Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Synthesis of benzimidazoles by potassium *tert*-butoxide-promoted intermolecular cyclization reaction of 2-iodoanilines with nitriles

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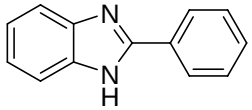
General Remarks

General: All manipulations were conducted with Schlenk tube. ^{1}H NMR spectra were recorded on the Varian 400 MHz WB spectrometers. Chemical shifts (in ppm) were referenced to tetramethylsilane (δ = 0 ppm) as an internal standard in $CDCl_3$ and DMSO-d6. ^{13}C NMR spectra were obtained by the same NMR spectrometers and were calibrated with $CDCl_3$ (δ = 77.00 ppm) or DMSO-d6 (δ = 39.50 ppm). Mass spectra were obtained using electrospray ionization (ESI) mass spectrometer. All nitriles and solvents were freshly distilled over CaH_2 (This operation is very important for these reactions). Unless otherwise noted, other materials obtained from commercial suppliers were used without further purification.

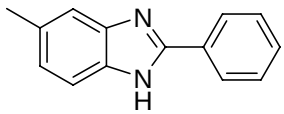
Experimental Section

2-Phenyl-benzimidazole (3a)[1]



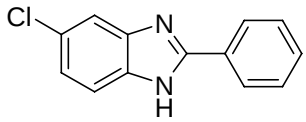
Typical Procedure: General procedure for the synthesis of 2-phenyl-benzoimidazole **3a**: 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), KOBu^{t} (102 mg, 0.9 mmol), DMAc (1.0 mL) were mixed in a Schlenck tube under Ar. To the mixture was added benzonitrile **2a** (61.8 mg, 0.3 mmol) under Ar. The reaction mixture was stirred for 24 h at 120 °C under Ar. The solution was cooled to room temperature and cold saturated aqueous solution of NaCl was added slowly. The crude product was purified by column chromatography on silica gel. (eluent: petroleum ether / acetone = 5:1) to afford 54.2 mg (93%) white solid **3a**; mp: 294-295°C; IR:(KBr) ν_{max} 3047, 1622, 1591, 1463, 1277, 1005, 687, 495 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6 , ppm) δ 12.96 (br s, 1H), 8.21-8.18 (m, 2H), 7.68 (br s, 1H), 7.59-7.45 (m, 4H), 7.26-7.17 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 151.3, 143.8, 135.0, 130.2, 129.9(broad peak), 129.0 (broad peak), 126.5 (broad peak), 122.6 (broad peak), 121.8 (broad peak), 118.9, 111.5 (broad peak); MS (ESI) m/z: $[M+H]^+$ 195.1.

5-Methyl-2-phenyl-benzimidazole (3b)[2]



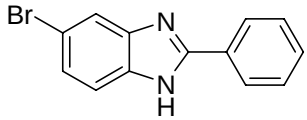
The reaction of 2-iodo-5-methylaniline **1c** (69.9 mg, 0.3 mmol), benzonitrile **2a** (61.9 mg, 0.6 mmol), KOBu^{t} (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 120 °C afforded 56.5 mg (91%) white solid **3b**; mp: 251-252°C; IR:(KBr) ν_{max} 1630, 1463, 1400, 1112, 766 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 12.78 (br s, 1H), 8.16 (d, *J* = 7.6 Hz, 2H), 7.57-7.31 (m, 5H), 7.06-7.00 (m, 1H), 2.43(s, 3H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ151.1, 150.7, 144.2, 142.0, 135.3, 133.1, 131.9, 130.6, 130.3, 129.7, 128.9, 126.3, 124.0, 123.3, 118.6, 118.5, 111.1, 110.9, 21.4, 21.3; MS (ESI)m/z: $[M+H]^+$209.1.

5-Chloro-2-phenyl-benzimidazole (**3c**) [2]



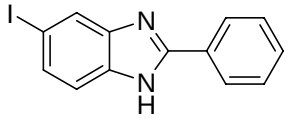
The reaction of 4-chloro-2-iodoaniline **1d** (75.9 mg, 0.3 mmol), benzonitrile **2a** (61.9 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 80 °C afforded 56.3 mg (82%) white solid **3c**; mp: 294-295°C; IR:(KBr) ν_{max} 3043, 1624, 1451, 1439, 1384, 1309, 1107, 1062 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 13.16 (br s, 1H), 8.20-8.18 (m, 2H), 7.76-7.51(m, 5H), 7.27-7.22(m, 1H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 152.9, 152.5, 144.8, 142.7, 135.8, 133.9, 132.6, 130.2, 129.7, 129.2, 129.0, 128.7, 128.4, 126.9, 126.6, 126.1, 122.7, 122.1, 120.1, 118.3, 112.7, 111.1; MS (ESI) m/z: $[M+H]^+$ 229.1.

5-Bromo-2-phenyl-benzimidazole (3d)[2]



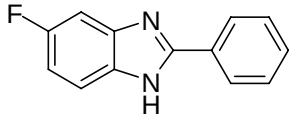
The reaction of 4-bromo-2-iodoaniline **1f** (69.9 mg, 0.3 mmol), benzonitrile **2a** (61.9 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 100 °C afforded 68.5 mg (84%) brown solid **3d**; mp: 206-208°C; IR:(KBr) ν_{max} 1621, 1467, 1444, 1422, 1397, 1304, 1277, 1109 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 13.16 (br s, 1H), 8.19 (d, *J* = 6.8 Hz, 2H), 7.88 (br s, 1H), 7.71-7.50 (m, 4H), 7.37 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 152.7, 152.4, 145.3, 143.0, 136.3, 134.1, 130.2, 129.7, 129.2, 129.0, 126.9, 126.6, 125.2, 124.8, 121.3, 120.6, 114.8, 114.0, 113.1; MS (ESI) m/z: $[M+H]^+$ 273.1.

5-Iodo-2-phenyl-benzimidazole (3e)[3]



The reaction of 2,4-diiodoaniline **1h** (103.5 mg, 0.3 mmol), benzonitrile **2a** (61.9 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 100 °C afforded 71.9 mg (75%) beige solid **3e**; mp: 209-210°C; IR:(KBr) ν_{max} 1464, 1394 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 13.08 (br s, 1H), 8.17 (d, *J* = 6.8 Hz 2H), 7.95 (br s, 1H), 7.60-7.38 (m, 5H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 152.1, 151.9, 145.9, 143.3, 136.9, 134.5, 130.7, 130.3, 130.2, 129.6, 129.0, 127.3, 126.6, 121.0, 119.8, 113.6, 86.3, 85.3; MS (ESI) m/z: $[M+H]^+$ 321.0.

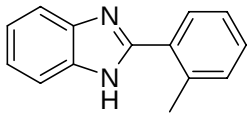
5-Fluoro-2-phenyl-benzimidazole (3f)[3]



The reaction of 4-fluoro-2-iodoaniline **1i** (71.1 mg, 0.3 mmol), benzonitrile **2a** (61.9

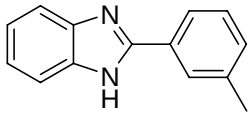
mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 80 °C afforded 26.1 mg (41%) brown solid **3f**; mp: 245-246°C; IR:(KBr) ν_{max} 3043, 1467, 1407 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 13.07 (br s, 1H), 8.18-8.16 (m, 2H), 7.70-7.32 (m, 5H), 7.12-7.03 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 159.8, 157.6, 153.0, 144.2, 140.5, 135.2, 131.7, 130.1, 129.9, 129.0, 126.5, 119.8, 112.1, 110.4, 104.4, 97.8; MS (ESI) m/z: $[M+H]^+$ 213.1.

2-(2-Methylphenyl)-benzimidazole (3g)[4]



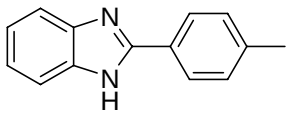
The reaction of 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), 2-methylbenzonitrile **2b** (70.3 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 120 °C afforded 22.0 mg (35%) white solid **3g**; mp: 226-227°C; IR:(KBr)ν_{max} 2876, 1605, 1541, 1445, 1409, 1366, 1315, 1273, 1227, 1091, 766, 747, 733, 455 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 12.62 (br s, 1H), 7.75 (d, J = 7.6 Hz, 1H), 7.68 (d, J = 7.2 Hz, 1H), 7.53 (d, J = 7.2 Hz, 1H), 7.41-7.36 (m, 3H), 7.24-7.19 (m, 2H), 2.61(s, 3H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 151.9, 143.7, 137.0, 134.4, 131.3, 130.1, 129.5, 129.3, 126.0, 122.4, 121.4, 118.9, 111.3, 21.1; MS (ESI) m/z: $[M+H]^+$ 209.1.

2-(3-Methylphenyl)-benzimidazole (3h)[2]



The reaction of 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), 3-methylbenzonitrile **2c** (70.3 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 120 °C afforded 52.4 mg (84%) white solid **3h**; mp: 224-225℃; IR:(KBr) ν_{max} 2878, 1624, 1447, 1404, 1359, 1316, 1275, 1228 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6 , ppm) δ 12.87 (br s, 1H), 8.04-8.03 (m, 1H), 7.97 (d, J = 7.6 Hz, 1H), 7.65-7.52 (m, 2H), 7.44 (t, J = 7.6 Hz, 1H), 7.33-7.30 (m, 1H), 7.21-7.19 (m, 2H), 2.42(s, 3H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 151.4, 138.2, 130.5, 130.1, 128.8, 127.0, 123.0, 122.1, 118.5, 111.5, 21.1; MS (ESI) m/z: $[M+H]^+$ 209.1.

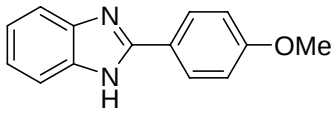
2-(4-Methylphenyl)-benzimidazole (3i)[1]



The reaction of 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), 4-methylbenzonitrile **2d** (70.3 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 120 °C afforded 57.4 mg (92%) white solid **3i**; mp: 279-280℃; IR:(KBr) ν_{max} 3052, 2962, 2914, 1619, 1588, 1500, 1446, 1430, 1273 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6 ,

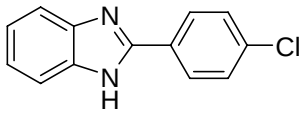
ppm) δ 12.84 (br s, 1H), 8.07 (d, *J* = 8.0 Hz, 2H), 7.64-7.52 (m, 2H), 7.36 (d, *J* = 8.4 Hz, 2H), 7.20-7.18 (m, 2H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 151.4, 139.6, 129.5, 127.5, 126.4, 122.0, 21.0; MS (ESI) m/z: $[M+H]^+$ 209.1.

2-(4-Methoxyphenyl)-benzimidazole (3j)[1]



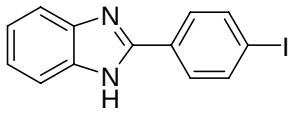
The reaction of 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), 4-methoxybenzonitrile **2e** (79.8 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 120 °C afforded 61.2 mg (91%) white solid **3j**; mp: 232-233°C; IR:(KBr) ν_{max} 3052, 1611, 1581, 1454, 1233, 764, 694cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 12.77 (br s, 1H), 8.12 (d, *J* = 8.8 Hz, 2H), 7.62 (d, *J* = 7.2 Hz, 1H), 7.50 (d, *J* = 7.2 Hz, 1H), 7.20-7.10 (m, 4H), 3.84(s, 3H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 160.6, 151.4, 128.1, 122.7, 121.8, 114.4, 55.3; MS (ESI) m/z: $[M+H]^+$ 225.1.

2-(4-Chlorophenyl)-benzimidazole (3k)[1]



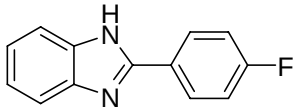
The reaction of 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), 4-chlorobenzonitrile **2f** (82.2 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 120 °C afforded 40.8 mg (60%) white solid **3k**; mp: 304-305°C; IR:(KBr) ν_{max} 3051, 1586, 1491, 1273, 1108, 1016, 765, 288, 471, 434cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 13.02 (br s, 1H), 8.22-8.19 (m, 2H), 7.70-7.63 (m, 3H), 7.55 (br s, 1H), 7.27-7.18 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 150.2, 143.8, 135.0, 134.5, 129.1, 129.07, 128.2, 122.8, 121.9, 119.0, 111.4; MS (ESI) m/z: $[M+H]^+$ 229.1.

2-(4-Iodophenyl)-benzimidazole (3l)[5]



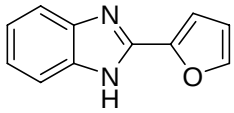
The reaction of 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), 4-iodobenzonitrile **2g** (137.4 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 120 °C afforded 55.1mg (57%) white solid **3l**; mp: 303-304°C; IR:(KBr) ν_{max} 2970, 1447, 1424, 1315, 1059, 828, 742cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 13.00 (br s, 1H), 7.98-7.92 (m, 4H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 7.6 Hz, 1H), 7.24-7.19 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 150.4, 143.7, 137.8, 135.0, 129.7, 128.3, 122.8, 121.9, 119.0, 111.5, 96.8; MS (ESI) m/z: $[M+H]^+$321.0.

2-(4-Fluorophenyl)-benzimidazole (3m)[6]



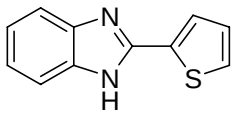
The reaction of 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), 4-fluorobenzonitrile **2h** (72.6 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 80 °C afforded 38.5 mg (61%) orange solid **3m**; mp: 254-255°C; IR:(KBr) ν_{max} 3053, 1603, 1397, 1228 1064, 795, 747cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 12.93 (br s, 1H), 8.24-8.20 (m, 2H), 7.68-7.65 (m, 2H), 7.55-7.39 (m, 2H), 7.22-7.21(m, 2H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 163.1(d, J = 246.5 Hz), 150.5, 143.8, 135.1, 128.8, 128.7, 126.9 (d, J = 2.3 Hz); 122.2 (d, J = 81.2 Hz); 118.9, 116.1 (d, J = 21.6 Hz), 111.4; MS (ESI) m/z: $[M+H]^+$ 213.1.

2-(Furan-2-yl)-benzimidazole (3n)[1]



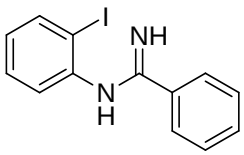
The reaction of 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), furan-2-carbonitrile **2i** (55.8 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 100 °C afforded 34.2 mg (62%) white solid **3n**; mp: 279-280°C; IR:(KBr) ν_{max} 3058, 1631, 1525, 1417, 1364, 1279, 1234 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 12.94 (br s, 1H), 7.96-7.95 (m, 1H), 7.63 (d, J = 8.0 Hz, 1H), 7.50 (d, J = 7.2 Hz, 1H), 7.24-7.17 (m, 3H), 6.75-6.73 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 145.6, 144.7, 143.7, 134.3, 122.7, 121.8, 118.8, 112.4, 111.4, 110.5; MS (ESI) m/z: $[M+H]^+$185.1.

2-(Thien-2-yl)-benzimidazole (3o)[1]



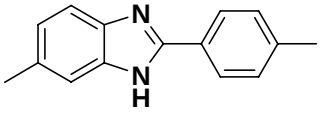
The reaction of 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), thiophene-2-carbonitrile **2j** (65.4 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 100 °C afforded 45.8 mg (76%) yellow solid **3o**; mp: 312-313°C; IR:(KBr) ν_{max} 3047, 3009, 1571, 1451, 1424, 1314, 1276, 1235 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 12.94 (br s, 1H), 7.84 (d, J = 2.8 Hz, 1H), 7.73 (d, J = 4.8 Hz, 1H), 7.59-7.52 (m, 2H), 7.25-7.19 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 147.1, 133.7, 128.8, 128.7, 128.33, 128.28, 126.73, 126.68, 122.2(broad peak), 118.5(broad peak), 111.1(broad peak); MS (ESI) m/z: $[M+H]^+$ 201.1.

N-(2-Iodophenyl)benzimidamide (5a)[4]



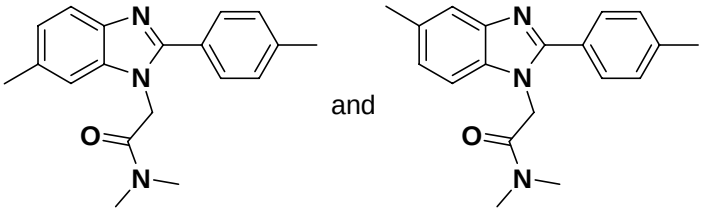
The reaction of 2-iodoaniline **1a** (65.7 mg, 0.3 mmol), benzonitrile **2b** (70.3mg, 0.6mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 25 °C afforded 87.1 mg (90%) white solid **5a**; mp: 110-112°C; IR:(KBr) ν_{max} 3446, 3296, 3147, 3051, 1632, 1564, 1386, 1014, 746 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 8.02 (d, *J* = 5.6 Hz, 2H), 7.82 (d, *J* = 8.0Hz, 1H), 7.47 (d, *J* = 7.2 Hz, 3H), 7.35-7.31 (m, 1H), 6.89 (d, *J* = 7.2 Hz, 1H), 6.78-6.74 (m, 1H), 6.42 (br s, 2H), ; ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 154.0, 151.8, 138.6, 135.5, 130.3, 129.4, 128.0, 127.4, 123.7, 121.8, 94.0; MS (ESI) m/z: $[M+H]^+$ 323.0.

