Electronic Supplementary Material (ESI) for Chemical Communications

Palladium/Copper-Cocatalyzed Intramolecular Oxidative Coupling: an Efficient and Atom-economical Strategy for the Synthesis of 3-Acylindoles

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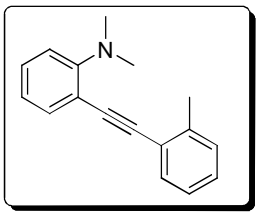
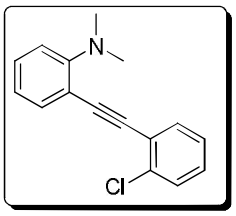
General Remarks: Column chromatography was carried out on silica gel. Unless noted ^{1}H NMR spectra were recorded on 400 MHz or 300 MHz in $CDCl_3$, ^{13}C NMR spectra were recorded on 100 MHz or 75 MHz in $CDCl_3$. IR spectra were recorded on an FT-IR spectrometer and only major peaks are reported in cm^{-1}. Melting points were determined on a microscopic apparatus and were uncorrected. All products were further characterized by HRMS (high resolution mass spectra); copies of their ^{1}H NMR and ^{13}C NMR spectra are provided. Commercially available reagents and solvents were used without further purification. Some starting metarials such as **1a**, **1b**, **1c**, **1d**, **1e**, **1g**, **1h**, **1j**, **1l**, **1n**, **1o**, **1p**, **1s**, **1z** have been reported in our previous work.[1]

Characterization data of compound 1.



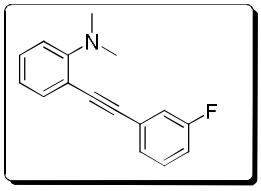
1f

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48-7.51 (m, 2 H), 7.12-7.25 (m, 4 H), 6.86-6.92 (m, 2 H), 2.98 (s, 6 H), 2.53 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 154.5, 139.7, 134.3, 131.6, 129.4, 129.1, 127.9, 125.5, 123.6, 120.5, 116.9, 115.6, 93.6, 92.7, 43.5, 20.7; IR (neat): 2943, 2916, 2784, 1592, 1489, 1452, 1328, 1049, 947, 764;



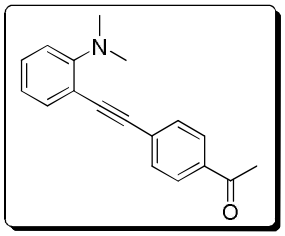
1i

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52-7.57 (m, 2 H), 7.39-7.42 (m, 1 H), 7.20-7.28 (m, 3 H), 6.87-6.92 (m, 2 H), 3.01 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 154.7, 135.5, 134.7, 133.0, 129.6, 129.2, 128.9, 126.3, 123.8, 120.3, 116.9, 114.5, 94.0, 91.4, 43.6; IR (neat): 2944, 2837, 2787, 1590, 1495, 1052, 947, 753; HRMS (APCI) m/z: calcd for $C_{16}H_{15}NCl$: M+H = 256.0888; found: 256.0888.



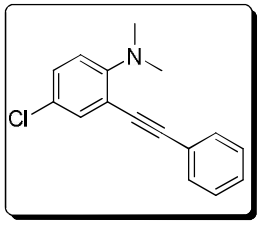
1k

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46-7.48 (m, 1 H), 7.20-7.31 (m, 4 H), 7.00-7.02 (m, 1 H), 6.86-6.99 (m, 2 H), 2.98 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 163.6, 161.2, 154.9, 134.4, 129.8, 129.6, 127.1, 125.8, 125.7, 120.4, 118.0, 117.8, 116.9, 115.3, 115.1, 114.4, 93.3, 89.9, 43.4; IR (neat): 2919, 2851, 1579, 1383, 1065, 1017, 774, 679; HRMS (ESI) m/z: calcd for $C_{16}H_{15}NF$: M+H = 240.1183; found: 240.1182.



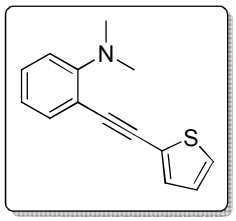
1m

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92-7.94 (m, 2 H), 7.59-7.61 (m, 2 H), 7.48-7.50 (m, 1 H), 7.25-7.29 (m, 1 H), 6.88-6.94 (m, 2 H), 3.01 (s, 6 H), 2.60 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 197.2, 154.9, 135.9, 134.5, 131.2, 129.8, 128.8, 128.2, 120.4, 116.9, 114.2, 93.9, 92.5, 43.5, 26.5; IR (neat): 2920, 2853, 2787, 1681, 1596, 1263, 951, 835, 760, 635;



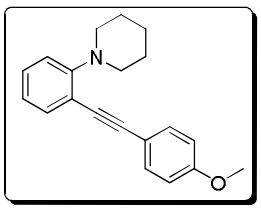
1q

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.53 (m, 2 H), 7.44 (s, 1 H), 7.32-7.37 (m, 3 H), 7.16-7.19 (m, 1 H), 6.81-6.83 (m, 1 H), 2.97 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 153.3, 133.5, 131.3, 129.0, 128.3, 125.0, 123.4, 118.1, 116.4, 95.6, 87.6, 43.4; IR (neat): 2919, 2848, 1634, 1493, 1391, 811, 758, 689; HRMS (APCI) m/z: calcd for $C_{16}H_{15}NCl$: M+H = 256.0888; found: 256.0887.



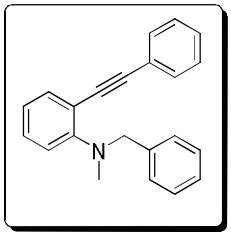
1r

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43-7.45 (m, 1 H), 7.19-7.23 (m, 3 H), 6.95-6.97 (m, 1 H), 6.83-6.88 (m, 2 H), 2.95 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 154.6, 133.3, 131.1, 129.3, 126.9, 126.8, 123.8, 120.2, 116.8, 114.4, 92.6, 87.7, 43.3; IR (neat, cm^{-1}): 3099, 3066, 2942, 2862, 2835, 2787, 1592, 1489, 1452, 1427, 1330, 1271, 1207, 945, 752, 700; HRMS (APCI) m/z: calcd for $C_{14}H_{14}NS$: M+H = 228.0841; found: 228.0842.



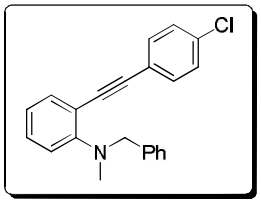
1t

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46-7.48 (m, 3 H), 7.21-7.25 (m, 1 H), 6.86-6.94 (m, 4 H), 3.81 (s, 3 H), 3.16-3.19 (m, 4 H), 1.76-1.82 (m, 4 H), 1.56-1.62 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): 159.4, 155.1, 133.6, 132.8, 128.9, 121.1, 117.7, 117.1, 116.1, 113.9, 94.4, 86.9, 55.2, 52.8, 26.4, 24.3; IR (neat): 2919, 2850, 1582, 1443, 1382, 1027, 831, 769; HRMS (APCI) m/z: calcd for $C_{20}H_{22}NO$: M+H = 292.1696; found: 292.1696.



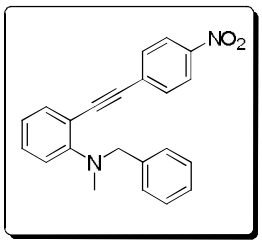
1u

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52-7.54 (m, 1 H), 7.42-7.44 (m, 2 H), 7.27-7.31 (m, 2 H), 7.19-7.25 (m, 7 H), 6.88-6.95 (m, 2 H), 4.59 (s, 2 H), 2.78 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 153.9, 139.0, 134.5, 131.2, 129.2, 128.2, 128.1, 127.9, 127.8, 126.9, 123.6, 120.5, 117.8, 115.2, 94.7, 88.7, 60.1, 39.1; IR (neat): 3059, 3027, 2850, 2797, 1593, 1490, 1446, 1362, 754, 693; HRMS (APCI) m/z: calcd for $C_{22}H_{20}N$: M+H = 298.1590; found: 298.1595.



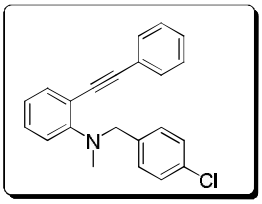
1v

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58-7.59 (m, 1 H), 7.55-7.57 (m, 1 H), 7.42-7.51 (m, 2 H), 7.21-7.32 (m, 3 H), 7.15-7.18 (m, 1 H), 7.02-7.07 (m, 3 H), 6.88-6.96 (m, 2 H), 4.58 (s, 2 H), 2.77 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 154.0, 139.1, 138.7, 134.5, 133.7, 132.4, 130.5, 129.5, 128.4, 128.3, 127.7, 126.9, 122.1, 120.5, 117.7, 114.6, 93.5, 89.6, 60.1, 38.9; IR (neat): 3060, 2922, 2850, 2797, 1591, 1490, 1090, 826, 751, 699; HRMS (APCI) m/z: calcd for $C_{22}H_{19}NCl$: M+H = 332.1201; found: 332.1202.



1w

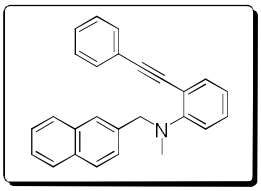
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02-8.05 (m, 2 H), 7.52-7.54 (m, 1 H), 7.44-7.46 (m, 2 H), 7.27-7.35 (m, 4 H), 7.10-7.14 (m, 2 H), 6.98-6.99 (m, 1 H), 6.90-6.94 (m, 1 H), 4.63 (s, 2 H), 2.82 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 154.4, 146.5, 139.1, 134.9, 131.7, 130.6, 130.4, 128.4, 128.2, 127.5, 127.0, 123.3, 120.3, 117.5, 113.2, 94.4, 92.9, 60.2, 38.9; IR (neat): 2919, 2851, 1589, 1383, 1075, 1024, 771; HRMS (APCI) m/z: calcd for $C_{22}H_{19}N_2O_2$: M+H = 343.1441; found: 343.1443.



1x

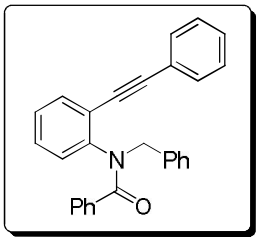
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51-7.53 (m, 1 H), 7.35-7.37 (m, 2 H), 7.18-7.25 (m, 8 H), 6.89-6.93 (m, 2 H), 4.51 (s, 2 H), 2.75 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 153.7, 137.6, 134.5, 132.5, 131.2, 129.3, 128.4, 128.2, 127.9, 123.5, 120.8, 117.9, 115.3, 94.8, 88.5, 59.5, 39.1; IR

(neat): 3059, 2921, 2851, 2797, 1592, 1489, 1443, 1360, 1090, 754, 690; HRMS (APCI) m/z: calcd for $C_{22}H_{19}NCl$: M+H = 332.1201; found: 332.1208.



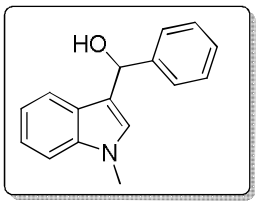
1y

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (s, 1 H), 7.73-7.80 (m, 3 H), 7.54-7.59 (m, 2 H), 7.40-7.43 (m, 2 H), 7.09-7.25 (m, 6 H), 6.90-6.98 (m, 2 H), 4.70 (s, 2 H), 2.80 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 154.1, 136.7, 134.5, 133.5, 132.8, 131.2, 129.3, 128.0, 127.9, 127.8, 127.7, 126.5, 126.4, 125.9, 125.4, 123.6, 120.7, 117.9, 115.4, 94.8, 88.7, 60.4, 39.2; IR (neat, cm^{-1}): 2923, 2846, 1592, 1487, 939, 811, 749; HRMS (ESI) m/z: calcd for $C_{26}H_{22}N$: M+H = 348.1747; found: 348.1752.



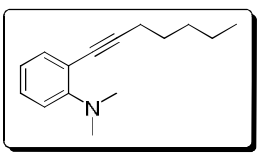
N-benzyl-*N*-(2-(phenylethynyl)phenyl)benzamide

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55-7.57 (m, 2 H), 7.32-7.40 (m, 8 H), 7.03-7.25 (m, 8 H), 6.83-6.85 (m, 1 H), 5.63-5.66 (m, 1 H), 4.74-4.78 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): 170.9, 144.3, 137.1, 136.2, 132.7, 131.6, 129.7, 129.5, 129.2, 128.7, 128.6, 128.4, 128.2, 127.4, 127.3, 127.2, 122.8, 122.7, 95.1, 86.1, 52.8; IR (neat): 3061, 3030, 2925, 1642, 1495, 1446, 1384, 1299, 911, 764, 695; HRMS (APCI) m/z: calcd for $C_{28}H_{22}NO$: M+H = 388.1696; found: 388.1698.



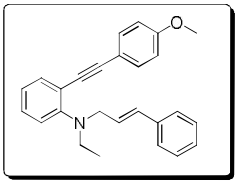
3

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53-7.55 (m, 1 H), 7.42-7.44 (m, 2 H), 7.16-7.31 (m, 6 H), 7.02-7.06 (m, 1 H), 6.68 (s, 1 H), 6.04 (s, 1 H), 3.57 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 143.7, 137.2, 128.3, 128.1, 127.4, 127.3, 126.8, 126.3, 126.2, 121.8, 119.6, 119.2, 118.2, 109.2, 69.9, 32.5; IR (neat, cm^{-1}): 3055, 2922, 2873, 1617, 1545, 1464, 1377, 1329, 1056, 739, 701;



2-(hept-1-ynyl)-N,N-dimethylaniline (1z)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34-7.36 (m, 1 H), 7.13-7.17 (m, 1 H), 6.80-6.86 (m, 2 H), 2.89 (s, 6 H), 2.44-2.47 (m, 2 H), 1.58-1.66 (m, 2 H), 1.30-1.48 (m, 4 H), 0.89-0.93 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 154.4, 134.1, 128.1, 120.5, 116.7, 116.4, 95.5, 79.4, 43.3, 31.0, 28.3, 22.1, 19.7, 13.8;

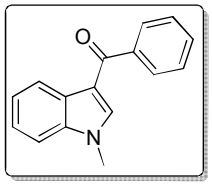

N-cinnamyl-*N*-ethyl-2-((4-methoxyphenyl)ethynyl)aniline

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44-7.50 (m, 3 H), 7.18-7.32 (m, 6 H), 6.94-6.96 (m, 1 H), 6.85-6.89 (m, 1 H), 6.80-6.82 (m, 2 H), 6.58 (d, *J* = 7.8 Hz, 1 H), 6.32-6.39 (m, 1 H), 4.09 (d, *J* = 6.0 Hz, 2 H), 3.76 (s, 3 H), 3.39 (dd, J = 13.4, 6.4 Hz, 2 H), 1.12-1.16 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 159.3, 152.4, 137.1, 134.3, 132.7, 131.6, 128.6, 128.4, 127.9, 127.2, 126.3, 120.5, 119.2, 116.4, 116.0, 113.9, 94.1, 87.7, 55.2, 54.9, 45.1, 12.5; HRMS (APCI) m/z: calcd for $C_{26}H_{26}NO$: M+H = 368.2009; found: 368.2024.

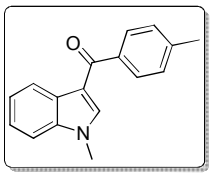
Typical procedure for the synthesis of product 2

To a solution of *N*,*N*-dimethyl-2-(phenylethynyl)aniline (**1a**) (44.2 mg, 0.200 mmol) in toluene (2 mL) was added $PdBr_2$ (5.3 mg, 10 mol%), CuI (3.8 mg, 10 mol%), LiCl (17 mg, 2.0 equiv.) and 120 uL TBHP (5 M in decane). The reaction mixture was then stirred in air at 100 ºC for 8 h. The resulting mixture was quenched with water and extracted twice with EtOAc. The combined organic extracts were washed with brine, dried over $MgSO_4$ and concentrated. Purification of the crude product by flash column chromatography afforded **2a** in 61% yield (28.7 mg).

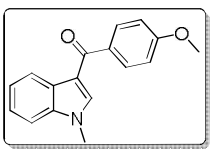
Characterization data of compound 2.


2a, m.p. = 114-116 ^{o}C

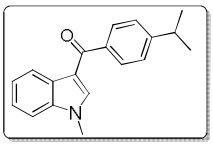
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.42-8.44 (m, 1 H), 7.79-7.82 (m, 2 H), 7.46-7.56 (m, 4 H), 7.33-7.38 (m, 3 H), 3.83 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 190.8, 140.9, 137.9, 137.5, 131.0, 128.6, 128.5, 128.2, 127.1, 126.9, 123.6, 122.7, 115.6, 109.6, 33.5; IR (neat, cm^{-1}): 2957, 2926, 1732, 1617, 1524, 1465, 1368, 1233, 1073, 873, 748, 718, 669; HRMS (APCI) m/z: calcd for $C_{16}H_{14}NO$: M+H = 236.1070; found: 236.1080.


2b, m.p. = 134-135 ^{o}C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.40-8.42 (m, 1 H), 7.71-7.74 (m, 2 H), 7.52 (s, 1 H), 7.25-7.52 (m, 5 H), 3.82 (s, 3 H), 2.43 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 190.6, 141.5, 138.1, 137.5, 128.8, 127.2, 123.5, 122.7, 122.5, 115.6, 109.5, 33.5, 21.5; IR (neat, cm^{-1}): 2919, 1617, 1524, 1465, 1368, 1234, 1072, 878, 748; HRMS (APCI) m/z: calcd for $C_{17}H_{16}NO$: M+H = 250.1226; found: 250.1237.

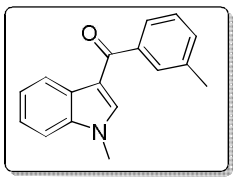

2c, m.p. = 114-116 ^{o}C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.36-8.38 (m, 1 H), 7.81-7.84 (m, 2 H), 7.52-7.57 (m, 1 H), 7.29-7.34 (m, 3 H), 6.86-6.98 (m, 2 H), 3.87 (s, 3 H), 3.82 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$):189.7, 162.1, 137.4, 137.0, 133.4, 130.8, 127.3, 123.4, 122.6, 122.4, 113.5, 109.5, 55.4, 33.4; IR (neat, cm^{-1}): 2930, 1740, 1608, 1465, 1367, 1255, 1233, 1174, 1124, 1028, 878, 844, 751; HRMS (APCI) m/z: calcd for $C_{17}H_{16}NO_2$: M+H = 266.1176; found: 266.1181.



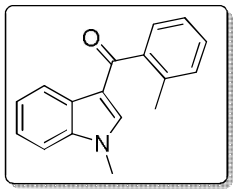
2d

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.41-8.44 (m, 1 H), 7.75-7.77 (m, 2 H), 7.54 (s, 1 H), 7.31-7.35 (m, 5 H), 3.82 (s, 3 H), 2.95-3.02 (m, 1 H), 1.29-1.31 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 190.6, 152.3, 138.5, 137.6, 137.5, 128.9, 127.2, 126.3, 123.5, 122.7, 122.5, 115.7, 109.5, 34.1, 33.4, 23.8; IR (neat, cm^{-1}): 2960, 2922, 1705, 1612, 1524, 1464, 1381, 1234, 1073, 878, 749; HRMS (APCI) m/z: calcd for $C_{19}H_{20}NO$: M+H = 278.1539; found: 278.1551.



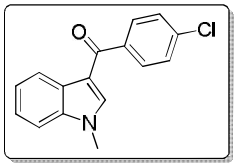
2e, m.p. = 102-104 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.41-8.43 (m, 1 H), 7.58-7.62 (m, 2 H), 7.52 (s, 1 H), 7.32-7.38 (m, 5 H), 3.83 (s, 3 H), 2.43 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 191.0, 140.9, 138.1, 137.8, 137.5, 131.8, 129.1, 128.5, 128.0, 127.6, 127.2, 126.9, 125.8, 123.6, 122.7, 122.6, 115.7, 109.6, 33.5, 21.4; IR (neat, cm^{-1}): 2921, 1697, 1621, 1580, 1524, 1464, 1368, 1243, 1123, 1074, 750; HRMS (APCI) m/z: calcd for $C_{17}H_{16}NO$: M+H = 250.1226; found: 250.1234.



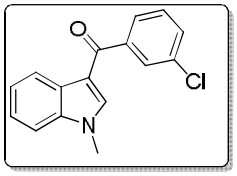
2f, m.p. = 130-132 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.36-8.38 (m, 1 H), 7.32-7.39 (m, 5 H), 7.28-7.30 (m, 1 H), 7.22-7.26 (m, 2 H), 3.78 (s, 3 H), 2.38 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 192.8, 141.1, 138.5, 137.7, 135.8, 130.7, 129.2, 127.4, 126.6, 125.0, 123.6, 122.8, 122.6, 117.1, 109.7, 33.5, 19.5; IR (neat, cm^{-1}): 2920, 1621, 1524, 1464, 1370, 1232, 1126, 1071, 879, 752, 660; HRMS (APCI) m/z: calcd for $C_{17}H_{16}NO$: M+H = 250.1226; found: 250.1232.



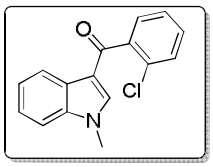
2g, m.p. = 99-100 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.35-8.38 (m, 1 H), 7.72-7.74 (m, 2 H), 7.48 (s, 1 H), 7.42-7.44 (m, 2 H), 7.34-7.35 (m, 3 H), 3.82 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 189.4, 139.1, 137.7, 137.5, 137.2, 130.0, 128.5, 128.4, 127.5, 127.0, 126.9, 123.7, 122.8, 122.6, 115.3, 109.7, 33.5; IR (neat, cm^{-1}): 2919, 1617, 1524, 1463, 1381, 1233, 1088, 1014, 771, 753; HRMS (APCI) m/z: calcd for $C_{16}H_{13}NOCl$: M+H = 270.0680; found: 270.0689.



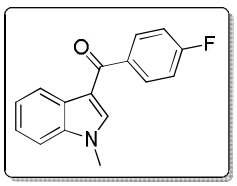
2h, m.p. = 130-132 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.38-8.40 (m, 1 H), 7.76 (s, 1 H), 7.64-7.66 (m, 1 H), 7.48 (s, 1 H), 7.34-7.41 (m, 5 H), 3.82 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 188.9, 142.5, 137.8, 137.6, 134.3, 130.9, 129.6, 128.5, 127.5, 127.0, 126.9, 126.7, 123.8, 122.9, 122.6, 115.2, 109.7, 33.5; IR (neat, cm^{-1}): 2919, 1613, 1521, 1370, 1226, 1075, 880, 749, 726; HRMS (APCI) m/z: calcd for $C_{16}H_{13}NOCl$: M+H = 270.0680; found: 270.0688.



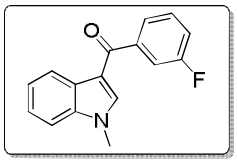
2i, m.p. = 150-152 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.35-8.38 (m, 1 H), 7.40-7.48 (m, 3 H), 7.34-7.38 (m, 4 H), 7.29 (s, 1 H), 3.79 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 188.8, 140.5, 138.8, 137.8, 130.9, 130.3, 130.0, 128.7, 128.5, 126.9, 126.4, 123.8, 123.0, 122.6, 116.4, 109.7, 33.6; IR (neat, cm^{-1}): 2920, 1627, 1526, 1464, 1378, 1229, 1078, 770, 751; HRMS (APCI) m/z: calcd for $C_{16}H_{13}NOCl$: M+H = 270.0680; found: 270.0687.



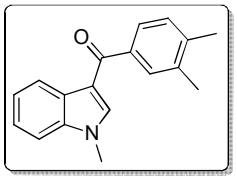
2j, m.p. = 130-132 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.37-8.39 (m, 1 H), 7.83-7.86 (m, 2 H), 7.52 (s, 1 H), 7.36-7.38 (m, 3 H), 7.14-7.18 (m, 2 H), 3.86 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 189.3, 137.5, 137.1, 131.0, 130.9, 128.5, 127.1, 126.9, 123.7, 122.7, 115.5, 115.4, 115.2, 109.6, 33.6; IR (neat, cm^{-1}): 2919, 1620, 1595, 1464, 1380, 1231, 1072, 771; HRMS (APCI) m/z: calcd for $C_{16}H_{13}NOF$: M+H = 254.0976; found: 254.0982.



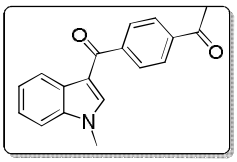
2k

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.39-8.42 (m, 1 H), 7.51-7.59 (m, 1 H), 7.41-7.50 (m, 3 H), 7.34-7.37 (m, 3 H), 7.20-7.25 (m, 1 H), 3.83 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 189.0, 163.7, 161.3, 142.9, 137.8, 137.5, 129.9, 127.0, 124.3, 123.8, 122.9, 122.6, 118.0, 117.8, 115.6, 115.4, 115.2, 109.7, 33.5; IR (neat, cm^{-1}): 3065, 2924, 1618, 1580, 1523, 1465, 1368, 1242, 1120, 747; HRMS (ESI) m/z: calcd for $C_{16}H_{13}NFO$: M+H = 254.0976; found: 254.0973.



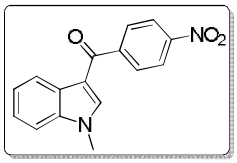
2l, m.p. = 160-162 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.40-8.42 (m, 1 H), 7.62 (s, 1 H), 7.54-7.57 (m, 2 H), 7.32-7.36 (m, 3 H), 7.22-7.24 (m, 1 H), 3.84 (s, 3 H), 2.34 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): 190.8, 140.2, 138.6, 137.5, 136.6, 129.9, 129.3, 127.3, 126.4, 123.5, 122.8, 122.5, 115.8, 109.5, 33.5, 19.8; IR (neat, cm^{-1}): 2919, 1609, 1570, 1523, 1463, 1382, 1071, 1023, 773, 750; HRMS (APCI) m/z: calcd for $C_{18}H_{18}NO$: M+H = 264.1383; found: 264.1390.



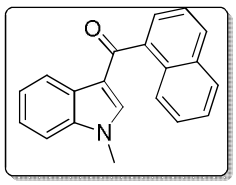
2m, m.p. = 120-122 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.39-8.41 (m, 1 H), 8.01-8.03 (m, 2 H), 7.82-7.84 (m, 2 H), 7.48 (s, 1 H), 7.33-7.36 (m, 3 H), 3.83 (s, 3 H), 2.64 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 197.6, 189.7, 144.6, 138.6, 138.1, 137.5, 128.6, 128.2, 126.9, 123.8, 122.9, 122.5, 115.3, 109.7, 33.5, 26.7; IR (neat, cm^{-1}): 3010, 2919, 1683, 1616, 1524, 1464, 1377, 1264, 1233, 1075, 749, 706; HRMS (APCI) m/z: calcd for $C_{18}H_{16}NO_2$: M+H = 278.1176; found: 278.1189.



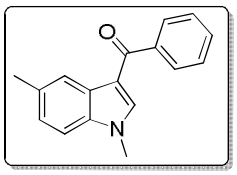
2n, m.p. = 192-194 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.38-8.40 (m, 1 H), 8.30-8.32 (m, 2 H), 7.91-7.93 (m, 2 H), 7.49 (s, 1 H), 7.35-7.40 (m, 3 H), 3.87 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 188.3, 149.1, 146.2, 138.1, 137.7, 129.3, 128.5, 127.6, 126.9, 126.8, 124.1, 123.5, 123.2, 122.6, 115.2, 109.8, 33.7; IR (neat, cm^{-1}): 2920, 1737, 1596, 1383, 1070, 1026, 770; HRMS (APCI) m/z: calcd for $C_{16}H_{13}N_2O_3$: M+H = 281.0921; found: 281.0923.



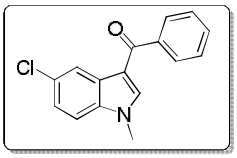
2o, m.p. = 180-182 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49-8.51 (m, 1 H), 8.15-8.17 (m, 1 H), 7.88-7.96 (m, 2 H), 7.62-7.64 (m, 1 H), 7.50-7.53 (m, 3 H), 7.34-7.49 (m, 3 H), 7.29 (s, 1 H), 3.73 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 191.9, 139.0, 138.9, 137.7, 133.7, 130.7, 129.9, 128.5, 128.1, 127.6, 126.9, 126.8, 126.7, 126.2, 125.9, 125.6, 124.5, 123.7, 122.9, 122.8, 117.6, 109.7, 33.5; IR (neat, cm^{-1}): 2920, 1614, 1523, 1464, 1367, 1233, 1089, 887, 791, 751; HRMS (APCI) m/z: calcd for $C_{20}H_{16}NO$: M+H = 286.1226; found: 286.1235.



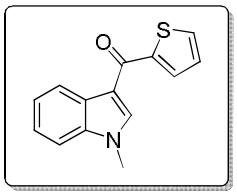
2p

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26 (s, 1 H), 7.79-7.81 (m, 2 H), 7.45-7.54 (m, 4 H), 7.25-7.27 (m, 1 H), 7.17-7.19 (m, 1 H), 3.81 (s, 3 H), 2.51 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 190.9, 141.0, 138.0, 135.9, 132.4, 130.9, 128.6, 128.2, 127.4, 125.2, 122.4, 115.1, 109.2, 33.5, 21.5; IR (neat, cm^{-1}): 2920, 1618, 1524, 1459, 1364, 1237, 1068, 760, 718, 669; HRMS (APCI) m/z: calcd for $C_{17}H_{16}NO$: M+H = 250.1226; found: 250.1236.



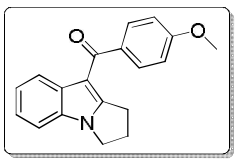
2q, m.p. = 140-142 ^{o}C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (s, 1 H), 7.77-7.79 (m, 2 H), 7.46-7.57 (m, 4 H), 7.25-7.30 (m, 2 H), 3.82 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 190.4, 140.5, 138.5, 135.9, 131.2, 128.8, 128.6, 128.3, 128.2, 124.0, 122.3, 115.2, 110.6, 33.7; IR (neat, cm^{-1}): 2924, 1690, 1610, 1522, 1458, 1363, 1232, 1080, 806, 709, 667; HRMS (APCI) m/z: calcd for $C_{16}H_{13}NOCl$: M+H = 270.0680; found: 270.0684.



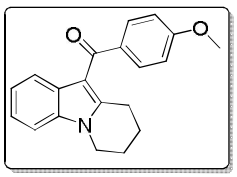
2r

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.39-8.43 (m, 1 H), 7.79 (s, 1 H), 7.73-7.74 (m, 1 H), 7.58-7.59 (m, 1 H), 7.25-7.36 (m, 3 H), 7.13-7.15 (m, 1 H), 3.85 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 181.3, 145.4, 137.4, 136.0, 131.2, 130.9, 127.4, 127.2, 123.7, 122.6, 115.4, 109.6, 33.5; IR (neat, cm^{-1}): 3108, 2926, 1729, 1600, 1521, 1464, 1420, 1368, 1233, 1123, 809, 748; HRMS (APCI) m/z: calcd for $C_{14}H_{12}NOS$: M+H = 242.0634; found: 242.0635.



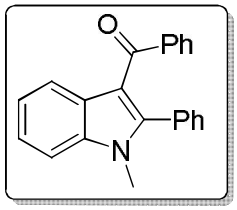
2s, m.p. = 190-192 ^{o}C

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96-7.98 (m, 1 H), 7.71-7.73 (m, 2 H), 7.19-7.27 (m, 3 H), 6.93-6.96 (m, 2 H), 4.09-4.12 (m, 2 H), 3.87 (s, 3 H), 2.95-2.98 (m, 2 H), 2.54-2.58 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): 190.7, 161.9, 152.7, 134.0, 132.9, 131.4, 130.5, 122.1, 121.9, 113.3, 109.7, 109.4, 55.3, 44.4, 26.9; IR (neat, cm^{-1}): 2924, 1712, 1604, 1510, 1456, 1418, 1382, 1251, 1220, 1163, 1028, 751; HRMS (APCI) m/z: calcd for $C_{19}H_{18}NO_2$: M+H = 292.1332; found: 292.1337.



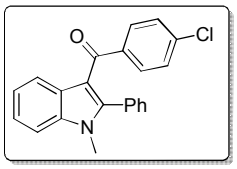
2t

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75-7.77 (m, 1 H), 7.34-7.38 (m, 2 H), 7.29-7.31 (m, 1 H), 7.16-7.20 (m, 1 H), 7.07-7.11 (m, 1 H), 6.92-6.94 (m, 2 H), 4.09-4.13 (m, 2 H), 3.88 (s, 3 H), 3.15-3.19 (m, 2 H), 2.09-2.15 (m, 2 H), 1.87-1.93 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): 191.2, 162.2, 145.5, 135.9, 134.1, 131.1, 128.5, 127.0, 126.9, 121.5, 120.9, 113.3, 112.2, 108.9, 55.3, 42.5, 25.0, 22.5, 20.2; IR (neat, cm^{-1}): 2922, 1601, 1506, 1456, 1420, 1379, 1253, 1162, 1027, 750; HRMS (APCI) m/z: calcd for $C_{20}H_{20}NO_2$: M+H = 306.1489; found: 306.1493.



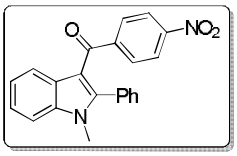
2u, m.p. = 118-120 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01-8.03 (m, 1 H), 7.51-7.52 (m, 2 H), 7.42-7.44 (m, 1 H), 7.33-7.37 (m, 1 H), 7.28-7.30 (m, 1 H), 7.23-7.26 (m, 6 H), 7.09-7.13 (m, 2 H), 3.67 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 192.8, 146.3, 140.1, 137.2, 130.9, 130.8, 130.7, 129.1, 128.7, 127.9, 127.6, 127.5, 123.2, 122.3, 121.8, 114.7, 109.7, 31.2; IR (neat, cm^{-1}): 2920, 1616, 1468, 1392, 1212, 751, 698, 661; HRMS (APCI) m/z: calcd for $C_{22}H_{18}NO$: M+H = 312.1383; found: 312.1390.



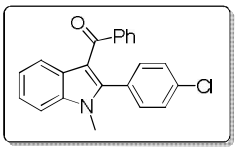
2v, m.p. = 110-112 °C

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03-8.05 (m, 1 H), 7.42-7.44 (m, 3 H), 7.20-7.38 (m, 7 H), 7.06-7.08 (m, 2 H), 3.68 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 191.4, 146.5, 138.5, 137.2, 136.9, 130.9, 130.6, 130.5, 130.1, 129.2, 128.8, 128.3, 128.1, 127.7, 127.5, 123.4, 122.5, 121.8, 114.5, 109.8, 31.3; IR (neat, cm^{-1}): 2920, 1617, 1578, 1467, 1387, 1087, 750, 701; HRMS (APCI) m/z: calcd for $C_{22}H_{17}NOCl$: M+H = 346.0993; found: 346.1000.



2w, m.p. = 206-208 °C

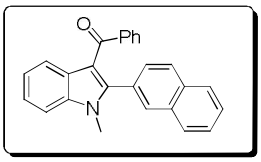
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.23-8.25 (m, 1 H), 7.87-7.89 (m, 2 H), 7.50-7.52 (m, 2 H), 7.34-7.47 (m, 4 H), 7.14-7.34 (m, 4 H), 3.68 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 190.6, 148.3, 147.4, 145.9, 137.3, 130.9, 130.2, 129.5, 129.4, 129.2, 129.1, 128.2, 127.3, 126.9, 123.9, 123.6, 123.2, 122.6, 122.0, 114.3, 109.9, 31.4; IR (neat, cm^{-1}): 2920, 1618, 1598, 1521, 1466, 1387, 1345, 845, 750, 704; HRMS (APCI) m/z: calcd for $C_{22}H_{17}N_2O_3$: M+H = 357.1234; found: 357.1238.



2x, m.p. = 154-156 °C

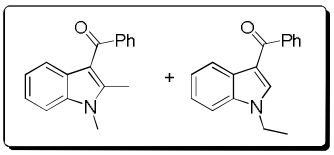
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.92 (m, 1 H), 7.51-7.54 (m, 2 H), 7.41-7.43 (m, 1 H), 7.21-7.37 (m, 4 H), 7.15-7.19 (m, 5 H), 3.66 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 192.6, 144.8, 140.0, 137.2, 135.0, 132.1, 131.1, 129.2, 128.3, 127.7, 127.4, 123.4, 122.3, 121.9, 115.0, 109.8,

31.2; IR (neat, cm^{-1}): 2919, 1608, 1465, 1387, 1090, 1017, 827, 750, 695; HRMS (APCI) m/z: calcd for $C_{22}H_{17}NOCl$: M+H = 346.0993; found: 346.0997.



2y

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99-8.01 (m, 1 H), 7.70-7.79 (m, 4 H), 7.45-7.54 (m, 5 H), 7.37-7.39 (m, 2 H), 7.25-7.35 (m, 1 H), 6.97-7.07 (m, 3 H), 3.72 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 192.9, 146.4, 140.2, 137.3, 132.9, 132.5, 131.1, 130.7, 128.9, 128.1, 127.7, 127.6, 127.4, 126.8, 126.4, 123.3, 122.3, 121.9, 115.1, 109.8, 31.3; IR (neat, cm^{-1}): 2926, 2842, 1726, 1615, 1464, 1395, 1228, 750, 698; HRMS (ESI) m/z: calcd for $C_{26}H_{19}NNaO$: M+Na = 384.1359; found: 384.1363.

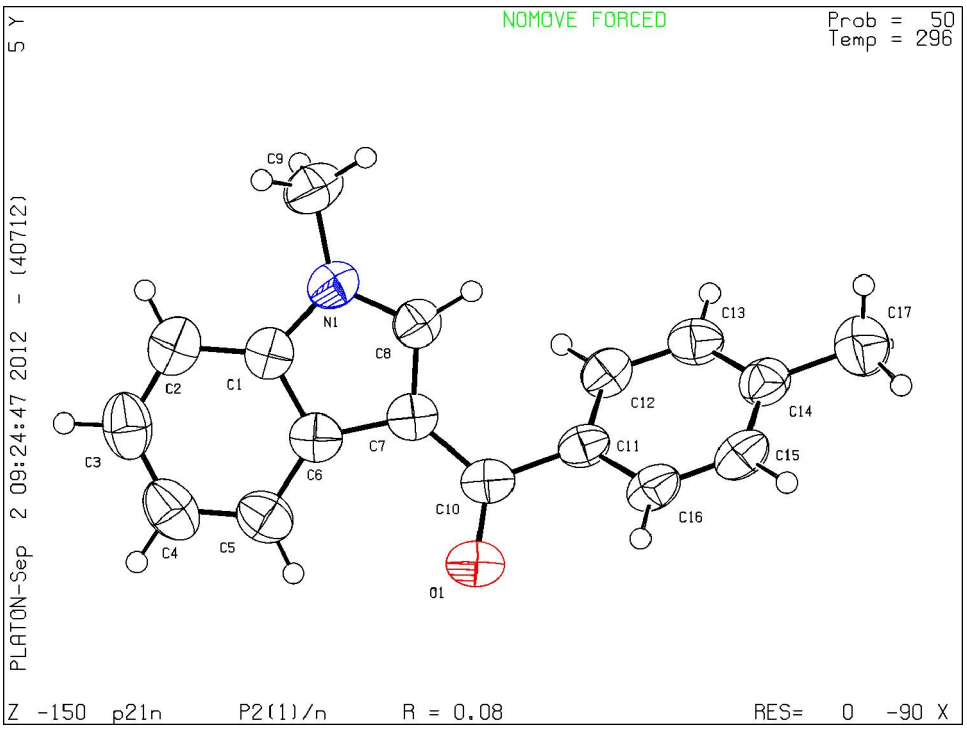


2z (1: 1.3)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.41-8.43 (m, 1 H), 7.80-7.82 (m, 2 H), 7.74-7.76 (m, 1.6 H), 7.59 (s, 1 H), 7.40-7.74 (m, 8 H), 7.29-7.39 (m, 4.3 H), 7.18-7.22 (m, 1.3 H), 7.04-7.08 (m, 1 H), 4.18-4.24 (m, 2.2 H), 3.74 (s, 2.4 H), 2.58 (s, 2.4 H), 1.51 (t, J = 7.2 Hz, 3.3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 192.8, 190.8, 144.7, 141.5, 141.0, 136.6, 136.1, 131.4, 131.0, 129.0, 128.7, 128.2, 127.4, 127.1, 123.5, 122.9, 122.6, 122.0, 121.4, 120.9, 115.7, 109.7, 109.1, 41.7, 29.7, 15.2, 12.5; IR (neat, cm^{-1}): 2924, 1673, 1619, 1519, 1462, 1383, 1223, 1056, 1021, 869, 747; HRMS (ESI) m/z: calcd for $C_{17}H_{15}NNaO$: M+Na = 272.1046; found: 272.1047.

Crystal Data and Structure Refinement for 2b.

Datablock: p21n



Bond precision: C-C = 0.0068 A Wavelength=0.71073

Cell: a=11.180(9) b=7.292(6) c=16.780(13)
alpha=90 beta=100.945(7) gamma=90

Temperature: 296 K

	Calculated	Reported
Volume	1343.1(19)	1343.1(19)
Space group	P 21/n	P2(1)/n
Hall group	-P 2yn	?
Moiety formula	C17 H15 N O	?
Sum formula	C17 H15 N O	C17 H15 N O
Mr	249.30	249.30
Dx,g cm-3	1.233	1.233
Z	4	4
Mu (mm-1)	0.077	0.077
F000	528.0	528.0
F000'	528.21	
h,k,lmax	13,8,20	13,8,20
Nref	2500	2470
Tmin,Tmax	0.981,0.988	0.981,0.988
Tmin'	0.981	

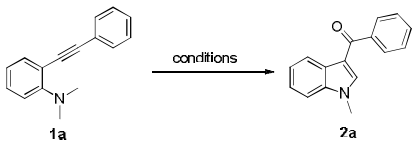
Correction method= MULTI-SCAN

Data completeness= 0.988 Theta(max)= 25.500

R(reflections)= 0.0774(1613) wR2(reflections)= 0.2290(2470)

S = 1.187 Npar= 175

Table S1. Screening optimal conditions.[a]



1a 2a

Entry	[Pd] (mol %)	[Cu] (mol %)	[O] (equiv)	Additive (equiv)	solvent	Yield (%)[b]
1	$PdCl_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	toluene	30
2	$PdCl_2(CH_3CN)_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	toluene	34
3	PdI_2 (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	toluene	34
4	$PdBr_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	toluene	51
5	$Pd(OAc)_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	toluene	20
6	$Pd(OCOCF_3)_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	toluene	15
7	$PdBr_2$ (10)	CuBr (20)	TBHP (3eq)	LiCl (2 eq)	toluene	35
8	$PdBr_2$ (10)	CuCl (20)	TBHP (3eq)	LiCl (2 eq)	toluene	30
9	$PdBr_2$ (10)	$CuCl_2$ (20)	TBHP (3eq)	LiCl (2 eq)	toluene	33

10	$PdBr_2$ (10)	$CuBr_2$ (20)	TBHP (3eq)	LiCl (2 eq)	toluene	25
11	$PdBr_2$ (10)	CuI (20)	TBHP (3eq)	LiBr (2 eq)	toluene	36
12	$PdBr_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq) + NaI (20%)	toluene	31
13	$PdBr_2$ (10)	CuI (20)	TBHP (3eq)	NaCl (2 eq)	toluene	15
14	$PdBr_2$ (10)	CuI (20)	TBHP (3eq)	LiI (2 eq)	toluene	20
15[c]	$PdBr_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	CH3CN	_
16	$PdBr_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	DMF	_
17[c]	$PdBr_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	DCE	15
18	$PdBr_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	anisole	40
19	$PdBr_2$ (10)	CuI (20)	TBHP (3eq)	LiCl (2 eq)	PhCF3	38
20	***$PdBr_2$ (10)***	***CuI (10)***	***TBHP (3eq)***	***LiCl (2 eq)***	***toluene***	***61***
21	$PdBr_2$ (10)	CuI (10)	tBuOOtBu (3eq)	LiCl (2 eq)	toluene	_
22	$PdBr_2$ (10)	CuI (10)	H_2O_2 (3eq)	LiCl (2 eq)	toluene	_
23	$PdBr_2$ (10)	CuI (10)	(PhCO)2O2 (3 eq)	LiCl (2 eq)	toluene	30
24	$PdBr_2$ (10)	CuI (10)	O_2 (1 atm)	LiCl (2 eq)	toluene	_
25	$PdBr_2$ (10)	CuI (10)	TBHP (4eq)	LiCl (2 eq)	toluene	48
26	$PdBr_2$ (10)	_	TBHP (3eq)	LiCl (2 eq)	toluene	<5
27	_	CuI (10)	TBHP (3eq)	LiCl (2 eq)	toluene	<5

[a] Reaction conditions: **1a**, [Pd], [Cu], oxidant, additive, solvent (2 mL), and 100 °C for 8 h. [b] Yield of isolated product. [c] under 80 °C.

