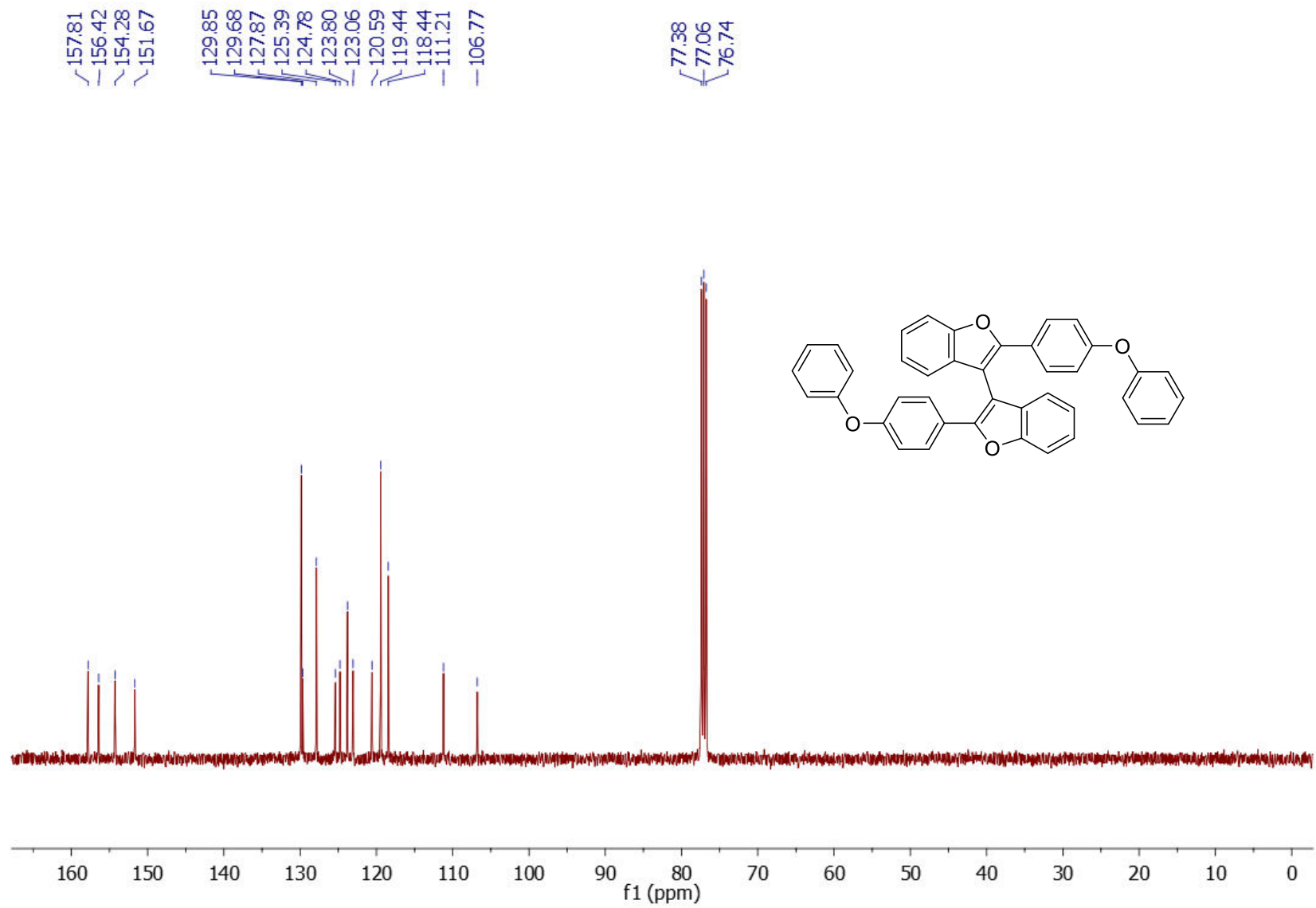
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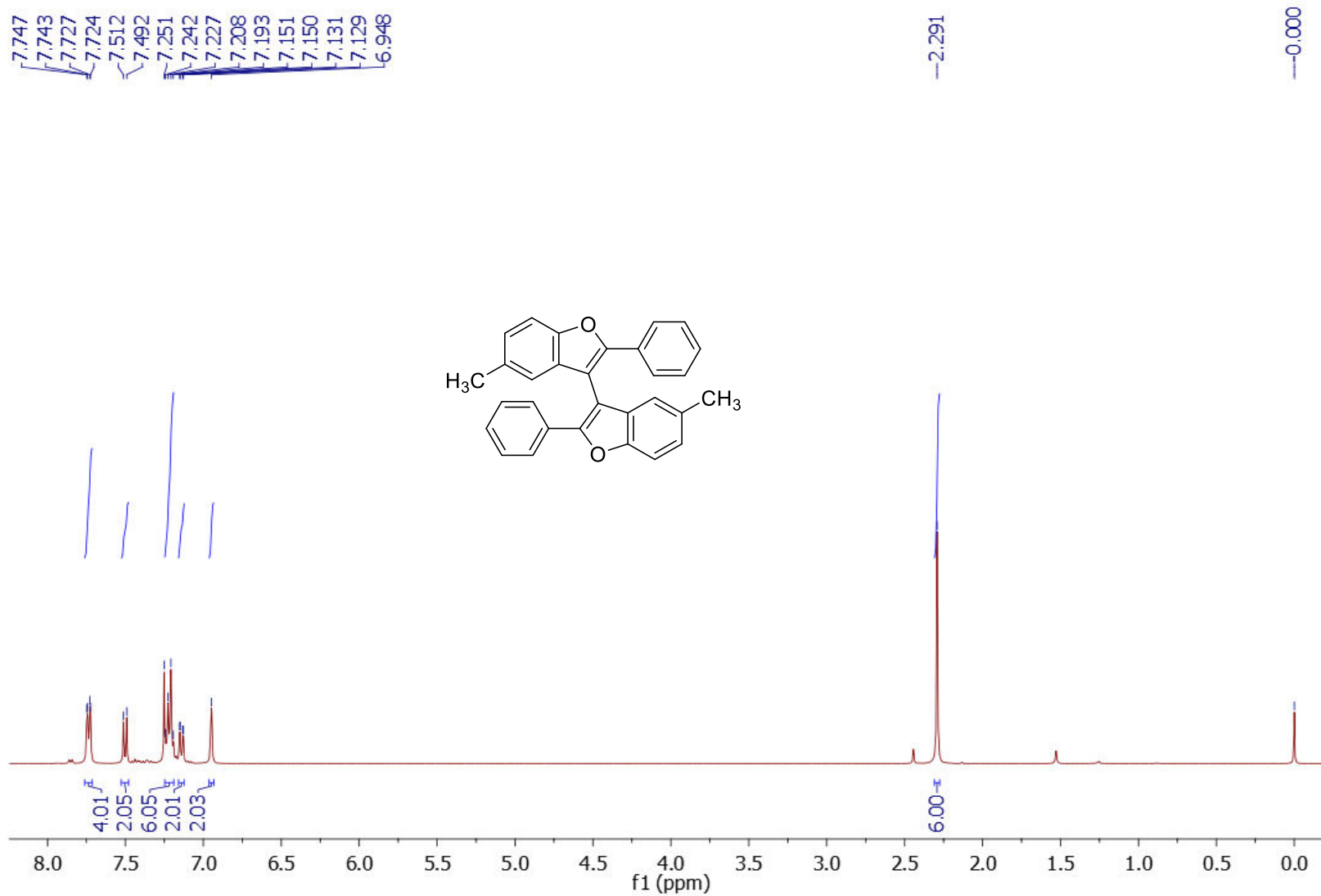


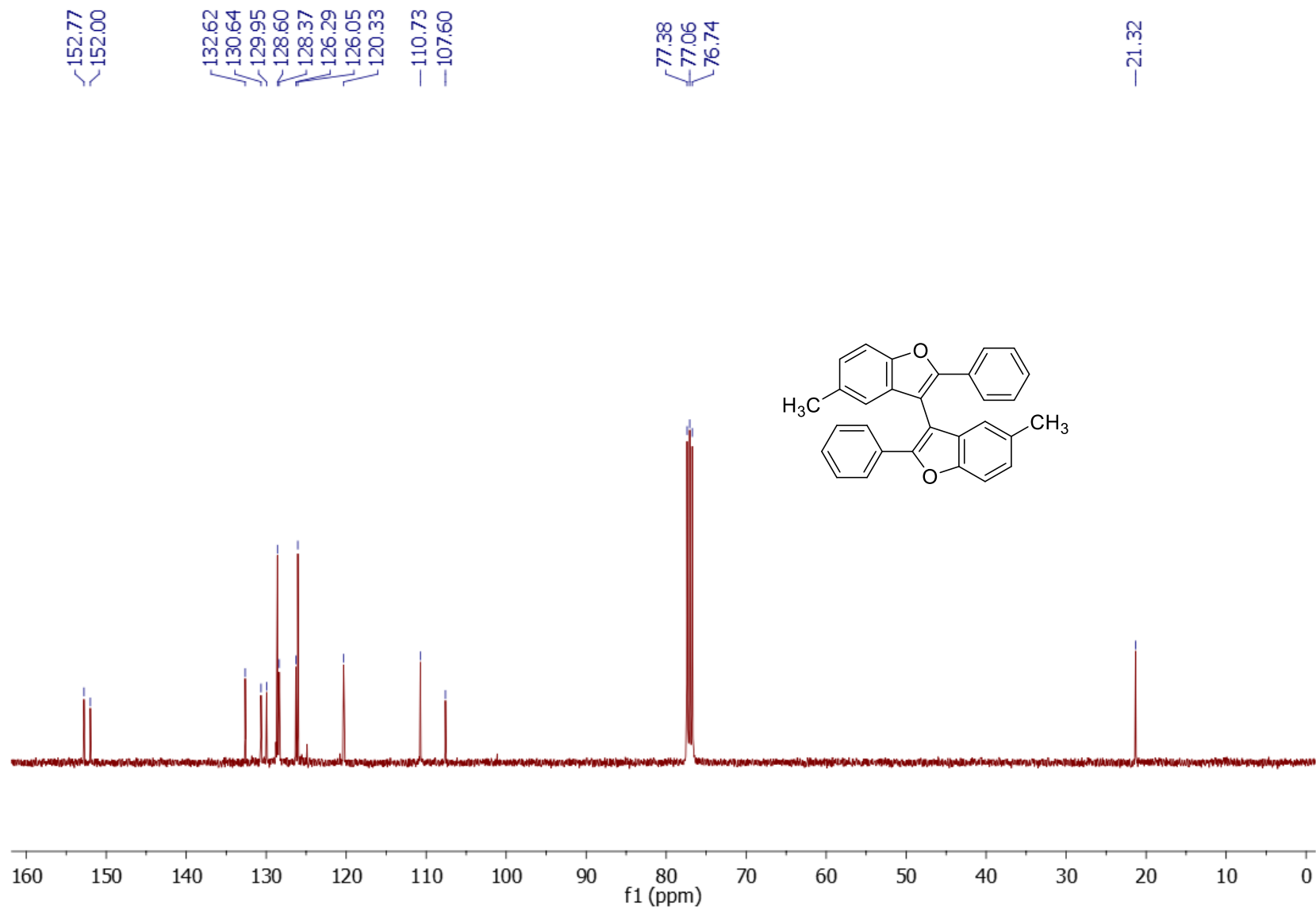