6-Methyl-2-(*p*-tolyl)-benzimidazole (3p)[7]



The reaction of 2-iodo-4-methylaniline **1c** (69.9 mg, 0.3 mmol), 4-methylbenzonitrile **2d** (70.3 mg, 0.6 mmol), KOBu^t (101.1 mg, 0.9 mmol), DMAc (1.0 mL) under Ar at 100°C afforded 53.0 mg (80%) white solid **3p**; mp: 168-169°C; IR:(KBr) ν_{max} 3024, 2955, 2920, 2855, 1911, 1717, 1660, 1631, 1447, 803 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 12.69 (br s, 1H), 8.06 (d, *J* = 8.0 Hz, 2H), 7.49-7.33 (m, 4H), 7.02 (d, *J* = 8.0 Hz, 1H), 2.43 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d6, ppm) δ 151.1, 139.4, 131.1, 129.5, 127.6, 126.4, 123.4, 114.9, 21.4, 21.0; MS (ESI) m/z: $[M+H]^+$ 223.1.

***N,N*-Dimethyl-2-(6-methyl-2-(*p*-tolyl)-benzimidazol-1-yl)acetamide** and ***N,N*-Dimethyl-2-(5-methyl-2-(*p*-tolyl)-benzoimidazol-1-yl)acetamide (7 and 7')**[7]



7 **7'**

The compounds **7** and **7'** were synthesized according to literature's method[7] with yield of 66% (7/7' = 1:1, the ratio was determined by ^{1}H NMR on the crude products); white solid; mp: 110-113°C; IR:(KBr) ν_{max} 3030, 2920, 1653, 1449,1146, 807 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6, ppm) δ 7.69 (d, *J* = 8.8 Hz, 1H), 7.59-7.56 (m, 5H), 7.30-7.28 (m, 4H), 7.13-7.07 (m, 3H), 7.01 (s, 1H), 4.88 (s, 2H), 4.87 (s, 2H), 3.05-2.30 (m, 12H), 2.48 (s, 6H), 2.42 (s, 6H); MS (ESI) m/z: $[M+H]^+$ 308.2.

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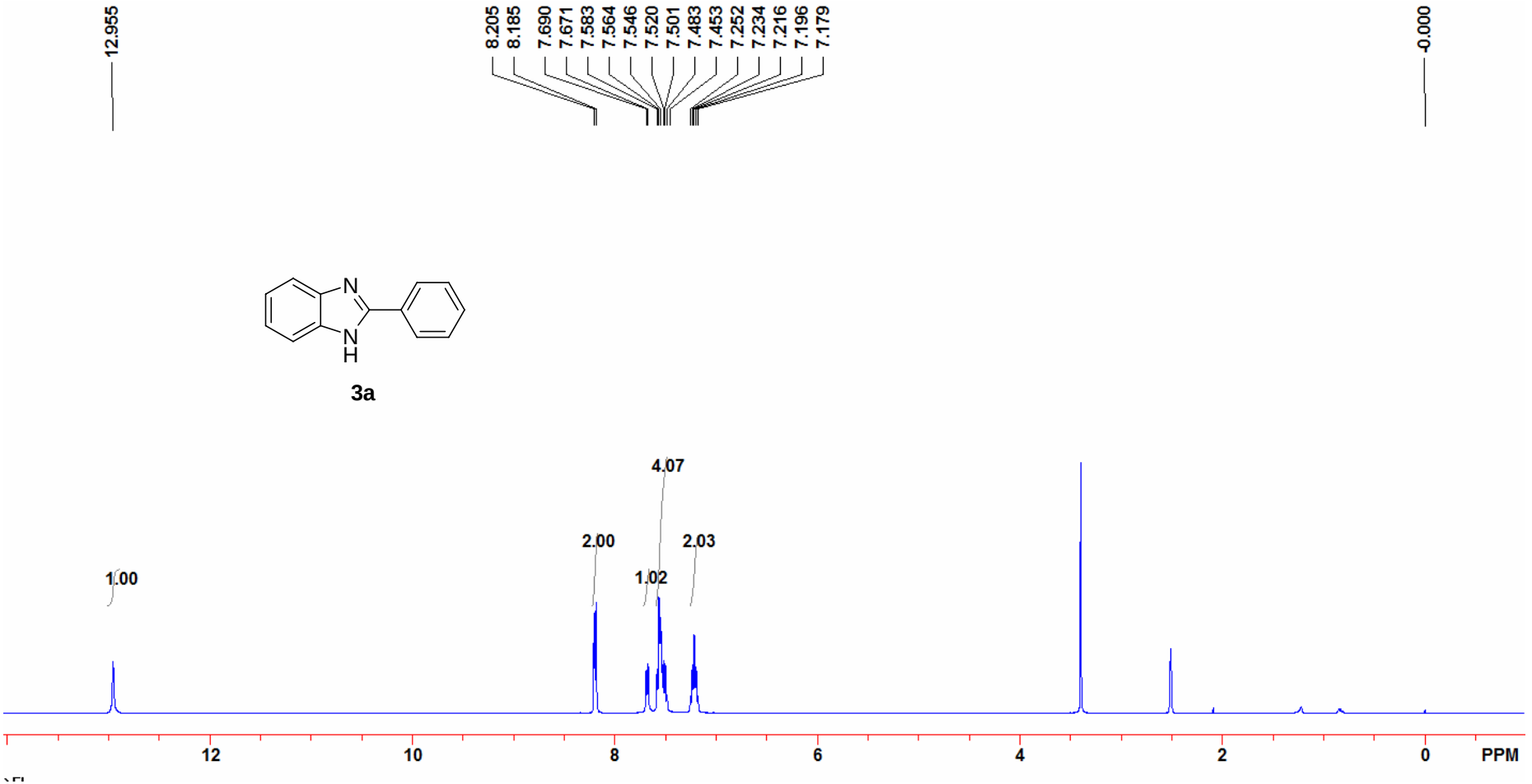

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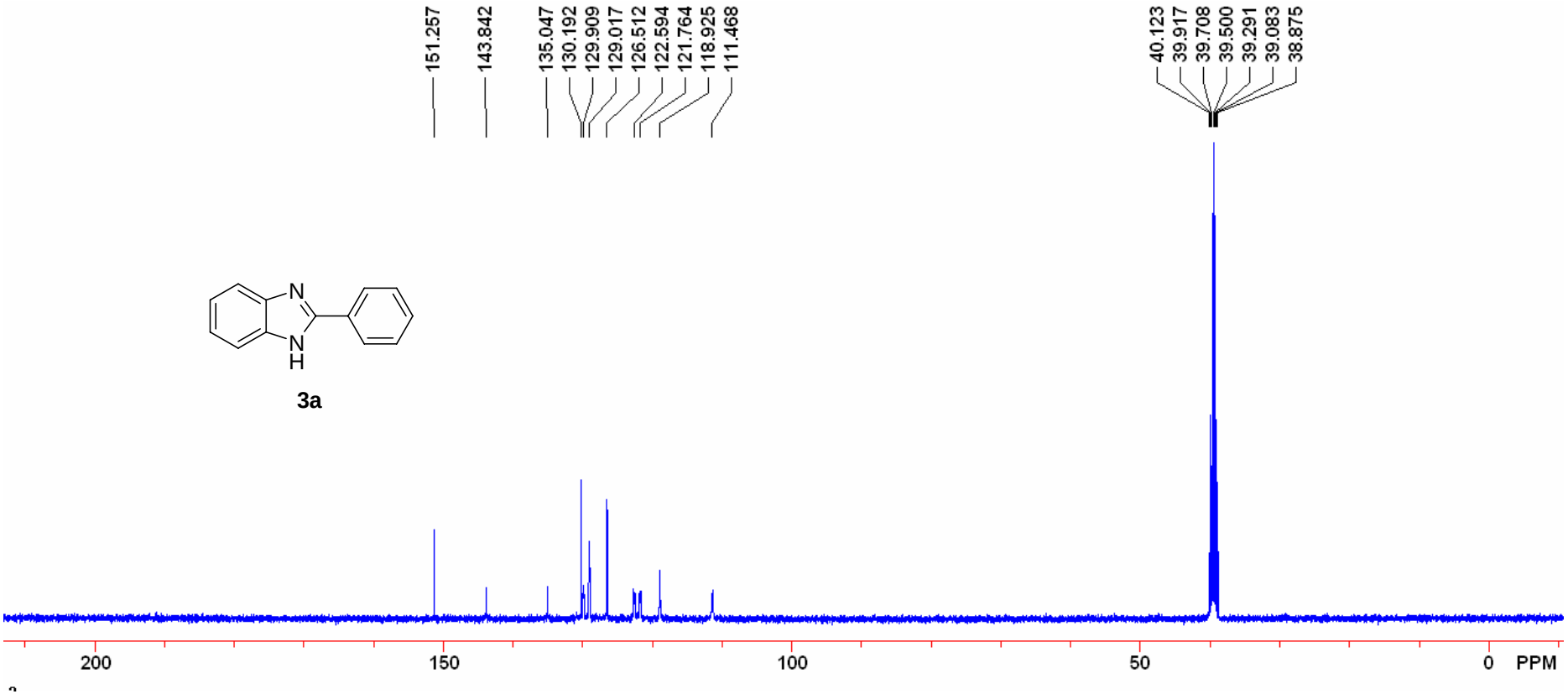

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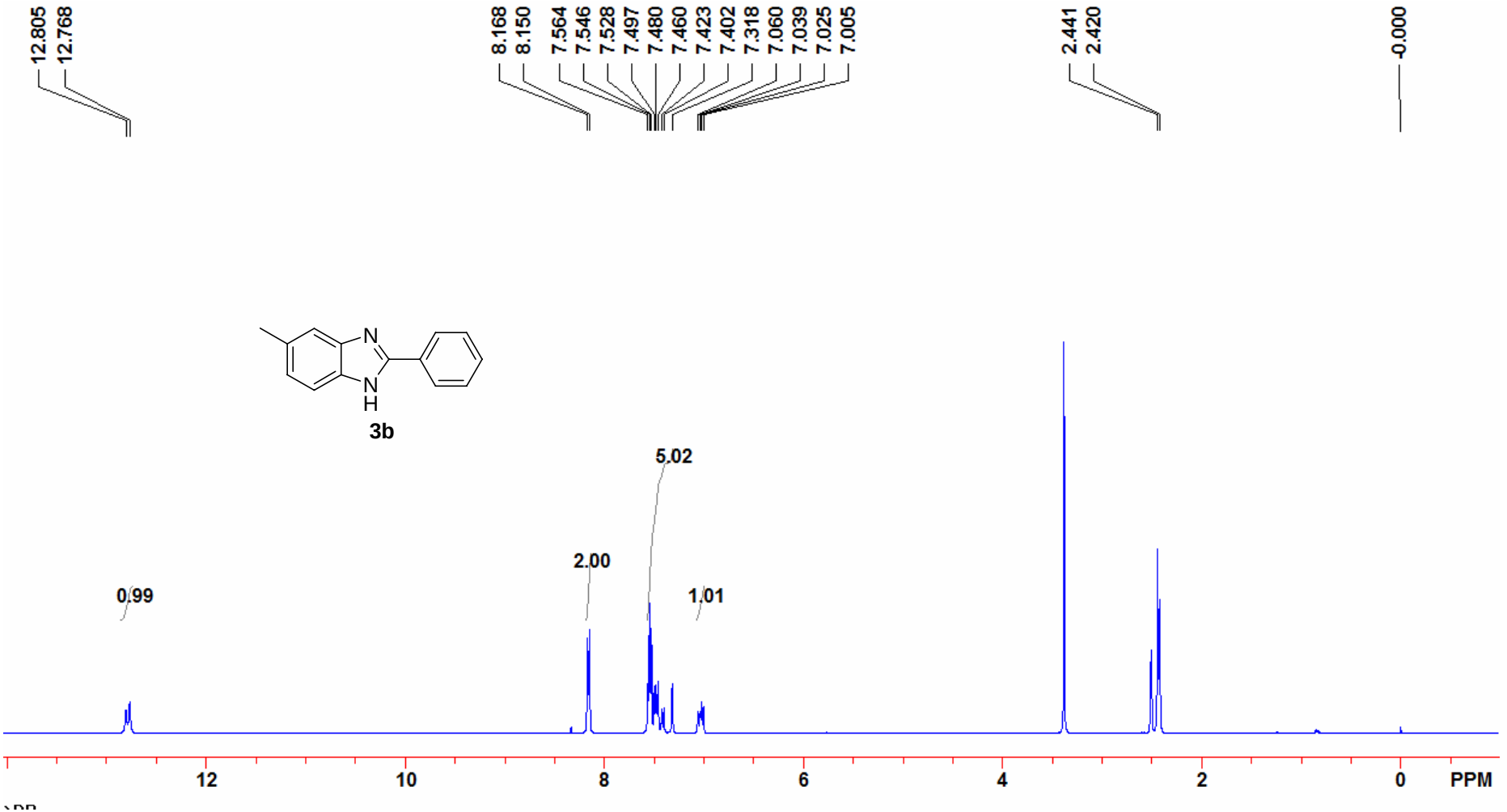