Analytical data of ESI-MS:

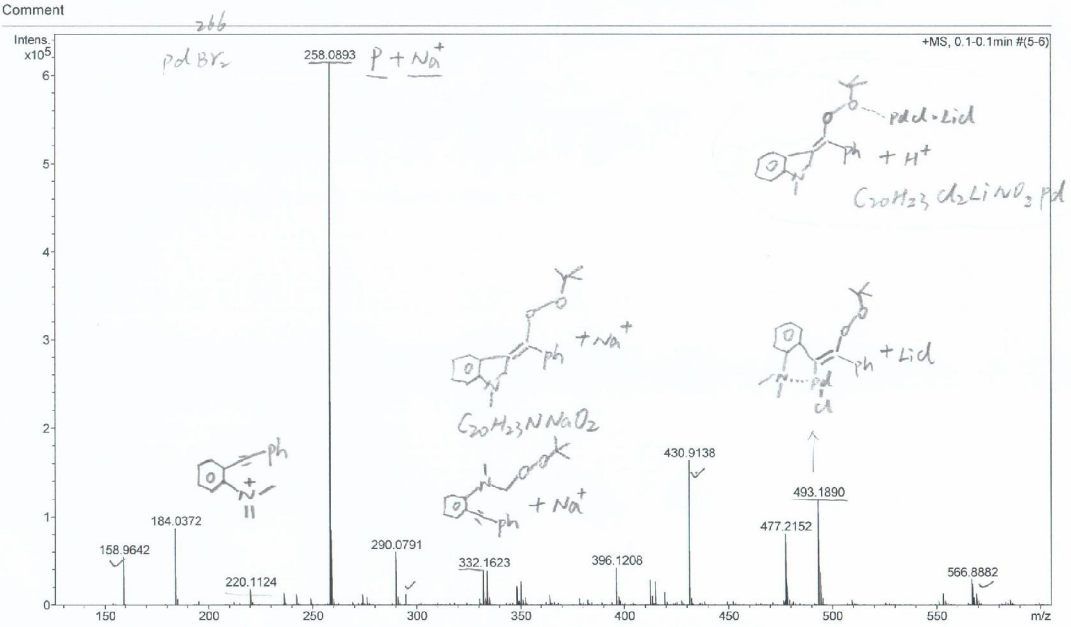
To a solution of *N*,*N*-dimethyl-2-(phenylethynyl)aniline (**1a**) (44.2 mg, 0.200 mmol) in toluene (2 mL) was added $PdBr_2$ (5.3 mg, 10 mol%), CuI (3.8 mg, 10 mol%), LiCl (17 mg, 2.0 equiv.) and 120 uL TBHP (5 M in decane). The reaction mixture was then stirred under air atmosphere at 100 °C for 2 h. The reaction was cooled to room temperature and diluted with CH_3CN (1/100) prior to the injection into the mass spectrometer. The positive-ion mode of ESI-MS spectrum showed the signals corresponding to the iminium intermediate **B (**m/z 220.1124**)** and peroxide **A (M+ Na,** m/z =332.1623**).**

NaTFA

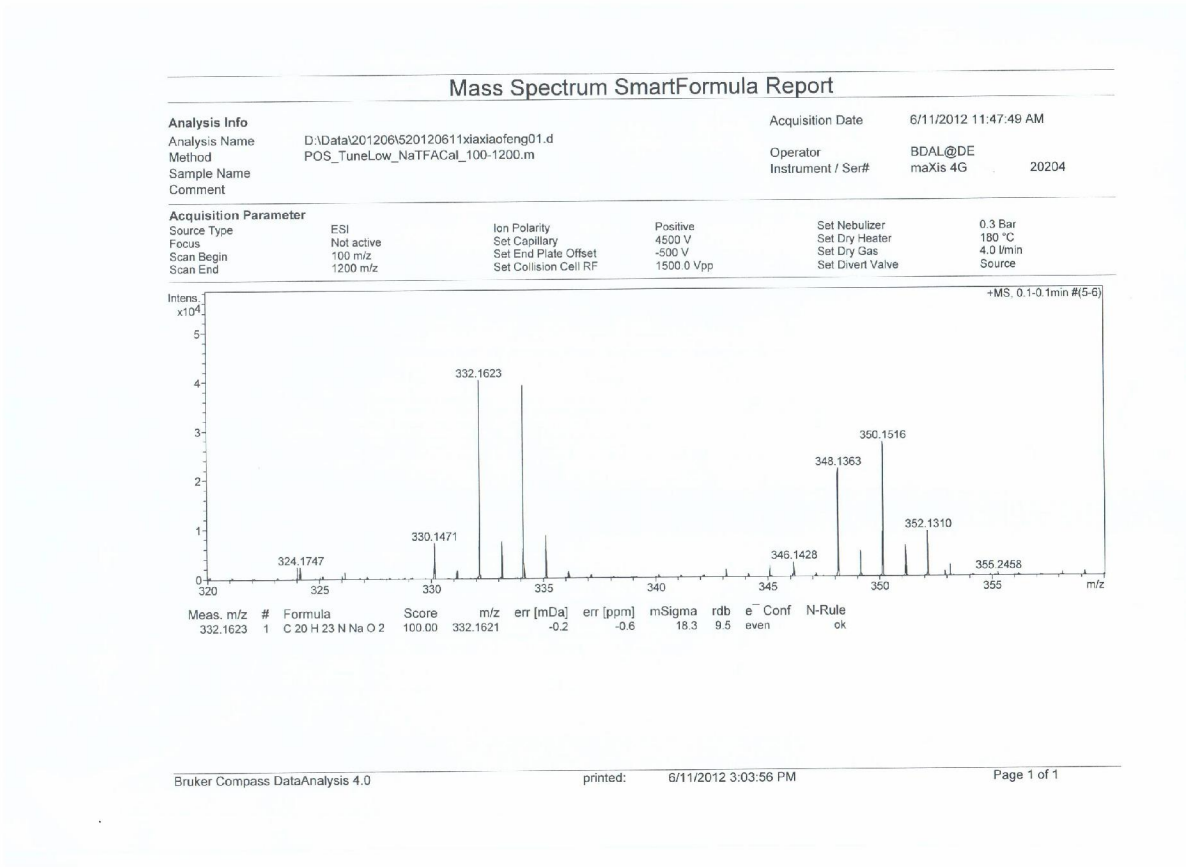
Generic Display Report

Analysis Info

Analysis Name	D:\Data\201206\520120611xiaxiaofeng01.d	Acquisition Date	6/11/2012 11:47:49 AM
Method	POS_TuneLow_NaTFACal_100-1200.m	Operator	BDAL@DE
Sample Name		Instrument	maXis 4G
Comment			

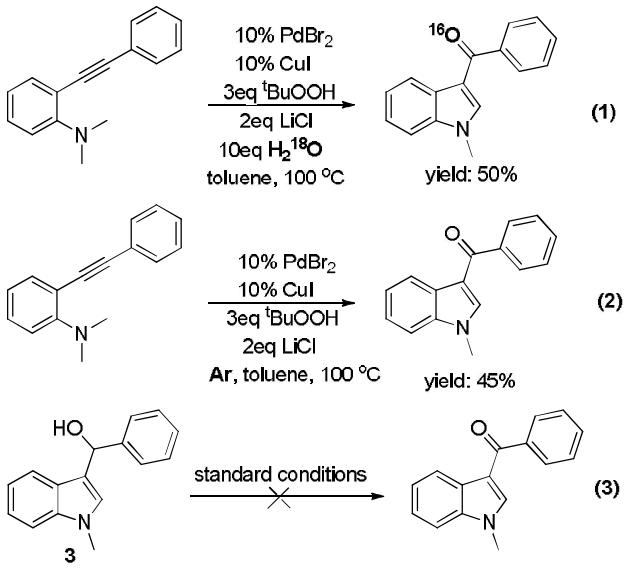


Bruker Compass DataAnalysis 4.0 printed: 6/11/2012 11:48:54 AM Page 1 of 1

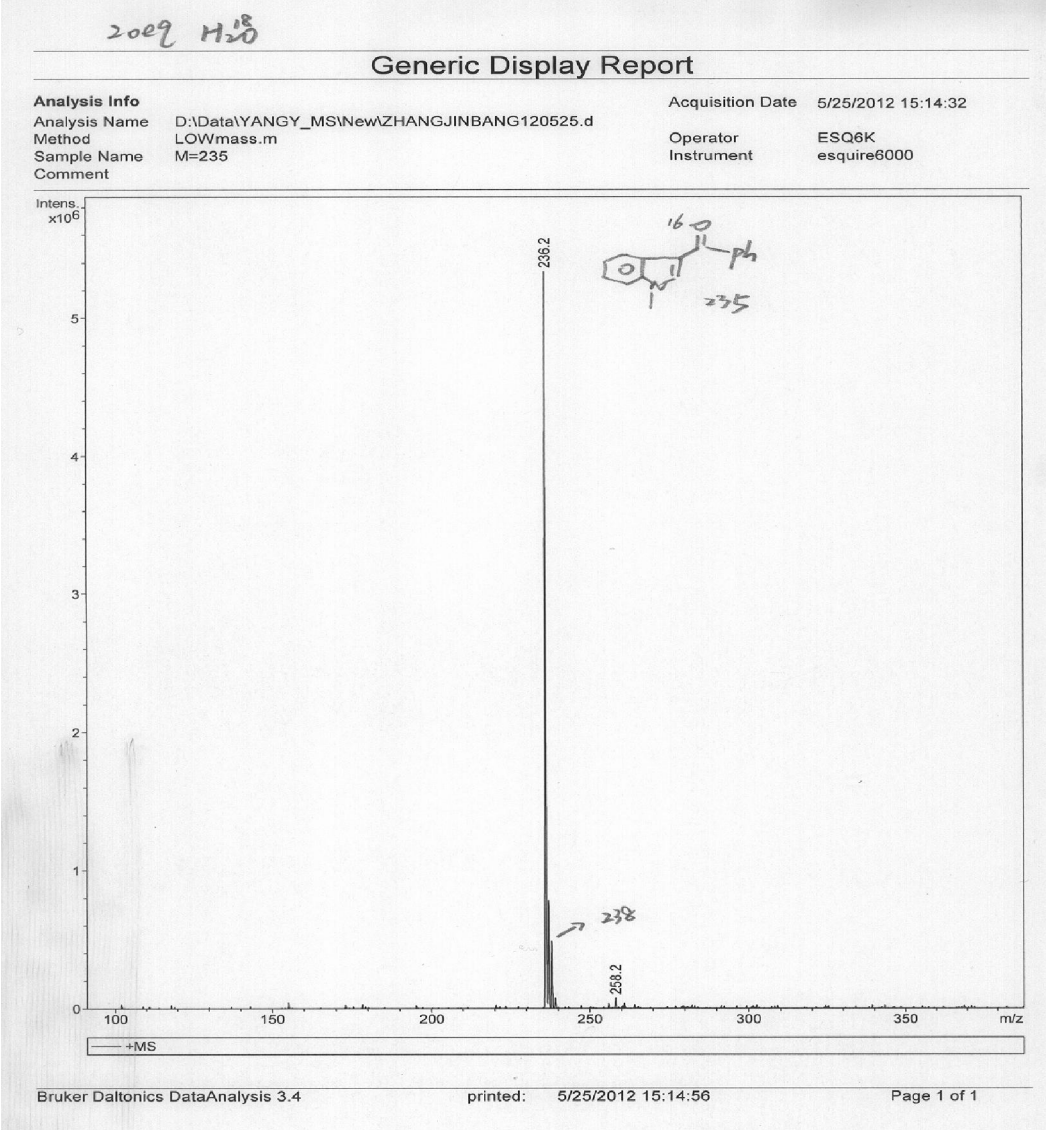


For the Labeling experiment and Control experiments:

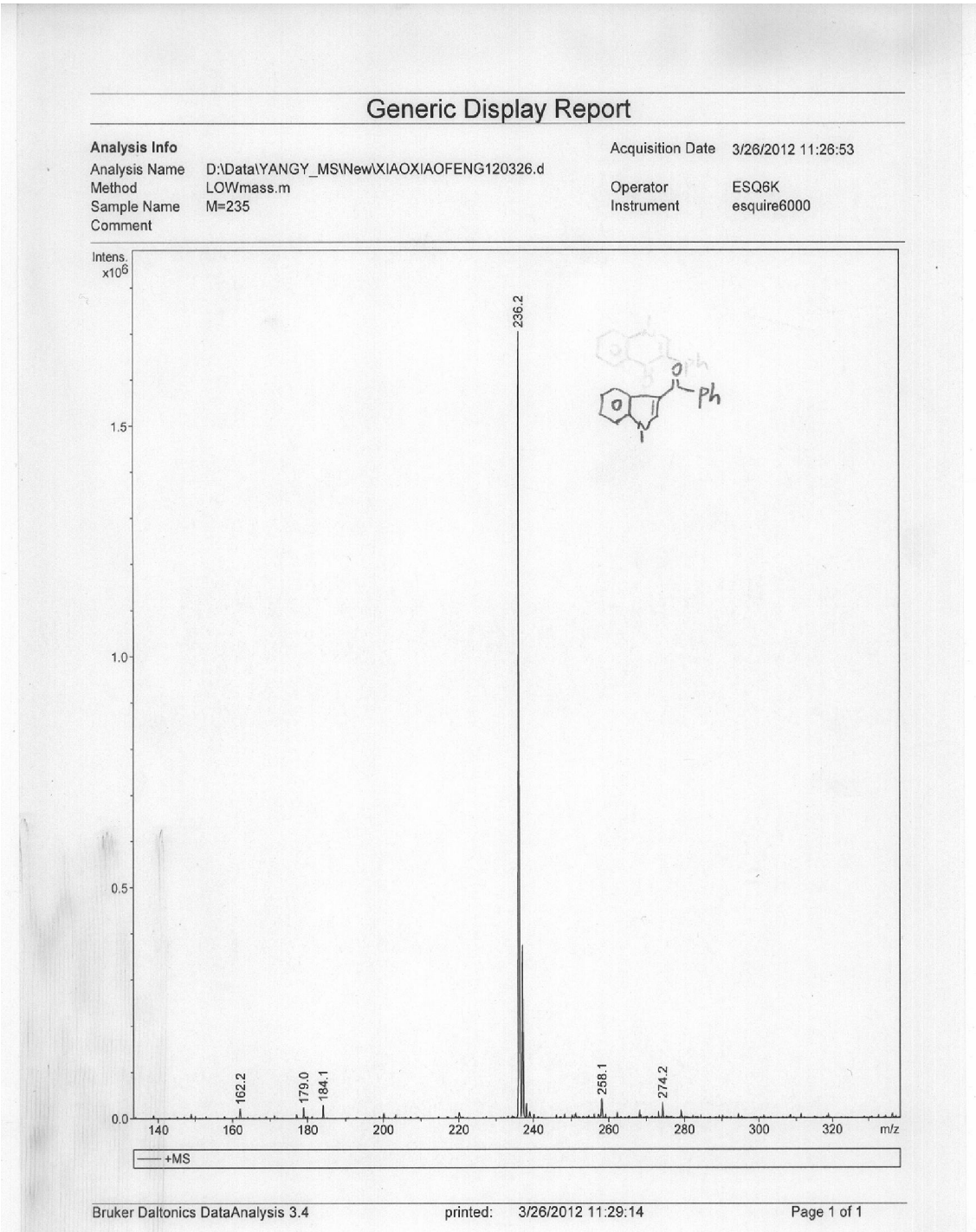
To a solution of *N*,*N*-dimethyl-2-(phenylethynyl)aniline (**1a**) (44.2 mg, 0.200 mmol) in toluene (2 mL) was added $PdBr_2$ (5.3 mg, 10 mol%), CuI (3.8 mg, 10 mol%), LiCl (17 mg, 2.0 equiv.), $H_2{}^{18}O$ (10 equiv., 40 uL) and 120 uL TBHP (5 M in decane). The reaction mixture was then stirred in air at 100 ºC for 8 h. The resulting mixture was quenched with water and extracted twice with EtOAc. The combined organic extracts were washed with brine, dried over $MgSO_4$ and concentrated. Purification of the crude product by flash column chromatography afforded **2a** in 50 % yield.



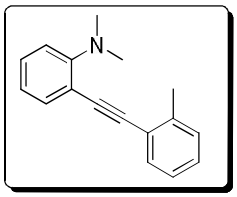
Add $H_2{}^{18}O$ in the reaction system, data of MS:

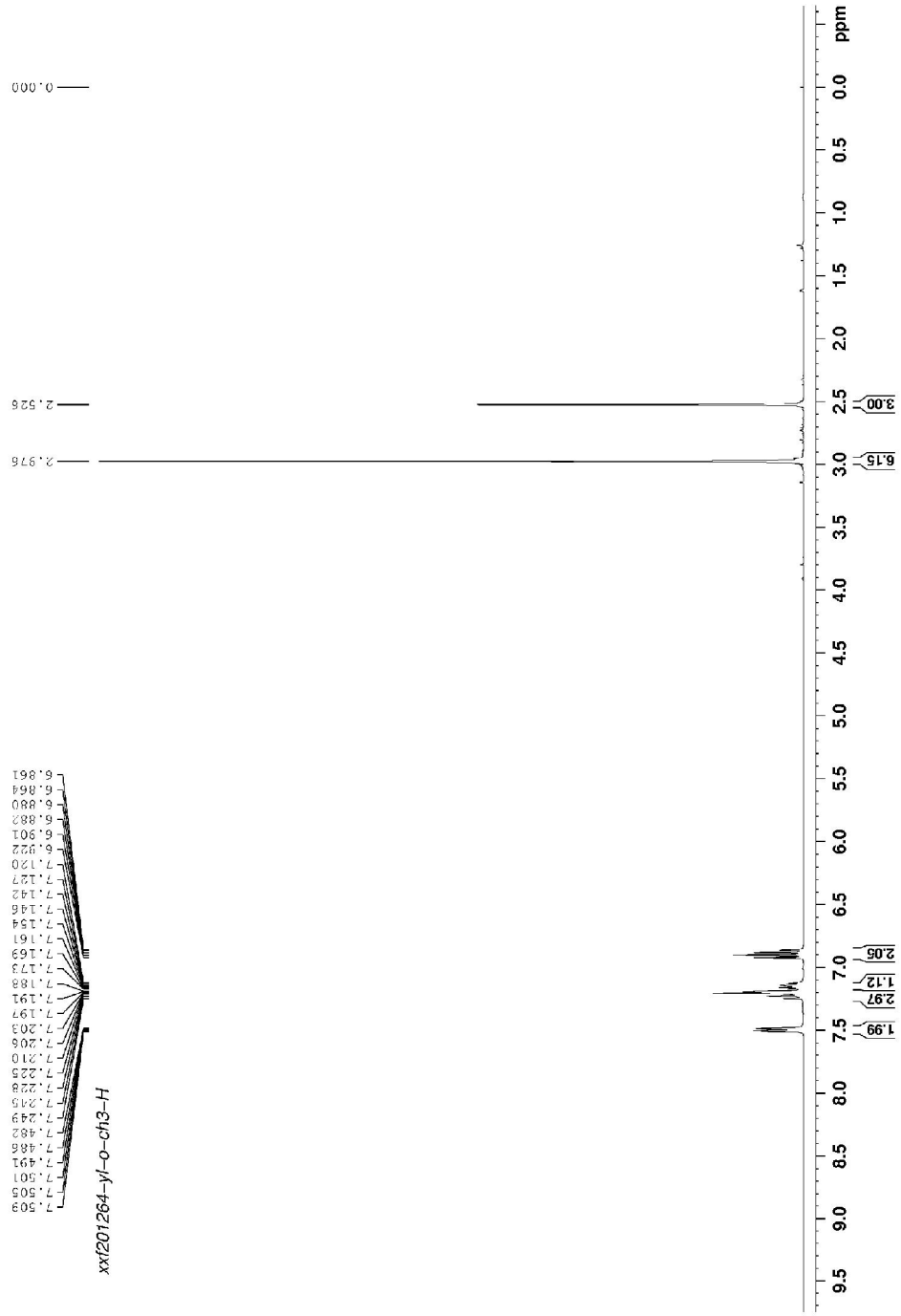


No $H_2{}^{18}O$ in the reaction system, data of MS:



Reference: 1. Xia, X.-F.; Wang, N.; Zhang, L.-L.; Song, X.-R.; Liu, X.-Y.; Liang, Y.-M. *J. Org. Chem.* 2012, **77**, 9163−9170.

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7.501
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7.482
7.249
7.245
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7.210
7.206
7.203
7.197
7.191
7.188
7.173
7.169
7.161
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7.146
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7.120
6.922
6.901
6.882
6.880
6.864
6.861
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3.0
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4.0
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5.0
5.5
6.0
6.5
7.0
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8.0
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9.0
9.5
3.00
6.15
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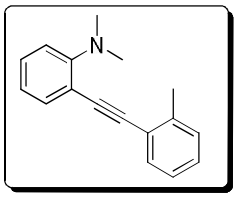
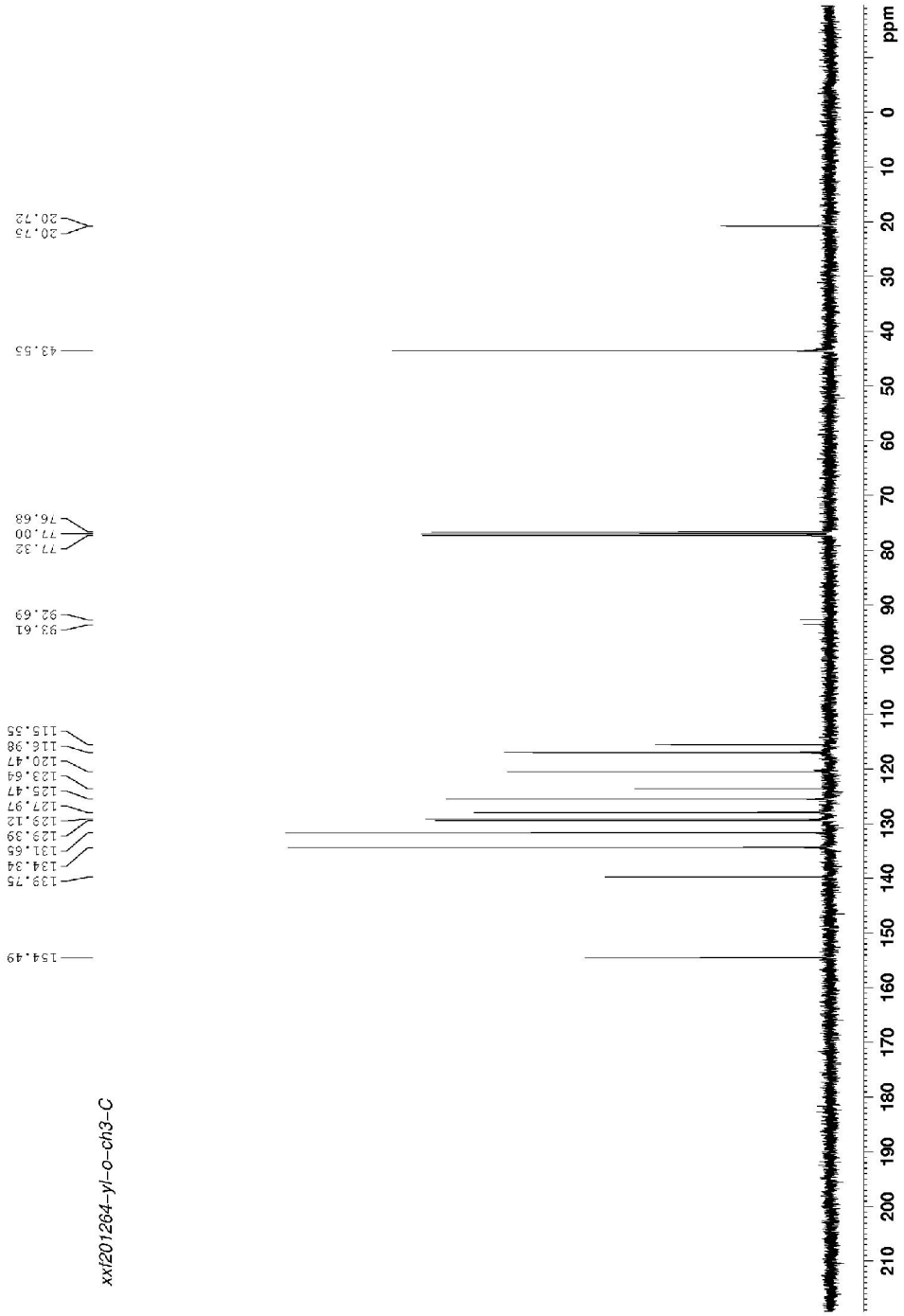
1.12
2.97
1.99

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134.34
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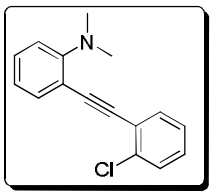
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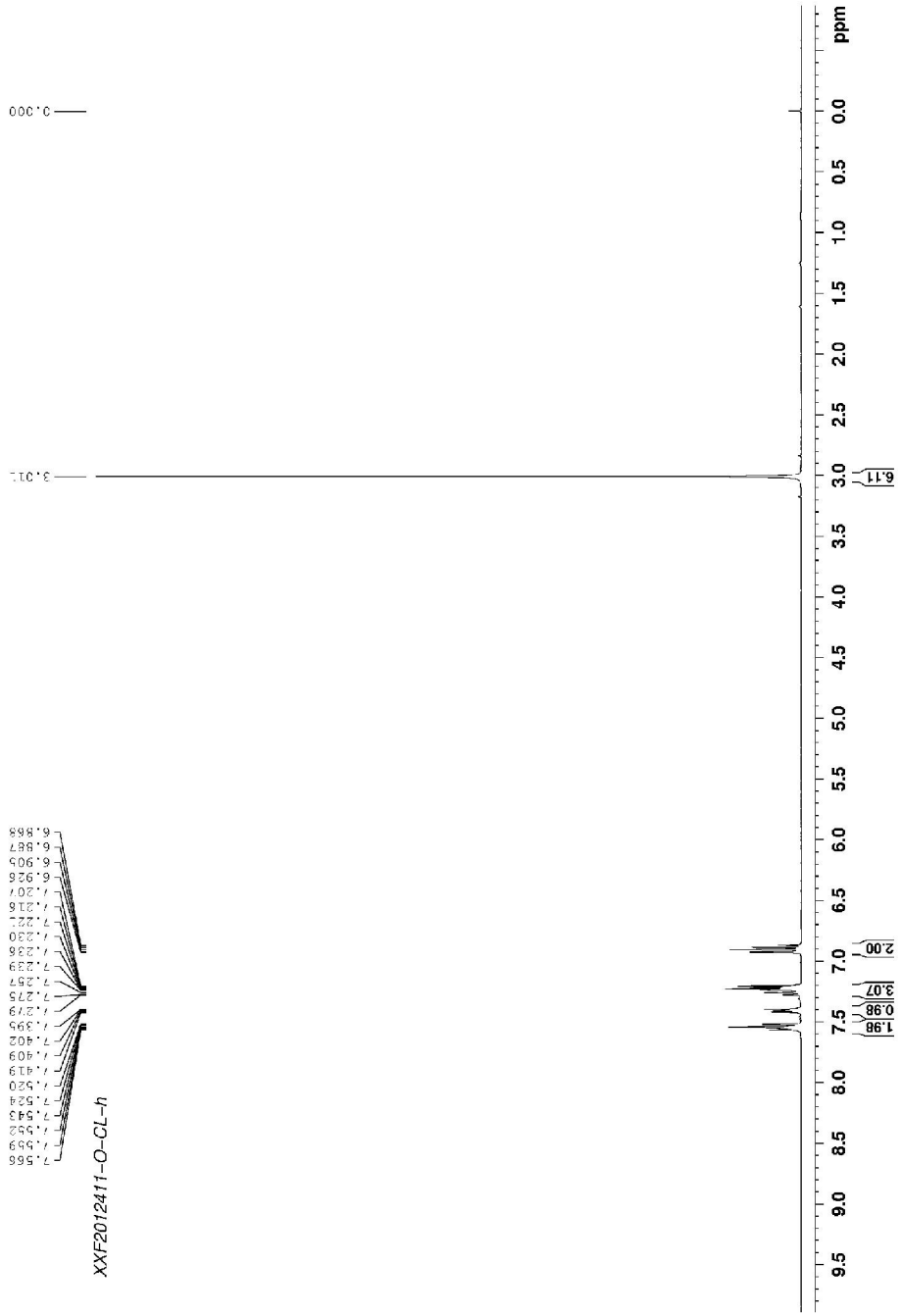
Cl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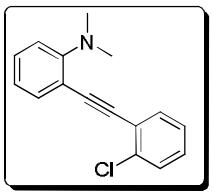
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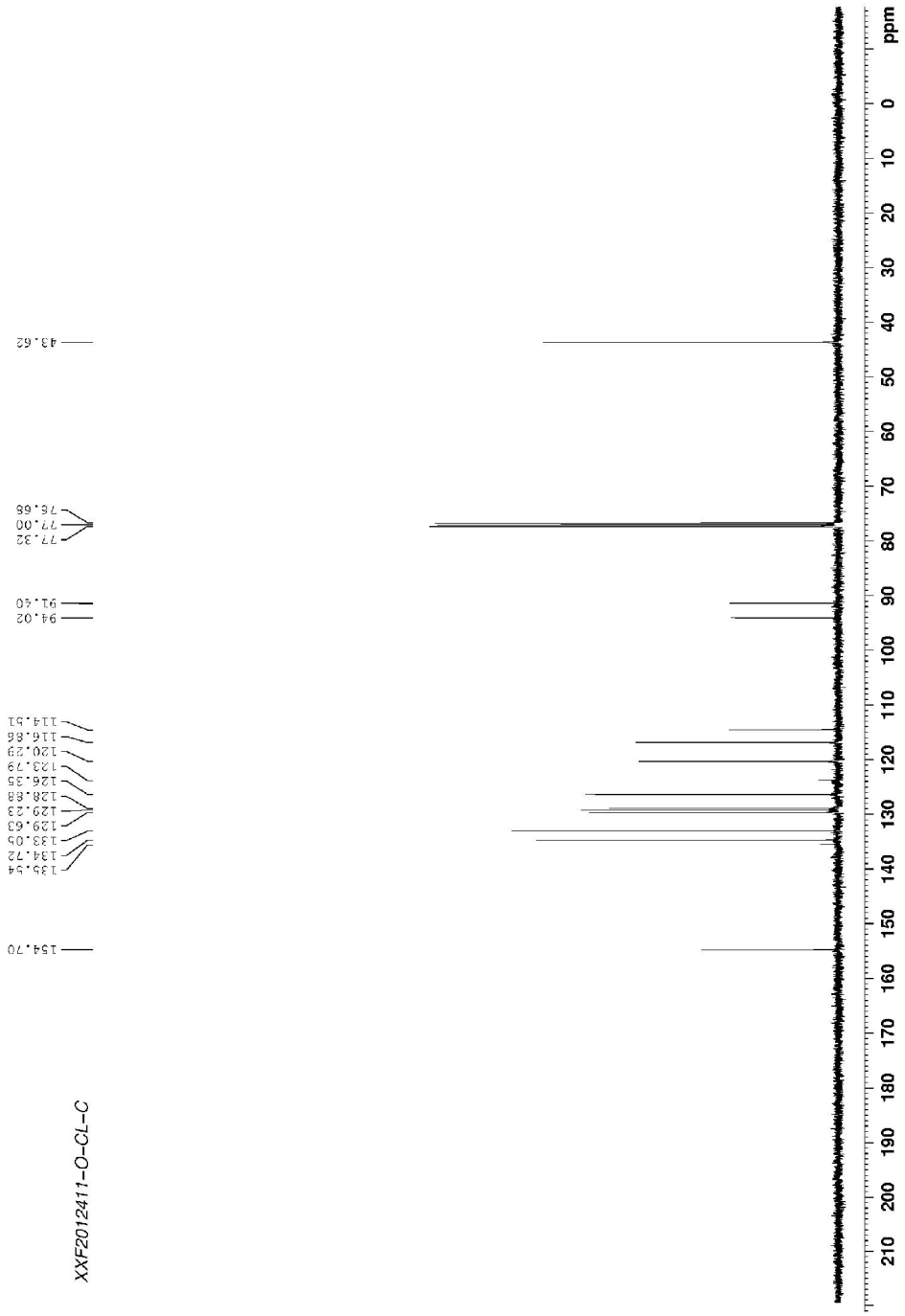
Cl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133.05
134.72
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ppm
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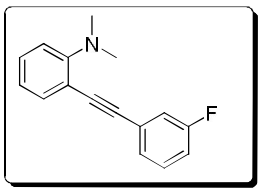
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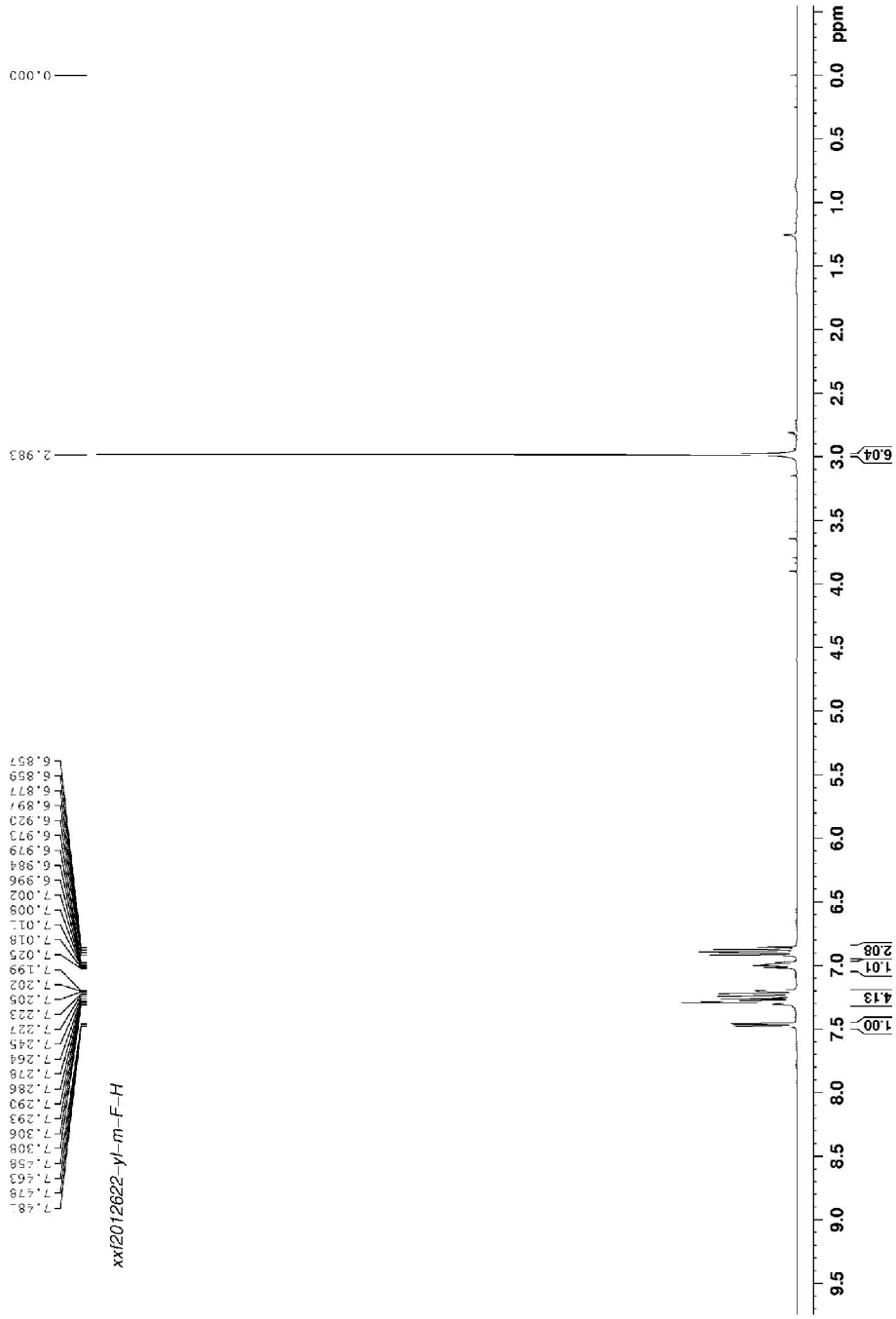
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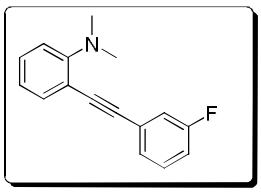
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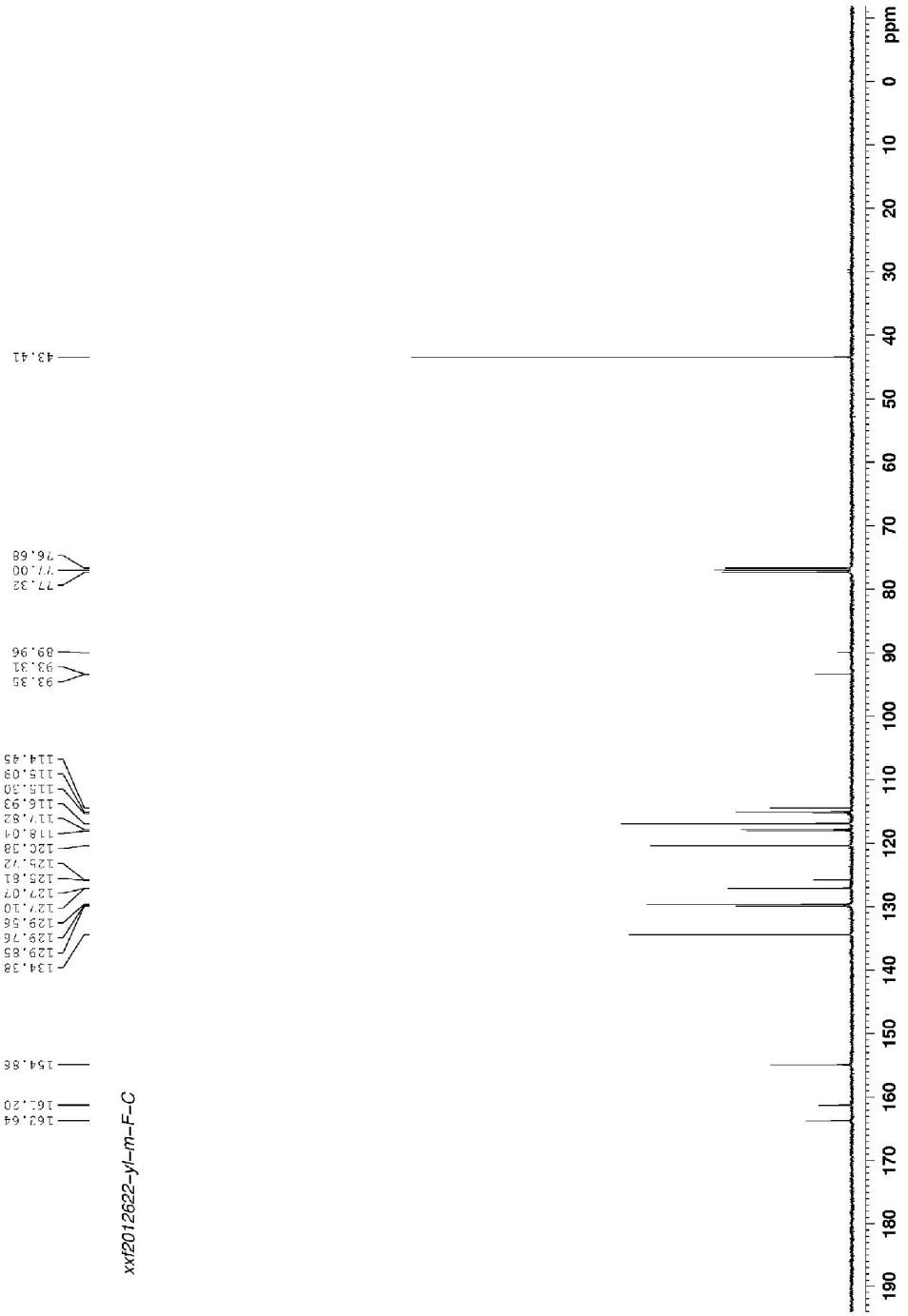
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127.10
129.56
129.76
129.85
134.38
154.86
162.20
162.64
xxtf2012622-yl-m-F-C
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80
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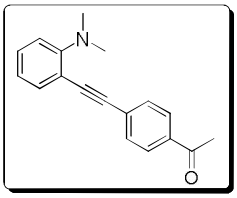

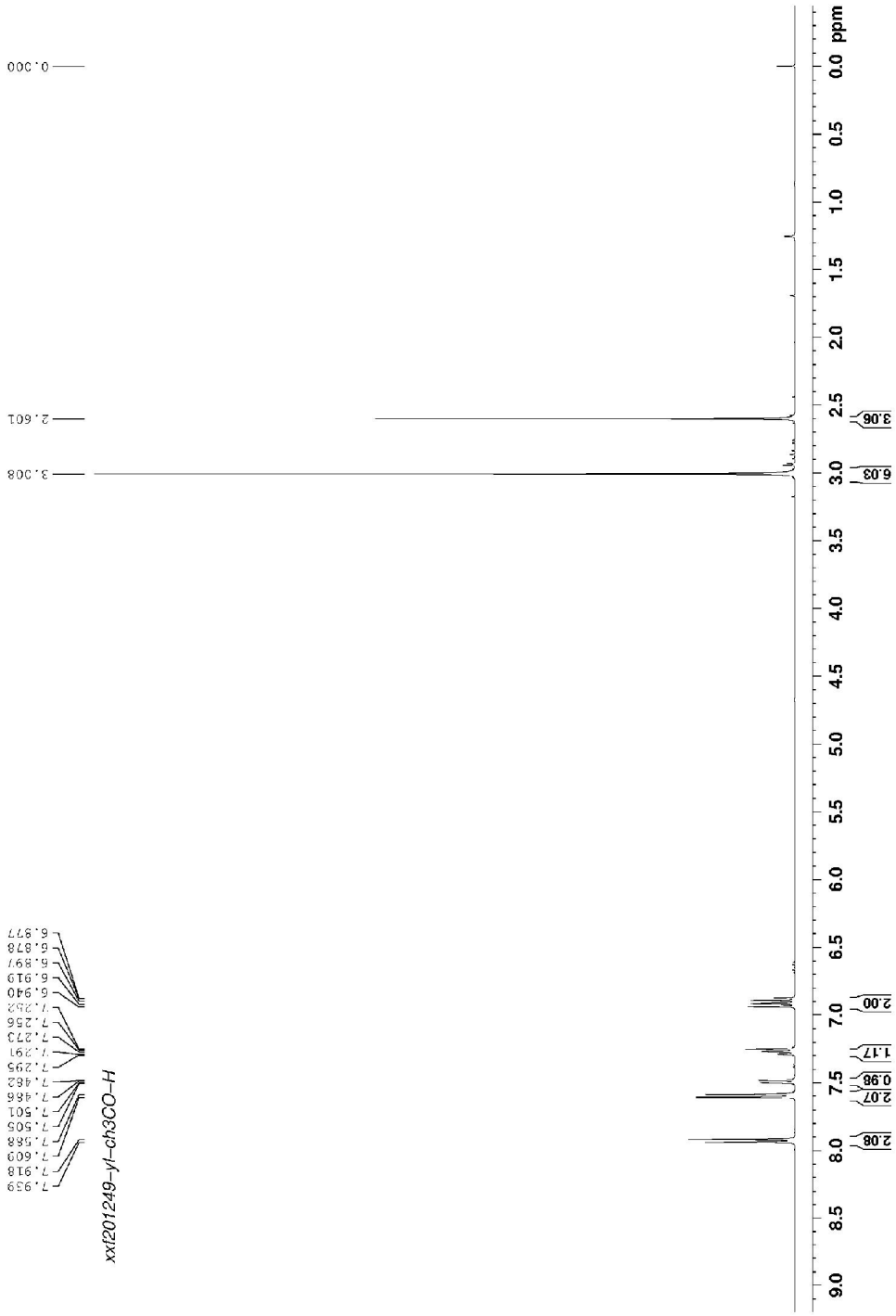
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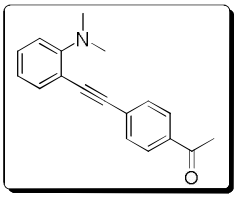

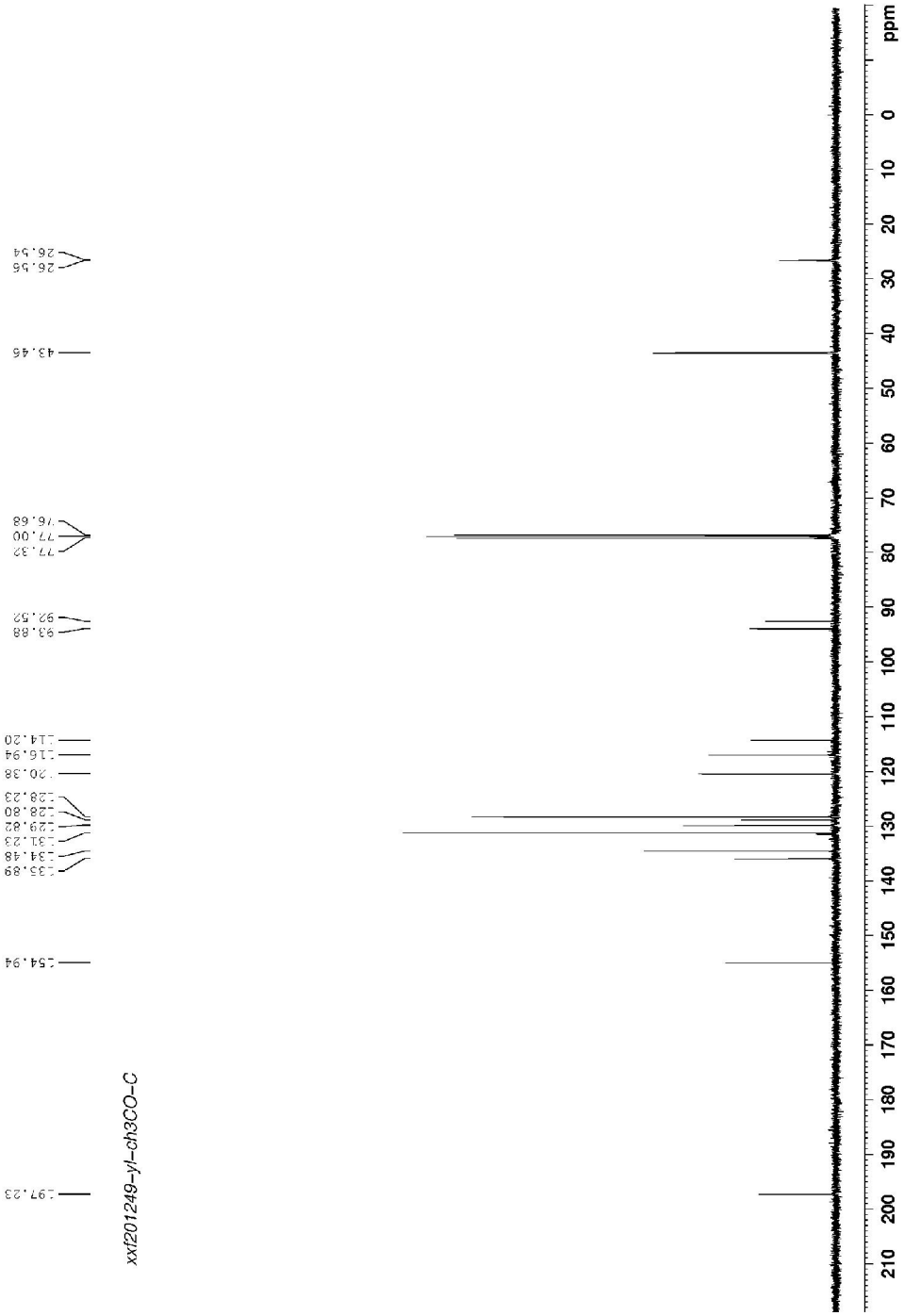
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77.00
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93.88
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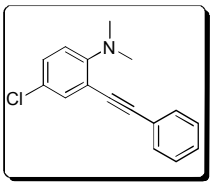
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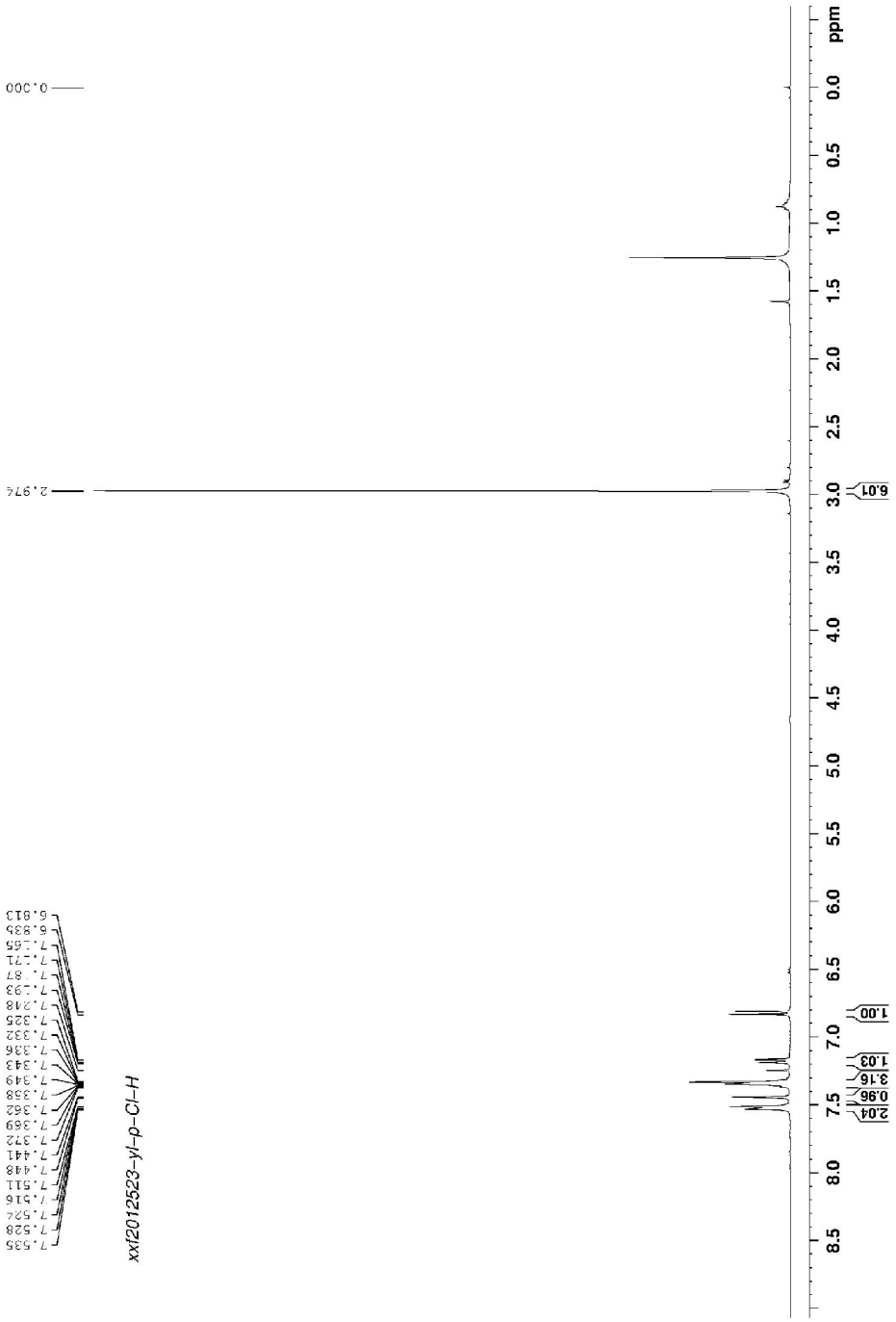
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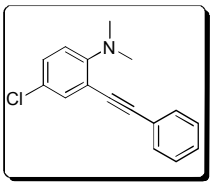
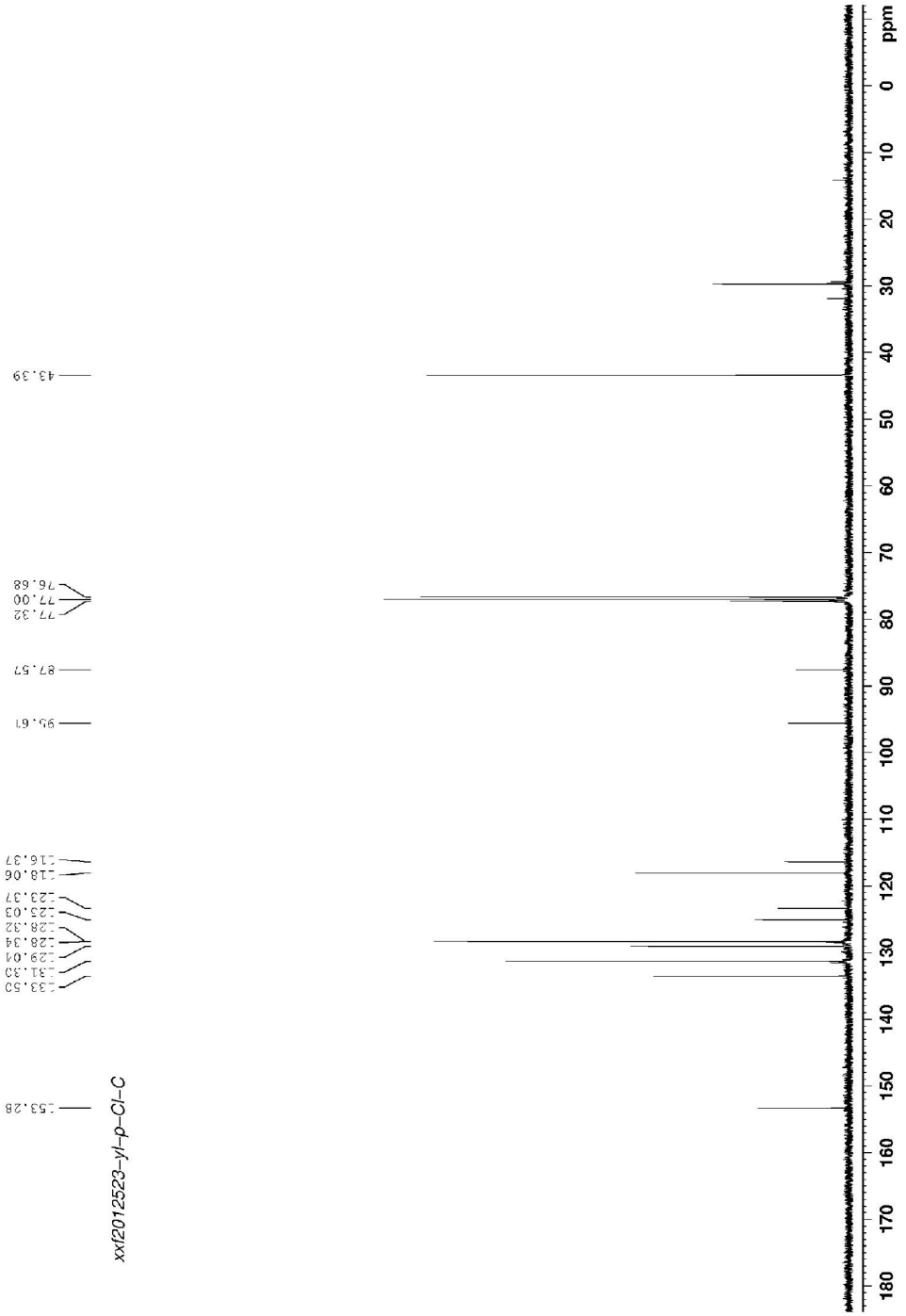
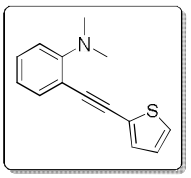
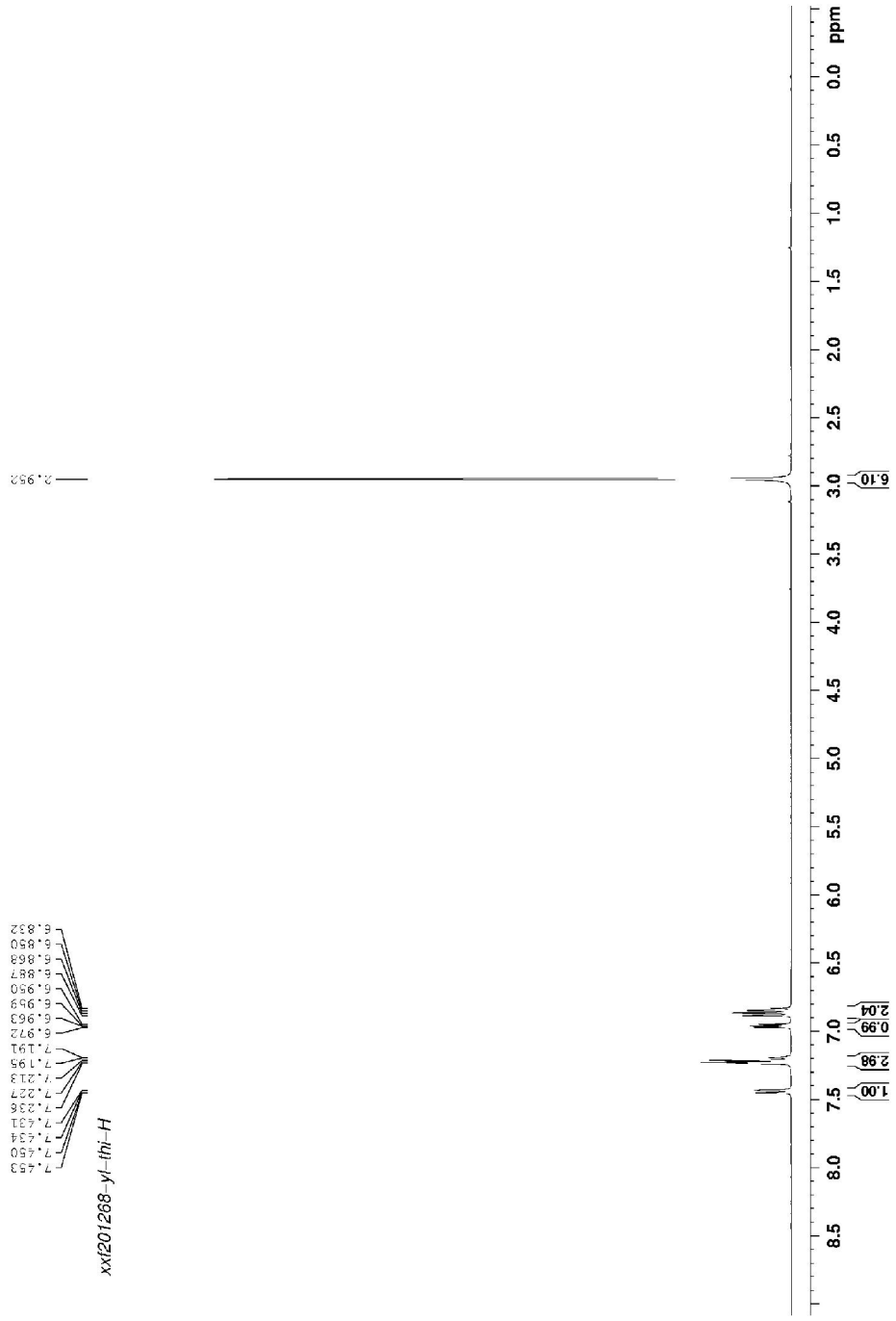
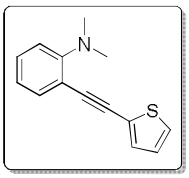
7.565
7.771
7.787
7.793
7.218
7.325
7.332
7.336
7.343
7.349
7.358
7.362
7.369
7.372
7.441
7.448
7.511
7.516
7.524
7.528
7.535
xxl20120523-yl-p-Cl-H
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
6.01
1.00
1.03
3.16
0.96
2.04