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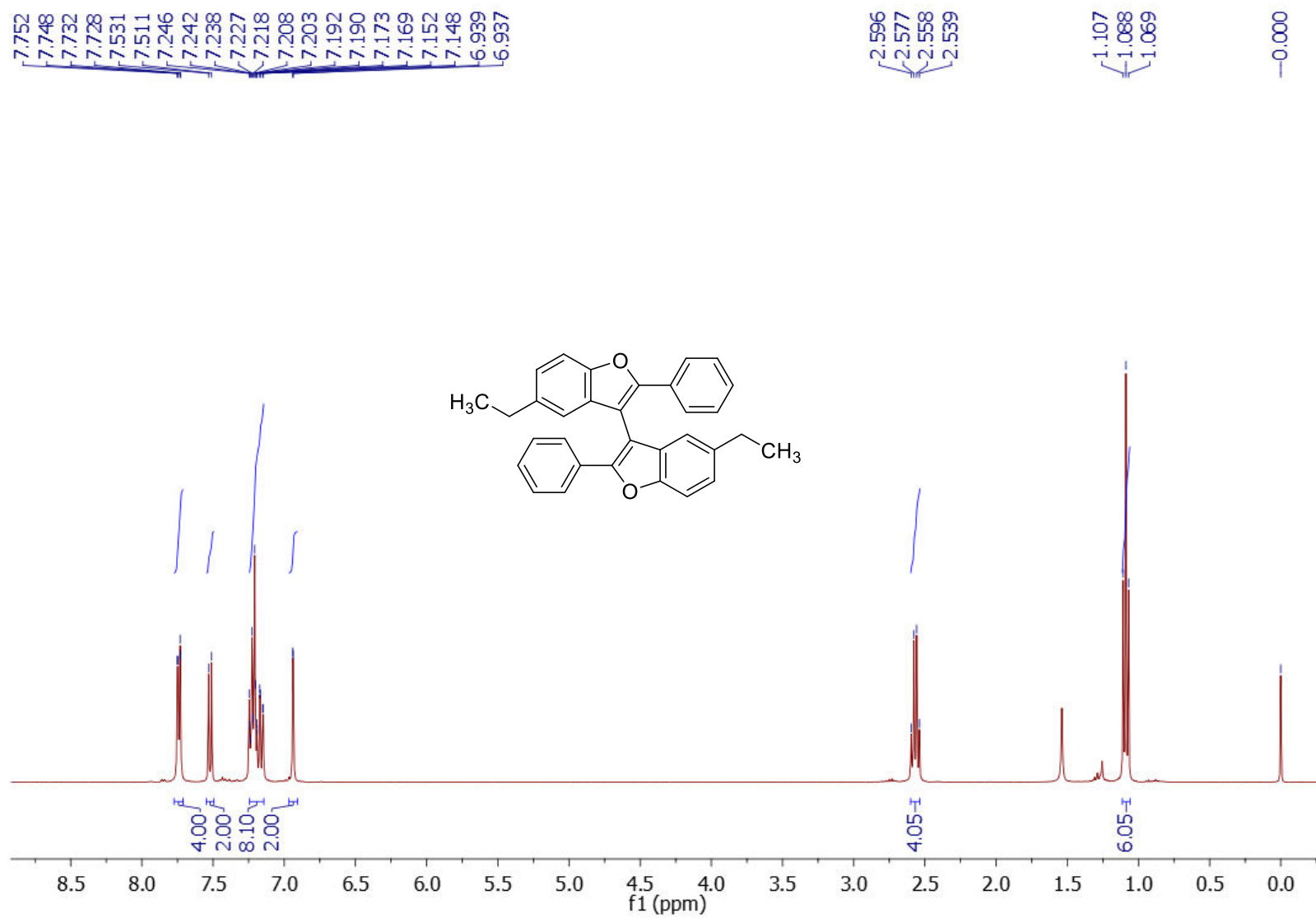


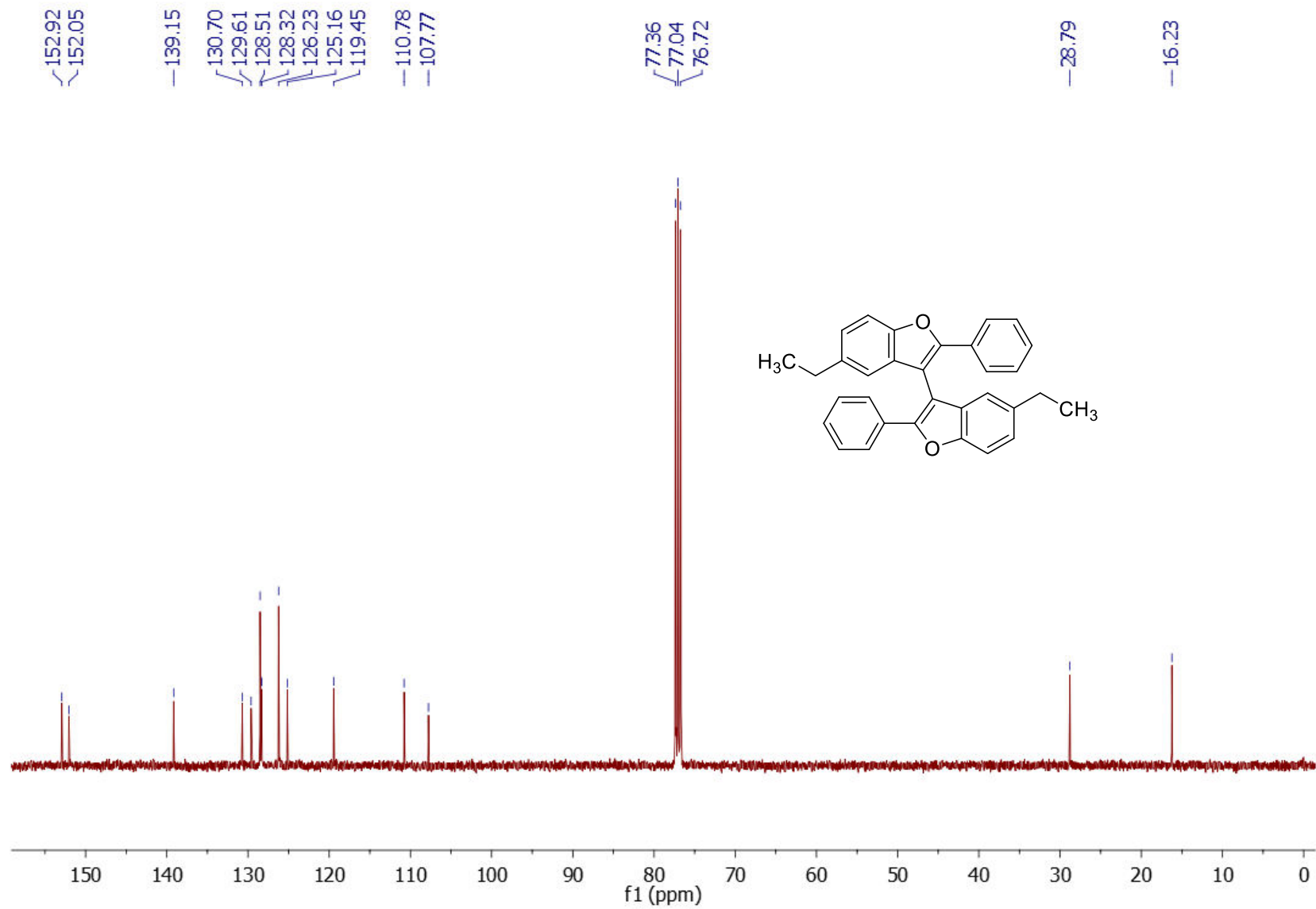
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