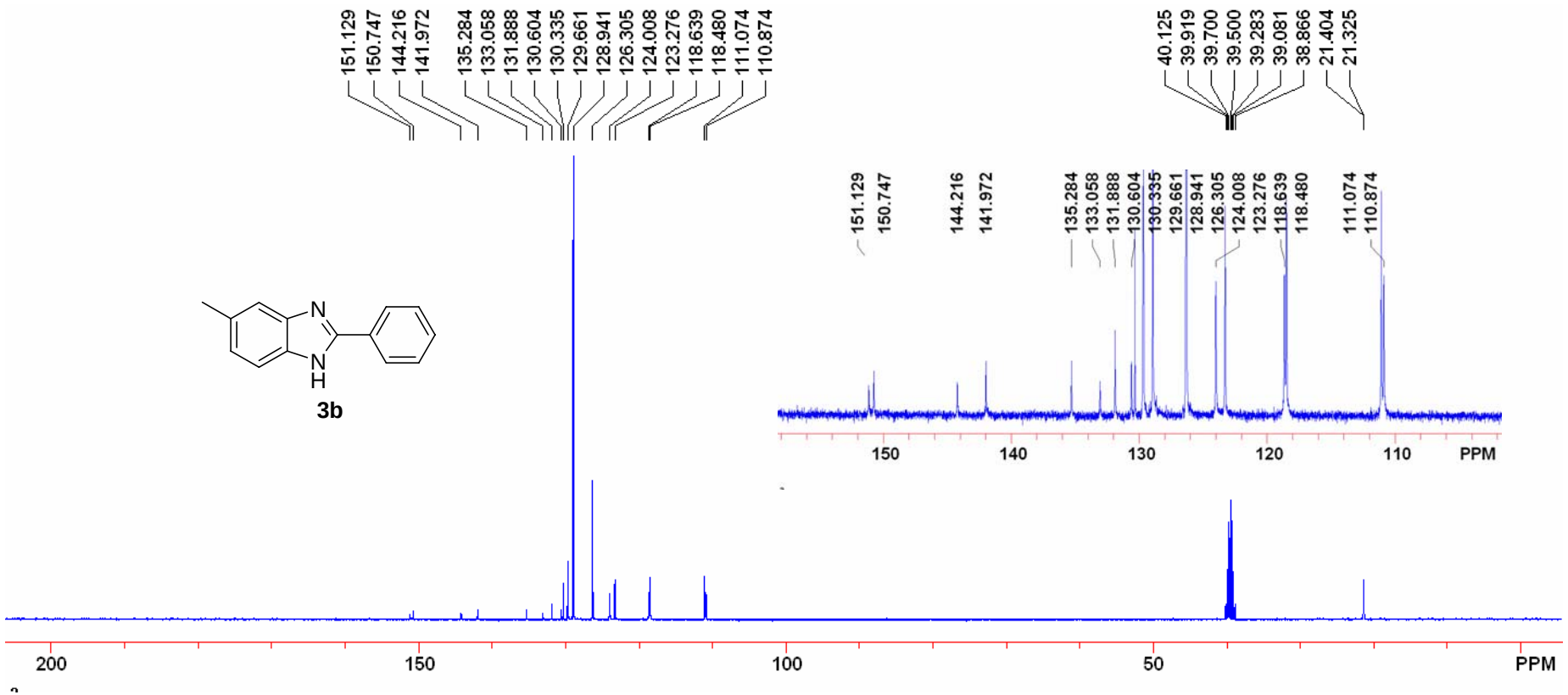

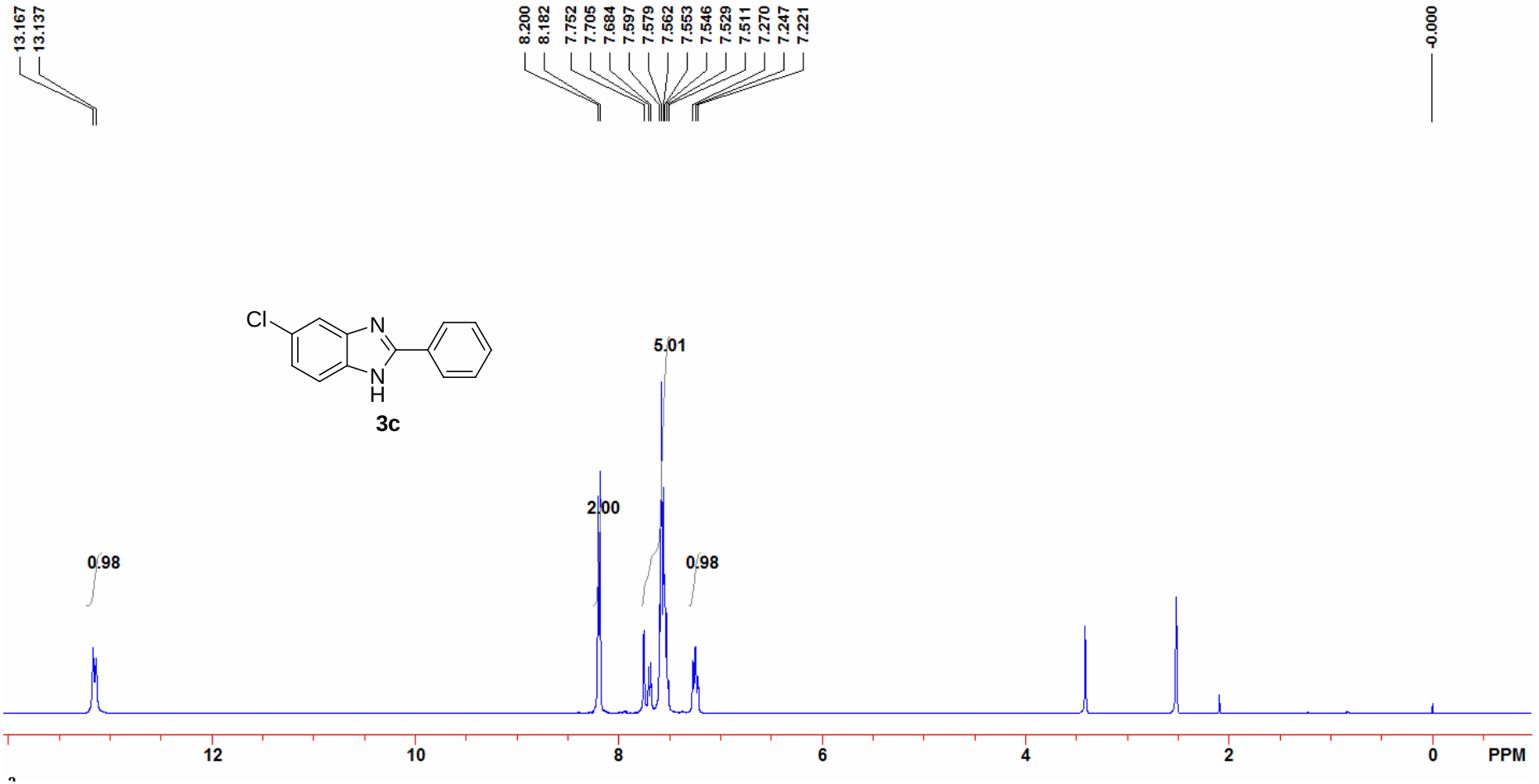

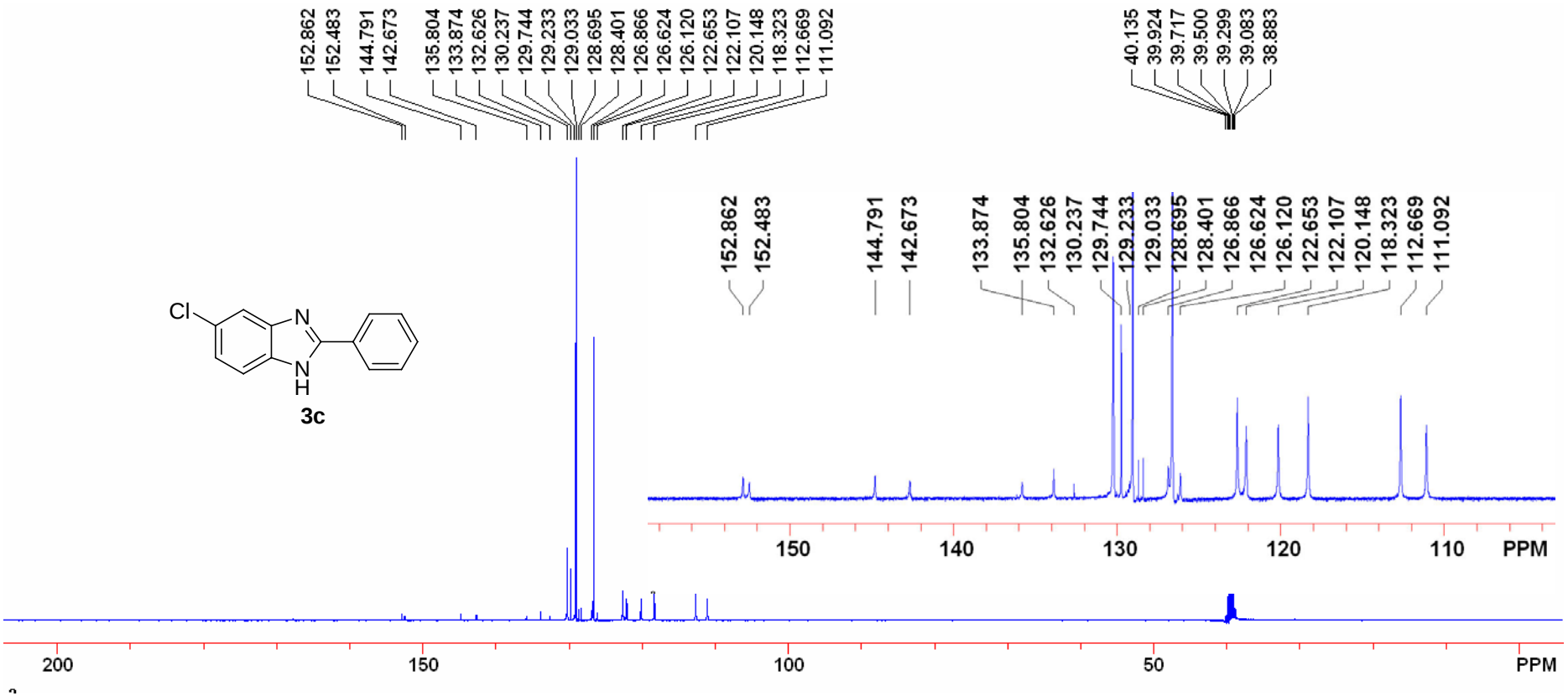

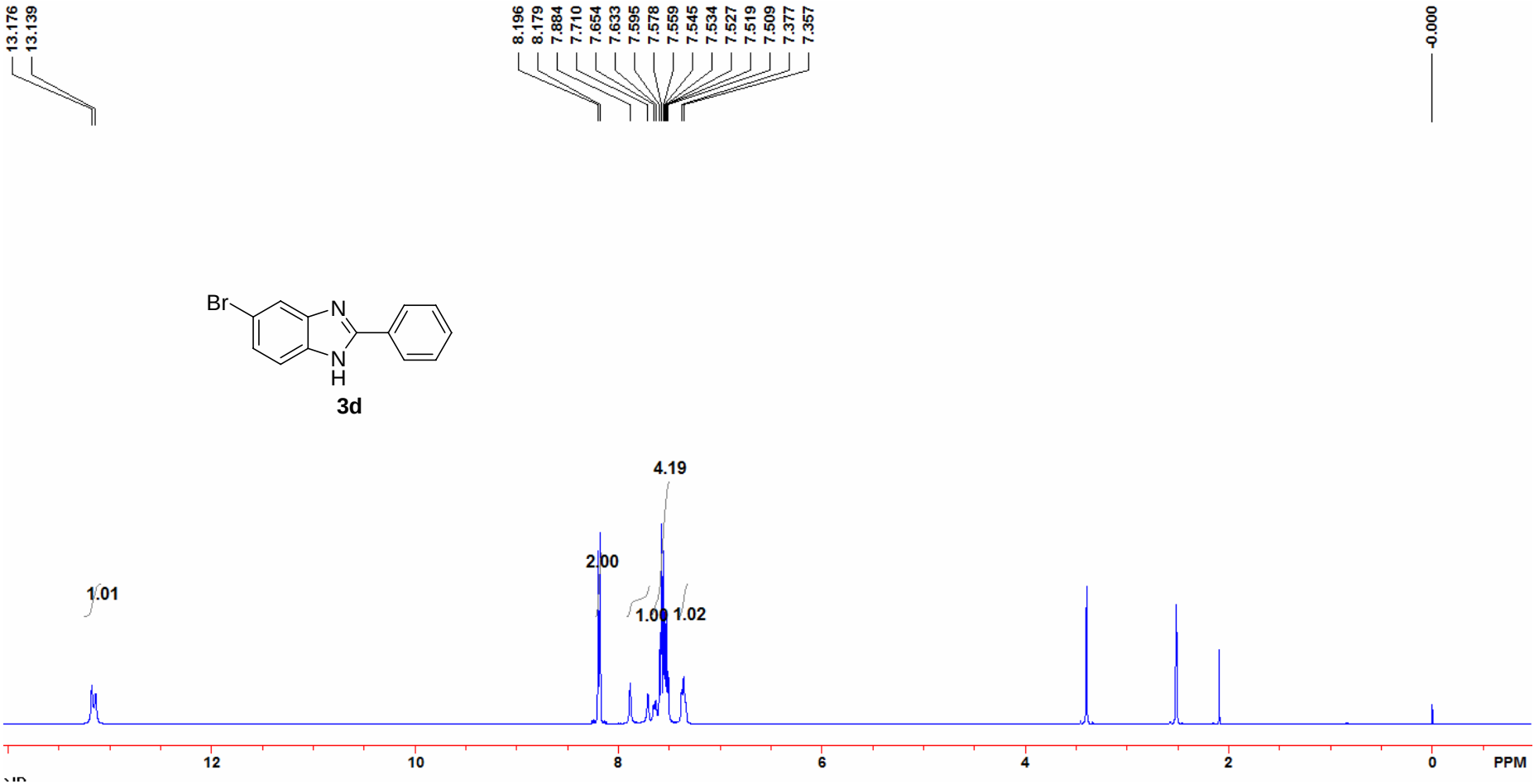

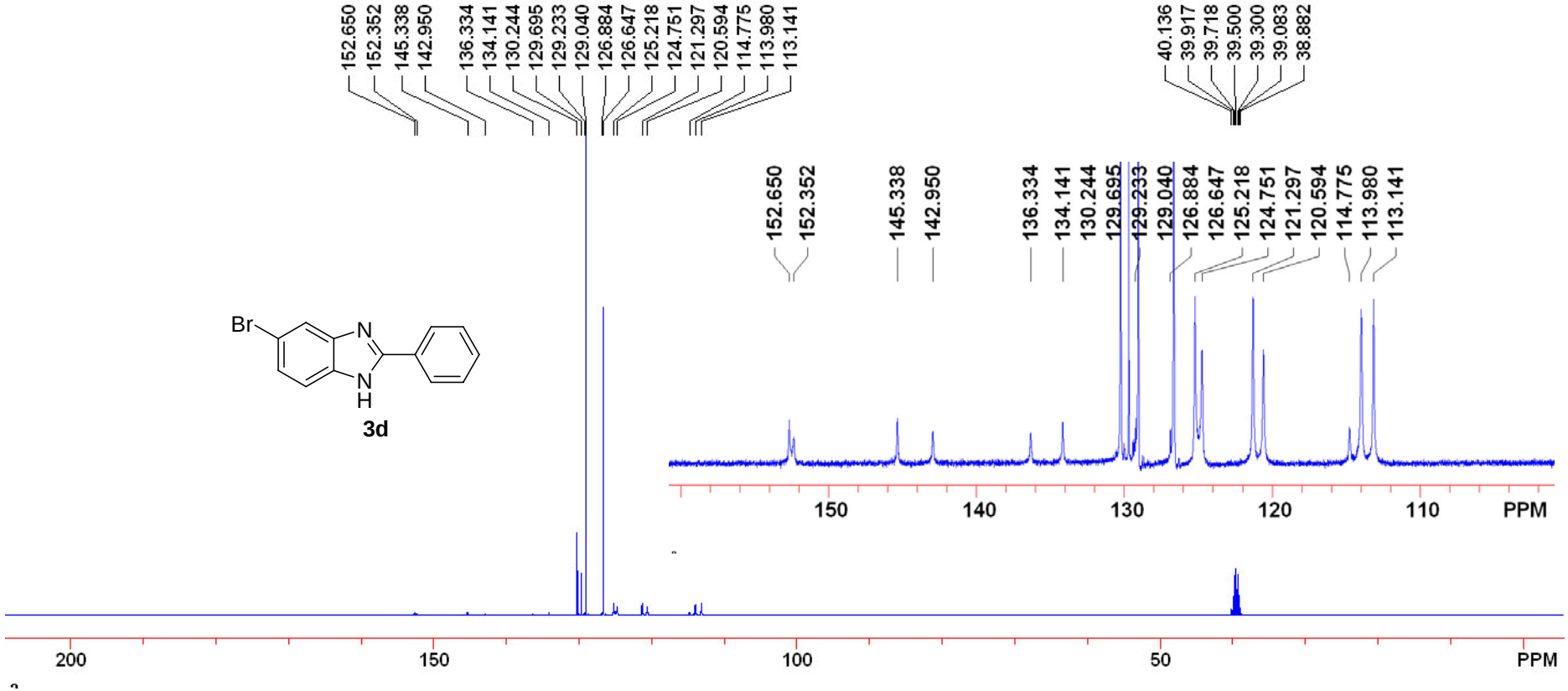

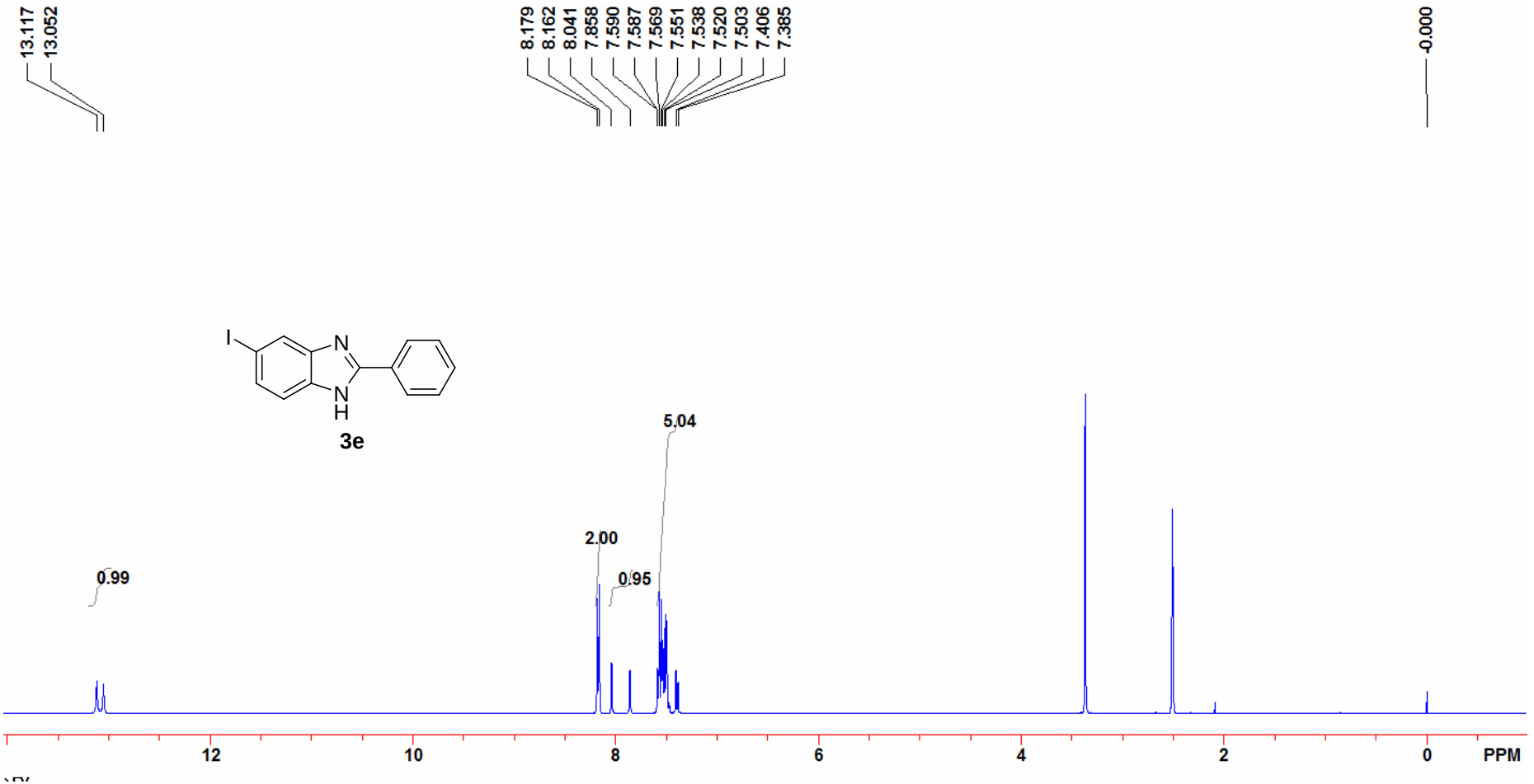