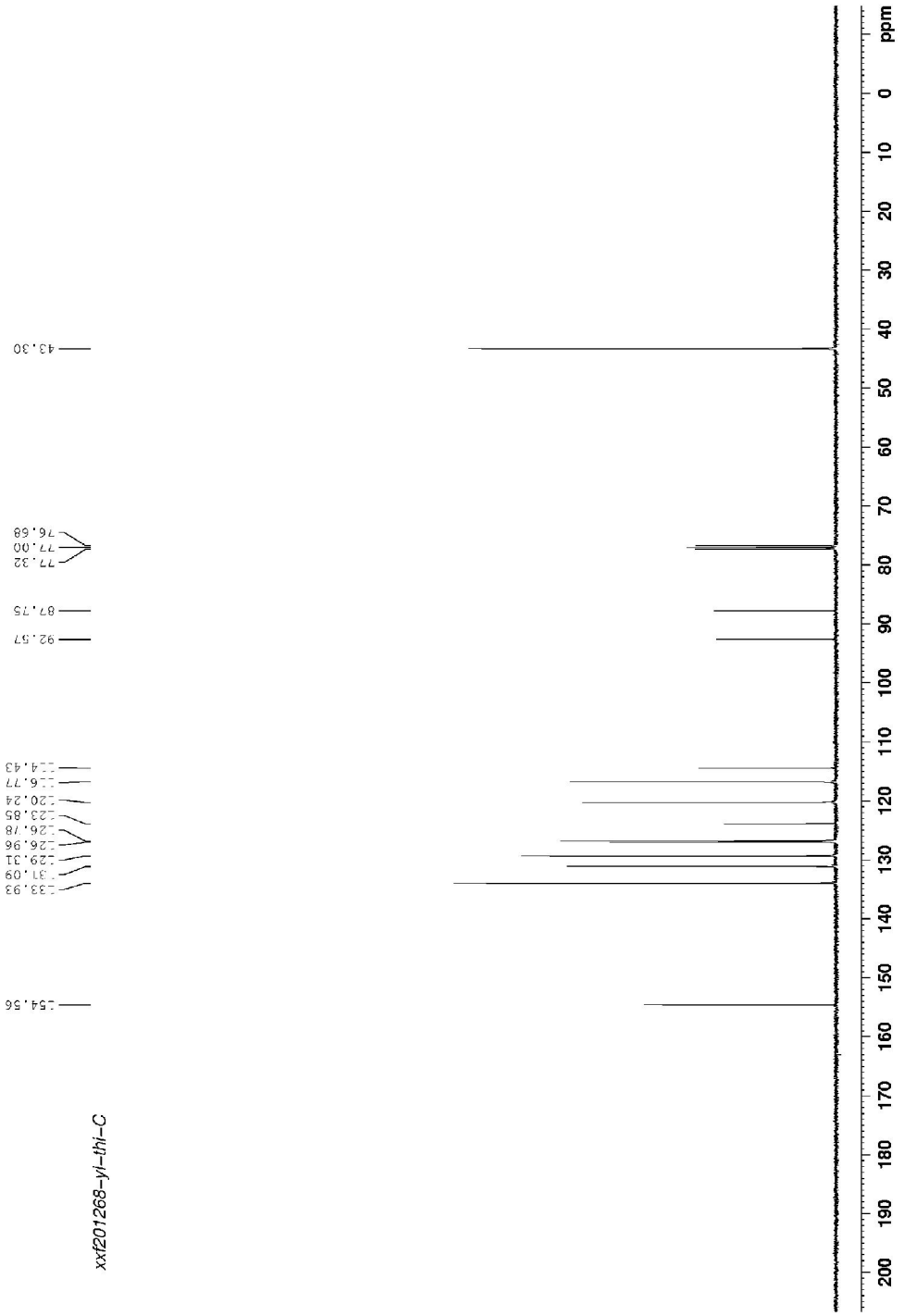

N
Cl

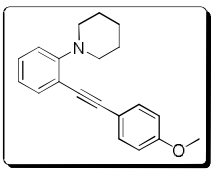

43.39
76.68
77.00
77.32
87.57
95.67
116.37
118.06
123.37
125.03
128.32
128.34
129.04
131.33
133.50
153.28
xxf2012523-yl-p-Cl-C
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

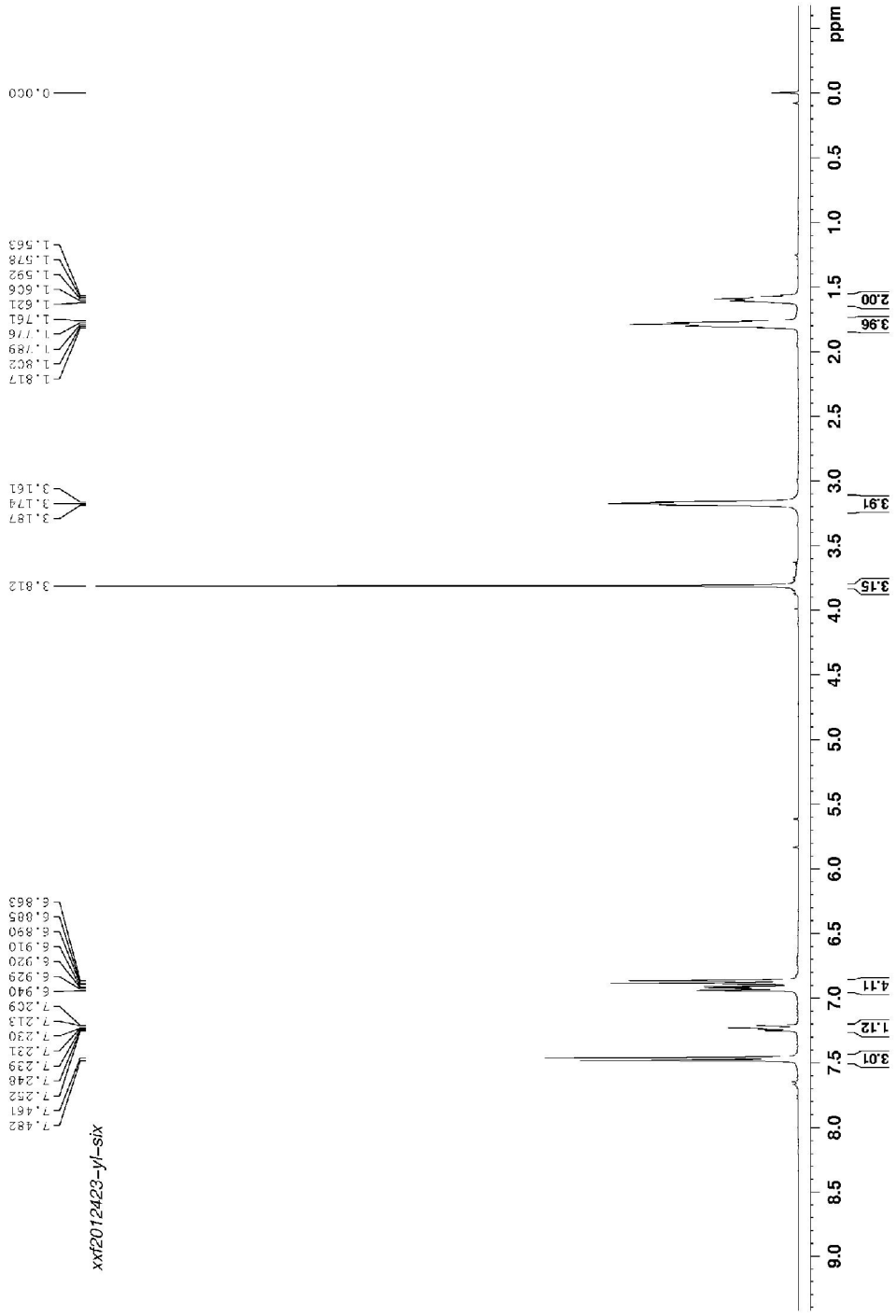

N
S

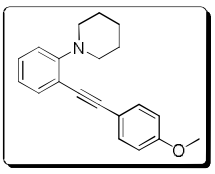

ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
6.10
2.04
0.99
2.98
1.00
2.952
6.832
6.850
6.868
6.887
6.950
6.959
6.963
6.972
7.191
7.195
7.213
7.227
7.236
7.431
7.434
7.450
7.453
xxf201268_yl_thi_H

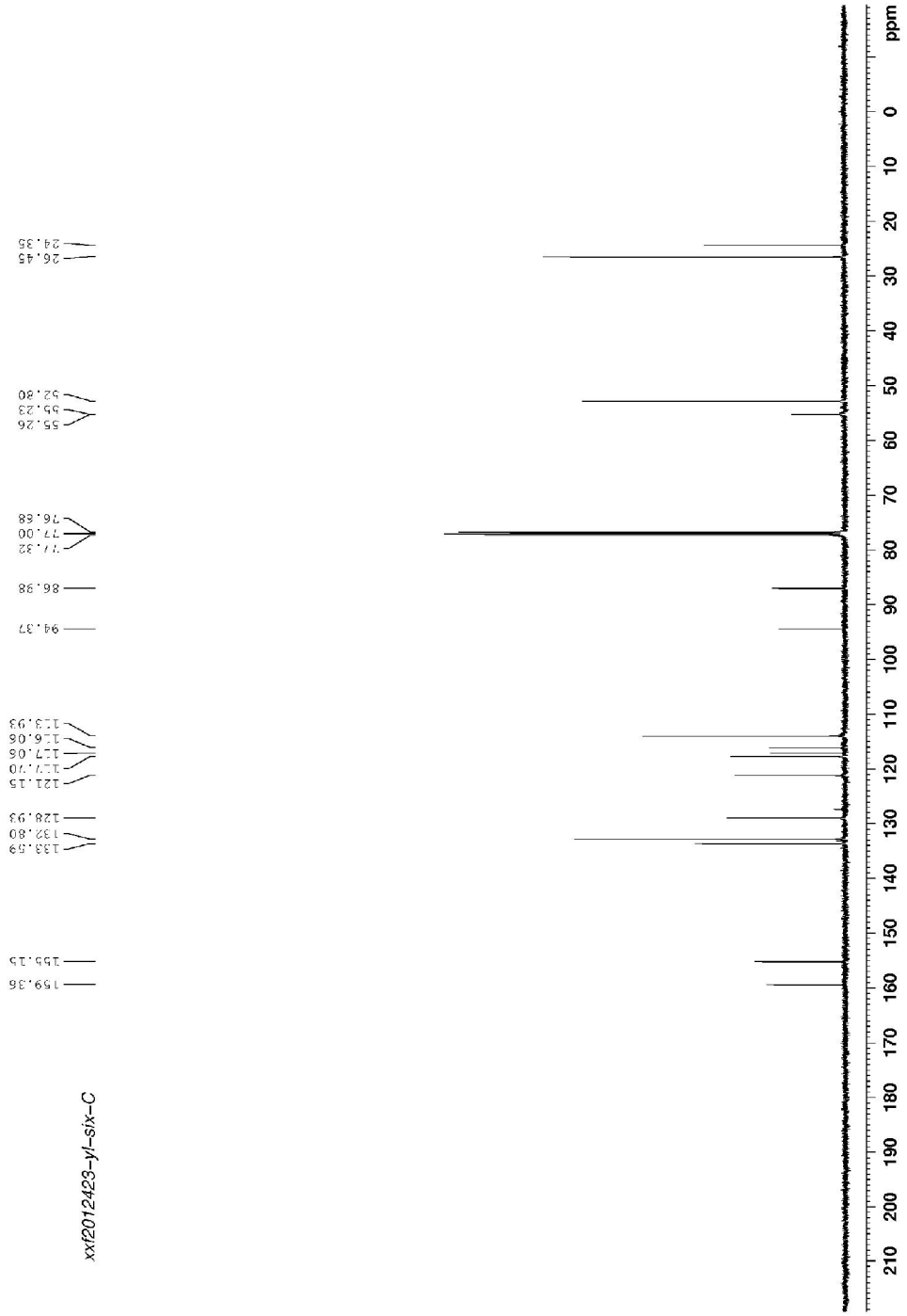

N
S

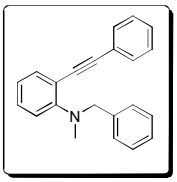

43.30
76.68
77.00
77.32
87.75
92.57
114.43
116.77
120.24
123.85
126.78
126.96
129.31
131.09
133.93
154.56
xxf201268-yl-thi-C
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200

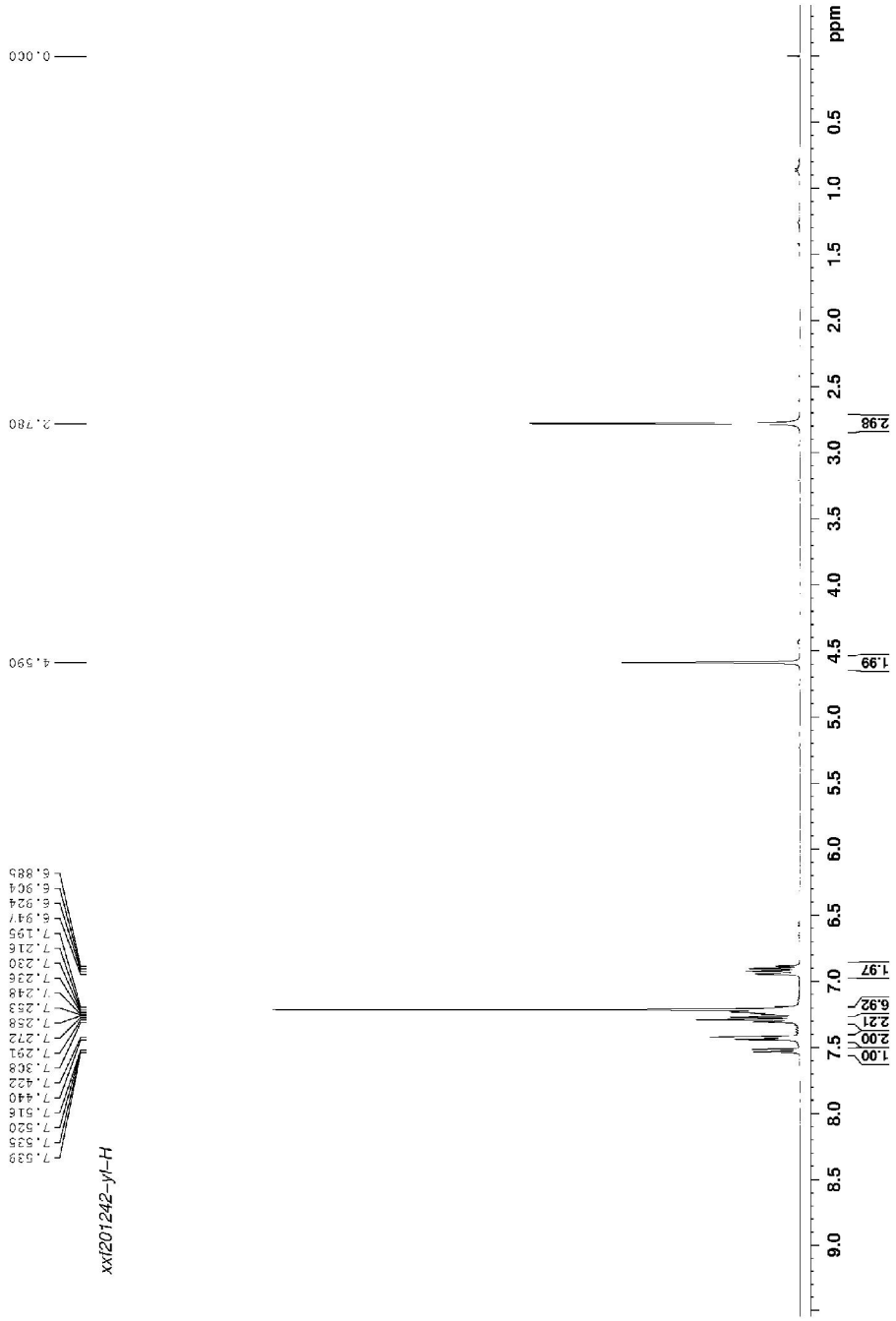


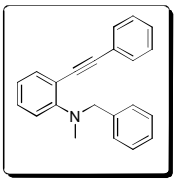

0.000
1.563
1.578
1.592
1.606
1.621
1.761
1.776
1.789
1.802
1.817
3.161
3.174
3.187
3.812
6.863
6.885
6.890
6.910
6.920
6.929
6.940
7.209
7.213
7.230
7.231
7.239
7.248
7.252
7.461
7.482
xxf20120423-yl-six
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
2.00
3.96
3.91
3.15
4.11
1.12
3.01

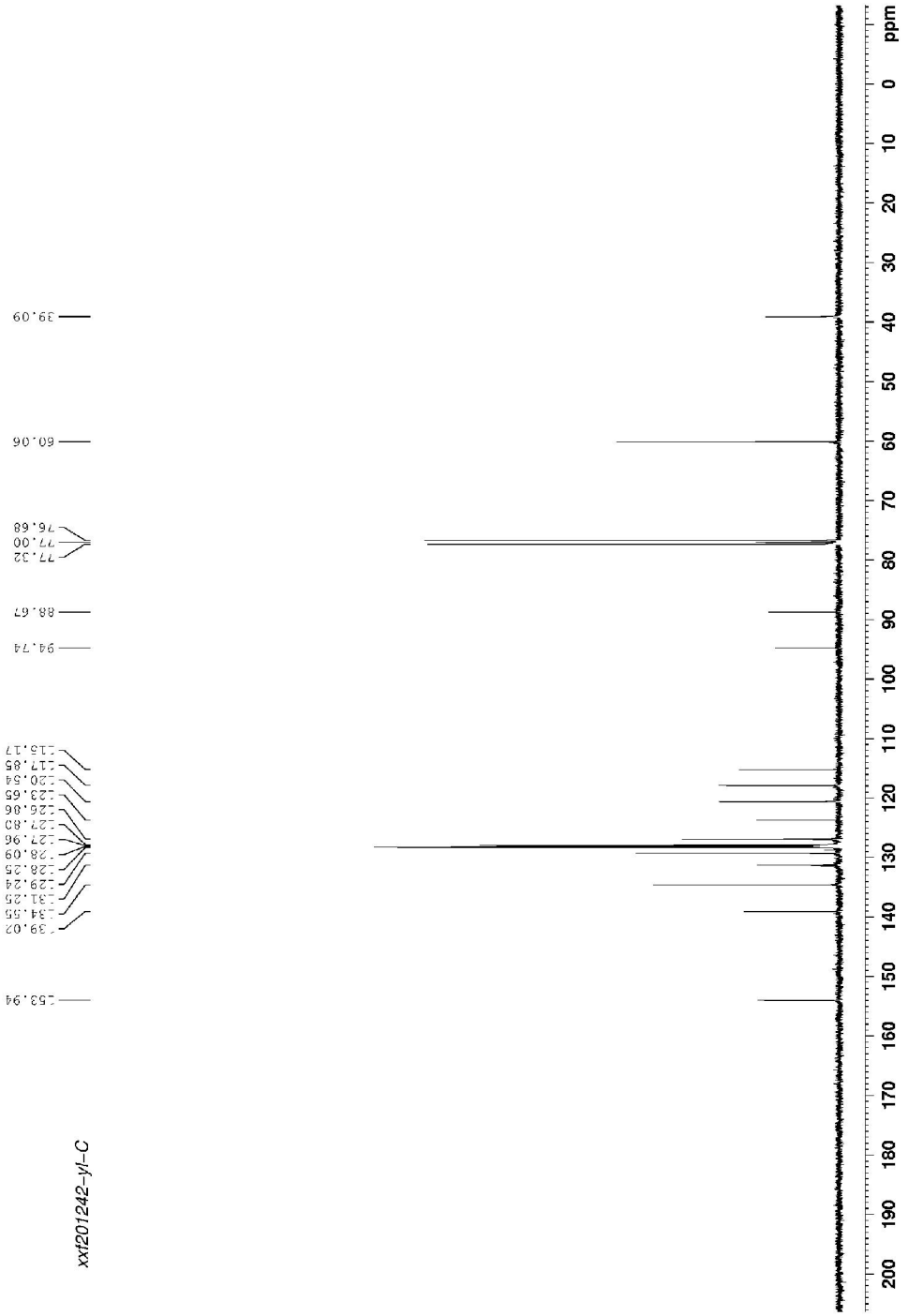


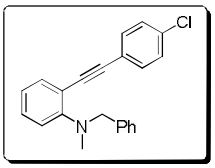

24.35
26.45
55.26
55.23
62.80
76.68
77.00
77.32
86.98
94.37
113.93
116.06
117.06
117.70
121.15
128.93
132.80
133.59
155.15
159.36
xxf2012423-yl-six-C
ppm

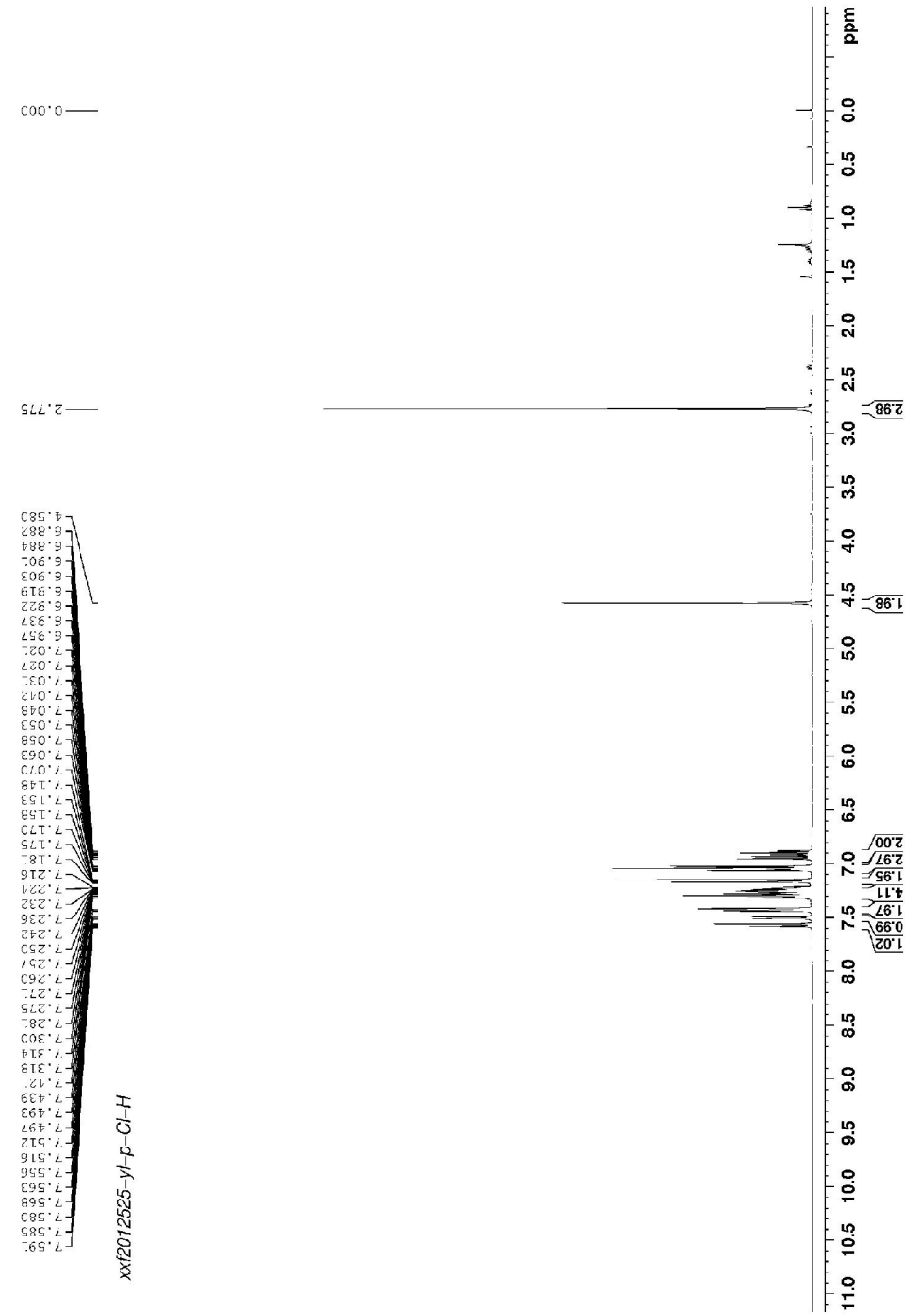



xxf201242-yl-H
ppm
0.000
2.780
4.590
7.539
7.535
7.520
7.516
7.440
7.422
7.368
7.291
7.272
7.258
7.253
7.248
7.236
7.230
7.216
7.195
6.947
6.924
6.904
6.885
2.98
1.99
1.97
6.92
2.21
2.00
1.00




39.09
60.06
76.68
77.00
77.32
88.67
94.74
115.17
117.85
120.54
123.65
126.06
127.80
127.96
128.09
128.25
129.24
131.25
134.55
139.02
153.94
xxf201242-yl-C
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200


Cl
N
Ph


xxf20120525-yl-p-Cl-H
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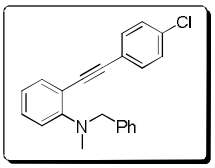
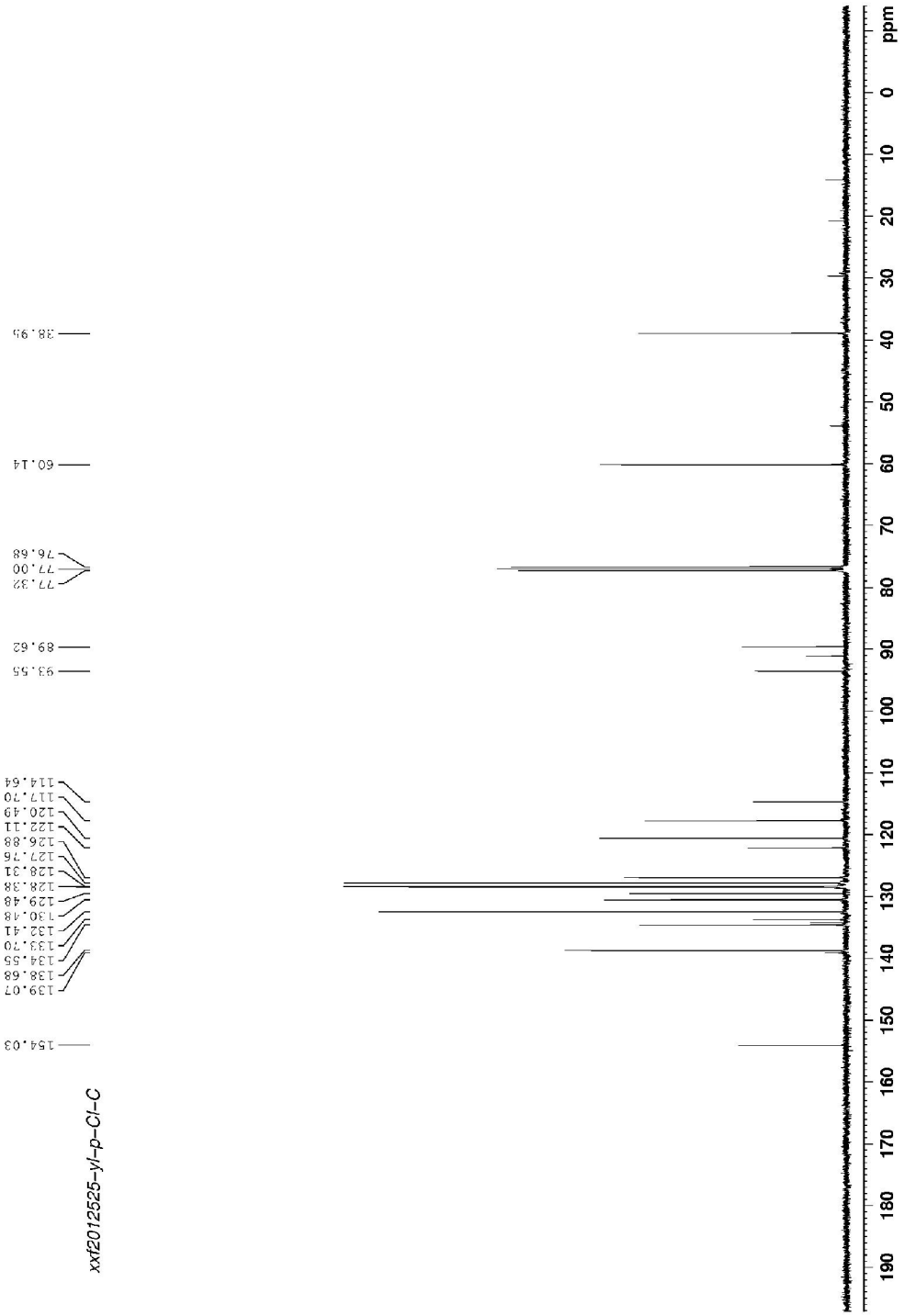
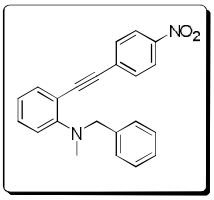
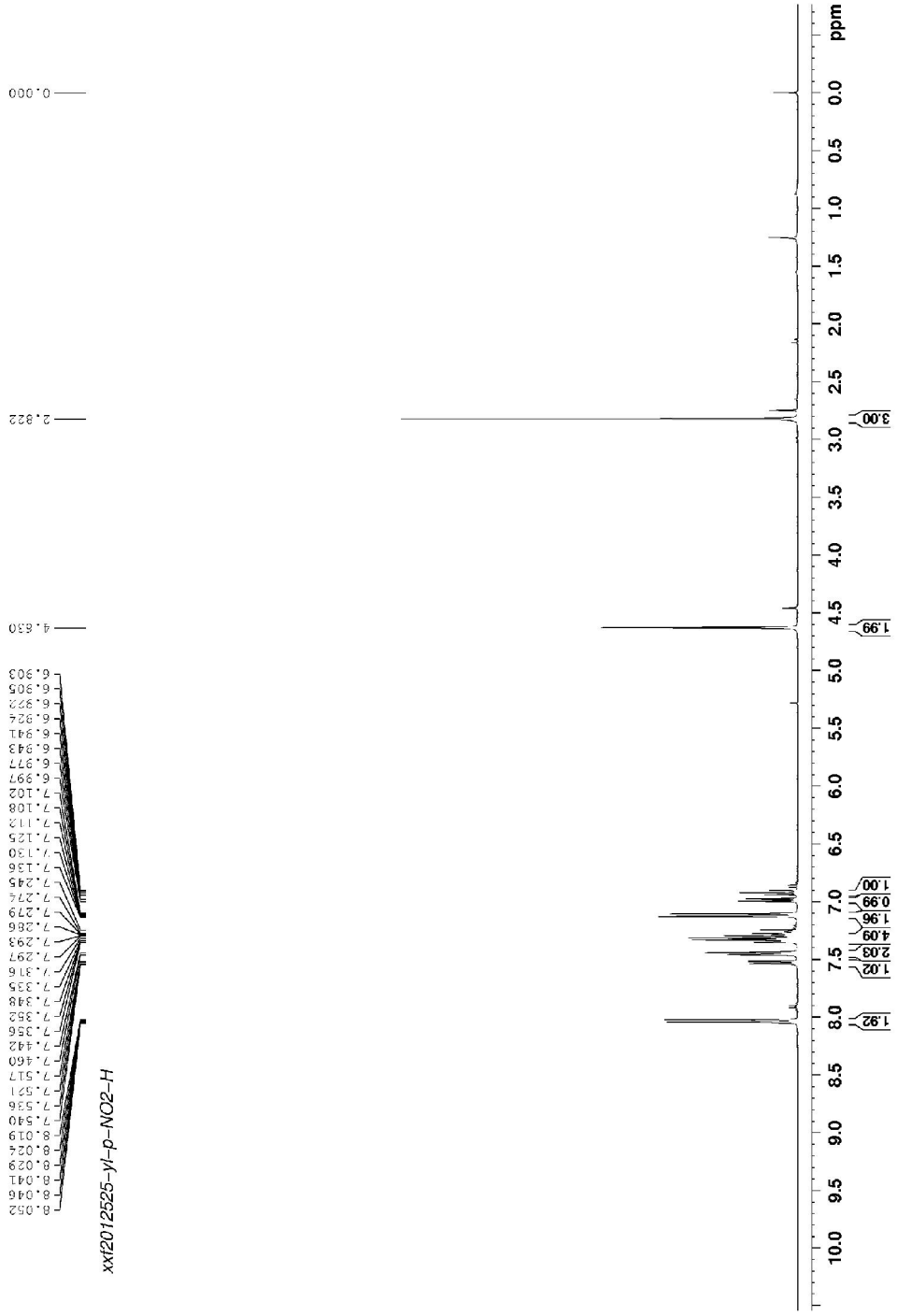
2.98
1.98
2.00
2.97
1.95
4.11
1.97
0.99
1.02


Cl
N
Ph


38.95
60.14
76.68
77.00
77.32
89.62
93.55
114.64
117.70
120.49
122.11
126.88
127.76
128.31
128.38
129.48
130.48
132.41
133.70
134.55
138.68
139.07
154.03
xxf20120525-yl-p-Cl-C
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190


NO2
N


xxf20120525-yl-p-NO2-H
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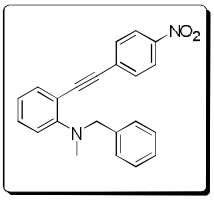
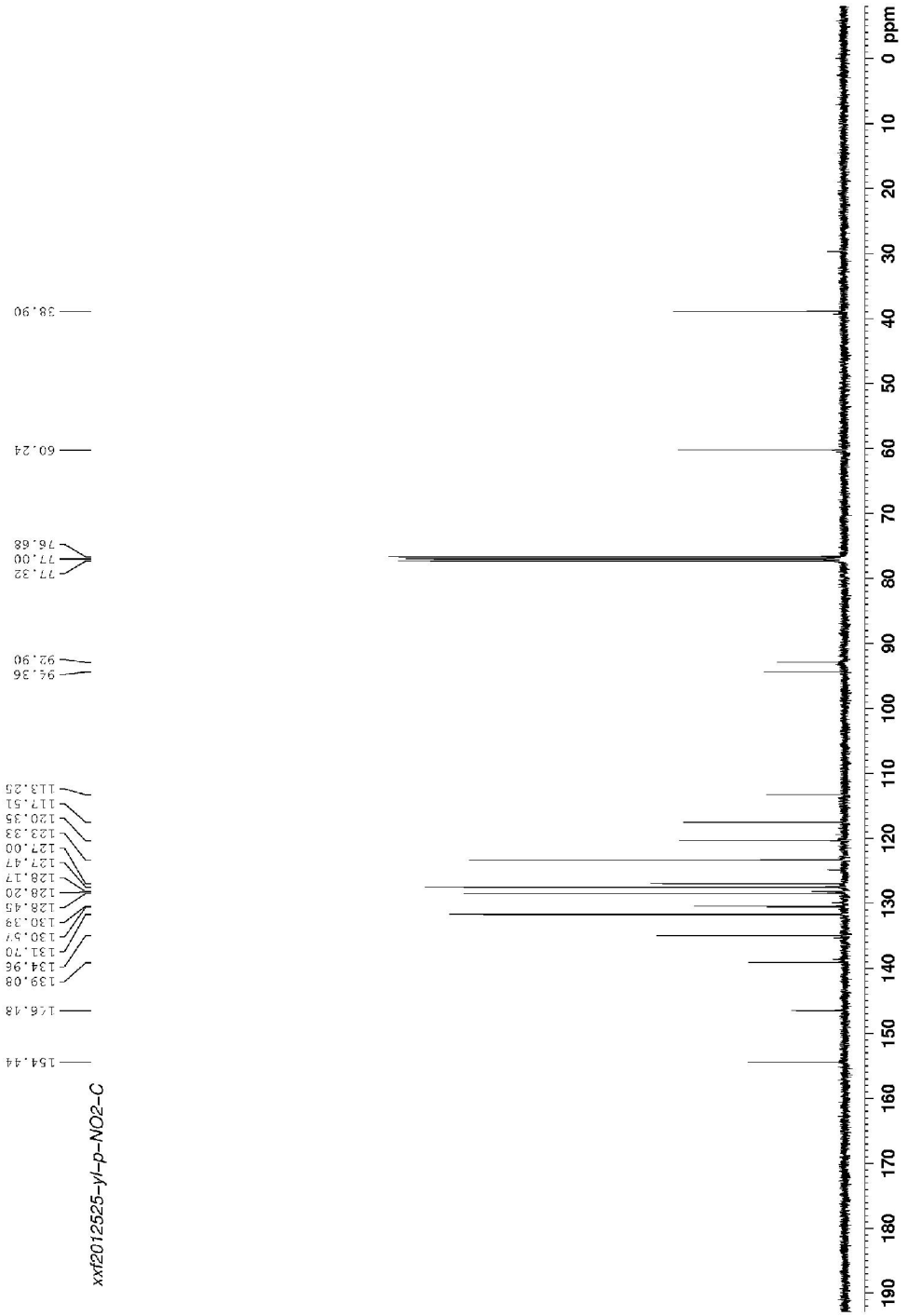
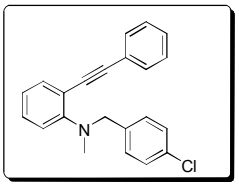
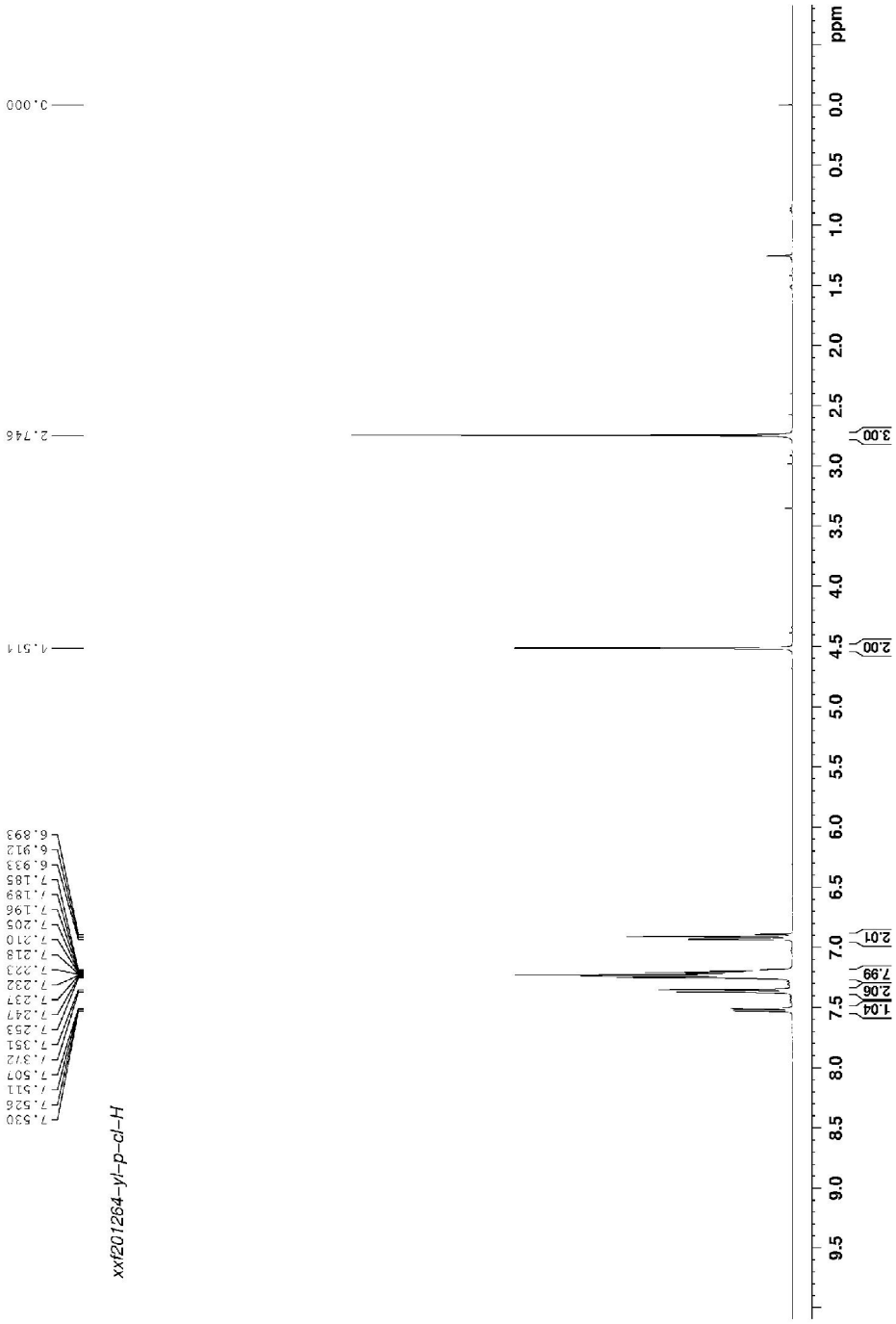
2.822
4.830
6.903
6.925
6.943
6.977
6.997
7.102
7.108
7.112
7.125
7.130
7.136
7.245
7.274
7.279
7.286
7.293
7.297
7.316
7.335
7.348
7.352
7.356
7.442
7.460
7.517
7.521
7.536
7.540
8.019
8.024
8.029
8.041
8.046
8.052
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
10.0
3.00
1.99
1.00
0.99
1.96
4.09
2.03
1.02
1.92


NO2
N


38.90
60.24
76.68
77.00
77.32
92.90
94.36
113.25
117.51
120.35
123.33
127.00
127.47
128.17
128.20
128.45
130.39
130.57
131.70
134.96
139.08
176.18
154.44
xxf2012525-yl-p-NO2-C
0 ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190


N
Cl


xxf201264-yl-p-cl-H
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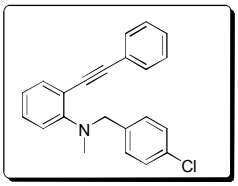
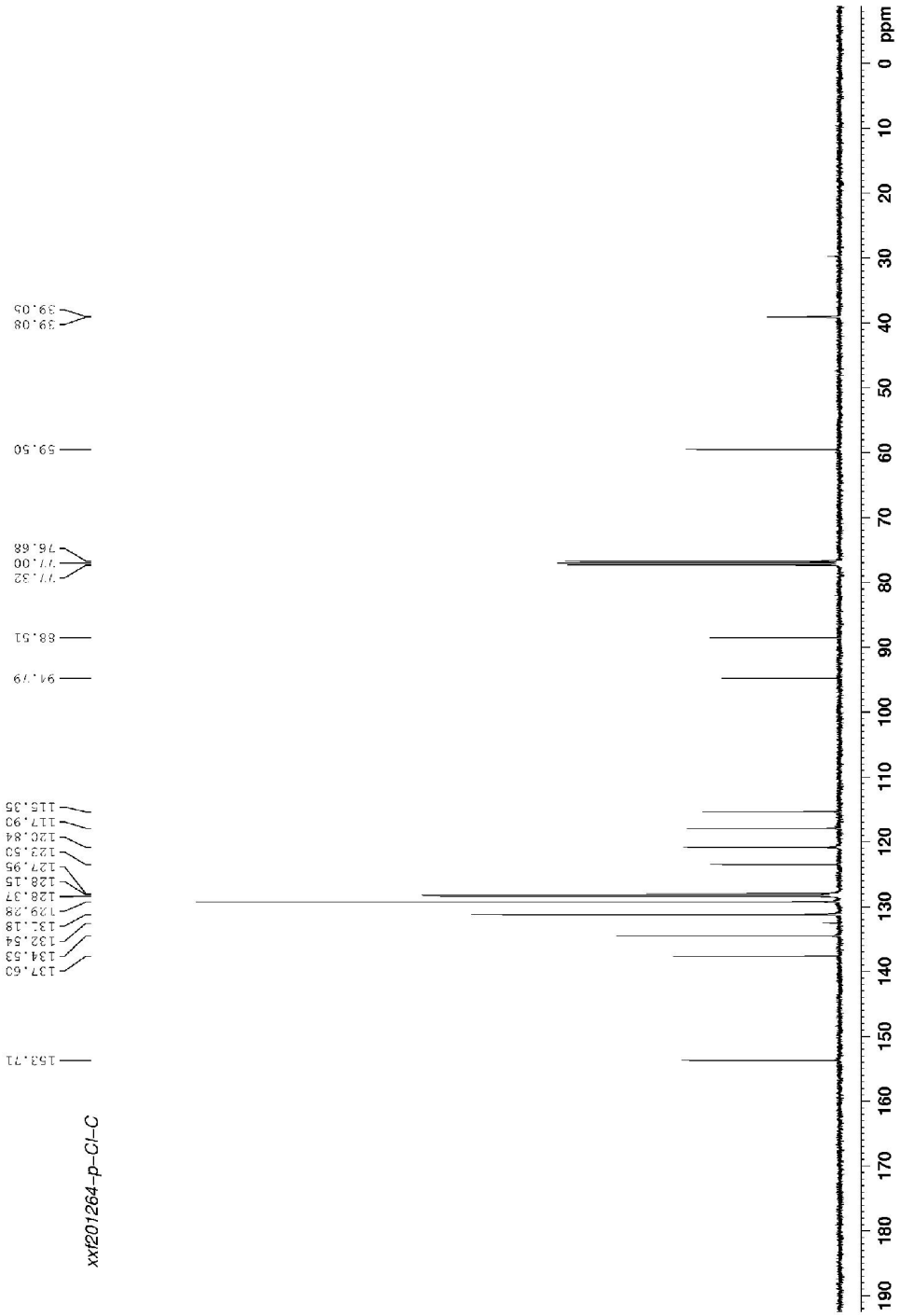
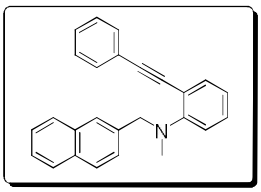
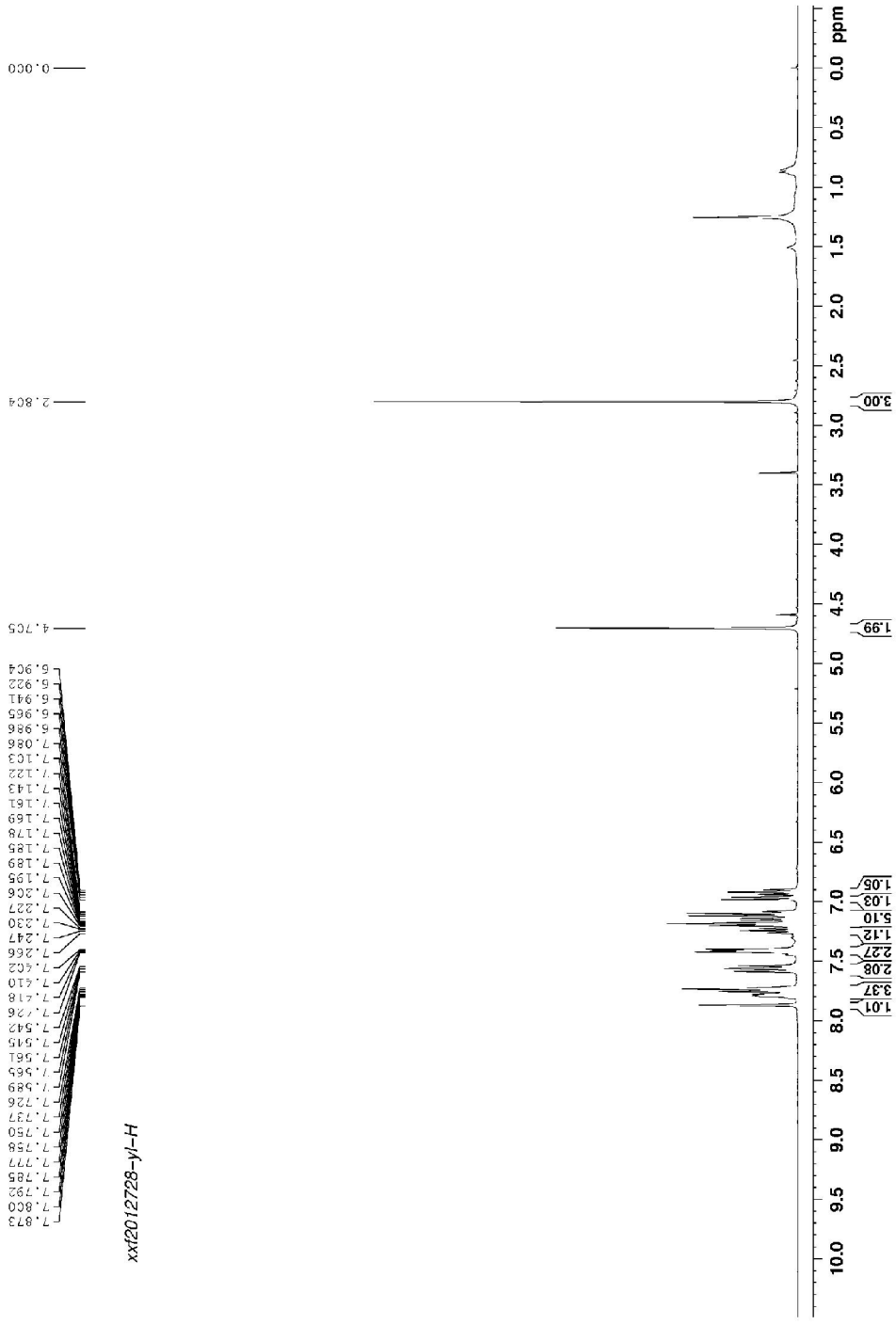
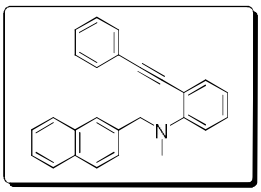
2.00
2.01
2.06
7.99
1.04
ppm

