Synthesis of 2,3-Disubstituted Benzo[*b*]furans by the Palladium-Catalyzed Coupling of *o*-Iodoanisoles and Terminal Alkynes, Followed by Electrophilic Cyclization

Dawei Yue, Tuanli Yao and Richard C. Larock*

Department of Chemistry, Iowa State University, Ames, IA 50011

larock@iastate.edu

General. ^1H and ^{13}C NMR spectra were recorded at 300 and 75.5 MHz or 400 and 100 MHz respectively. Thin layer chromatography was performed using commercially prepared 60-mesh silica gel plates, and visualization was effected with short wavelength UV light (254 nm) or a basic $KMnO_4$ solution [3 g of $KMnO_4$ + 20 g of K_2CO_3 + 5 mL of NaOH (5 %) + 300 mL of H_2O]. All melting points are uncorrected. Low resolution mass spectra were recorded on a Finnigan TSQ700 triple quadrupole mass spectrometer. High resolution mass spectra were recorded on a double focusing magnetic sector mass spectrometer using EI at 70 eV. All reagents were used directly as obtained commercially unless otherwise noted. Anhydrous forms of ethyl ether, hexanes, ethyl acetate, CH_2Cl_2 and DMF, 2-iodoanisole, 2-bromo-1,4-dimethoxybenzene, 1-bromo-2,4-dimethoxybenzene, 2-iodo-4-nitroanisole, 2-iodo-5-nitroanisole, resorcinol, 4-iodoanisole, 4-nitroiodobenzene, phenylacetylene, 1-decyne, 1-cyclohexenyl acetylene, 1-octyne, *tert*-butyl acetylene, trimethylsilyl acetylene, the palladium salts and Et_3N were purchased from supplier.

General procedure for the palladium/copper-catalyzed formation of the *o*-(1-alkynyl)anisoles. To a solution of Et_3N (12.5 mL), $PdCl_2(PPh_3)_2$ (2 mol %), 5 mmol of *o*-iodoanisole and 6 mmol of terminal acetylene (stirring for 5 min beforehand), CuI (1 mol %) was added and stirring was continued for another 2 min before flushing with Ar and the flask was then sealed. The mixture was allowed to stir at room temperature for 3-6 h and the resulting solution was filtered and washed with a saturated aq NaCl solution and extracted with diethyl ether (2 x 15 mL). The combined ether fractions were dried over anhydrous Na_2SO_4 and concentrated under vacuum to yield the crude product. The crude product was purified by flash chromatography on silica gel using ethyl acetate/hexane as the eluent.

2-(Phenylethynyl)anisole (1). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 3.90 (s, 3H), 6.89 (d, *J* = 8.4 Hz, 1H), 6.93 (t, *J* = 7.6 Hz, 1H), 7.29-7.33 (m, 4H), 7.50 (d, *J* = 7.2 Hz, 1H), 7.55-7.57 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 56.0, 85.9, 93.6, 110.9, 112.6, 120.7, 123.7, 128.2, 128.3, 129.9, 131.8, 133.7, 160.1; IR (neat, cm^{-1}) 3058, 2926, 2855, 2226; HRMS calcd for $C_{15}H_{12}O$ 208.0888, found 208.0894. The spectral properties were identical to those previously reported.[1]

2-[(Cyclohex-1-enyl)ethynyl]anisole (5). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) 7.05-7.26 (m, 4H), 6.20 (s, 1H), 3.77 (s, 3H), 2.09-2.35 (m, 4H), 1.20-1.67 (m, 4H); IR (neat, cm^{-1}) 3048, 2944, 2222; HRMS calcd for $C_{15}H_{16}O$ 212.1201, found 212.1206. The spectral properties were identical to those previously reported.[2]

2-[(4-Methoxyphenyl)ethynyl]anisole (8). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 3.77 (s, 3H), 3.87 (s, 3H), 6.83-6.93 (m, 4H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.46-7.50 (m, 3H); ^{13}C NMR ($CDCl_3$) δ 55.4, 55.9, 84.5, 93.6, 110.8, 112.8, 114.0, 115.8, 120.6, 129.6, 133.2, 133.5, 159.6, 159.9; IR (neat, cm^{-1}) 2227; HRMS calcd for $C_{16}H_{14}O_2$ 238.0994, found 238.1000. The spectral properties were identical to those previously reported.[3]

4-Methoxy-2-(phenylethynyl)anisole (11). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 3.76 (s, 3H), 3.86 (s, 3H), 6.80-6.84 (m, 2H), 7.05 (s, 1H), 7.30-7.34 (m, 3H), 7.56 (d, *J* = 6.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 56.0, 56.7, 85.9, 93.6, 112.3, 113.1, 115.9, 118.3, 123.6, 128.4, 128.4, 131.8, 153.4, 154.7; IR (neat, cm^{-1}) 3058, 2926, 2855, 2227, 1464, 1435, 749; HRMS calcd for $C_{16}H_{14}O_2$ 238.0994, found 238.0999. The spectral properties were identical to those previously reported.[4]

4-Nitro-2-(phenylethynyl)anisole (14). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 4.01 (s, 3H), 6.96 (d, *J* = 9.2 Hz, 1H), 7.36-7.38 (m, 3H), 7.56-7.58 (m, 2H), 8.20 (dd, *J* = 9.2, 2.8 Hz, 1H), 8.38 (d, *J* = 2.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 56.8, 83.5, 95.6, 110.5, 113.9, 122.7, 125.7, 128.6, 129.1, 129.2, 132.0, 141.3, 164.6; IR (neat, cm^{-1}) 3058, 2926, 2855, 2225; HRMS calcd for $C_{15}H_{11}NO_3$ 253.0739, found 253.0742.

5-Nitro-2-(phenylethynyl)anisole (16). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 4.01 (s, 3H), 7.37-7.38 (m, 3H), 7.57-7.62 (m, 3H), 7.75 (d, *J* = 2.0 Hz, 1H), 7.83 (dd, *J* = 8.4, 2.0 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 56.6, 84.3, 98.6, 105.8, 115.9, 119.9, 122.7, 128.6, 129.3, 132.1, 133.7, 148.2, 160.3; IR (neat, cm^{-1}) 3047, 2215; HRMS calcd for $C_{15}H_{11}NO_3$ 253.0739, found 253.0741.

3-Methoxy-6-methyl-2-(phenylethynyl)pyridine (18). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 2.52 (s, 3H), 3.90 (s, 3H), 7.06-7.15 (m, 2H), 7.33-7.36 (m, 3H), 7.61-7.64 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 55.8, 85.7, 93.7, 118.8, 122.9, 123.5, 128.4, 128.9, 132.3, 150.6, 155.3; IR (neat, cm^{-1}) 2217; HRMS calcd for $C_{15}H_{13}NO$ 233.0997, found 233.1002.

2-Acetoxy-2’-methoxydiphenylacetylene (20). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 2.37 (s, 3H), 3.87 (s, 3H), 6.88 (d, *J* = 8.7 Hz, 1H), 6.92 (td, *J* = 7.5, 0.9 Hz, 1H), 7.11 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.20 (td, *J* = 7.5, 1.5 Hz, 1H), 7.26-7.35 (m, 2H), 7.46 (dd, *J* = 7.5, 1.8 Hz, 1H), 7.59 (dd, *J* = 7.5, 1.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 21.0, 55.8, 88.3, 90.9, 110.9, 112.3, 118.0, 120.6, 122.4, 126.0, 129.4, 130.2, 133.1, 133.7, 151.6, 160.1, 169.1; IR (neat, cm^{-1}) 2213, 1770; HRMS calcd for $C_{17}H_{14}O_3$ 266.0943, found 266.0946.

1,4-Bis[(2-methoxyphenyl)ethynyl]benzene (22). The product was obtained as a yellow oil: ^1H NMR ($CDCl_3$) δ 3.91 (s, 6H), 6.88-6.96 (m, 4H), 7.30 (t, J = 7.6 Hz, 2H), 7.48-7.53 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 56.0, 87.7, 93.4, 110.8, 112.4, 120.7, 123.4, 130.1, 131.7, 133.7, 160.1; IR (neat, cm^{-1}) 2210; HRMS calcd for $C_{24}H_{18}O_2$ 338.13068, found 338.13150.

2,5-Dimethoxy-1,4-di(phenylethynyl)benzene (24). The product was obtained as a yellow oil: ^1H NMR ($CDCl_3$) δ 3.90 (s, 6H), 7.04 (s, 2H), 7.32-7.37 (m, 6H), 7.56-7.59 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 56.7, 85.9, 95.2, 113.6, 115.9, 123.4, 128.5, 128.6, 131.9, 154.1; IR (neat, cm^{-1}) 2227; HRMS calcd for $C_{24}H_{18}O_2$ 338.13068, found 338.13151. The spectral properties were identical to those previously reported.[5]

2,4-Dimethoxy-1-(phenylethynyl)benzene (26). The product was obtained as a yellow oil: ^1H NMR ($CDCl_3$) δ 3.79 (s, 3H), 3.87 (s, 3H), 6.45 (s, 1H), 6.48 (d, J = 6.0 Hz, 1H), 7.27-7.34 (m, 3H), 7.42 (d, J = 9.0 Hz, 1H), 7.54 (d, J = 9.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 55.5, 56.0, 86.0, 92.1, 98.6, 105.0, 105.1, 124.0, 127.9, 128.4, 131.6, 134.5, 161.3, 161.4; IR (neat, cm^{-1}) 3058, 2926, 2855, 2227, 1464, 1435, 749; HRMS calcd for $C_{16}H_{14}O_2$ 238.0994, found 238.1000. The spectral properties were identical to those previously reported.[4]

2-(1-Octynyl)anisole (27). The product was obtained as a yellow oil: ^1H NMR ($CDCl_3$) δ 0.88-0.93 (m, 3H), 1.30-1.34 (m, 4H), 1.46-1.50 (m, 2H), 1.60-1.65 (m, 2H), 2.47 (t, J = 7.2 Hz, 2H), 3.87 (s, 3H), 6.85 (d, J = 8.4 Hz, 1H), 6.88 (td, J = 7.5, 0.9 Hz, 1H), 7.24 (td, J = 8.1, 1.8 Hz, 1H), 7.37 (dd, J = 7.5, 1.8 Hz, 1H); HRMS calcd for

$C_{15}H_{20}O$ 216.1514, found 216.1519. The spectral properties were identical to those previously reported.[2]

2-[(4-Nitrophenyl)ethynyl]anisole (28). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 3.92 (s, 3H), 6.92-6.99 (m, 2H), 7.37 (t, *J* =8.4 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.70 (d, *J* = 8.8 Hz, 2H), 8.19 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 56.0, 91.6, 91.4, 110.9, 111.4, 120.8, 123.7, 130.8, 131.1, 132.4, 133.9, 147.0, 160.4; HRMS calcd for $C_{15}H_{11}NO_2$ 253.0739, found 253.0745.

2-(Trimethylsilylethynyl)anisole (29). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.08 (s, 9H), 3.68 (s, 3H), 6.64-6.71 (m, 2H), 7.08 (td, *J* = 8.4, 2.0 Hz, 1H), 7.24 (dd, *J* = 7.6, 1.6 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 0.3, 56.0, 98.6, 101.5, 110.8, 112.5, 120.5, 130.2, 134.4, 160.5. The spectral properties were identical to those previously reported.[6]

2,4-Dimethoxy-5-(phenylethynyl)pyrimidine (30). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 4.01 (s, 3H), 4.06 (s, 3H), 7.32-7.35 (m, 3H), 7.51-7.54 (m, 2H), 8.40 (s, 1H); ^{13}C NMR ($CDCl_3$) δ 54.6, 55.2, 80.8, 95.5, 100.2, 122.9, 128.4, 128.6, 131.6, 161.3, 164.2, 170.4; IR (neat, cm^{-1}) 2207 cm^{-1}; HRMS calcd for $C_{14}H_{12}N_2O_2$ 240.0899, found 240.0901. The spectral properties were identical to those previously reported.[7]

2-(1-Decynyl)-3-methoxy-6-methyl-pyridine (32). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.28-1.33 (m, 8H), 1.43-1.48 (m, 2H), 1.61-1.68 (m, 2H), 2.47 (s, 3H), 2.49 (t, *J* = 7.2 Hz, 2H), 3.85 (s, 3H), 7.10 (d, *J* = 8.7 Hz, 1H), 7.73 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.2, 19.9, 22.8, 23.5, 28.6, 29.2, 29.2, 29.3, 31.9, 56.0, 77.1, 95.7, 118.5, 122.7, 133.0, 150.1, 154.8; IR (neat, cm^{-1}) 2217; HRMS calcd for $C_{17}H_{25}NO$ 259.1936, found 259.1941.

General procedure for the iodocyclizations. To a solution of 0.25 mmol of the *o*-(1-alkynyl)anisole and 3 mL of CH_2Cl_2, 2 equiv of I_2 or 1.2 equiv of ICl dissolved in 2 mL of CH_2Cl_2 was added gradually. The reaction mixture was flushed with Ar and allowed to stir at room temperature for 3 h or 12 h. The excess I_2 or Br_2 was removed by washing with a satd aq $Na_2S_2O_3$ solution. The mixture was then extracted by diethyl ether (2 x 10 mL). The combined ether layers were dried over anhydrous Na_2SO_4 and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel using ethyl acetate/hexanes as the eluent.

3-Iodo-2-phenylbenzo[*b*]furan (2). The product was obtained as a yellow oil: ^1H NMR ($CDCl_3$) δ 7.28-7.51 (m, 7H), 8.16-8.19 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 61.3, 111.4, 122.1, 123.7, 125.9, 127.7, 128.7, 129.4, 130.2, 132.7, 153.2, 154.1; IR (neat, cm^{-1}) 3058, 1450; HRMS calcd for $C_{14}H_{18}INO_3$ 364.9549, found 364.9551. The spectral properties were identical to those previously reported.[8]

2-(Cyclohex-1-enyl)-3-iodobenzo[*b*]furan (6). The product was obtained as a yellow oil: ^1H NMR ($CDCl_3$) δ 1.66-1.70 (m, 2H), 1.77-1.79 (m, 2H), 2.25-2.29 (m, 2H), 2.59-2.64 (m, 2H), 6.77-6.79 (m, 2H), 7.24-7.28 (m, 2H), 7.35-7.38 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 21.9, 22.7, 25.9, 26.9, 59.1, 111.0, 121.7, 123.3, 125.2, 128.2, 132.0, 132.2, 153.5, 155.3; IR (neat, cm^{-1}) 3058, 2199, 1475; HRMS calcd for $C_{14}H_{13}IO$ 324.0011, found 324.0020.

3-Iodo-2-(4-methoxyphenyl)benzo[*b*]furan (9). The product was obtained as a yellow oil: ^1H NMR (400 MHz, $CDCl_3$) δ 3.84 (s, 3H), 6.99 (d, J = 8.0 Hz, 2H), 7.28-

7.31 (m, 2H), 7.39-7.45 (m, 2H), 8.10 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.5, 59.7, 111.2, 114.1, 121.7, 122.8, 123.6, 125.4, 129.2, 132.8, 153.4, 153.9, 160.5; IR (neat, cm^{-1}) 3058, 2227, 1464; HRMS calcd for $C_{15}H_{11}IO_2$ 349.98038, found 349.98100.

3-Iodo-5-methoxy-2-phenylbenzo[*b*]furan (12). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 3.89 (s, 3H), 6.88 (s, 1H), 6.94 (d, *J* = 8.5 Hz, 1H), 7.36 (d, *J* = 8.8 Hz, 1H), 7.41 (t, *J* = 7.2 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 8.14 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 56.2, 61.3, 104.1, 112.1, 114.9, 127.6, 128.7, 129.4, 130.3, 133.3, 149.0, 154.0, 156.9; IR (neat, cm^{-1}) 3058, 2926, 2855, 2227, 1464, 1435, 749; HRMS calcd for $C_{15}H_{11}O_2I$ 349.9804, found 349.9810.

3-Iodo-5-nitro-2-phenylbenzo[*b*]furan (15). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 7.50-7.58 (m, 4H), 8.15-8.18 (m, 2H), 8.27 (dd, *J* = 12.0, 3.2 Hz, 1H), 8.37 (d, *J* = 3.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 60.9, 111.9, 118.7, 121.5, 127.8, 128.9, 129.0, 130.4, 133.6, 144.9, 156.5, 157.0; IR (neat, cm^{-1}) 3058, 2926; HRMS calcd for $C_{14}H_{18}INO_3$ 364.9549, found 364.9551.

3-Iodo-6-nitro-2-phenylbenzo[*b*]furan (17). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 7.50-7.56 (m, 4H), 8.18-8.21 (m, 2H), 8.23 (dd, *J* = 8.4, 2.0 Hz, 1H), 8.39 (d, *J* = 2.0 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 60.6, 107.8, 119.5, 122.1, 128.0, 129.0, 129.0, 130.7, 138.5, 146.1, 152.6, 158.4; IR (neat, cm^{-1}) 3058, 2926; HRMS calcd for $C_{14}H_{18}INO_3$ 364.9549, found 364.9552.

3-Iodo-5-methyl-2-phenylfuro[3,3-*b*]pyridine (19). The product was obtained as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 2.73 (s, 3H), 7.14 (d, *J* = 8.4 Hz, 1H), 7.46-7.54 (m, 3H), 7.62 (d, *J* = 8.4 Hz, 1H), 8.21-8.24 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 24.7, 64.4, 118.5, 120.6, 127.8, 128.8, 129.9, 130.0, 145.8, 148.9, 156.0, 156.2; IR (neat, cm^{-1}) 1775; HRMS calcd for $C_{14}H_{10}NIO$ 334.9808, found 334.9809.

2-(2-Acetoxyphenyl)-3-iodobenzo[*b*]furan (21). The product was obtained as a yellow oil: ^1H NMR ($CDCl_3$) δ 2.21 (s, 3H), 7.25 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.32-7.40 (m, 3H), 7.44-7.53 (m, 3H), 7.82 (dd, *J* = 7.8, 1.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 21.3, 65.4, 111.4, 122.1, 123.3, 123.7, 123.8, 126.0, 126.1, 131.2, 131.7, 131.9, 148.8, 152.3, 154.6, 169.4; IR (neat, cm^{-1}) 1771; HRMS calcd for $C_{16}H_{11}IO_3$ 377.9753, found 377.9758.

2,2'-(1,4-Phenylene)-bis-3-iodobenzofuran (23). The product was obtained as a white solid: mp > 210 ºC (decompose); ^1H NMR ($CDCl_3$) δ 7.34-7.42 (m, 4H), 7.47-7.53 (m, 4H), 8.36 (s, 4H); ^{13}C NMR ($CDCl_3$) δ 62.3, 111.5, 122.2, 123.9, 126.3, 127.5, 130.7, 132.8, 152.5, 154.2; IR (CH_2Cl_2) 3058, 2926; HRMS calcd for $C_{22}H_{12}I_2O_2$ 561.8927, found 561.8934.

3,7-Diiodo-2,6-diphenyl-benzo[1,2-*b*;4,5-*b*')difuran (25). The compound was obtained as a white solid: mp > 320 ºC (decompose); ^1H NMR (CD_2Cl_2) δ 7.48-7.59 (m, 7H), 8.23-8.26 (m, 5H); HRMS calcd for $C_{22}H_{12}I_2O_2$ 561.8927, found 561.8934. ^{13}C NMR could not be obtained because of the poor solubility of the product; IR (CH_2Cl_2) 3058, 2926.

2-(1,2-Diiodo-1-decenyl)-3-methoxy-6-methylpyridine (33). The product was obtained as a yellow oil: ^1H NMR ($CDCl_3$) δ 0.88-0.92 (m, 3H), 1.25-1.44 (m, 10H), 1.65-1.70 (m, 2H), 2.50 (s, 3H), 2.72-2.77 (m, 1H), 2.84-2.89 (m, 1H), 3.86 (s, 3H), 7.10 (dd, *J* = 13.5, 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 14.3, 22.9, 23.7, 28.5, 28.6, 29.4, 29.7, 32.1, 49.1, 56.1, 91.0, 107.4, 120.1, 124.1, 149.5, 149.7, 151.9; IR (neat, cm^{-1}) 1715.

The products in entries 17, 19 and 24 could not be fully characterized because of their instability.

General procedure for the *p*-$O_2NC_6H_4SCl$ and PhSeCl cyclizations. To a solution of 0.25 mmol of the *o*-(1-alkynyl)anisole and CH_2Cl_2 (5 mL), 0.375 mmol of *p*-$O_2NC_6H_4SCl$ or PhSeCl dissolved in CH_2Cl_2 (2 mL) was added. The mixture was flushed with Ar and allowed to stir at 25 ^oC for 3 h. The reaction mixture was then washed with 20 mL of water and extracted with diethyl ether. The combined ether layers were dried over anhydrous Na_2SO_4 and concentrated under vacuum to yield the crude product, which was further purified by flash chromatography on silica gel using ethyl acetate/hexanes as the eluent.

2-Phenyl-3-(phenylselenyl)benzo[*b*]furan (3). The product was obtained as a yellow oil: ^1H NMR ($CDCl_3$) δ 7.13-7.17 (m, 3H), 7.22 (t, *J* = 4.0 Hz, 1H), 7.27-7.32 (m, 4H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 1H), 8.19-8.21 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 99.9, 111.4, 121.4, 123.6, 125.4, 126.4, 128.0, 128.7, 129.4, 129.5, 130.3, 131.6, 132.1, 154.3, 157.4; IR (neat, cm^{-1}) 3058, 2926; HRMS calcd for $C_{20}H_{14}OSe$ 350.0211, found 350.0220.

2-Phenyl-3-(*p*-nitrophenylsulfenyl)benzo[*b*]furan (4). The product was obtained as a yellow oil: ^1H NMR ($CDCl_3$) δ 7.22 (d, *J* = 8.8 Hz, 2H), 7.26 (d, *J* = 7.6 Hz, 1H), 7.35-7.46 (m, 5H), 7.60 (d, *J* = 8.0 Hz, 1H), 8.02 (d, *J* = 8.8 Hz, 2H), 8.13 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 102.1, 111.9, 120.1, 124.1, 124.4, 125.9, 126.0, 127.6, 128.9, 129.3, 130.1, 130.1, 145.6, 146.4, 154.2, 158.6; IR (neat, cm^{-1}) 3058, 2926; HRMS calcd for $C_{20}H_{13}NO_3S$ 347.0617, found 347.0622.

2-(Cyclohex-1-enyl)-3-(phenylselenyl)benzofuran (7). The product was obtained as a yellow oil: ^{1}H NMR (CDCl$_3$) δ 1.64-1.66 (m, 2H), 1.72-1.74 (m, 2H), 2.19-2.21 (m, 2H), 2.50-2.51 (m, 2H), 6.09-6.10 (m, 1H), 7.10-7.15 (m, 5H), 7.28-7.31 (m, 2H), 7.76-7.78 (m, 2H); ^{13}C NMR (CDCl$_3$) δ 21.9, 23.0, 25.9, 30.4, 114.0, 122.2, 124.7, 125.0, 126.0, 129.3, 131.7, 132.0, 133.3, 138.4, 142.0, 152.1; IR (neat, cm^{-1}) 3025; HRMS calcd for $C_{20}H_{18}OSe$ 354.05240, found 354.05330.

2-(4-Methoxyphenyl)-3-(phenylselenyl)benzo[*b*]furan (10). The product was obtained as a yellow oil: ^{1}H NMR (400 MHz, CDCl$_3$) δ 3.82 (s, 3H), 6.95 (d, *J* = 8.0 Hz, 2H), 7.11-7.23 (m, 4H), 7.26-7.53 (m, 3H), 7.48-7.53 (dd, *J* = 8.4, 7.6 Hz, 2H), 8.15 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 55.1, 98.0, 111.2, 114.1, 121.1, 123.0, 123.5, 125.0, 126.3, 129.1, 129.5, 131.8, 132.3, 154.1, 157.7, 160.6; HRMS calcd for $C_{21}H_{16}SeO_2$ 380.0317, found 380.0328.

5-Methoxy-2-phenyl-3-(phenylselenyl)benzo[*b*]furan (13). The product was obtained as a yellow oil: ^{1}H NMR (400 MHz, CDCl$_3$) δ 3.75 (s, 3H), 6.90-6.93 (m, 2H), 7.12-7.17 (m, 3H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.36-7.44 (m, 4H), 7.18 (d, *J* = 2.0 Hz, 2H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 56.0, 99.6, 103.3, 112.0, 114.5, 126.4, 127.9, 128.6, 129.3, 129.4, 129.5, 130.4, 131.6, 132.9, 149.2, 156.7, 158.2; IR (neat, cm^{-1}) 3058, 2926, 2855, 2227, 1464, 1435, 749; HRMS calcd for $C_{21}H_{16}O_2Se$ 380.03166, found 380.03279.

The product in entry 18 could not be fully characterized because of its instability.