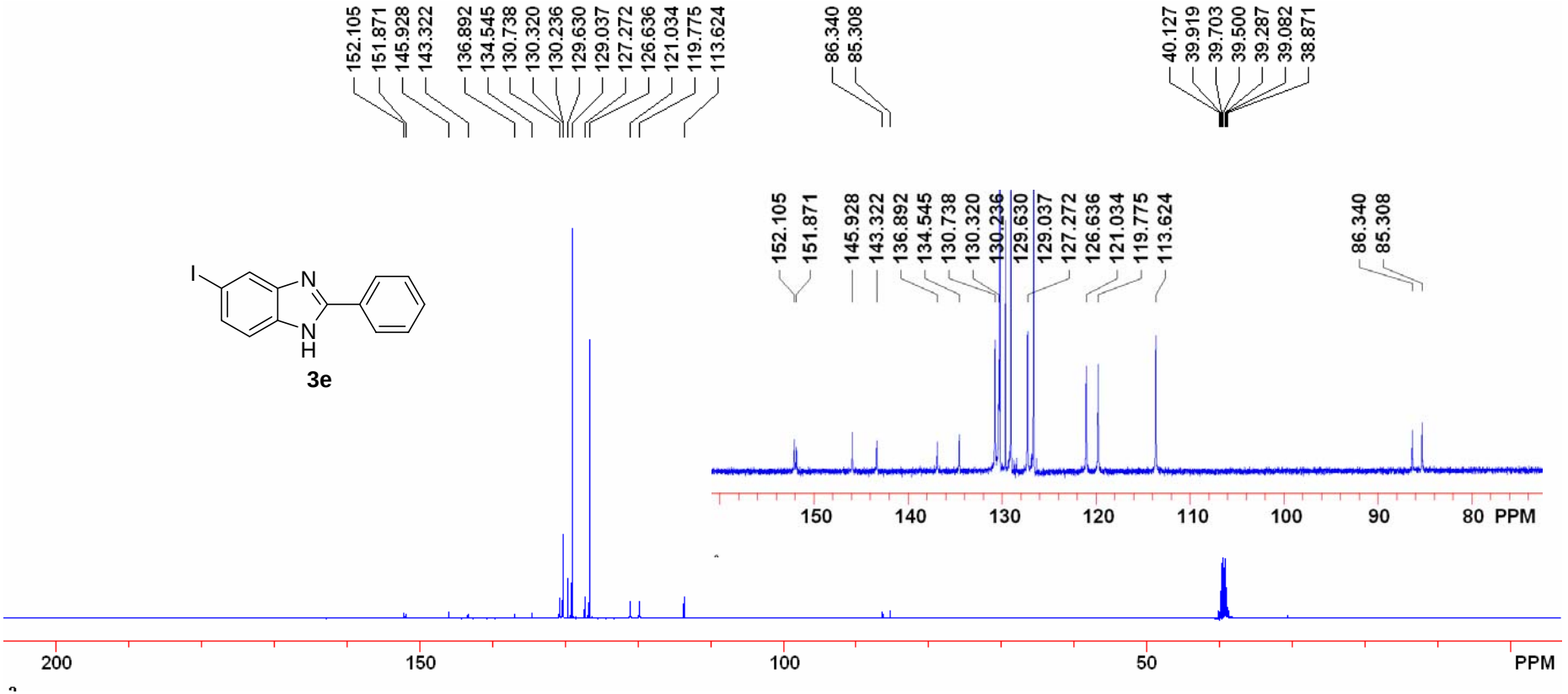

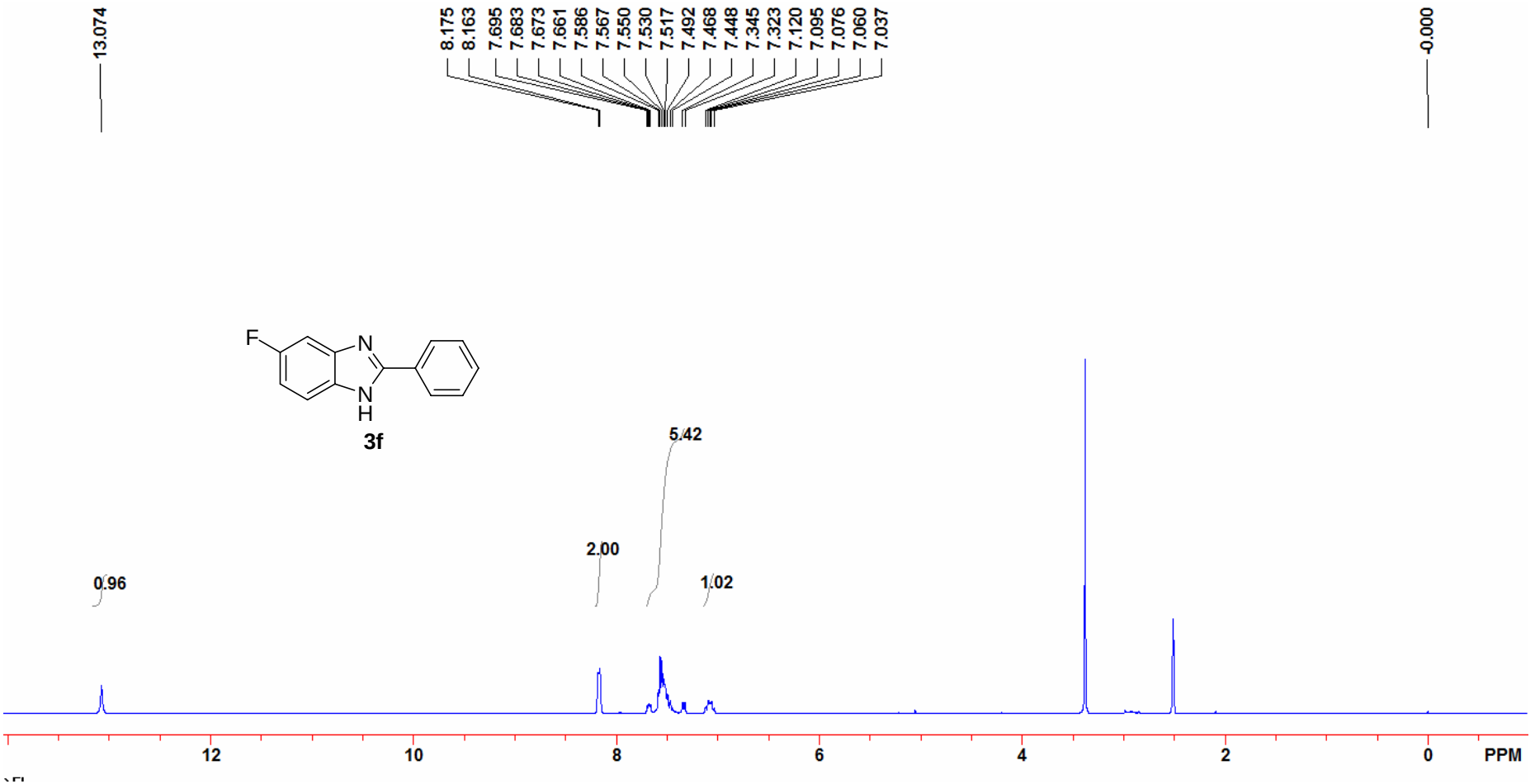

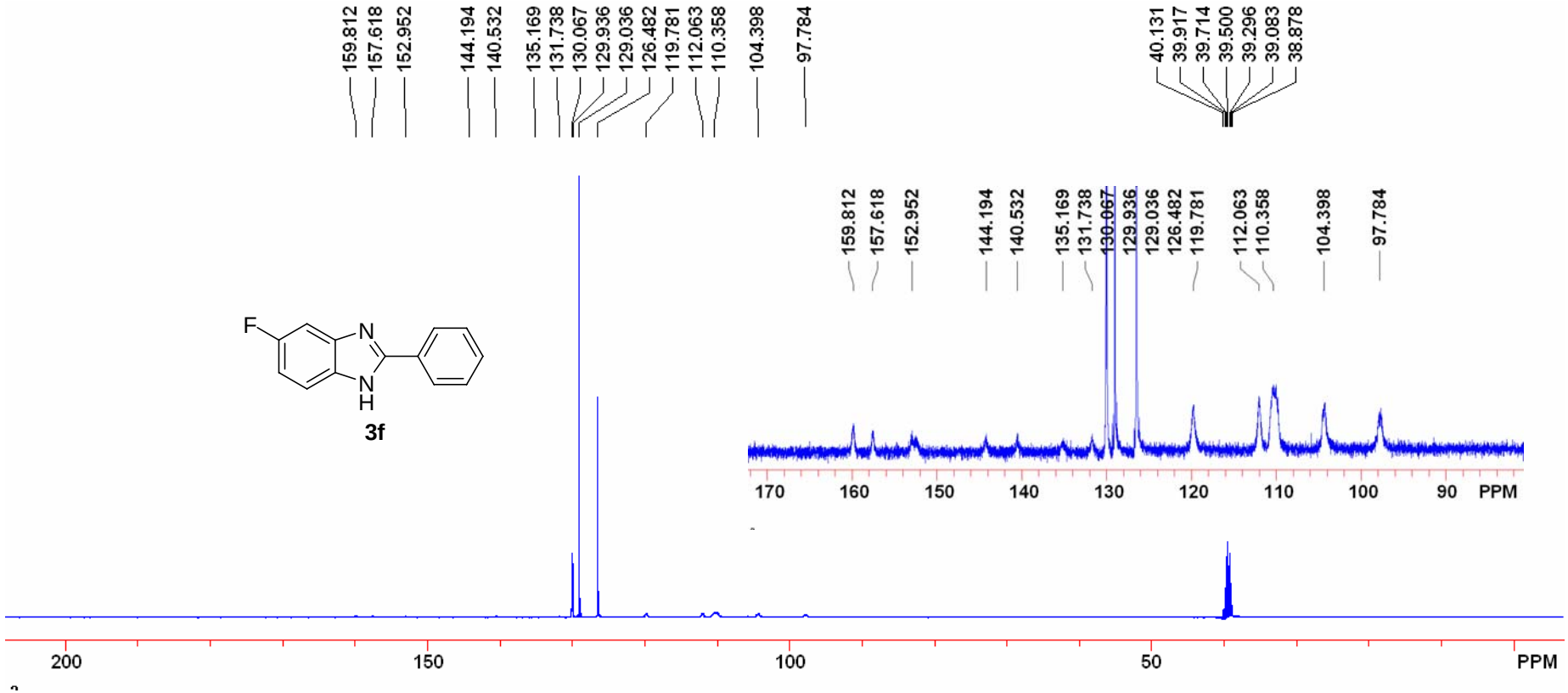

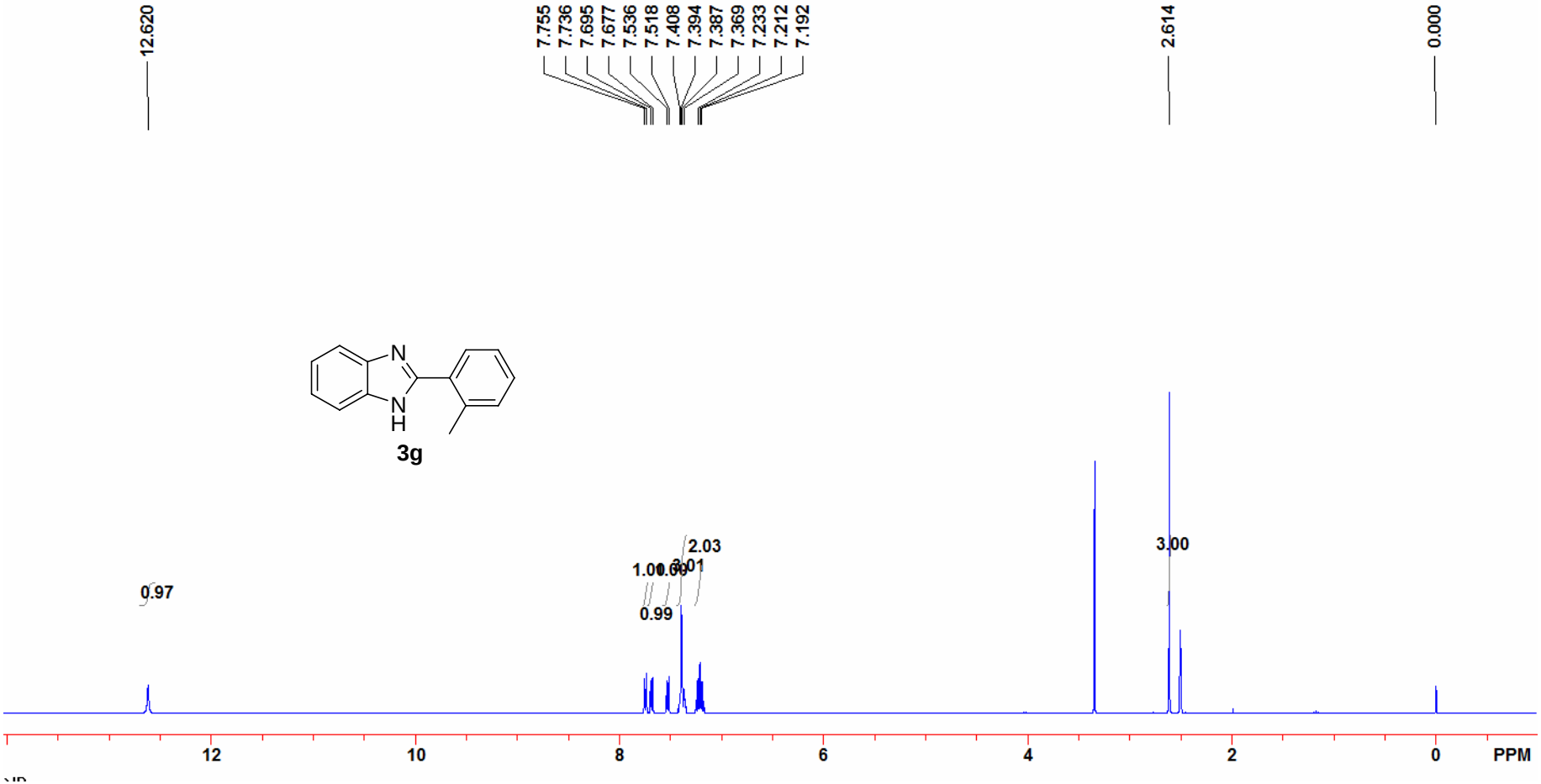

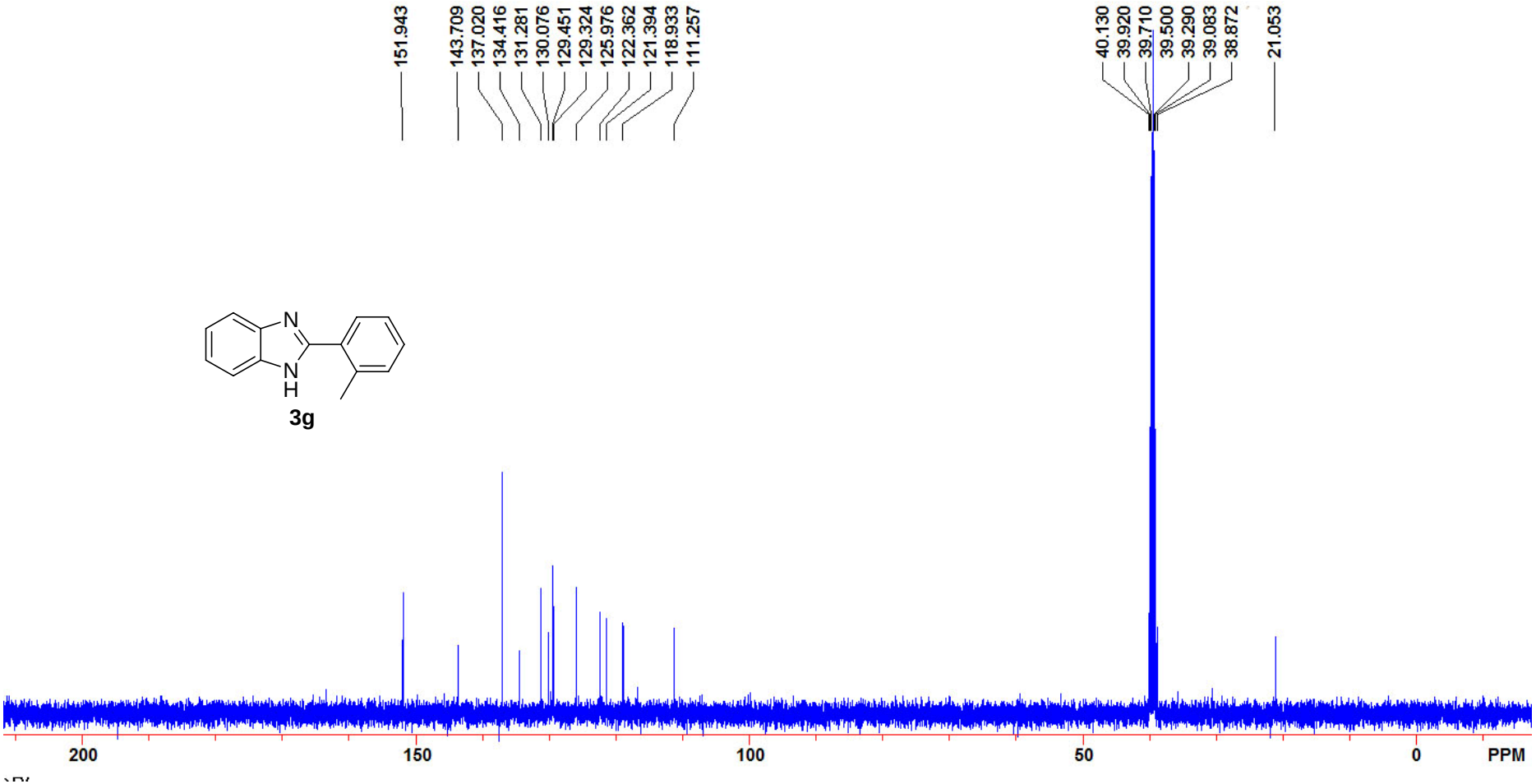

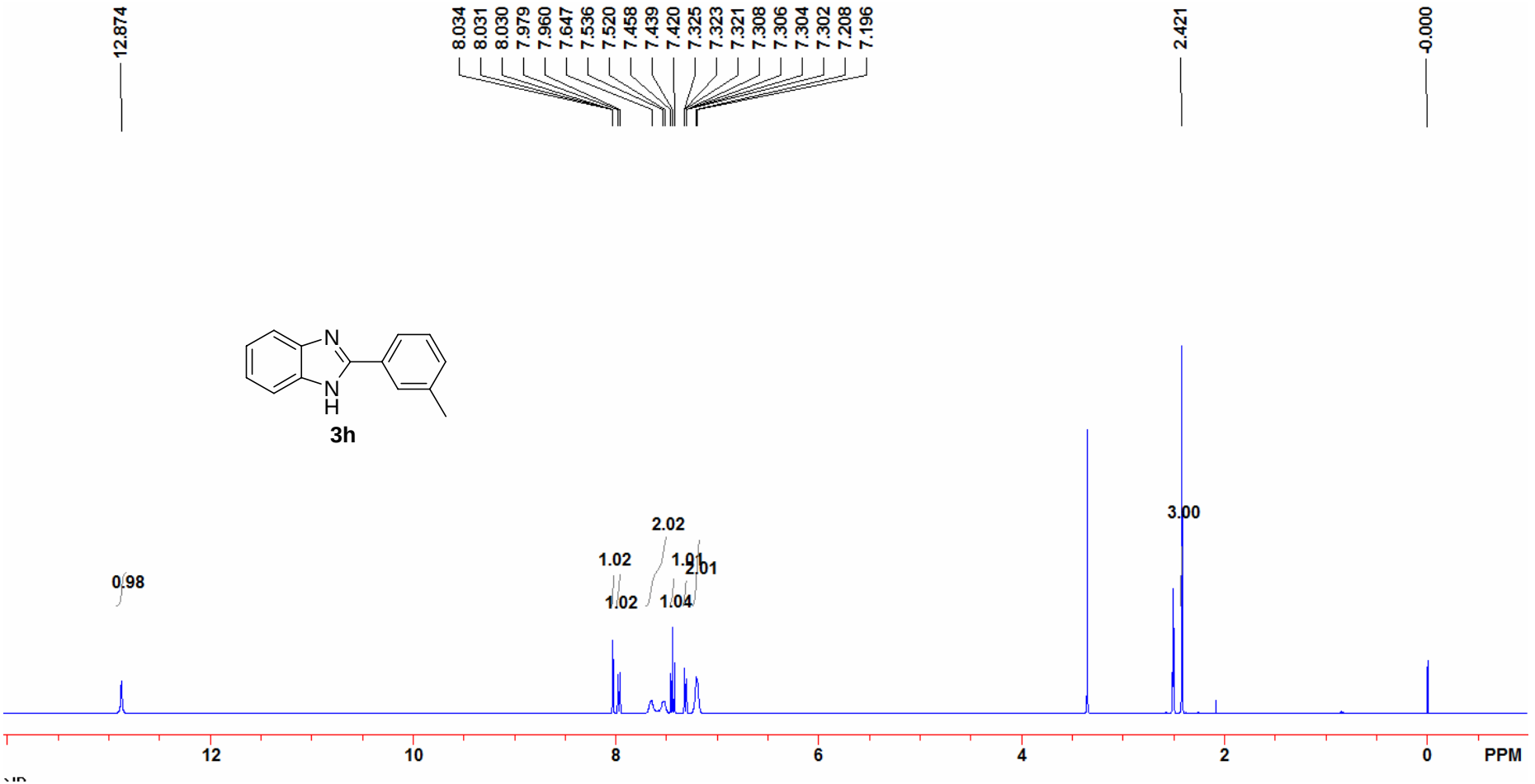