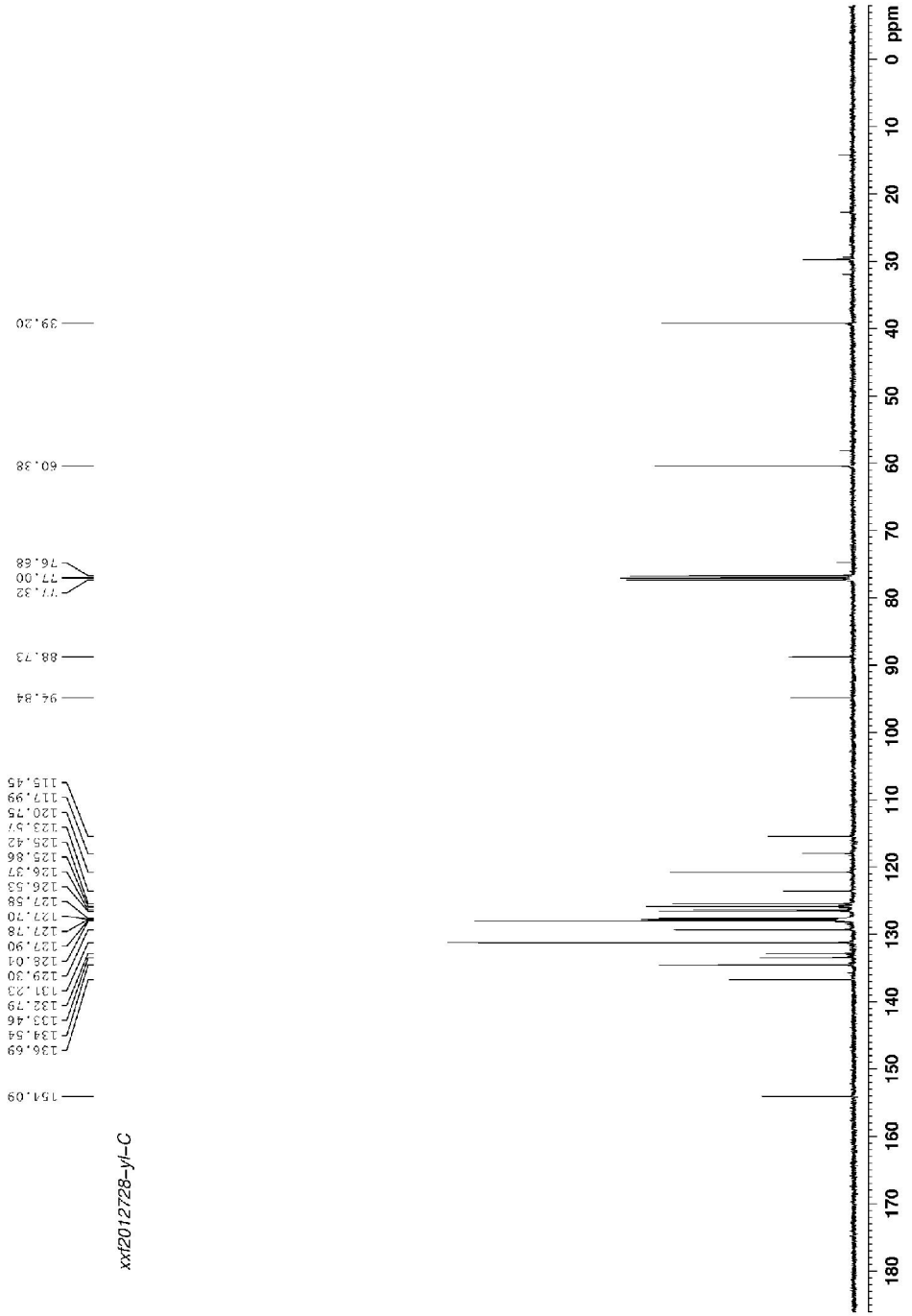

N
Cl

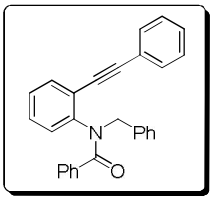

39.05
39.08
59.50
76.68
77.00
77.32
88.51
94.79
115.35
117.93
120.84
123.50
127.95
128.15
128.37
129.28
131.18
132.54
134.53
137.60
153.71
xxf201264-p-Cl-C
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190

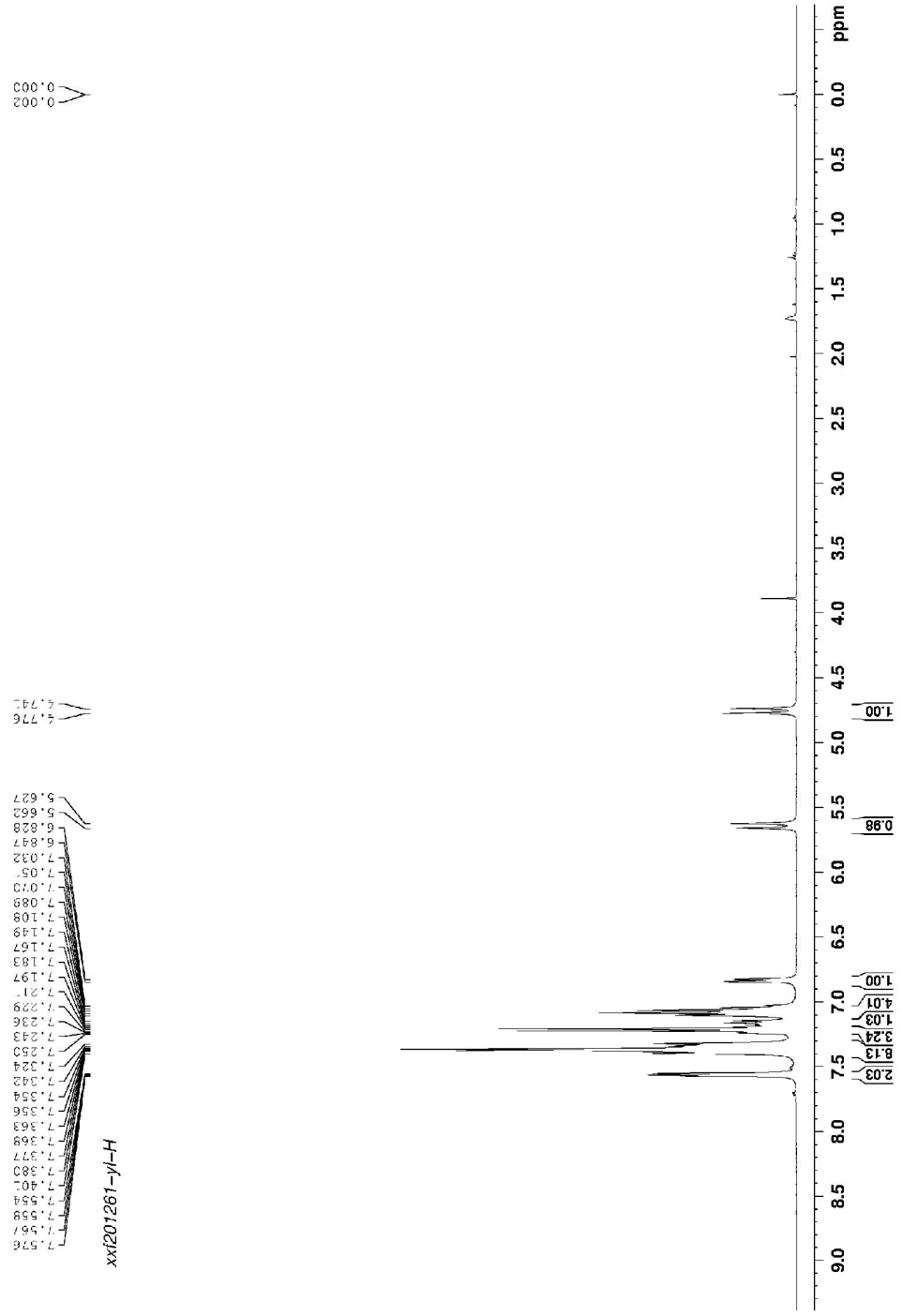


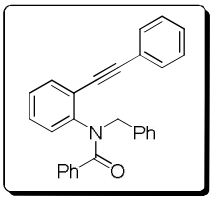

xxf2012728-yl-H
ppm
3.00
1.99
1.05
1.03
5.10
1.12
2.27
2.08
3.37
1.01

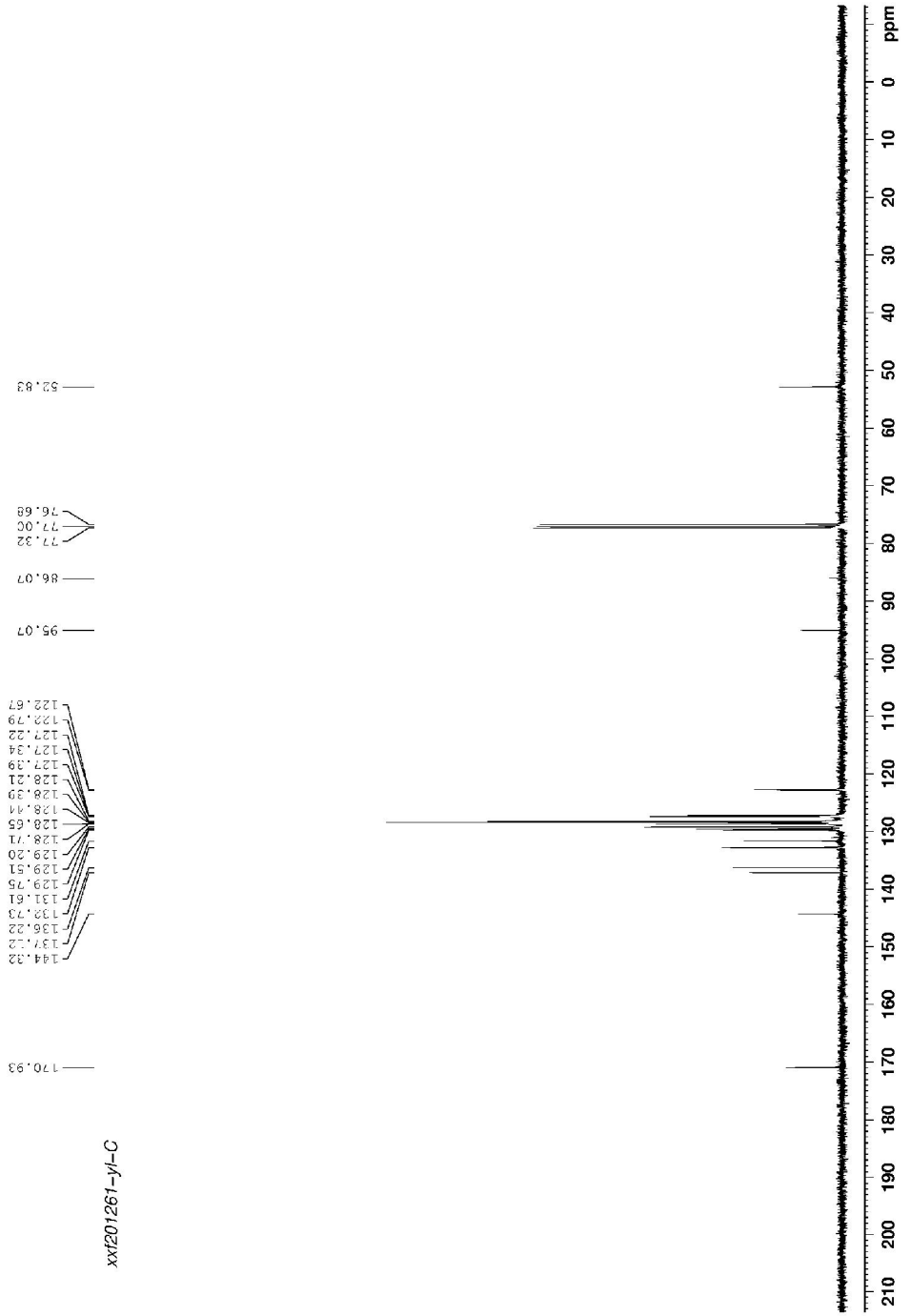


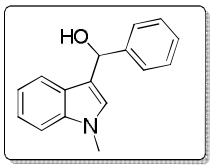

39.20
60.38
76.68
77.00
77.32
88.73
94.84
115.45
117.99
120.75
123.57
125.42
125.86
126.37
126.53
127.58
127.70
127.78
127.90
128.04
129.30
131.23
132.79
133.46
134.54
136.69
154.09
xxf2012728-yl-C
0 ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

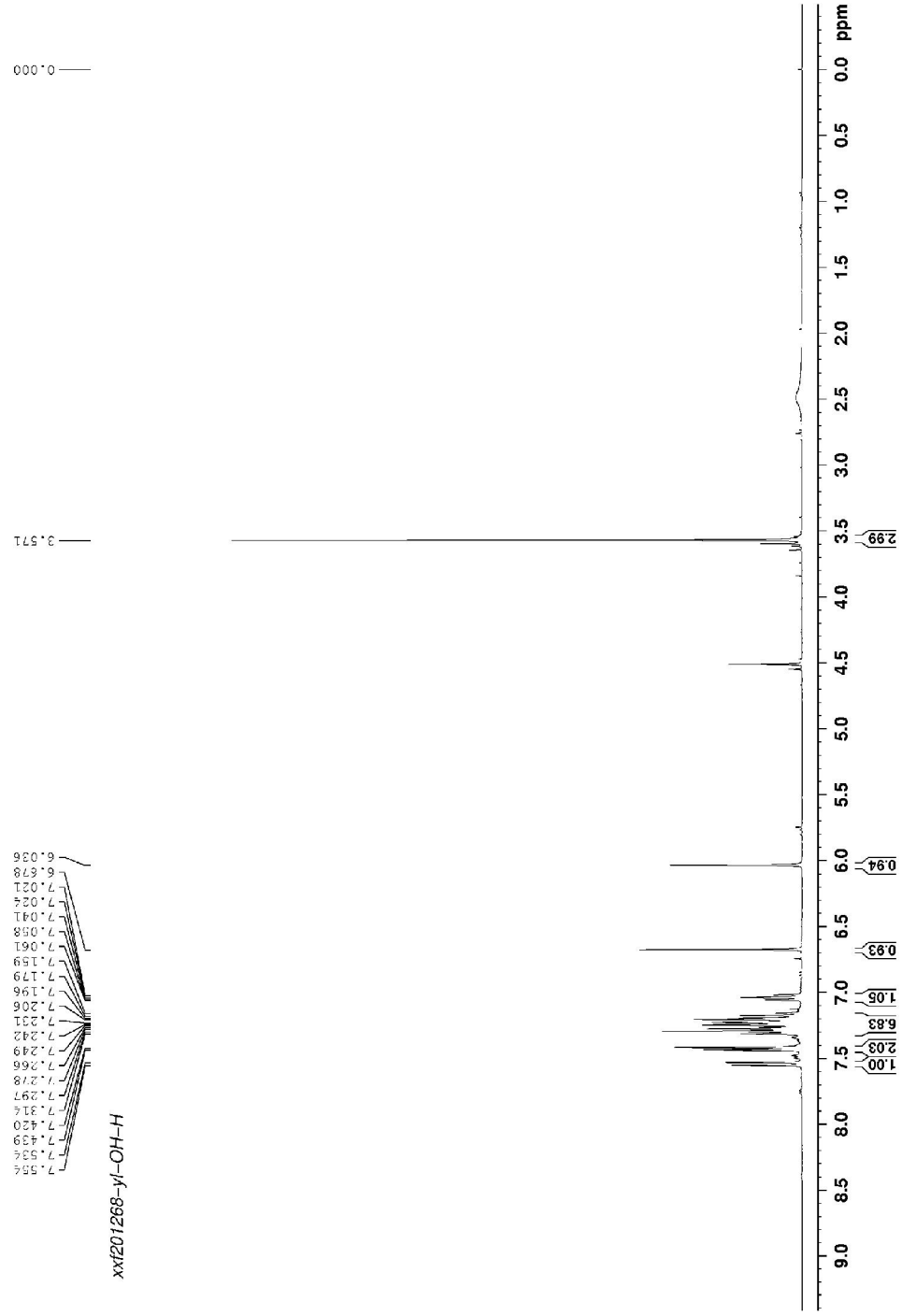

N
Ph
Ph
O

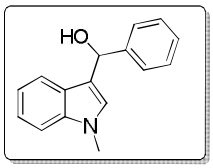

0.002
0.000
4.776
4.742
5.627
5.662
6.828
6.847
7.032
7.057
7.070
7.089
7.108
7.149
7.167
7.183
7.197
7.217
7.229
7.236
7.243
7.250
7.324
7.342
7.354
7.356
7.363
7.369
7.377
7.380
7.402
7.554
7.558
7.567
7.576
xxfi201261-yl-H
1.00
0.98
1.00
4.01
1.03
3.24
8.13
2.03
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0

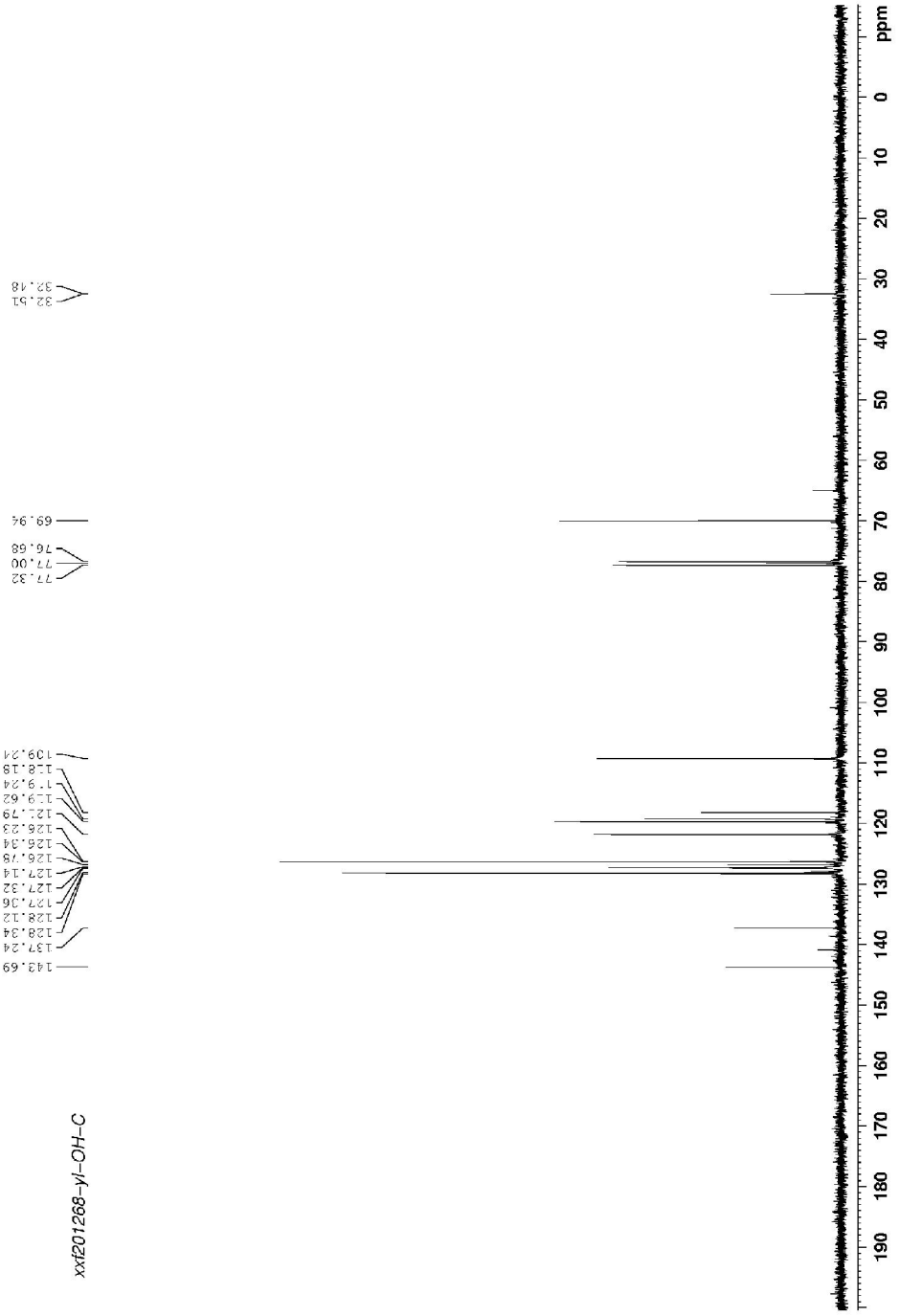

N
Ph
Ph
O


52.83
76.68
77.00
77.32
86.07
95.07
122.67
122.79
127.22
127.34
127.39
128.21
128.39
128.41
128.65
128.71
129.20
129.51
129.75
131.61
132.73
136.22
137.12
144.32
170.93
xxf201261-yl-C
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210


HO
N


xxf201268-yl-OH-H
0.000
3.571
2.99
0.94
0.93
1.05
6.83
2.03
1.00
ppm


HO
N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110
120
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140
150
160
170
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190
32.18
32.51
69.94
76.68
77.00
77.32
109.24
118.18
119.24
119.62
121.79
126.23
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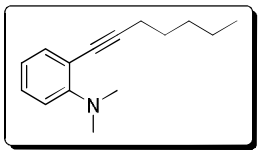
127.14
127.32
127.36
128.12
128.34
137.24
143.69
xxf201268-yl-OH-C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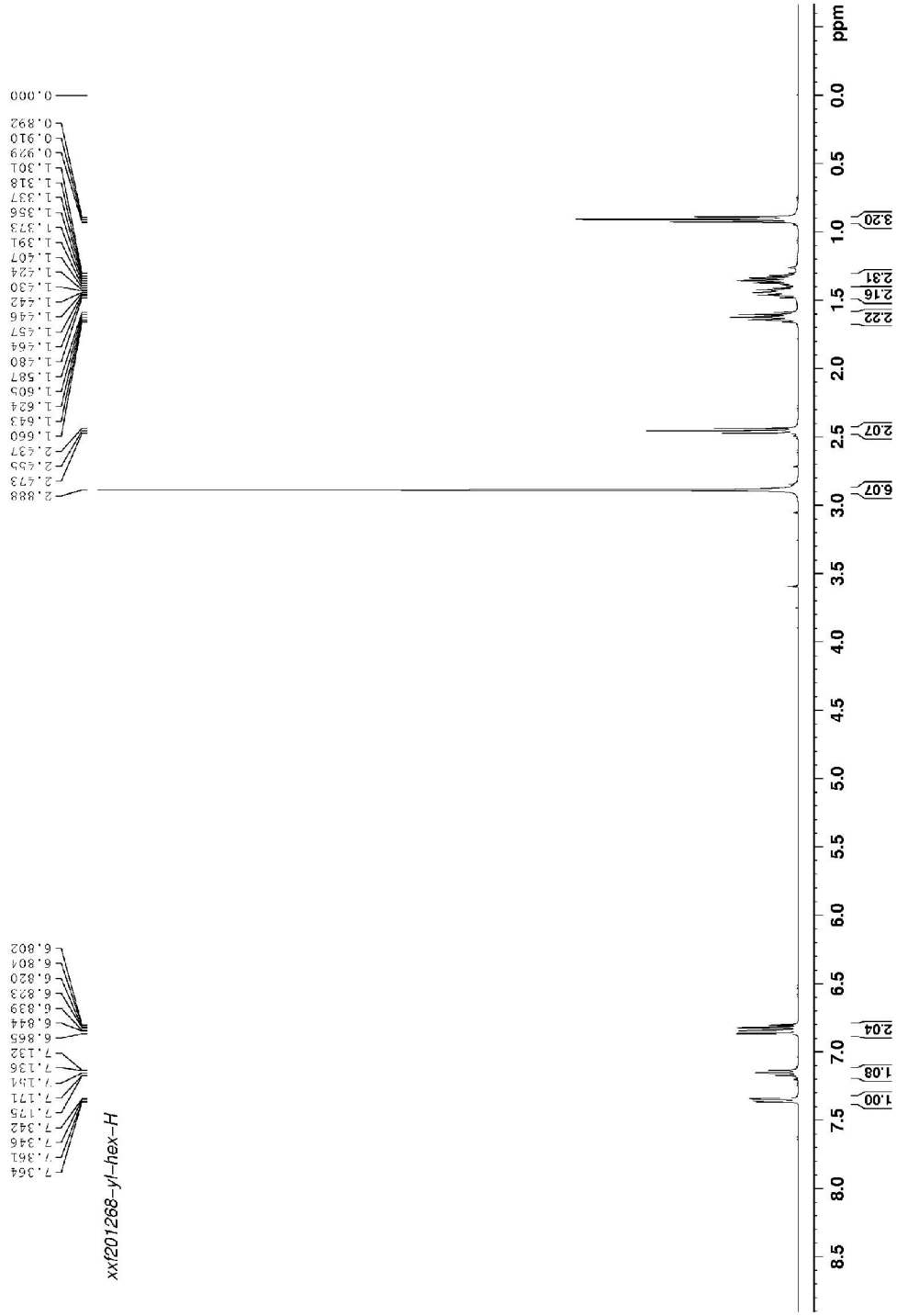
xxf201268-yl-hex-H
ppm
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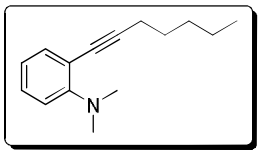
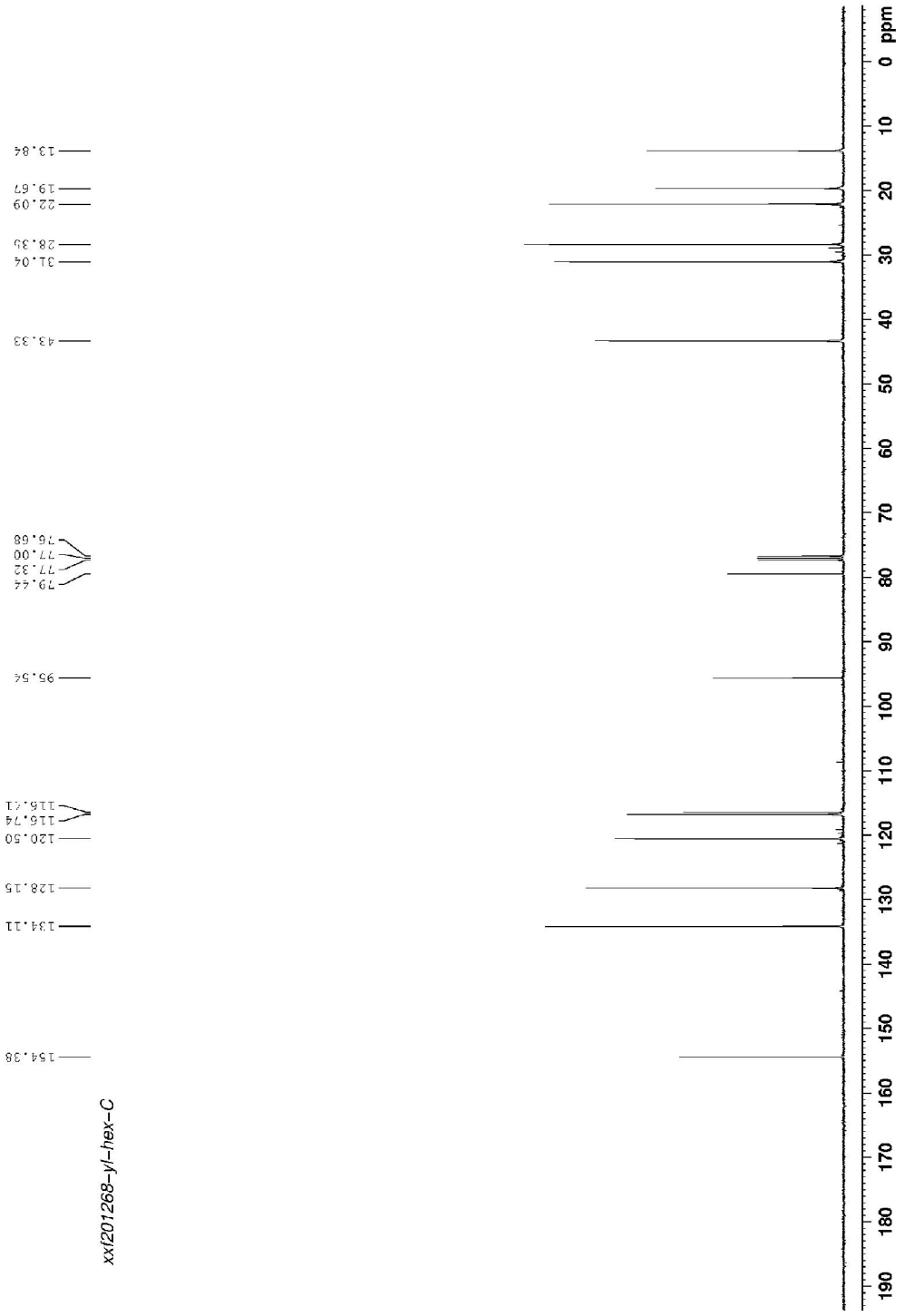
2.16
2.22
2.07
6.07
2.04
1.08
1.00

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19.67
22.09
28.35
31.04
43.33
76.68
77.00
77.32
79.44
95.54
116.21
116.74
120.50
128.15
134.11
154.38
xxl201268-yl-hex-C
ppm
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40
50
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70
80
90
100
110
120
130
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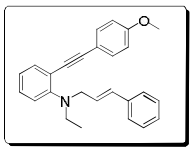
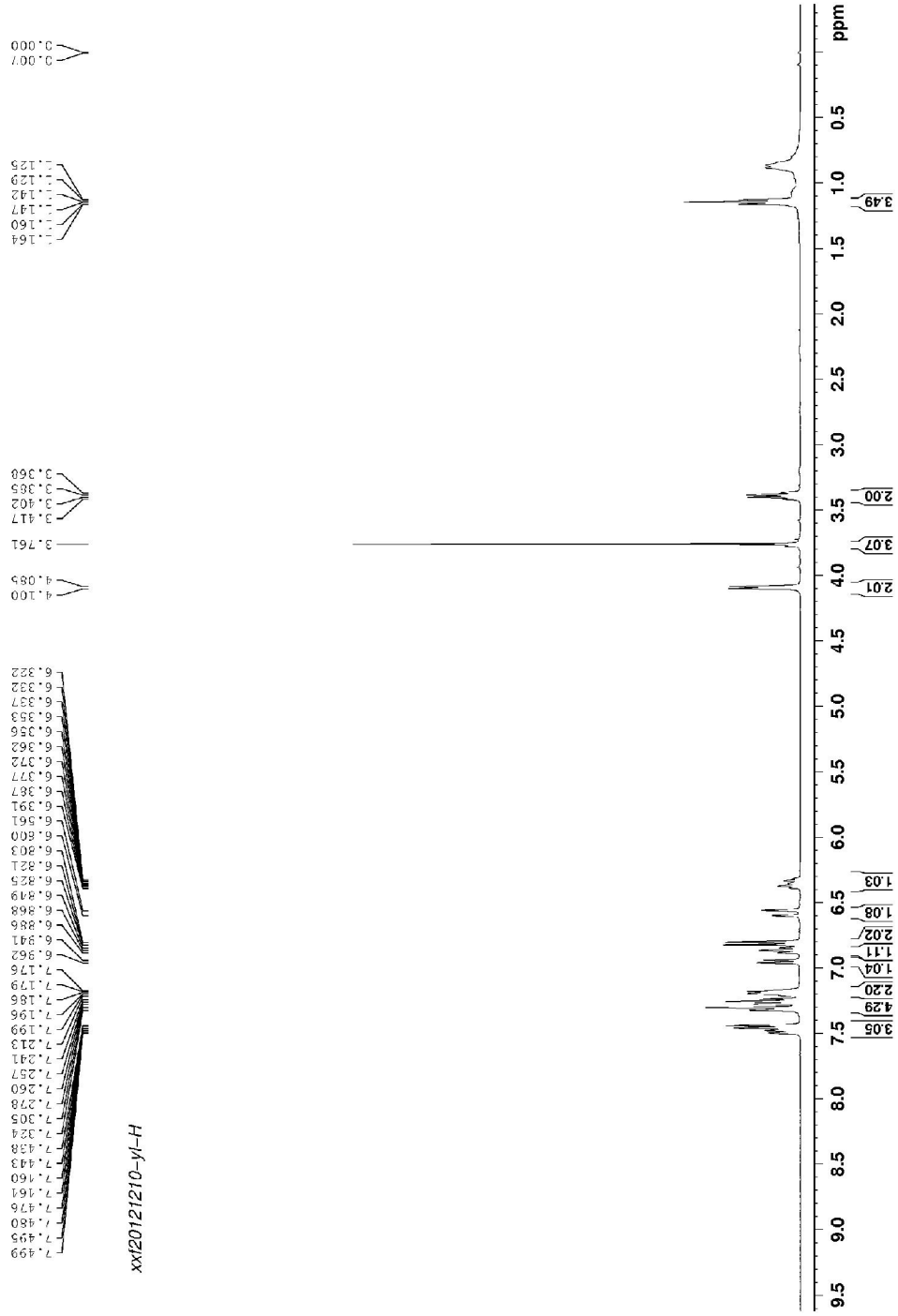
150
160
170
180
190

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4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
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3.07
2.01
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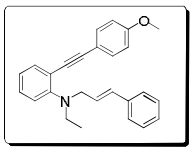
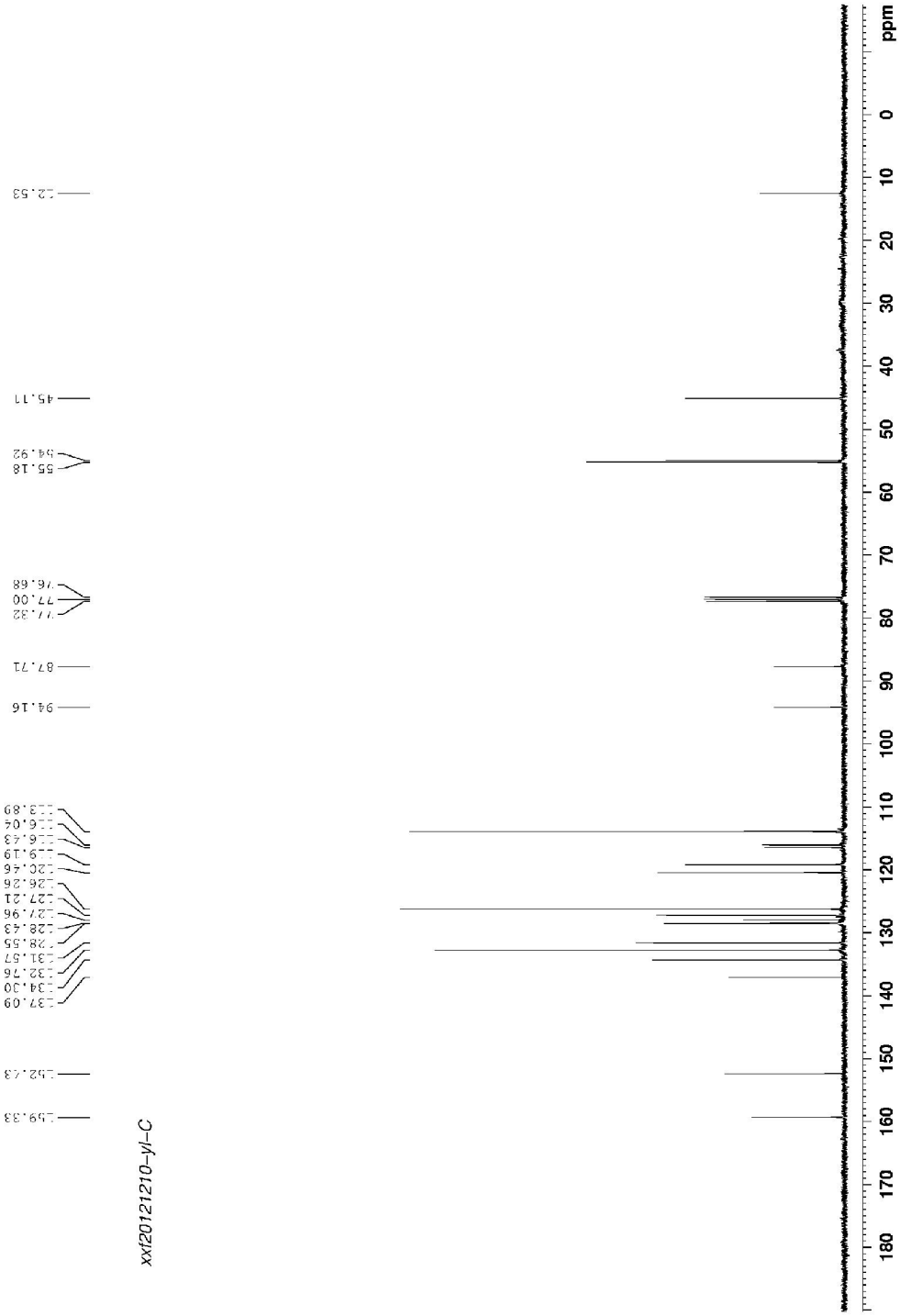
2.02
1.11
1.04
2.20
4.29
3.05

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54.92
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77.00
77.32
87.71
94.16
113.89
116.04
116.43
119.19
120.46
126.26
127.21
127.96
128.43
128.55
131.57
132.76
134.30
137.09
152.43
159.33
xxl20121210-yl-C
ppm
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30
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70
80
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150
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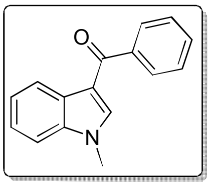