References

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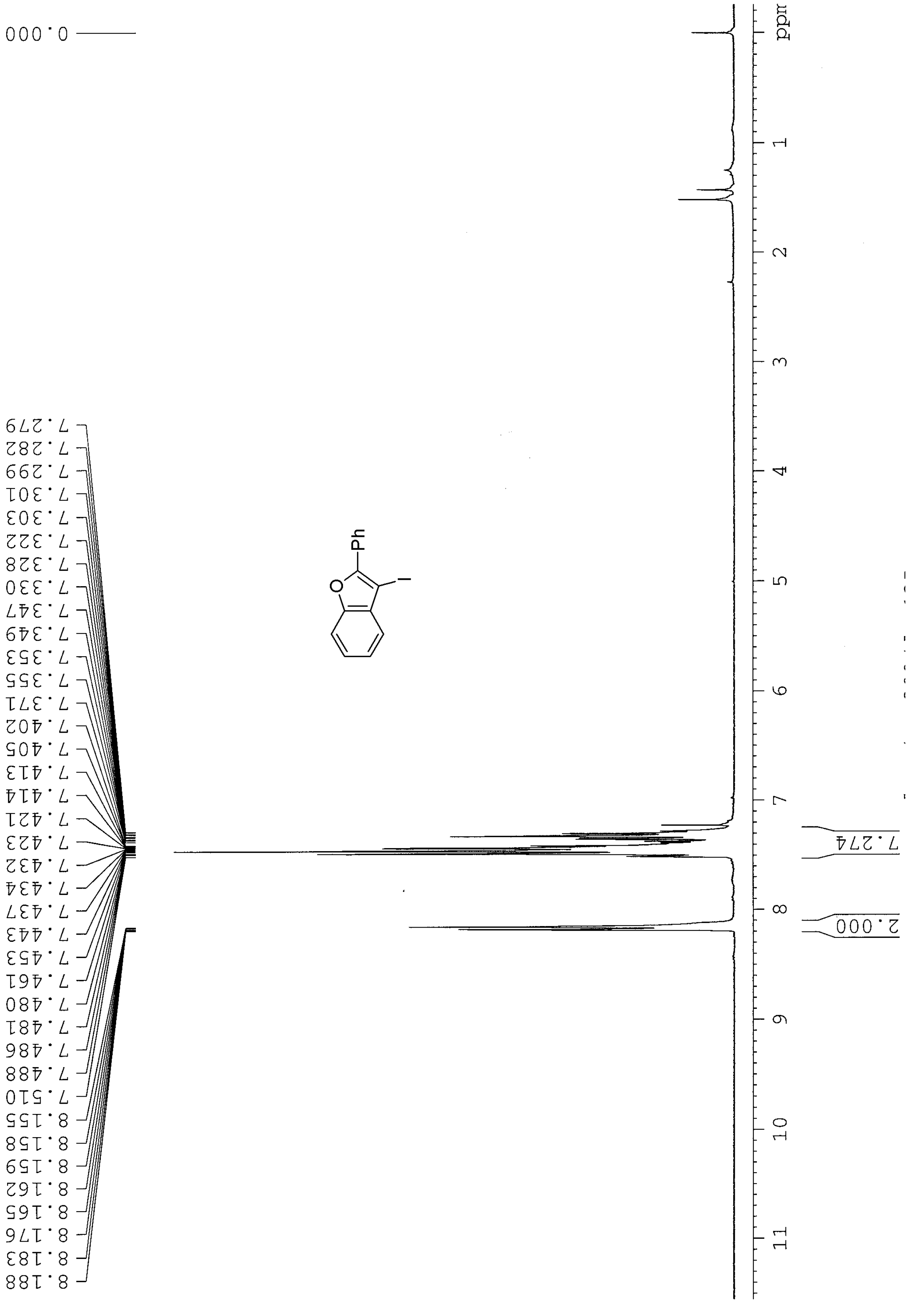
8. Arcadi, A.; Cacchi, S.; Giancarlo, F.; Marinelli, F.; Moro, L. *Synlet.* **1999**, 1432.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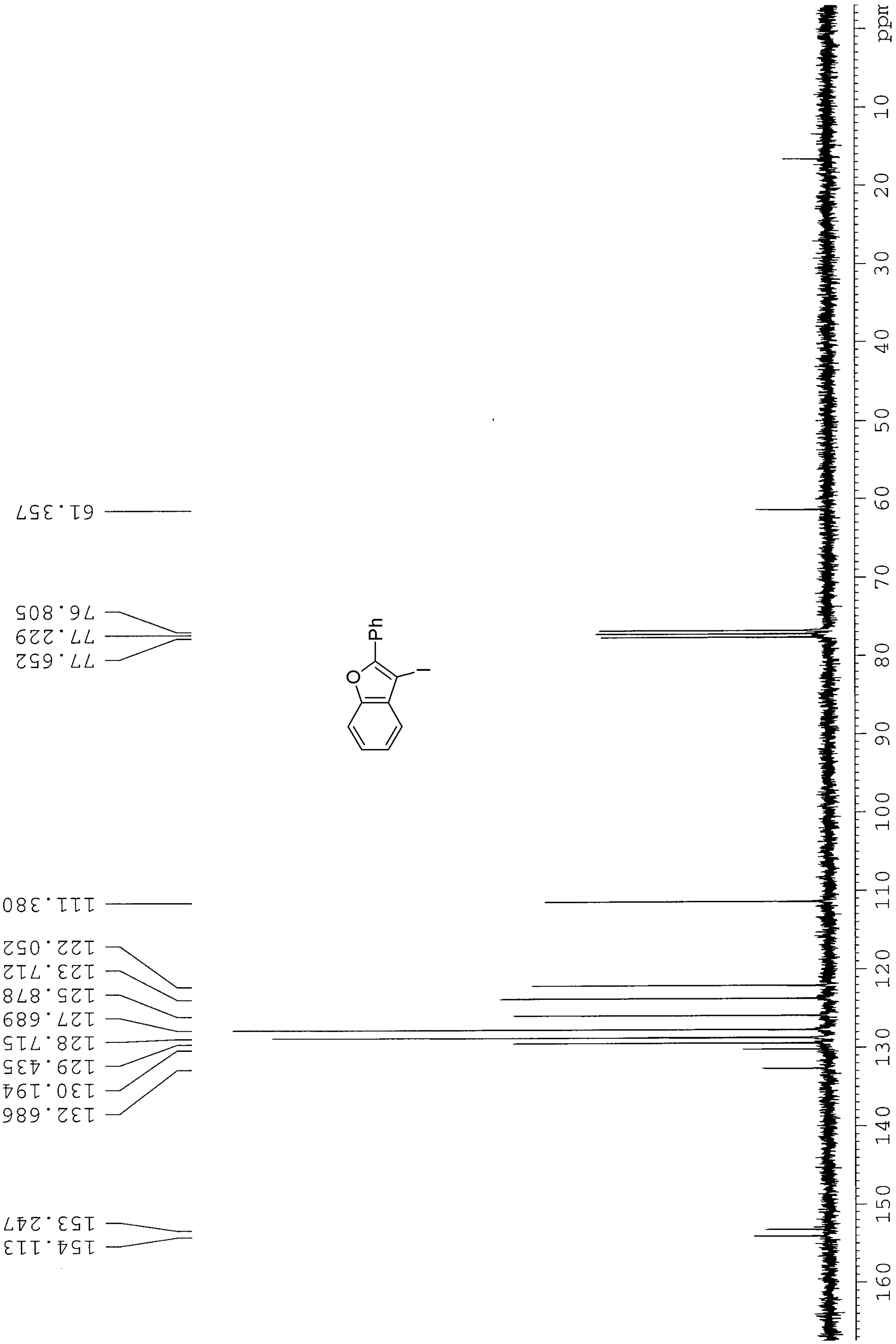
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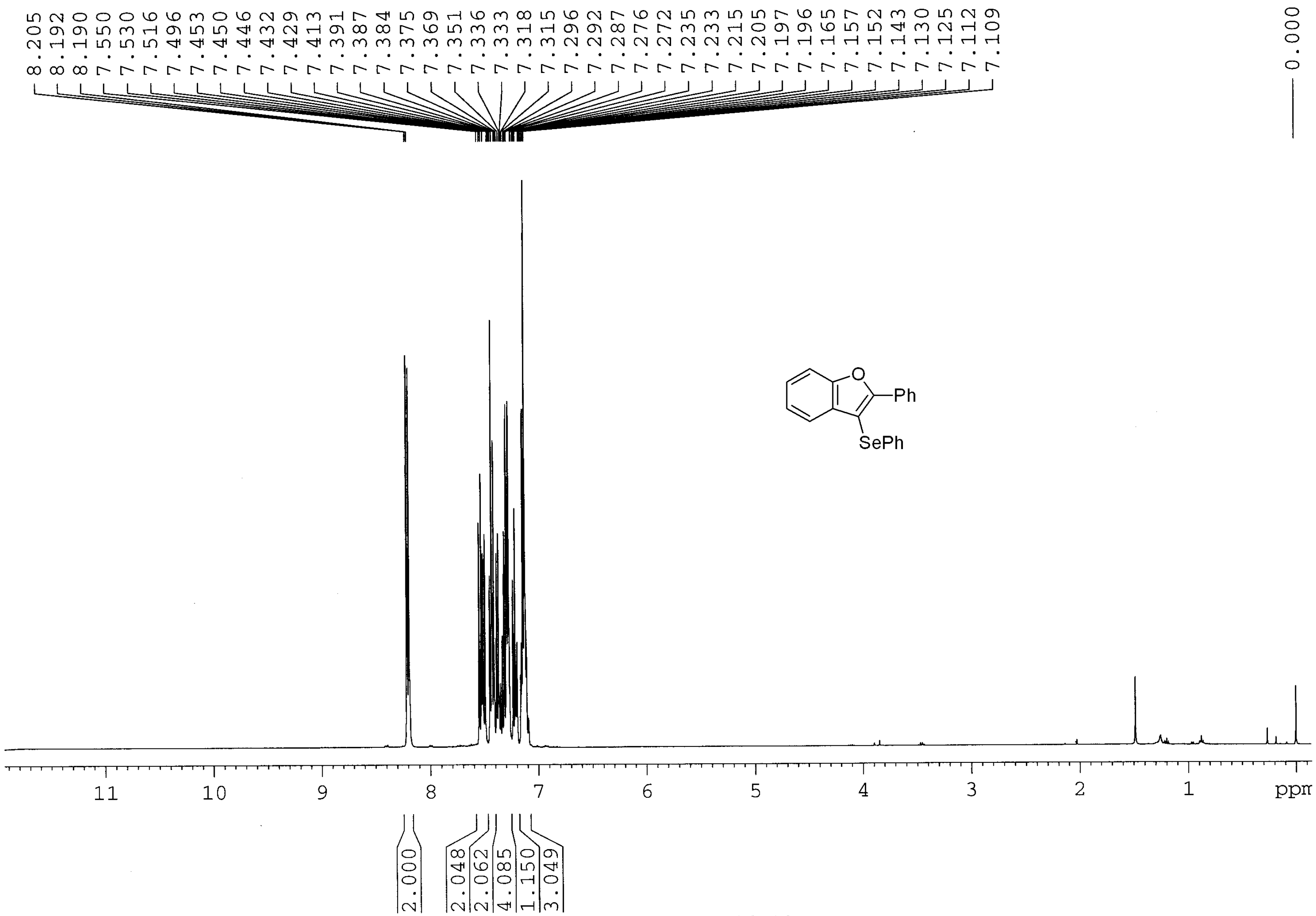
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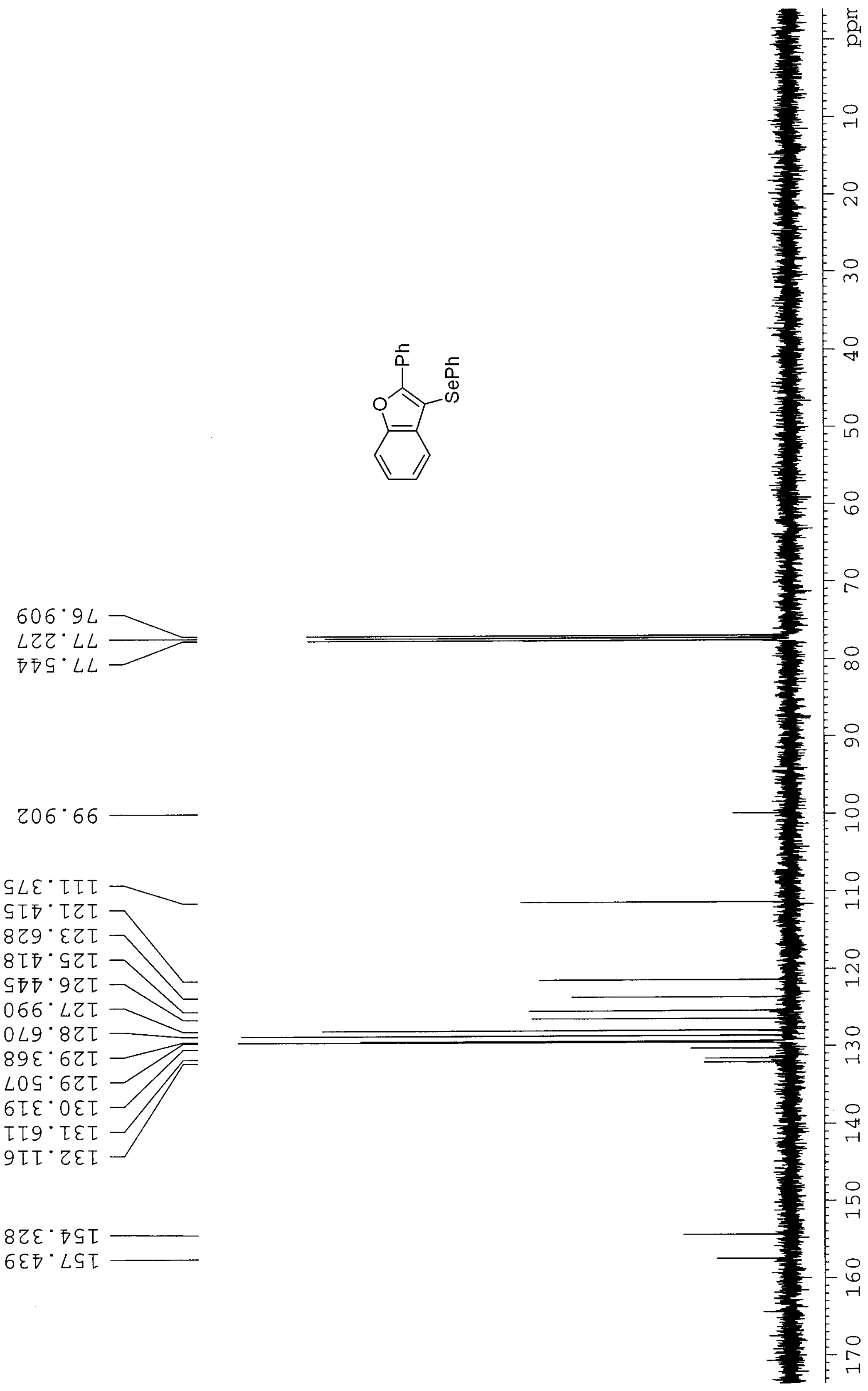

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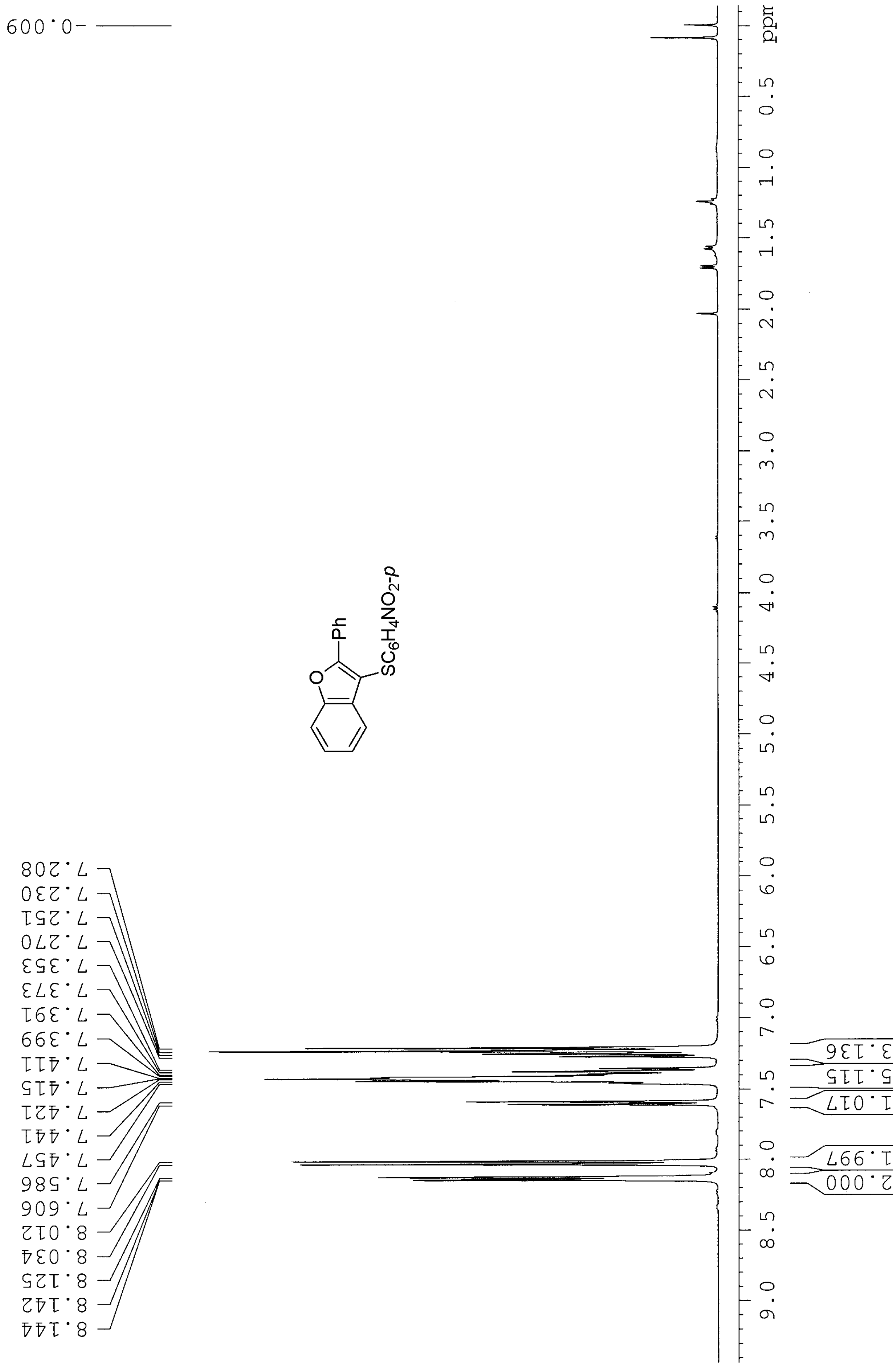

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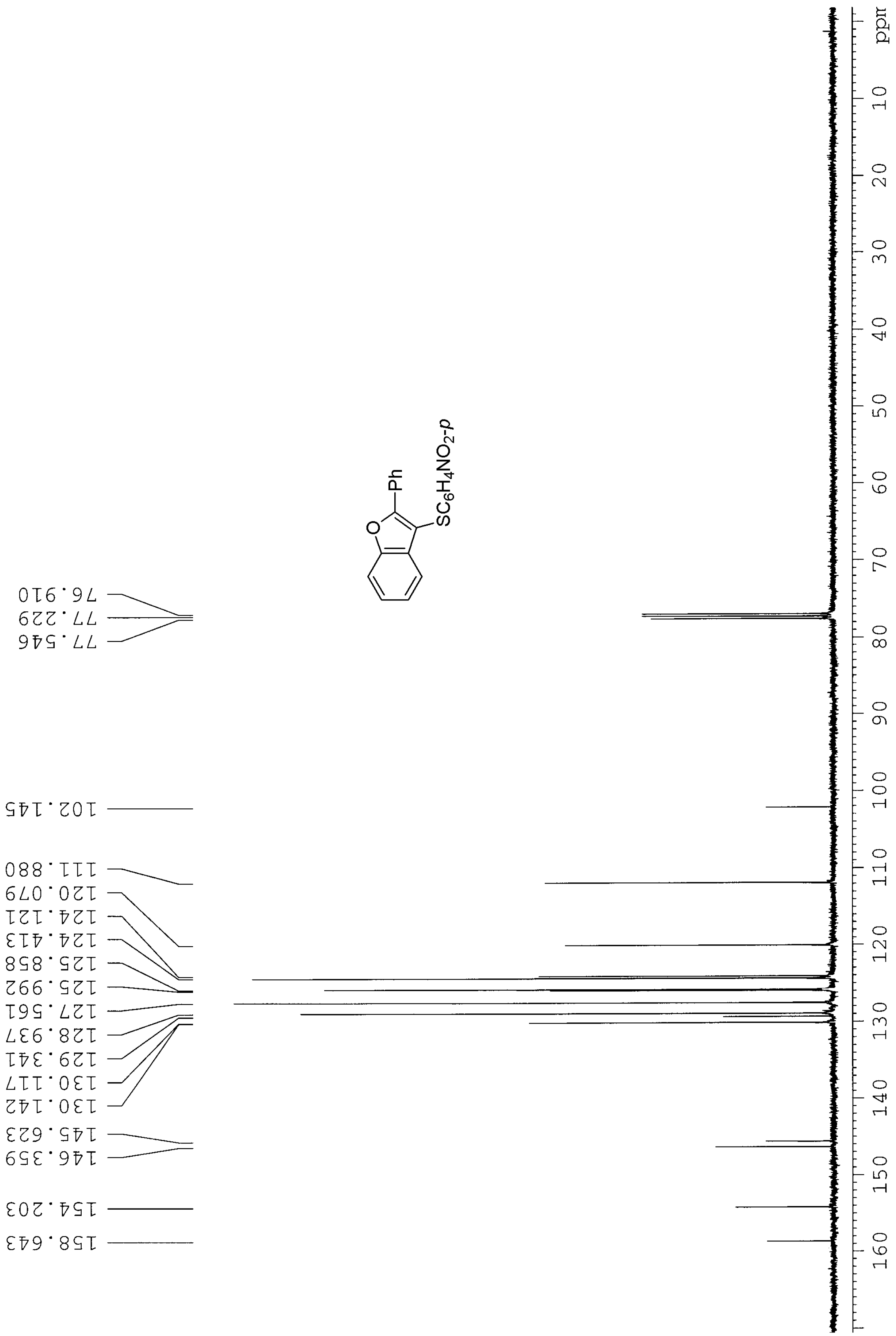
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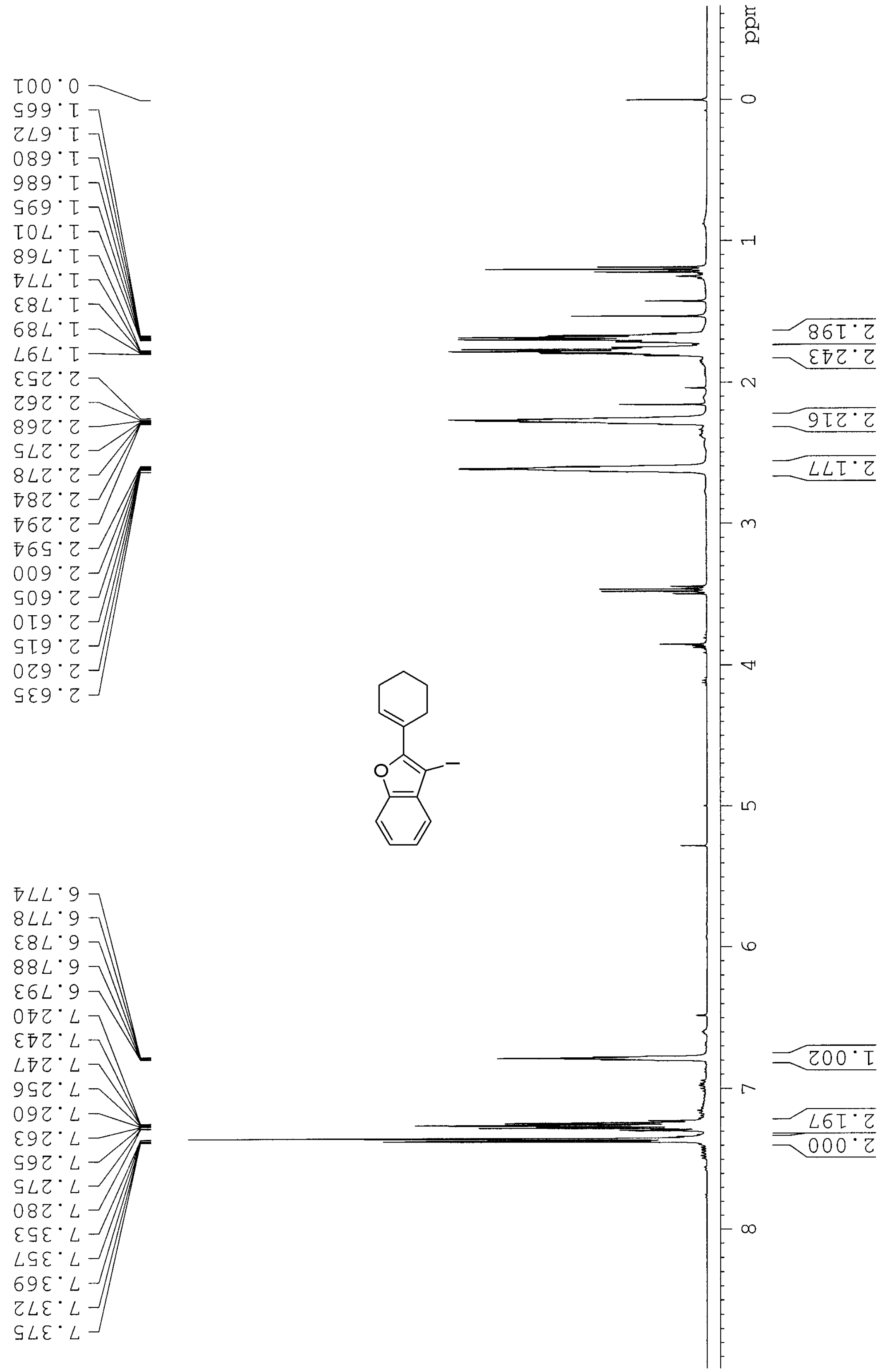