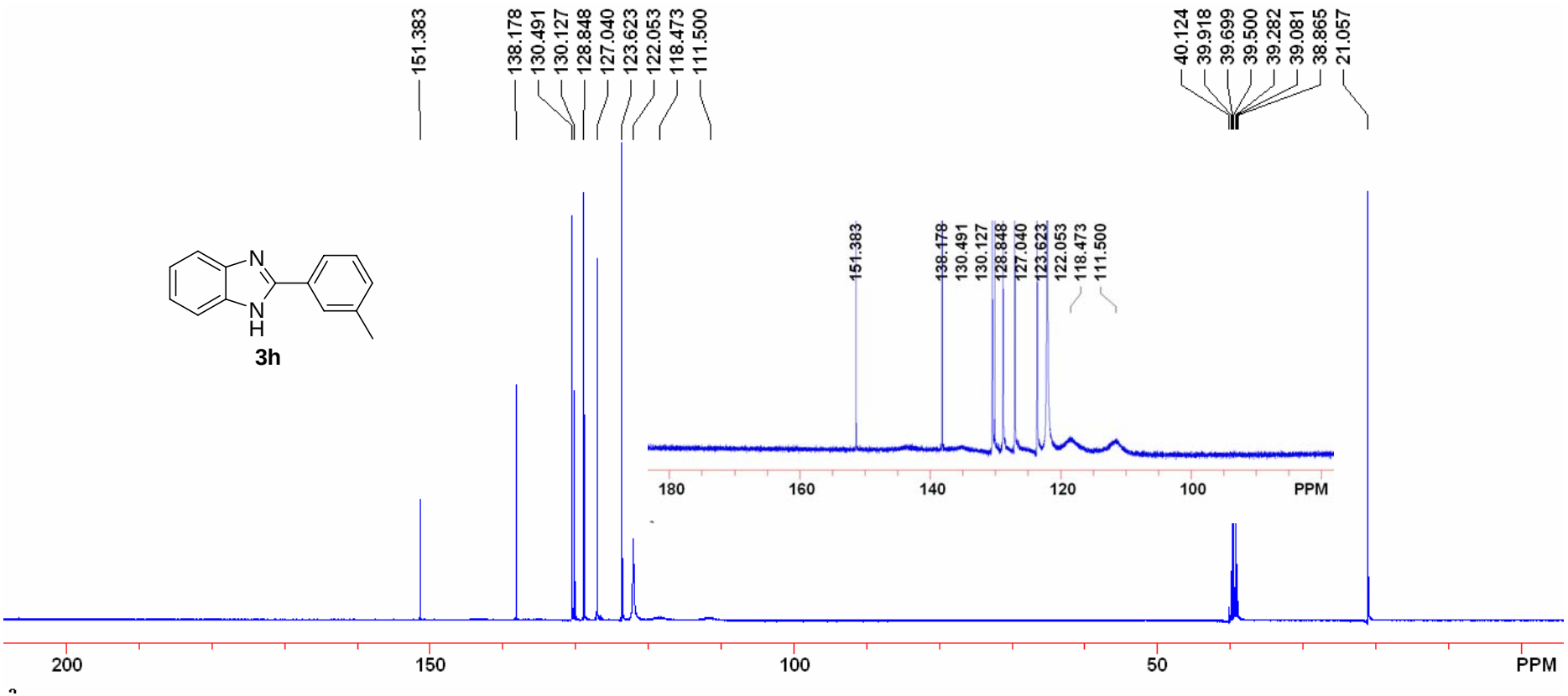

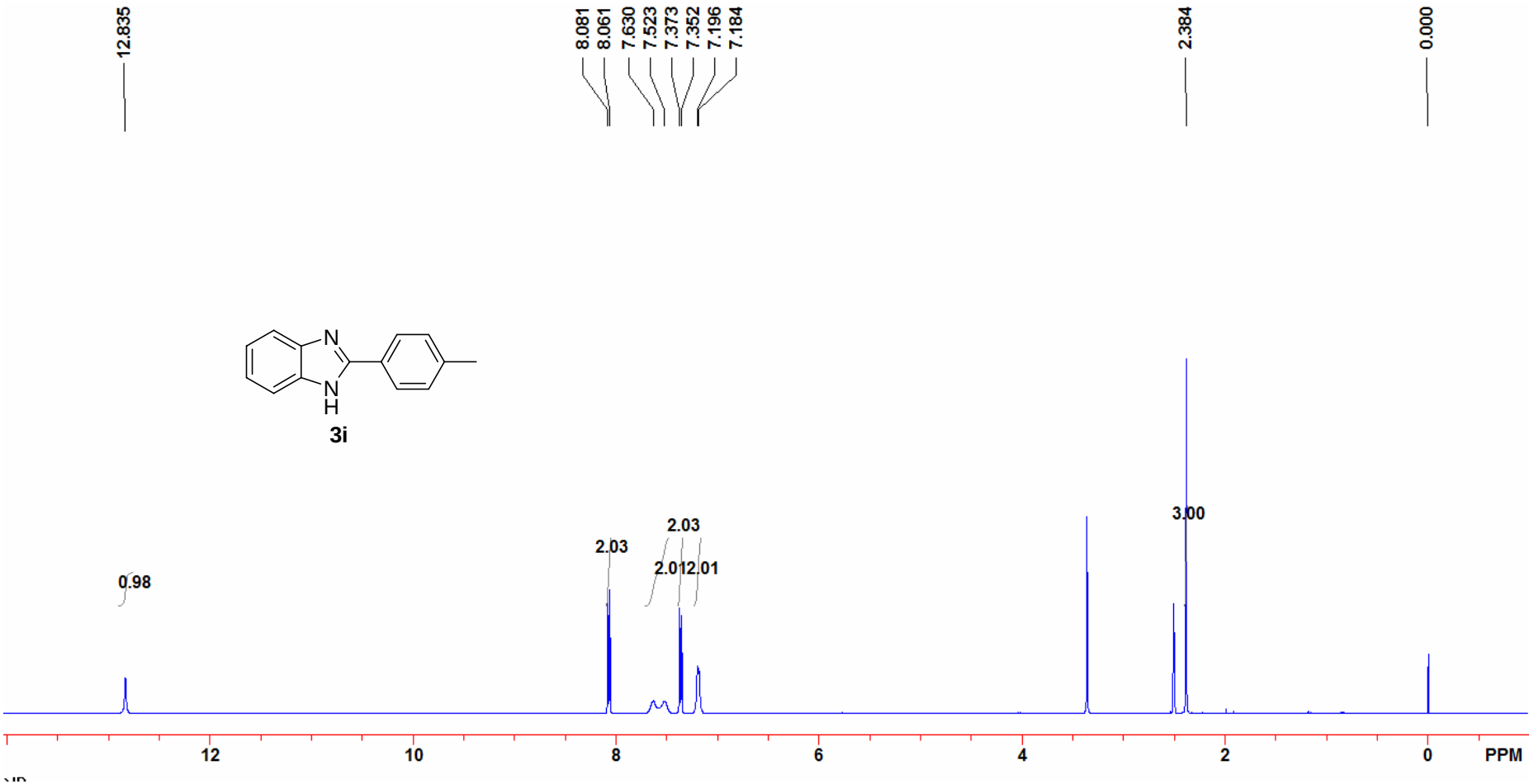

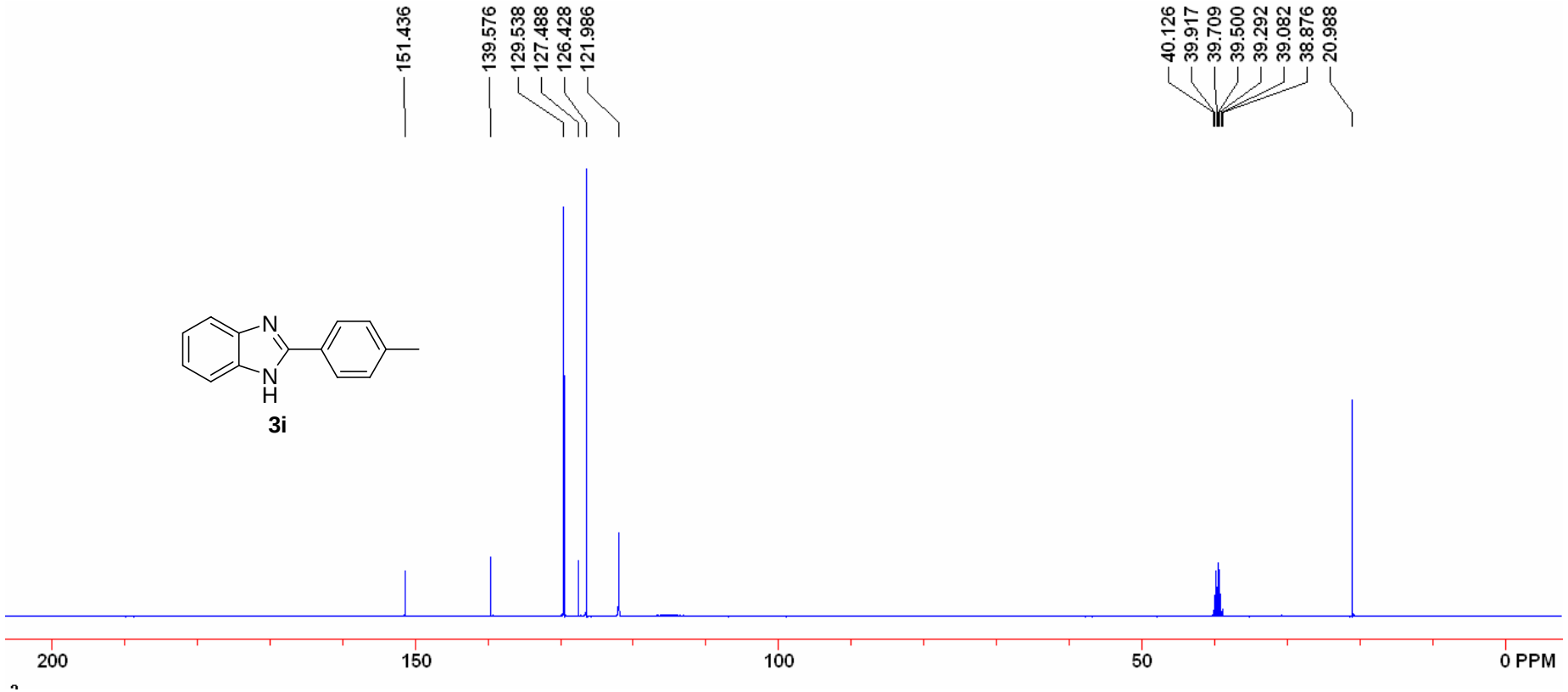

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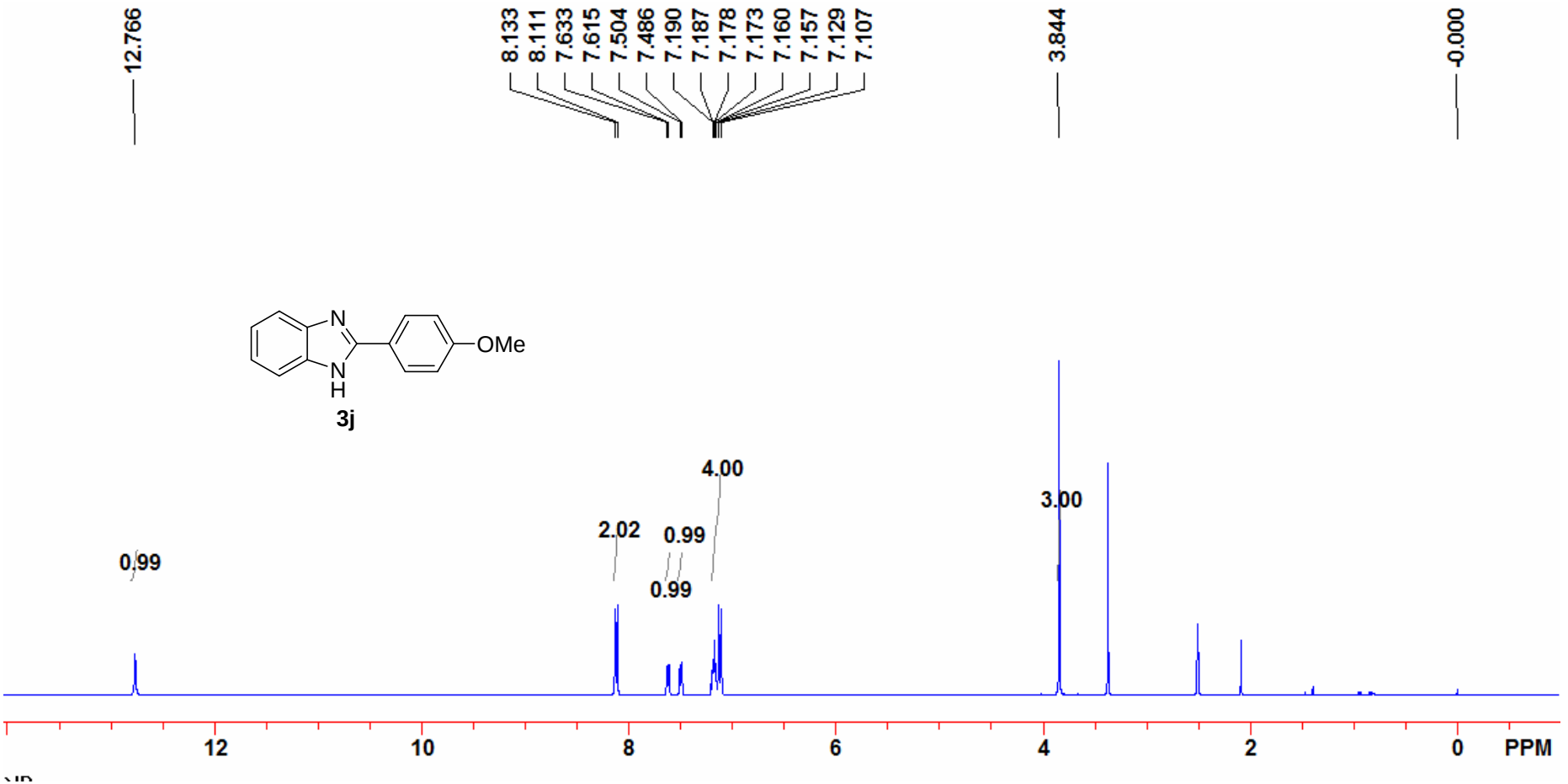