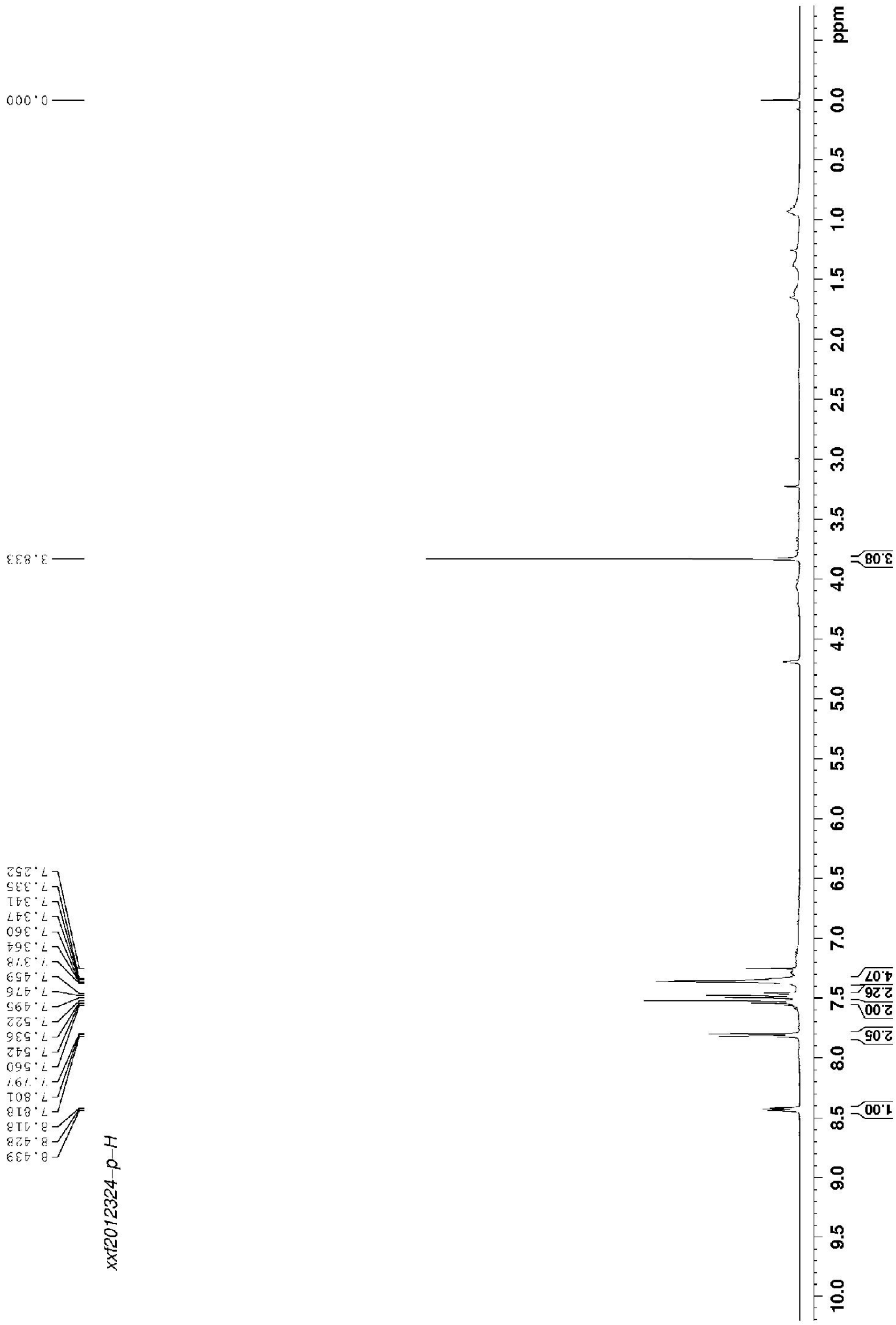

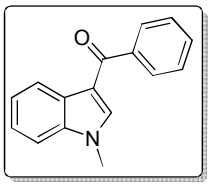
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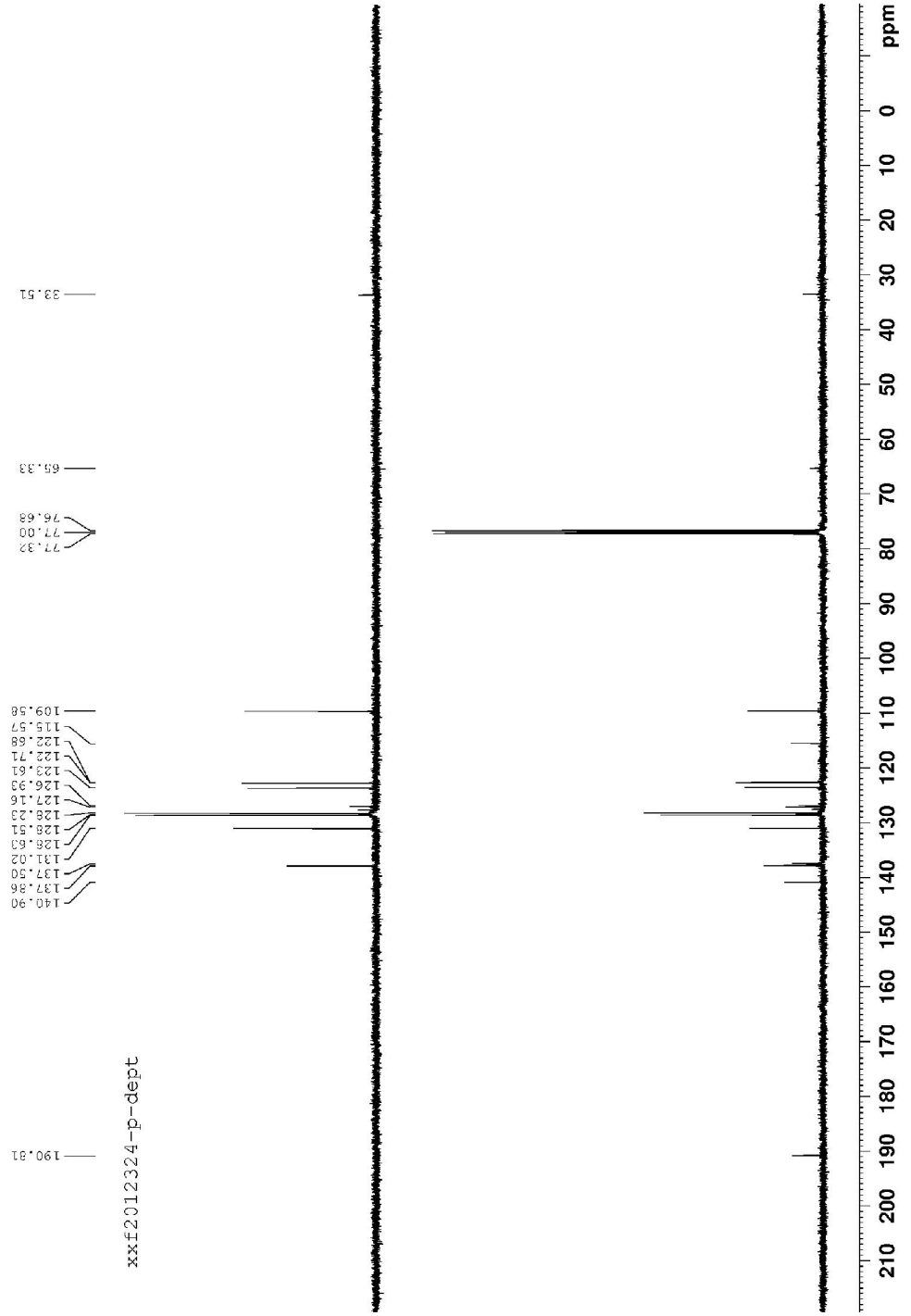
33.51
65.33
76.68
77.00
77.32
109.58
115.57
122.68
122.71
123.61
126.93
127.16
128.23
128.51
128.63
131.02
137.50
137.86
140.90
190.81
xxf2012324-p-dept
ppm
0
10
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30
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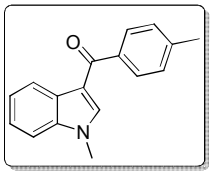
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210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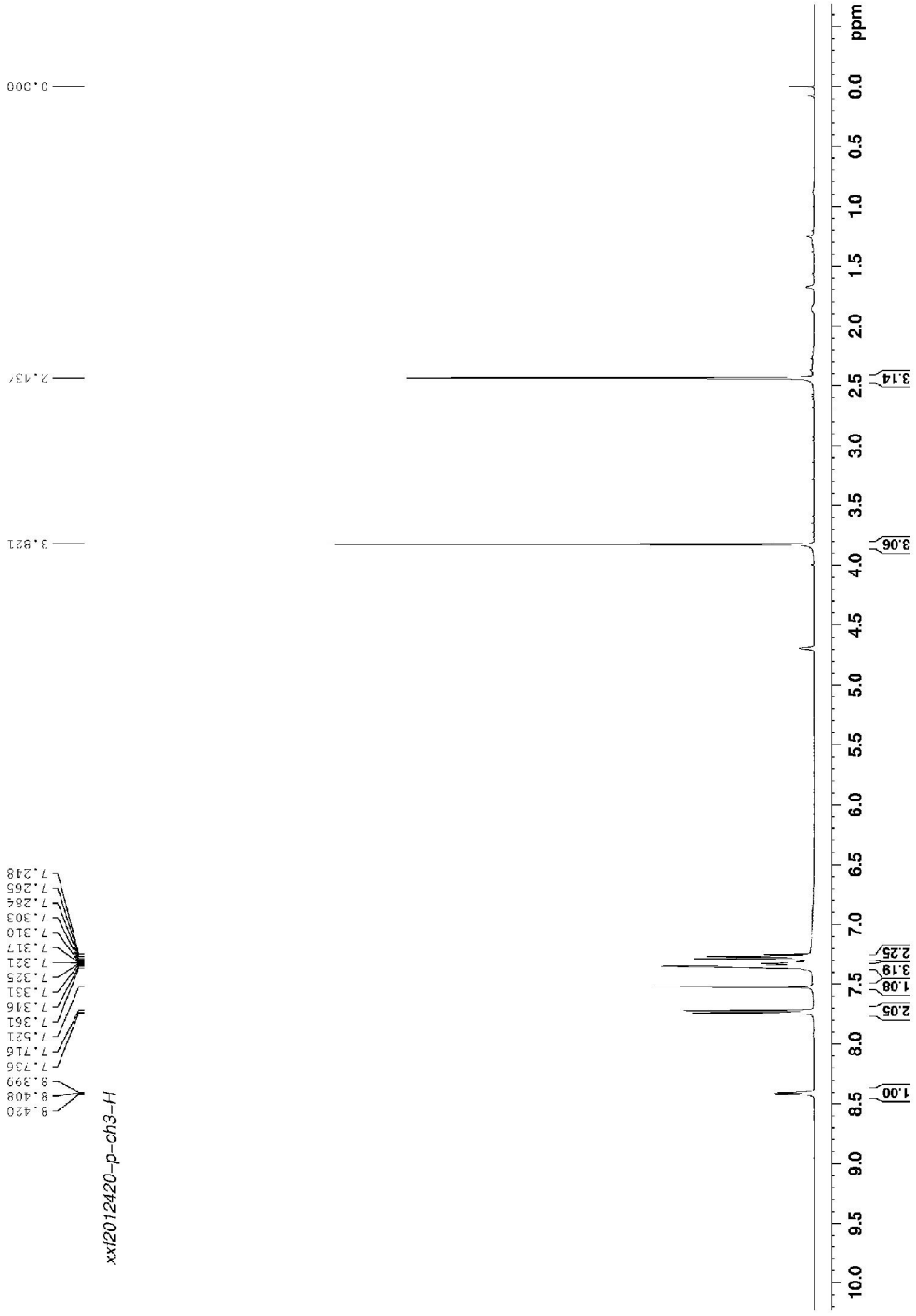
xxf20120420-p-ch3-H
8.420
8.408
8.399
7.736
7.716
7.521
7.361
7.346
7.331
7.325
7.321
7.317
7.310
7.303
7.284
7.265
7.248
7.437
3.821
0.300
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
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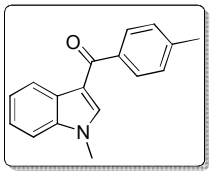
9.0
9.5
10.0
3.14
3.06
2.25
3.19
1.08
2.05
1.00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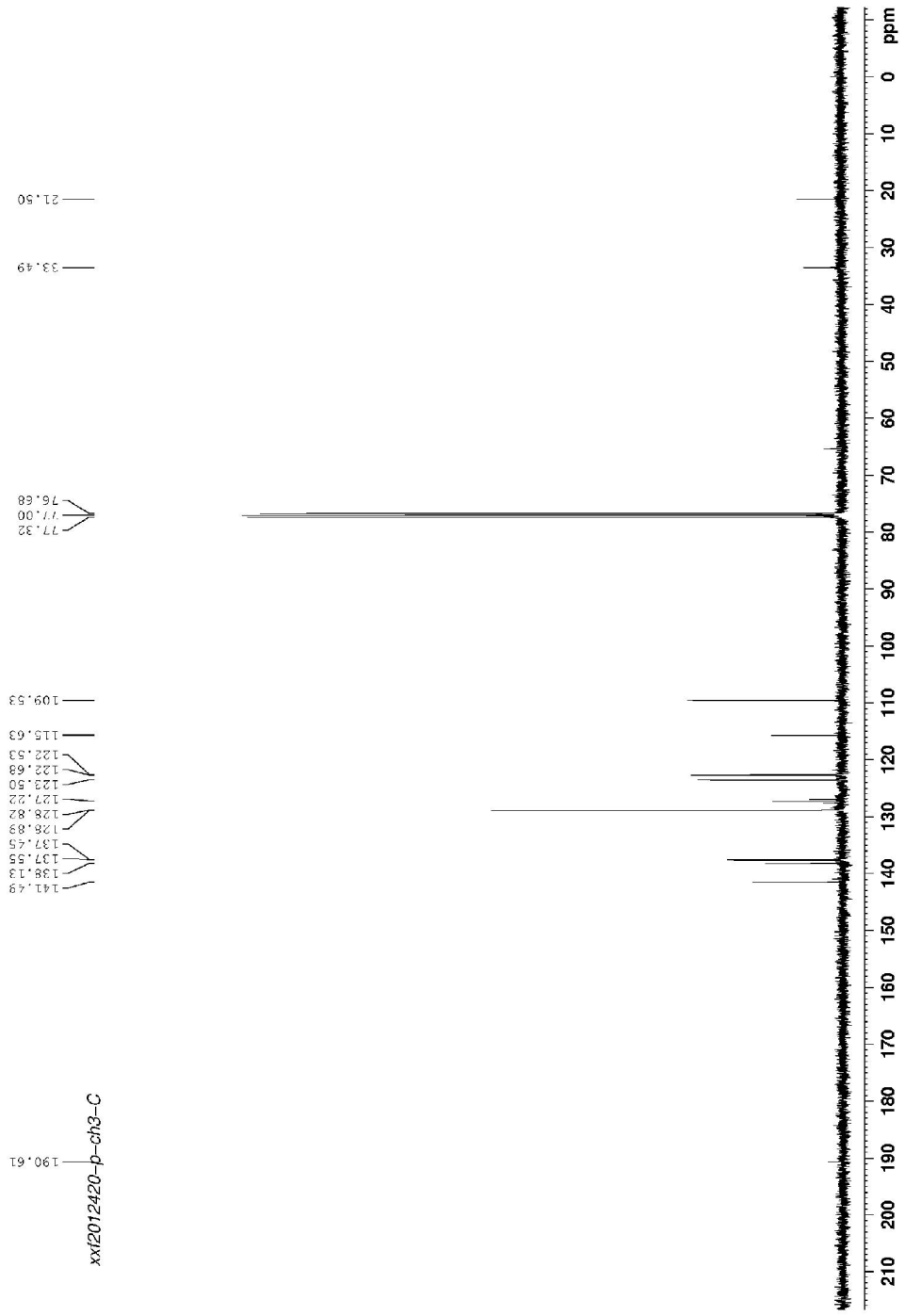
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33.49
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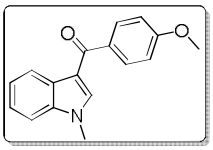
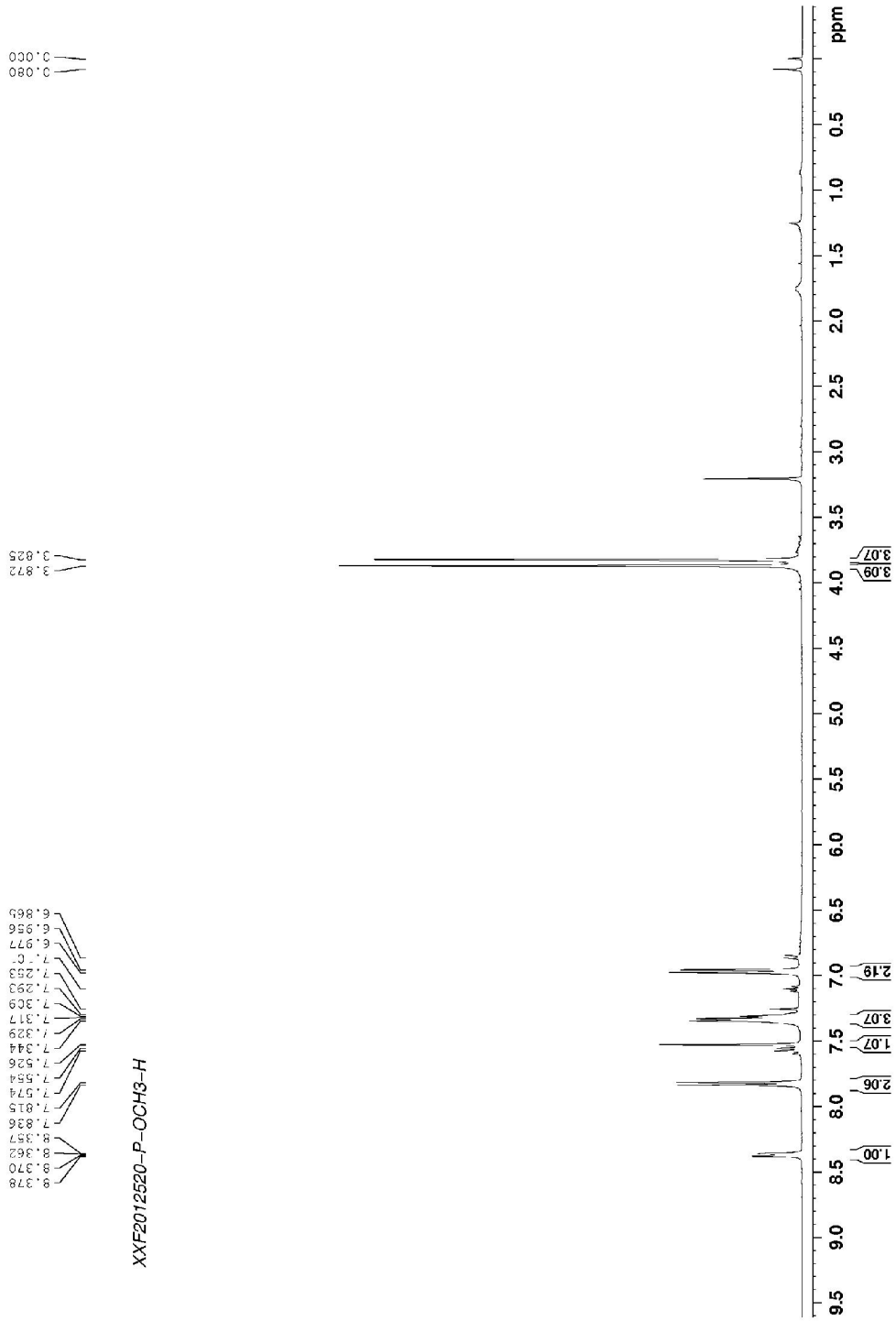
77.00
77.32
109.53
115.63
122.53
122.68
123.50
127.22
128.82
128.89
137.45
137.55
138.13
141.49
190.61
xxf2012420-p-ch3-C
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210

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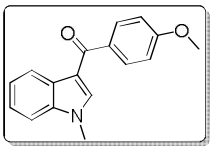
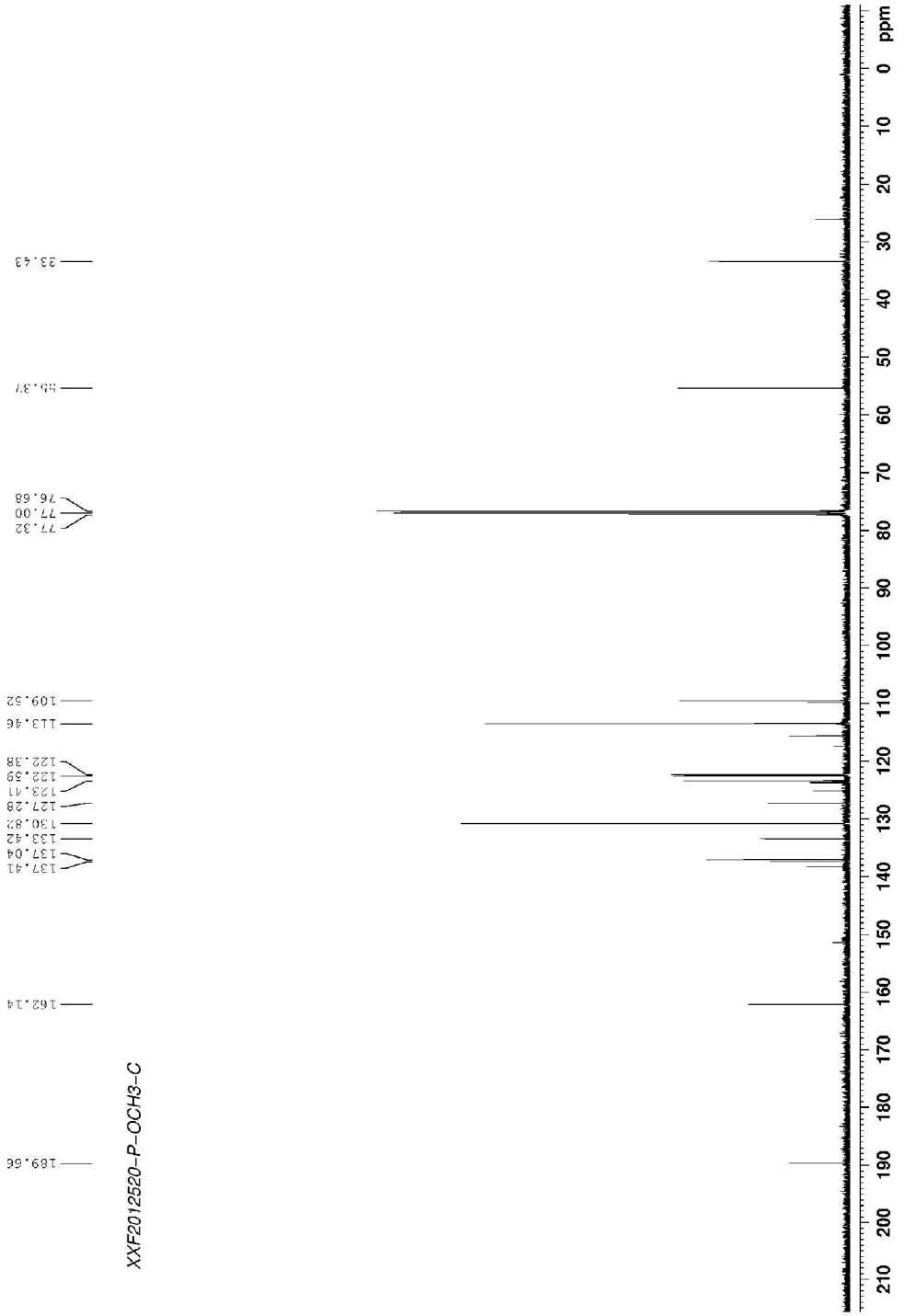
3.09
2.19
3.07
1.07
2.06
1.00

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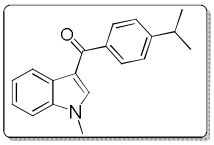
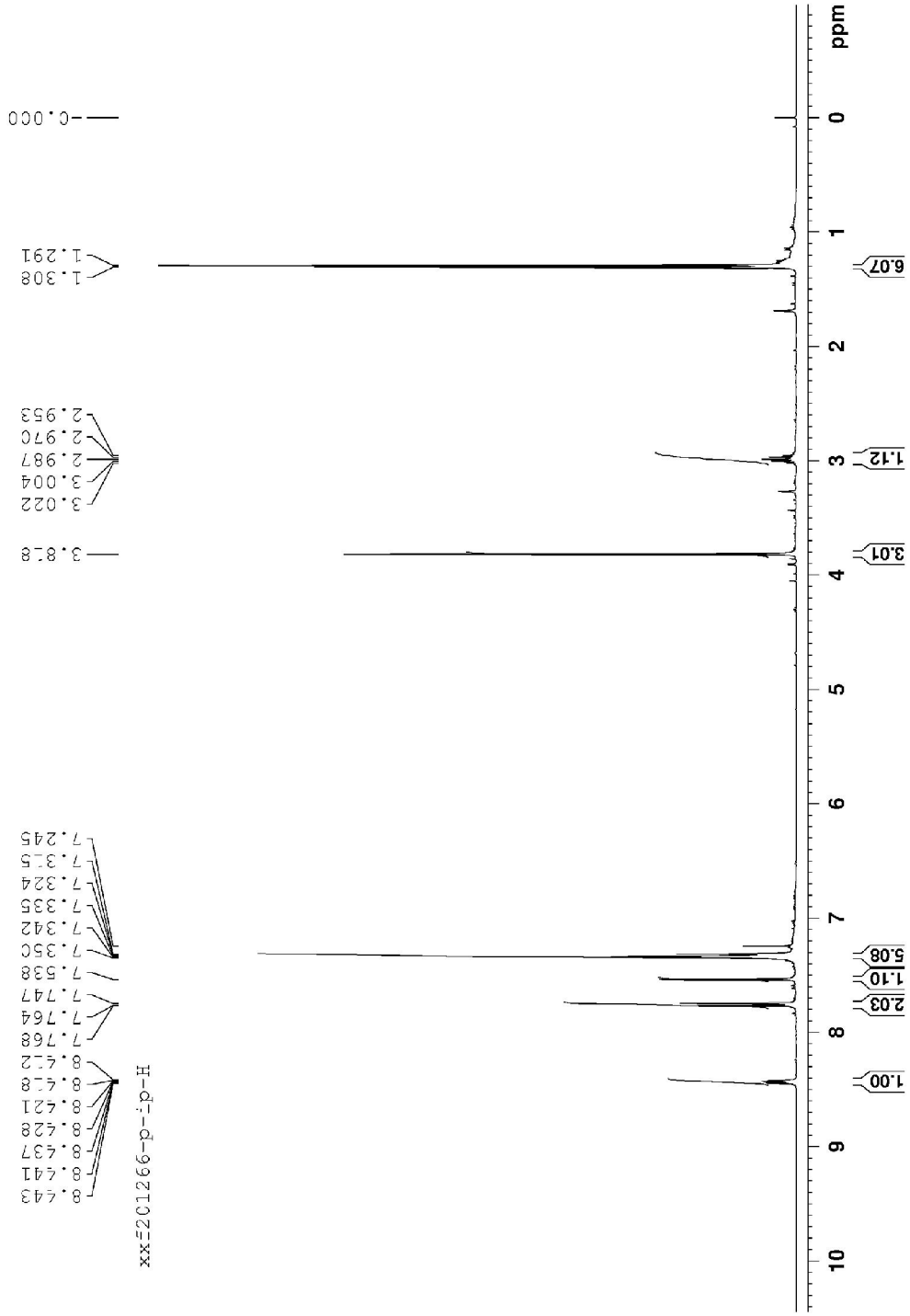
55.37
76.68
77.00
77.32
109.52
113.46
122.38
122.59
123.41
127.28
130.82
133.42
137.04
137.41
162.14
189.66
XXF2012520-P-OCH3-C
ppm
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210




H-d:-d-991201.xx
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8.437
8.428
8.421
8.418
8.412
7.768
7.764
7.747
7.538
7.350
7.342
7.335
7.324
7.315
7.245
3.822
3.004
2.987
2.970
2.953
1.308
1.291
0.000
ppm
0
1
2
3
4
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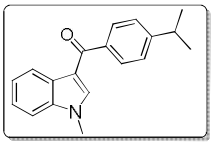
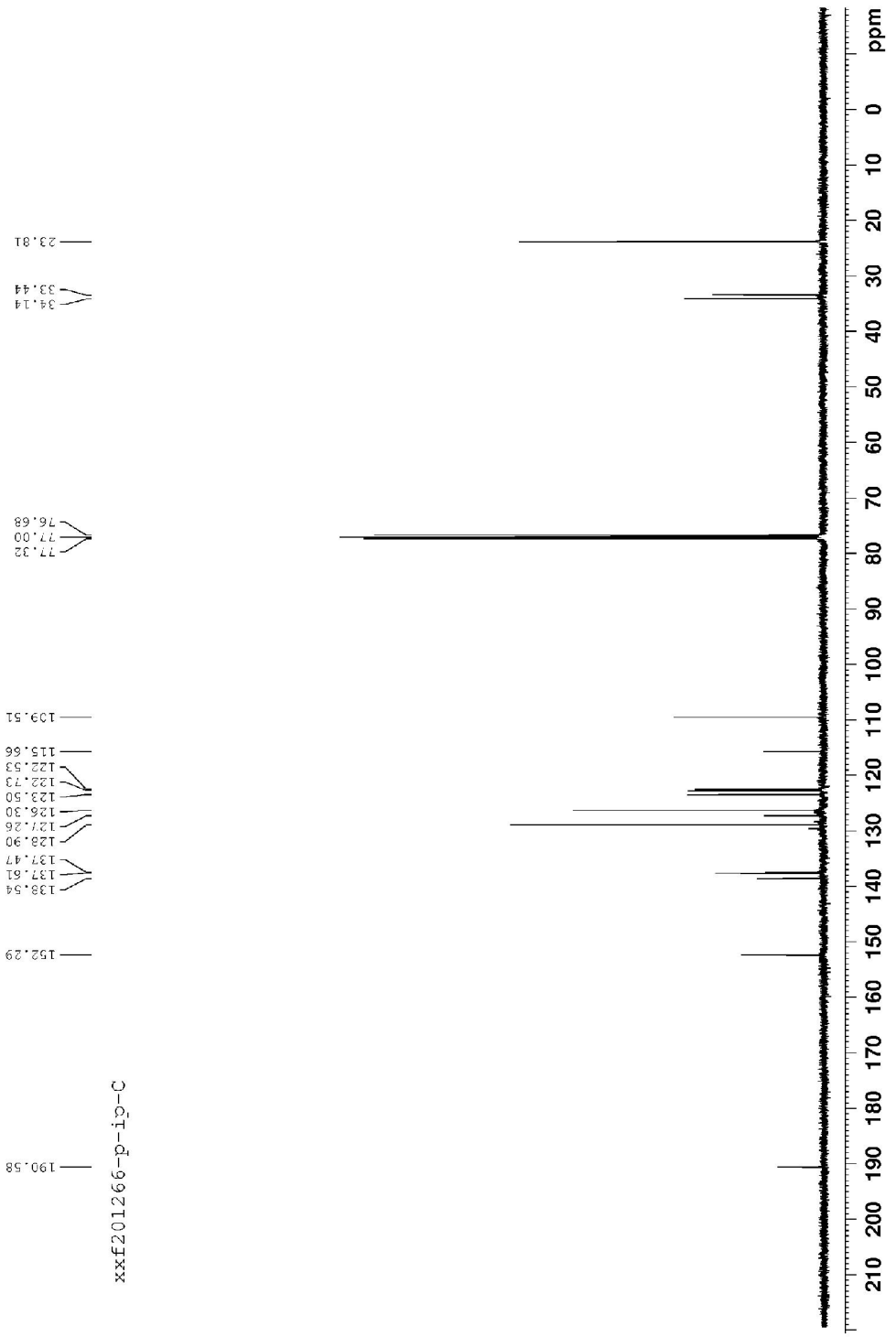
6
7
8
9
10
6.07
1.12
3.01
5.08
1.10
2.03
1.00

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77.32
139.51
115.66
122.53
122.73
123.50
126.30
127.26
128.90
137.47
137.61
138.54
152.29
190.58
xxf201266-p-ic-C
ppm
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60
70
80
90
100
110
120
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140
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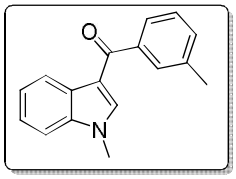
170
180
190
200
210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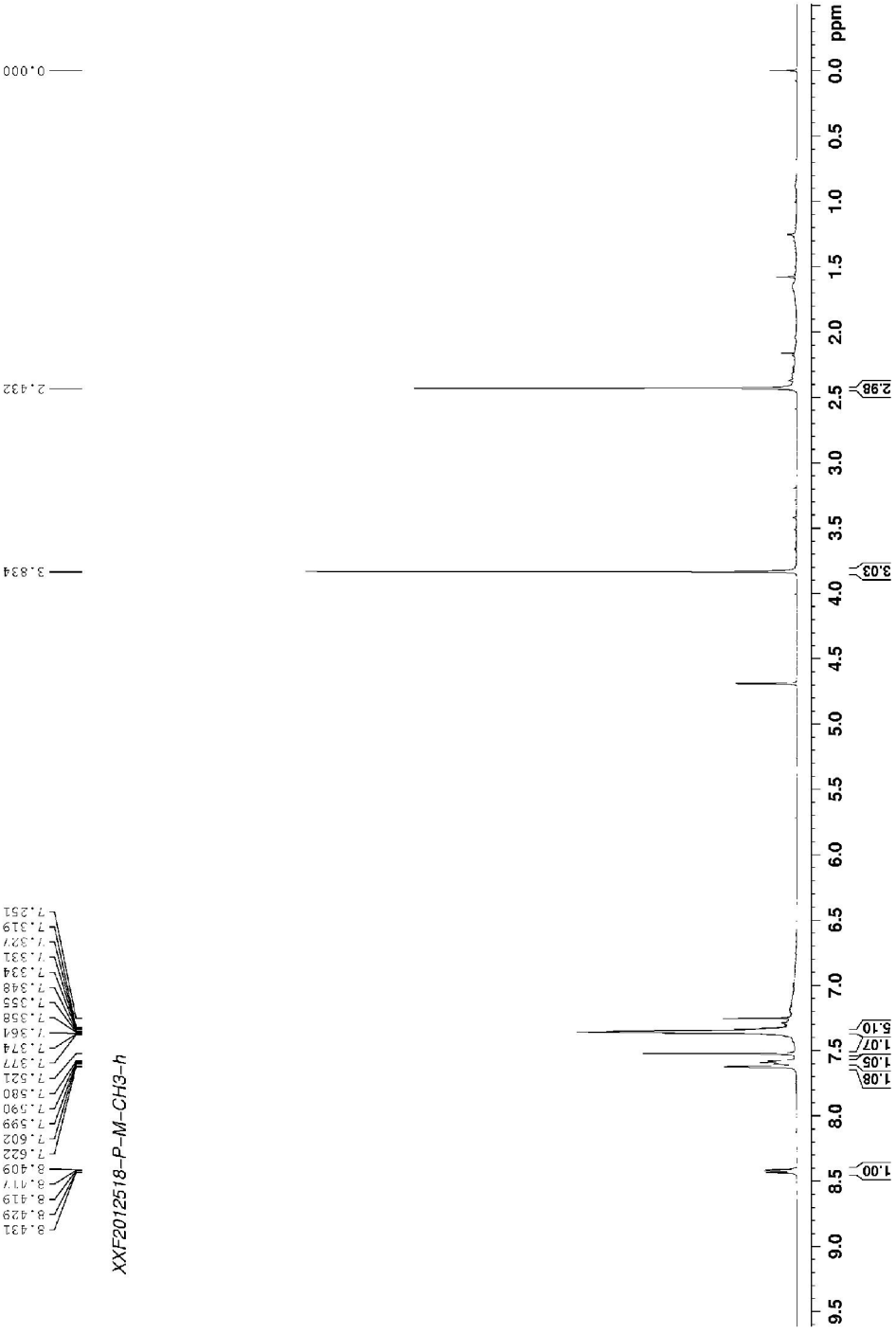
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7.602
7.599
7.590
7.580
7.521
7.377
7.374
7.364
7.358
7.355
7.348
7.334
7.331
7.327
7.319
7.251
XXF2012518-P-M-CH3-h
ppm
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0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
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5.10
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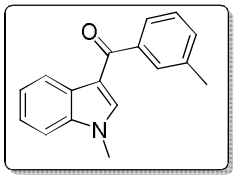
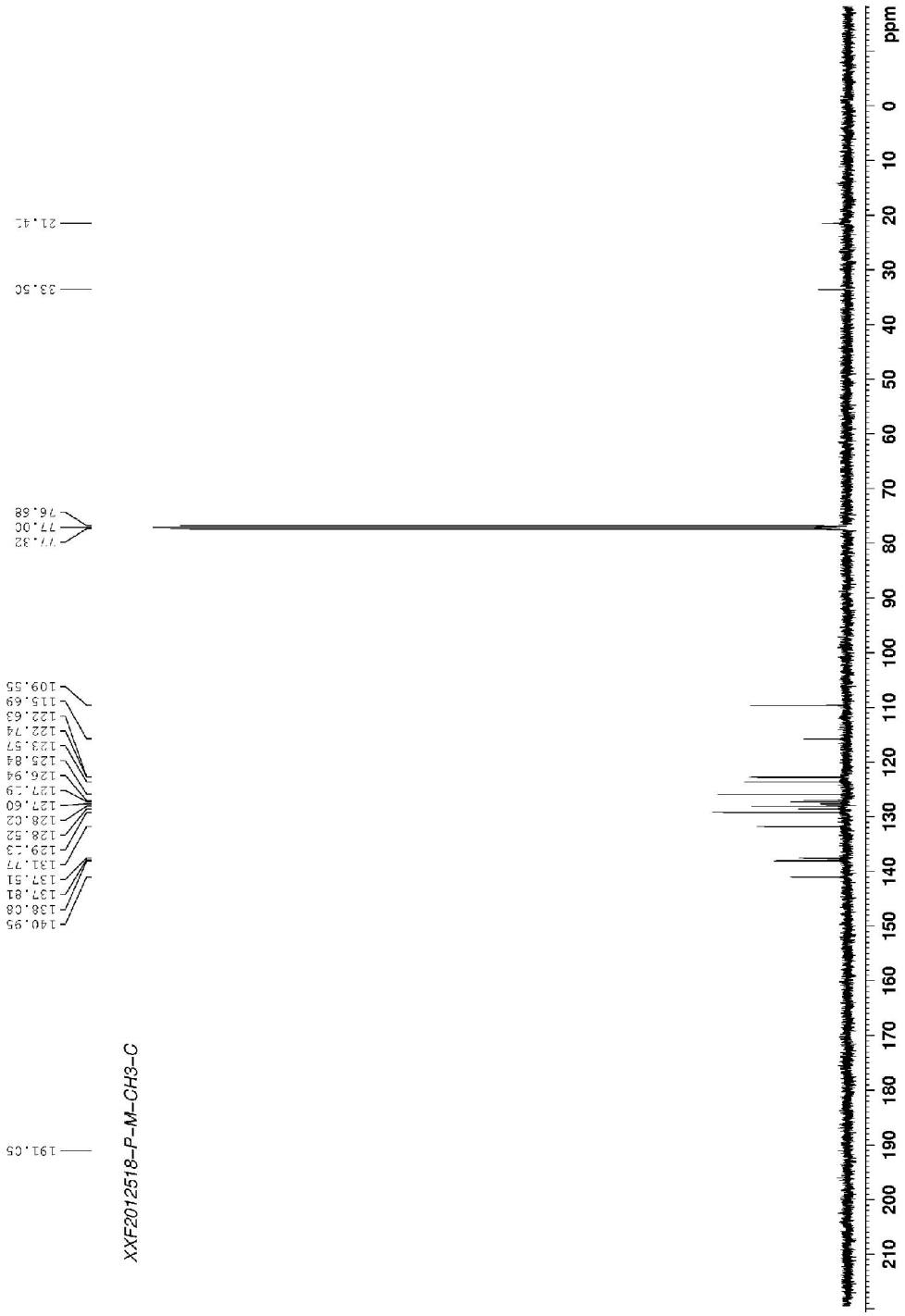
1.05
1.08
1.00


O
N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77.32
109.55
115.69
122.63
122.74
123.57
125.84
126.94
127.19
127.60
128.02
128.52
129.13
131.77
137.51
137.81
138.08
140.95
191.05
XXF2012518-P-M-CH3-C
ppm
0
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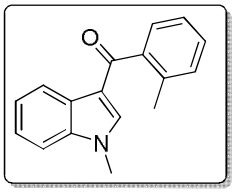
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210


O
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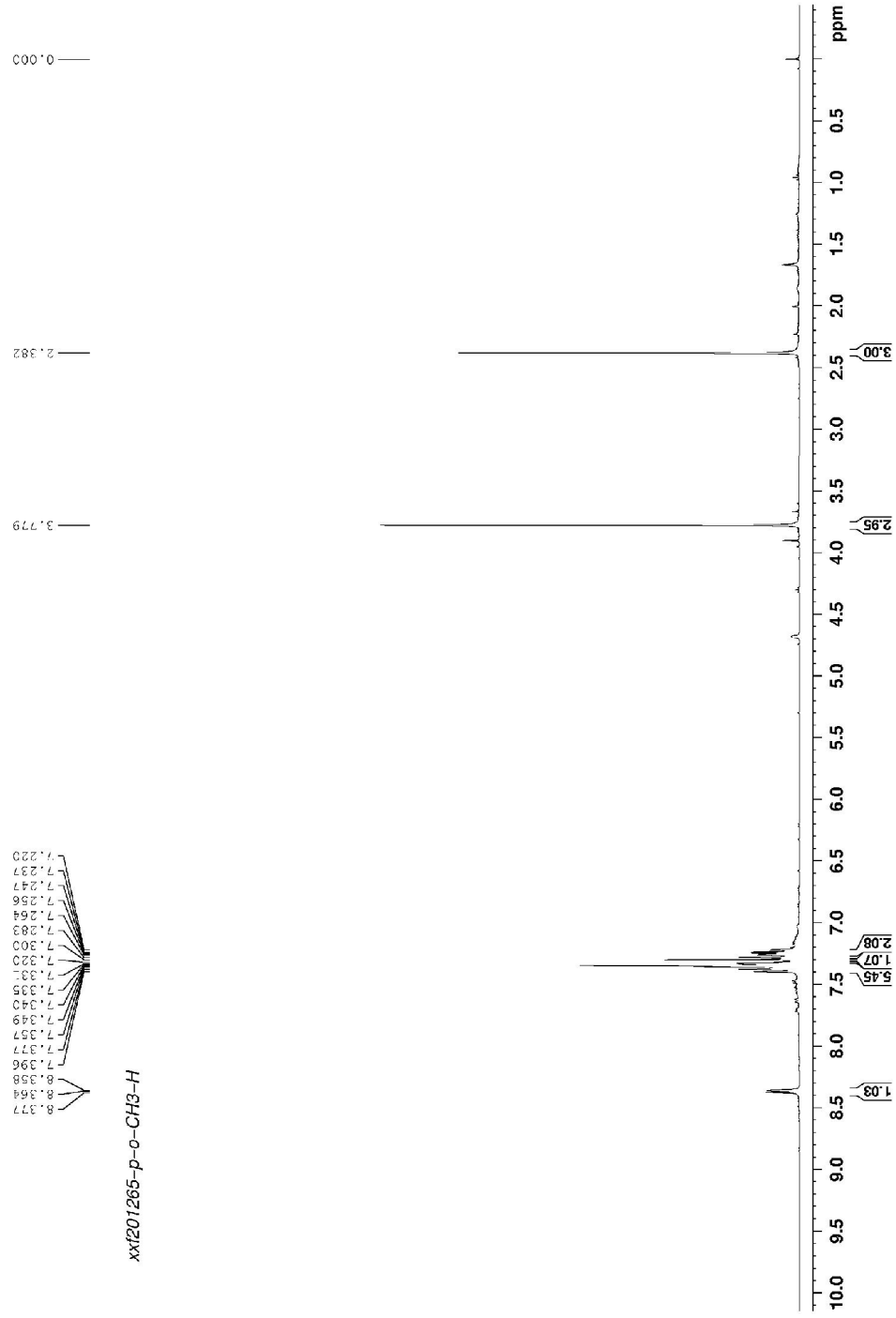
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8.358
7.396
7.377
7.357
7.349
7.340
7.335
7.330
7.320
7.303
7.283
7.264
7.256
7.247
7.237
7.220
xxl201265-p-o-CH3-H
ppm
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1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
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8.5
9.0
9.5
10.0
3.00
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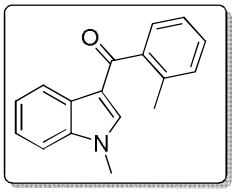
1.07
5.45
1.03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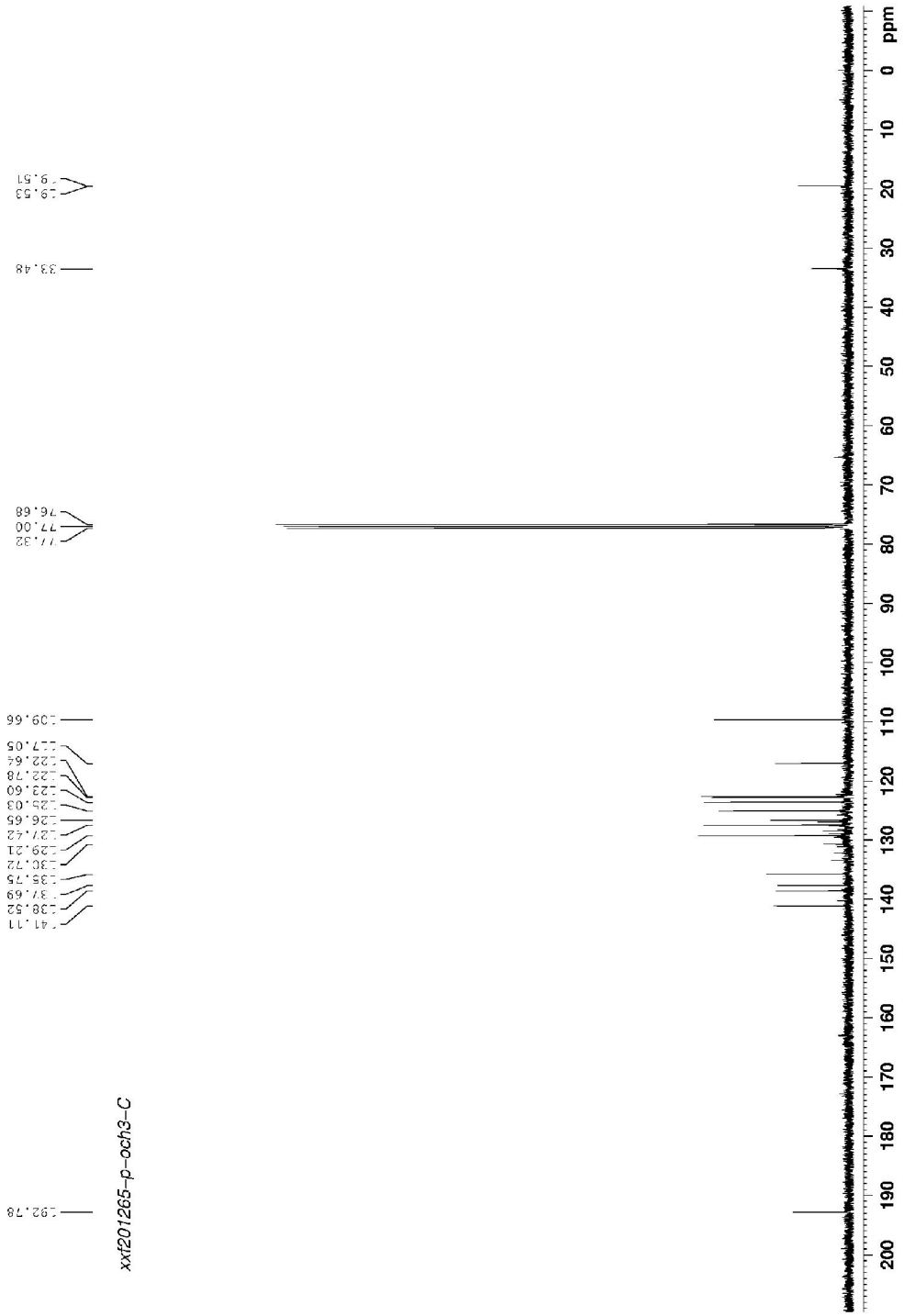
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77.32
109.66
117.05
122.64
122.78
123.60
125.03
126.65
127.42
129.21
130.72
135.75
137.69
138.52
141.11
192.78
xxf201265-p-och3-C
ppm
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50
60
70
80
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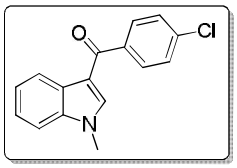
100
110
120
130
140
150
160
170
180
190
200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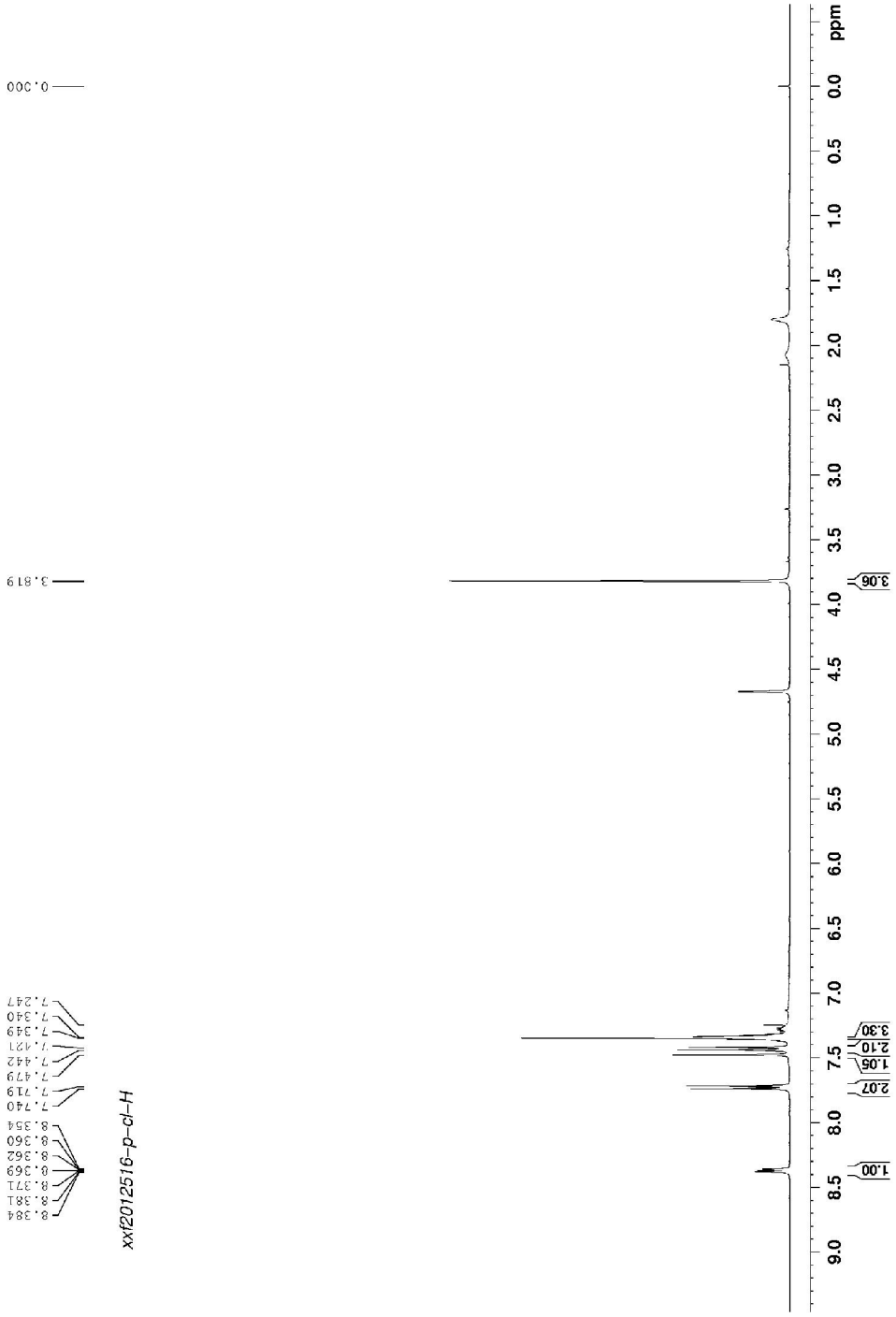
Cl
N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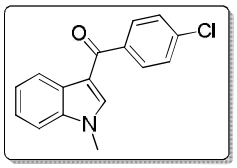
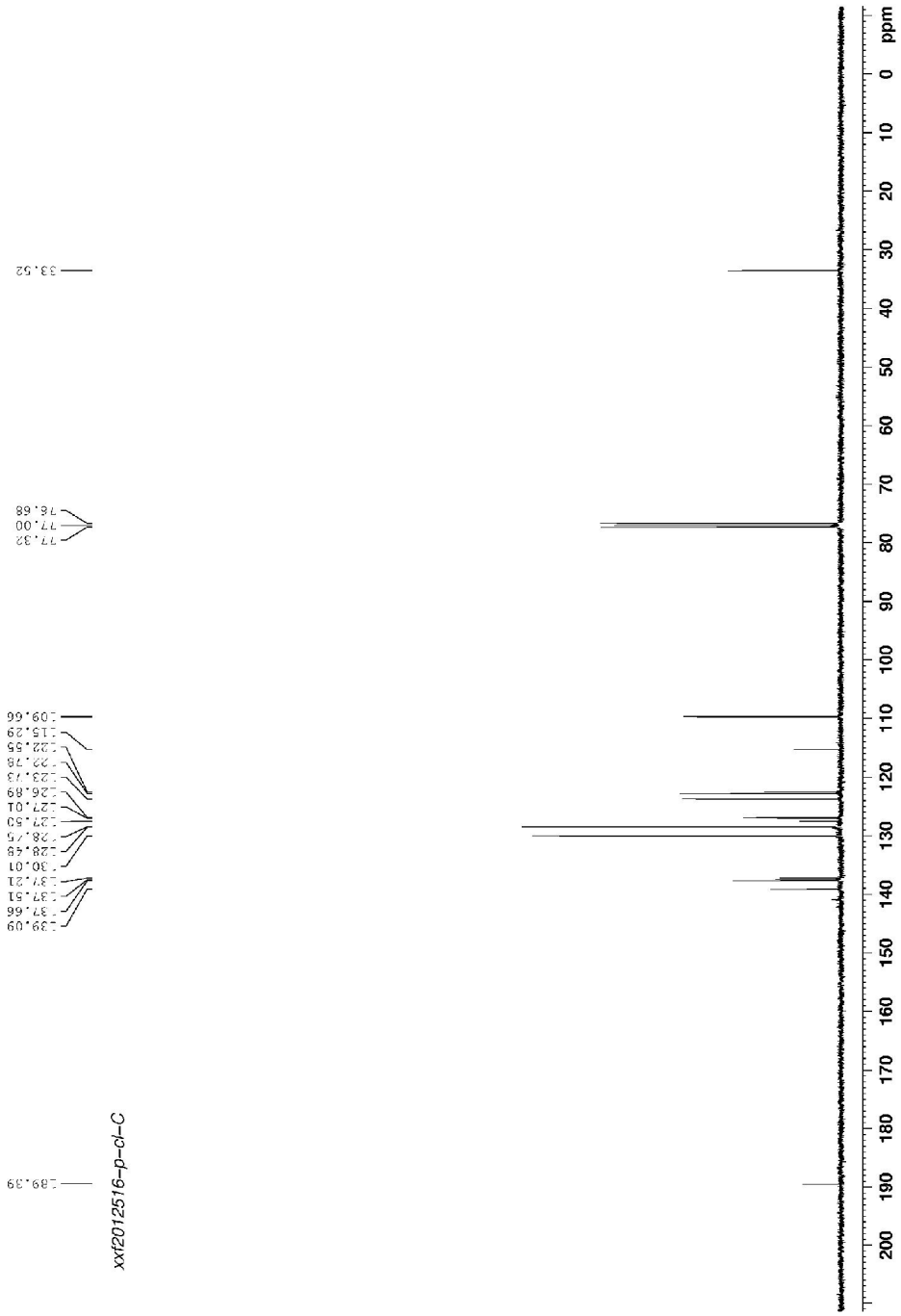
8.384
8.381
8.371
8.369
8.362
8.360
8.354
7.740
7.719
7.479
7.442
7.421
7.349
7.340
7.247
xxf2012516-p-cl-H
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
3.06
3.30
2.10
1.05
2.07
1.00


O
Cl
N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76.68
109.66
115.29
122.55
122.78
123.73
126.89
127.01
127.50
128.49
128.75
130.01
137.21
137.51
137.66
139.09
189.39
xxf20125l6-p-cl-C
ppm
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50
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70
80
90
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110
120
130
140
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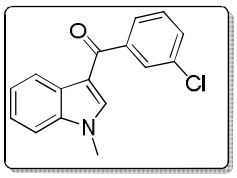
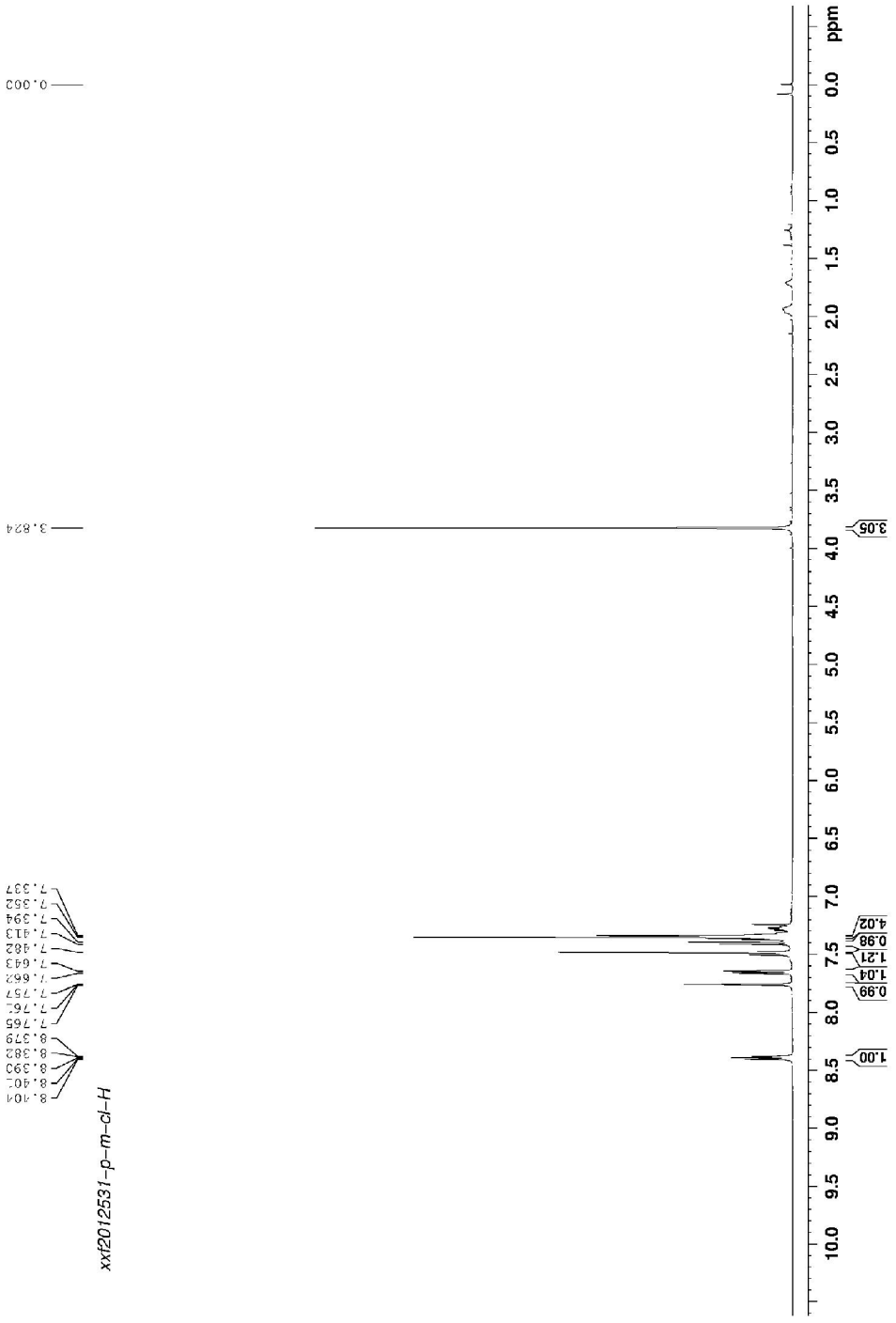
160
170
180
190
200