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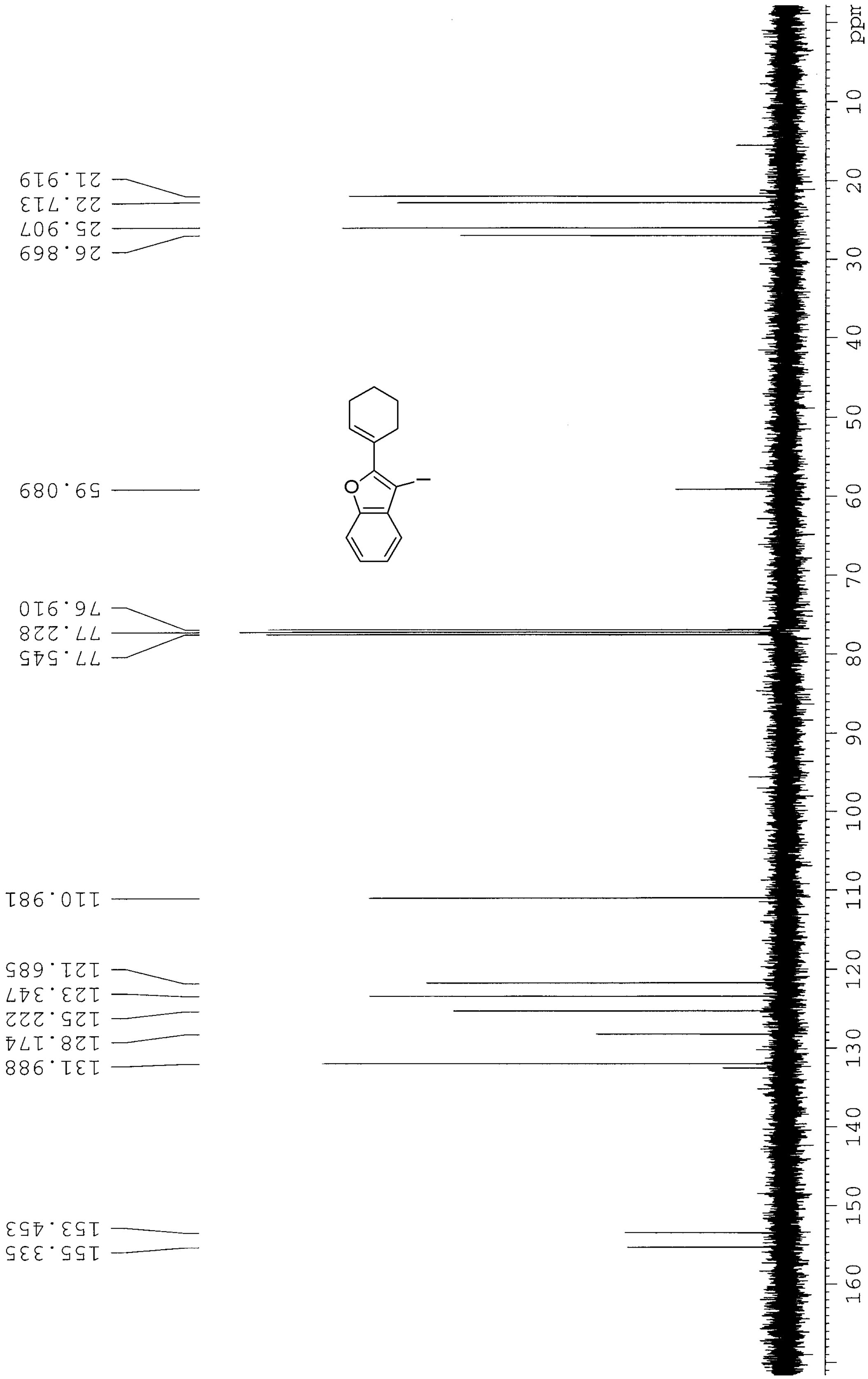
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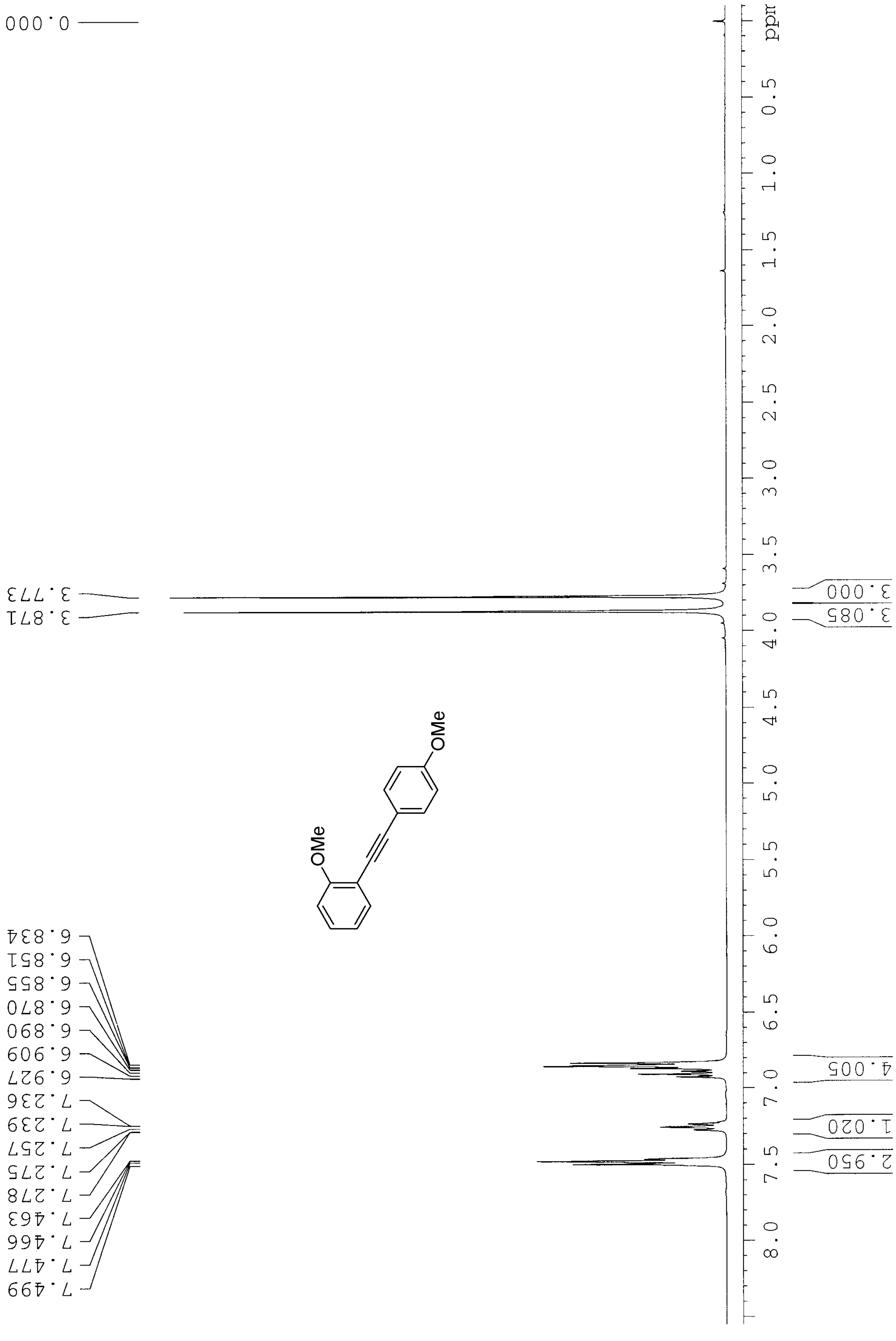
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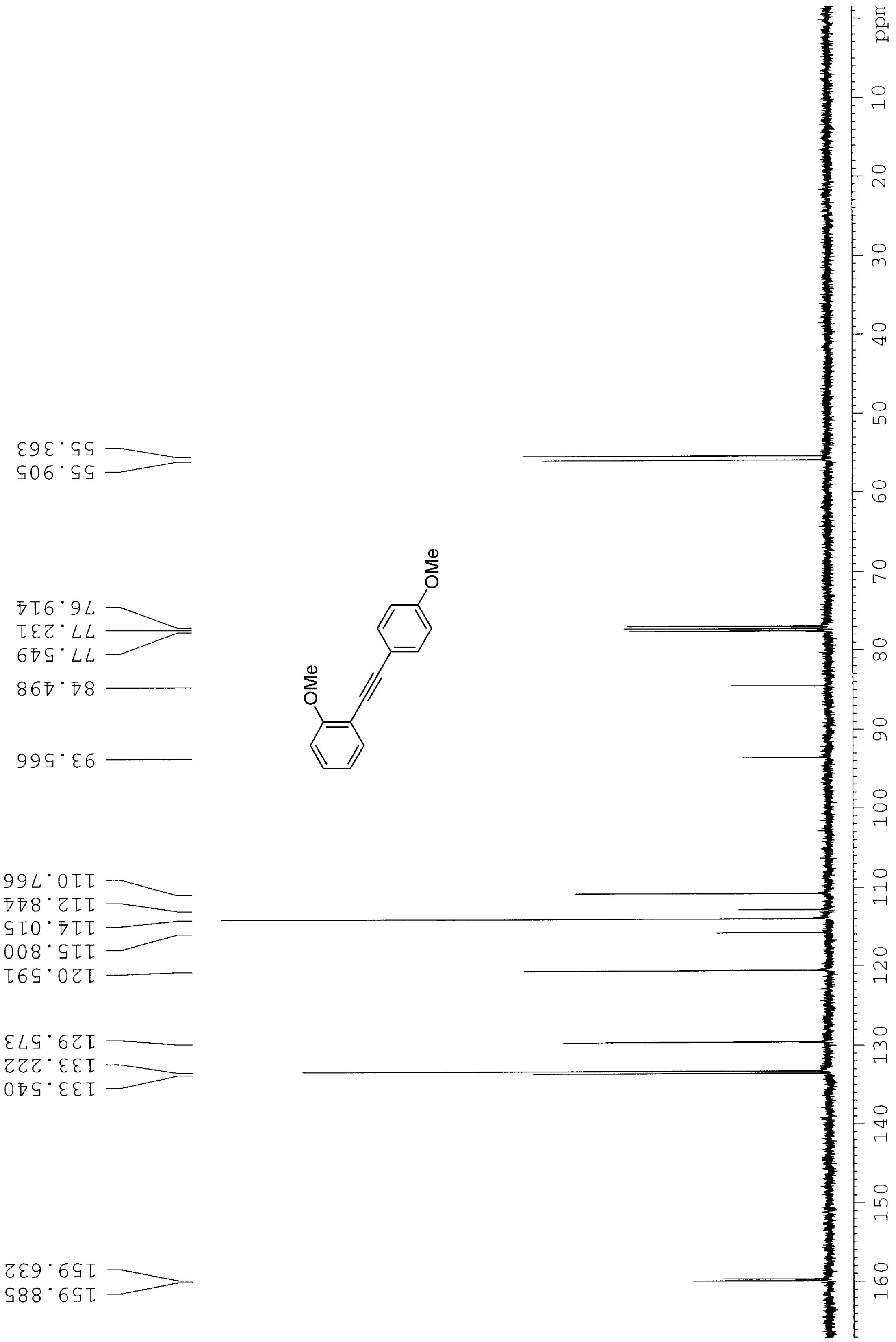
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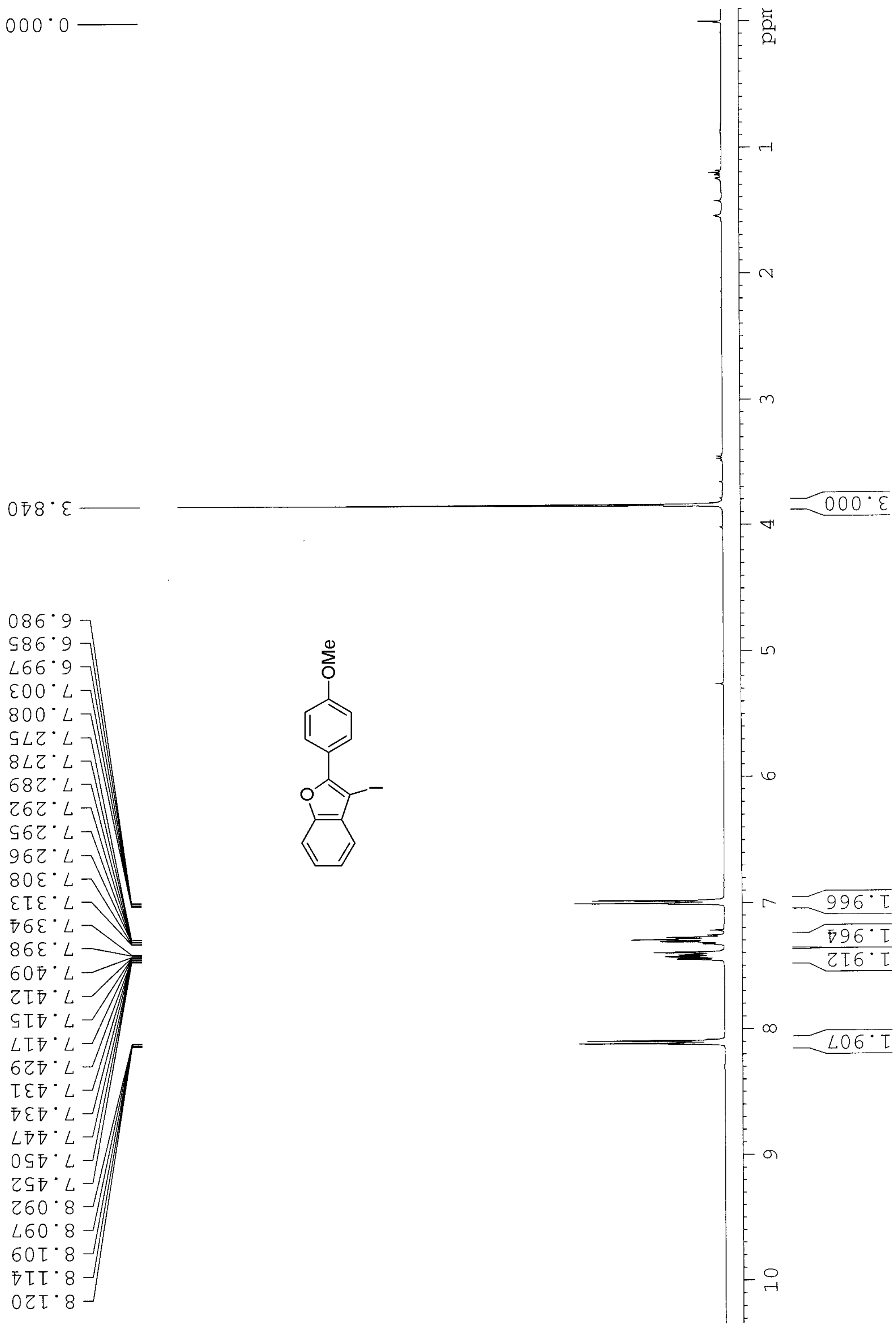
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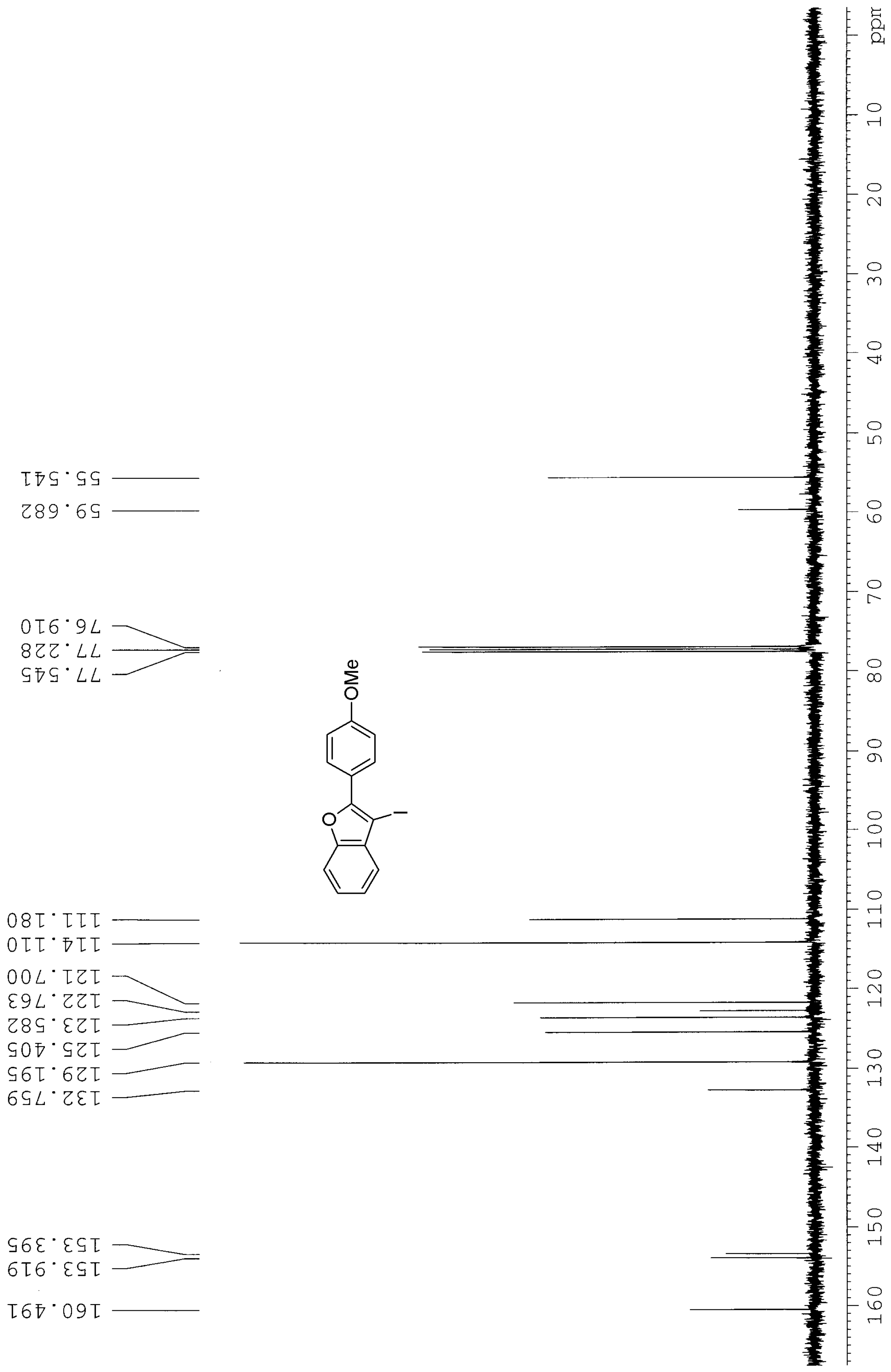
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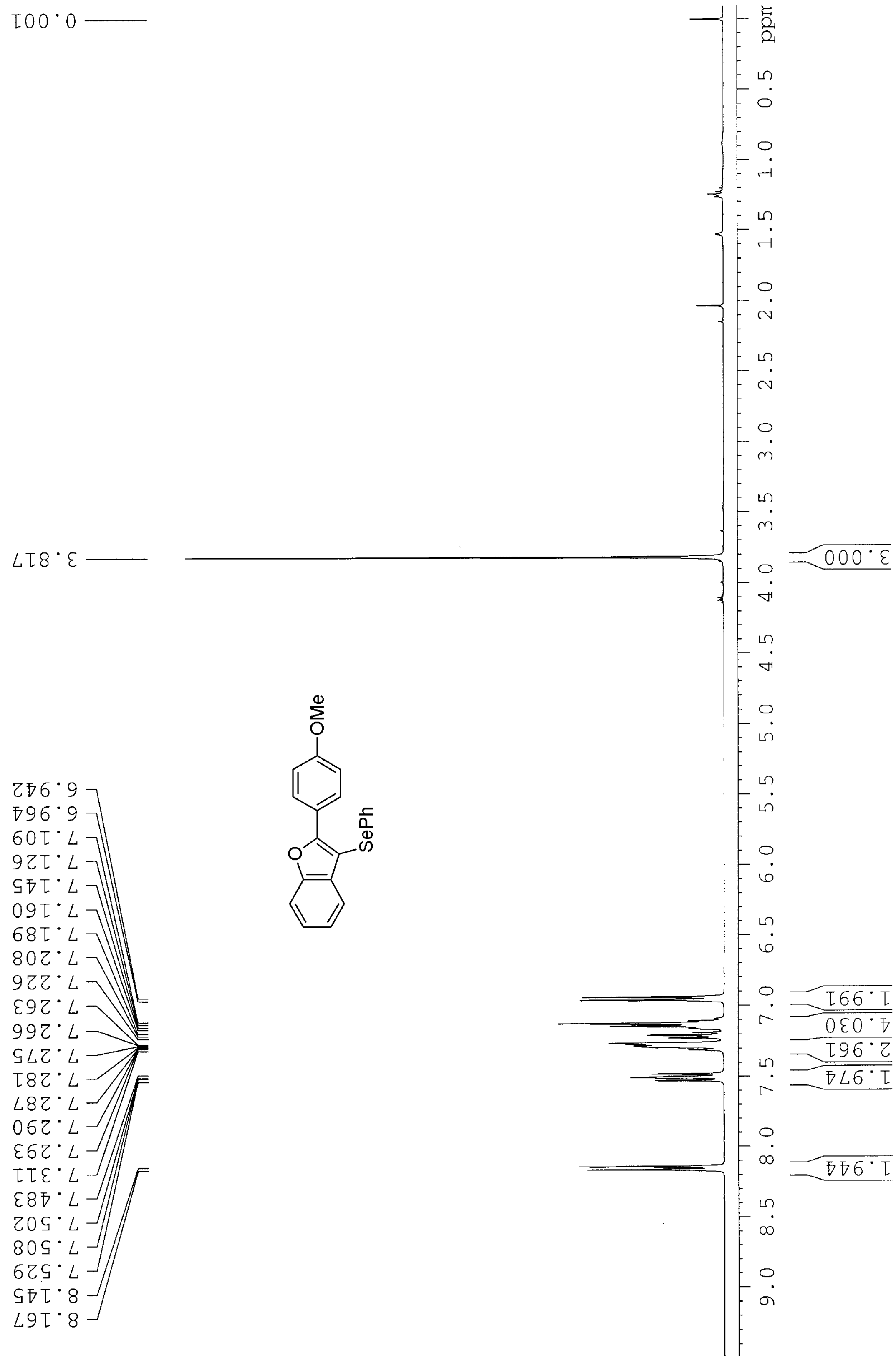
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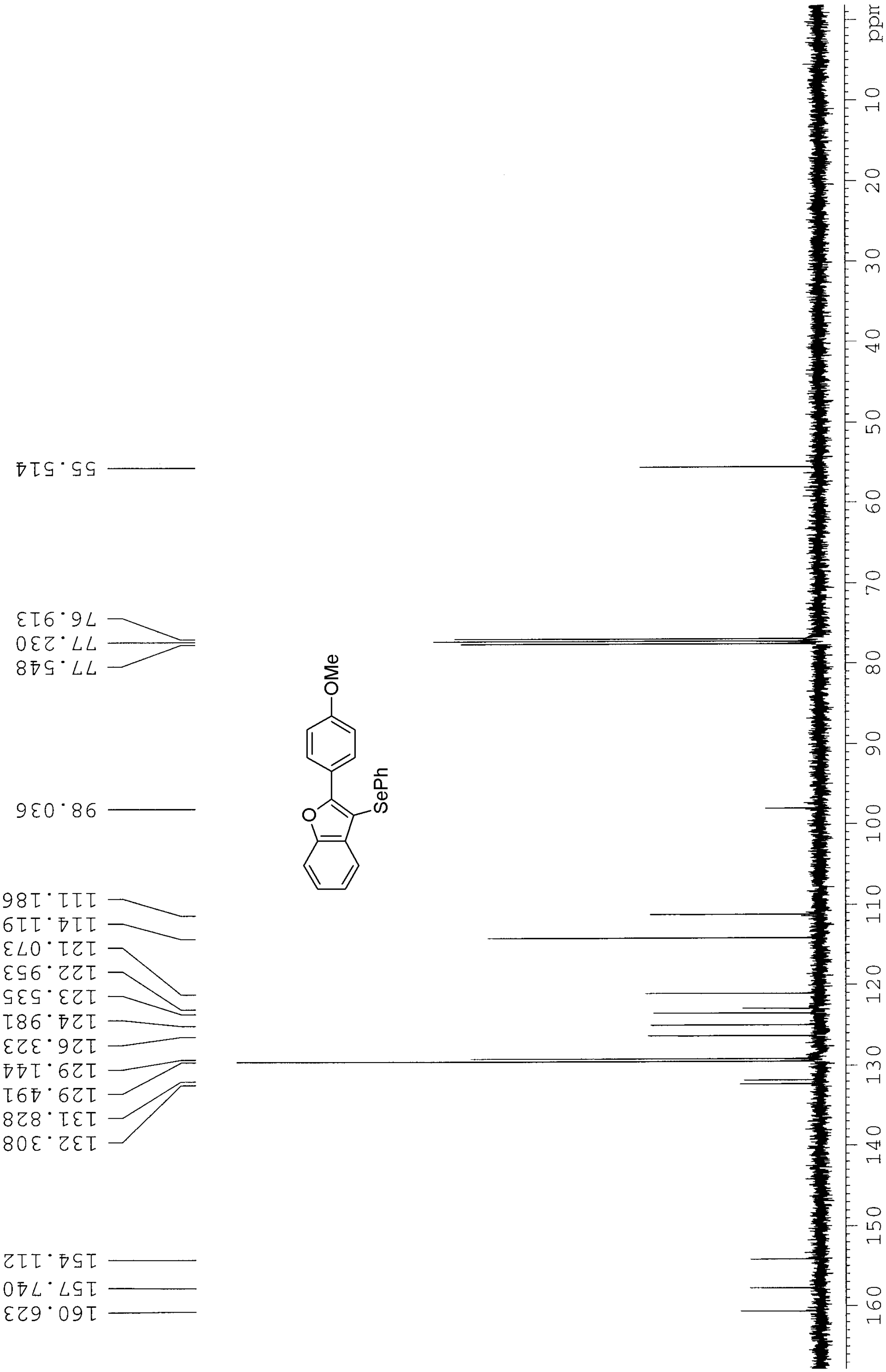
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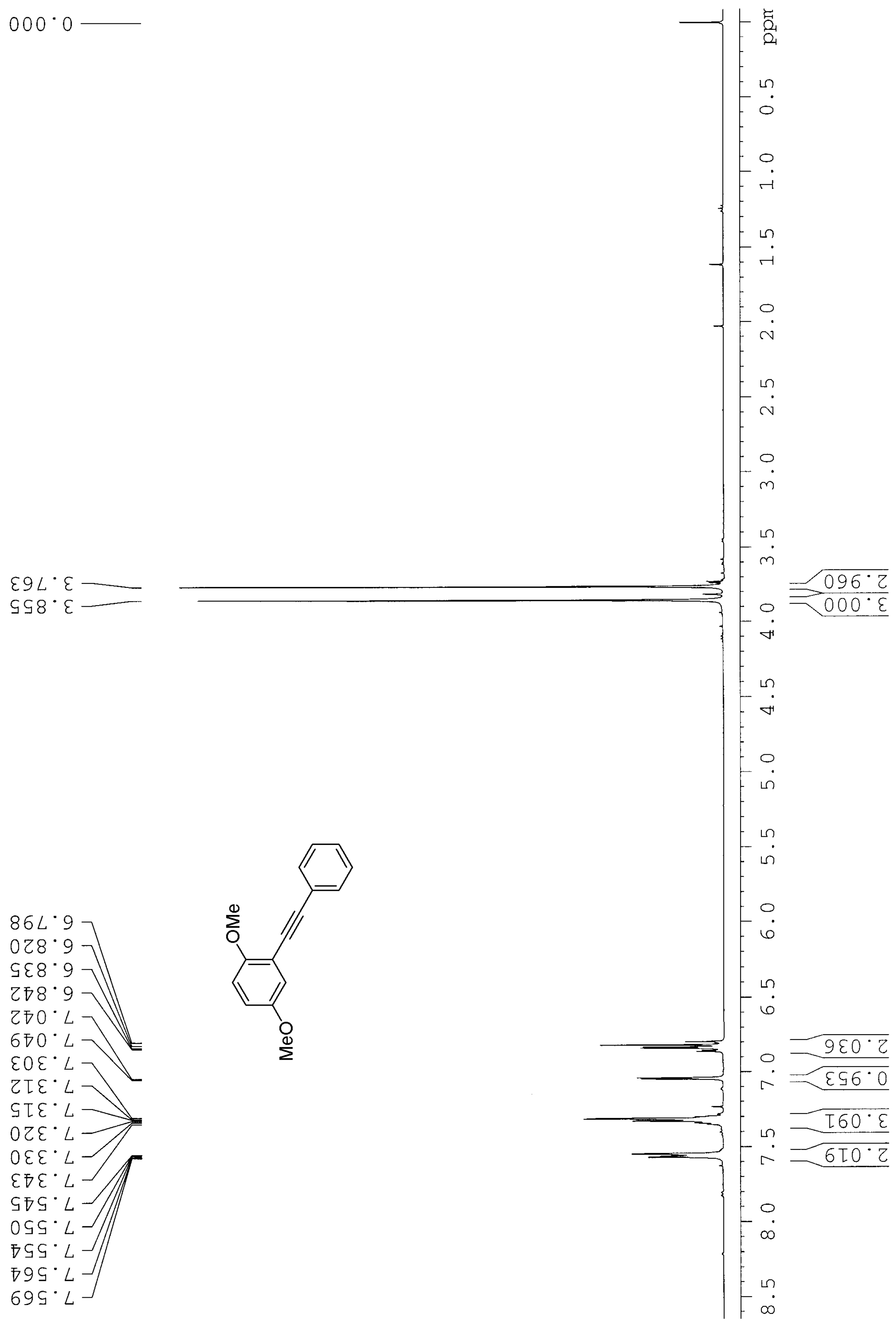
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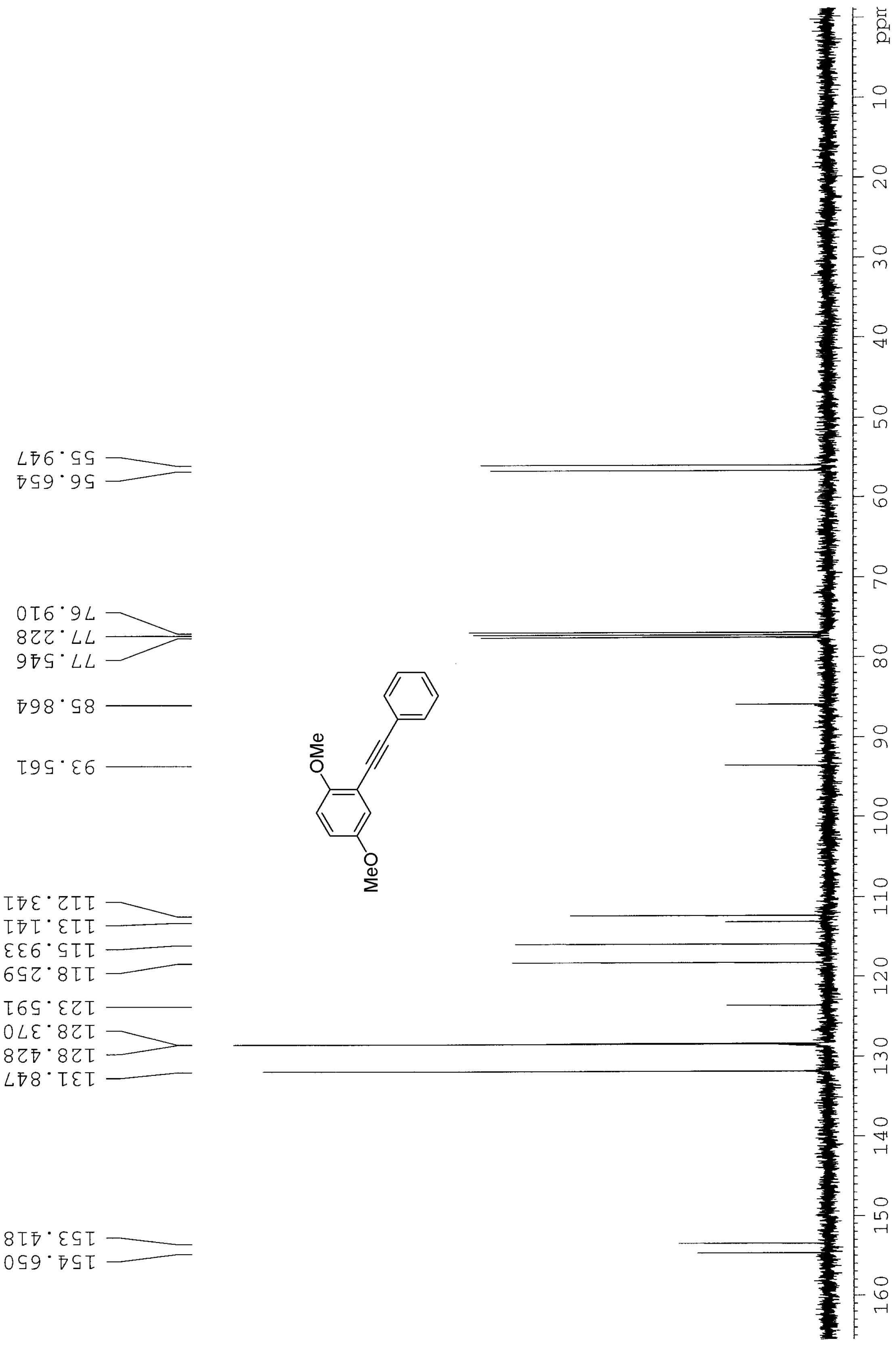
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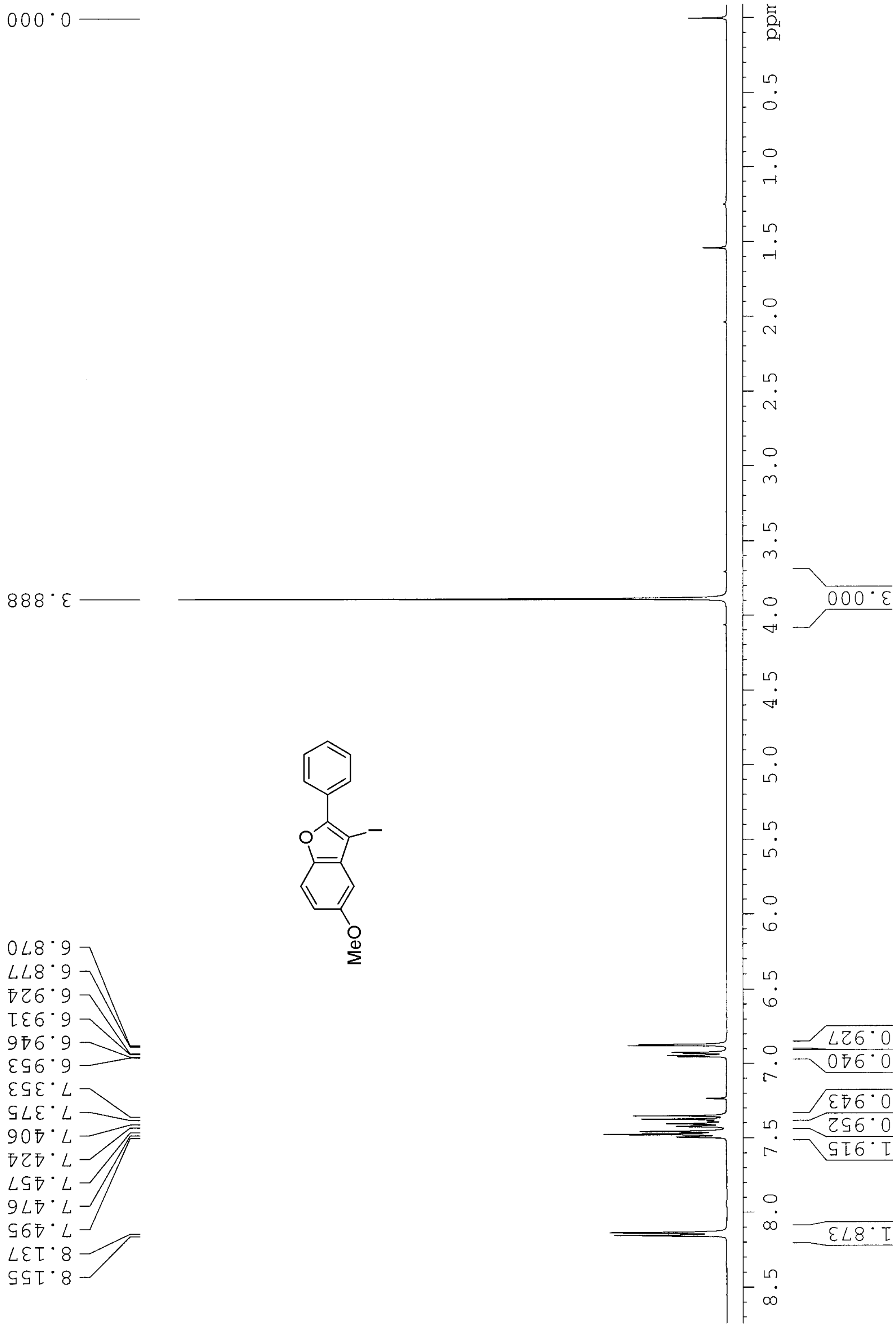
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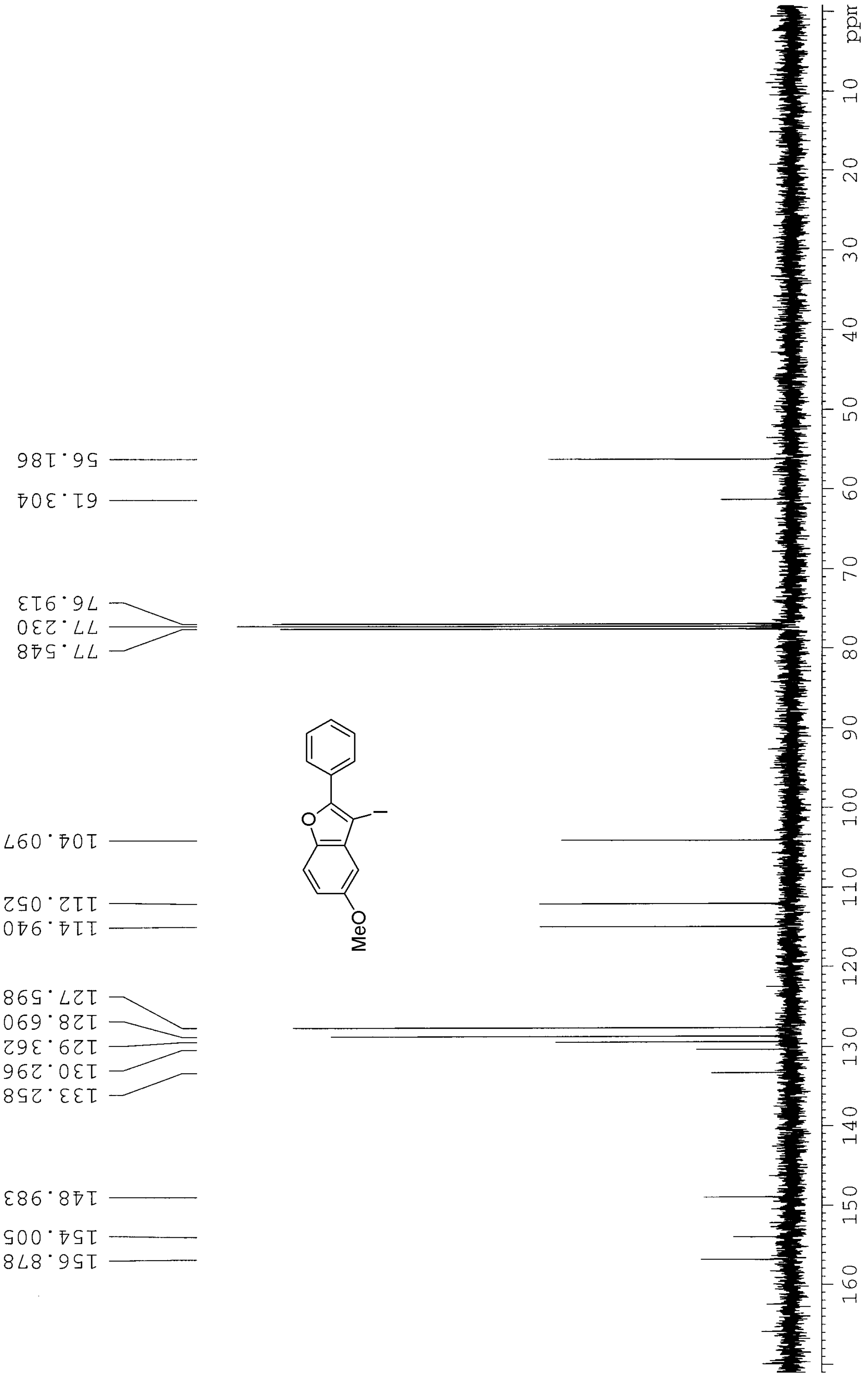