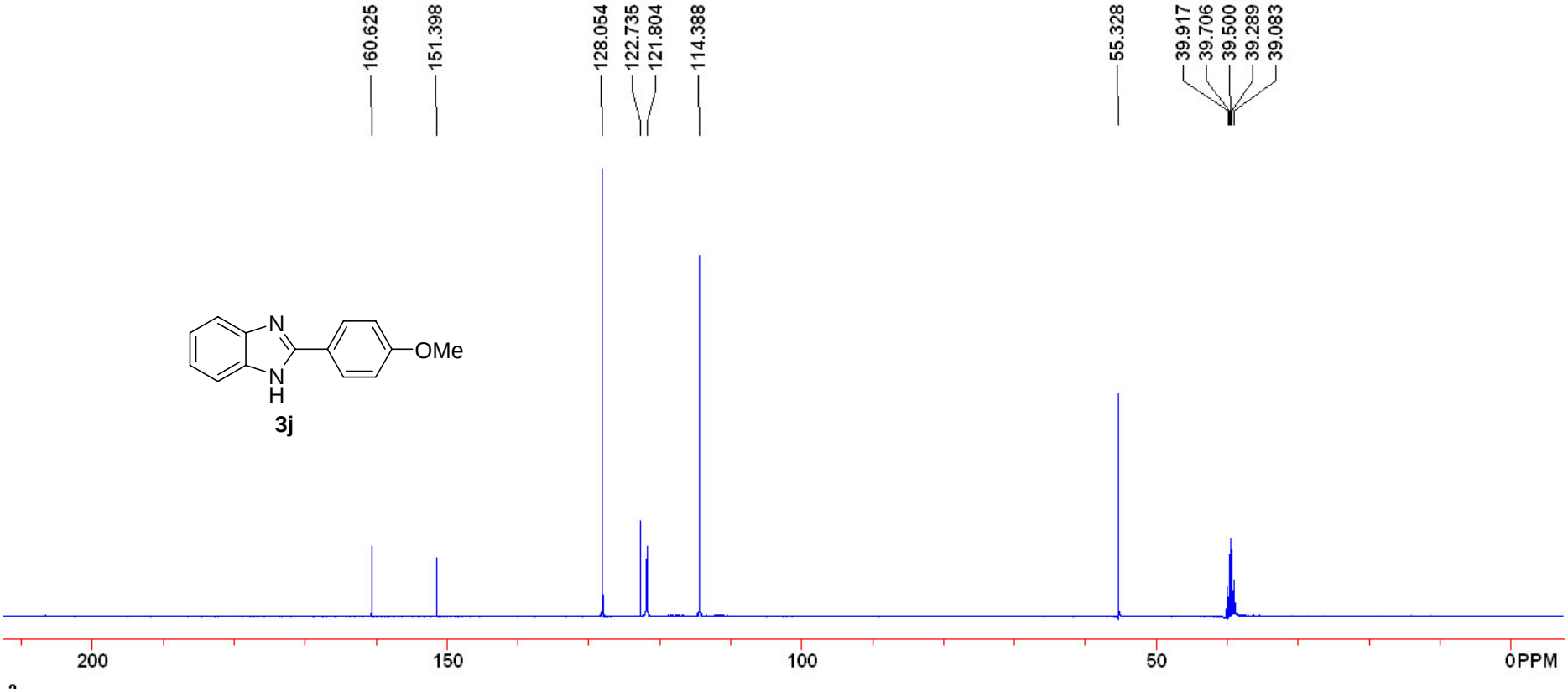

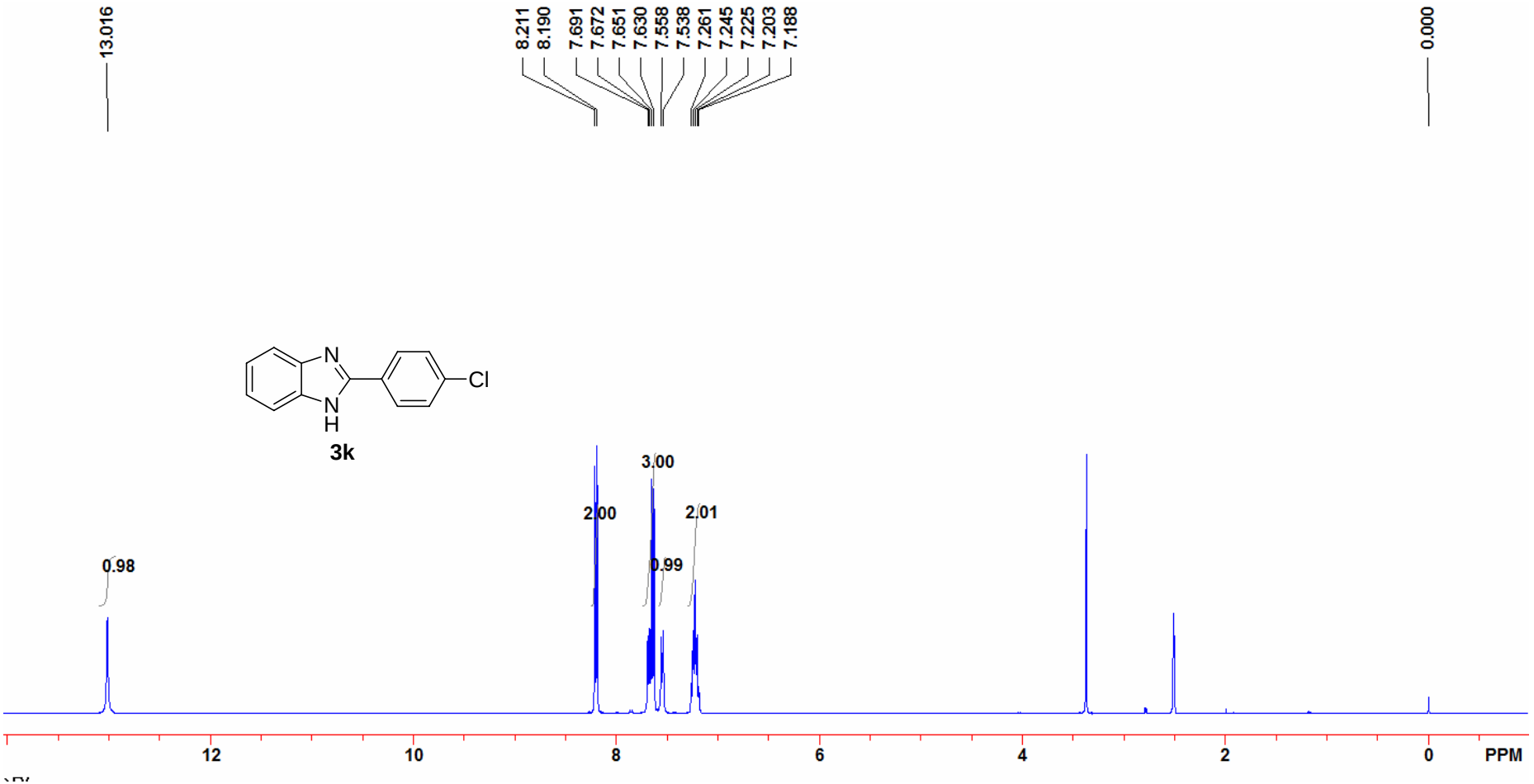

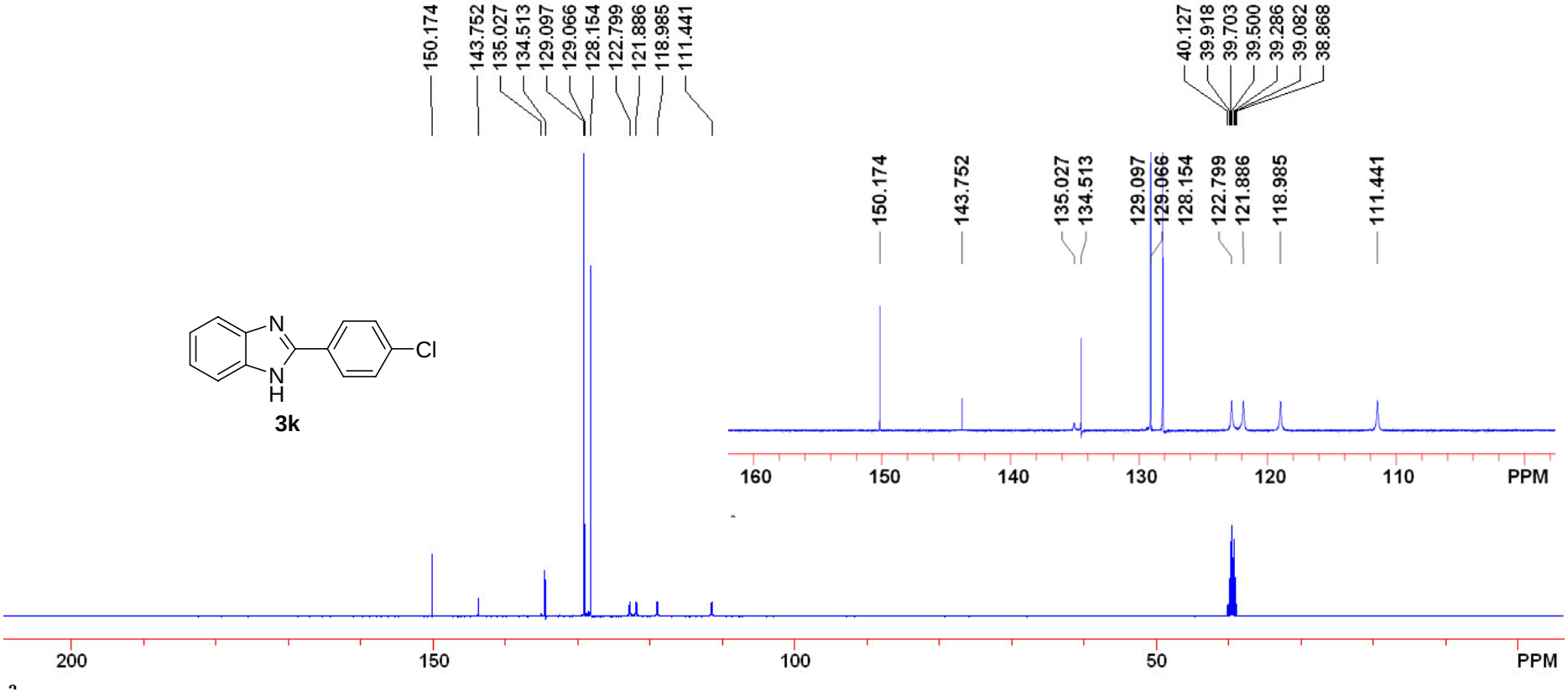

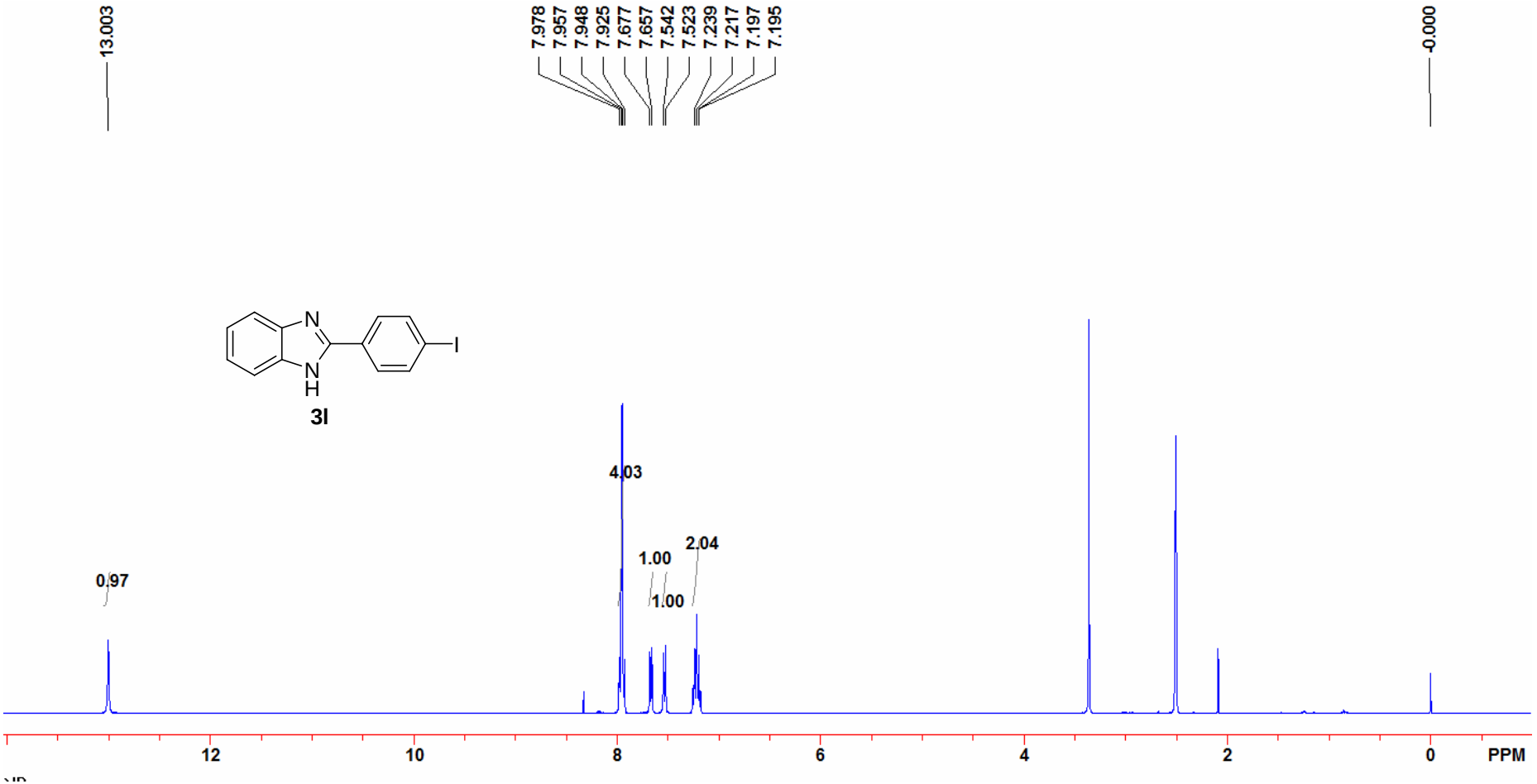

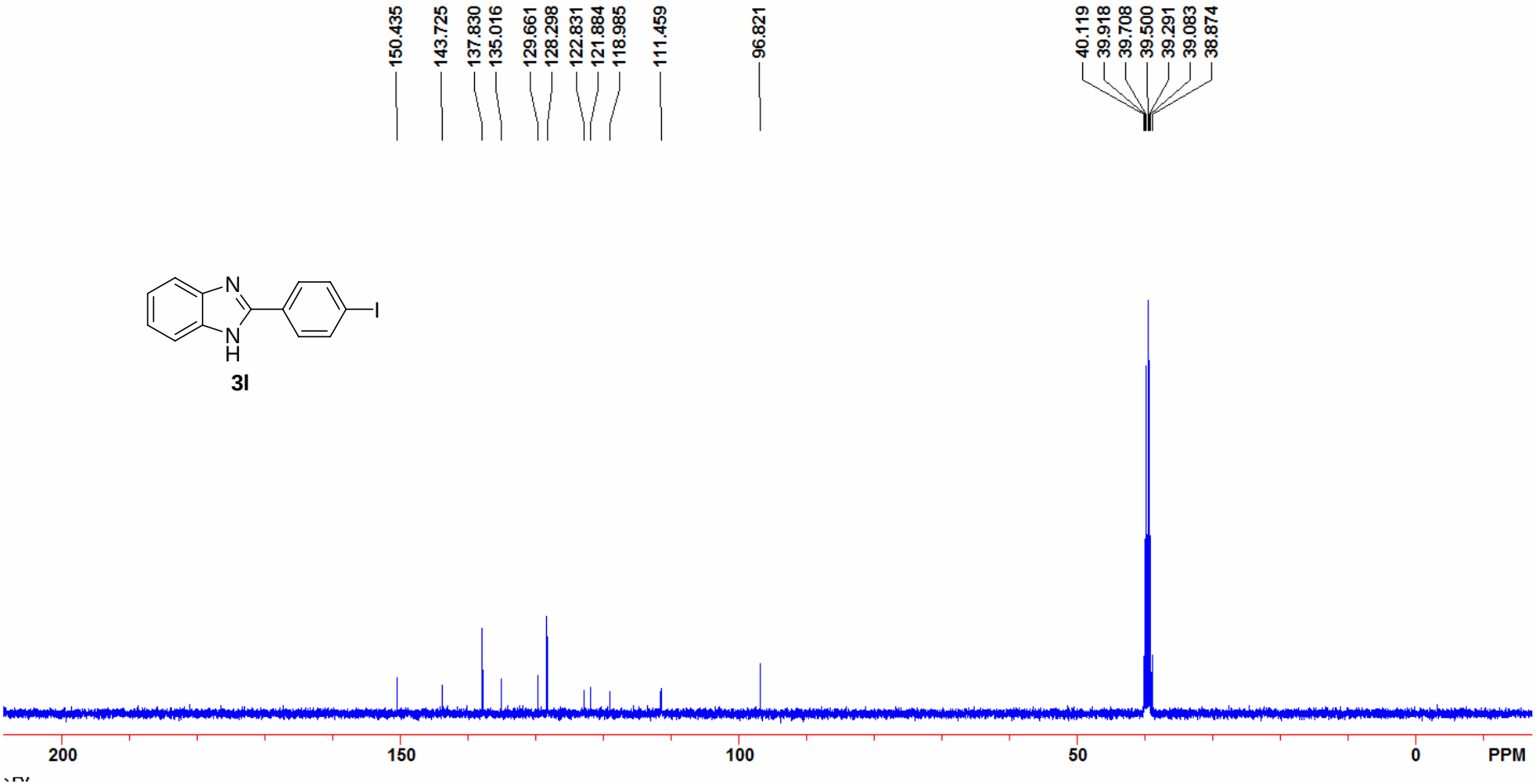

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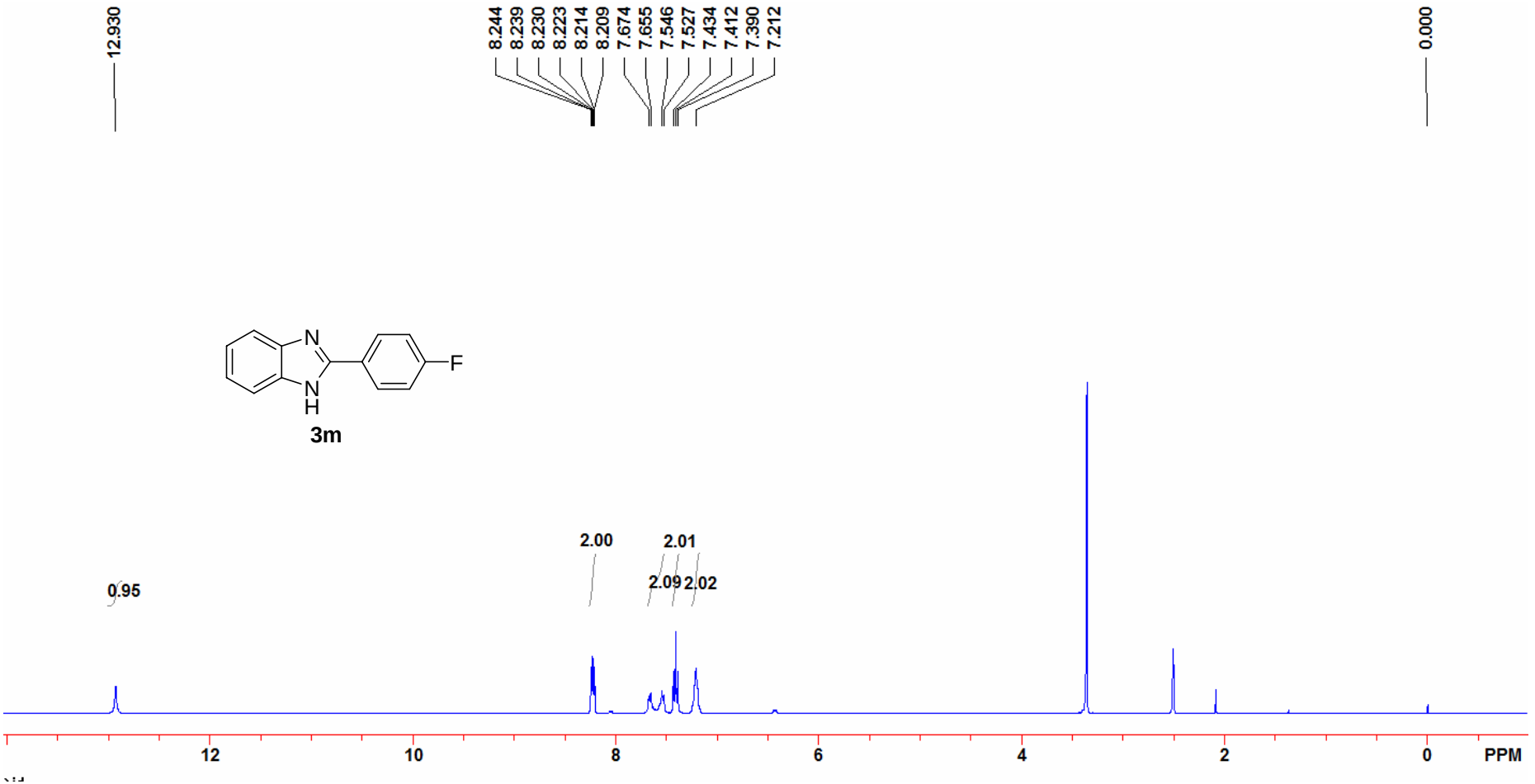