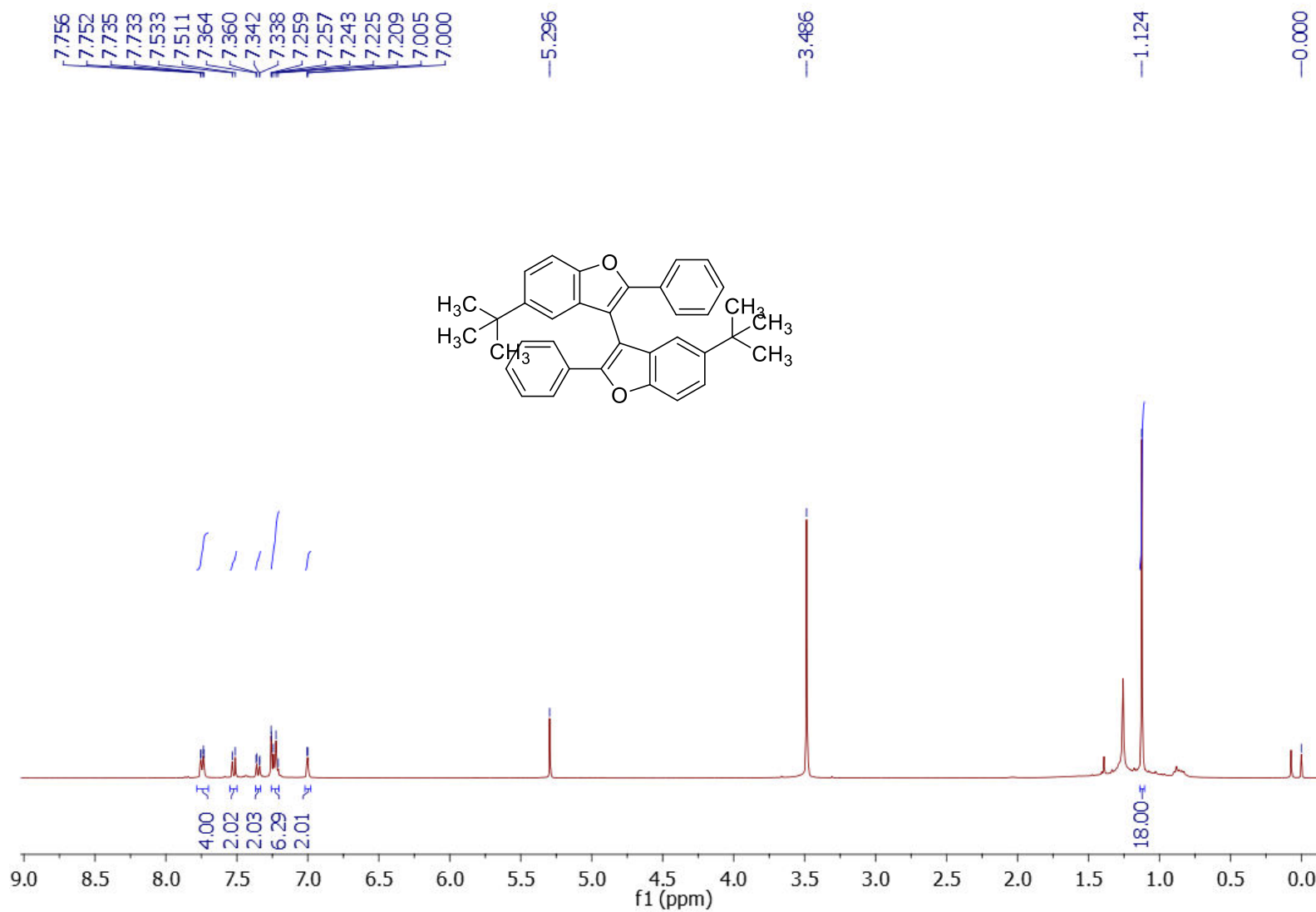


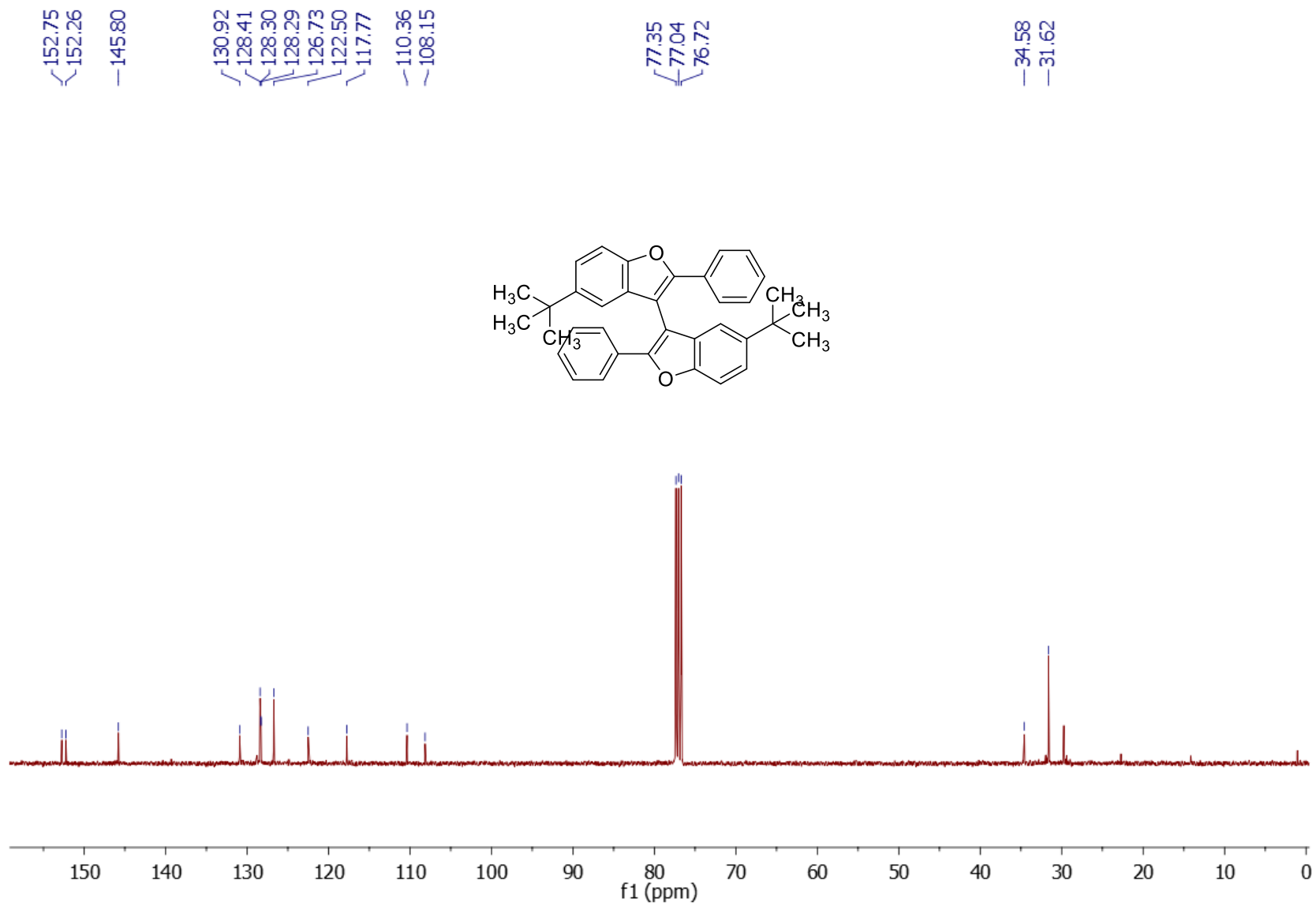
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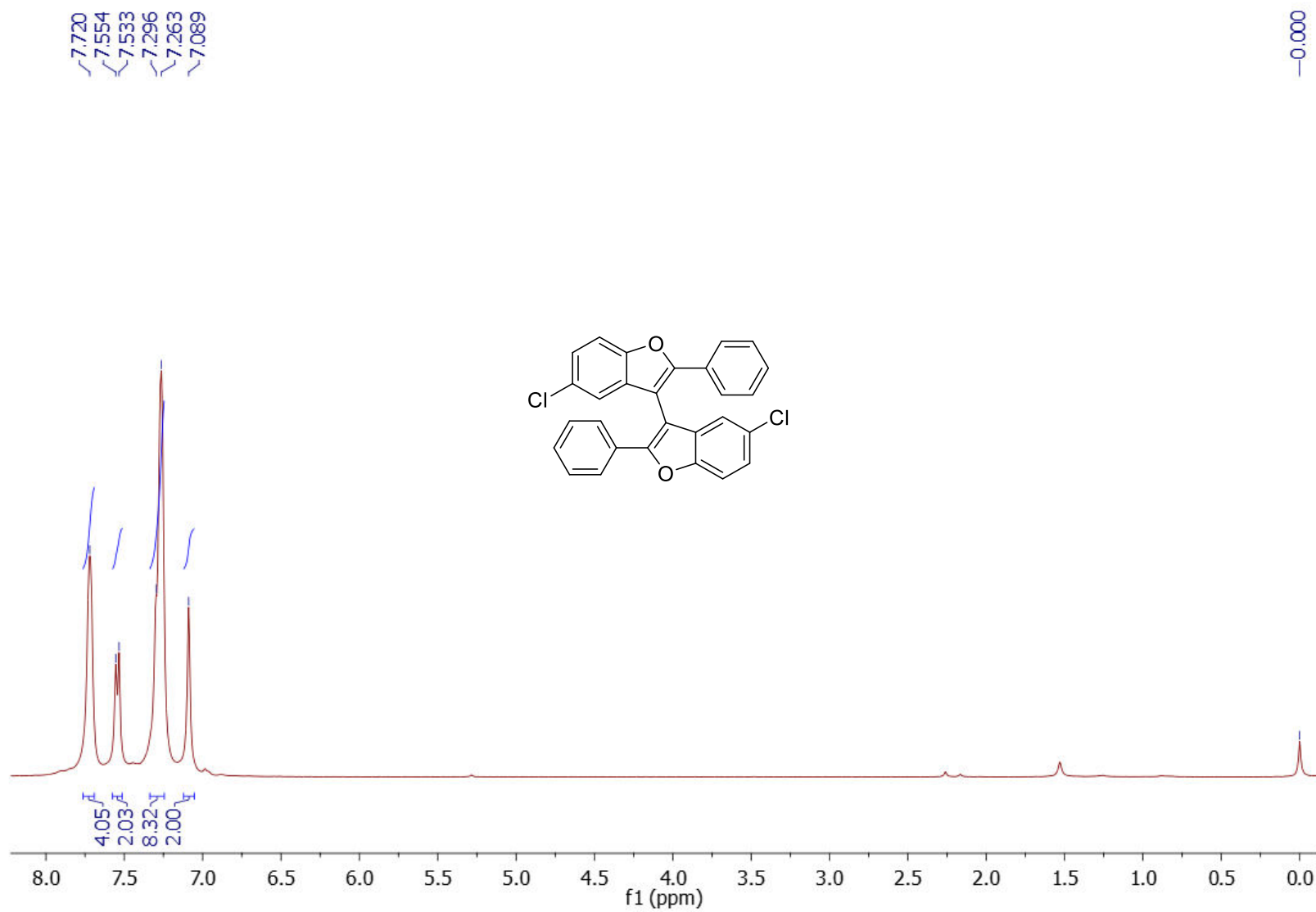