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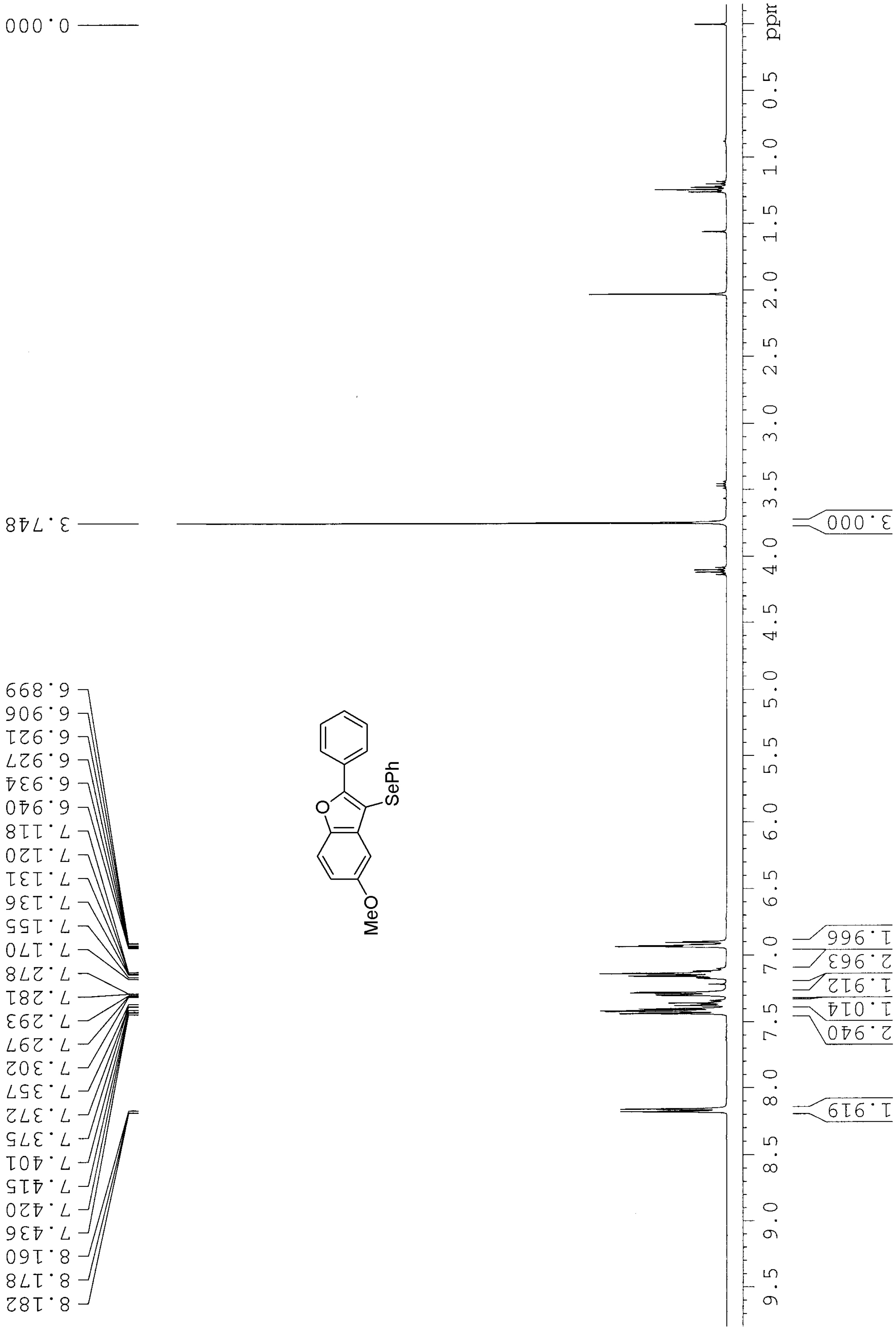
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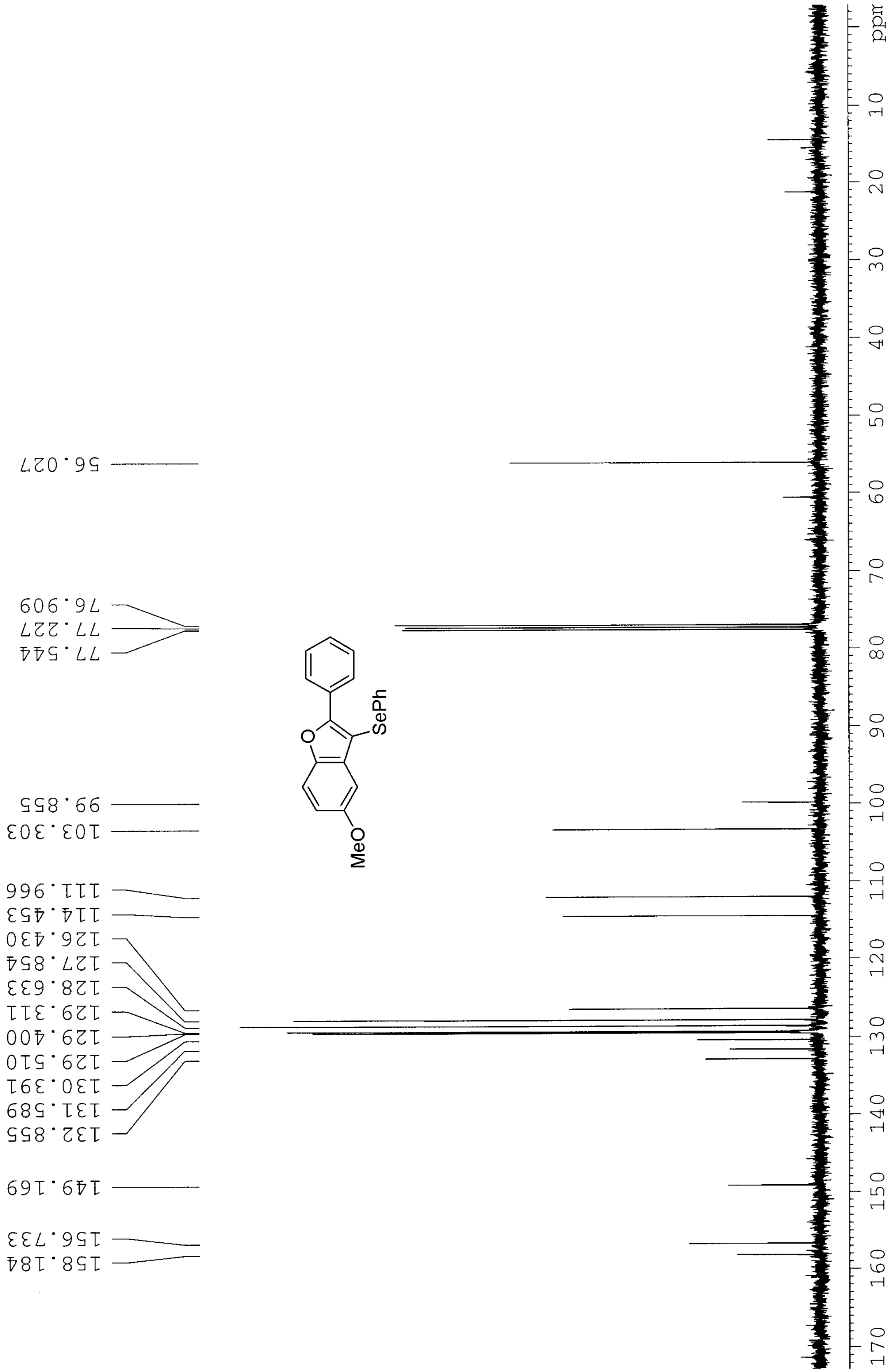

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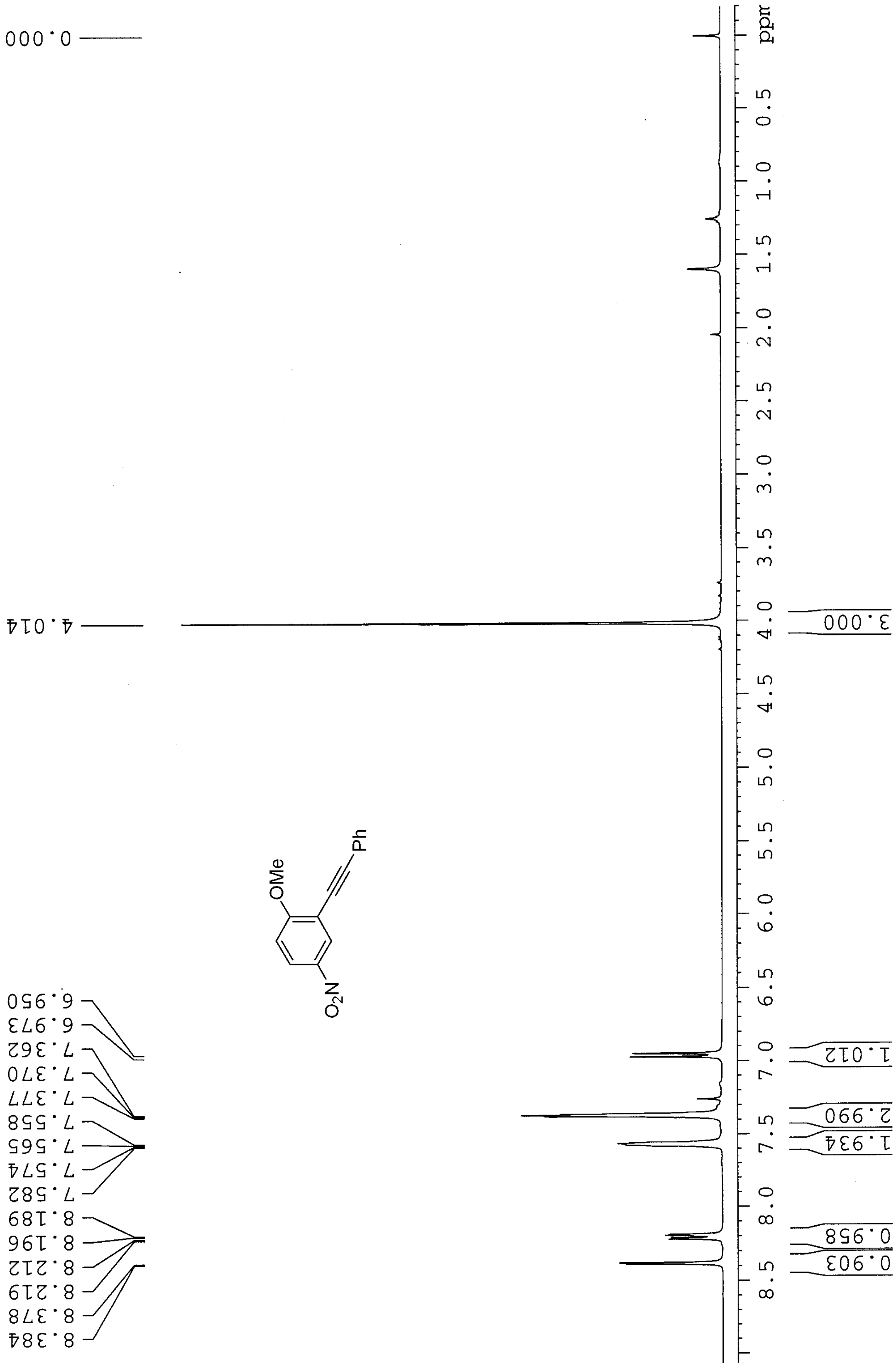
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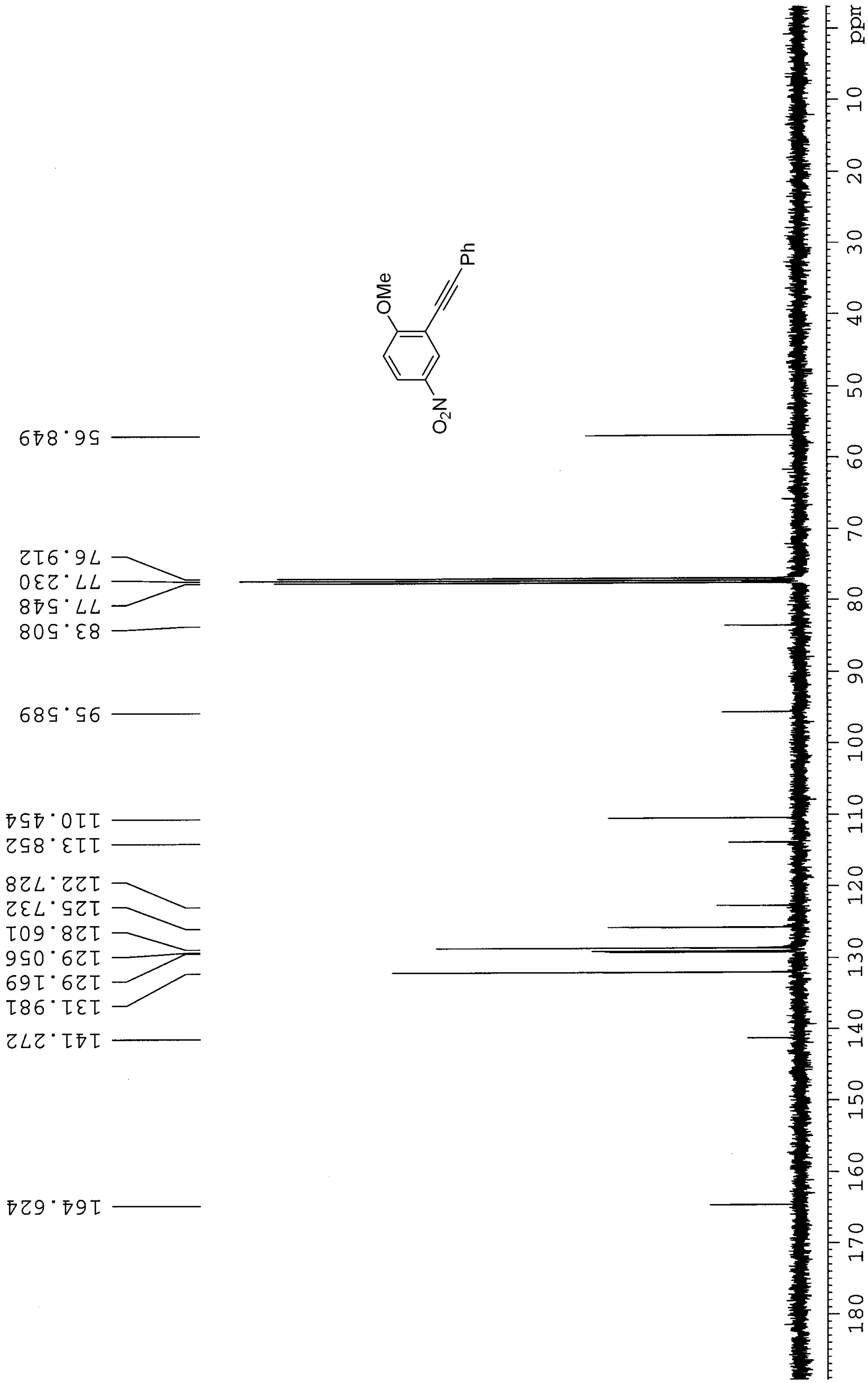
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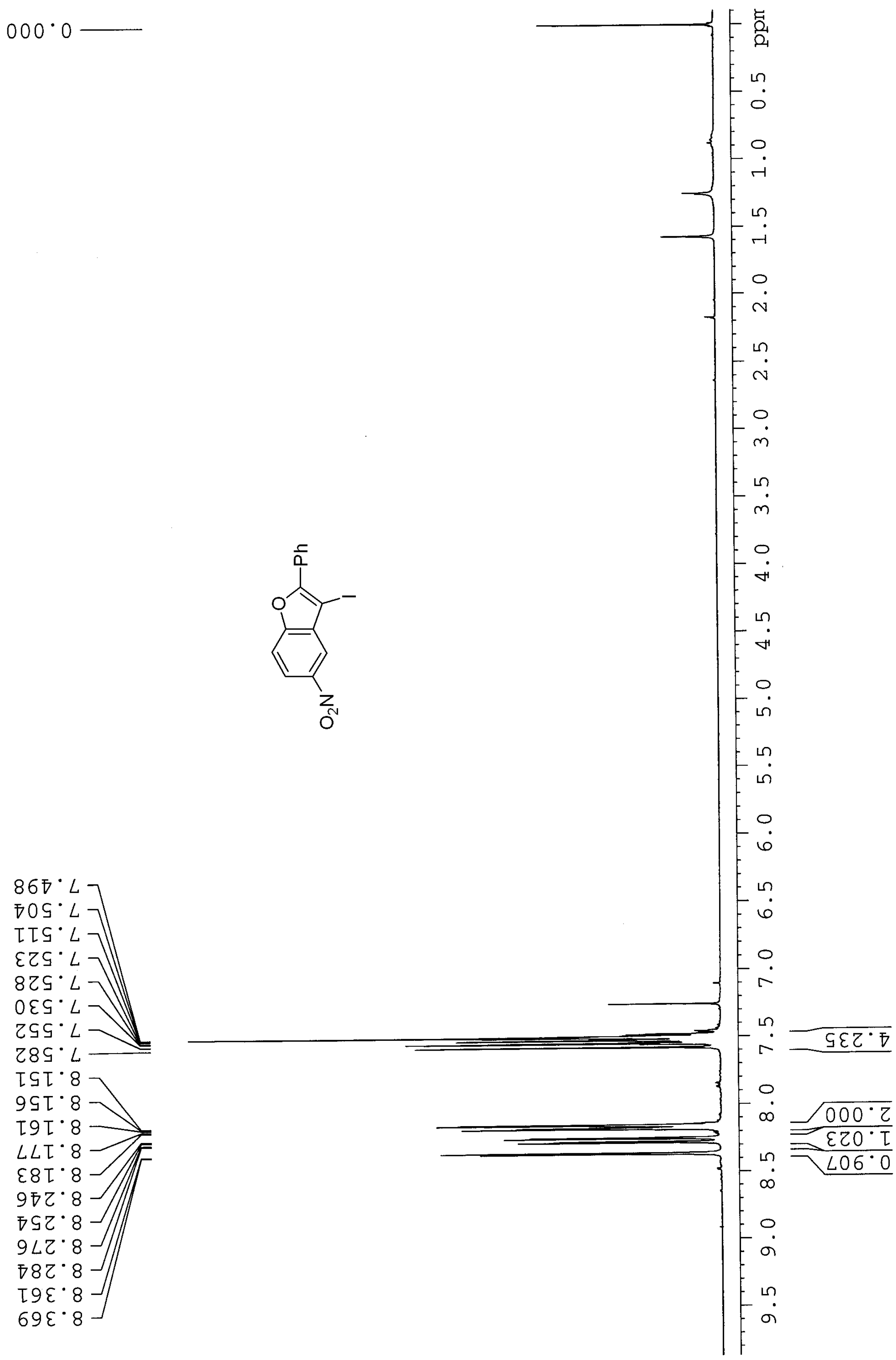
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8.161
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8.183
8.246
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8.276
8.284
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8.369
Ph
O
I
O2N
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3.0
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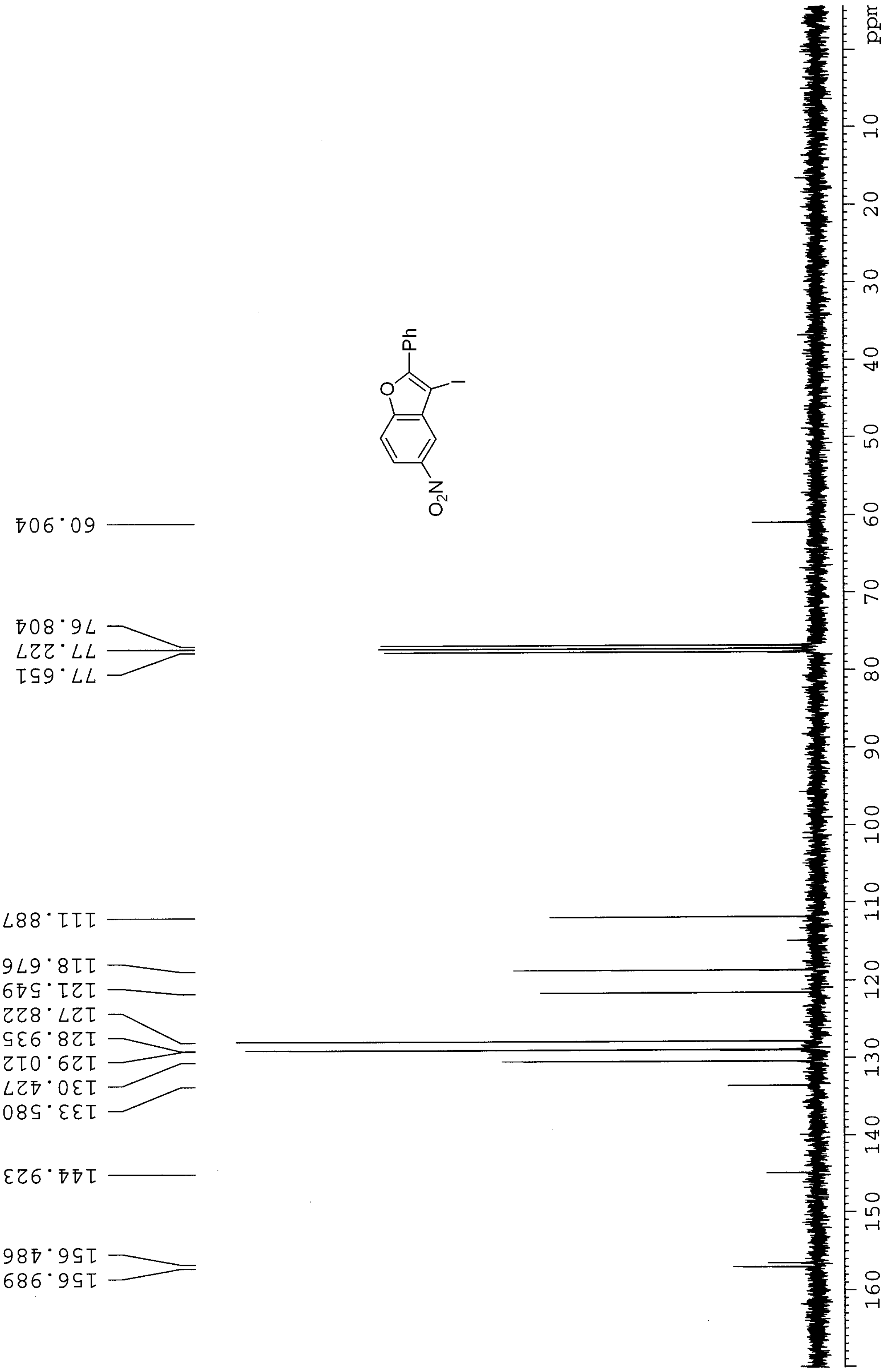
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156.989
Ph
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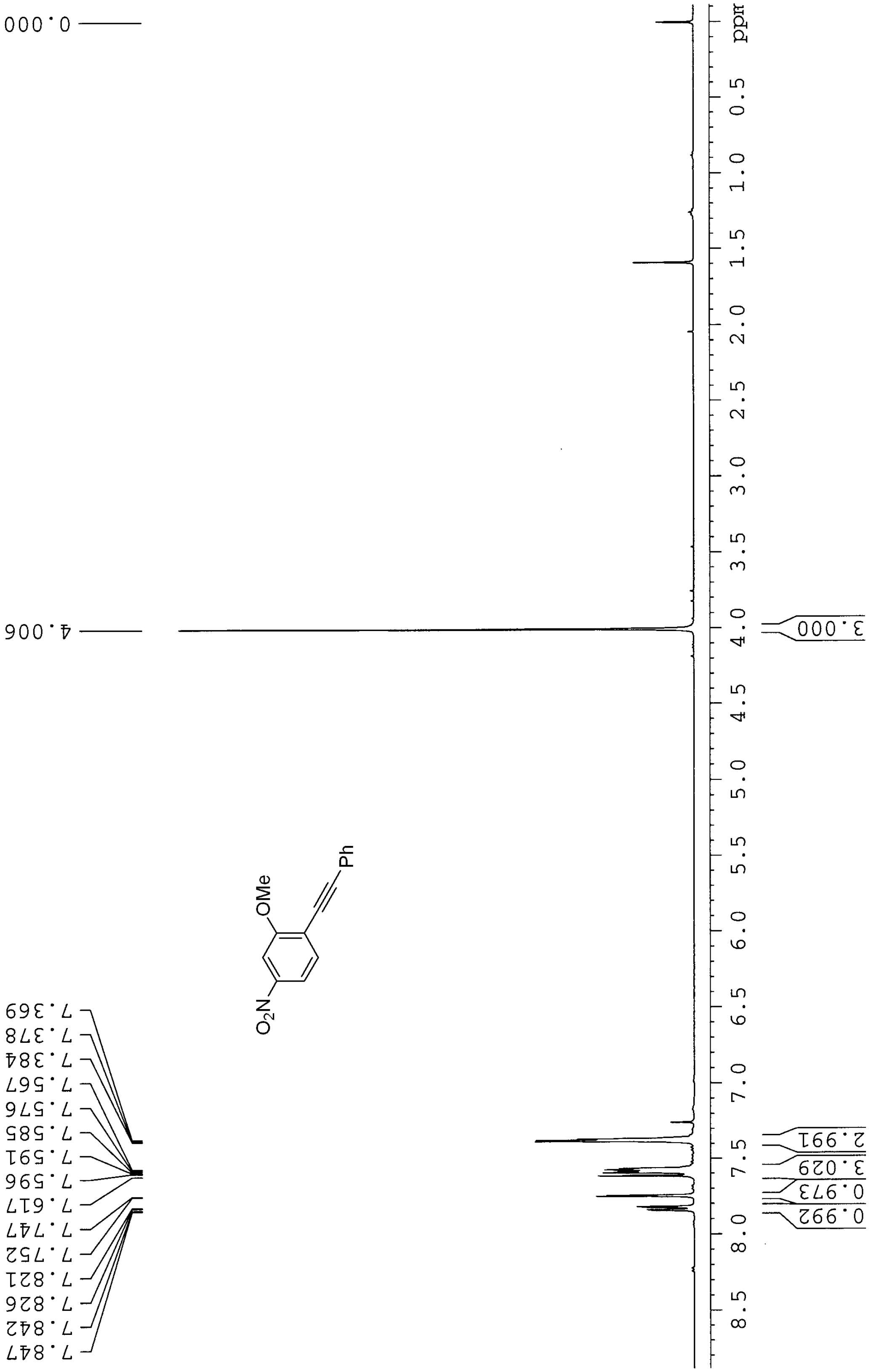
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7.596
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7.747
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7.821
7.826
7.842
7.847
O$_2$N
OMe
Ph
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ppm
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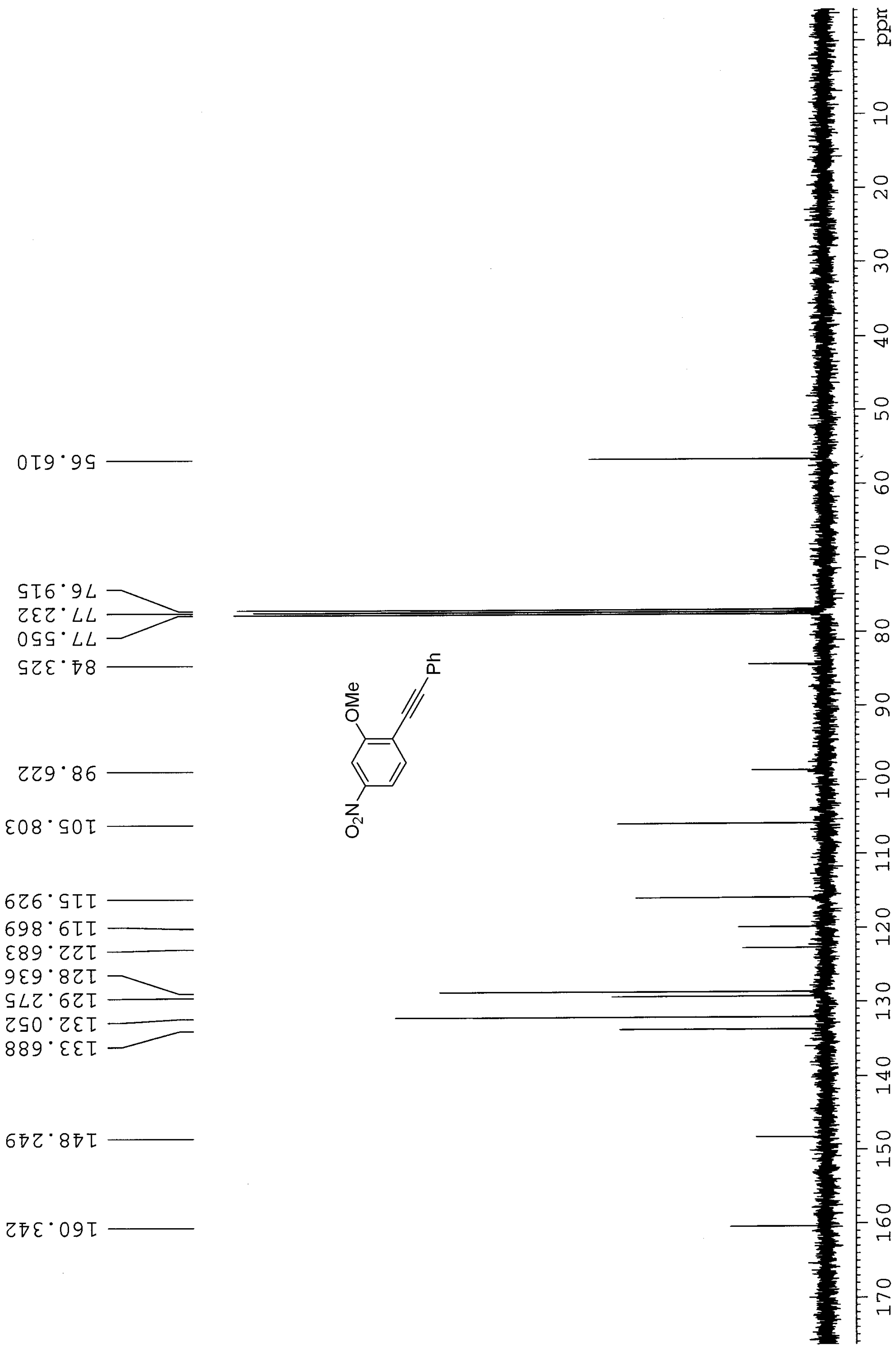
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115.929
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133.688
148.249
160.342
OMe
Ph
O_2N
ppm
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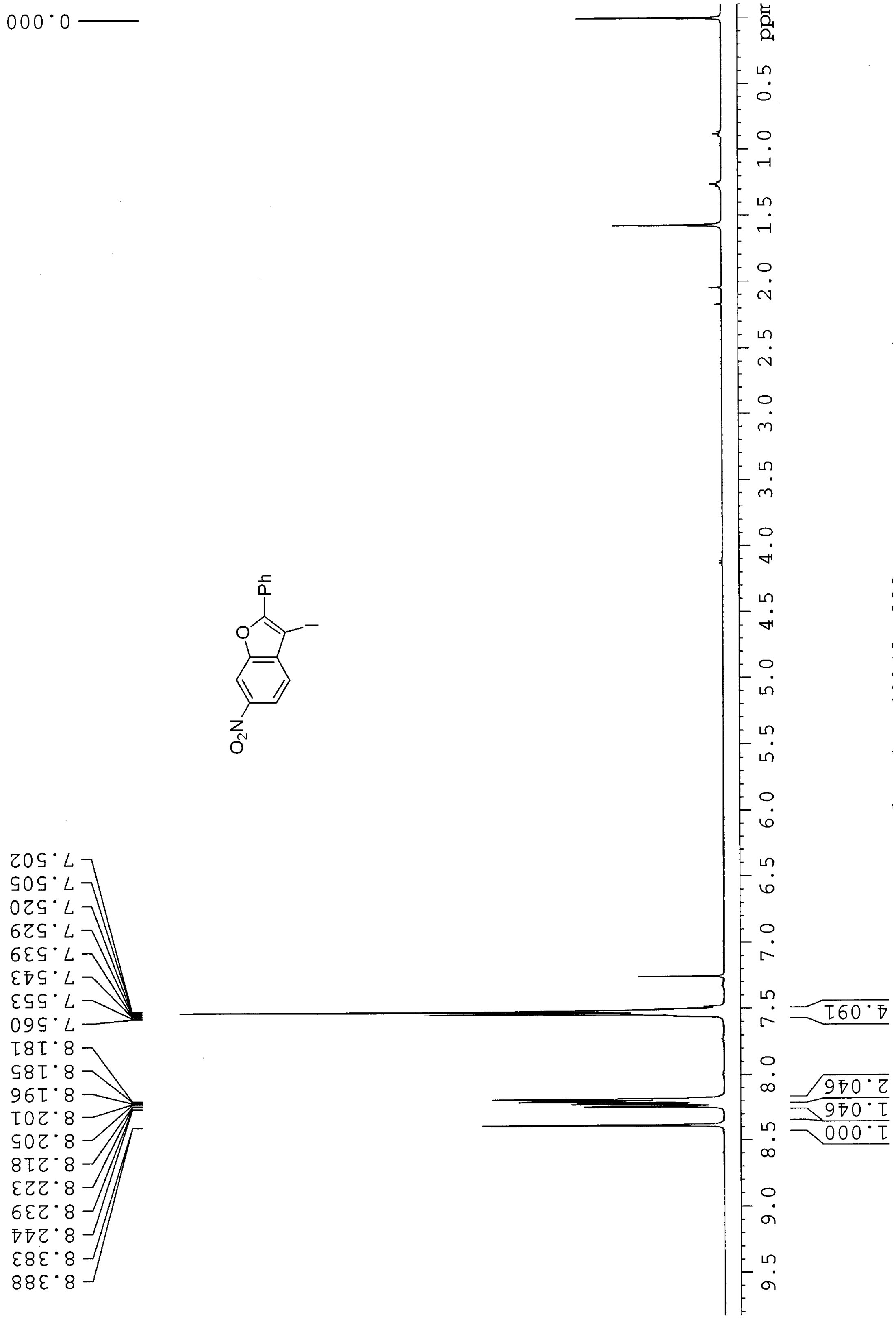
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8.201
8.205
8.218
8.223
8.239
8.244
8.383
8.388
Ph
O
I
O_2N
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
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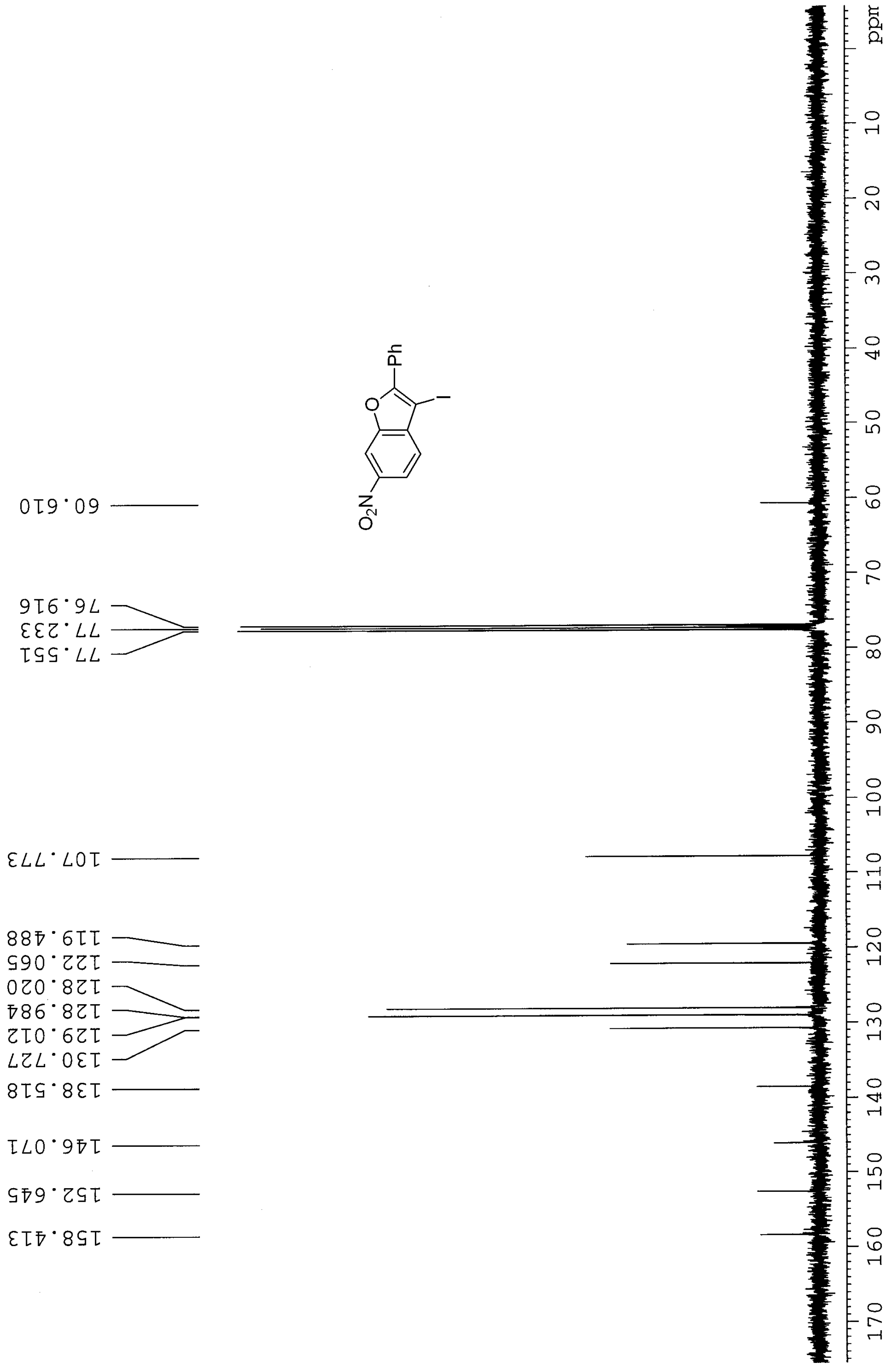