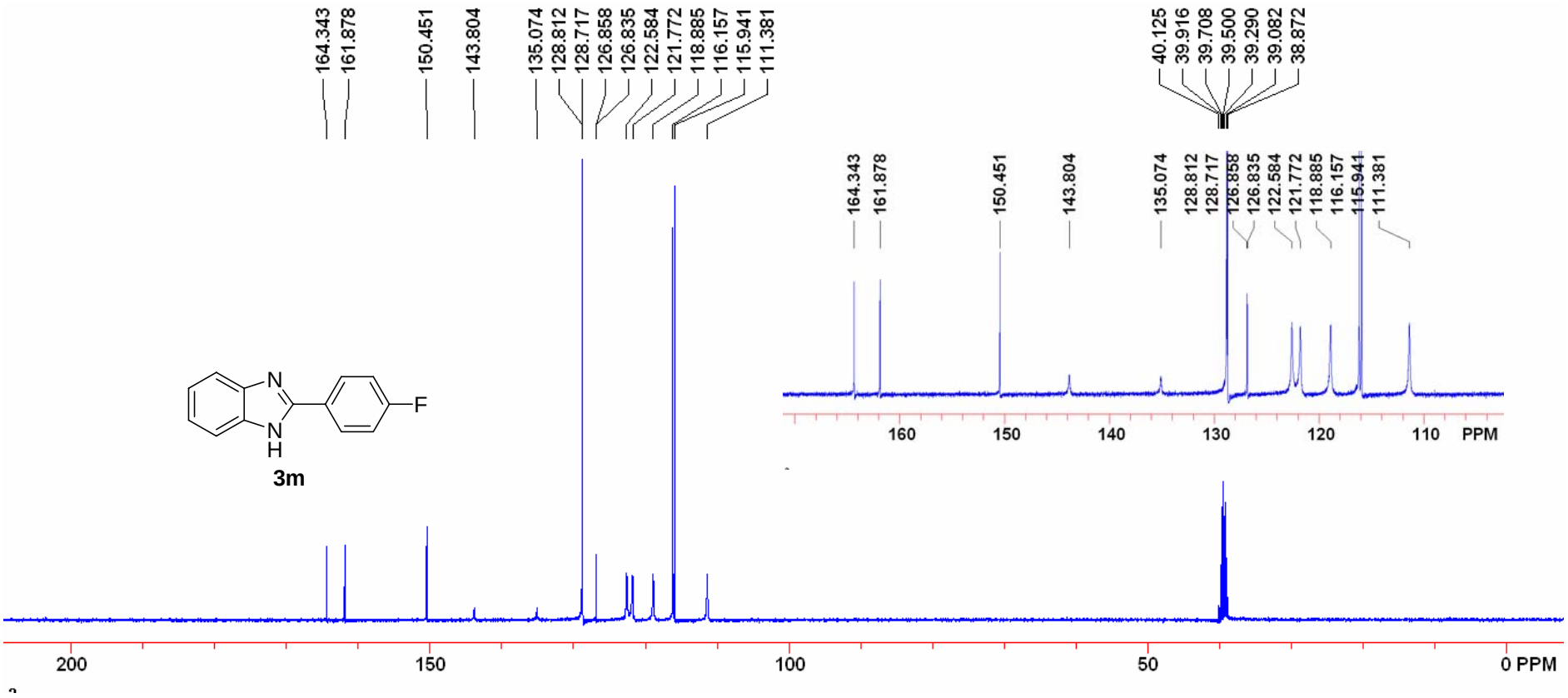

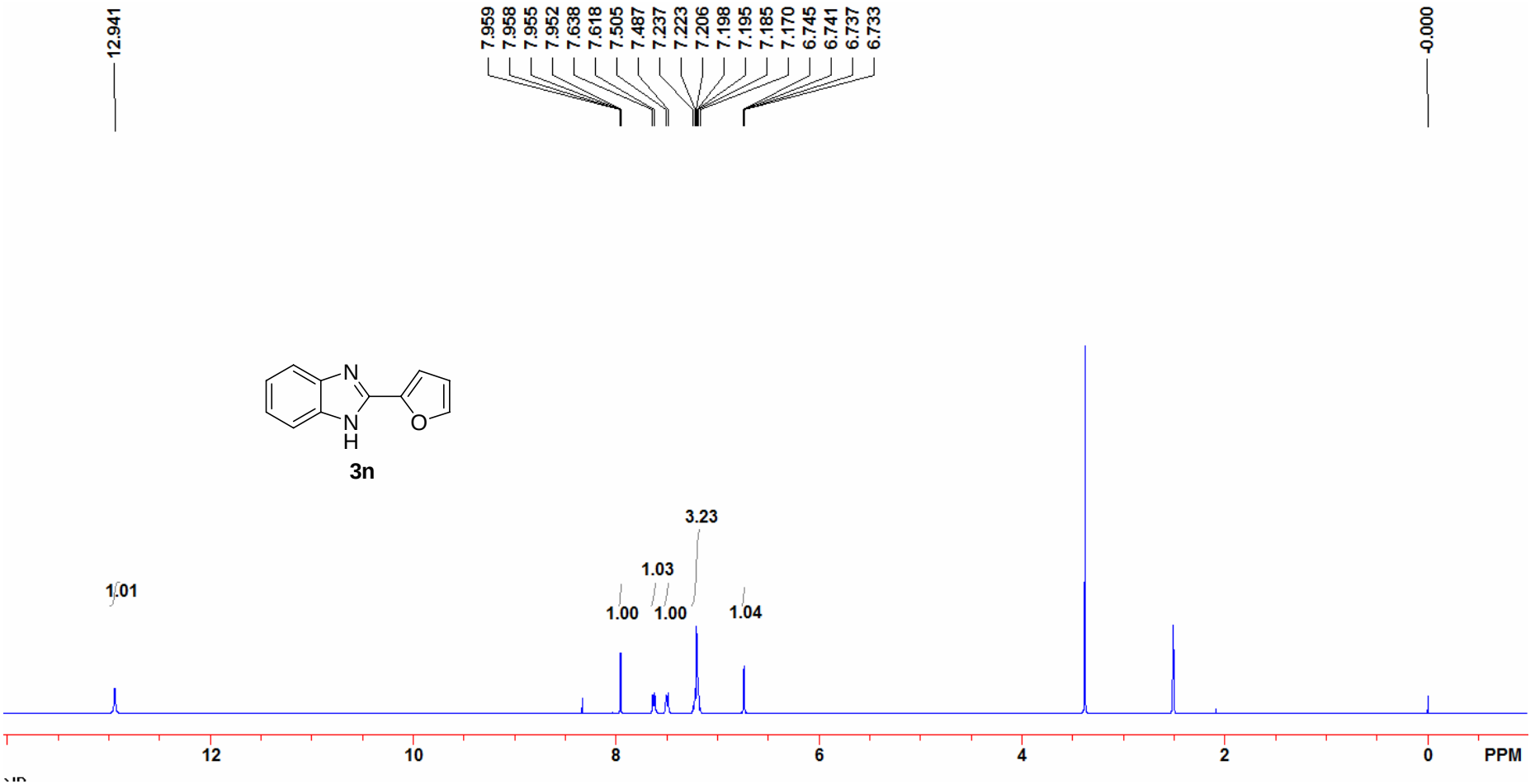

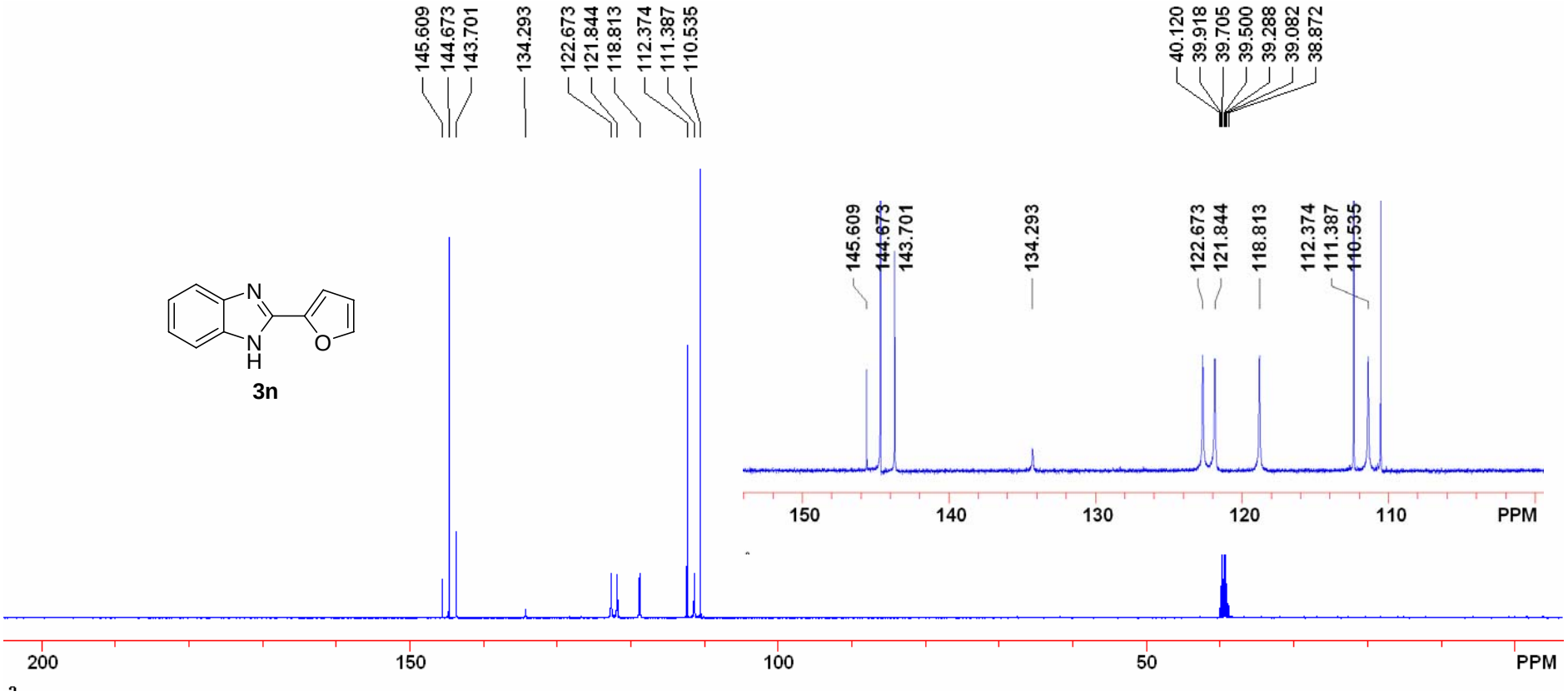

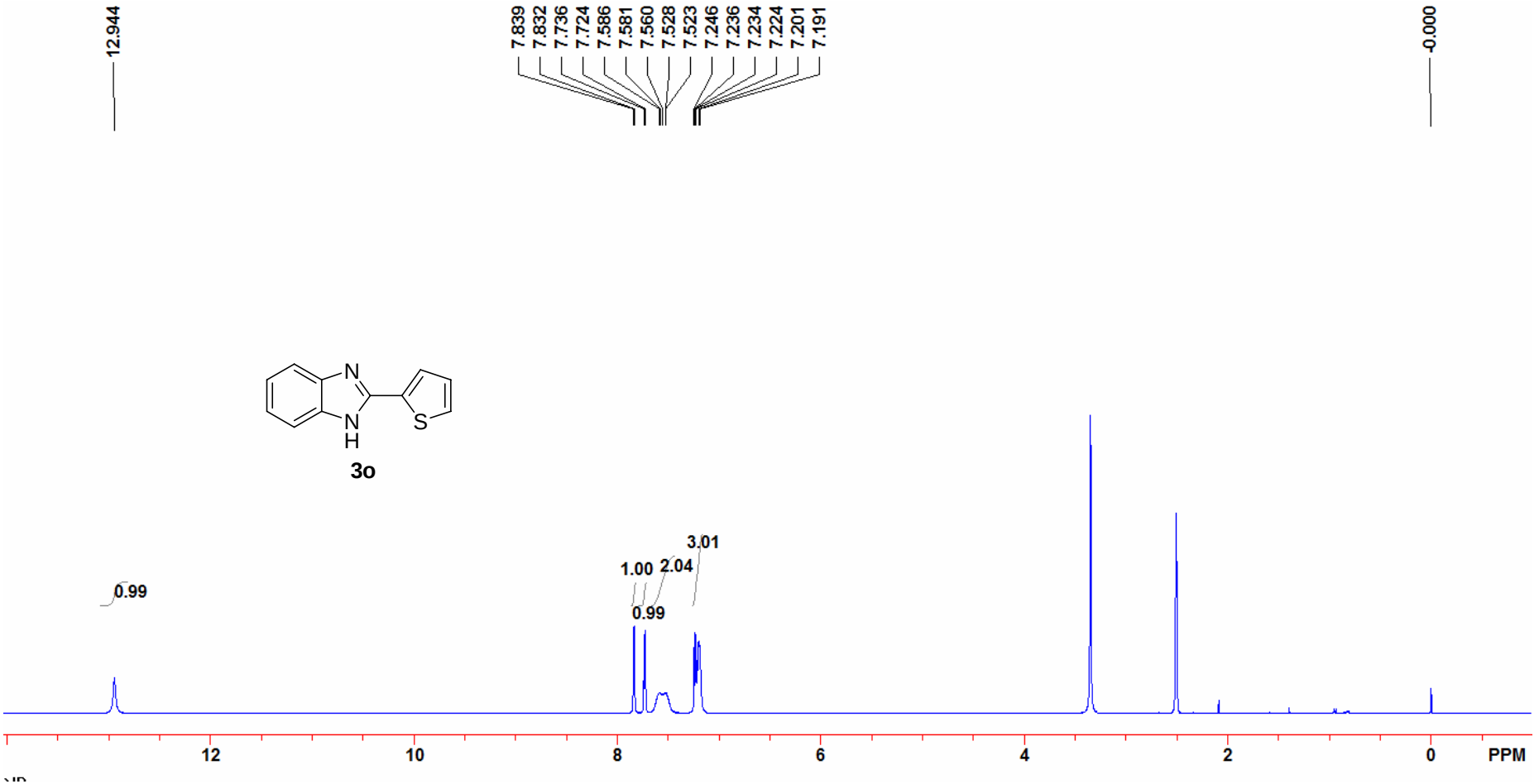