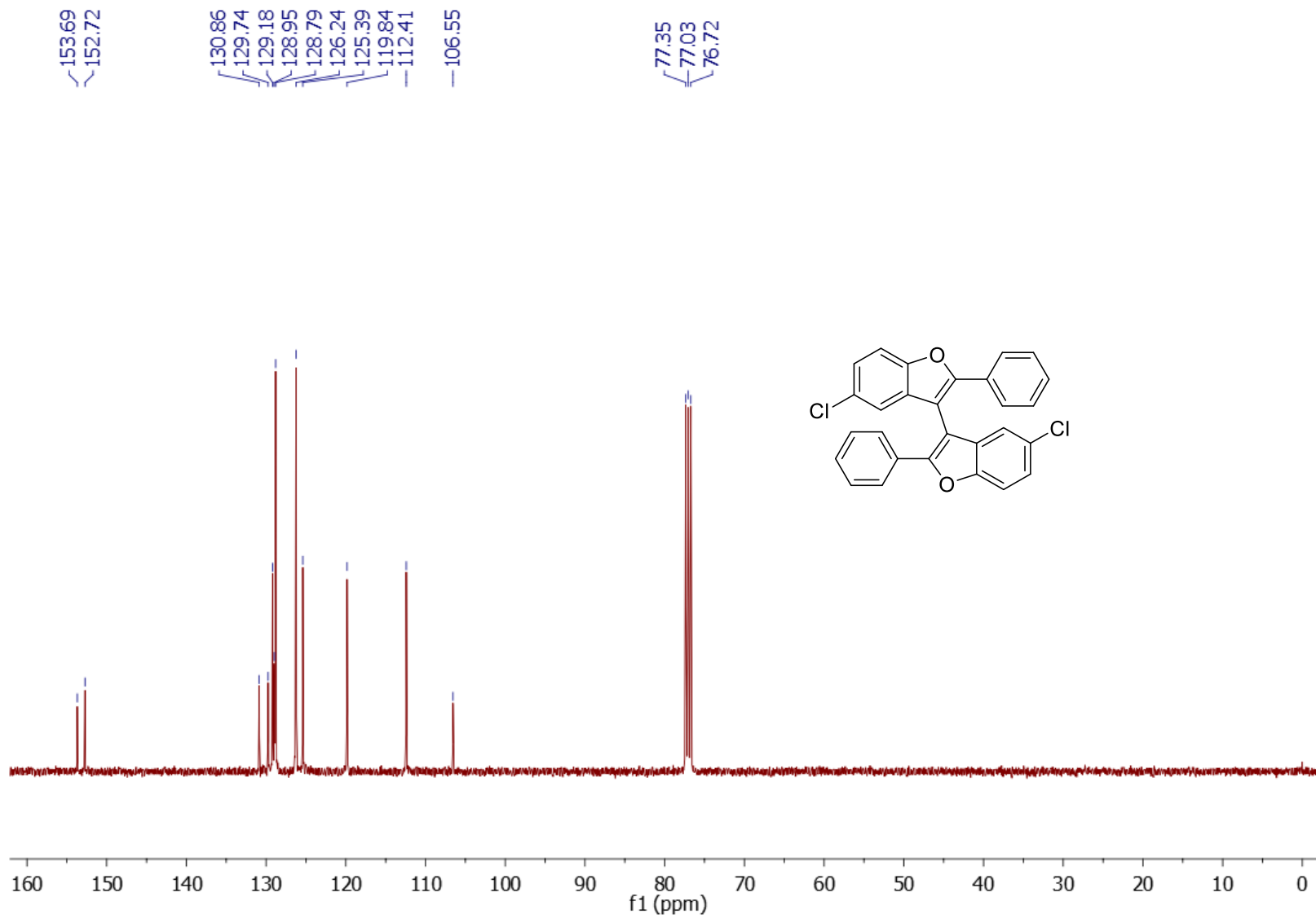
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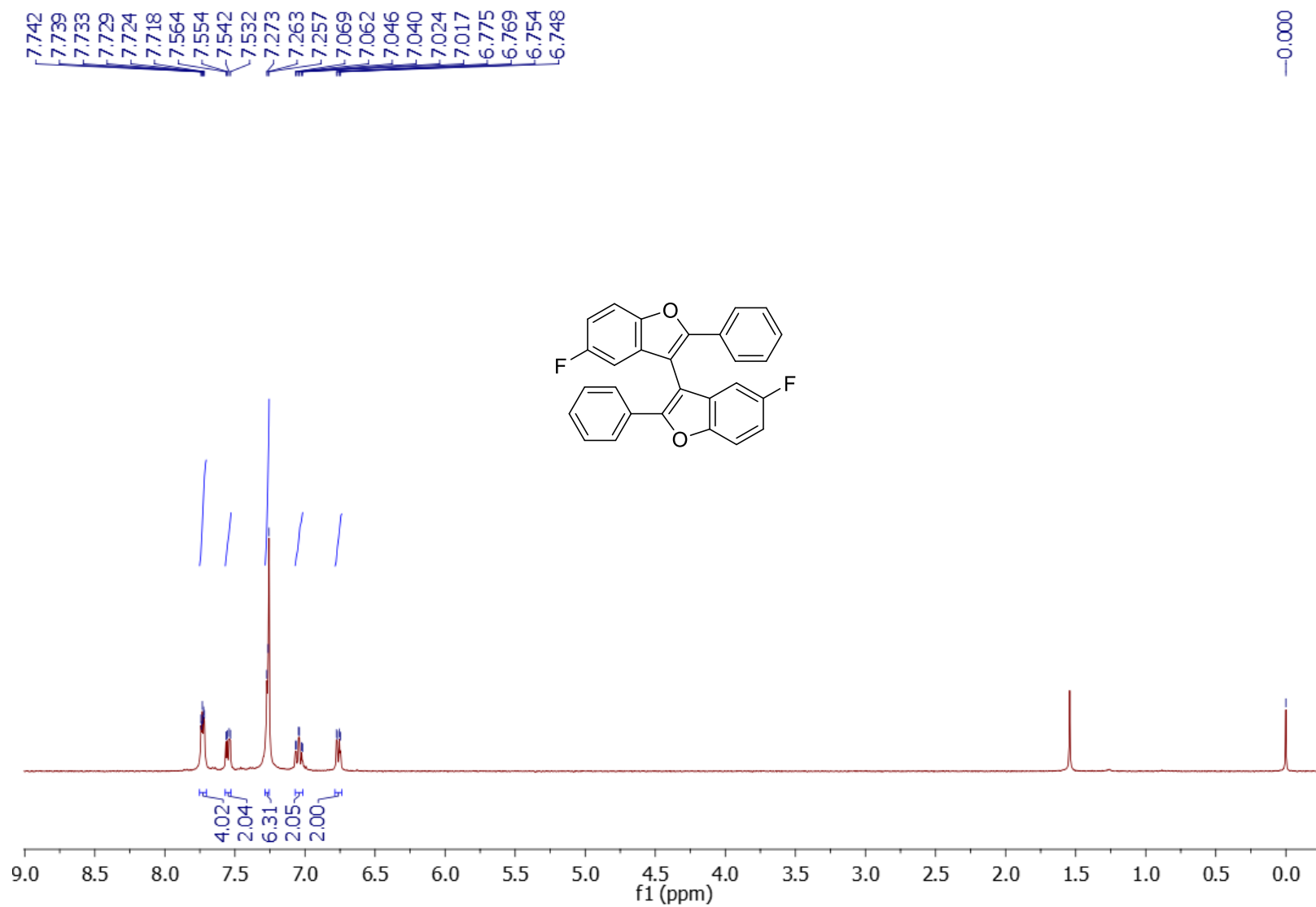


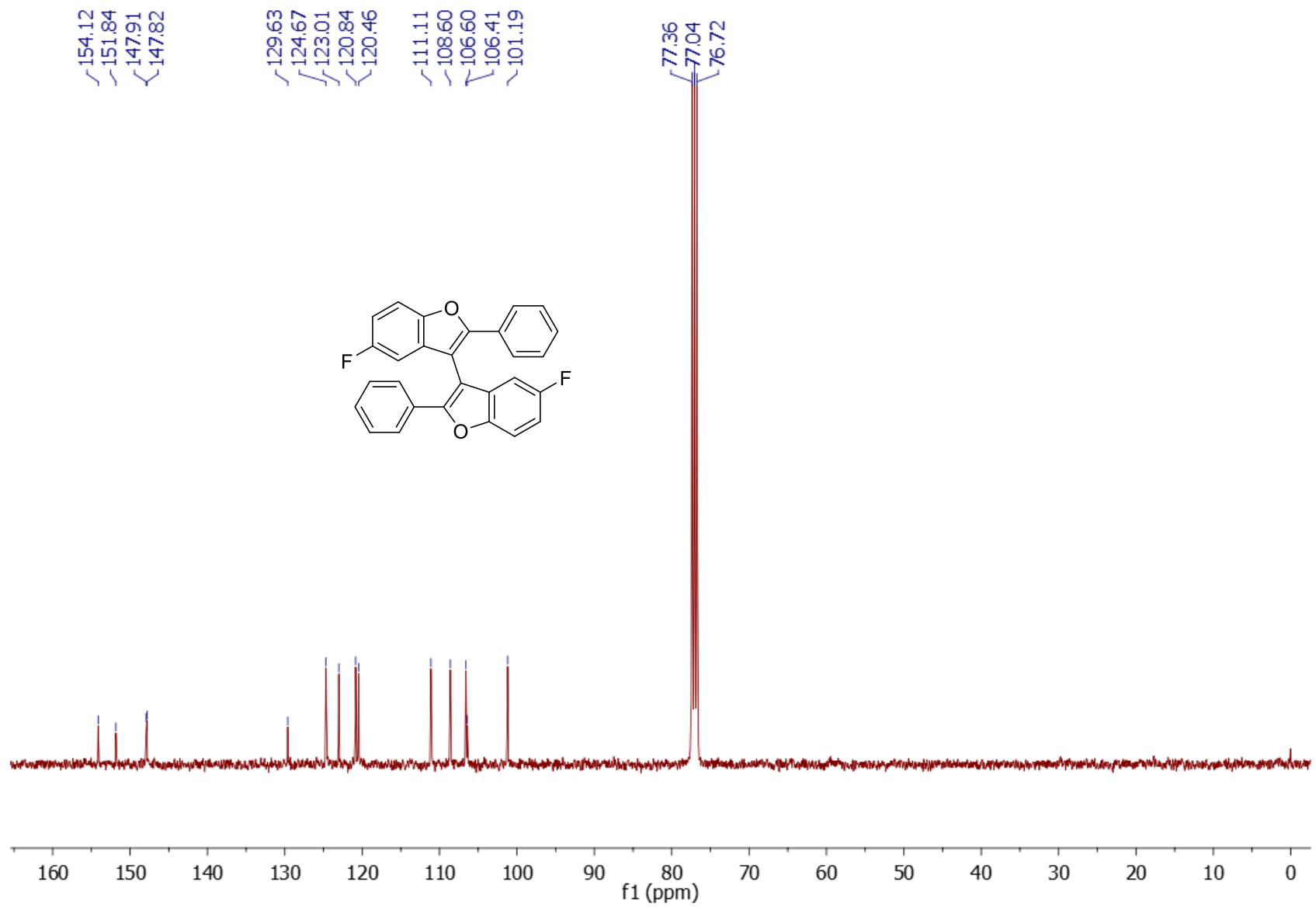