O2N
O
Ph
I
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77.551
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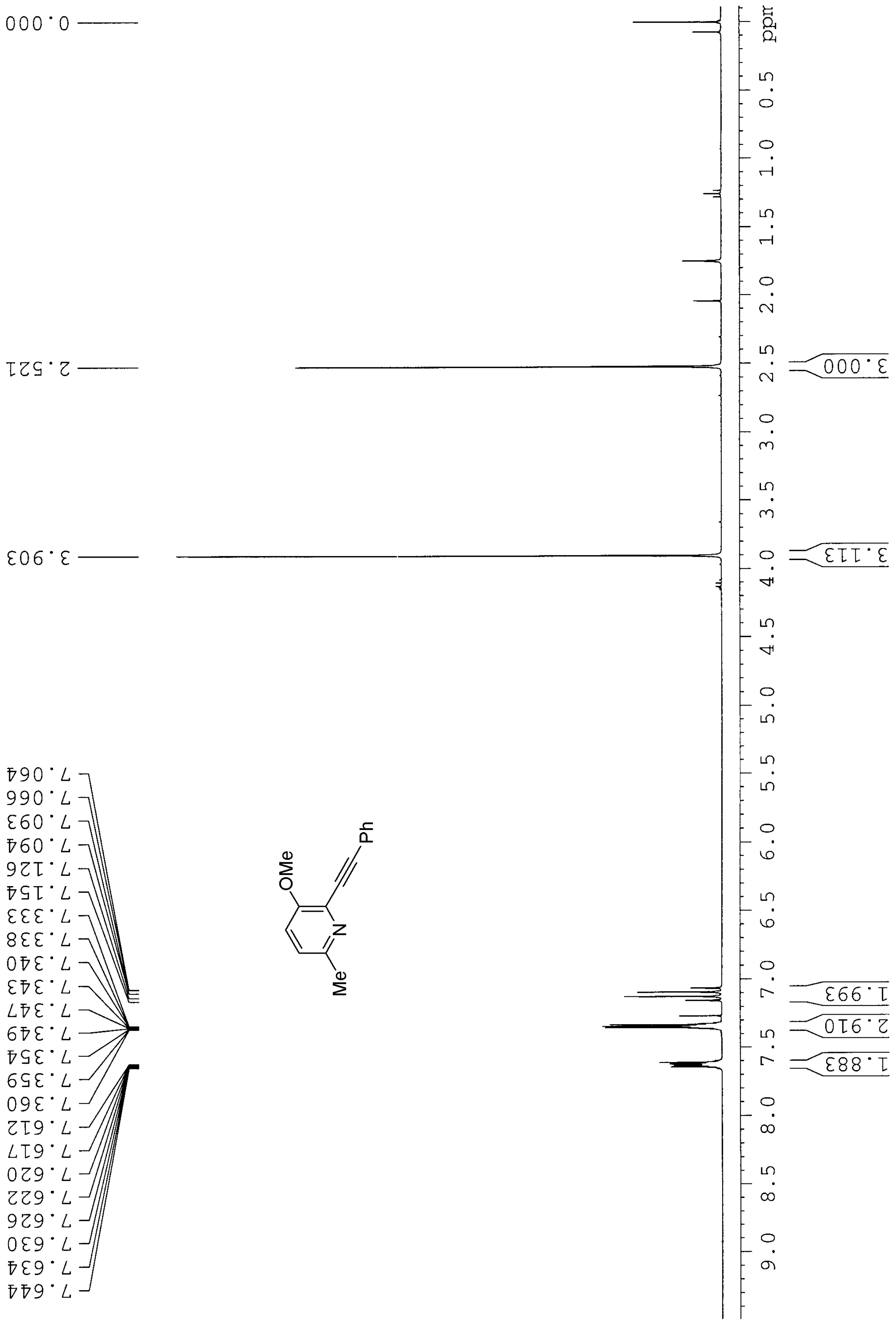
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7.343
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7.349
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7.359
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7.617
7.620
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7.630
7.634
7.644
OMe
Ph
N
Me
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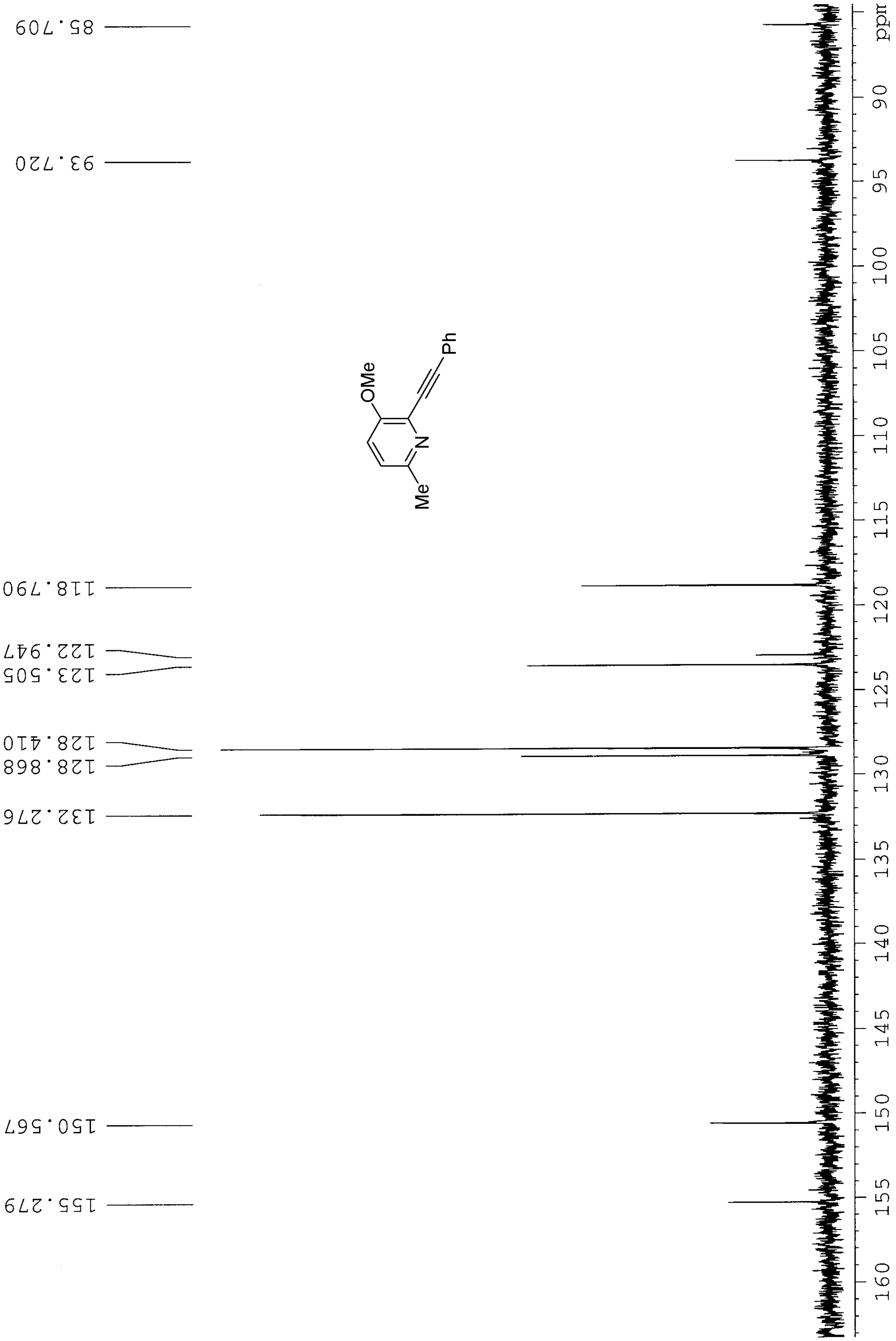
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85.709
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Ph
N
Me
ppm
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95
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115
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125
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135
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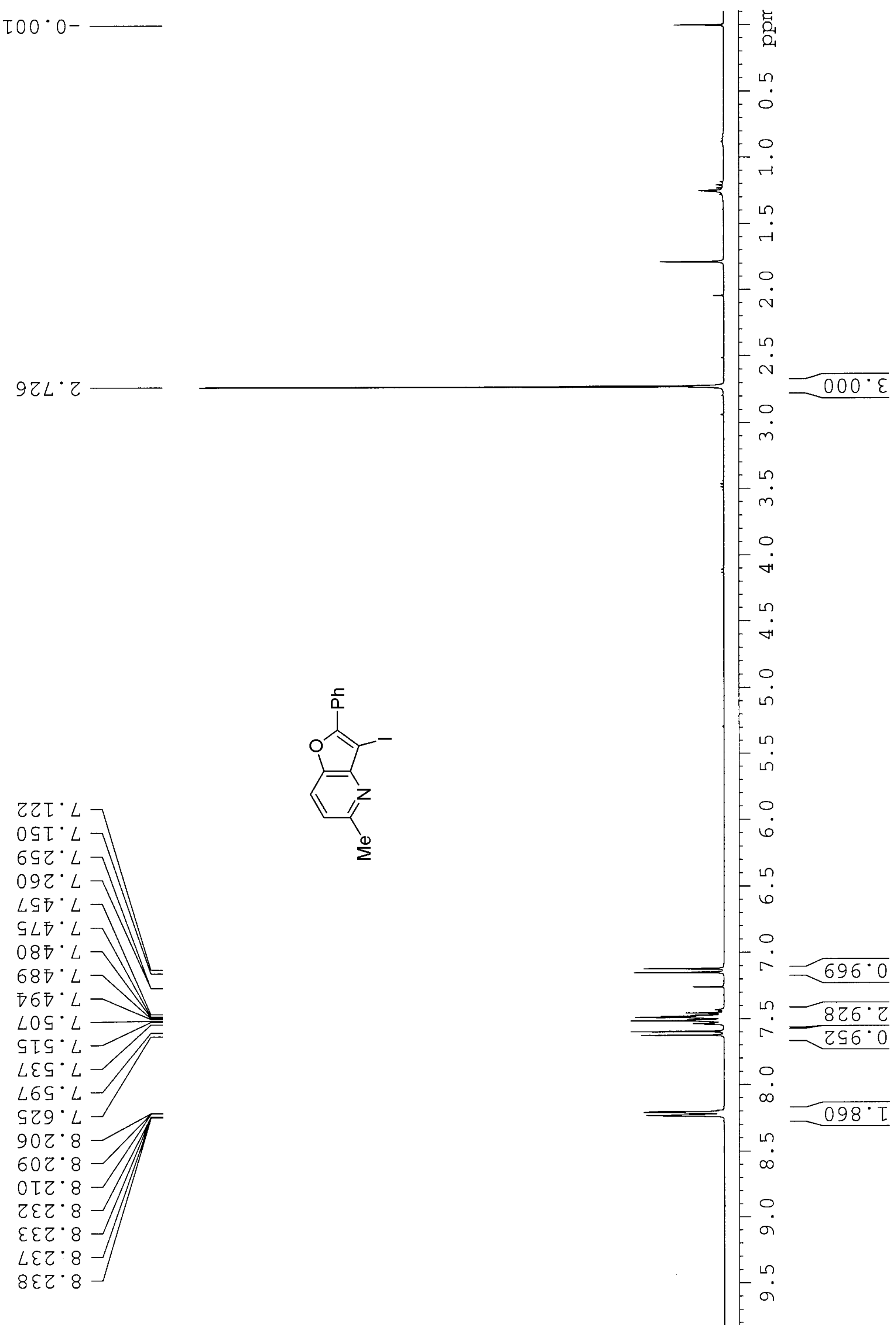
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7.475
7.480
7.489
7.494
7.507
7.515
7.537
7.597
7.625
8.206
8.209
8.210
8.232
8.233
8.237
8.238
Ph
O
I
N
Me
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0.952
1.860
ppm
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3.5
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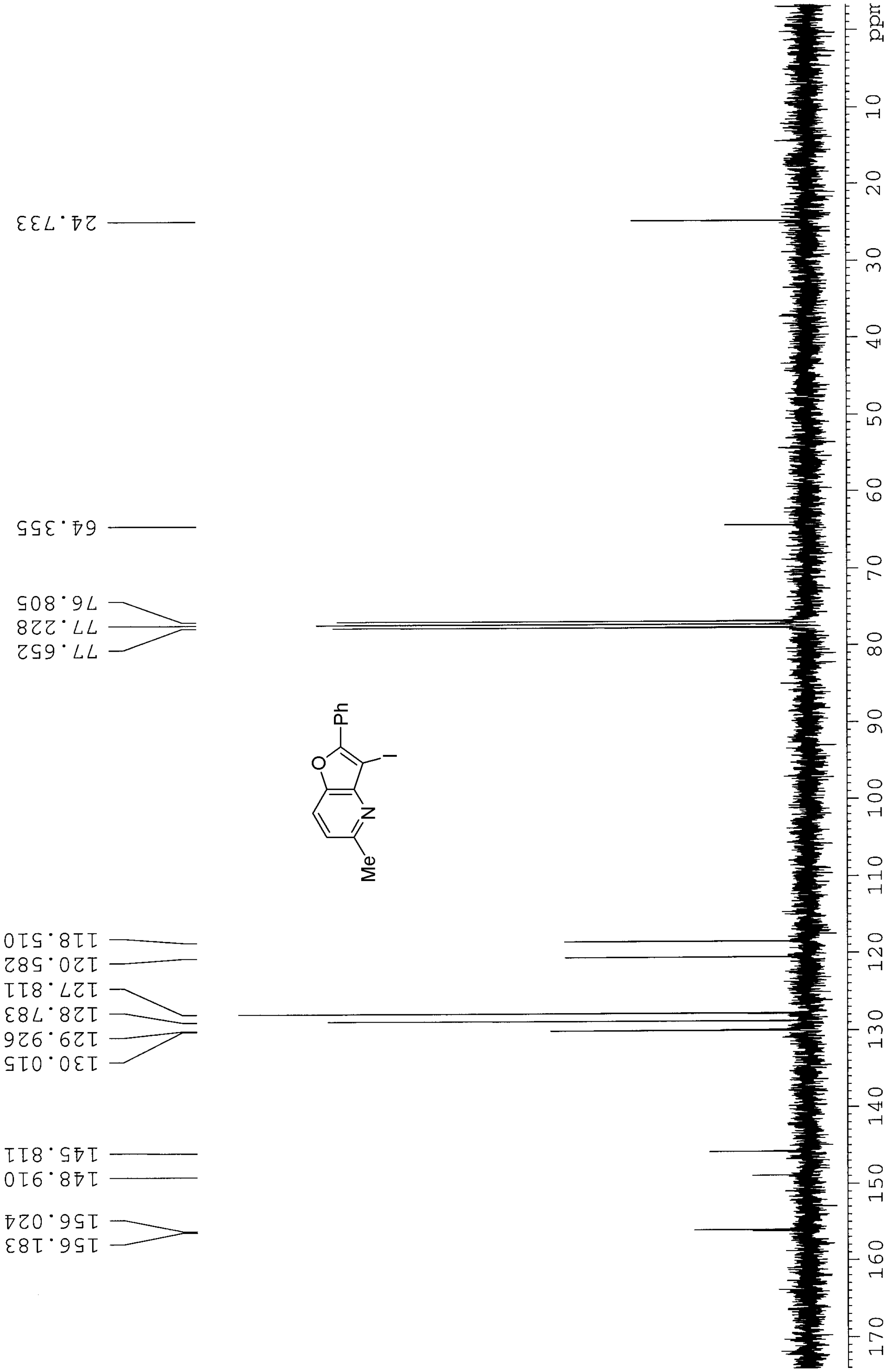
9.5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127.811
128.783
129.926
130.015
145.811
148.910
156.024
156.183
O
Ph
I
N
Me
ppm
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100
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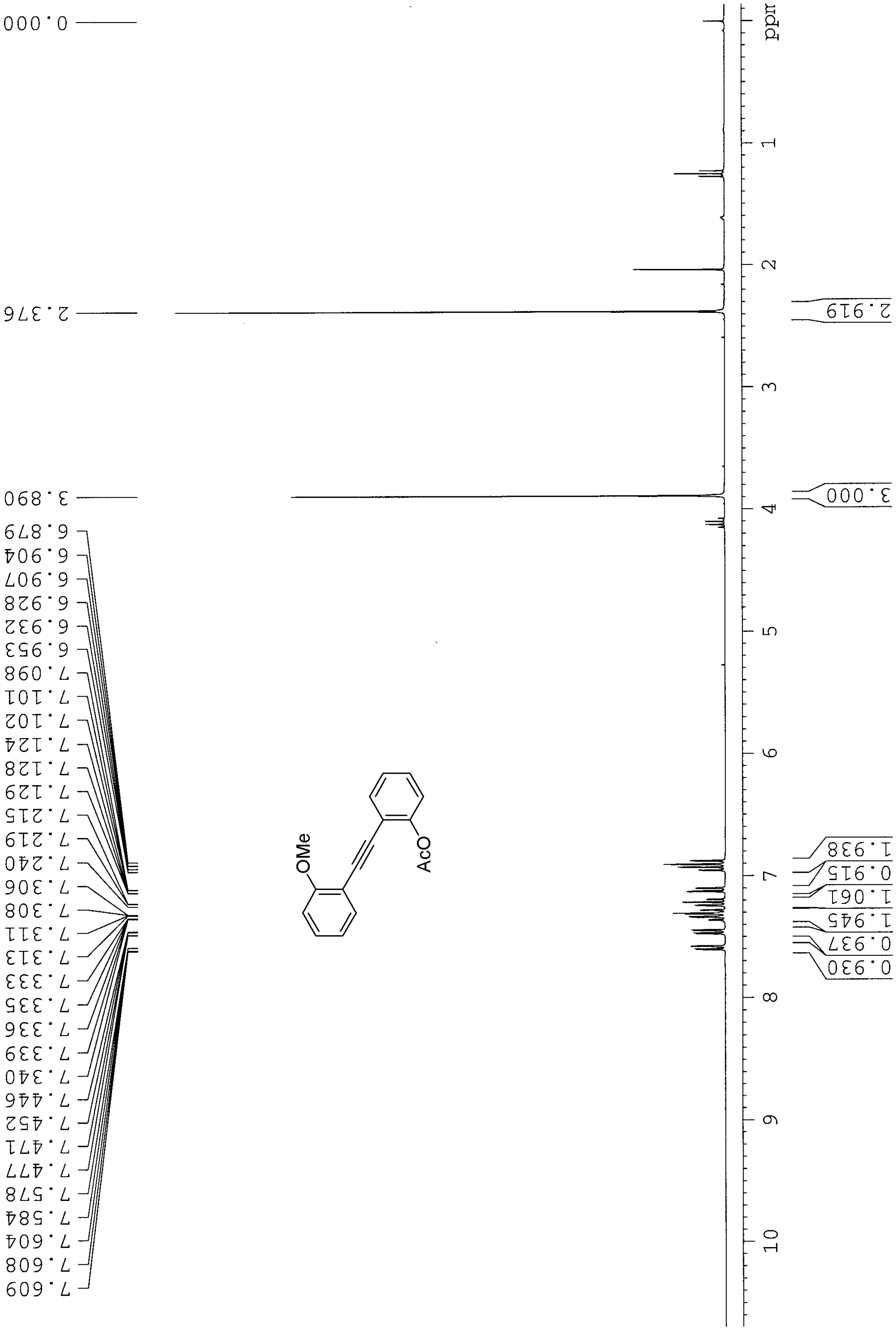
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6.953
7.098
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7.102
7.124
7.128
7.129
7.215
7.219
7.240
7.306
7.308
7.311
7.313
7.333
7.335
7.336
7.339
7.340
7.446
7.452
7.471
7.477
7.578
7.584
7.604
7.608
7.609
OMe
AcO
2.919
3.000
1.938
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1.061
1.945
0.937
0.930
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2
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4
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8
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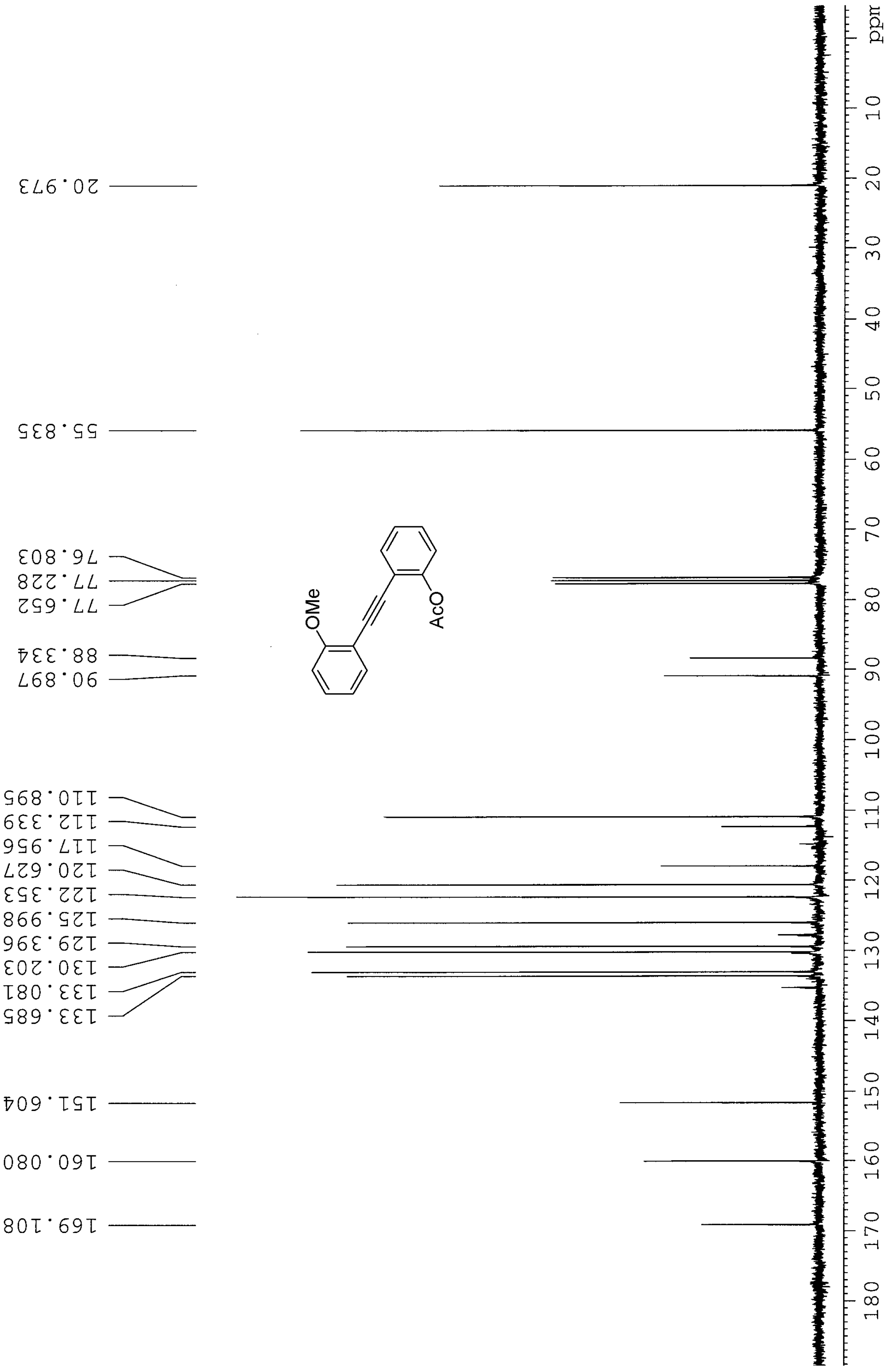
10
ppm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77.652
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90.897
110.895
112.339
117.956
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122.353
125.998
129.396
130.203
133.081
133.685
151.604
160.080
169.108
OMe
AcO
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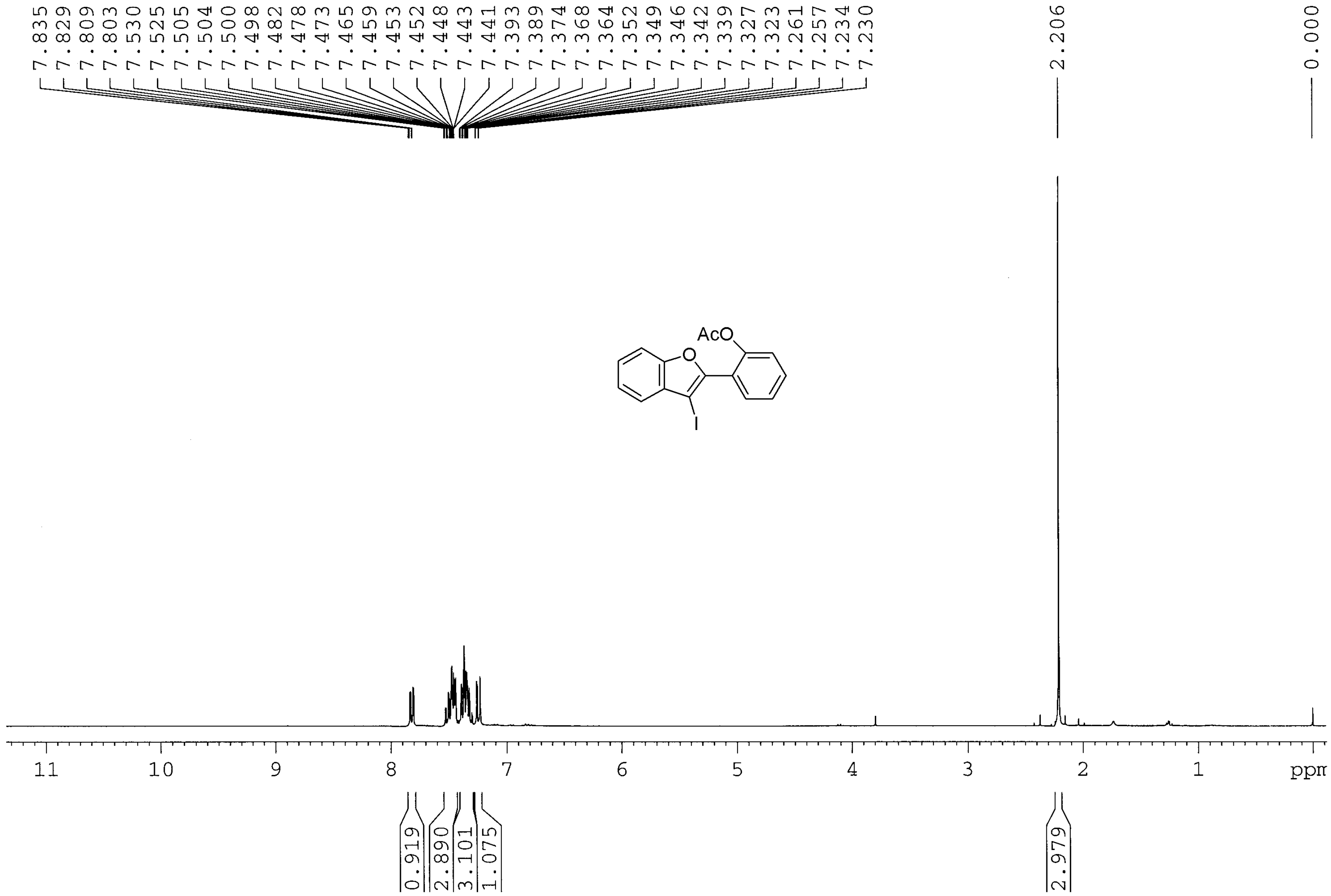
150
160
170
180