O
Cl
N


xxf20120531-p-m-cl-H
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7.482
7.413
7.394
7.352
7.337
3.824
0.000
ppm
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3.0
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4.5
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6.5
7.0
7.5
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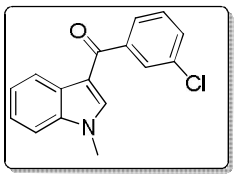
1.00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Cl
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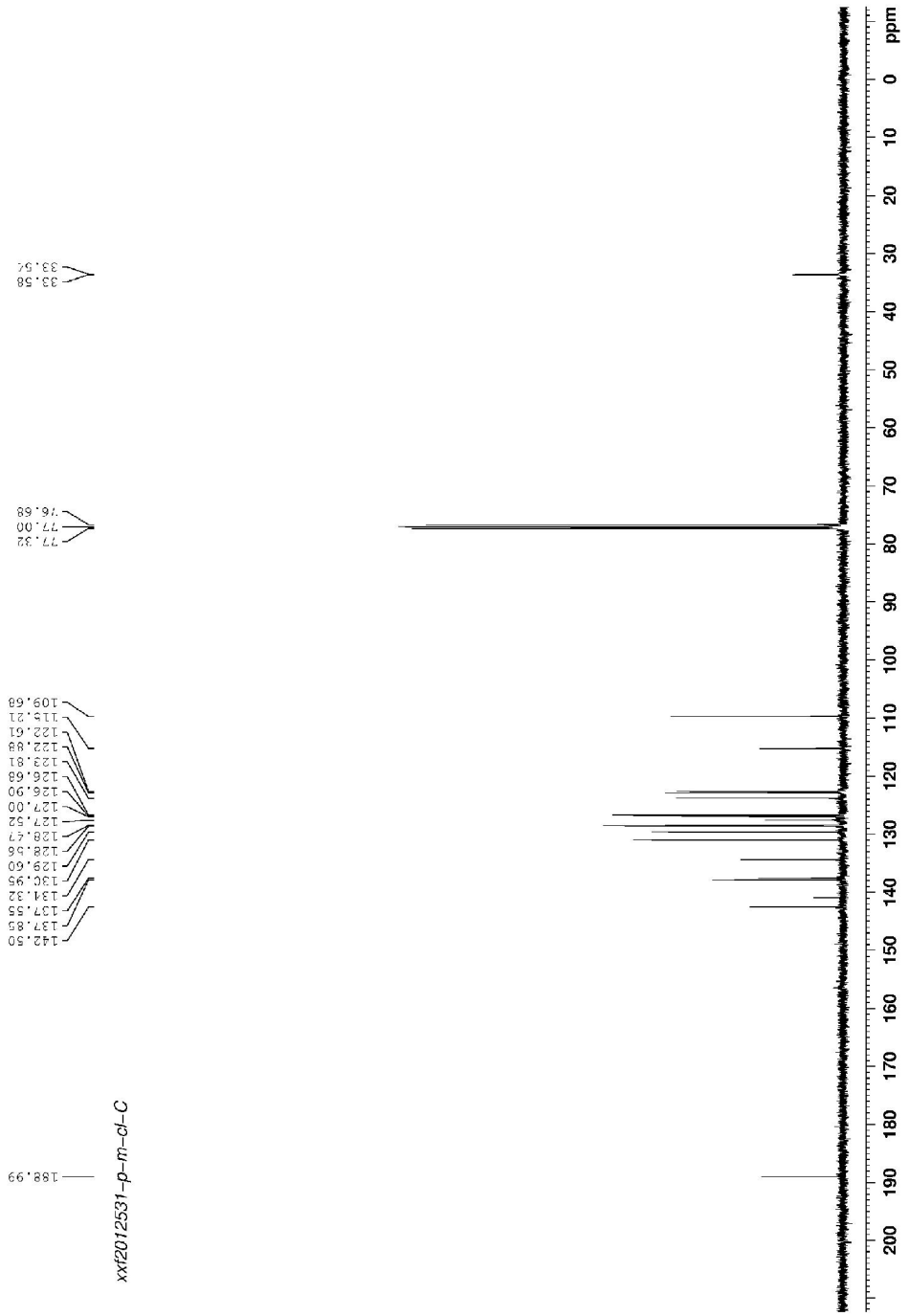
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126.90
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128.47
128.56
129.60
130.95
134.32
137.55
137.85
142.50
188.99
xxi20125531-p-m-cl-C
ppm
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50
60
70
80
90
100
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130
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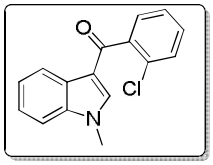
170
180
190
200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Cl
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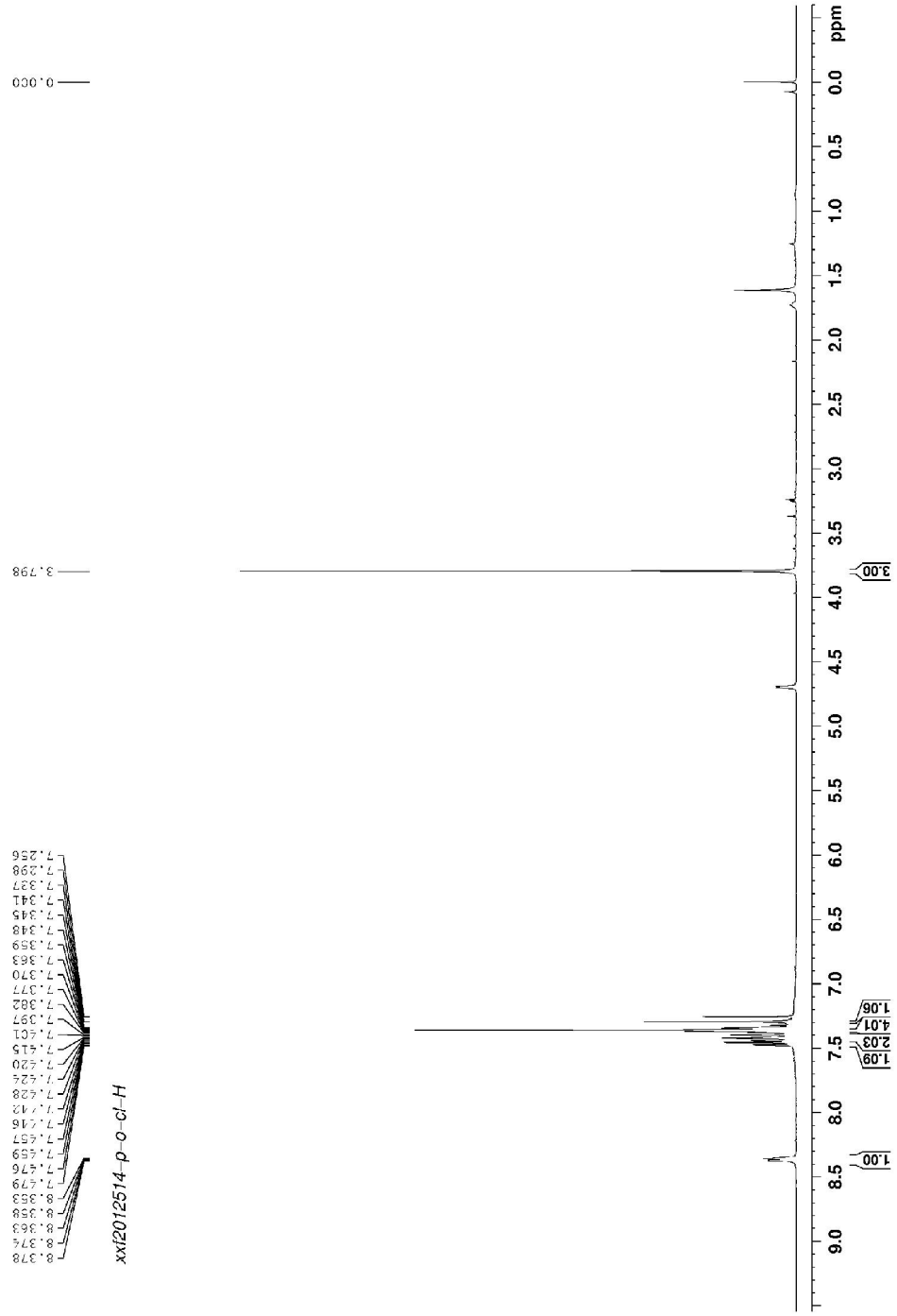
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7.377
7.382
7.397
7.401
7.415
7.420
7.424
7.428
7.443
7.446
7.457
7.459
7.476
7.479
8.353
8.358
8.363
8.374
8.378
xxi20120514_p-o-cl_H
ppm
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3.5
4.0
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5.0
5.5
6.0
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1.09
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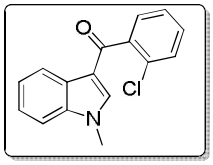
4.01
1.06
1.00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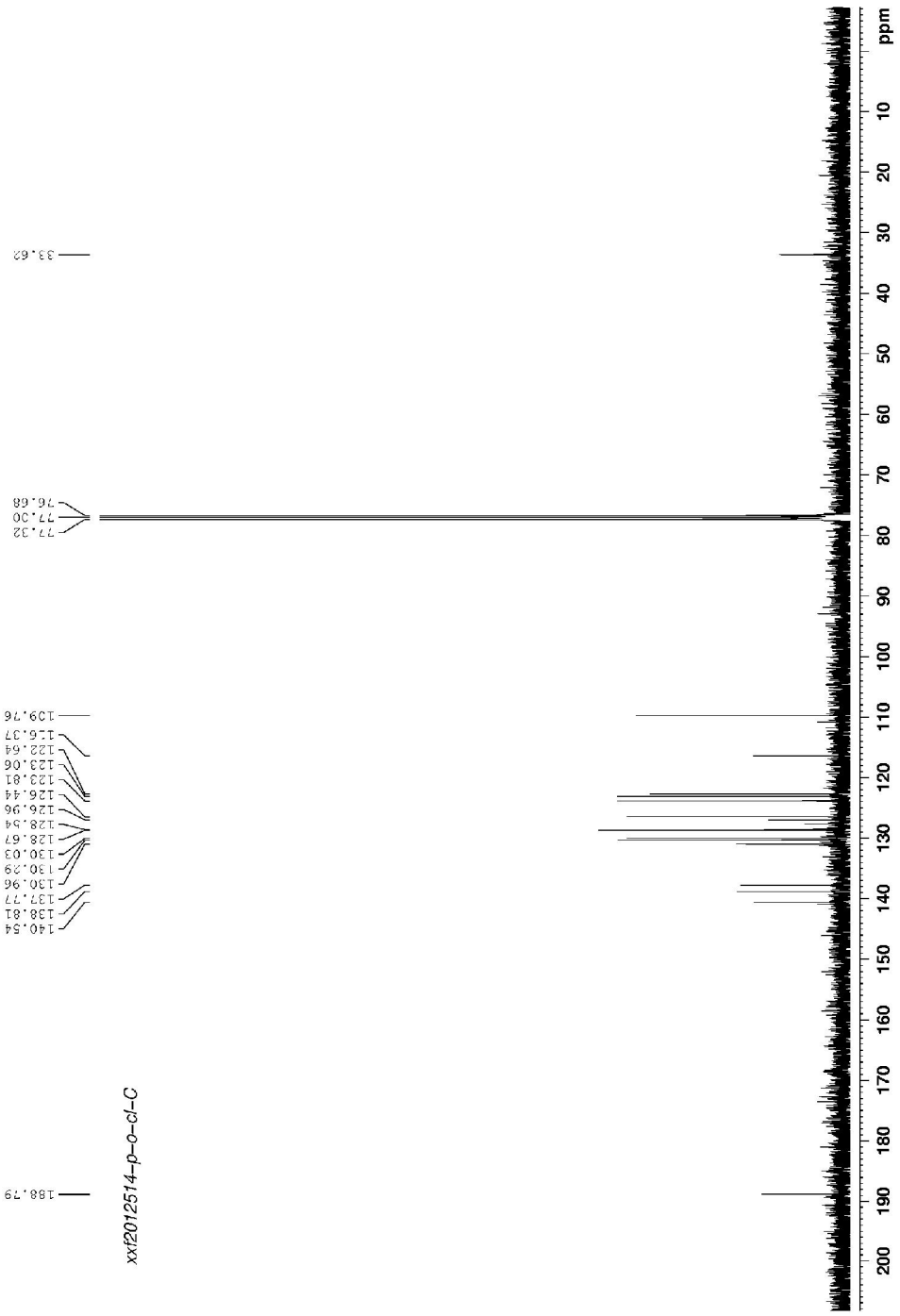
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123.06
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128.54
128.67
130.03
130.29
130.96
137.77
138.81
140.54
188.79
xxf2012514-p-o-cl-C
ppm
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50
60
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90
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130
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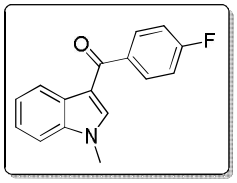
170
180
190
200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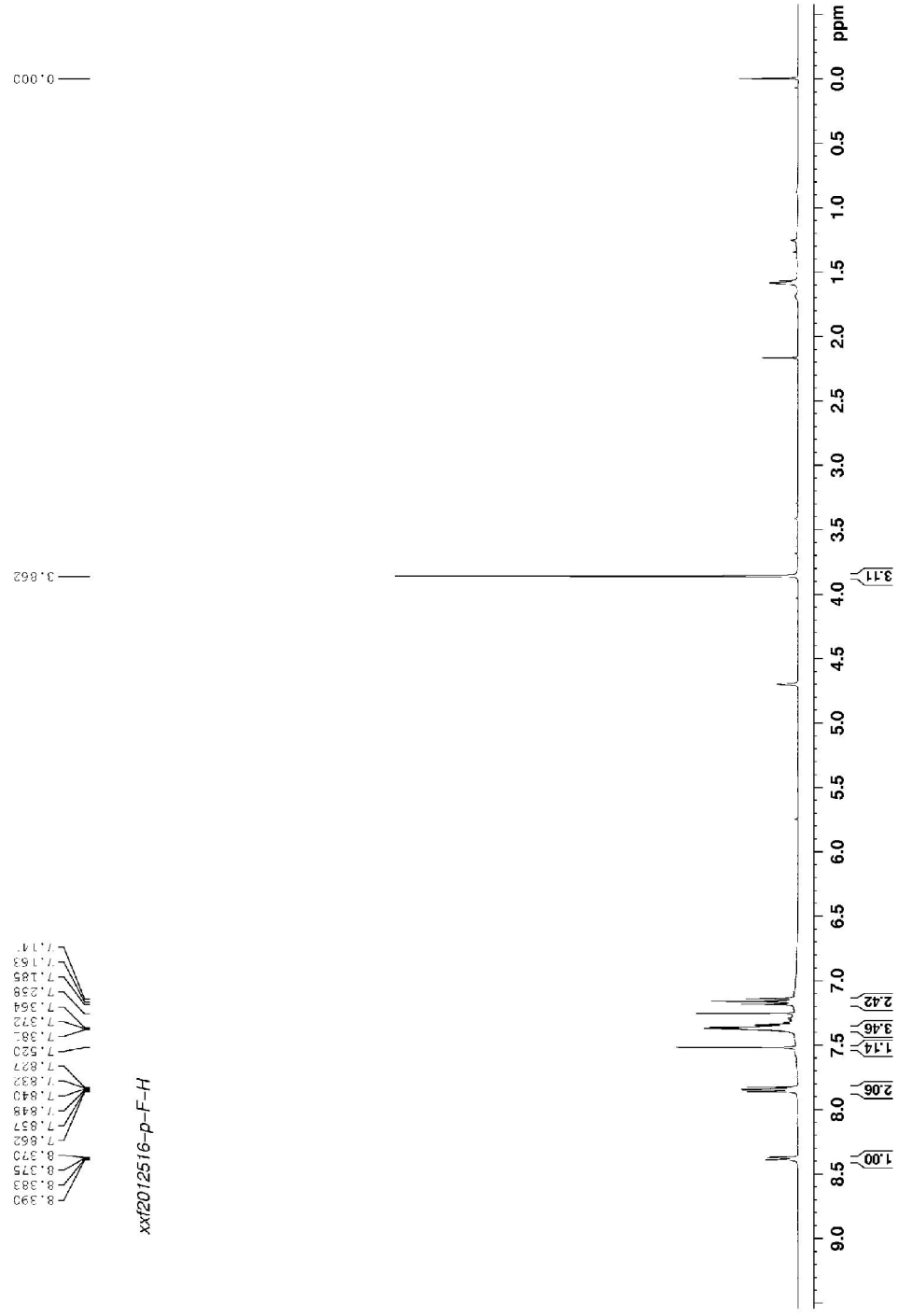
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7.857
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7.840
7.832
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7.520
7.381
7.372
7.364
7.238
7.185
7.163
7.147
xxf2012516-p-F-H
ppm
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2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
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8.0
8.5
9.0
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2.42
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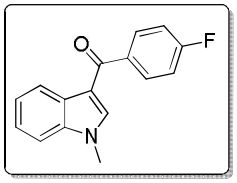
1.14
2.06
1.00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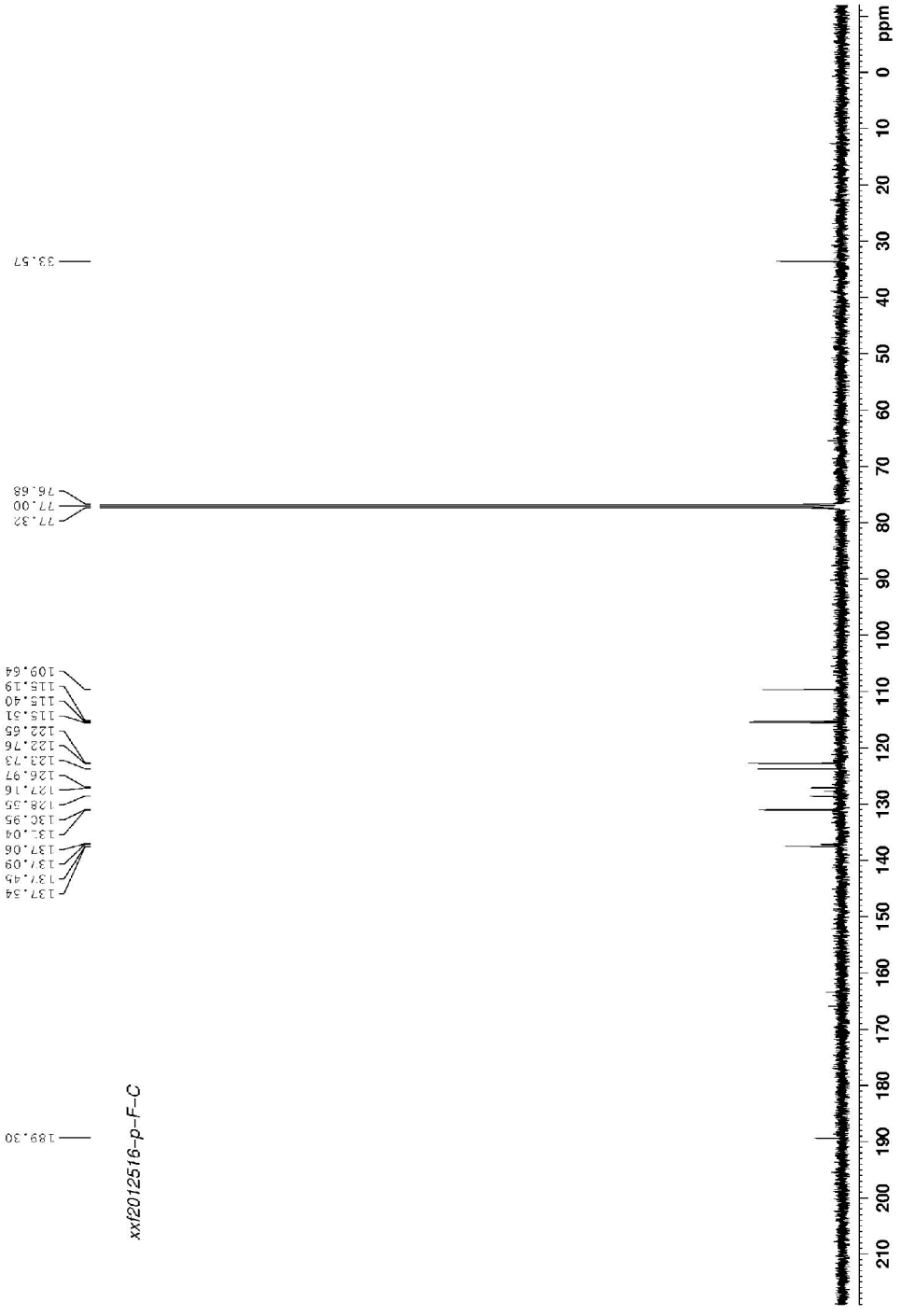
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115.51
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122.76
123.73
126.97
127.16
128.35
130.95
131.04
137.06
137.09
137.45
137.54
189.30
xxf2012516-p-F-C
ppm
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60
70
80
90
100
110
120
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140
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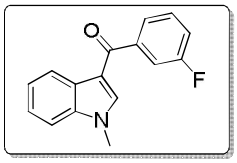
170
180
190
200
210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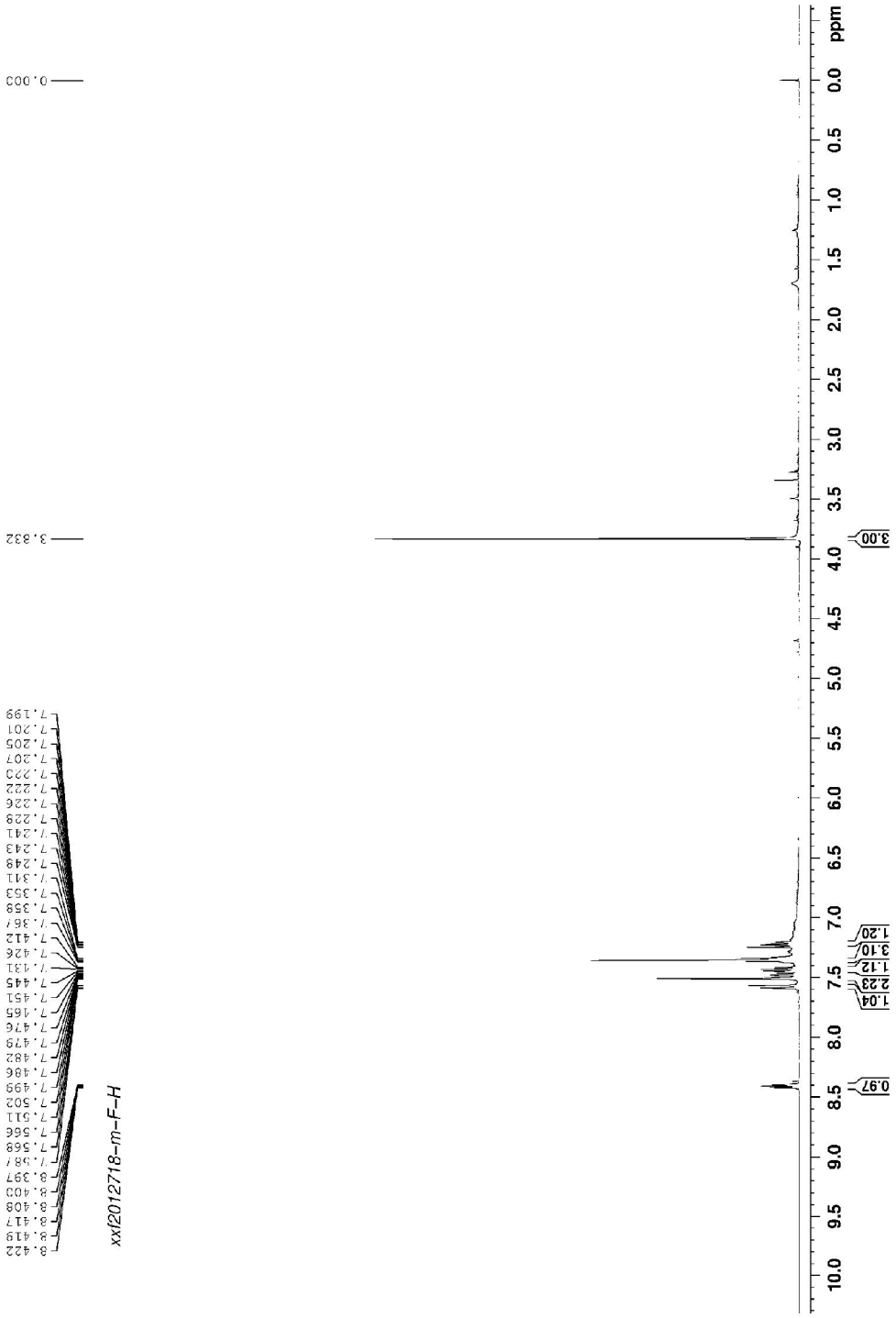
F
N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7.222
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7.229
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7.249
7.311
7.353
7.358
7.367
7.412
7.426
7.431
7.445
7.451
7.465
7.476
7.479
7.482
7.486
7.499
7.502
7.511
7.566
7.568
7.587
8.397
8.403
8.408
8.417
8.419
8.422
xxf2012718-m-F-H
ppm
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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
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9.0
9.5
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3.10
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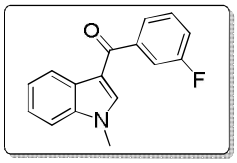
2.23
1.04
0.97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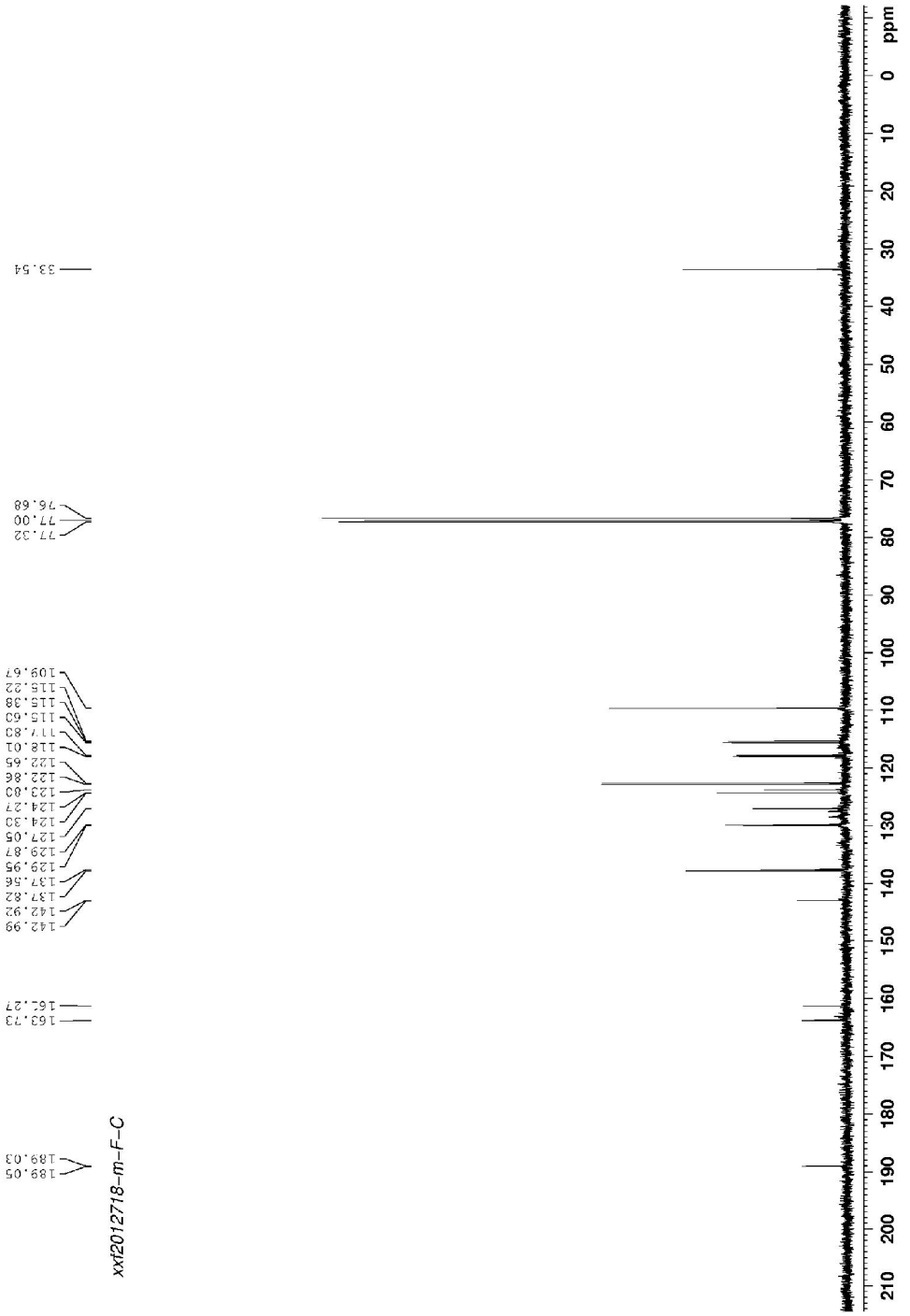
F
N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118.01
122.65
122.86
123.80
124.27
124.30
127.05
129.87
129.95
137.56
137.82
142.92
142.99
162.27
163.73
189.03
189.05
xxf20127l8-m-F-C
ppm
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60
70
80
90
100
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180
190
200
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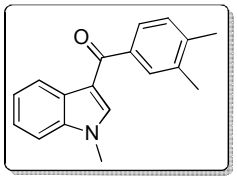
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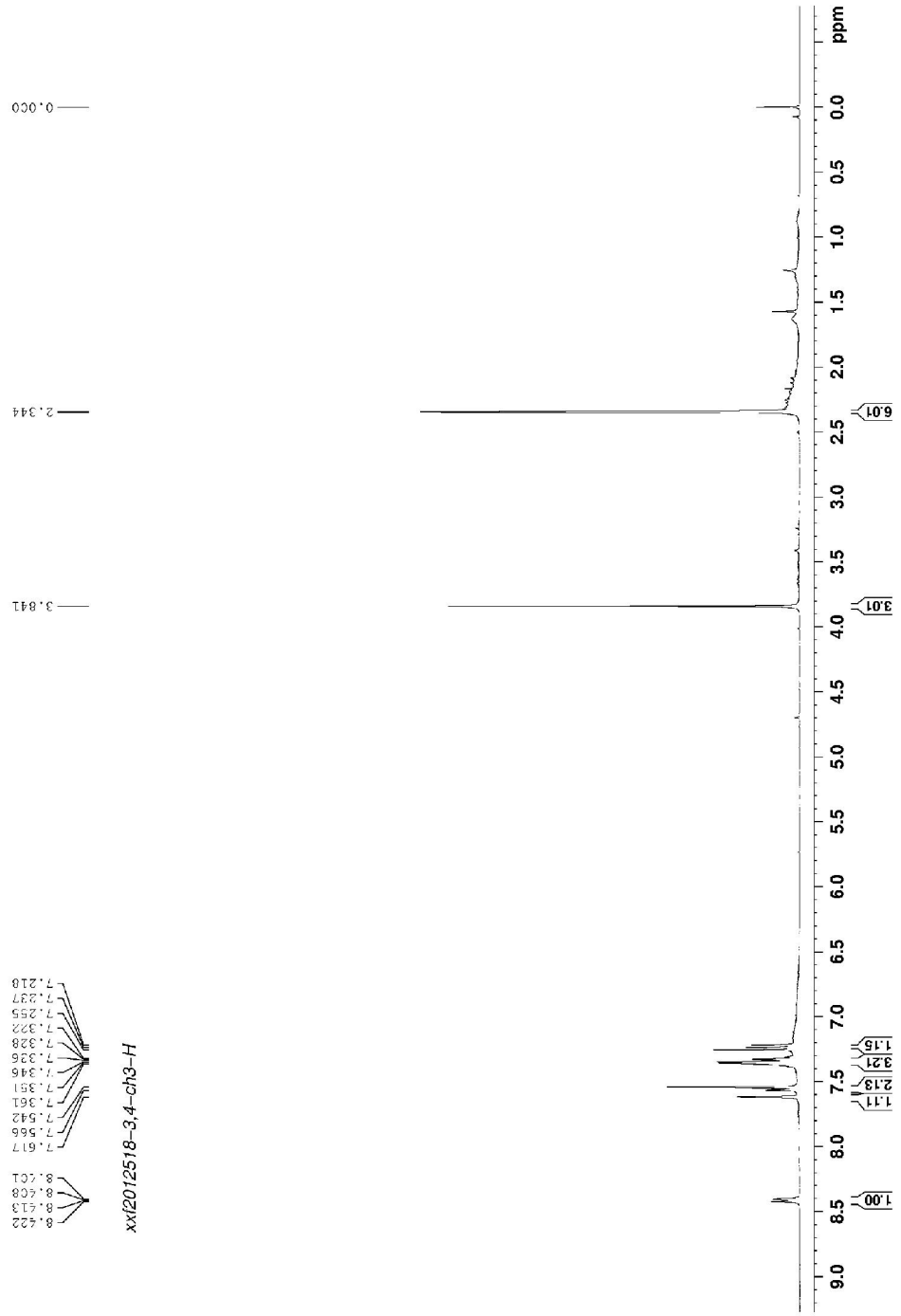
N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8.401
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7.542
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7.346
7.336
7.328
7.322
7.255
7.237
7.218
xxf20120518-3,4-ch3-H
ppm
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0.5
1.0
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
8.5
9.0
6.01
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1.75
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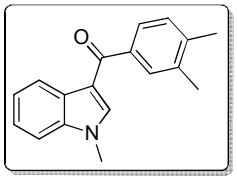
2.13
1.11
1.00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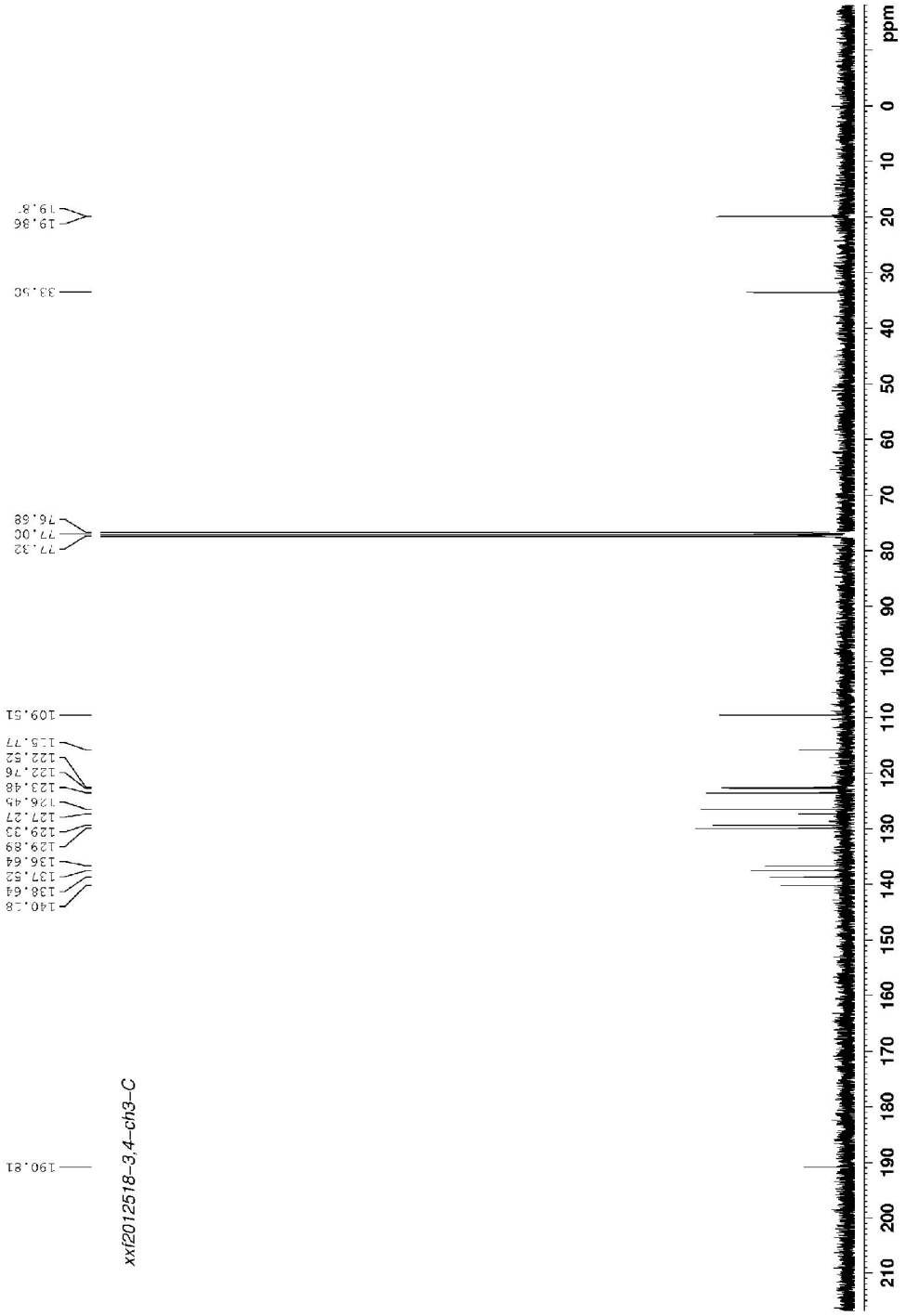
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77.32
109.51
115.77
122.52
122.76
123.48
126.45
127.27
129.33
129.89
136.64
137.52
138.64
140.18
190.81
xxf2012518-3,4-ch3-C
ppm
0
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60
70
80
90
100
110
120
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140
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200
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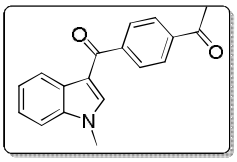

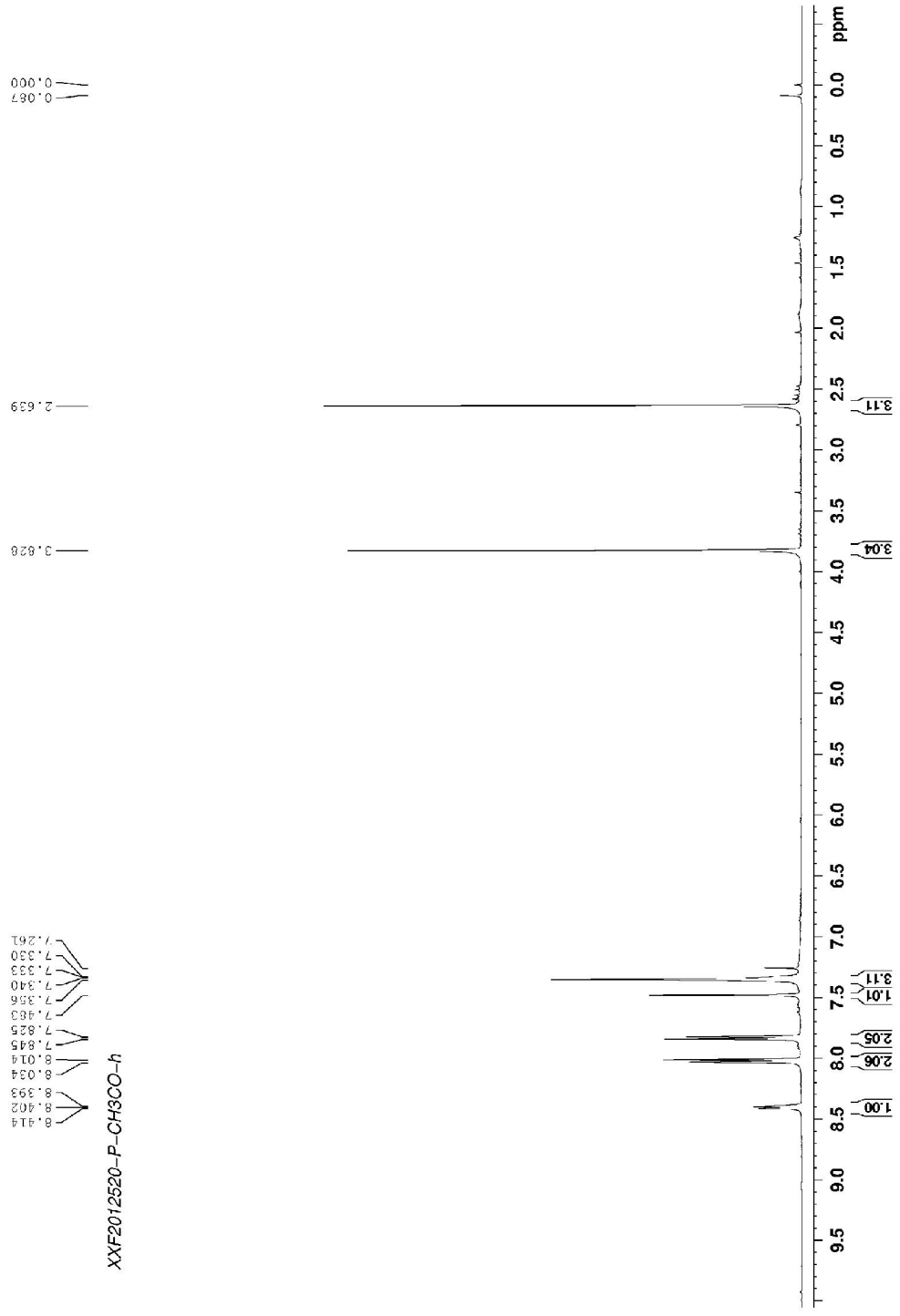
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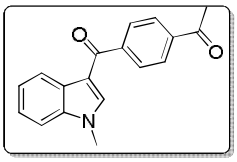
2.05
1.00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O
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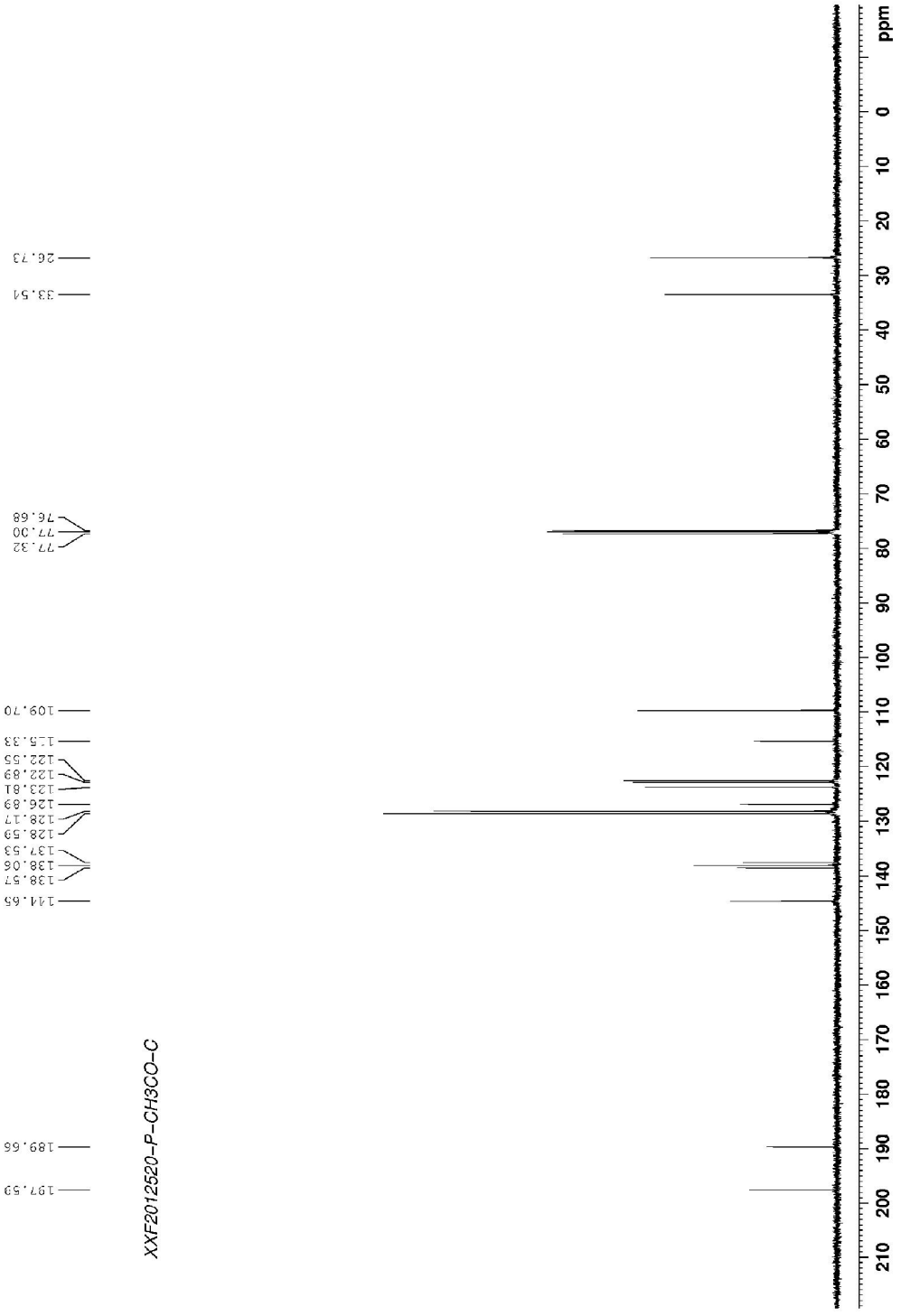
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122.55
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128.59
137.53
138.06
138.57
144.65
189.66
197.59
XXF2012520-P-CH3CO-C
ppm
0
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60
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80
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100
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200
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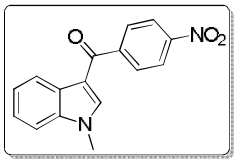
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NO2
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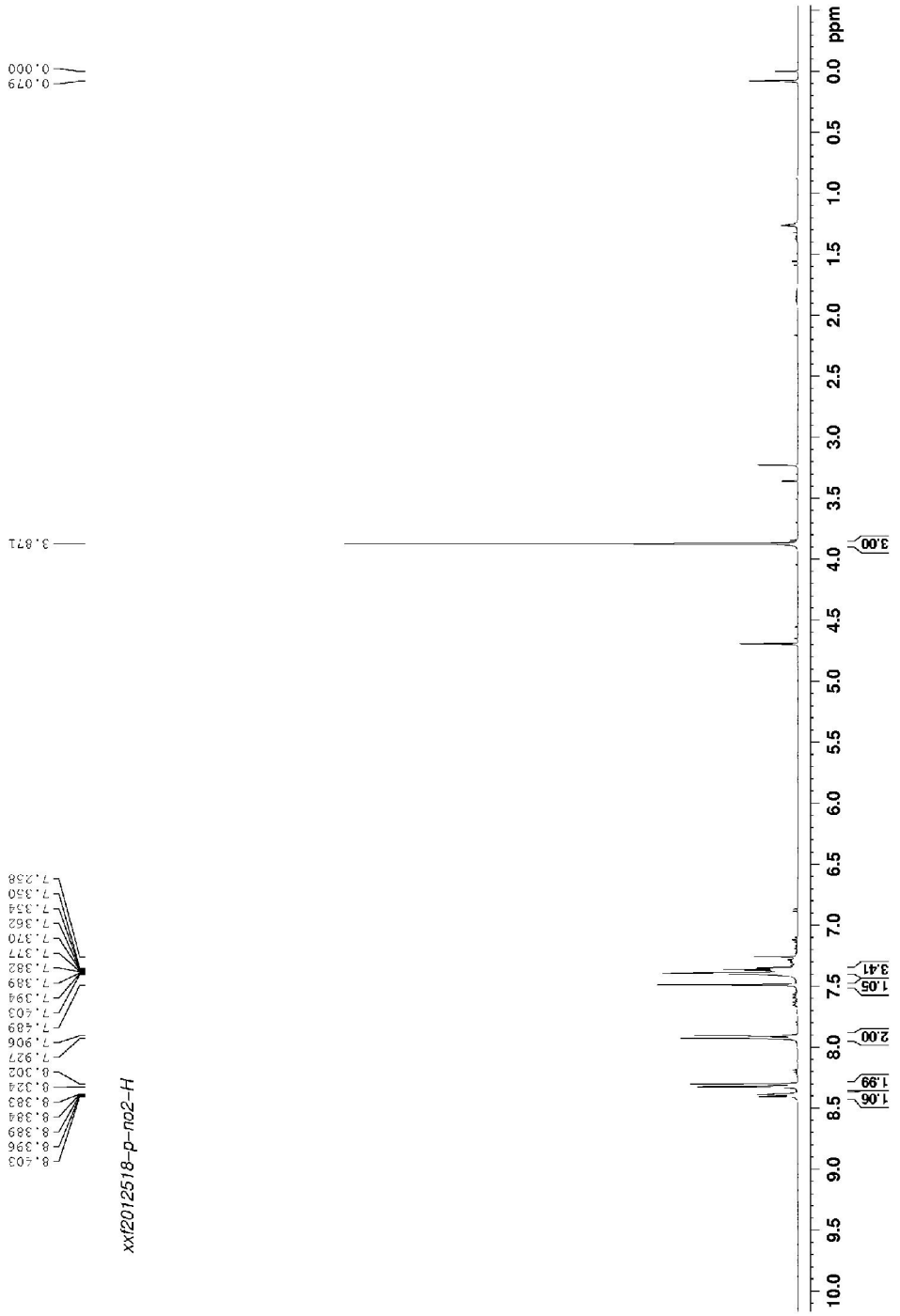
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7.377
7.382
7.389
7.394
7.403
7.409
7.906
7.927
8.302
8.324
8.383
8.384
8.389
8.396
8.403
xxf20125l8-p-no2-H
ppm
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1.0
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2.5
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3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
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3.41
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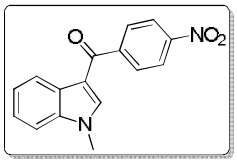
1.06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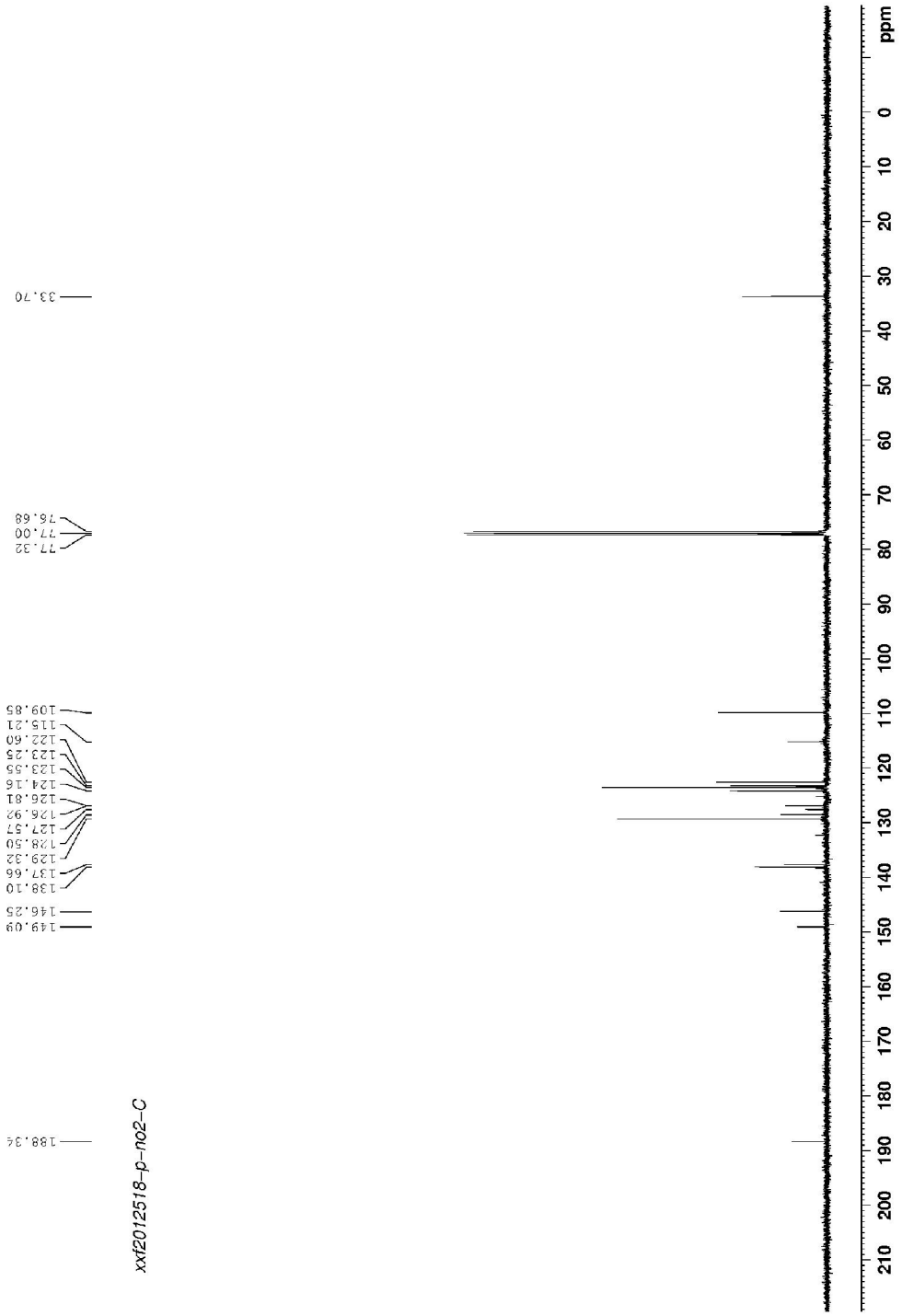
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124.16
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126.92
127.57
128.50
129.32
137.66
138.10
146.25
149.09
188.34
xxf2012518-p-no2-C
ppm
0
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80
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100
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140
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180
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200
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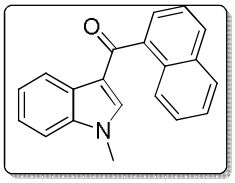
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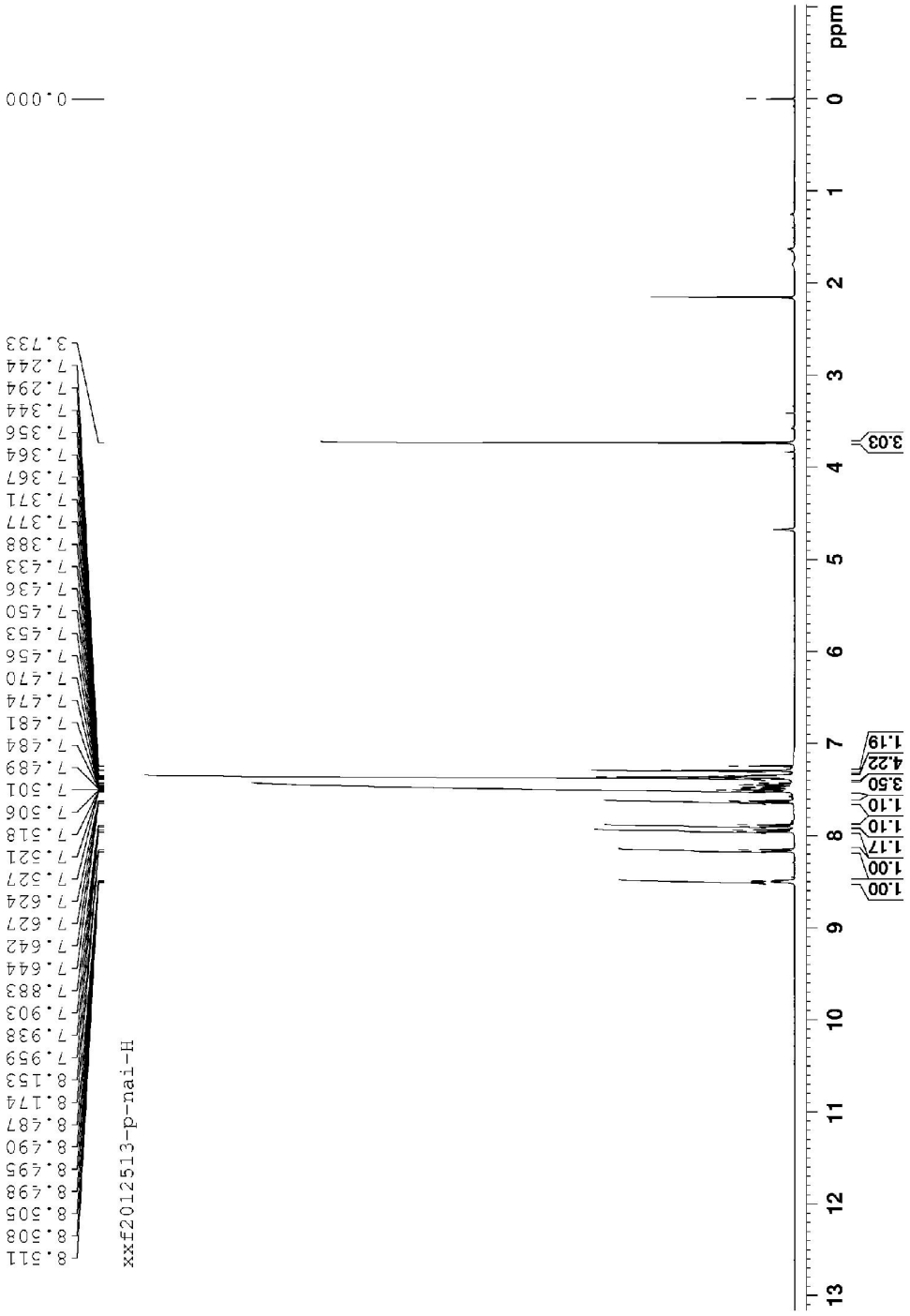
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10
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1.17
1.10
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1.19
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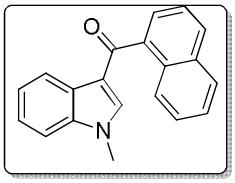
xxf2012513-p-nai-H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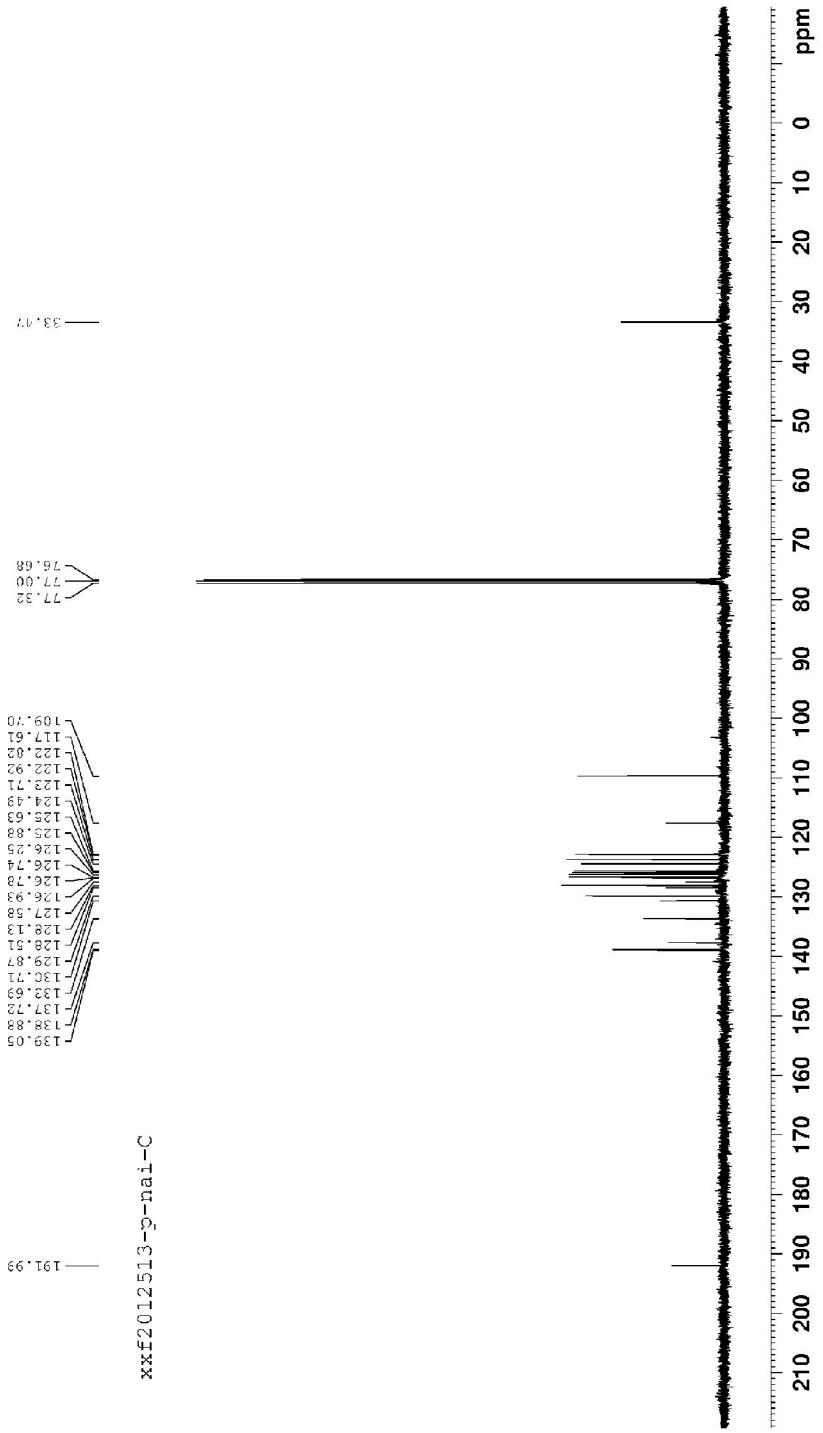
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128.13
128.51
129.87
130.71
133.69
137.72
138.88
139.05
191.99
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ppm
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60
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80
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200
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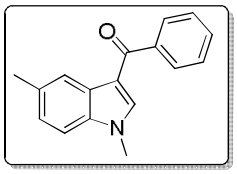