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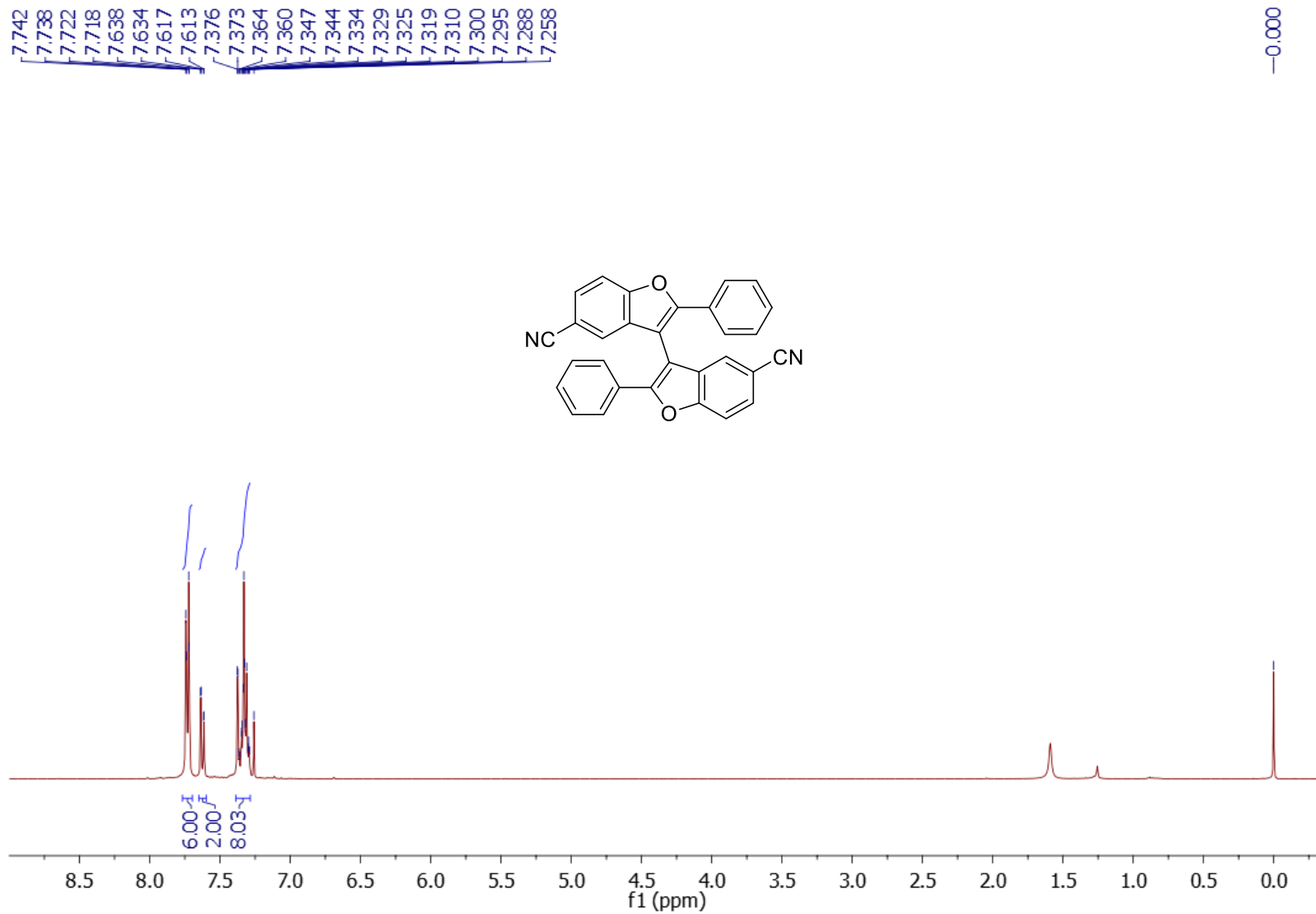


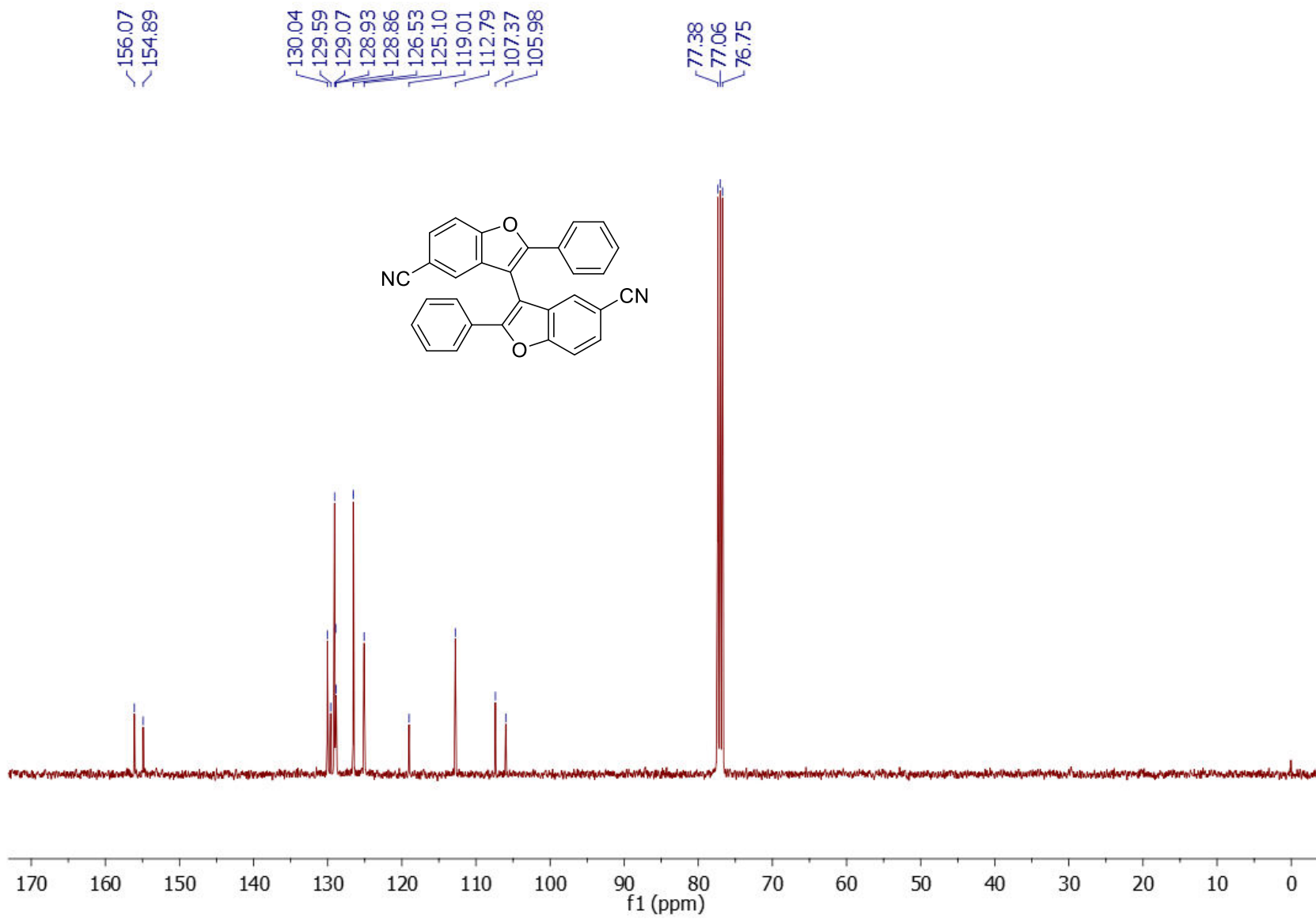
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