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7.809
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7.530
7.525
7.505
7.504
7.500
7.498
7.482
7.478
7.473
7.465
7.459
7.453
7.452
7.448
7.443
7.441
7.393
7.389
7.374
7.368
7.364
7.352
7.349
7.346
7.342
7.339
7.327
7.323
7.261
7.257
7.234
7.230
2.206
0.000
AcO
O
I
11
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8
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5
4
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2
1
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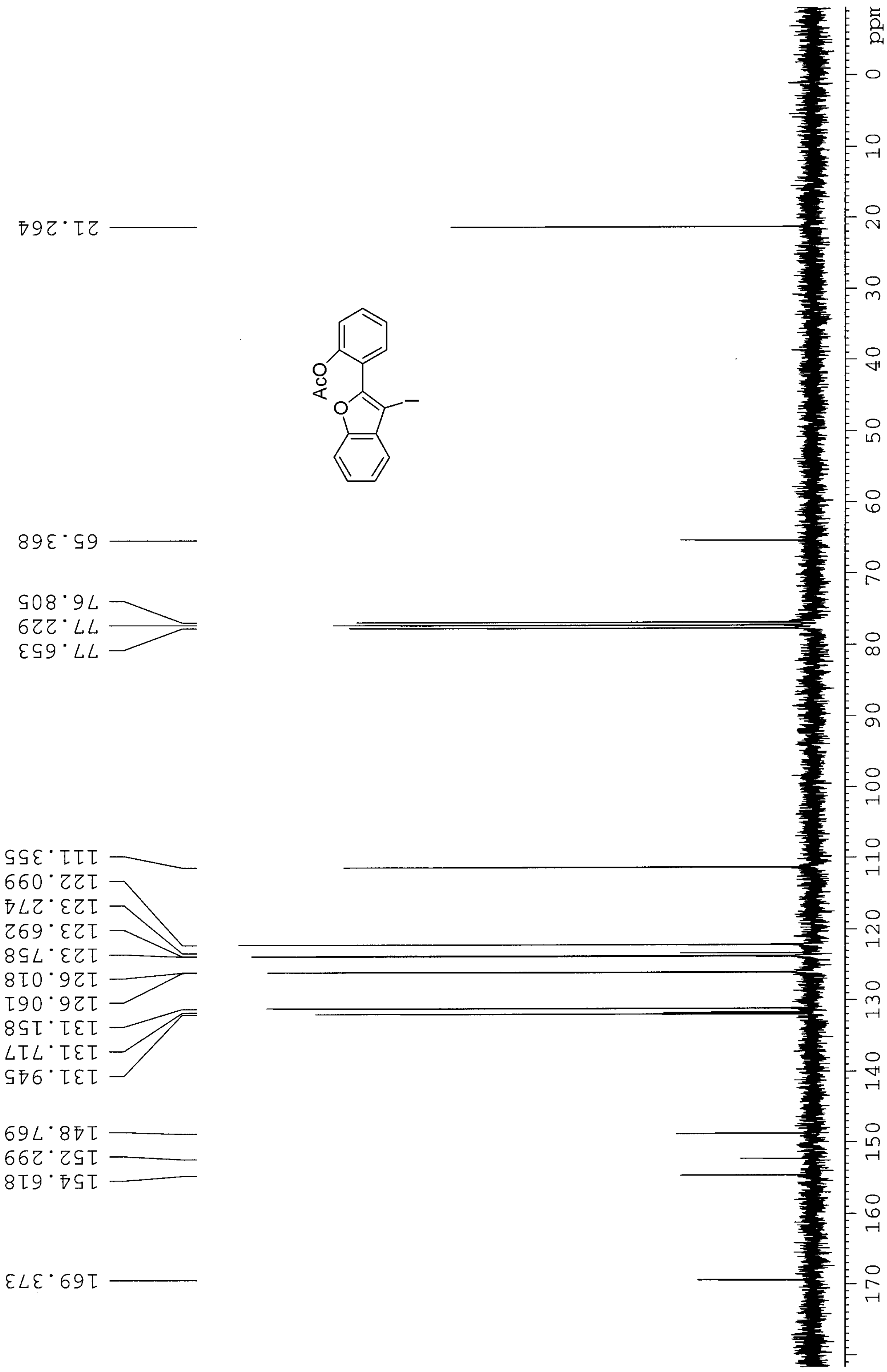
2.979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123.758
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126.061
131.158
131.717
131.945
148.769
152.299
154.618
169.373
AcO
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150
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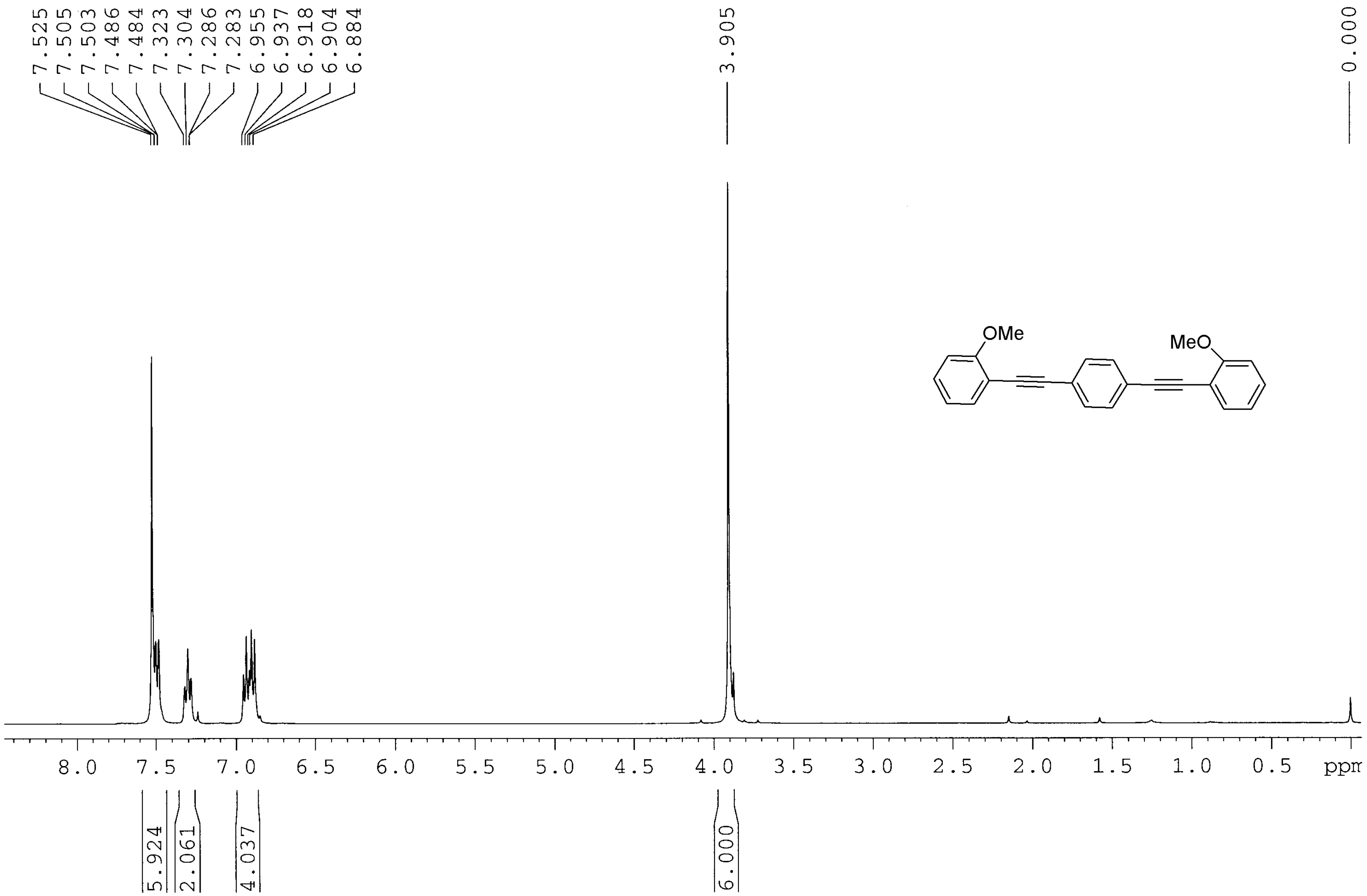
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6.918
6.904
6.884
3.905
0.000
OMe
MeO
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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ppm
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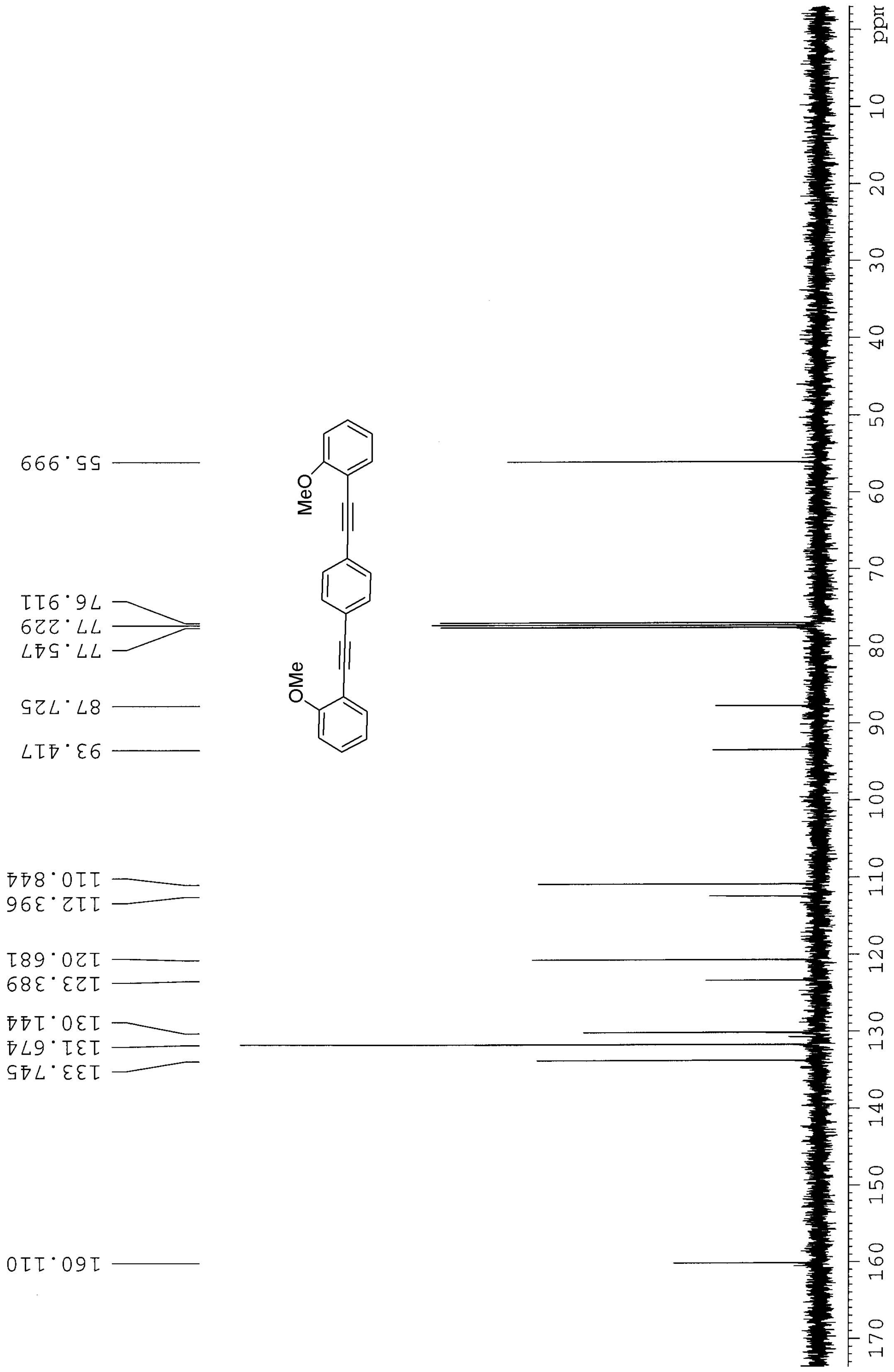
6.000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133.745
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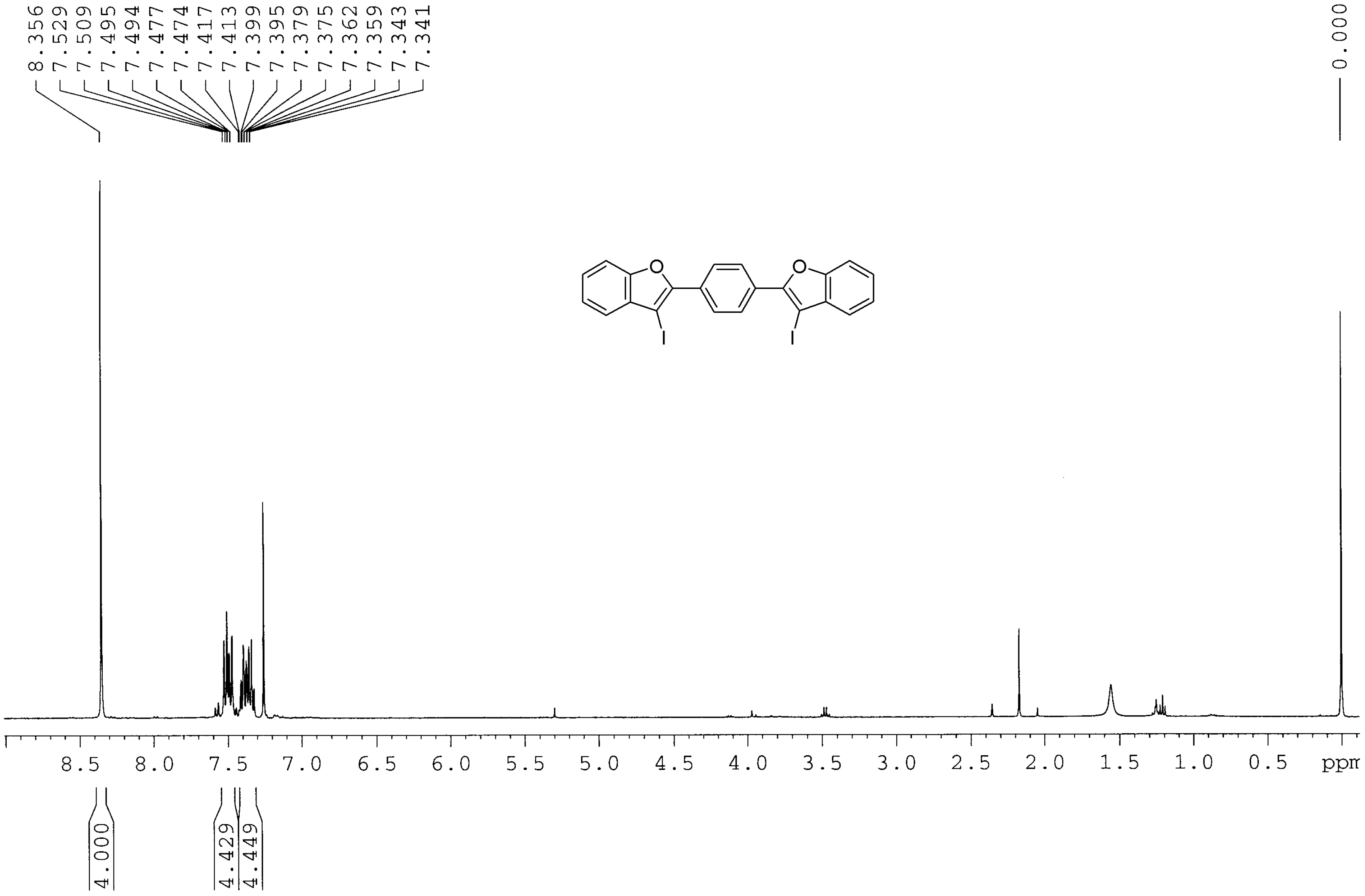
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7.375
7.362
7.359
7.343
7.341
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O
O
I
I
8.5
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7.5
7.0
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6.0
5.5
5.0
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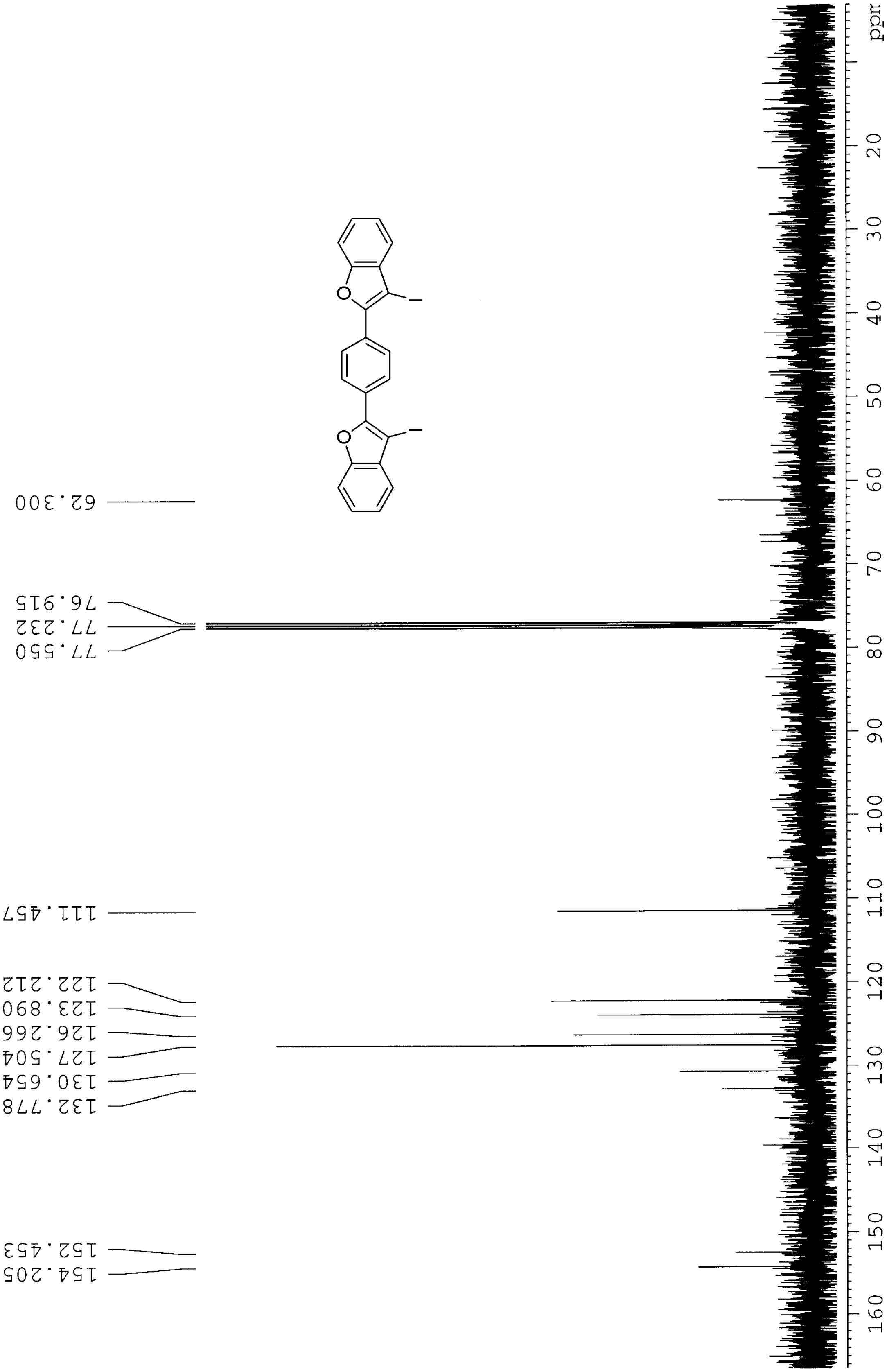
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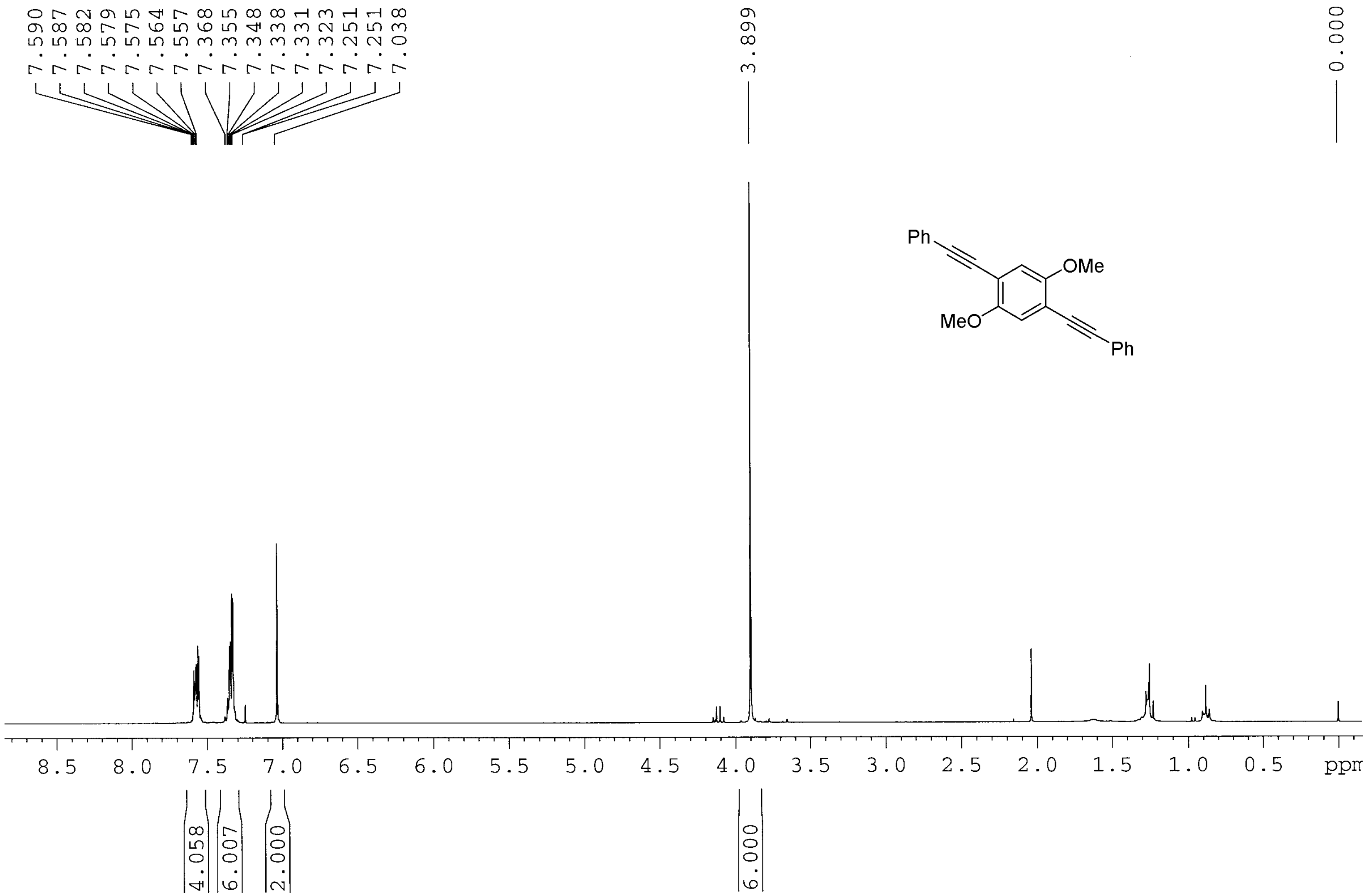
O
O
I
I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7.323
7.251
7.251
7.038
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0.000
Ph
OMe
MeO
Ph
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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1.0
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ppm
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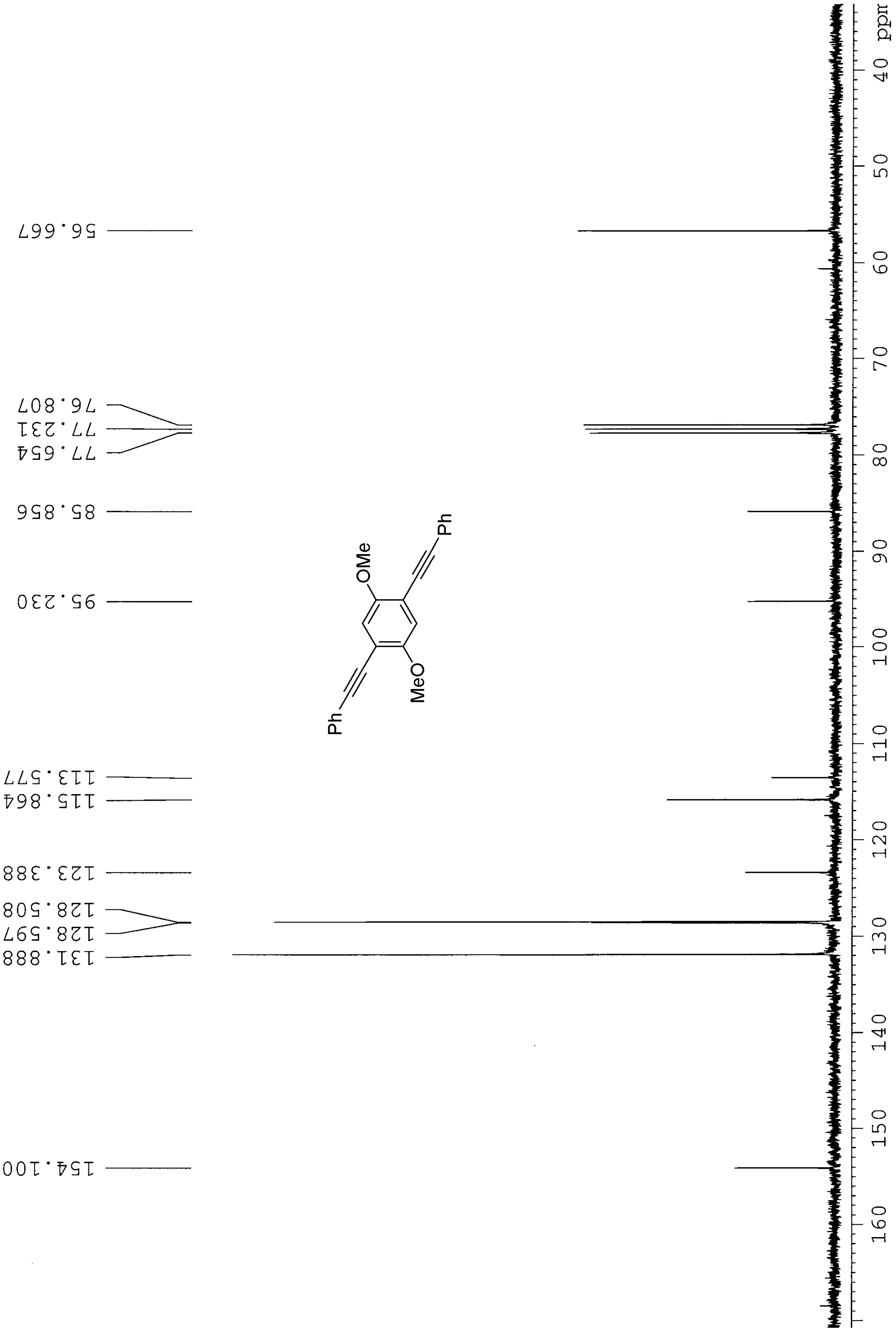
6.000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128.597
131.888
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MeO
Ph
Ph
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100
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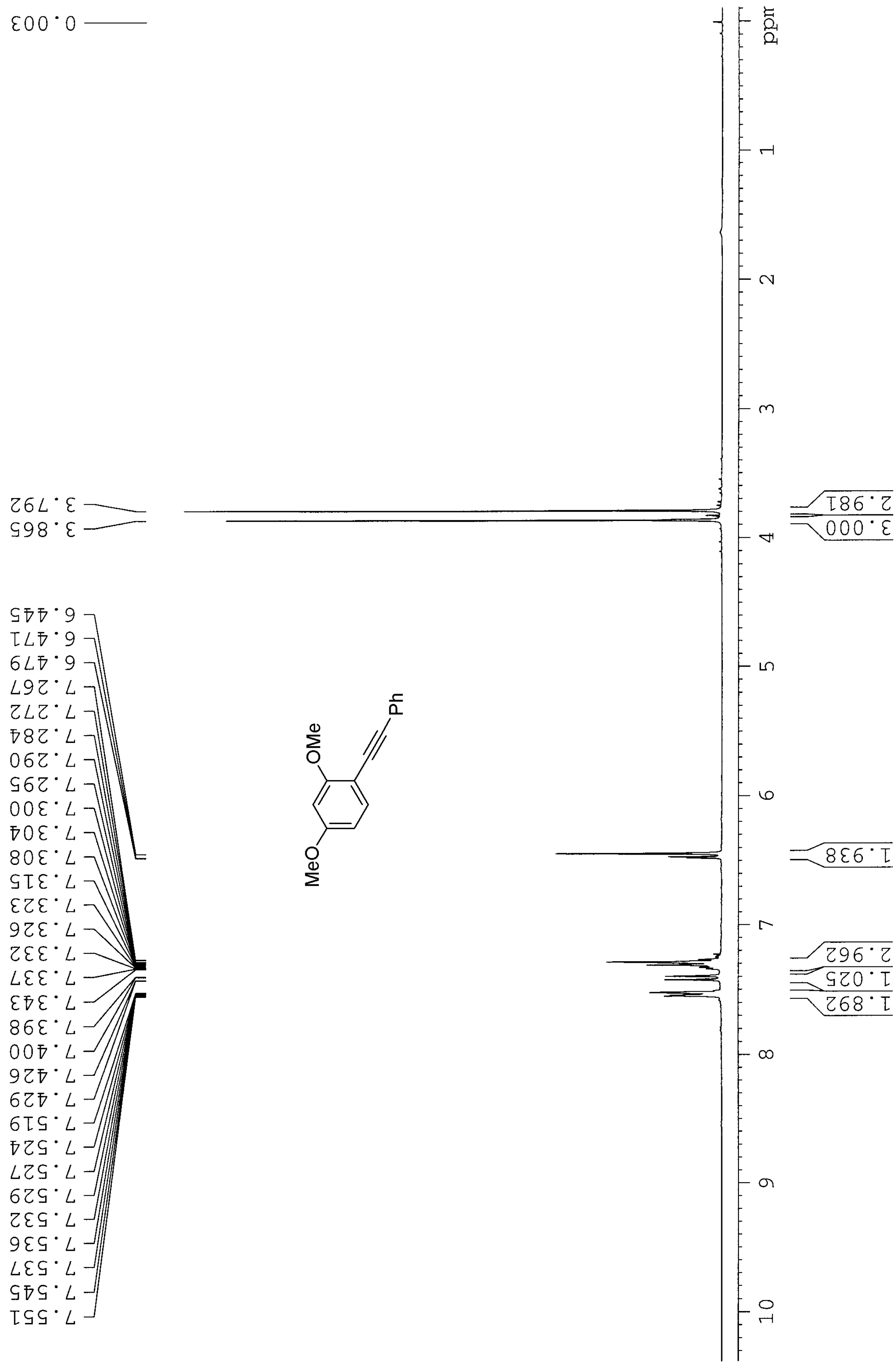
130
140
150
160
ppm