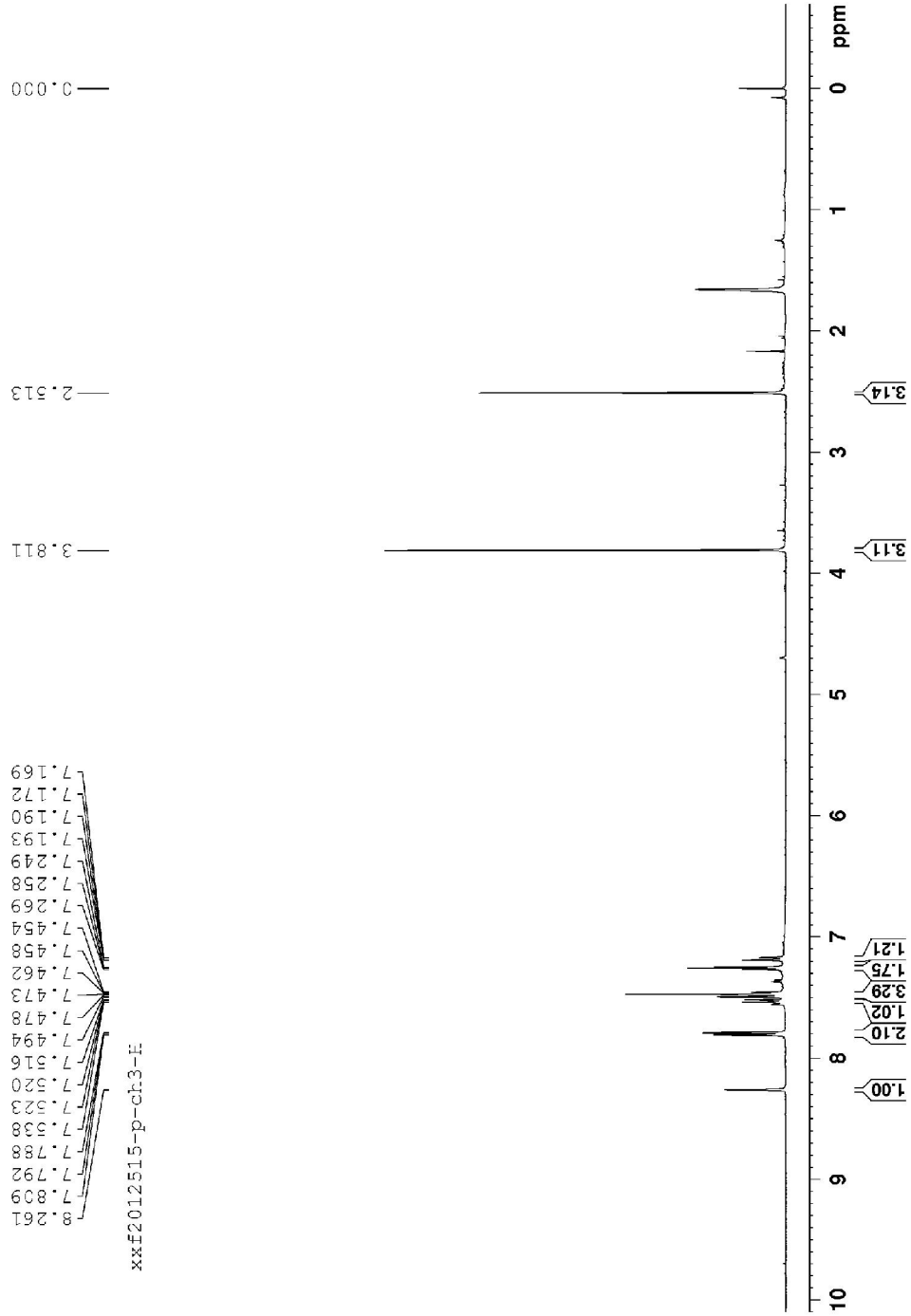
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7
8
9
10
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1.21
1.75
3.29
1.02
2.10
1.00
0.000
2.513
3.811
xxf2012515-p-ch3-E
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7.172
7.190
7.193
7.249
7.258
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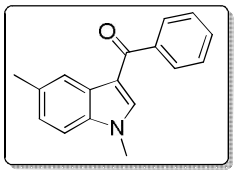
8.261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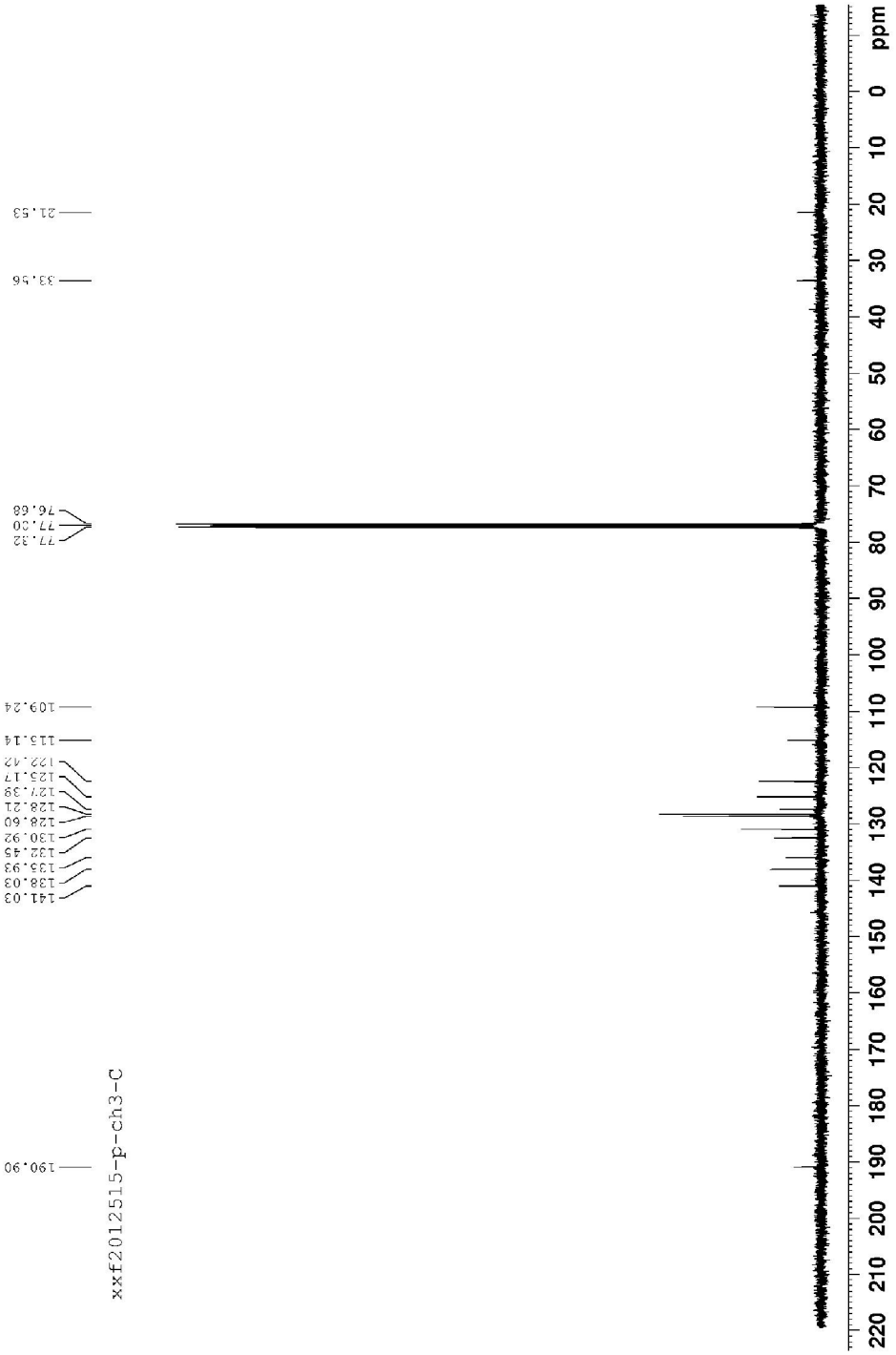
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115.14
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127.39
128.21
128.60
130.92
132.45
135.93
138.03
141.03
190.90
xxf2012515-p-ch3-C
ppm
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10
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30
40
50
60
70
80
90
100
110
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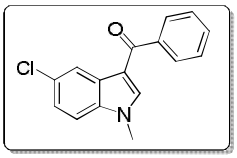
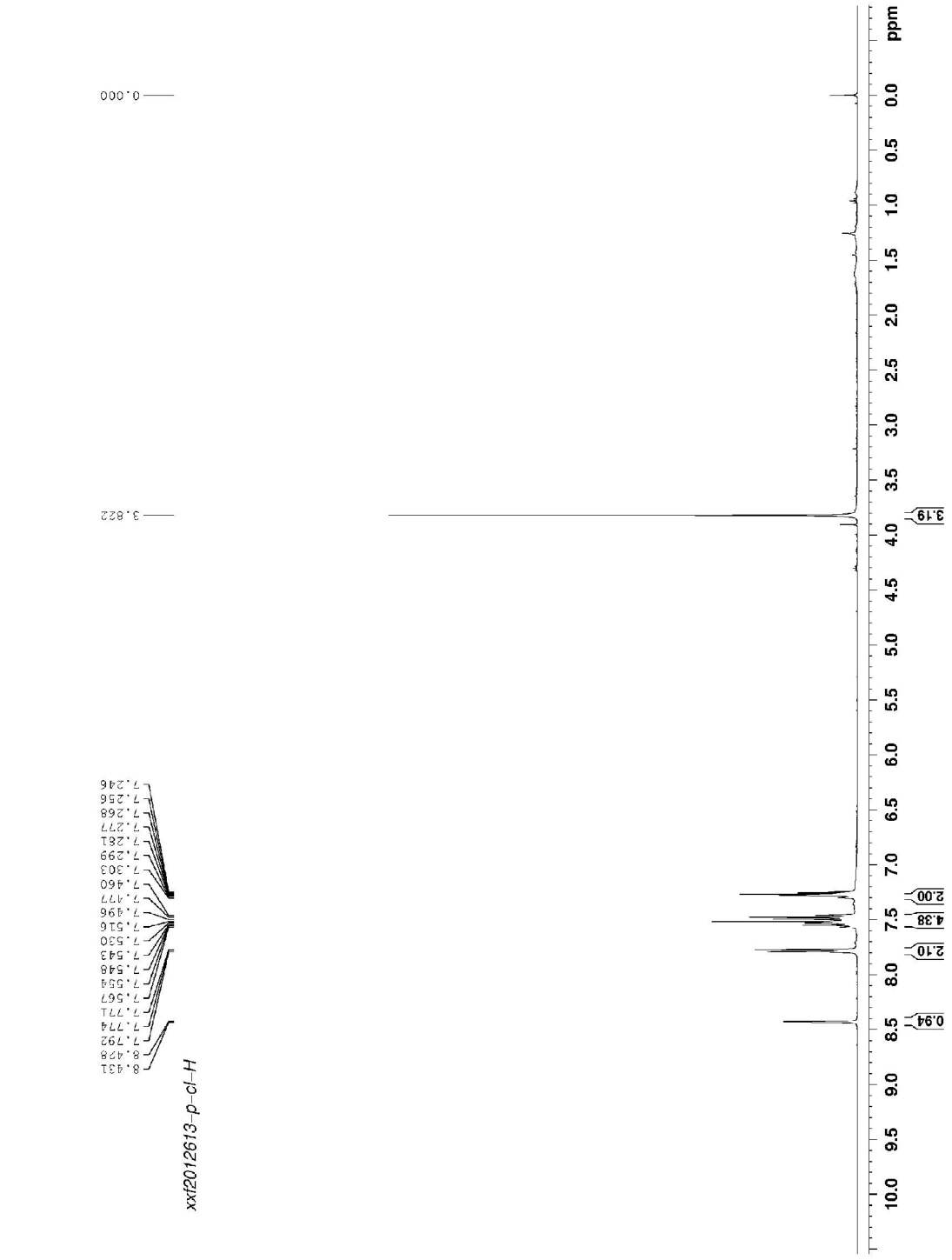
220


O
Cl
N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7.277
7.281
7.299
7.303
7.460
7.477
7.496
7.516
7.530
7.543
7.548
7.554
7.567
7.771
7.774
7.792
8.478
8.431
xxf2012613-p-cl-H
ppm
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1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
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8.5
9.0
9.5
10.0
3.19
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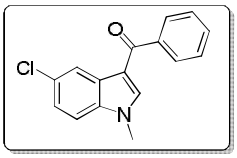
4.38
2.10
0.94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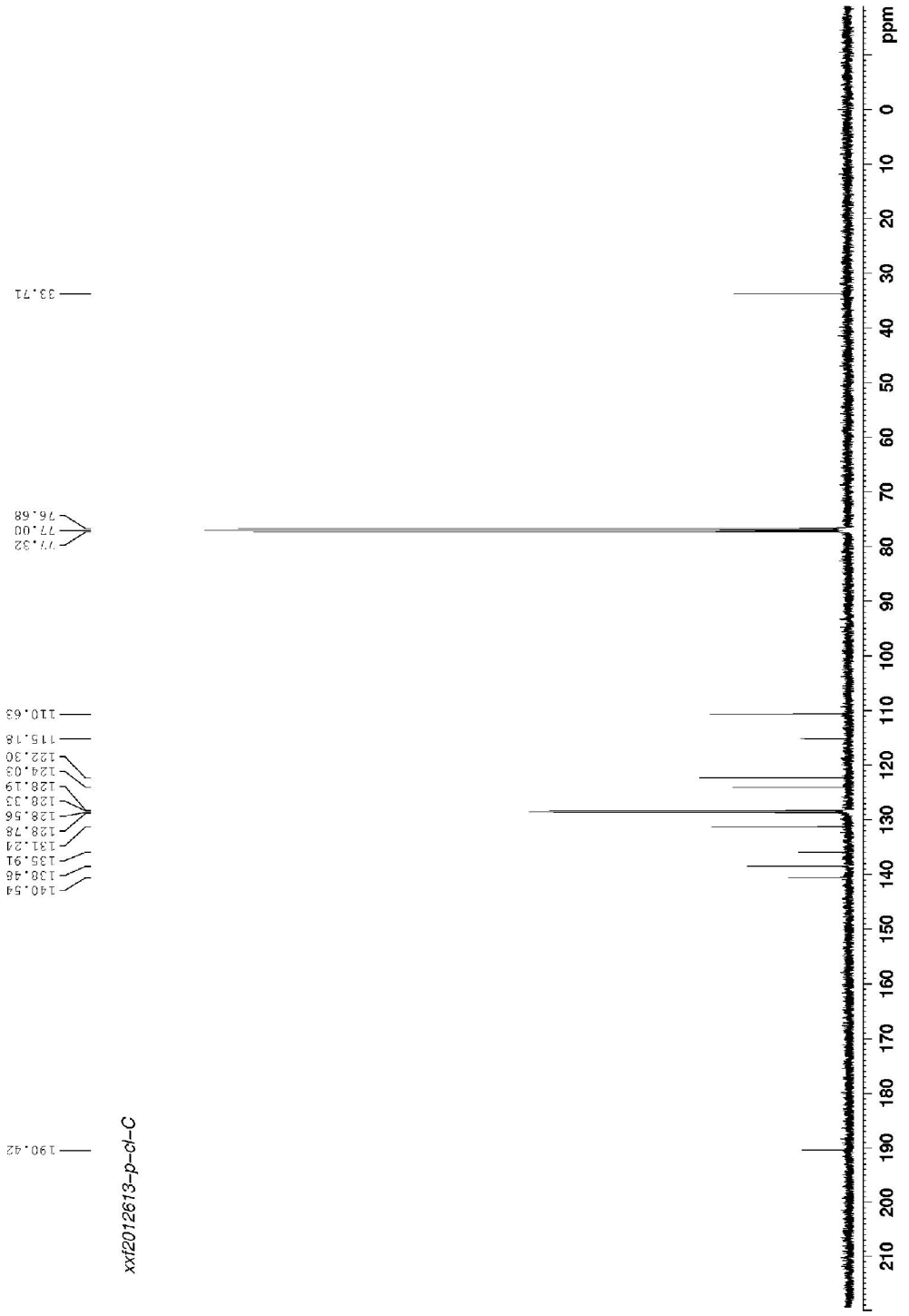
Cl
N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128.33
128.56
128.78
131.24
135.91
138.46
140.54
190.42
xxf2012613-p-cl-C
ppm
0
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30
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60
70
80
90
100
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140
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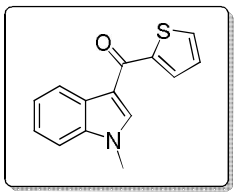
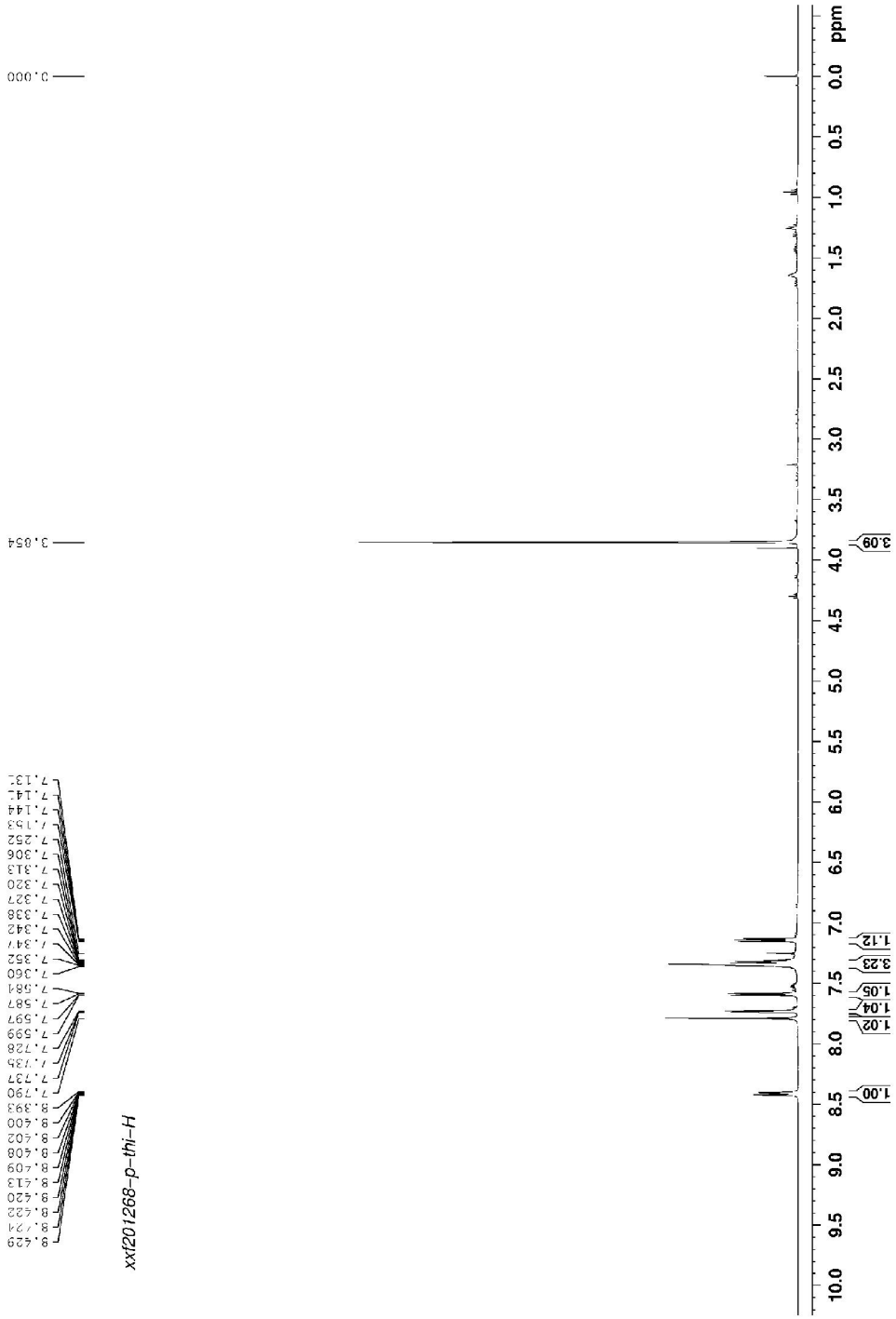
180
190
200
210


O
S
N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3.054
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7.141
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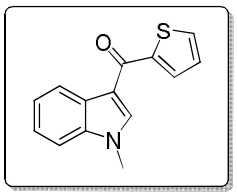
7.153
7.252
7.306
7.313
7.320
7.327
7.338
7.342
7.347
7.352
7.360
7.584
7.587
7.597
7.599
7.728
7.735
7.737
7.790
8.393
8.400
8.402
8.408
8.409
8.413
8.420
8.422
8.794
8.429
xxf201268-p-thi-H
ppm
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
3.09
1.12
3.23
1.05
1.04
1.02
1.00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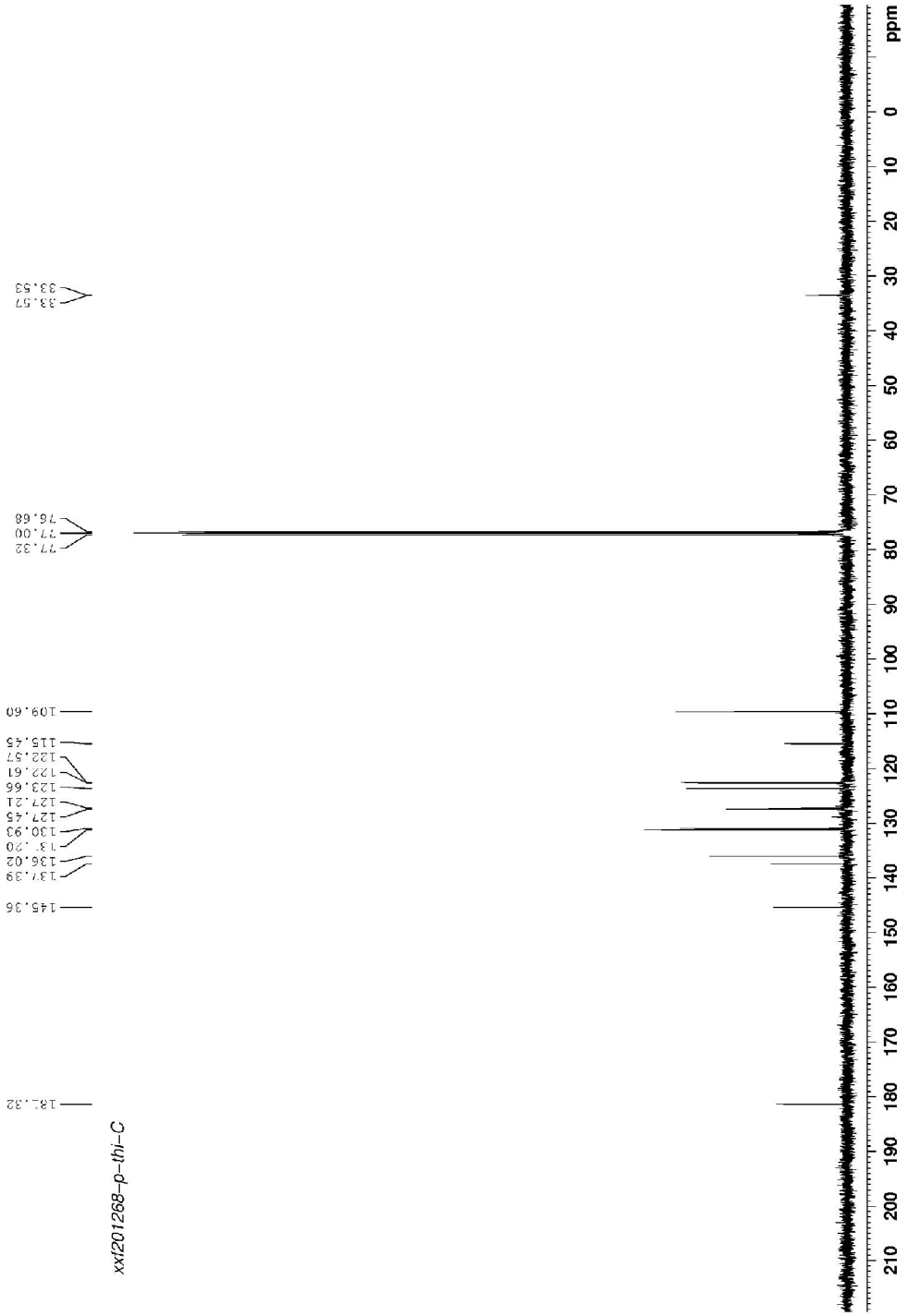
S
N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115.45
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123.66
127.21
127.45
130.93
136.20
136.02
137.39
145.36
182.32
xxf201268-p-thi-C
ppm
0
10
20
30
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50
60
70
80
90
100
110
120
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140
150
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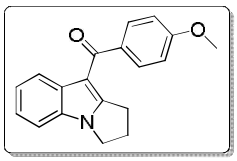
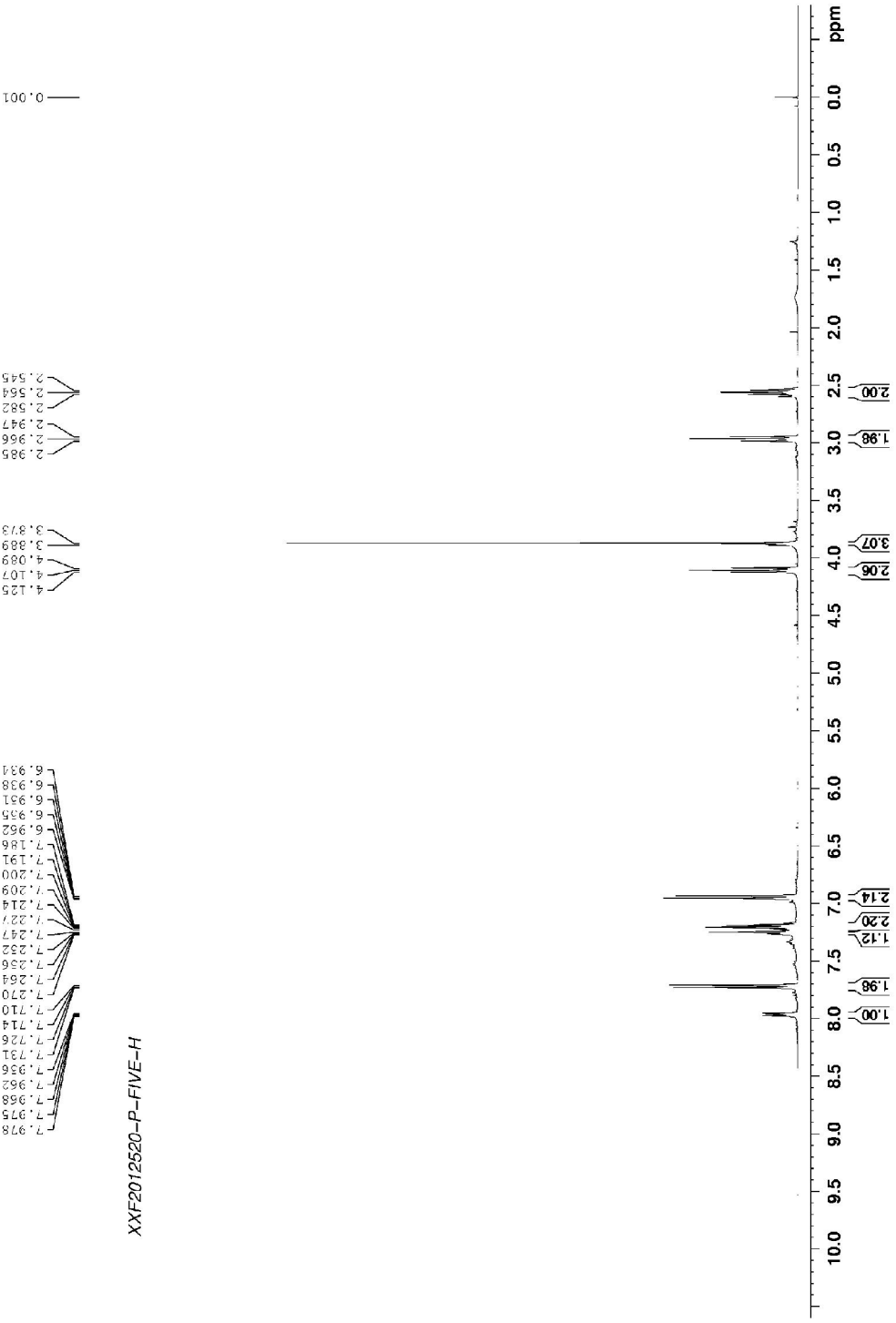
180
190
200
210




XXF2012520-P-FIVE-H
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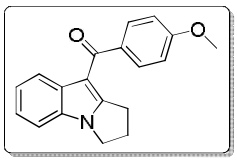
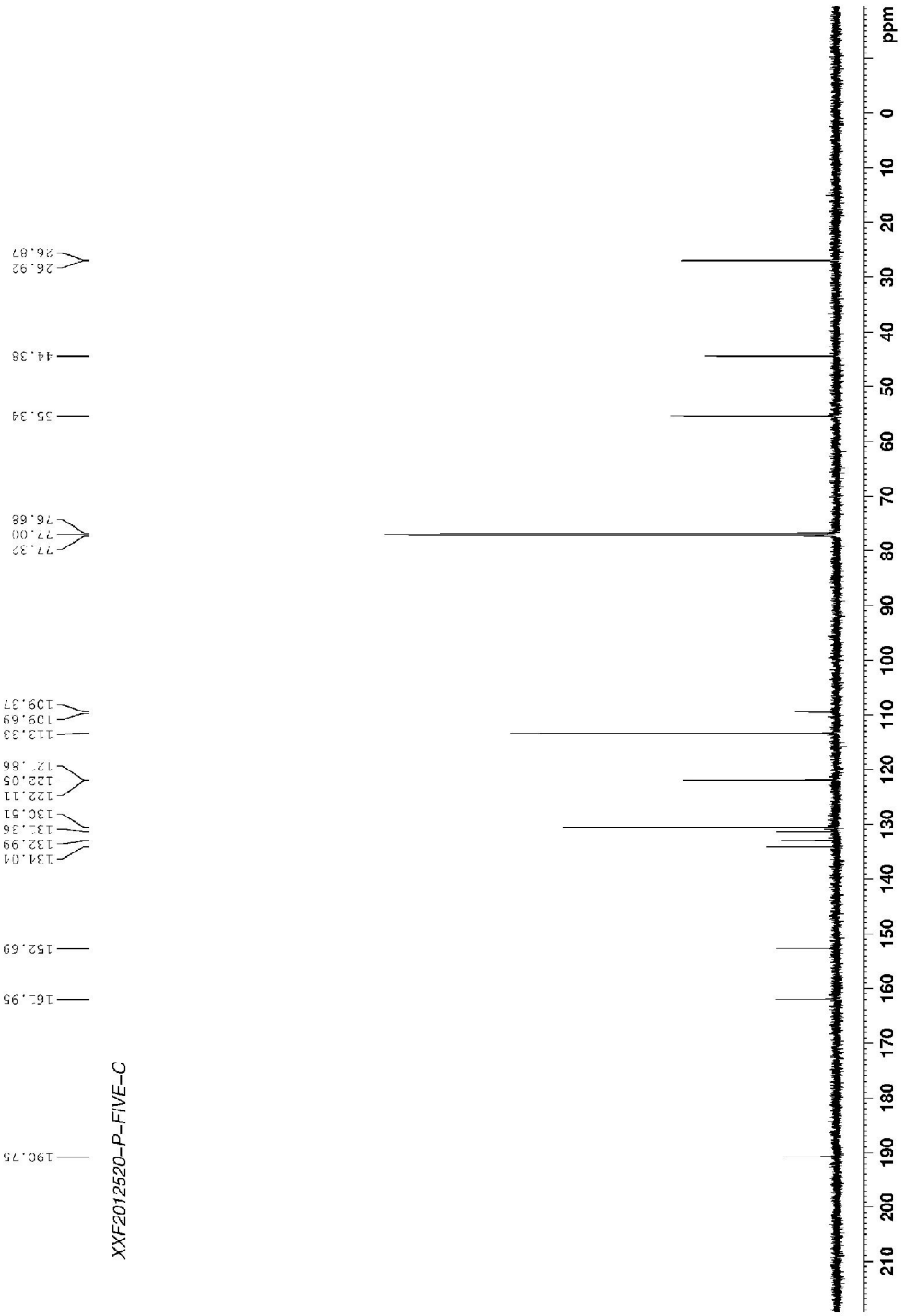
2.06
2.14
2.20
1.12
1.98
1.00

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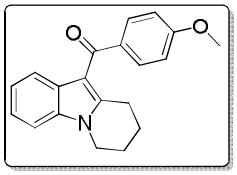
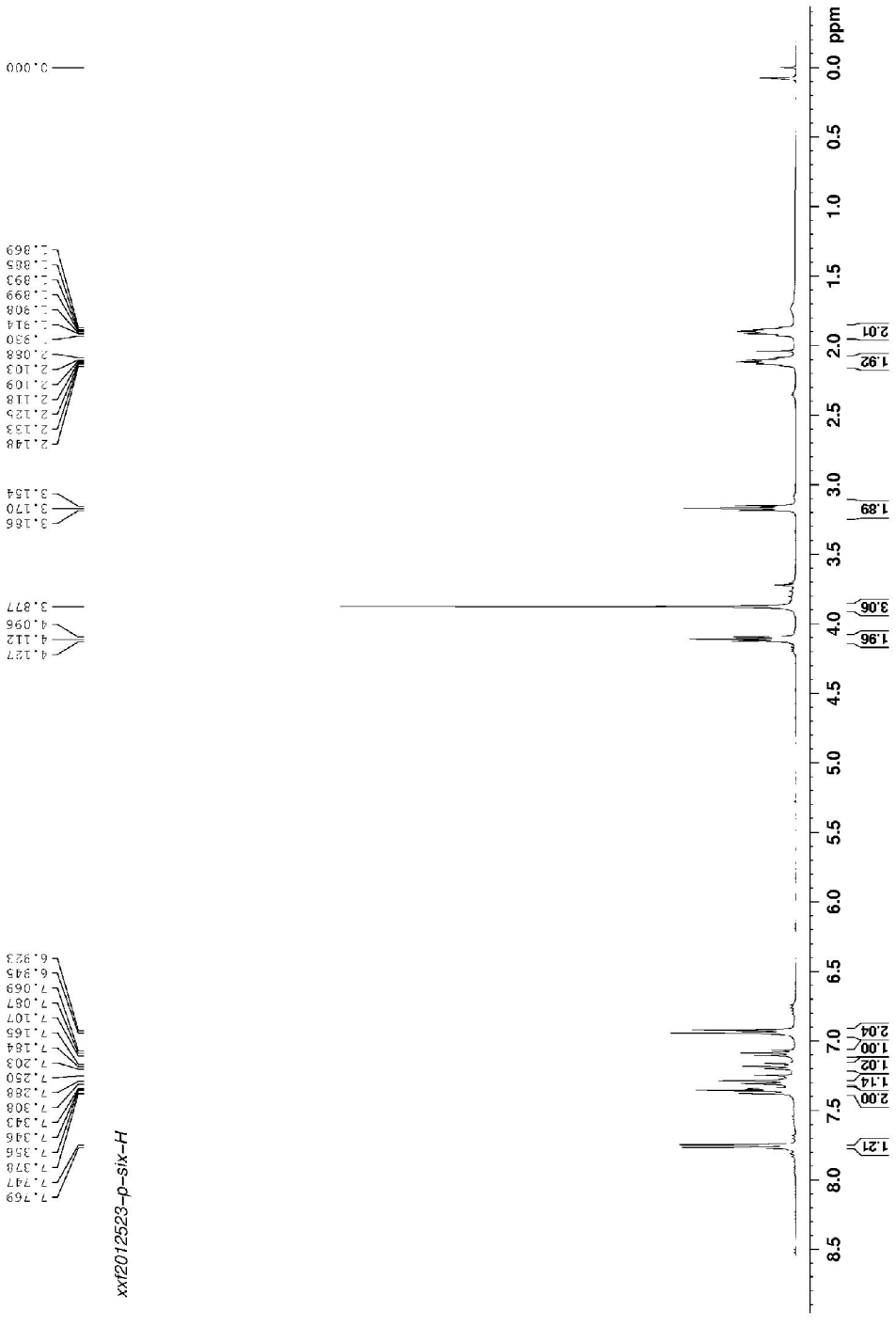
96.87
44.38
55.34
76.68
77.00
77.32
109.37
109.69
113.33
122.05
122.11
127.86
130.51
134.04
132.99
136.36
152.69
160.95
190.75
XXF2012520-P-FIVE-C
ppm

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7.356
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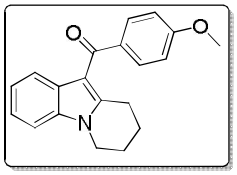
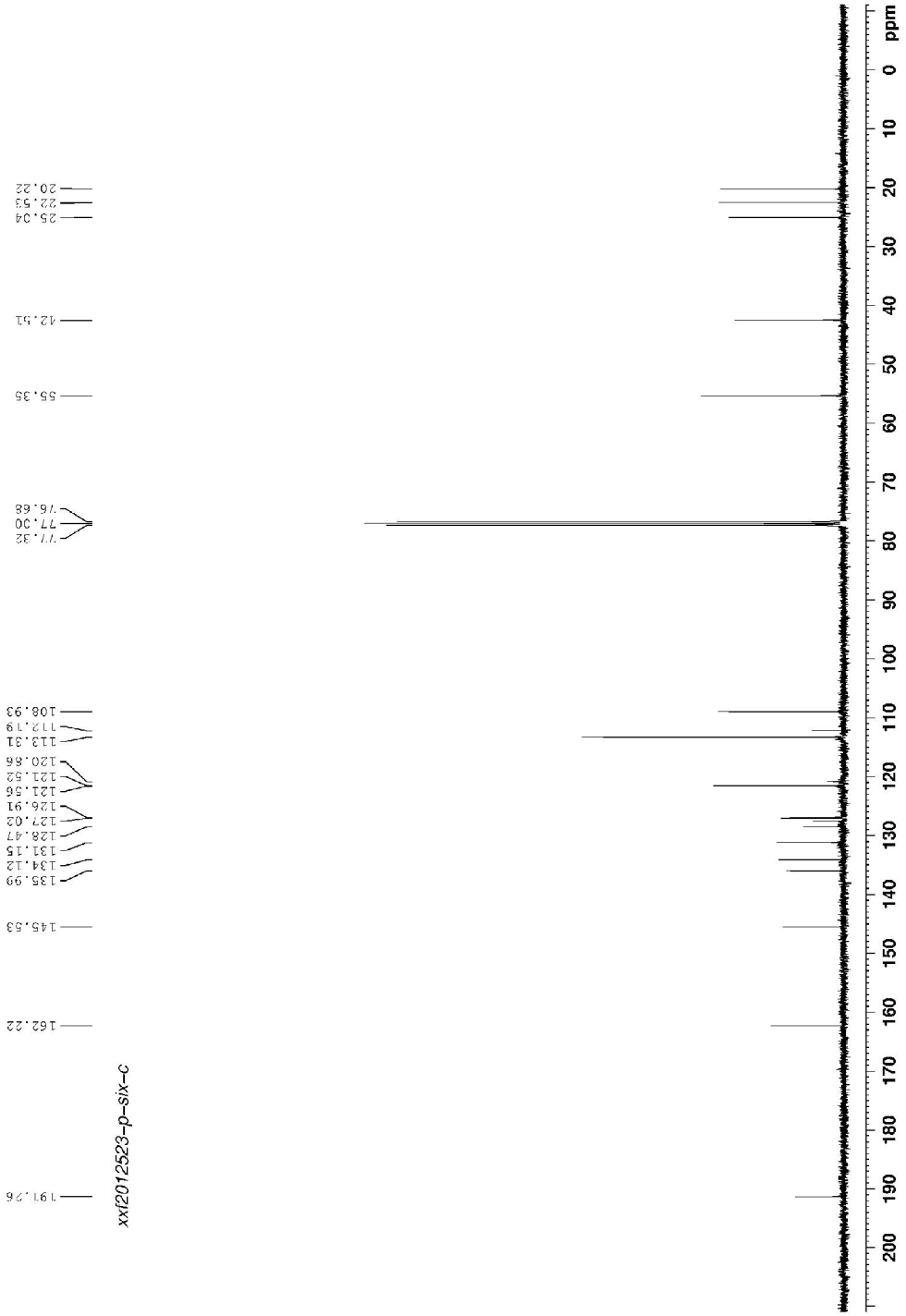
7.343
7.308
7.288
7.250
7.203
7.184
7.165
7.107
7.087
7.069
6.945
6.923
4.127
4.112
4.096
3.877
3.186
3.170
3.154
2.148
2.133
2.125
2.118
2.109
2.103
2.088
1.930
1.914
1.908
1.899
1.893
1.885
1.869
2.01
1.92
1.89
3.06
1.96
2.04
1.00
1.02
1.14
2.00
1.21
ppm

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70
80
90
100
110
120
130
140
150
160
170
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200
20.22
22.53
25.04
42.51
55.35
76.68
77.00
77.32
108.93
112.79
113.31
120.86
121.52
121.56
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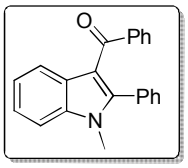
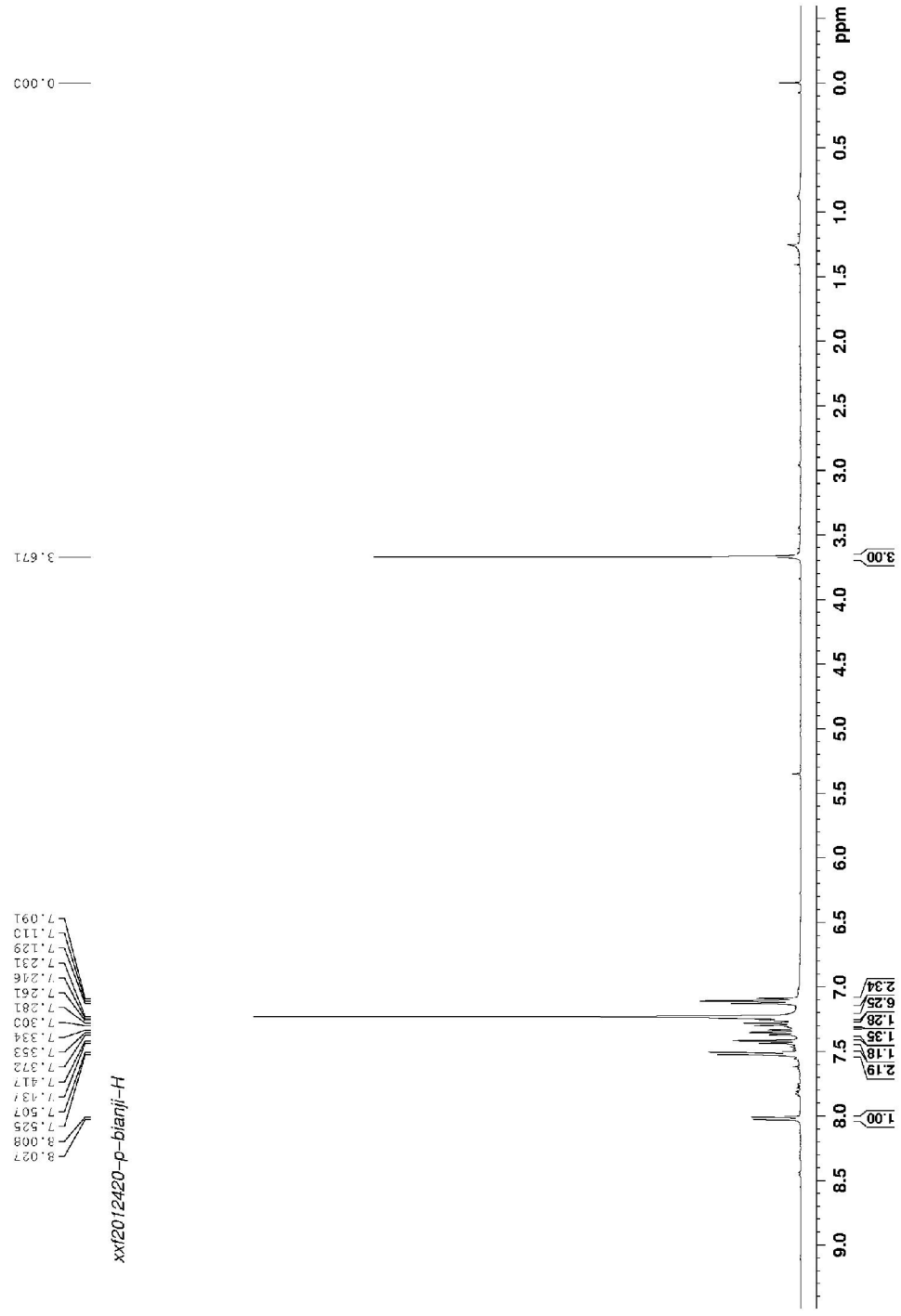
128.47
131.15
134.12
135.99
145.53
162.22
191.76
xxl2012523-p-six-c


O
Ph
Ph
N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4.0
4.5
5.0
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7.5
8.0
8.5
9.0
3.00
2.34
6.25
1.26
1.35
1.18
2.19
1.00
0.003
3.671
8.027
8.008
7.525
7.507
7.437
7.417
7.372
7.353
7.334
7.303
7.281
7.261
7.216
7.231
7.129
7.113
7.091
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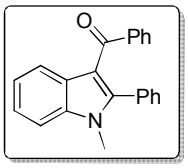
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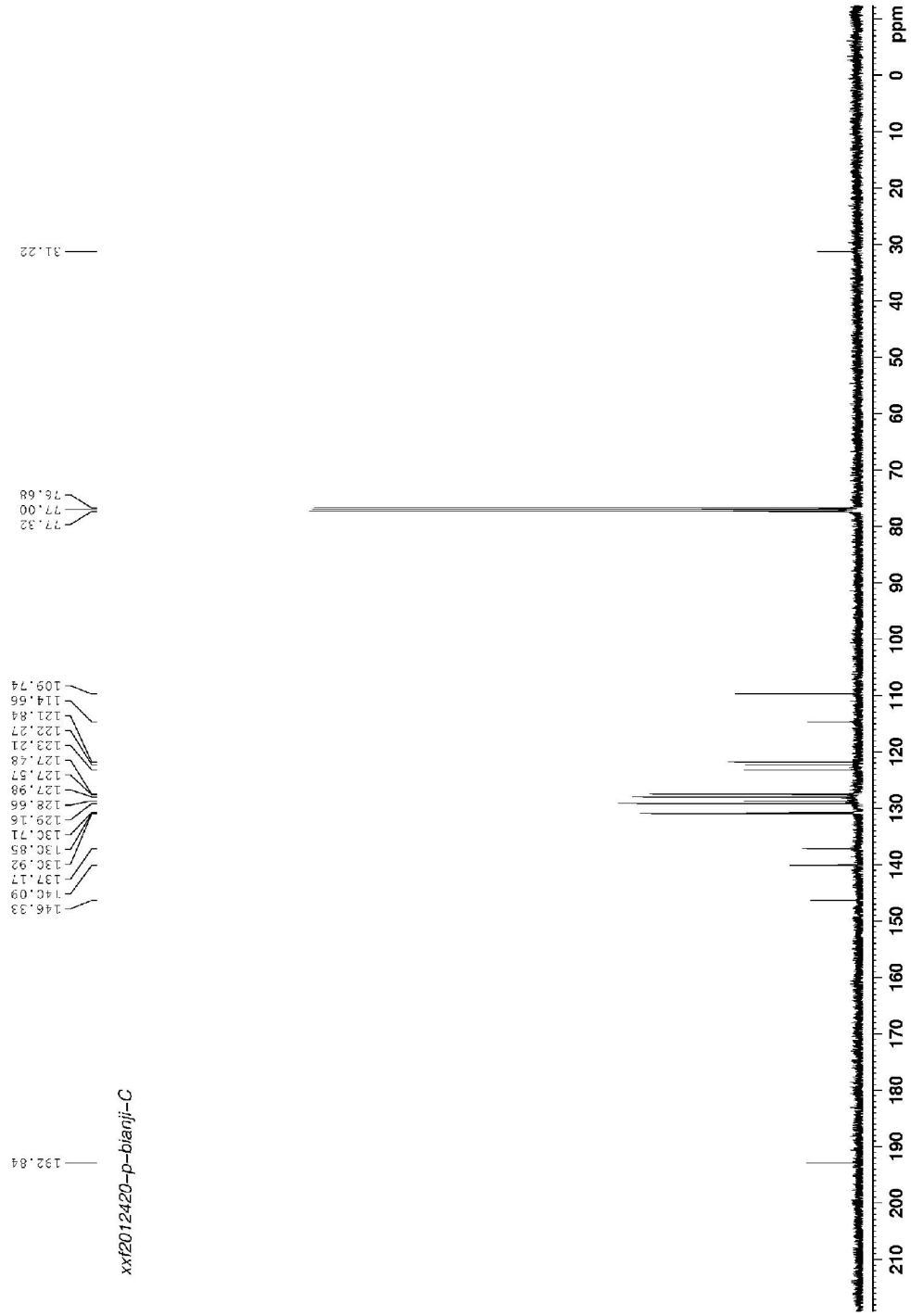
Ph
Ph
N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122.27
123.21
127.48
127.57
127.98
128.66
129.16
130.71
130.85
130.92
137.17
140.09
146.33
192.84
xxf2012420-p-bianji-C
ppm
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60
70
80
90
100
110
120
130
140
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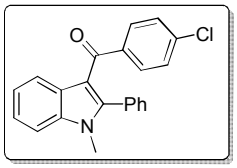
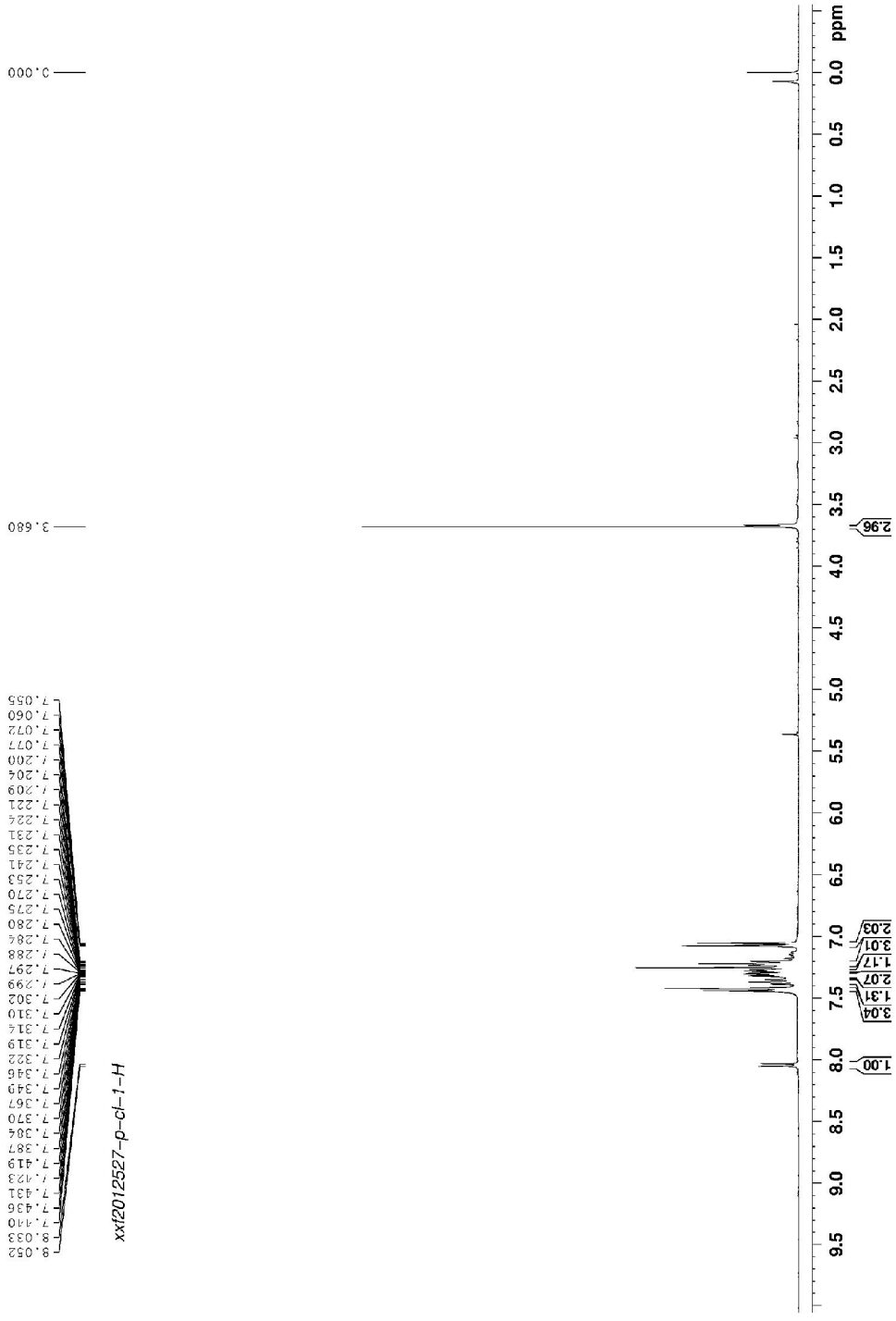
180
190
200
210


O
Cl
Ph
N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5.0
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6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
2.96
2.03
3.01
1.17
2.07
1.31
3.04
1.00
3.000
3.680
8.052
8.033
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7.436
7.431
7.423
7.419
7.387
7.384
7.370
7.367
7.349
7.346
7.322
7.319
7.314
7.310
7.302
7.299
7.297
7.288
7.284
7.280
7.275
7.270
7.253
7.241
7.235
7.231
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7.060
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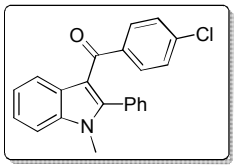
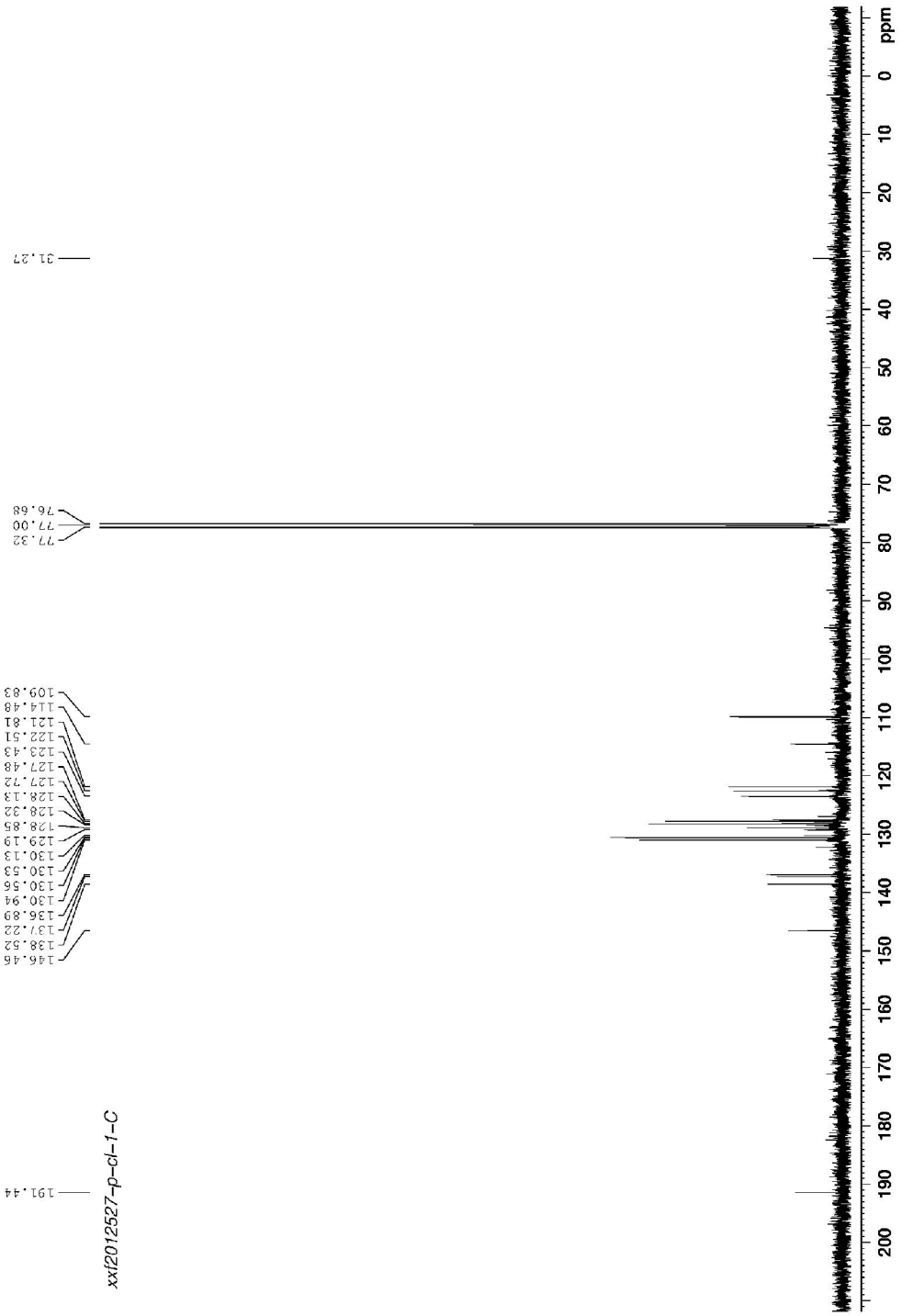

O
Cl
Ph
N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130.94
130.56
130.53
130.13
129.19
128.85
128.32
128.13
127.72
127.48
123.43
122.51
121.81
114.48
109.83
77.32
77.00
76.68
31.27
xxf20120527-p-cl-1-C
ppm
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50
60
70
80
90
100
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130
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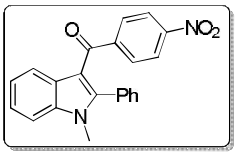
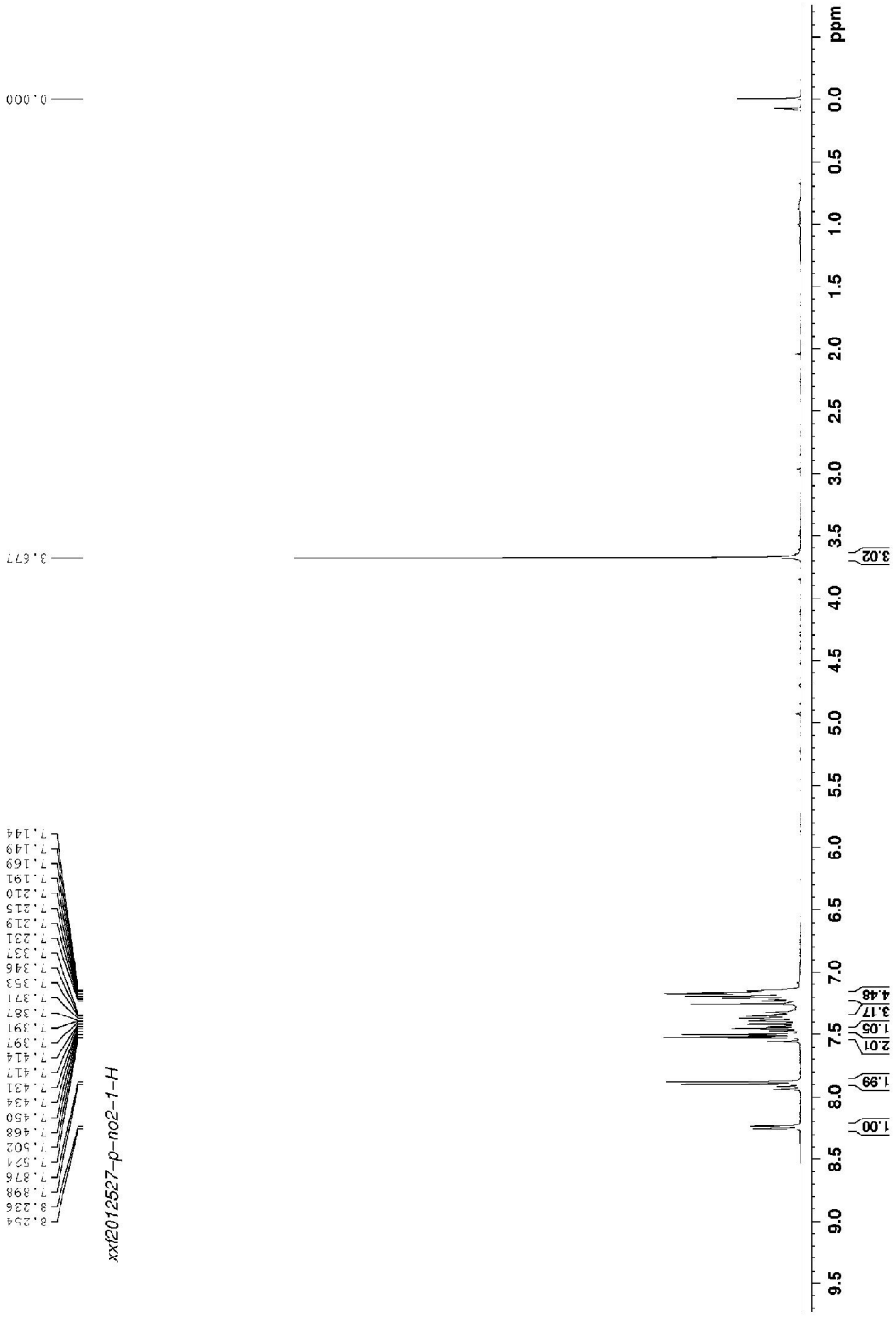
170
180
190
200


O
NO2
Ph
N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7.450
7.434
7.431
7.417
7.414
7.397
7.391
7.387
7.371
7.353
7.346
7.337
7.231
7.219
7.215
7.210
7.191
7.169
7.149
7.144
xxf2012527-p-no2-1-H
ppm
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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
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9.0
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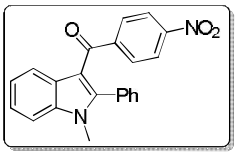
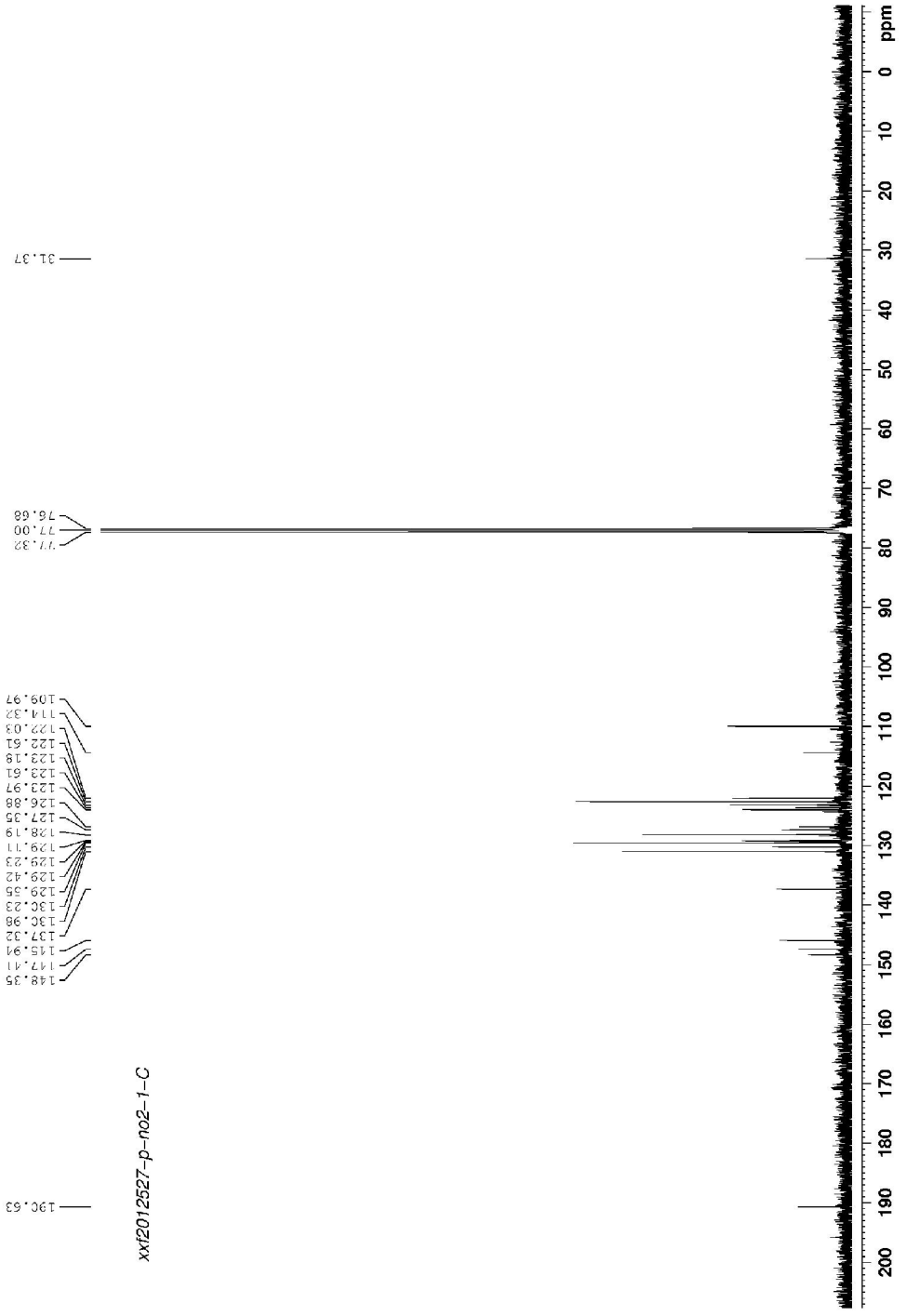
1.00


O
NO2
Ph
N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123.61
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126.88
127.35
128.19
129.11
129.23
129.42
129.55
130.23
130.98
137.32
145.94
147.41
148.35
196.63
xxf2012527_p_no2_1_C
ppm
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50
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80
90
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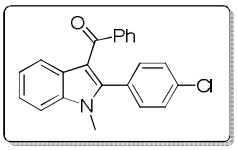
180
190
200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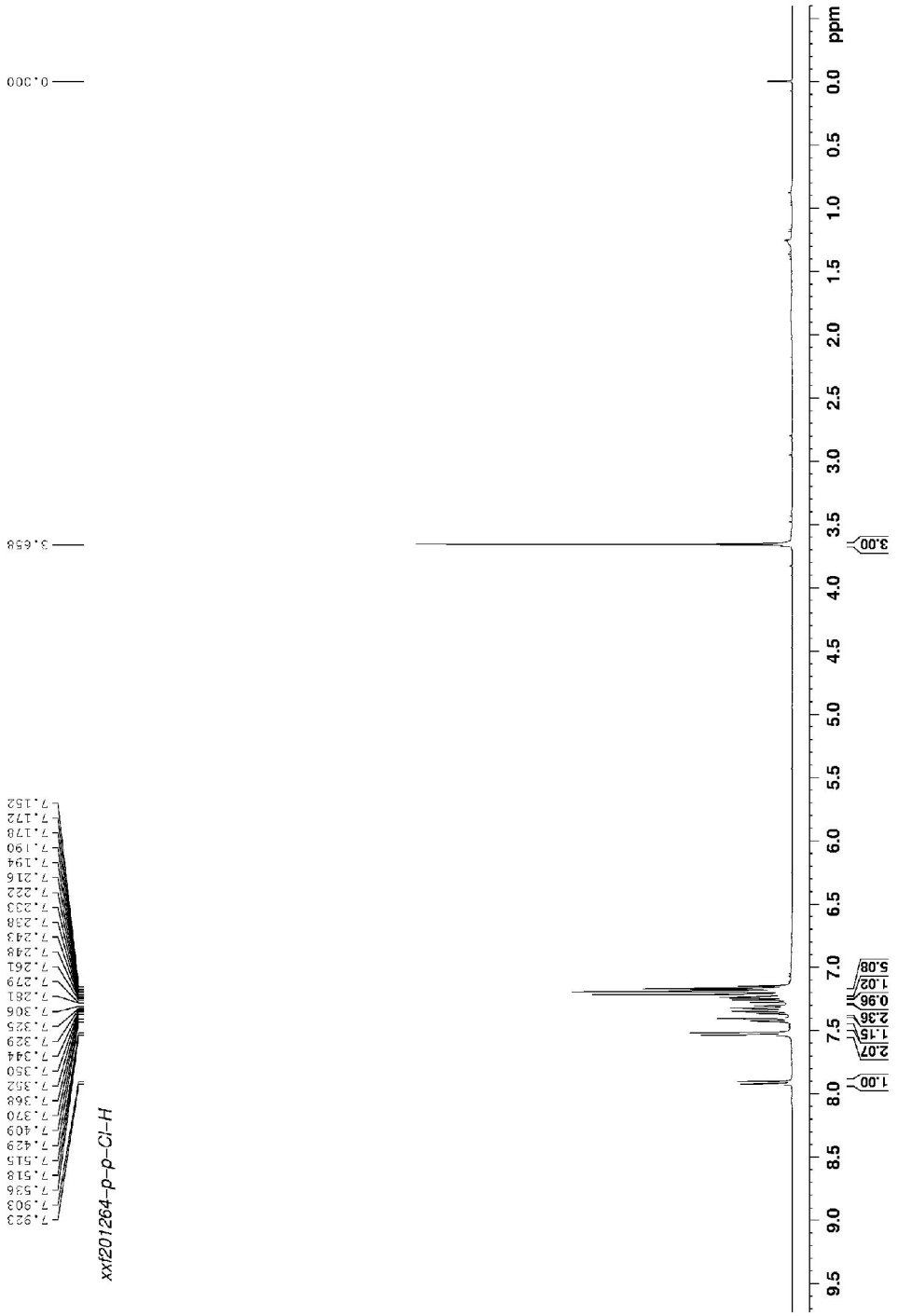
Ph
N
Cl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7.233
7.238
7.243
7.248
7.261
7.279
7.281
7.306
7.325
7.329
7.344
7.350
7.352
7.368
7.370
7.409
7.429
7.515
7.518
7.536
7.903
7.923
xxf201264-p-p-Cl-H
ppm
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2.5
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5.5
6.0
6.5
7.0
7.5
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1.02
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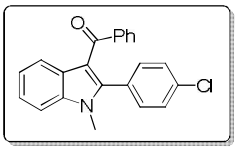
2.07
1.00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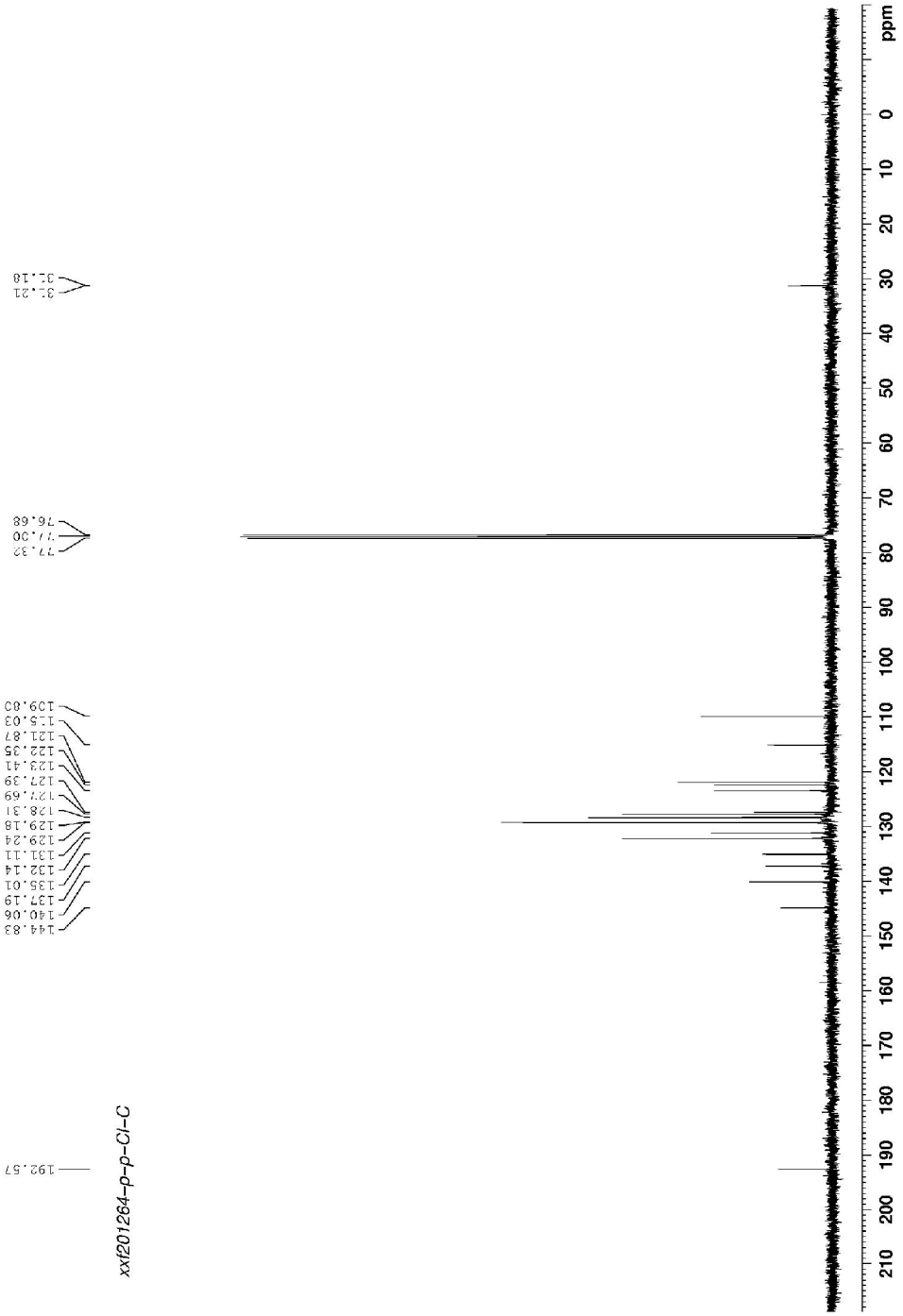
Ph
N
Cl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123.41
127.39
127.69
128.31
129.18
129.24
131.11
132.14
135.01
137.19
140.06
144.83
192.57
xxf201264-p-p-Cl-C
ppm
0
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60
70
80
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100
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140
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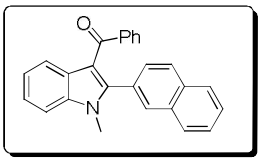
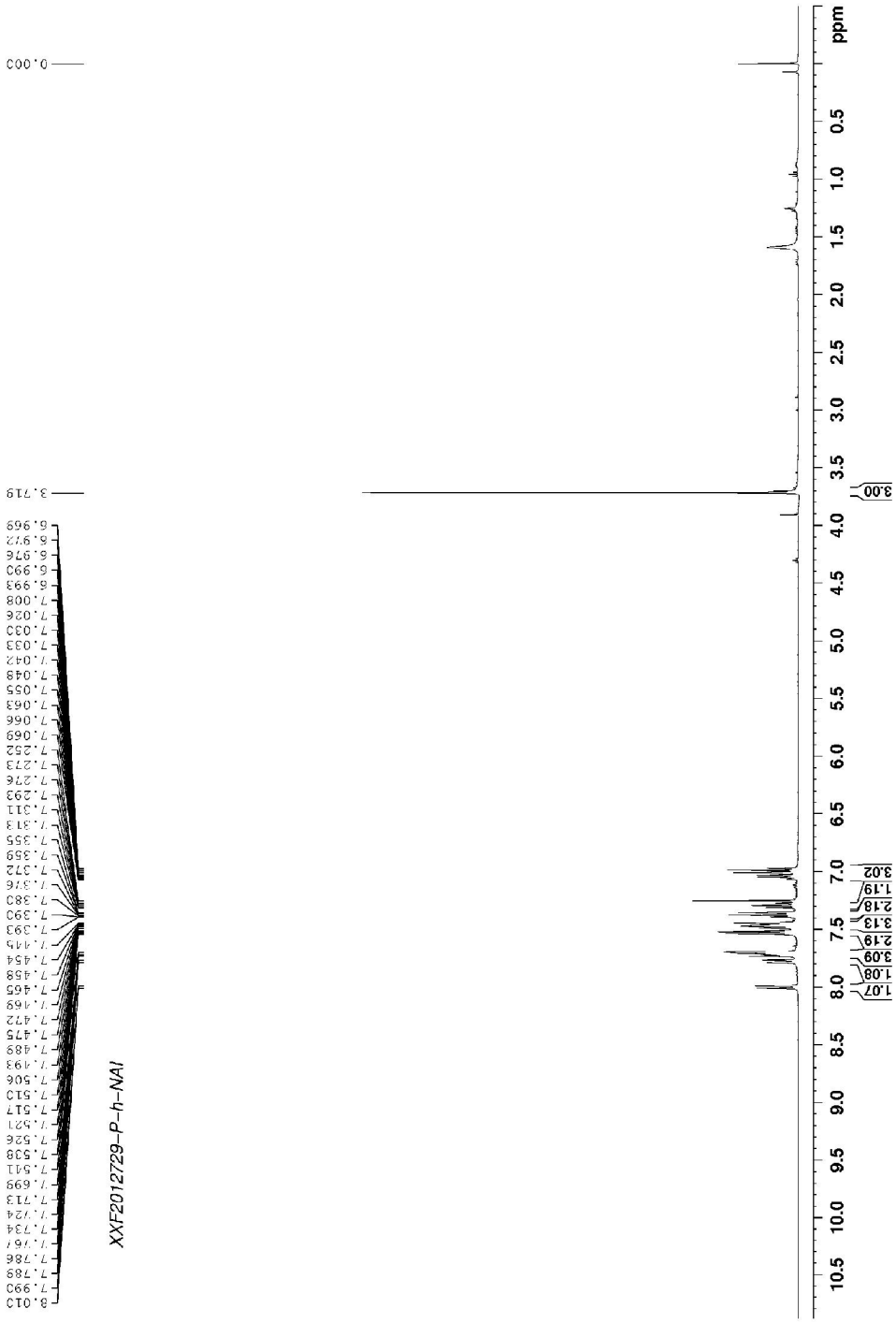
190
200
210


O
Ph
N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7.026
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7.042
7.048
7.055
7.063
7.066
7.069
7.252
7.273
7.276
7.293
7.311
7.313
7.355
7.359
7.372
7.376
7.380
7.390
7.393
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7.493
7.506
7.510
7.517
7.521
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7.538
7.541
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7.734
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7.786
7.789
7.990
8.010
XXF2012729-P-h-NAI
ppm
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1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
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7.0
7.5
8.0
8.5
9.0
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2.18
3.13
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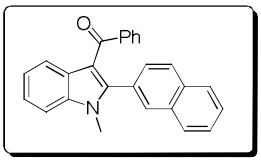
1.07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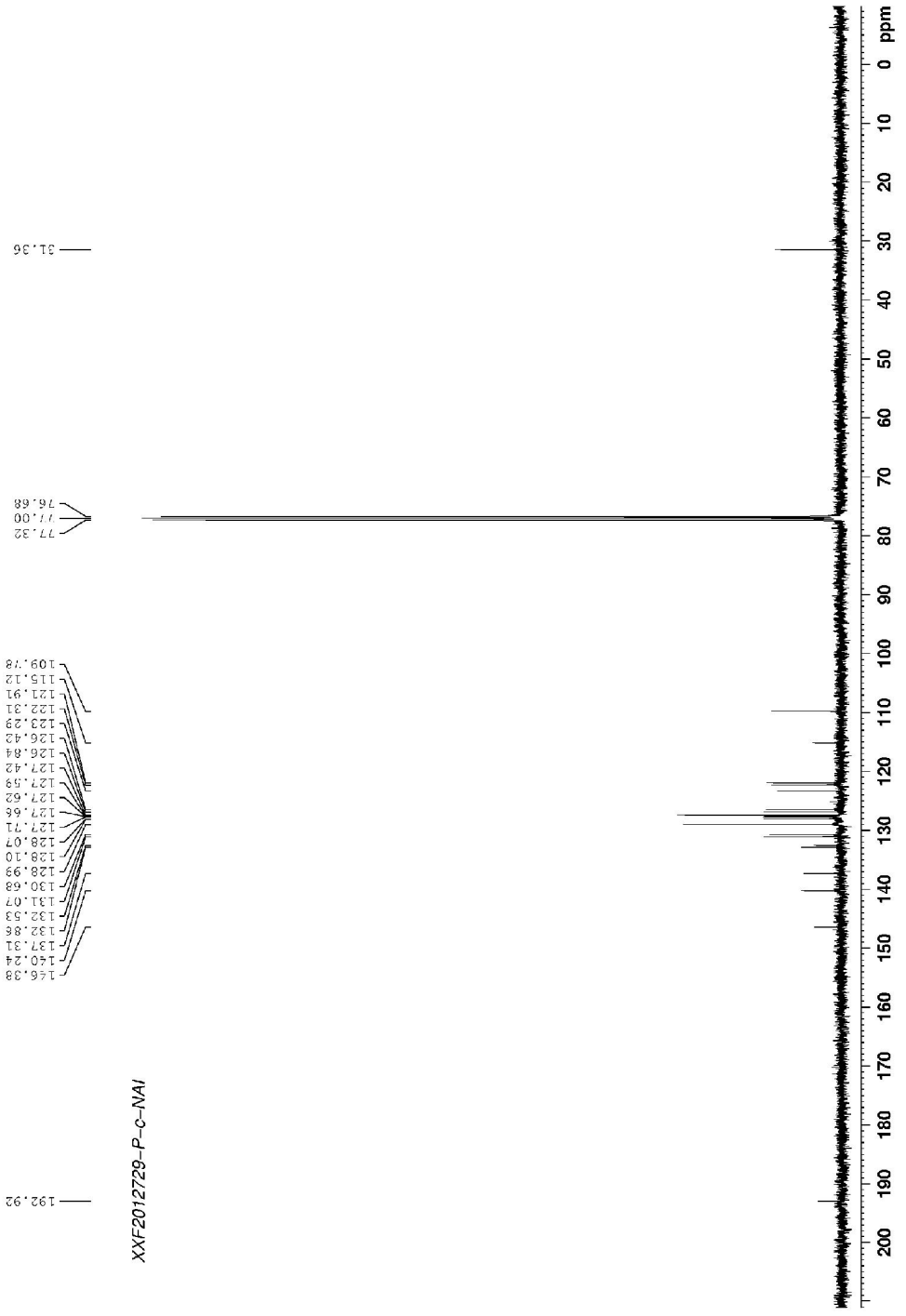
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127.62
127.66
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128.07
128.10
128.99
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131.07
132.53
132.86
137.31
140.24
146.38
192.92
XXF2012729-P-c-NAI
ppm
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130
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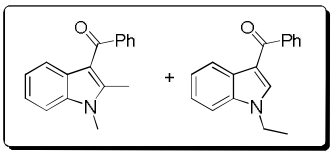
190
200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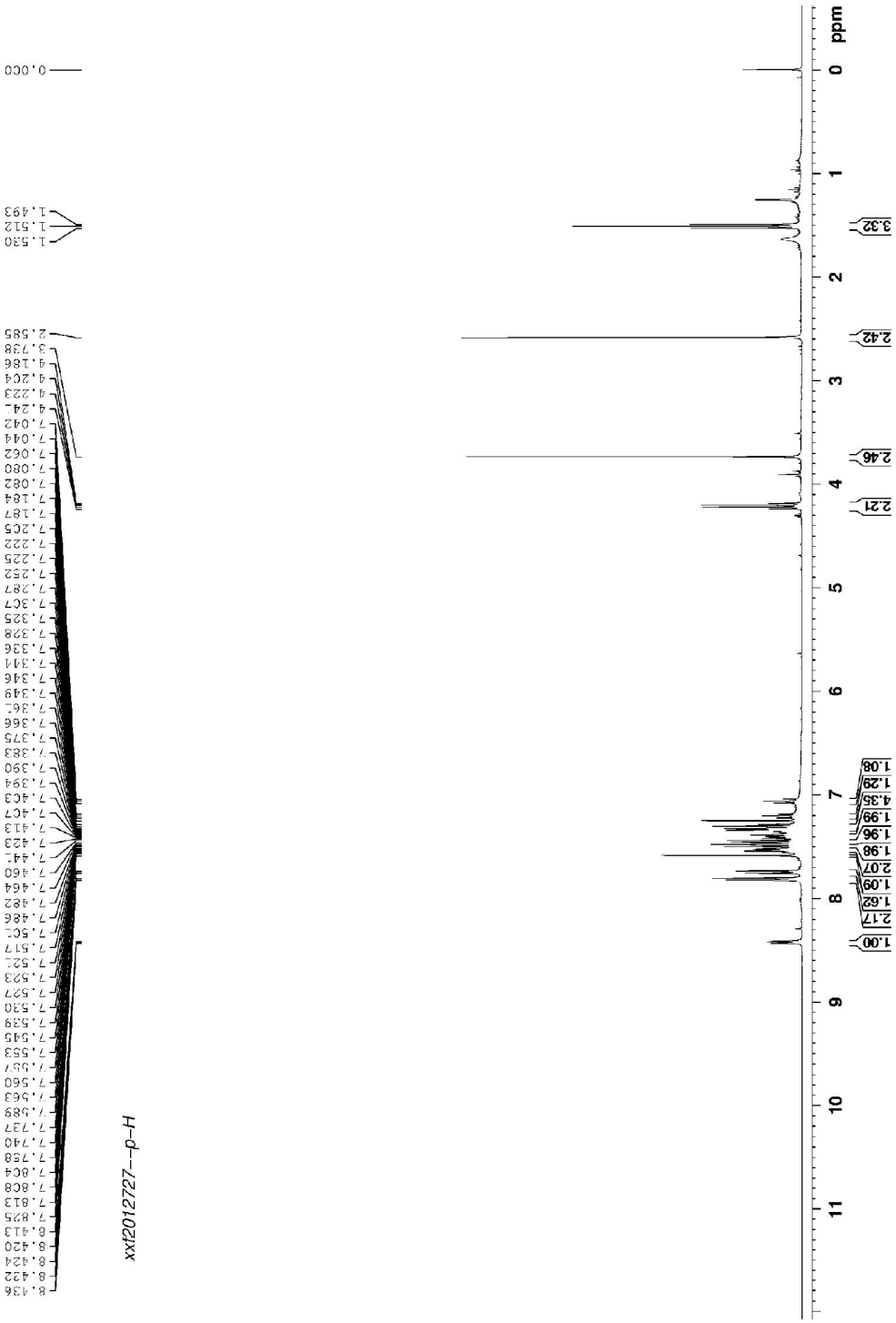
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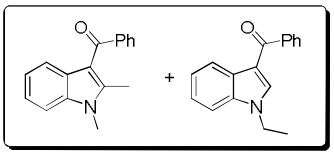
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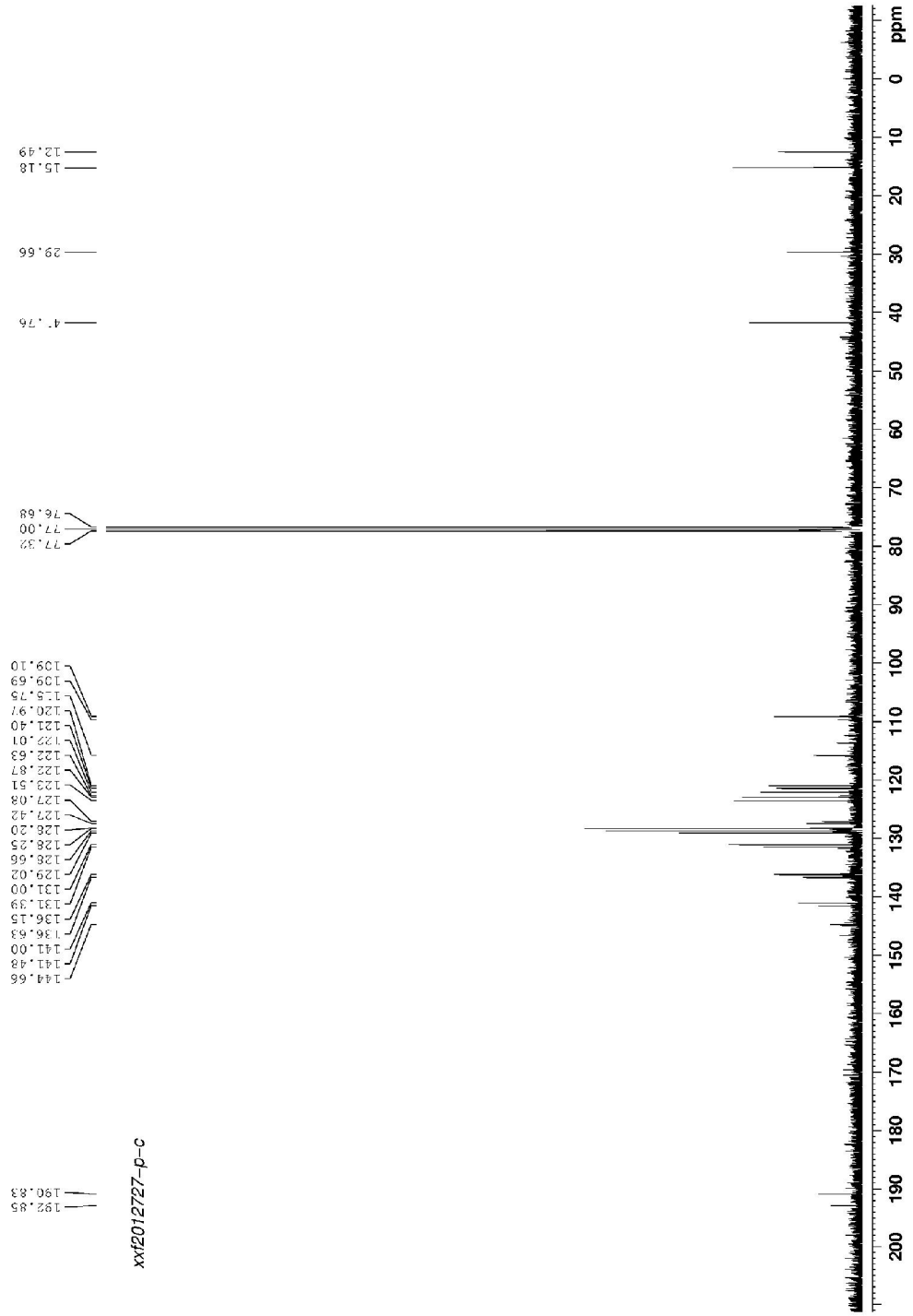
+
Ph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77.00
77.32
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122.63
122.87
123.51
127.08
127.42
128.20
128.25
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129.02
131.00
131.39
136.15
136.63
141.00
141.48
144.66
192.85
190.83
xxf2012727-p-c
ppm
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20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200