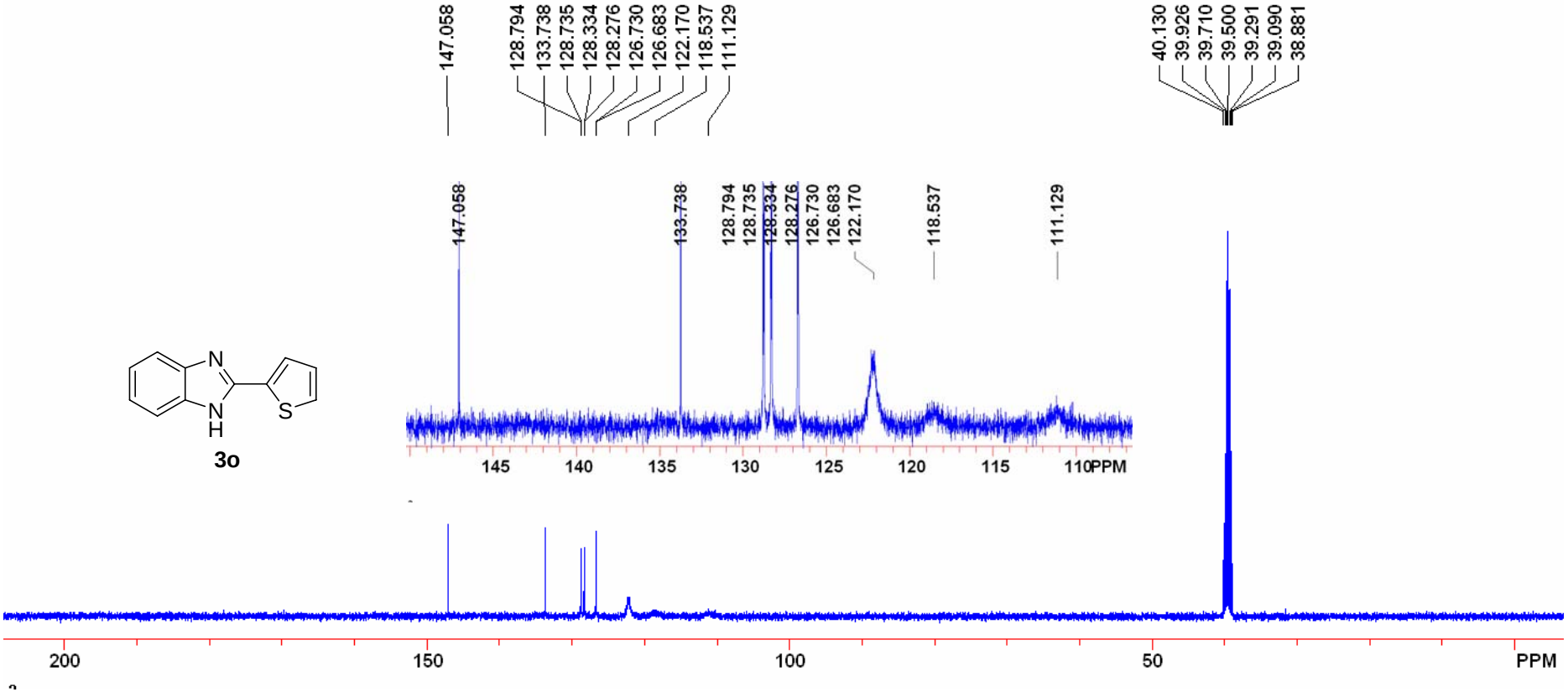

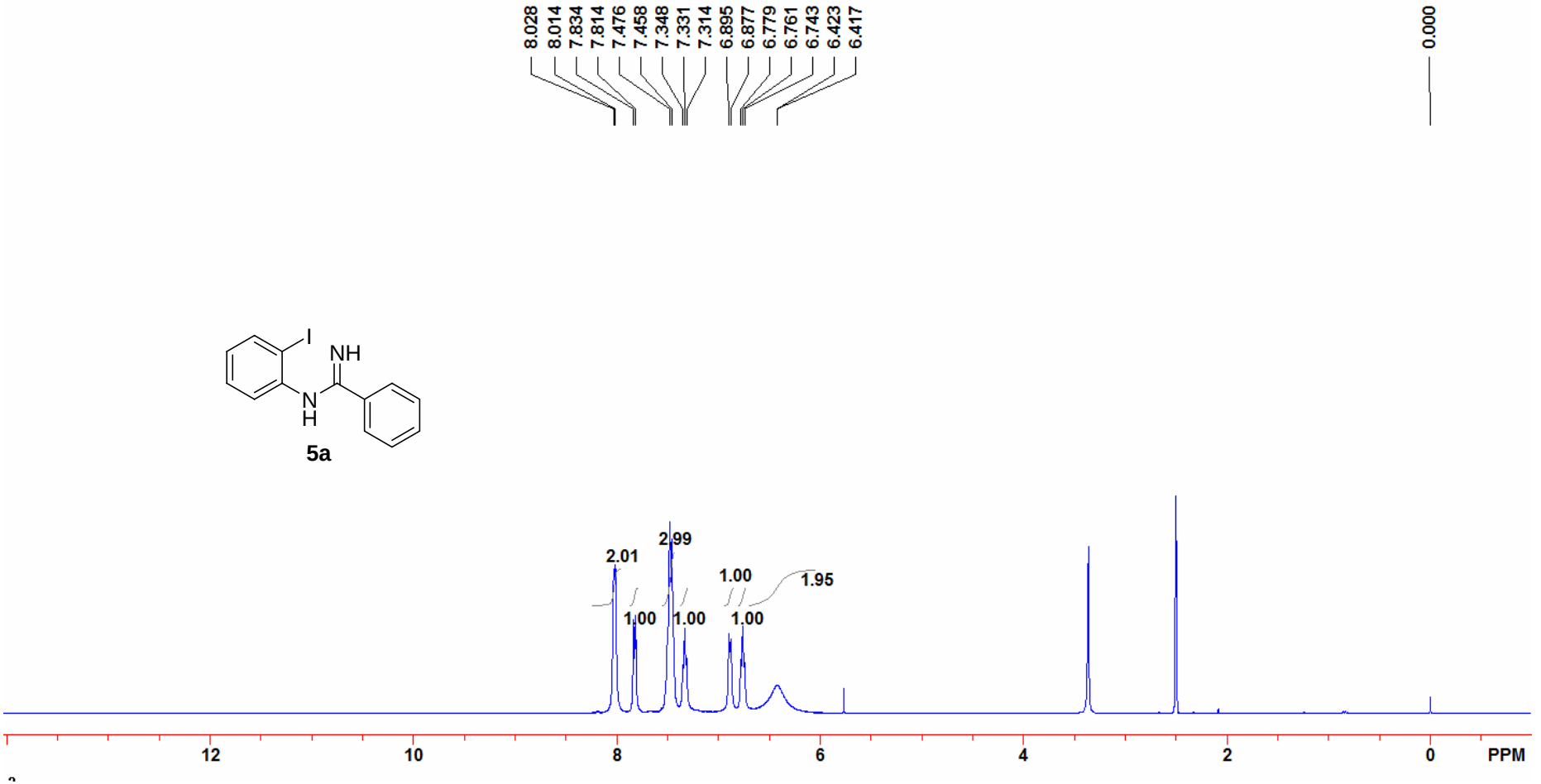

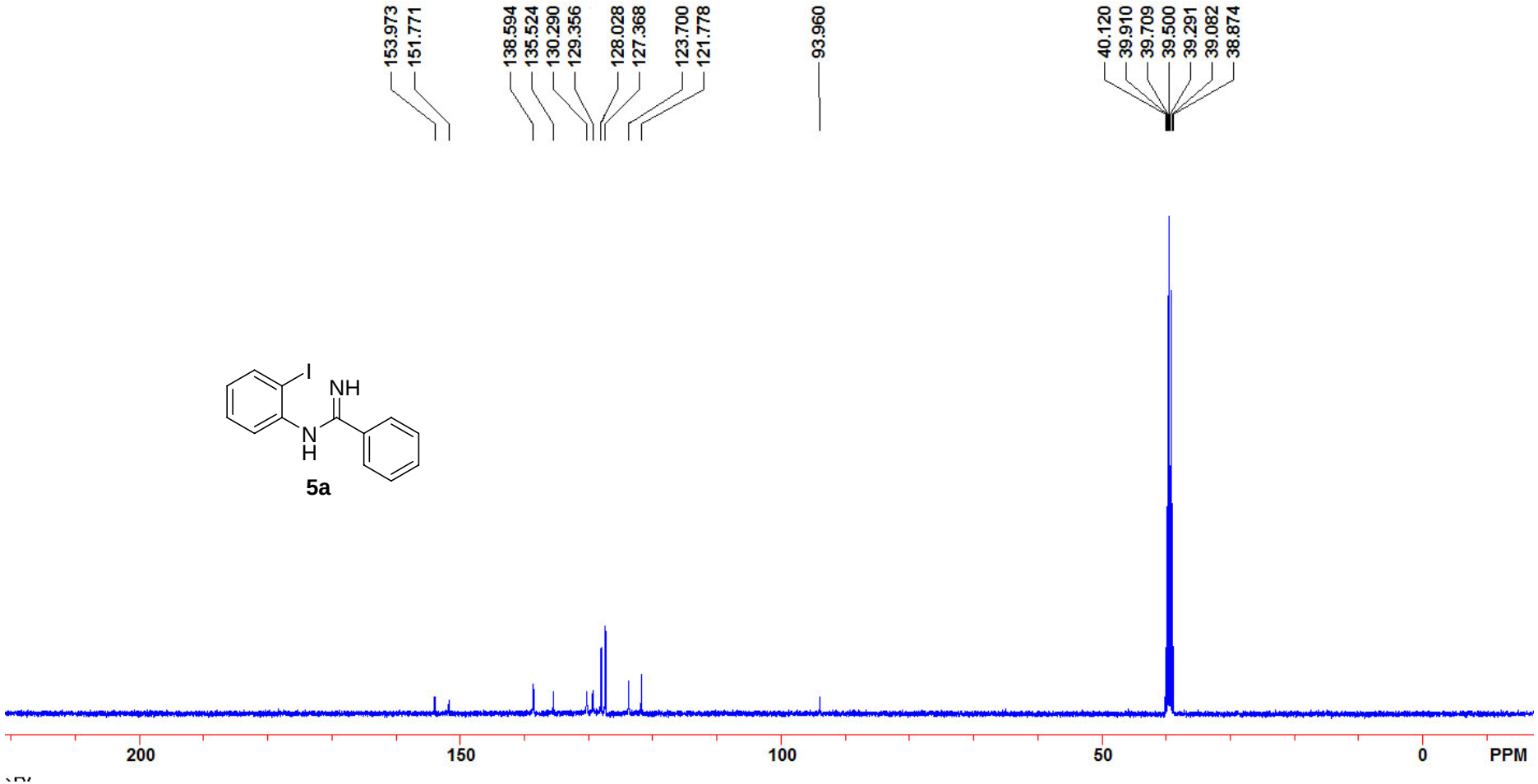

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