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7.304
7.308
7.315
7.323
7.326
7.332
7.337
7.343
7.398
7.400
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7.429
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7.524
7.527
7.529
7.532
7.536
7.537
7.545
7.551
MeO
OMe
Ph
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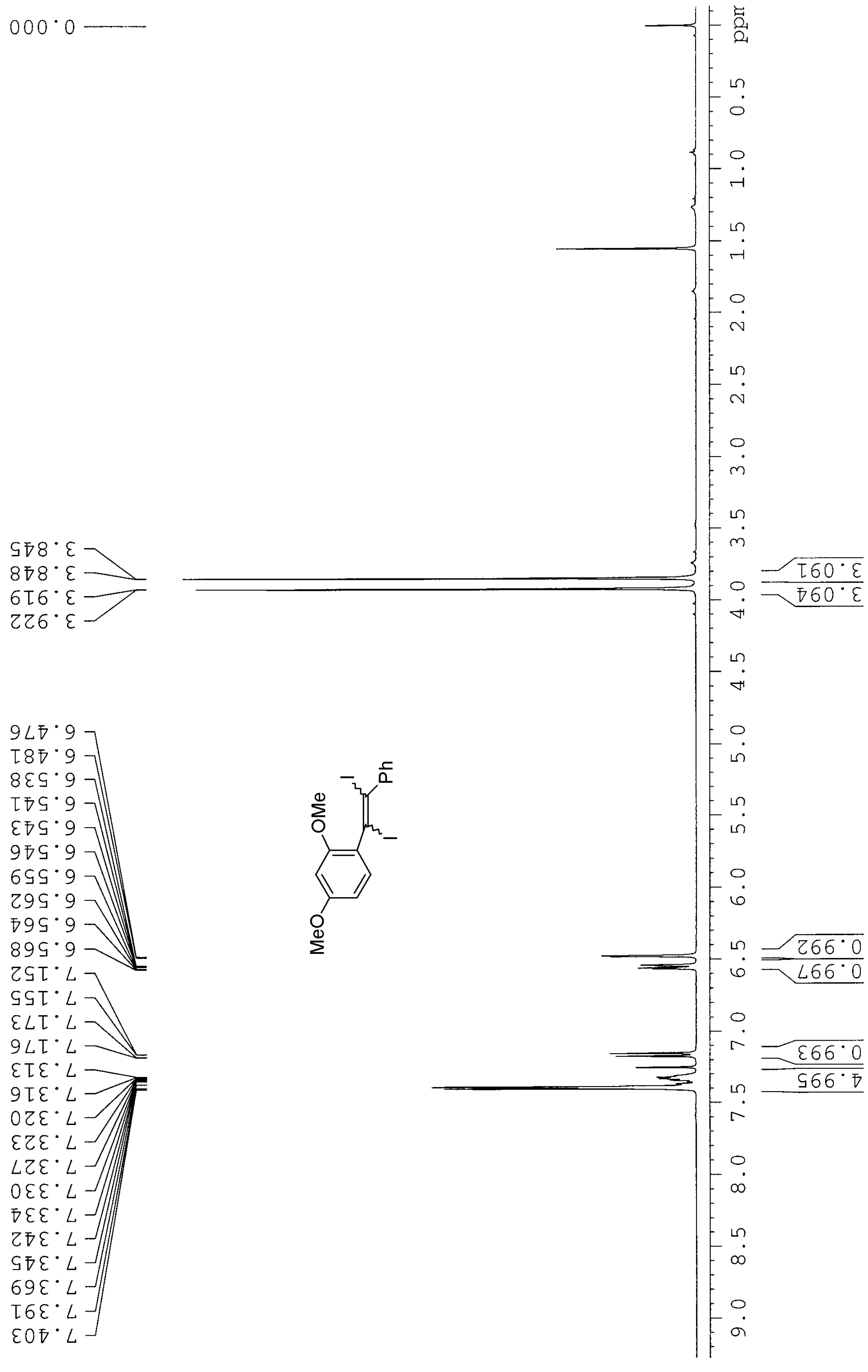
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6.559
6.562
6.564
6.568
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7.155
7.173
7.176
7.313
7.316
7.320
7.323
7.327
7.330
7.334
7.342
7.345
7.369
7.391
7.403
MeO
OMe
I
Ph
I
3.091
3.094
0.992
0.997
0.993
4.995
ppm
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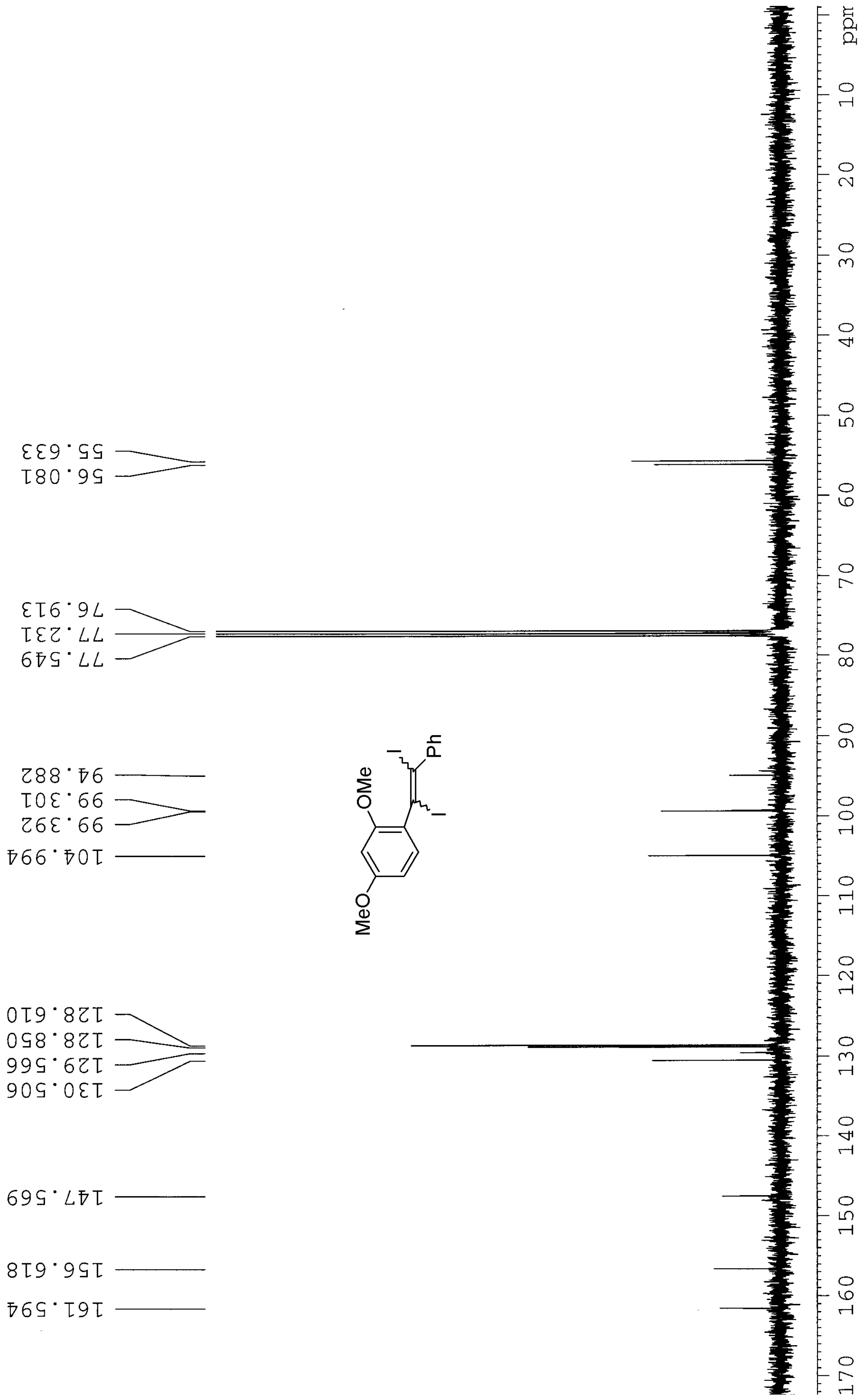
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99.392
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129.566
130.506
147.569
156.618
161.594
MeO
OMe
I
Ph
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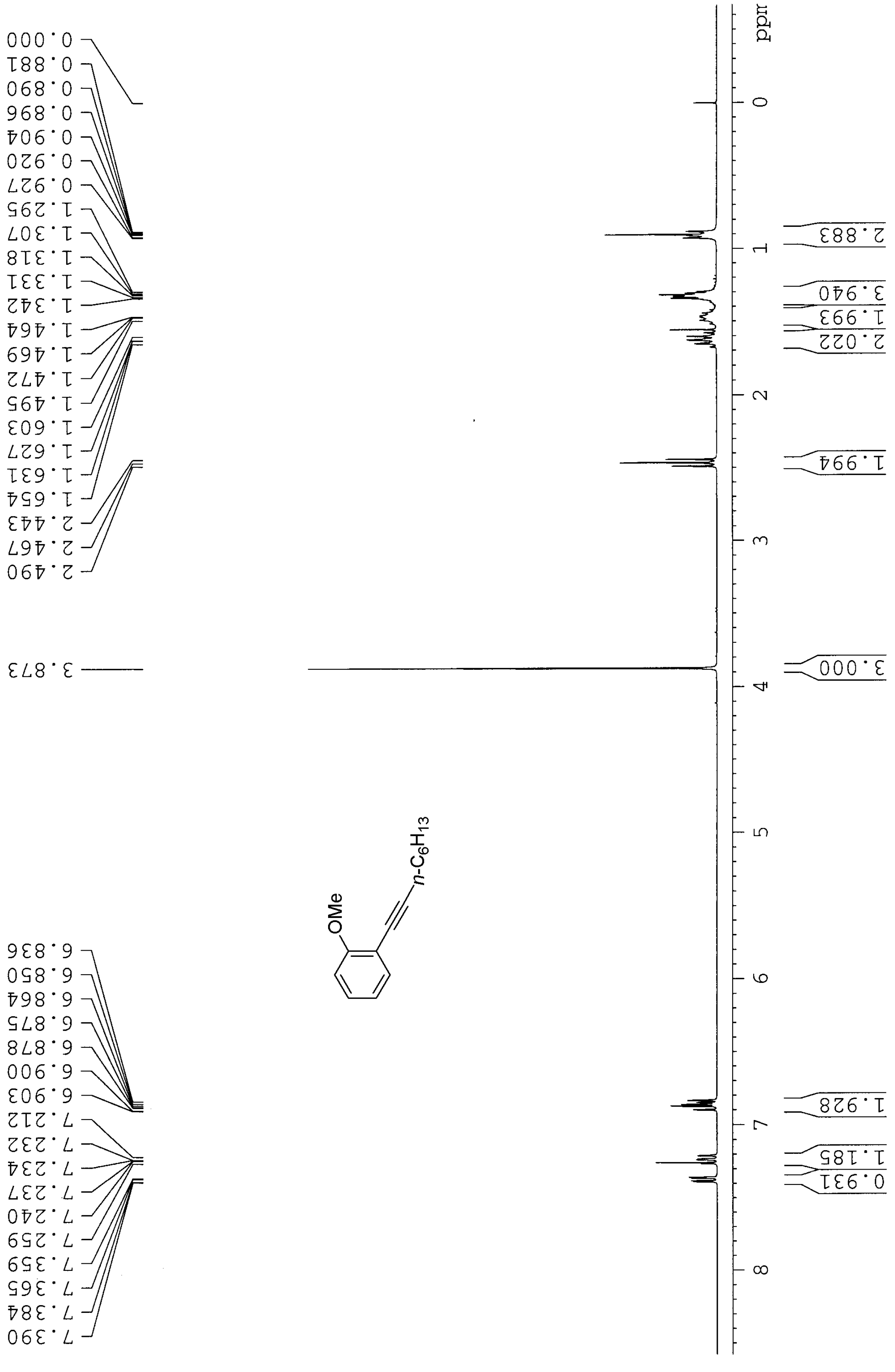
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1.342
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1.469
1.472
1.495
1.603
1.627
1.631
1.654
2.443
2.467
2.490
3.873
6.836
6.850
6.864
6.875
6.878
6.900
6.903
7.212
7.232
7.234
7.237
7.240
7.259
7.359
7.365
7.384
7.390
OMe
n-C6H13
2.883
3.940
1.993
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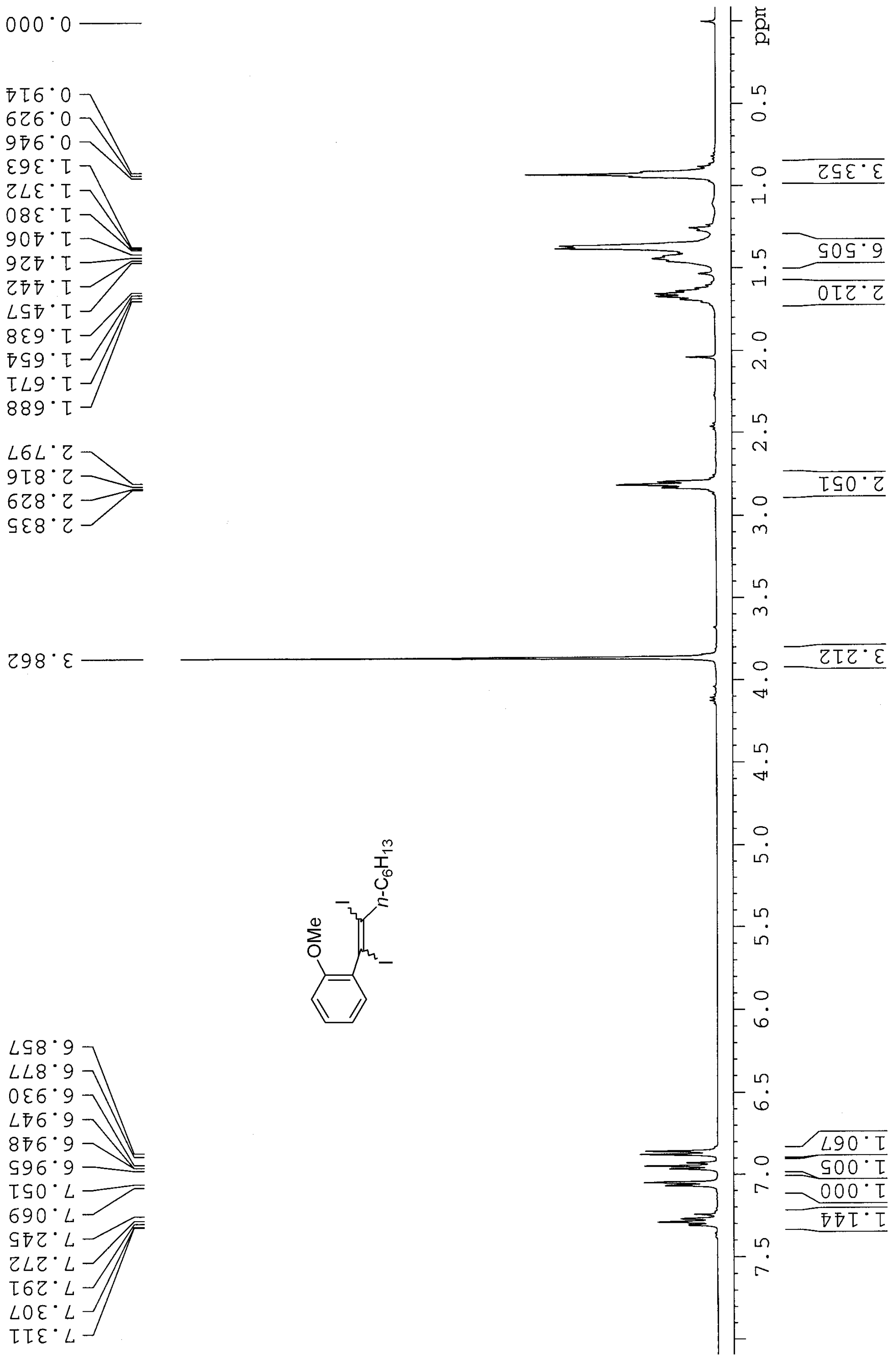
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1.457
1.638
1.654
1.671
1.688
2.797
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2.829
2.835
3.862
6.857
6.877
6.930
6.947
6.948
6.965
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7.245
7.272
7.291
7.307
7.311
OMe
I
I
n-C6H13
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6.505
2.210
2.051
3.212
1.067
1.005
1.000
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1.5
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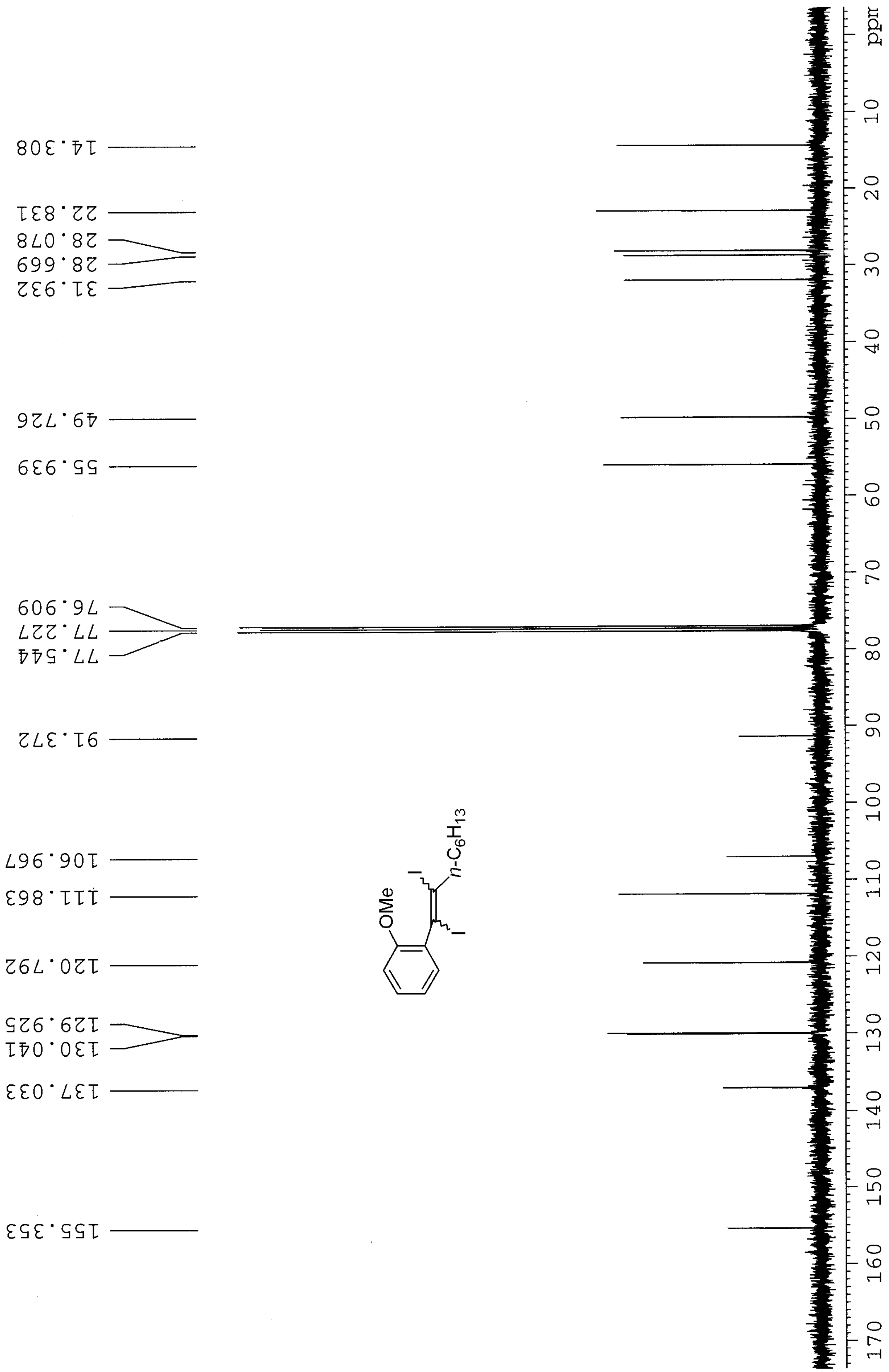
7.5



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76.909
77.227
77.544
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106.967
111.863
120.792
129.925
130.041
137.033
155.353
OMe
I
I
n-C_6H_{13}
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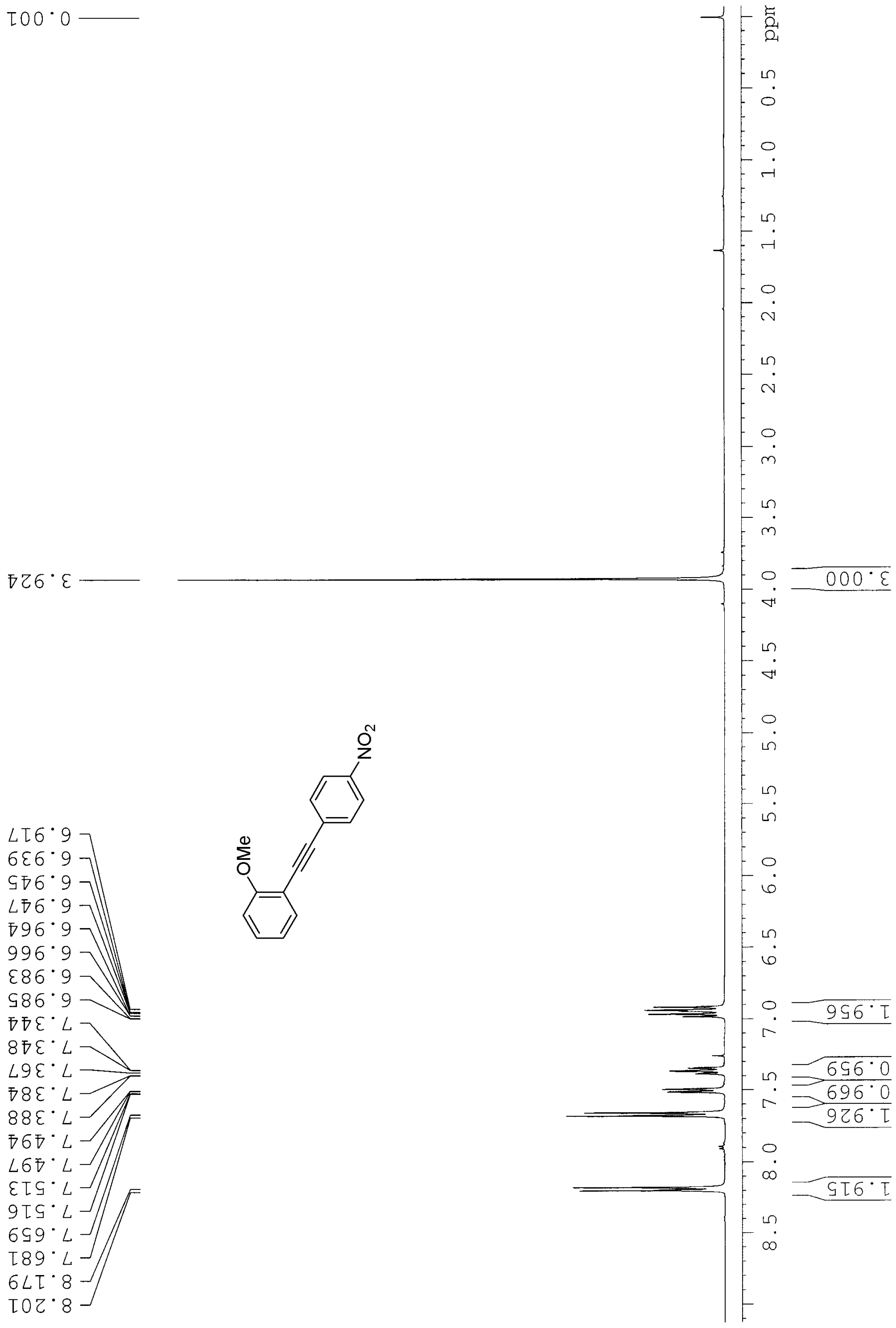
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7.367
7.384
7.388
7.494
7.497
7.513
7.516
7.659
7.681
8.179
8.201
OMe
NO2
3.000
1.956
0.959
0.969
1.926
1.915
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
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4.5
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6.0
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7.0
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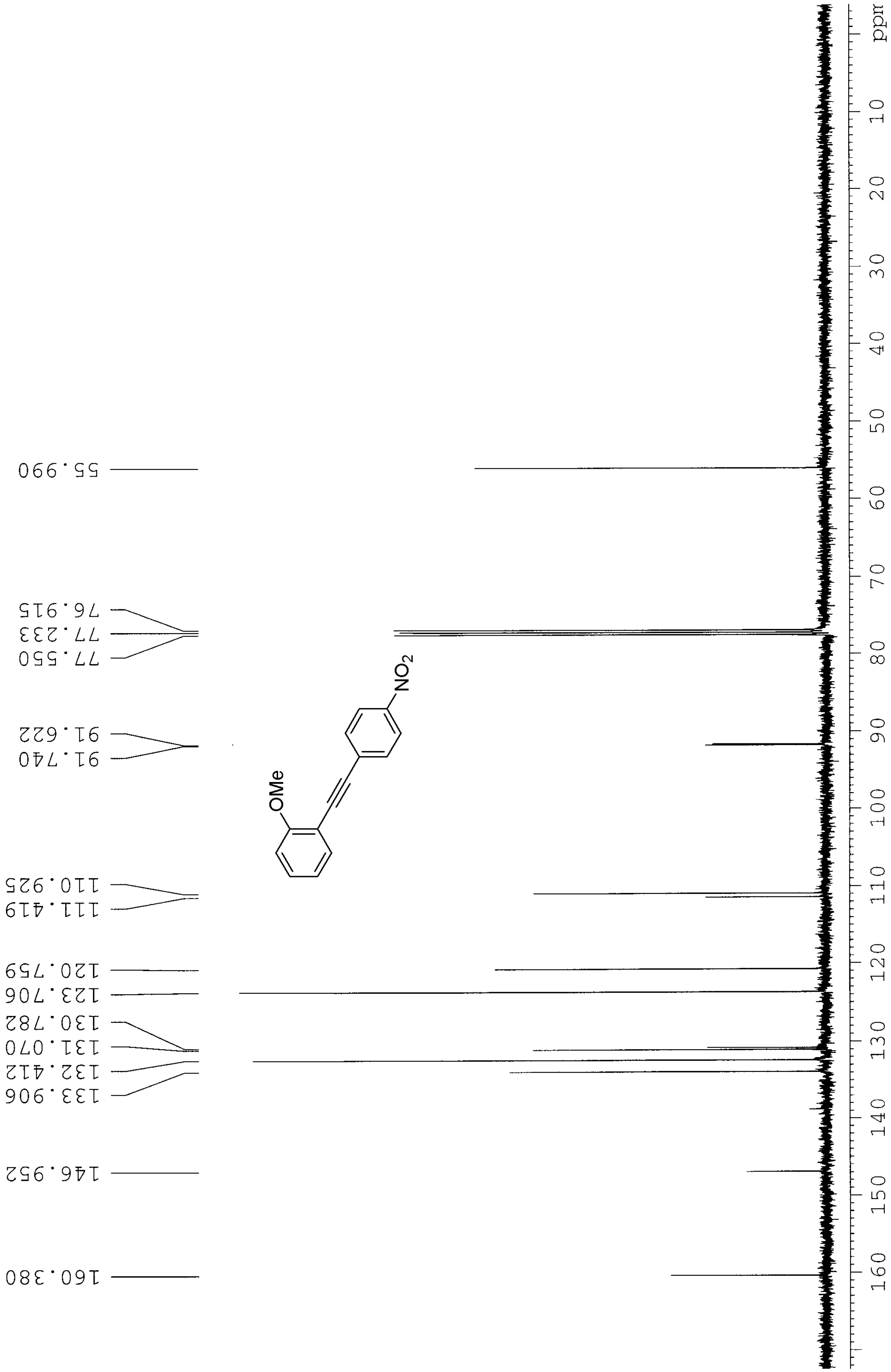
8.0
8.5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146.952
133.906
132.412
131.070
130.782
123.706
120.759
111.419
110.925
91.740
91.622
77.550
77.233
76.915
55.990
OMe
NO2
ppm
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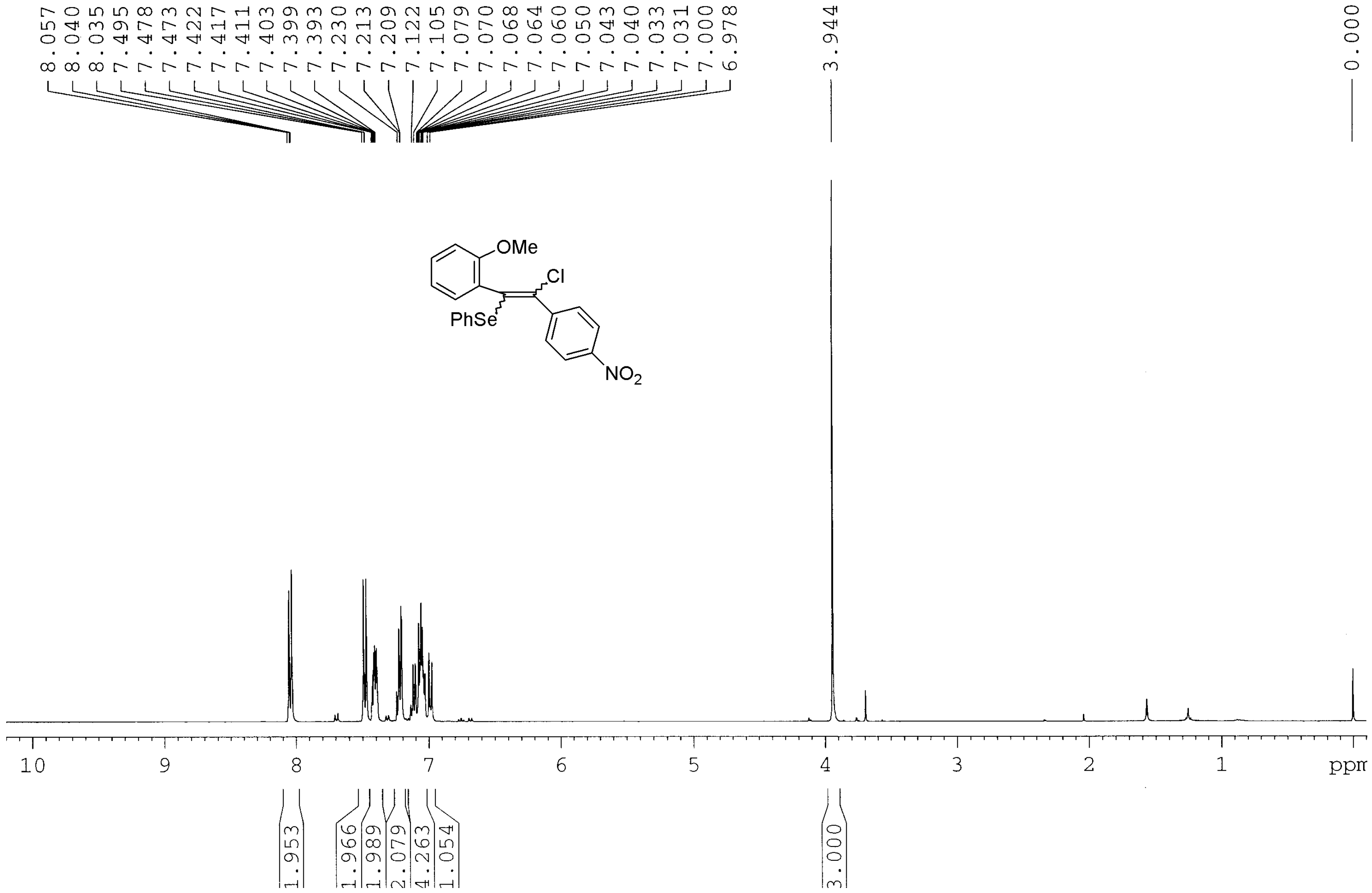
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160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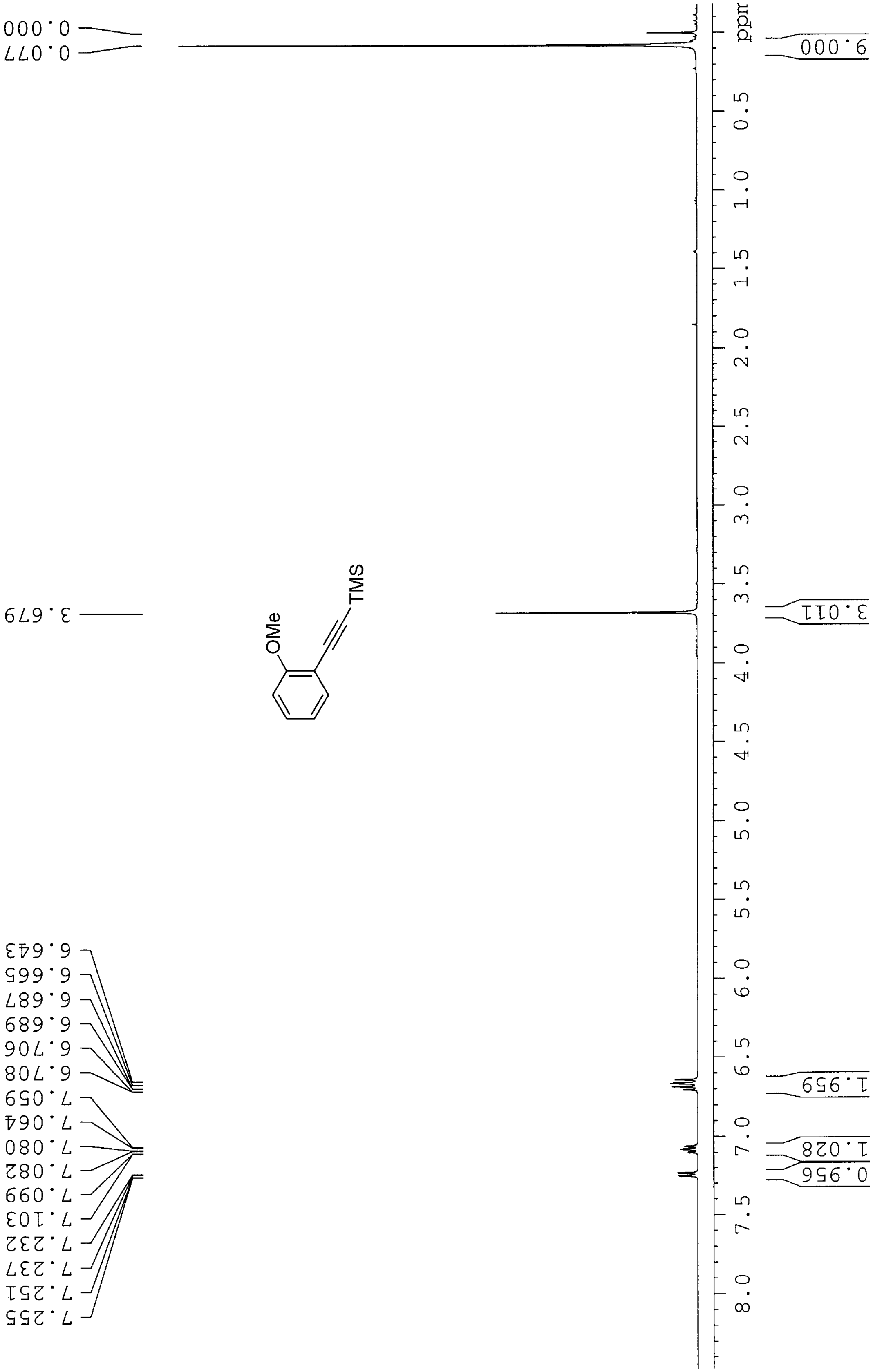
8.040
8.035
7.495
7.478
7.473
7.422
7.417
7.411
7.403
7.399
7.393
7.230
7.213
7.209
7.122
7.105
7.079
7.070
7.068
7.064
7.060
7.050
7.043
7.040
7.033
7.031
7.000
6.978
3.944
0.000
OMe
Cl
PhSe
NO2
10
9
8
7
6
5
4
3
2
1
ppm
1.953
1.966
1.989
2.079
4.263
1.054
3.000