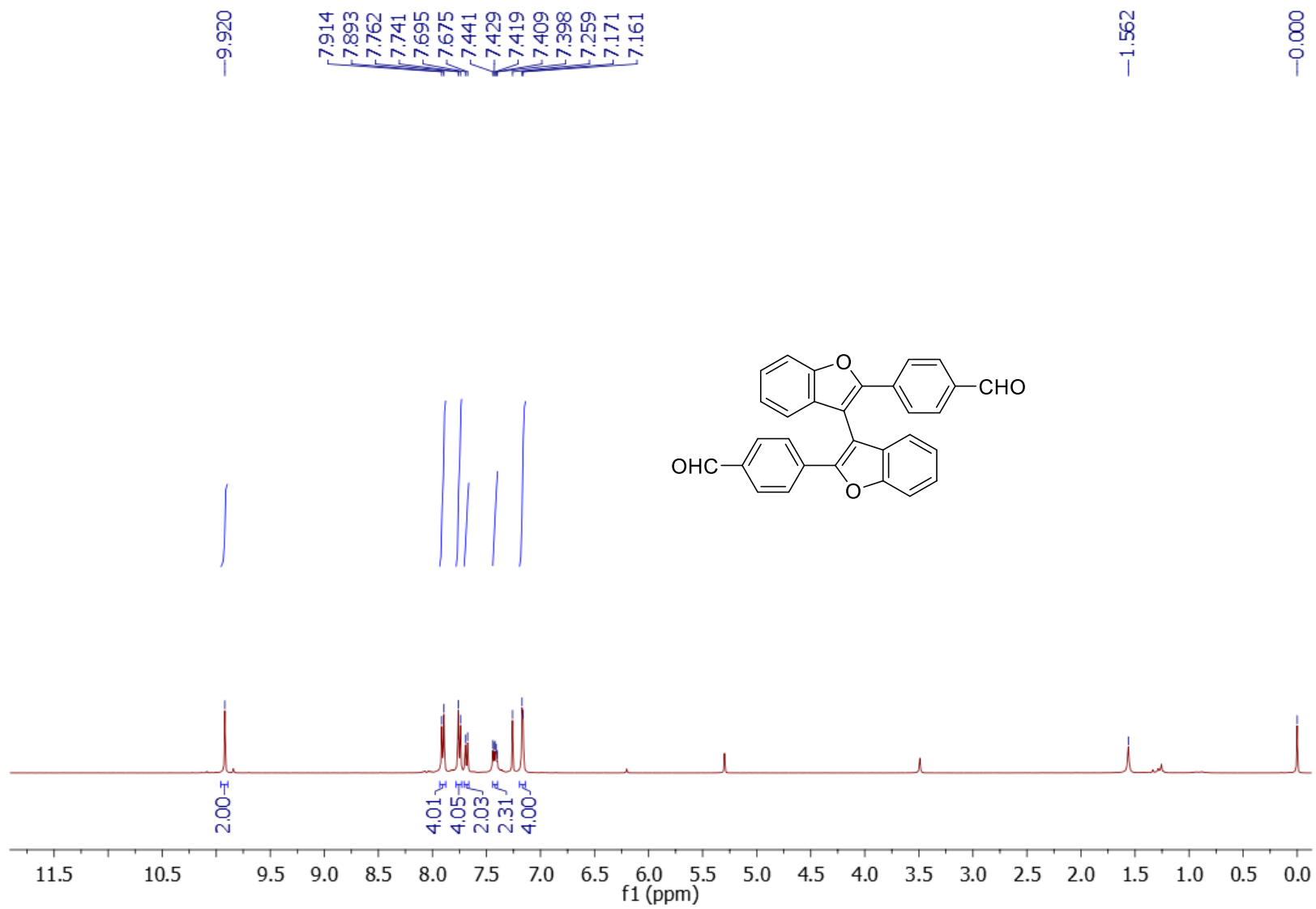


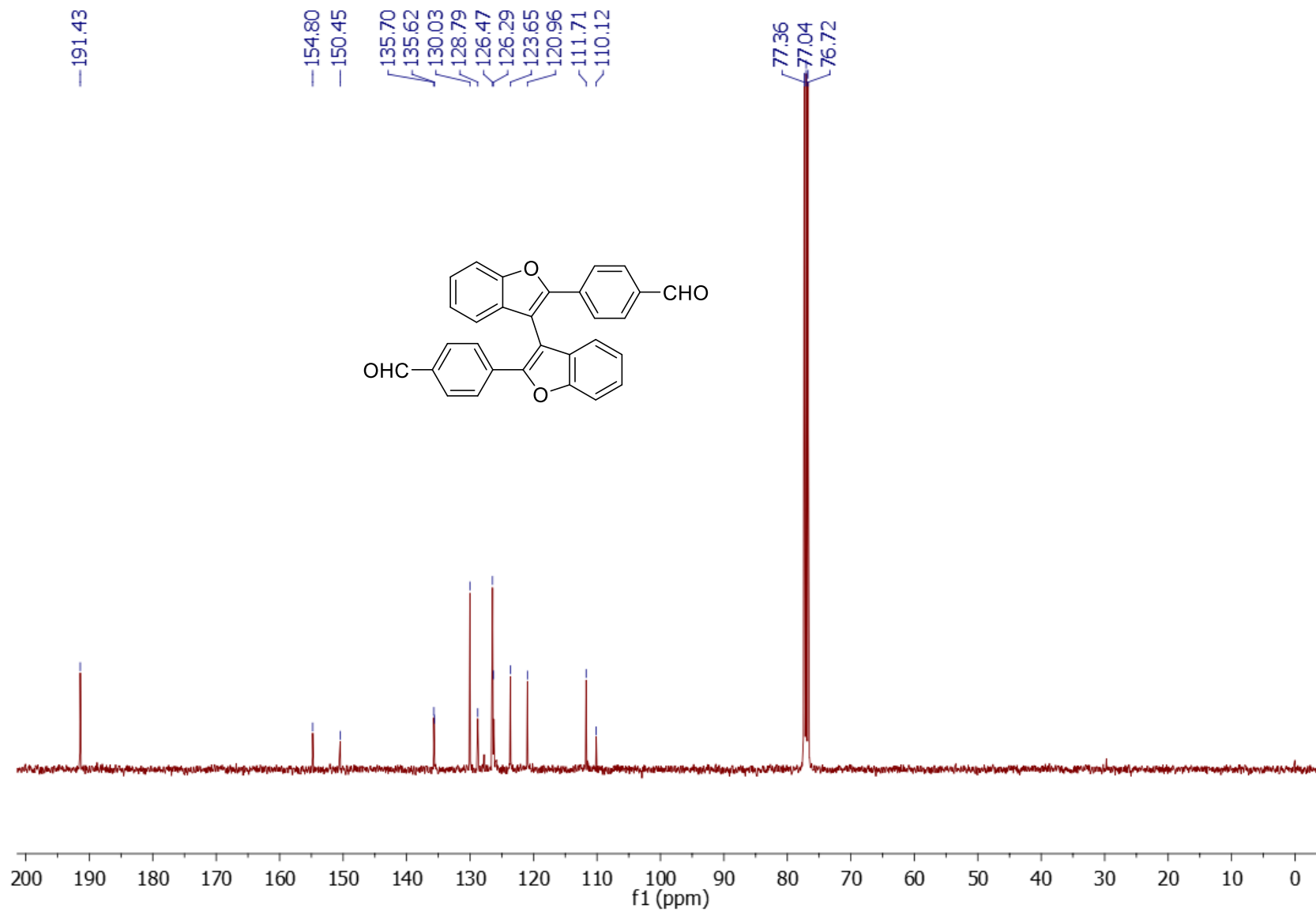
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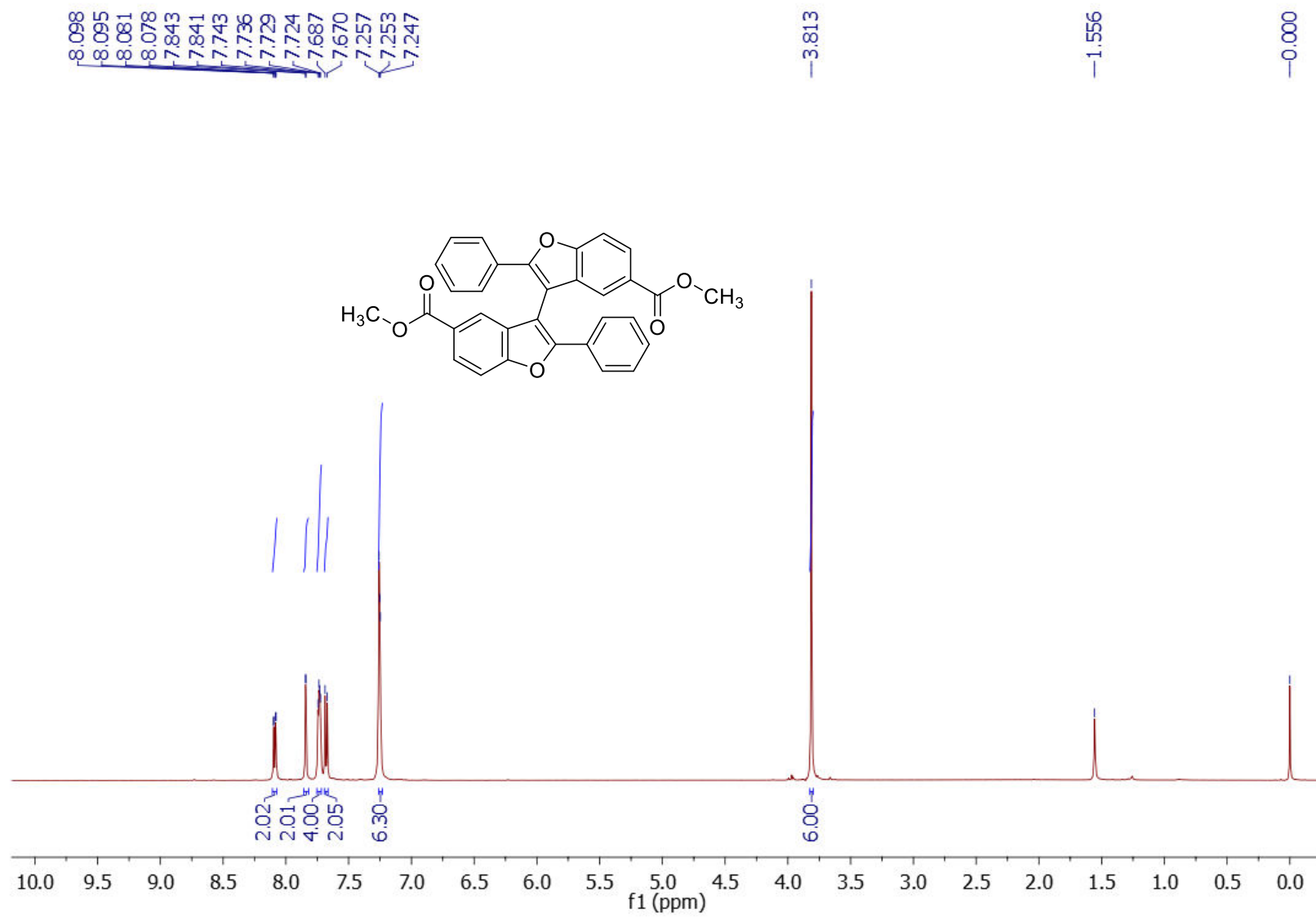


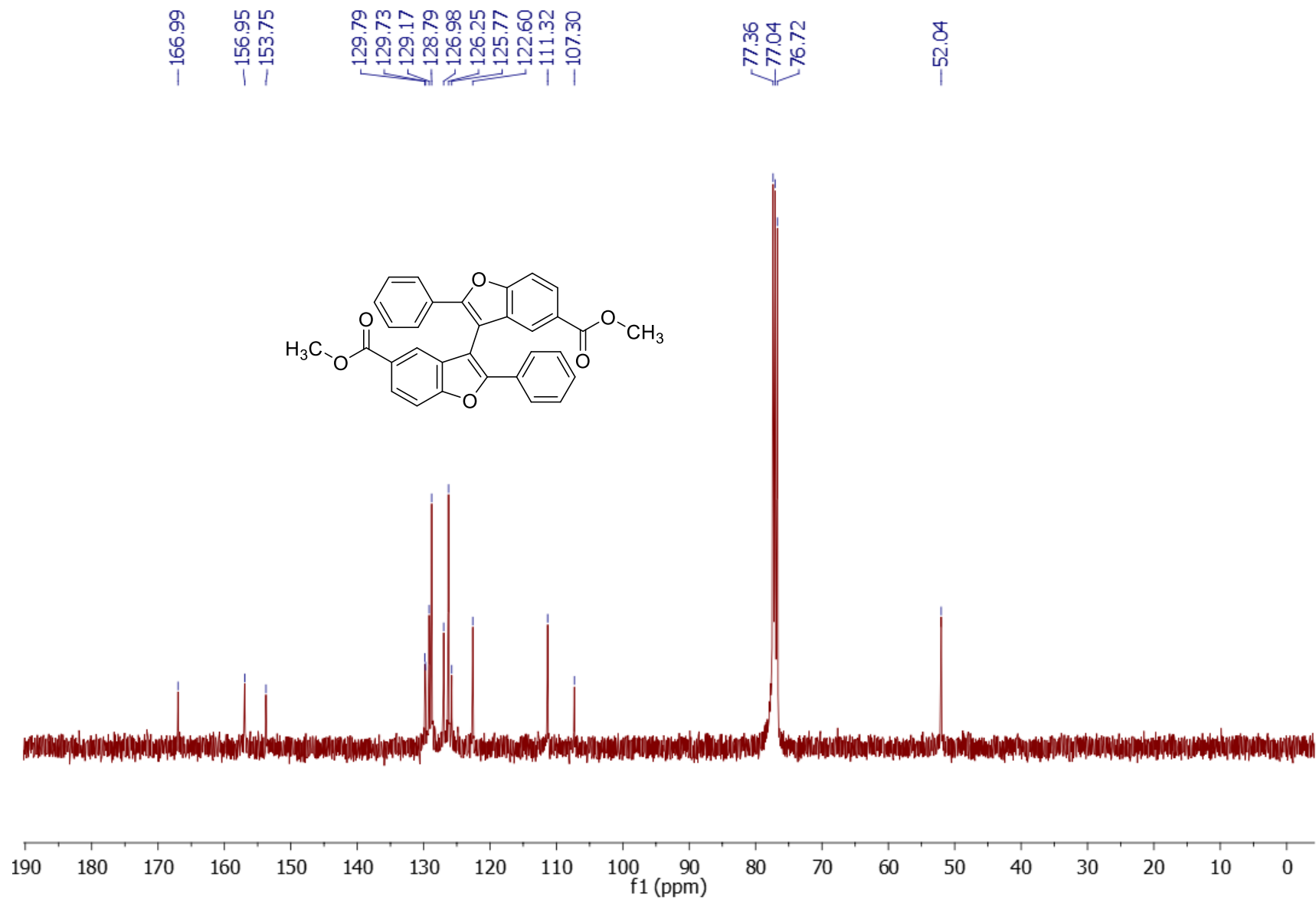
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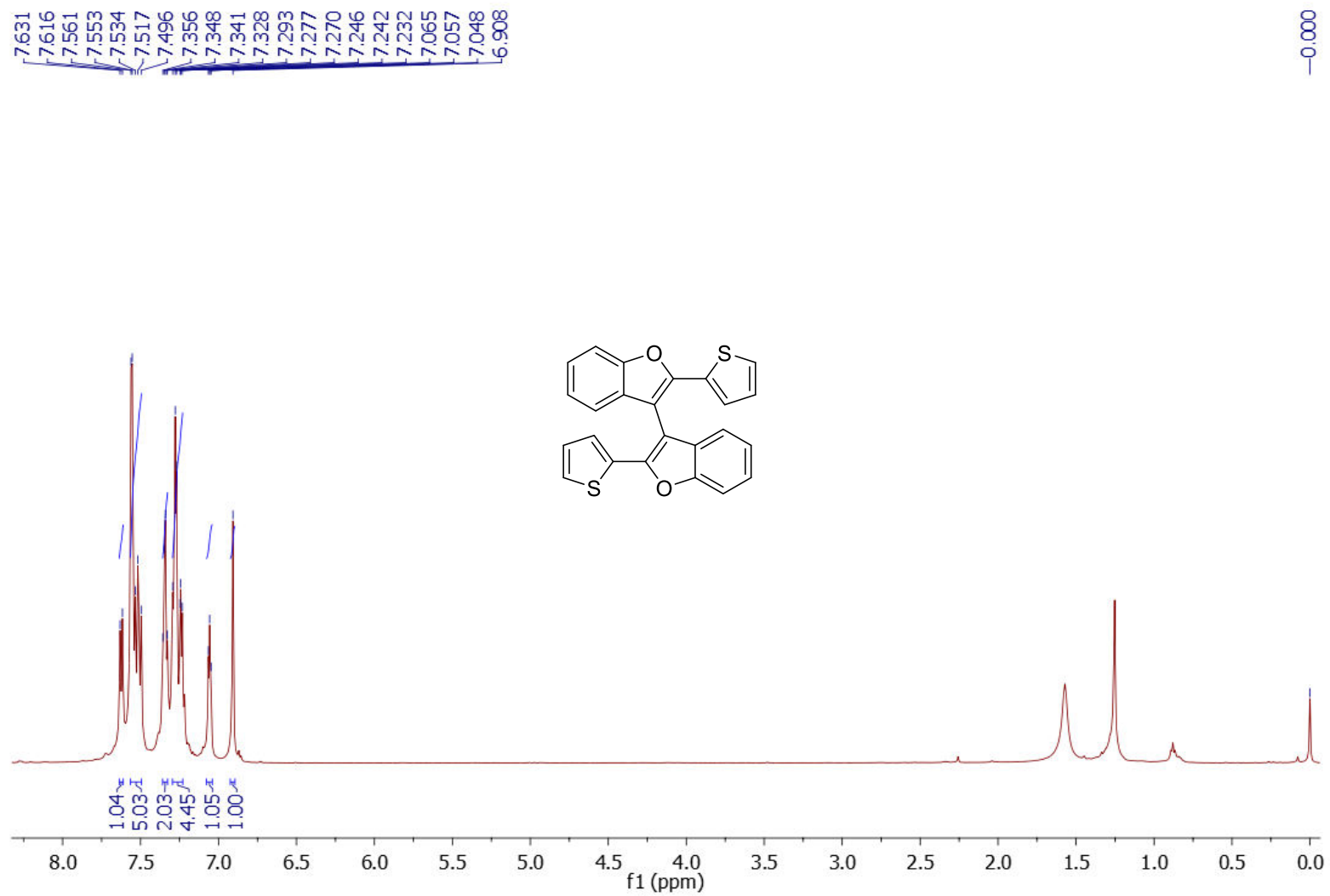


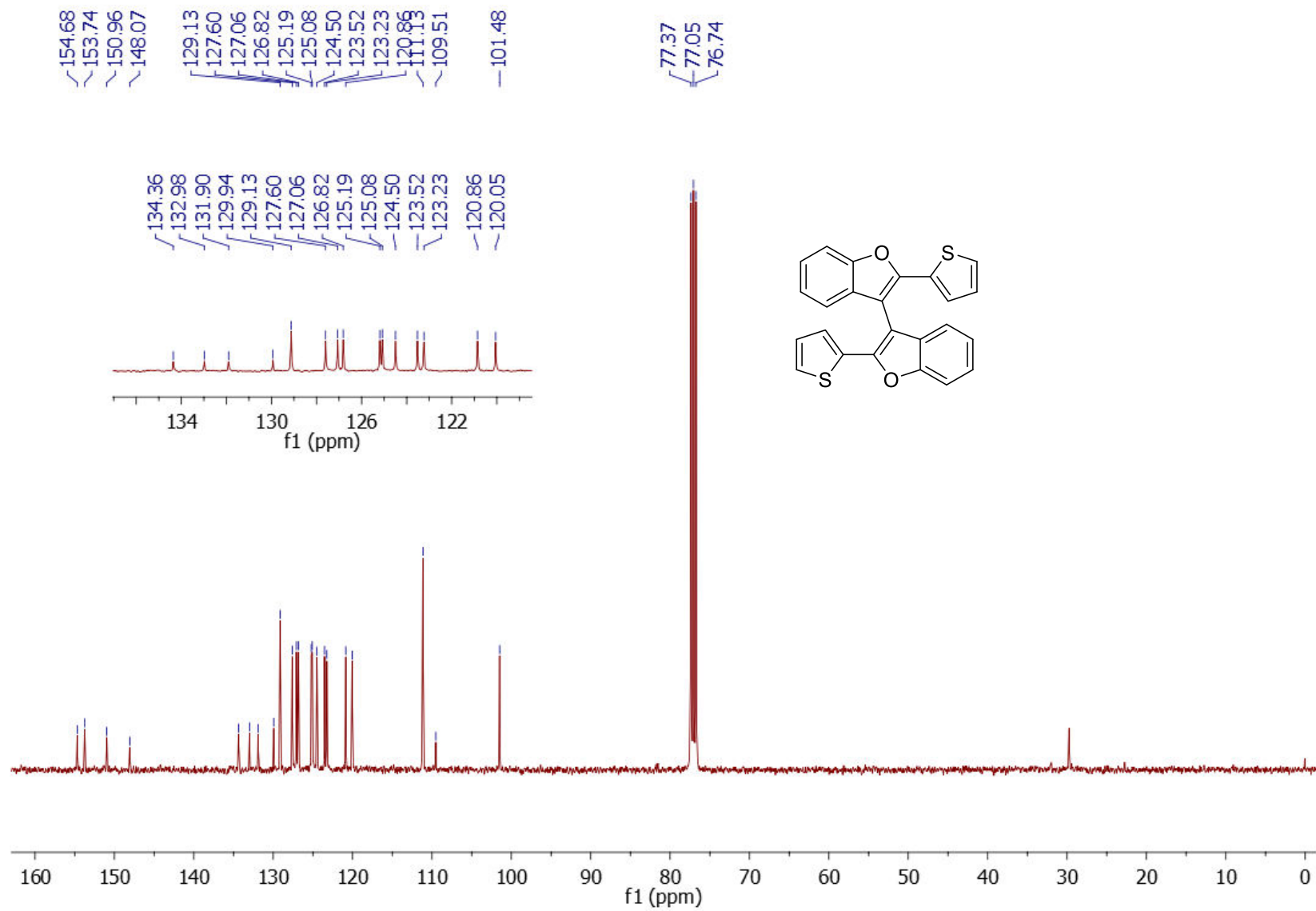
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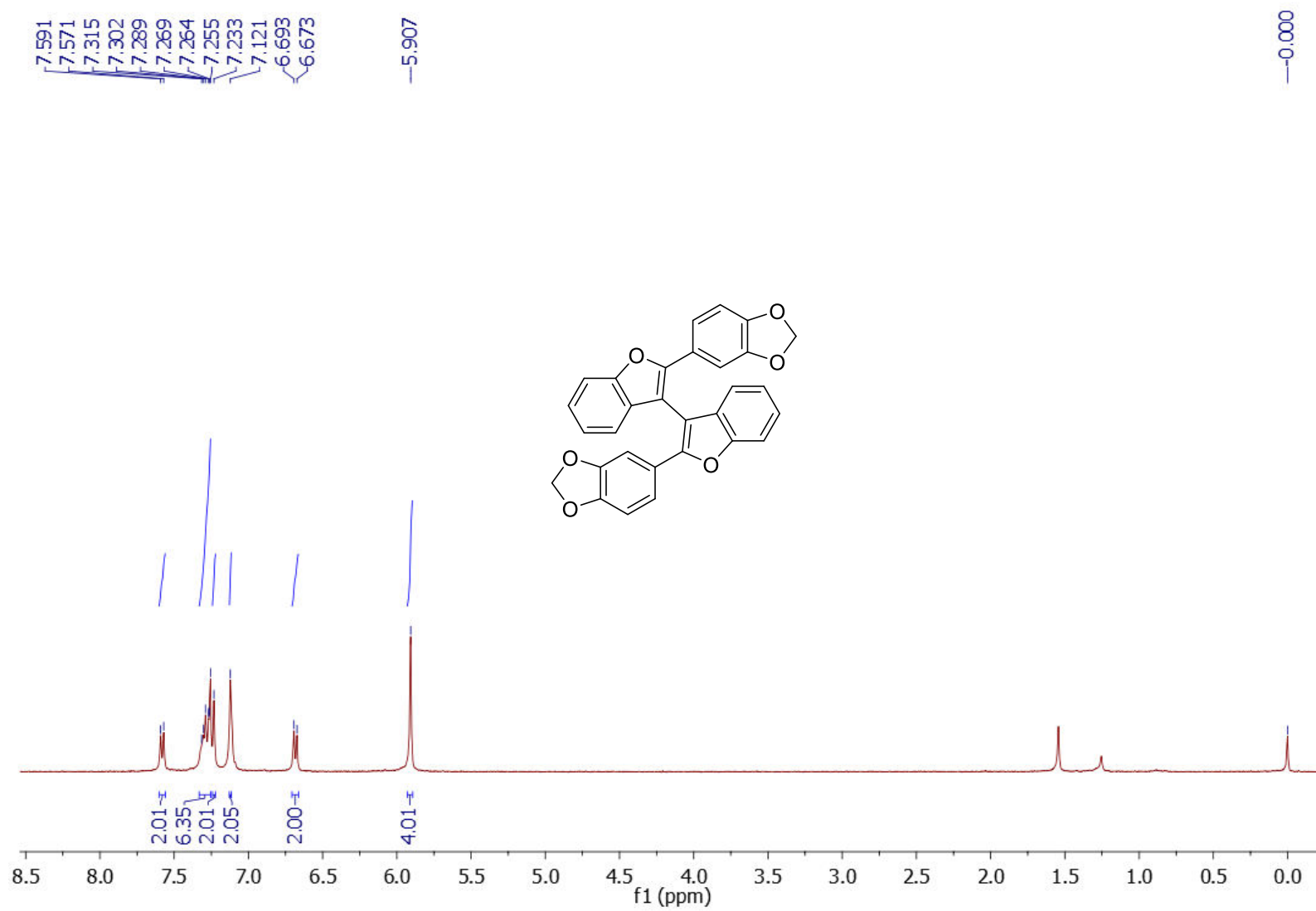


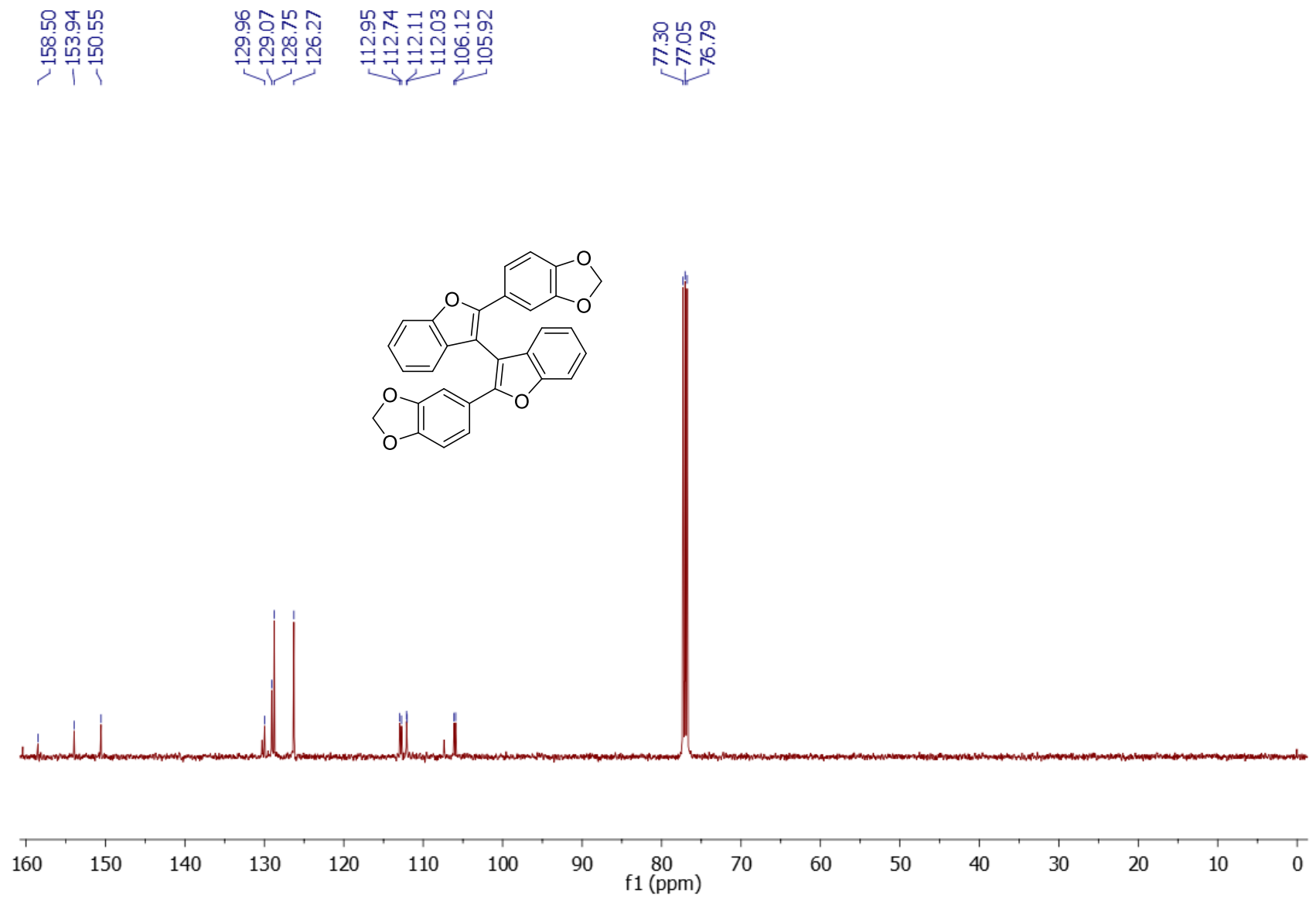
Compound 2s





Compound 2t





Compound 2u