0.000
3.944
6.978
7.000
7.031
7.033
7.040
7.043
7.050
7.060
7.064
7.068
7.070
7.079
7.105
7.122
7.209
7.213
7.230
7.393
7.399
7.403
7.411
7.417
7.422
7.473
7.478
7.495
8.035
8.040
8.057
3.000
1.054
4.263
2.079
1.989
1.966
1.953
ppm
1
2
3
4
5
6
7
8
9
10
OMe
Cl
NO$_2$
PhSe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0.077
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6.665
6.687
6.689
6.706
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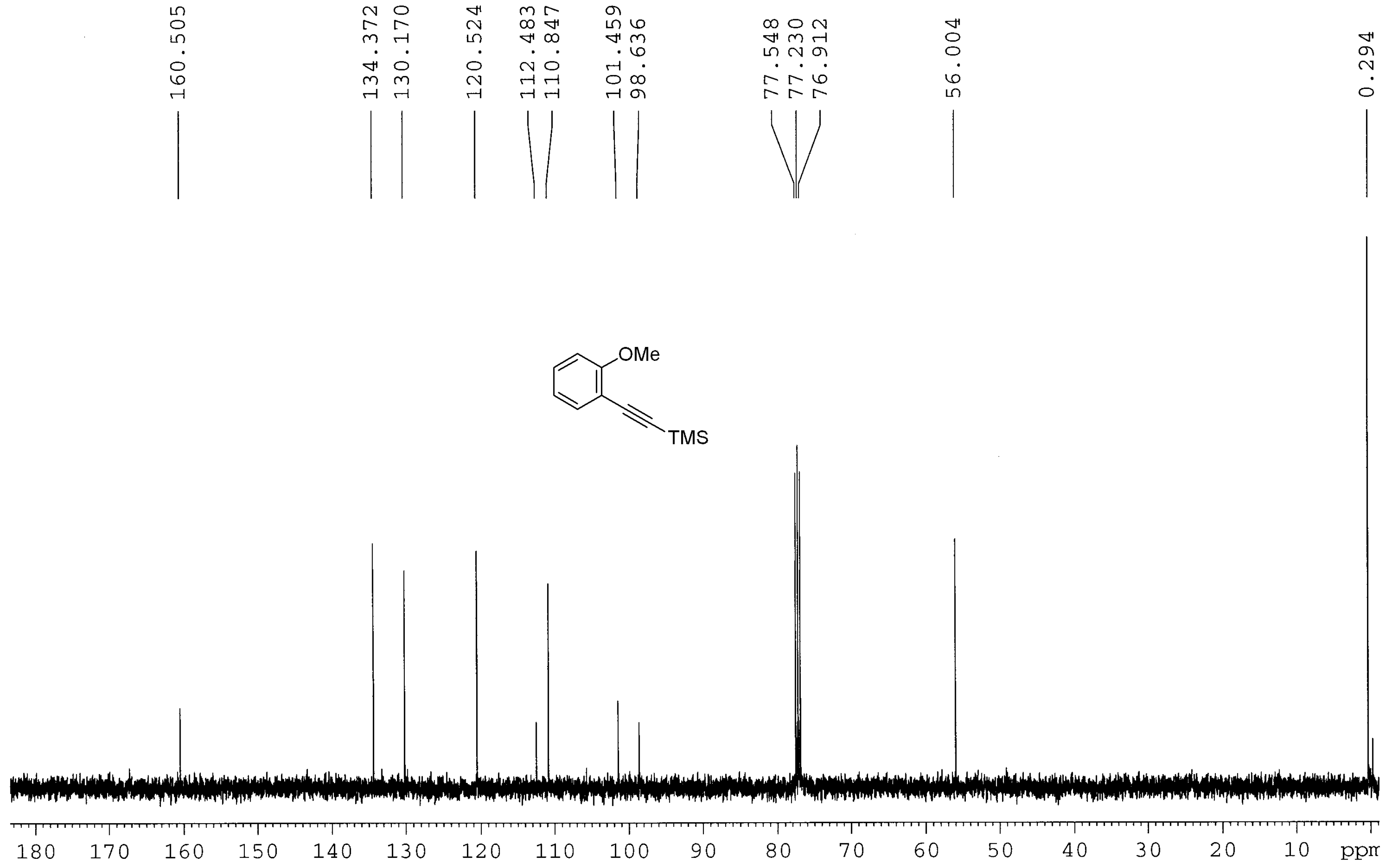
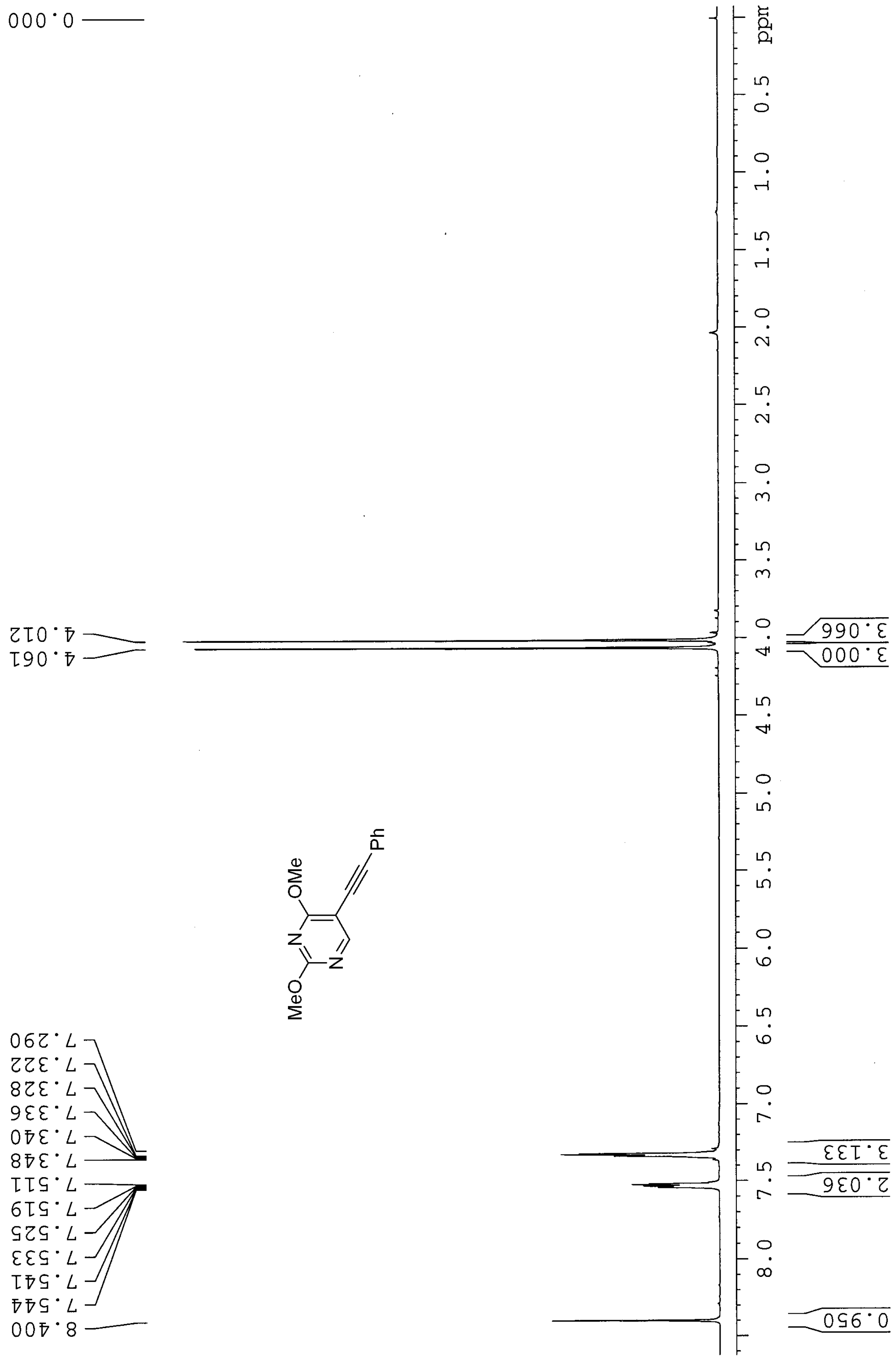
7.064
7.080
7.082
7.099
7.103
7.232
7.237
7.251
7.255
OMe
TMS
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
9.000
3.011
1.959
1.028
0.956


160.505
134.372
130.170
120.524
112.483
110.847
101.459
98.636
77.548
77.230
76.912
56.004
0.294
OMe
TMS
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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4.012
4.061
7.290
7.322
7.328
7.336
7.340
7.348
7.511
7.519
7.525
7.533
7.541
7.544
8.400
MeO
N
N
OMe
Ph
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
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6.0
6.5
7.0
7.5
8.0
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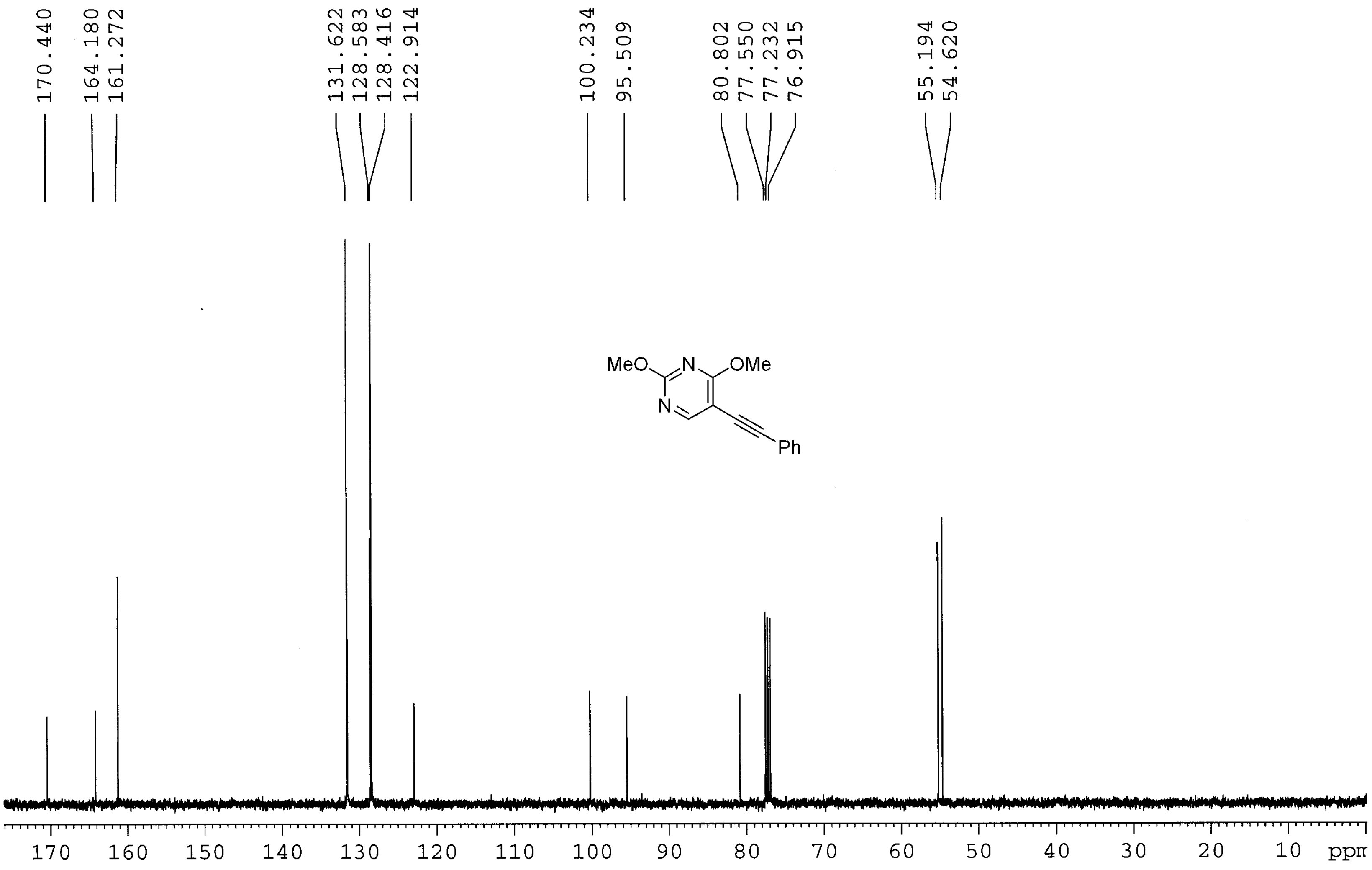
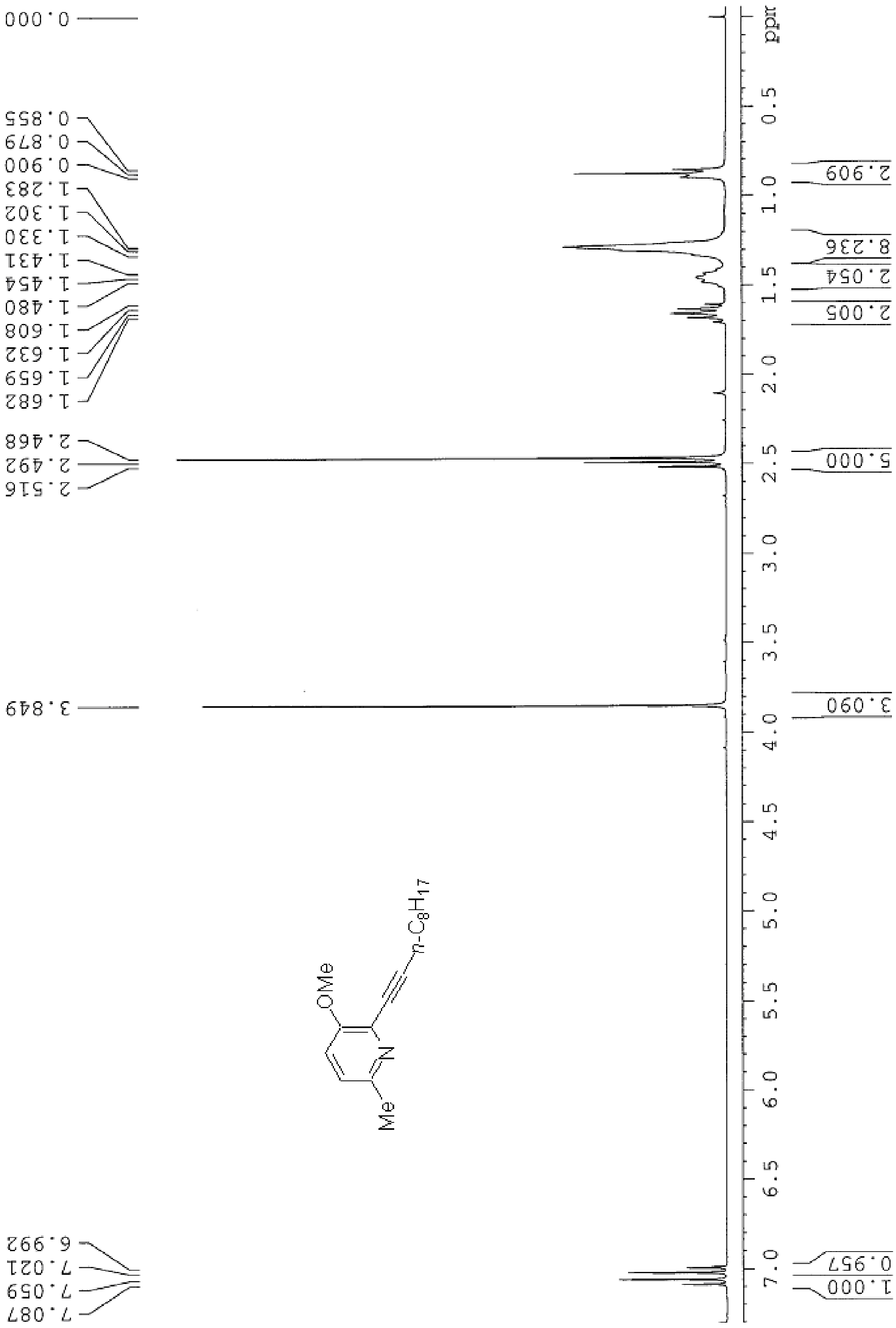
3.000
3.133
2.036
0.950


170.440
164.180
161.272
131.622
128.583
128.416
122.914
100.234
95.509
80.802
77.550
77.232
76.915
55.194
54.620
MeO
N
OMe
N
Ph
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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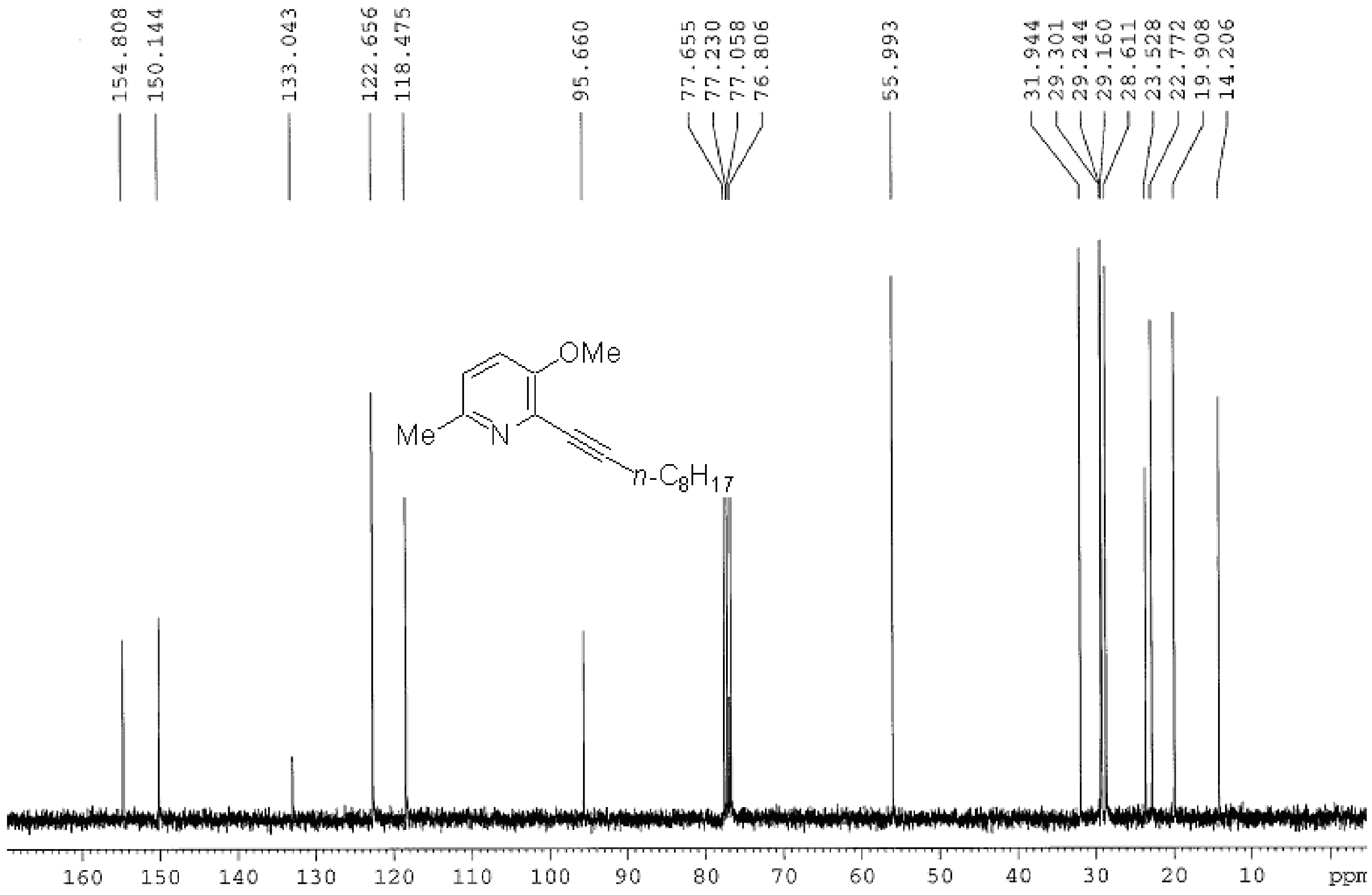
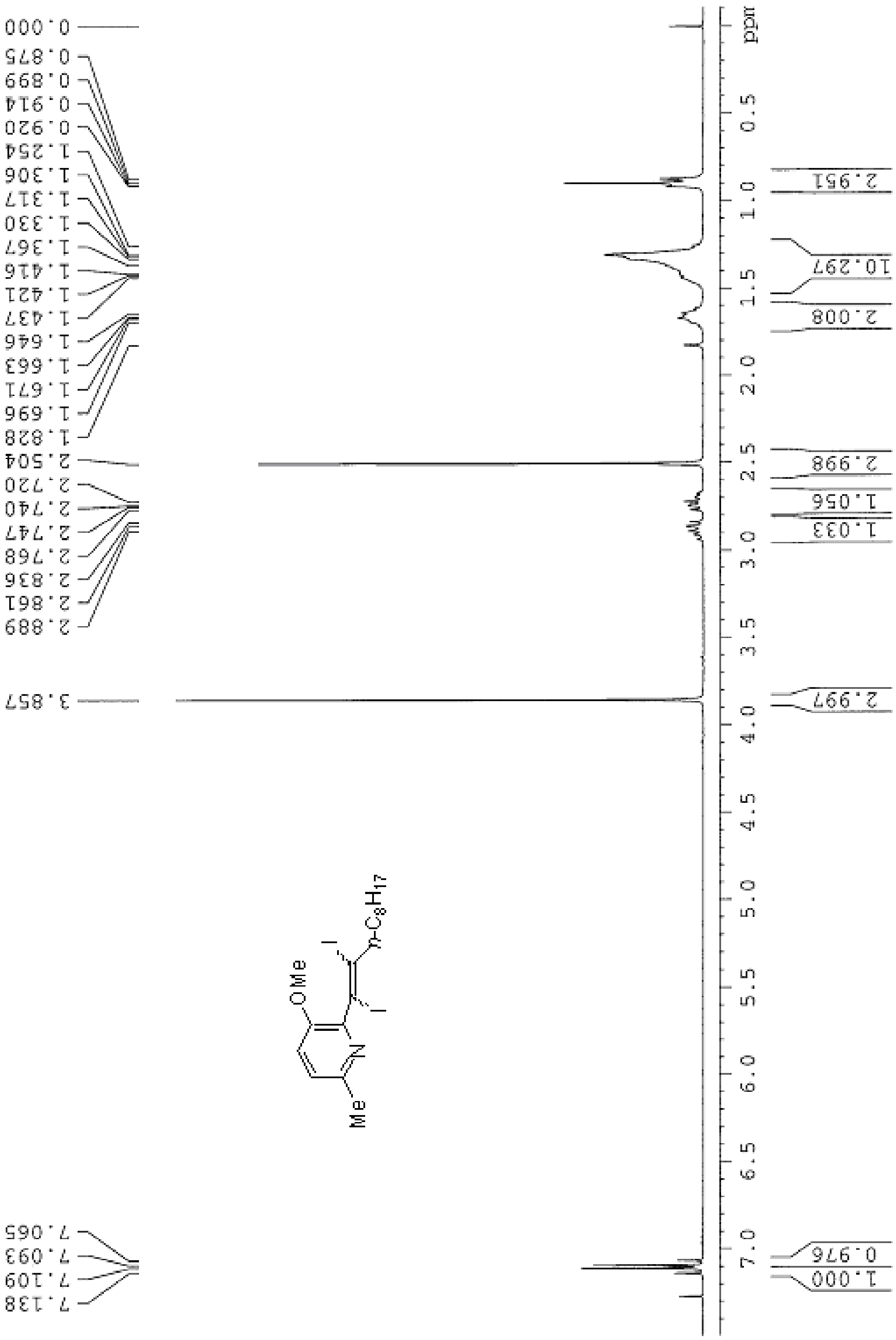
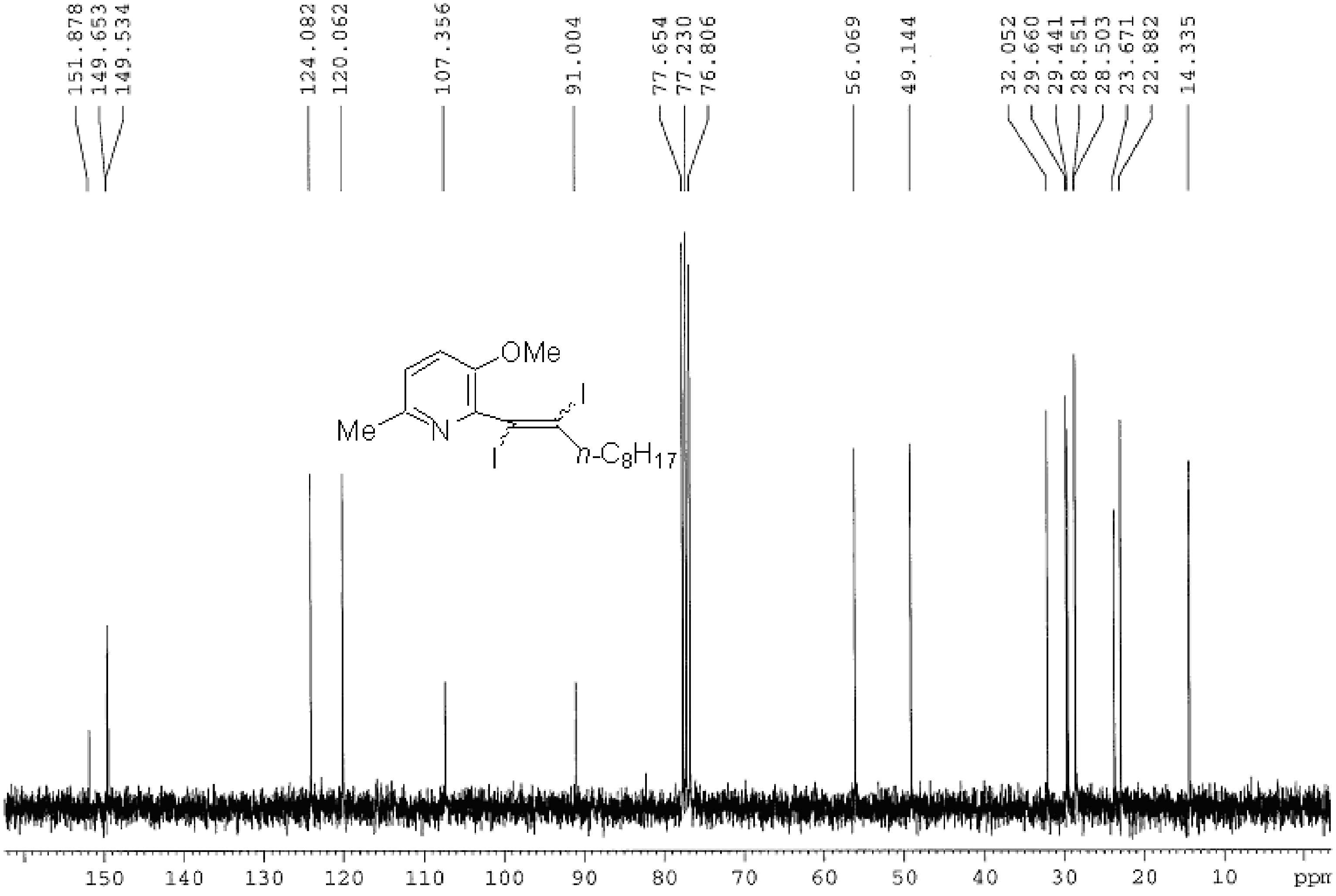
0.879
0.900
1.283
1.302
1.330
1.431
1.454
1.480
1.608
1.632
1.659
1.682
2.468
2.492
2.516
3.849
6.992
7.021
7.059
7.087
2.909
8.236
2.054
2.005
5.000
3.090
0.957
1.000
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
OMe
Me
N
n-C8H17


154.808
150.144
133.043
122.656
118.475
95.660
77.655
77.230
77.058
76.806
55.993
31.944
29.301
29.244
29.160
28.611
23.528
22.772
19.908
14.206
OMe
Me
N
n-C8H17
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


0.000
0.875
0.899
0.914
0.920
1.254
1.306
1.317
1.330
1.367
1.416
1.421
1.437
1.646
1.663
1.671
1.696
1.828
2.504
2.720
2.740
2.747
2.768
2.836
2.861
2.889
3.857
7.065
7.093
7.109
7.138
2.951
10.297
2.008
2.998
1.056
1.033
2.997
0.976
1.000
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
OMe
Me
N
I
I
n-C8H17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149.653
149.534
124.082
120.062
107.356
91.004
77.654
77.230
76.806
56.069
49.144
32.052
29.660
29.441
28.551
28.503
23.671
22.882
14.335
OMe
Me
N
I
I
n-C_8H_{17}
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm