Supporting Information

Metal-free Synthesis of Substituted Pyridines from aldehydes and NH_4OAc under Air

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General remark

^{1}H NMR and ^{13}C NMR spectra were recorded on 400MHz and 100MHz in $CDCl_3$. All chemical shifts are given as δ value (ppm) with reference to tetramethylsilane (TMS) as an internal standard. All compounds were further characterized by HRMS; copies of their ^{1}H NMR and ^{13}C NMR spectra are provided. Products were purified by flash chromatography on 200–300 mesh silica gels. All melting points were determined without correction. Unless otherwise noted, commercially available reagents and solvents were used without further purification.

Experimental Section

General procedure for the synthesis of the desired pyridines 3 and 4.

An oven-dried tube was charged with 0.3 mmol of aldehydes **1(1″)**, 0.9 mmol of NH_4OAc **2**, $NaHCO_3$ (0.3 mmol) and 1 mL 1, 4-dioxane. Then the reaction was stirred at 90 ^{o}C under air and the reaction time was monitored by TLC. After cooling to room temperature, the solvent was diluted with 20 ml of ethyl acetate, washed with 10 ml of brine, and dried over anhydrous Na_2SO_4. Then the solvent was evaporated in vacuo, the residues were purified by column chromatography, eluting with petroleum ether/EtOAc to afford the disired pyridines.

The detail experiment information of 3p.

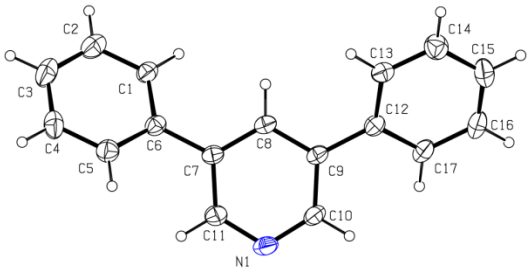
An oven-dried tube was charged with 0.3 mmol (40.2 mg) of 2-(p-tolyl)acetaldehyde **1b**, 0.3 mmol (45.0 mg) of 2-(4-methoxyphenyl)acetaldehyde **1g**, 0.9 mmol (69.3 mg) of NH_4OAc **2**, 0.3 mmol $NaHCO_3$ (25.2 mg) and 2 mL 1, 4-dioxane. Then the reaction was stirred at 90 ^{o}C under air and the reaction time was monitored by TLC. After cooling to room temperature, the solvent was diluted with 20 ml of ethyl acetate, washed with 10 ml of brine, and dried over anhydrous Na_2SO_4. Then the

solvent was evaporated in vacuo, the residues were purified by column chromatography, eluting with petroleum ether/EtOAc to afford **3p** of 18.5 mg (34% yield), **3b** of 10.1 mg (26% yield) and **3g** of 13.8mg (32% yield).

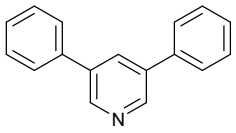
The detail experiment information of 3q.

An oven-dried tube was charged with 0.3 mmol (40.2 mg) of 2-(p-tolyl)acetaldehyde **1b**, 2-(3, 4-dimethoxyphenyl) acetaldehyde **1i**, 0.9 mmol (69.3 mg) of NH_4OAc **2**, 0.3 mmol $NaHCO_3$ (25.2 mg) and 2 mL 1, 4-dioxane. Then the reaction was stirred at 90 ^oC under air and the reaction time was monitored by TLC. After cooling to room temperature, the solvent was diluted with 20 ml of ethyl acetate, washed with 10 ml of brine, and dried over anhydrous Na_2SO_4. Then the solvent was evaporated in vacuo, the residues were purified by column chromatography, eluting with petroleum ether/EtOAc to afford **3q** of 21.9 mg (36% yield), **3b** of 8.2 mg (21% yield) and **3g** of 16.4 mg (38% yield).

X-ray data for 3a：

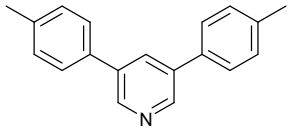


3,5-diphenylpyridine(3a)[1]



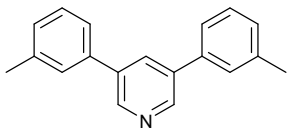
Yellow solid, melting point: 120-122 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.84 (s, 2H), 8.06 (t, *J* = 2.0 Hz, 1H), 7.66-7.64 (m, 4H), 7.53-7.42 (m, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 146.86, 137.74, 136.72, 133.01, 129.13, 128.25, 127.27. **HRMS calcd for** $C_{17}H_{14}N$ $[M+H]^+$ 232.1121, **found** 232.1126.

3,5-di-p-tolylpyridine(3b)[2]



Yellow solid, melting point: 175-177 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.78 (d, *J* = 2.0 Hz, 2H), 8.00 (t, *J* = 2.0 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 4H), 7.31 (d, *J* = 8.0 Hz, 4H), 2.42 (s, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 146.62, 138.11, 136.47, 134.94, 132.42, 129.81, 127.07, 21.16. **HRMS calcd for** $C_{19}H_{18}N$ $[M+H]^+$ 260.1434, **found** 260.1436.

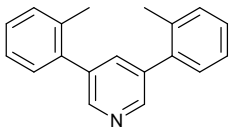
3,5-di-m-tolylpyridine(3c)[3]



Yellow solid, melting point: 136-138 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.79 (d, *J* = 2.0 Hz, 2H), 8.02 (t, *J* = 2.0 Hz, 1H), 7.45-7.37 (m, 6H), 7.25-7.23 (m, 2H), 2.45 (s, 6H). **^{13}C NMR (100 MHz,**

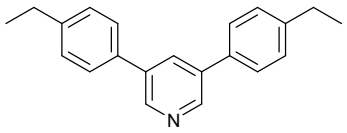
$CDCl_3$) δ 146.92, 138.79, 137.79, 136.67, 132.88, 129.00, 128.92, 127.99, 124.35, 21.51. **HRMS calcd for** $C_{19}H_{18}N$ $[M+H]^+$ 260.1434, **found** 260.1437.

3,5-di-o-tolylpyridine(3d)[2]



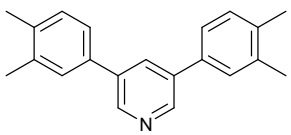
Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.58 (d, *J* = 2.0 Hz, 2H), 7.64 (t, *J* = 2.0 Hz, 1H), 7.31-7.26 (m, 8H), 2.33 (s, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 148.20, 137.89, 136.99, 136.71, 135.59, 130.59, 129.90, 128.13, 126.10, 20.42. **HRMS calcd for** $C_{19}H_{18}N$ $[M+H]^+$ 260.1434, **found** 260.1436.

3,5-bis(4-ethylphenyl)pyridine(3e)



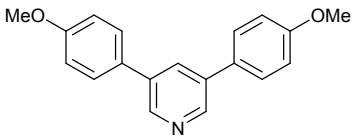
Yellow solid, melting point: 131-133 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.78 (d, *J* = 2.0 Hz, 2H), 8.02 (t, *J* = 2.0 Hz, 1H), 7.56 (d, *J* = 8.4 Hz, 4H), 7.33 (d, *J* = 8.4 Hz, 4H), 2.72 (q, *J* = 7.6 Hz, 4H), 1.29 (t, *J* = 7.6 Hz, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 146.63, 144.45, 136.50, 135.20, 132.49, 128.63, 127.16, 28.56, 15.51. **HRMS calcd for** $C_{21}H_{22}N$ $[M+H]^+$ 288.1747, **found** 288.1744.

3,5-bis(3,4-dimethylphenyl)pyridine(3f)



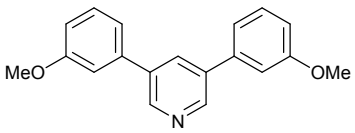
Yellow solid, melting point: 144-146 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.76 (d, *J* = 2.0 Hz, 2H), 8.00 (t, *J* = 2.0 Hz, 1H), 7.41-7.36 (m, 4H), 7.26-7.24 (m, 2H), 2.36-2.33 (d, 12H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 146.54, 137.34, 136.72, 136.52, 135.43, 132.43, 130.34, 128.41, 124.57, 19.90, 19.47. **HRMS calcd for** $C_{21}H_{22}N$ $[M+H]^+$ 288.1747, **found** 288.1749.

3,5-bis(4-methoxyphenyl)pyridine(3g)[4]



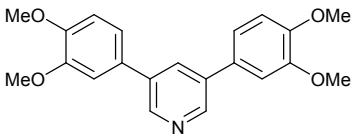
Yellow solid, melting point: 60-62 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.73 (d, *J* = 2.0 Hz, 2H), 7.96 (t, *J* = 2.0 Hz, 1H), 7.59-7.56 (m, 4H), 7.04-7.02 (m, 4H), 3.87 (s, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 159.82, 146.08, 136.15, 131.92, 130.27, 128.31, 114.57, 55.34. **HRMS calcd for** $C_{19}H_{18}NO_2$ $[M+H]^+$ 292.1332, **found** 292.1337.

3,5-bis(3-methoxyphenyl)pyridine(3h)[4]



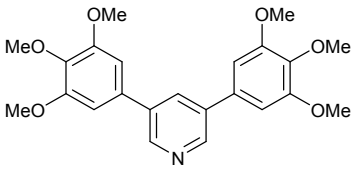
Yellow solid, melting point: 108-110 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.80 (d, *J* = 2.0 Hz, 2H), 8.03 (t, *J* = 2.0 Hz, 1H), 7.44-7.40 (m, 2H), 7.26-7.16 (m, 4H), 6.98-6.96 (m, 2H), 3.88 (s, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 160.15, 147.12, 139.17, 136.47, 132.95, 130.16, 119.67, 113.54, 113.03, 55.37. **HRMS calcd for** $C_{19}H_{18}NO_2$ $[M+H]^+$ 292.1332, **found** 292.1335.

3,5-bis(3,4-dimethoxyphenyl)pyridine(3i)



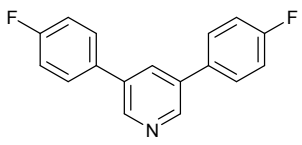
Yellow solid, melting point: 141-143 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.75 (d, *J* = 2.0 Hz, 2H), 7.96 (t, *J* = 2.0 Hz, 1H), 7.20 (dd, *J* = 8.0 Hz, 1.6 Hz, 2H), 7.13 (d, *J* = 1.6Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 2H), 3.97-3.95 (d, 12H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 149.45, 149.32, 146.34, 136.39, 132.16, 130.63, 119.70, 111.67, 110.38, 56.02, 55.97. **HRMS calcd for** $C_{21}H_{22}NO_4$ $[M+H]^+$ 352.1544, **found** 352.1547.

3,5-bis(3,4,5-trimethoxyphenyl)pyridine(3j)



Yellow solid, melting point: 228-230 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.77 (d, *J* = 2.0 Hz, 2H), 7.95 (t, *J* = 2.0 Hz, 1H), 6.81 (s, 4H), 3.95 (s, 12H), 3.92 (s, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 153.77, 146.90, 138.41, 136.84, 133.52, 132.78, 104.66, 60.94, 56.30. **HRMS calcd for** $C_{23}H_{26}NO_4$ $[M+H]^+$ 412.1755, **found** 412.1757.

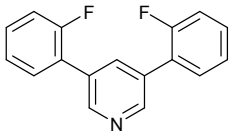
3,5-bis(4-fluorophenyl)pyridine(3k)



Yellow solid, melting point: 174-176 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.77 (d, *J* = 2.0 Hz, 2H), 7.95 (t, *J* = 2.0 Hz, 1H), 7.62-7.58 (m, 4H), 7.22-7.18 (m, 4H),. **^{13}C NMR (100 MHz, $CDCl_3$)** δ 163.02 (d, *J* = 247 Hz), 146.84, 135.73, 133.75 (d, *J* = 3 Hz,), 132.58, 128.92 (d, *J* = 9 Hz), 116.15 (d,

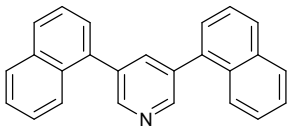
J = 21 Hz). **HRMS calcd for** $C_{17}H_{12}F_2N$ $[M+H]^+$ 268.0933, **found** 268.0931.

3,5-bis(2-fluorophenyl)pyridine(3l)



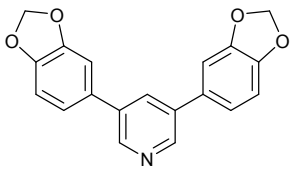
Yellow solid, melting point: 88-90 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.79 (t, *J* = 1.6 Hz, 2H), 8.06 (t, *J* = 1.6 Hz, 1H), 7.52-7.37 (m, 4H), 7.29-7.19 (m, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 159.92 (d, *J* = 247 Hz), 148.58 (d, *J* = 3 Hz), 136.58 (d, *J* = 3 Hz), 131.35, 130.58 (d, *J* = 3 Hz), 130.10 (d, *J* = 9 Hz), 125.40 (d, *J* = 13 Hz), 124.72 (d, *J* = 3 Hz), 116.34 (d, *J* = 23 Hz). **HRMS calcd for** $C_{17}H_{12}F_2N$ $[M+H]^+$ 268.0933, **found** 268.0930.

3,5-di(naphthalen-1-yl)pyridine(3m)



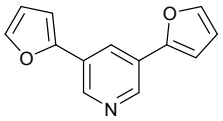
Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.84 (d, *J* = 2.0 Hz, 2H), 7.99-7.91 (m, 7H), 7.59-7.47 (m, 8H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 149.36, 138.62, 136.07, 135.91, 133.85, 131.46, 128.64, 128.53, 127.56, 126.64, 126.13, 125.42, 125.26. **HRMS calcd for** $C_{25}H_{18}N$ $[M+H]^+$ 332.1434, **found** 332.1437.

3,5-bis(benzo[d][1,3]dioxol-5-yl)pyridine(3n)



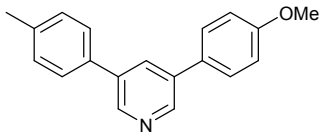
Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.71 (s, 2H), 7.89 (t, *J* = 2.0 Hz, 1H), 7.11-7.08 (m, 4H), 6.94-6.92 (m, 2H), 6.03 (s, 4H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 148.46, 147.87, 146.34, 132.25, 131.87, 120.95, 108.91, 107.59, 101.38. **HRMS calcd for** $C_{19}H_{14}NO_4$ $[M+H]^+$ 320.0918, **found** 320.0922.

3,5-di(furan-2-yl)pyridine(3o)



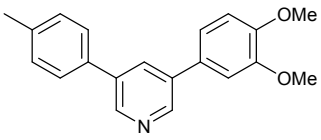
Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.80 (d, *J* = 2.0 Hz, 2H), 8.18 (t, *J* = 2.0 Hz, 1H), 7.55 (d, *J* = 1.6 Hz, 2H), 6.80 (d, *J* = 3.2 Hz, 2H), 6.53 (dd, *J* = 3.2 Hz, 1.6 Hz, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 150.83, 143.75, 143.14, 126.73, 125.38, 111.89, 106.80. **HRMS calcd for** $C_{13}H_{10}NO_2$ $[M+H]^+$ 212.0706, **found** 212.0710.

3-(4-methoxyphenyl)-5-(p-tolyl)pyridine(3p)



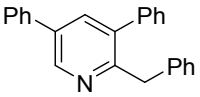
Yellow solid, melting point: 167-169 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.75 (d, *J* = 2.0 Hz, 2H), 7.98 (t, *J* = 2.0 Hz, 1H), 7.58-7.52 (m, 4H), 7.31-7.29 (m, 2H), 7.04-7.02 (m, 2H) 3.87 (s, 3H), 2.42 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 159.81, 146.37, 146.26, 138.09, 136.45, 136.15, 134.94, 132.16, 130.22, 129.80, 128.30, 127.05, 114.56, 53.37, 21.14. **HRMS calcd for** $C_{19}H_{18}NO$ $[M+H]^+$ 276.1383, **found** 276.1385.

3-(3,4-dimethoxyphenyl)-5-(p-tolyl)pyridine(3q)



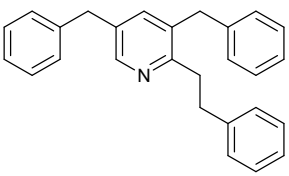
Yellow solid, melting point: 179-181 °C. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.76 (d, *J* = 2.0 Hz, 2H), 7.98 (t, *J* = 2.0 Hz, 1H), 7.54 (d, *J* = 8.0 Hz ,2H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.20 (dd, *J* = 8.4 Hz, 2.0 Hz, 1H), 7.13 (d, *J* = 2.0 Hz, 1H), 7.00 (d, *J* = 8.4 Hz, 1H), 3.97 (s, 3H), 3.95 (s, 3H), 2.43 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 149.53, 149.38, 146.51, 146.46, 138.16, 136.52, 136.44, 134.95, 132.34, 130.72, 129.83, 127.10, 119.73, 111.77, 110.45, 56.07, 56.04, 21.15. **HRMS calcd for** $C_{20}H_{20}NO_2$ $[M+H]^+$ 306.1489, **found** 306.1493.

2-benzyl-3,5-diphenylpyridine (3aa).



Yellow oil. **^{1}H NMR(400 MHz, $CDCl_3$)** δ 8.84-8.83 (d, *J* = 2.4 Hz, 1H), 7.75-7.74 (d, *J* = 2.4 Hz, 1 H), 7.62-7.59 (m, 2 H), 7.48-7.38 (m, 6 H), 7.29-7.27 (m, 2 H), 7.20-7.13 (m, 3 H), 7.06-7.04 (m, 2 H), 4.19 (s, 2 H).**^{13}C NMR (100 MHz, $CDCl_3$)** δ 156.6, 146.7, 140.0, 139.5, 137.5, 137.3, 136.1, 134.2, 129.2, 129.0, 128.8, 128.3, 128.2, 127.9, 127.6, 127.0, 125.9, 41.3. **HRMS calcd for** $C_{24}H_{20}N[M+H]^+$ 322.1590, **found** 322.1596.

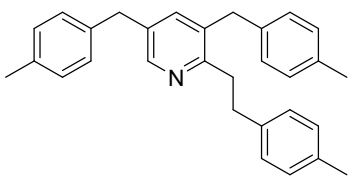
3,5-dibenzyl-2-phenethylpyridine(4a)



Yellow oil. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.36 (d, *J* = 2.0 Hz, 1H), 7.31-7.01 (m, 16H), 3.93 (s, 2H), 3.86 (s, 2H), 3.02-2.98 (m, 2H), 2.91-2.86 (m, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 157.62, 147.68, 141.97, 140.12, 139.57, 138.40, 133.97, 133.49, 128.74, 128.62, 128.59, 128.56, 128.45,

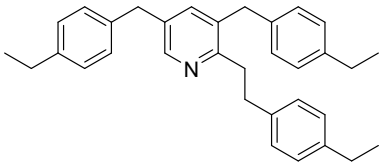
128.30, 126.33, 125.85, 38.60, 38.28, 36.82, 35.46. **HRMS calcd for** $C_{27}H_{26}N$ $[M+H]^+$ 364.2060, **found** 364.2064.

3,5-bis(4-methylbenzyl)-2-(4-methylphenethyl)pyridine (4b).



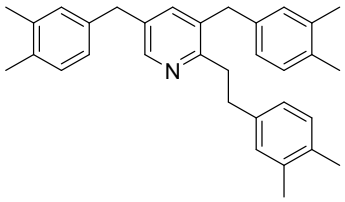
Yellow oil (20.2 mg, 50%). **^{1}H NMR(400 MHz, CDCl^3)** δ 8.34-8.33 (d, *J* = 2.0 Hz, 1H), 7.17-7.16 (d, *J* = 2.0 Hz, 1H), 7.11-6.99 (m, 10 H), 6.91-6.89 (m, 2 H), 3.88 (s, 2 H), 3.83 (s, 2H), 2.99-2.95 (m, 2 H), 2.86-2.82 (m, 2H), 2.32 (s, 3 H), 2.31 (s, 6 H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 157.6, 147.6, 139.0, 138.3, 137.1, 136.5, 135.83, 135.79, 135.3, 134.1, 133.7, 129.3, 129.2, 129.0, 128.6, 128.5, 128.3, 38.2, 37.9, 37.0, 35.1, 21.0. **HRMS calcd for** $C_{30}H_{32}N[M+H]^+$ 406.2530, **found** 406.2534.

3,5-bis(4-ethylbenzyl)-2-(4-ethylphenethyl)pyridine(4c).



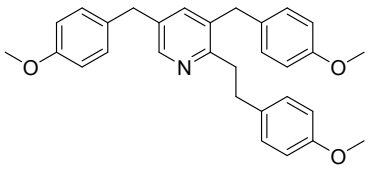
Yellow oil (24.1 mg, 54%). **^{1}H NMR(400 MHz, CDCl^3)** δ 8.35-8.34 (d, *J* = 2.0 Hz, 1H), 7.19-7.18 (d, *J* = 2.0 Hz, 1H), 7.13-7.06 (m, 8 H), 7.03-7.01 (m, 2 H), 6.95-6.93 (m, 2 H) 3.89 (s, 2 H), 3.84 (s, 2 H), 3.00-2.96 (m, 2 H), 2.86-2.82 (m, 2H), 2.65-2.58 (m, 6 H), 1.24-1.19 (m, 9 H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 157.7, 147.6, 142.2, 141.7, 139.2, 138.4, 137.4, 136.8, 134.1, 133.6, 128.65, 128.57, 128.4, 128.06, 128.01, 127.8, 38.2, 37.9, 37.0, 35.1, 28.4, 15.63, 15.61. **HRMS calcd for** $C_{33}H_{38}N[M+H]^+$ 448.2999, **found** 448.2996.

3,5-bis(3,4-dimethylbenzyl)-2-(3,4-dimethylphenethyl)pyridine(4d).



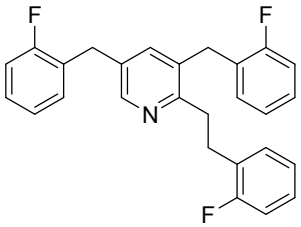
Yellow oil (22.8 mg, 51%). **^{1}H NMR(400 MHz, $CDCl_3$)** δ 8.35-8.34 (d, *J* = 2.4 Hz, 1H), 7.20-7.19 (d, *J* = 2.4 Hz, 1H), 7.05-7.00 (m, 3 H), 6.93-6.73 (m, 6 H), 3.85 (s, 2 H), 3.82 (s, 2 H), 3.00-2.95 (m, 2 H), 2.81-2.77 (m, 2H), 2.22-2.19 (m, 18 H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 157.7, 147.5, 139.5, 138.4, 137.7, 137.1, 136.7, 136.6, 136.3, 134.43, 134.39, 134.2, 133.9, 133.7, 130.02, 129.97, 129.85, 129.79, 129.75, 129.6, 126.06, 126.04, 125.8, 38.2, 37.9, 37.2, 35.1, 19.72, 19.68, 19.3. **HRMS calcd for** $C_{33}H_{38}N[M+H]^+$ 448.2999, **found** 448.2994.

3,5-bis(4-methoxybenzyl)-2-(4-methoxyphenethyl)pyridine(4e).



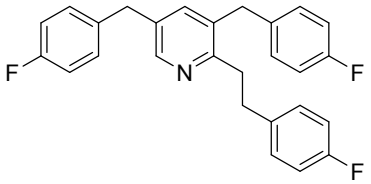
Yellow oil (23.6 mg, 52%). **^{1}H NMR(400 MHz, $CDCl_3$)** δ 8.33-8.32 (d, *J* = 1.6 Hz, 1H), 7.15-7.14 (d, *J* = 1.6 Hz, 1H), 7.08-7.01 (m, 4 H), 6.94-6.91 (m, 2 H), 6.84-6.78 (m, 6 H) 3.86 (s, 2 H), 3.80 (s, 2H), 3.78 (s, 3 H), 3.77 (s, 6 H), 2.99-2.95 (m, 2 H), 2.85-2.81 (m, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 158.11, 158.07, 157.8, 157.5, 147.5, 138.1, 134.3, 134.1, 133.8, 132.3, 131.6, 129.7, 129.6, 129.3, 113.99, 113.95, 113.7, 55.25, 55.23, 37.7, 37.4, 37.0, 34.6. **HRMS calcd for** $C_{30}H_{32}NO_3[M+H]^+$ 454.2377, **found** 454.2371.

3,5-bis(2-fluorobenzyl)-2-(2-fluorophenethyl)pyridine(4f).



Yellow oil (24.2 mg, 58%). **^{1}H NMR(400 MHz, $CDCl_3$)** δ 8.36-8.35 (d, *J* = 2.0 Hz, 1H), 7.22-6.97 (m, 12H),6.90-6.86 (m, 1 H), 3.93-3.91 (m, 4 H) 3.08-2.98 (m, 4 H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 162.4, 162.12, 162.08, 160.0, 159.7, 159.6, 157.3, 147.6, 137.9, 132.9, 132.4, 130.8, 130.73, 130.68,130.54, 130.50, 128.7, 128.6, 128.5, 128.4, 128.3, 128.25, 128.22, 128.17, 127.7, 127.6, 127.1, 127.0, 126.4, 126.3, 124.19, 124.15, 124.11, 123.93, 123.90, 115.5, 115.4, 115.3, 115.2, 115.1, 35.1, 31.72, 31.69, 30.93, 30.90, 28.72, 28.69. **HRMS calcd for** $C_{27}H_{23}F_3N[M+H]^+$ 418.1777, **found** 418.1780.

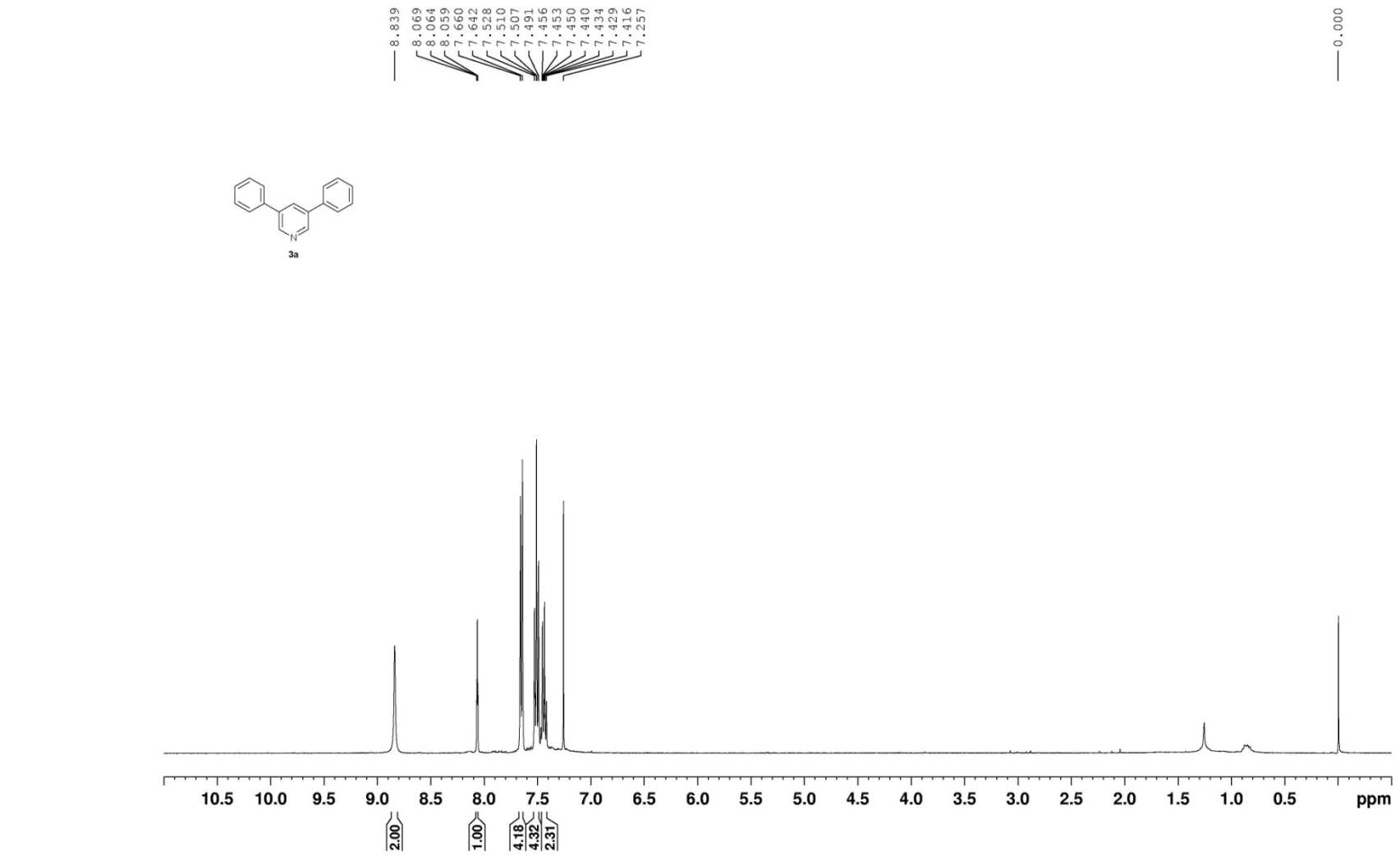
3,5-bis(4-fluorobenzyl)-2-(4-fluorophenethyl)pyridine (4g).



Yellow oil (25.8 mg, 62%). **^{1}H NMR(400 MHz, $CDCl_3$)** δ 8.34-8.33 (d, *J* = 2.0 Hz, 1H), 7.12-7.11 (d, *J* = 2.0 Hz, 1H), 7.11-7.09 (m, 3 H), 7.04-6.89 (m, 10 H), 3.90 (s, 2 H), 3.81 (s, 2 H), 2.96-2.94 (m, 2 H), 2.90-2.88 (m, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 162.77, 162.71, 162.5, 160.34, 160.28, 160.1, 157.4, 147.7, 138.1, 137.42, 137.39, 135.67, 135.64, 135.05, 135.01, 134.0, 133.4, 130.2, 130.1, 130.0, 129.9, 129.8, 129.7, 115.5, 115.3, 115.1, 114.9, 37.7, 37.4, 36.7, 34.4. **HRMS calcd for** $C_{27}H_{23}F_3N[M+H]^+$ 418.1777, **found** 418.1773.

Reference:

1. Q. Wang, C.-H. Wan, Y. Gu, J.-T. Zhang, L.-F. Gao, and Z.-Y. Wang, *Green Chem.*, 2011, **13**, 578-581.
2. S. M. Spinella, Z.-H. Guan, J. Chen, and X. Zhang, *Synthesis*, 2009, **18**, 3094-3098.
3. H. Adams, E. Chekmeneva, C. A. Hunter, M. C. Misuraca, C. Navarro, and S. M. Turega, *J. Am. Chem. Soc.* 2013, **135**, 1853−1863.
4. T.-H. Chuang, Y.-C. Chen, and S. Pola, *J. Org. Chem.* 2010, **75**, 6625–6630.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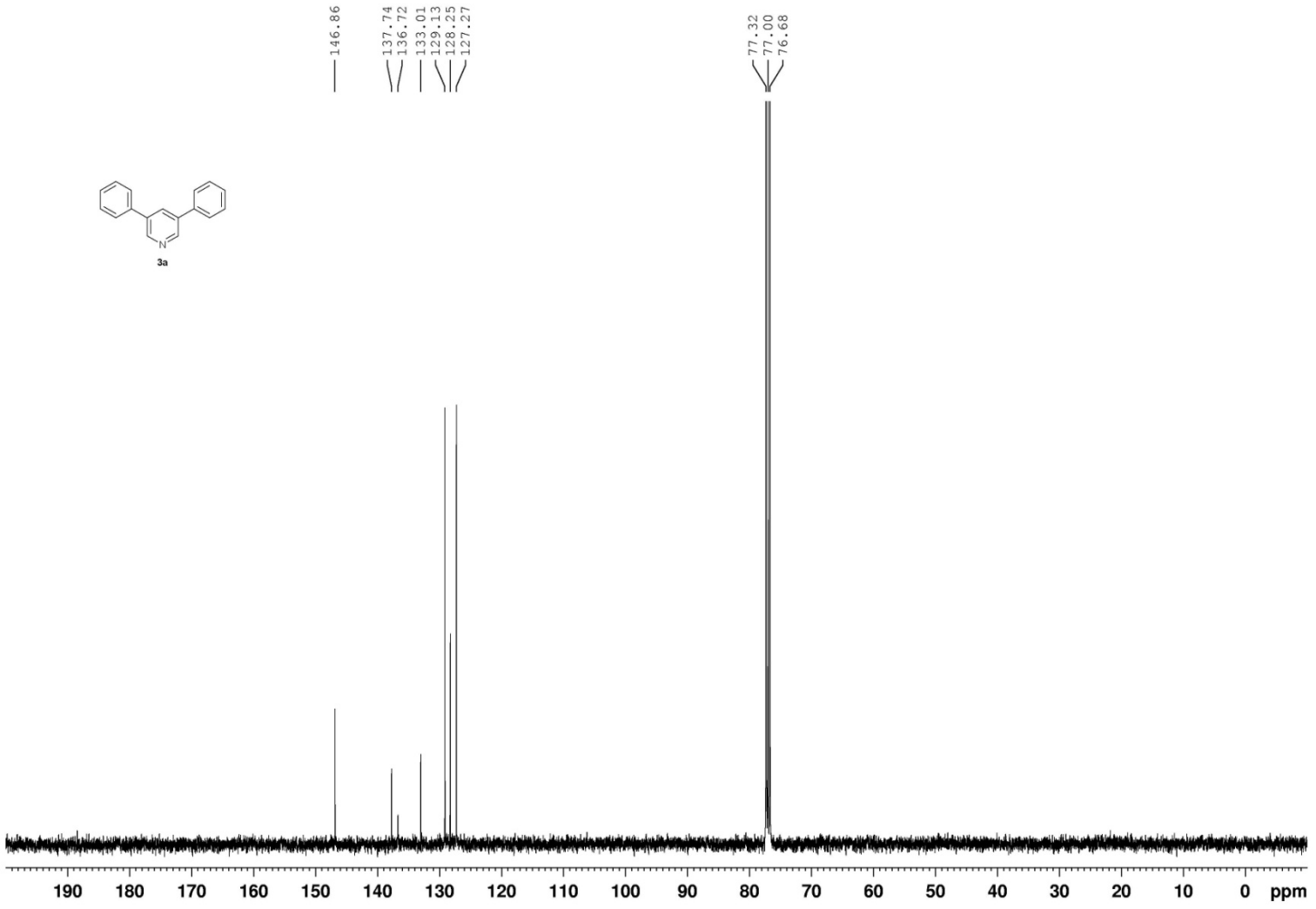
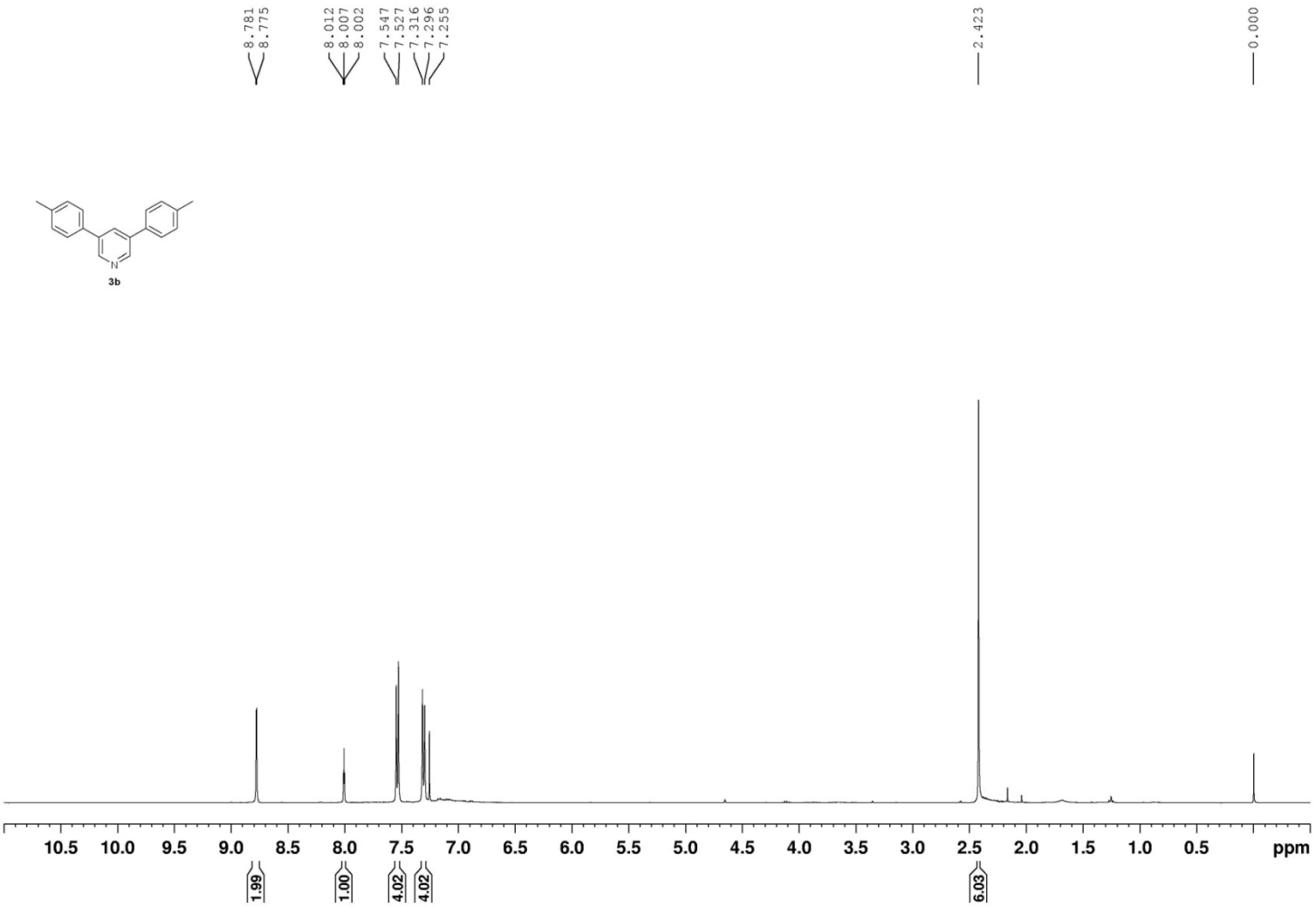
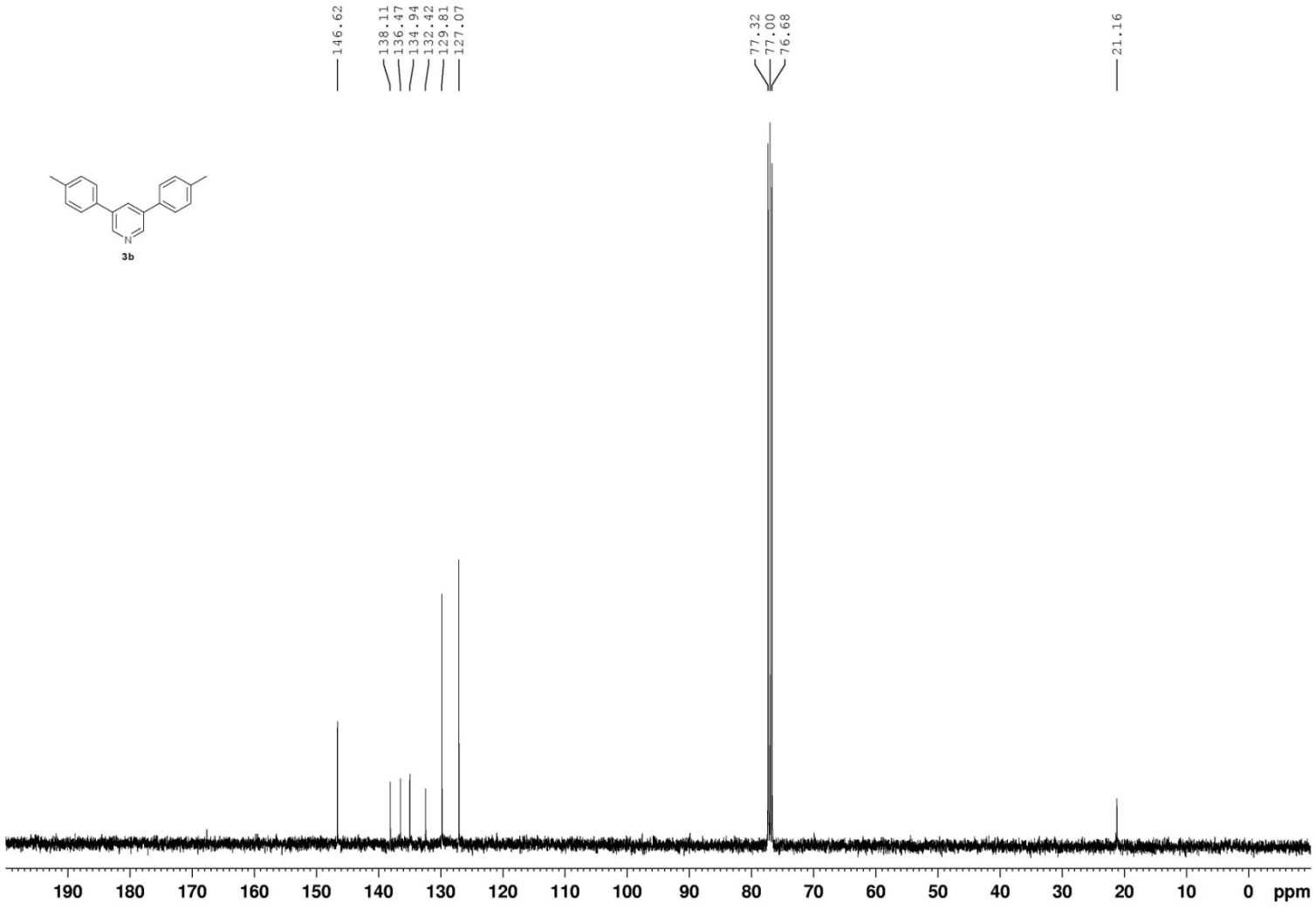
8.069
8.064
8.059
7.660
7.642
7.528
7.510
7.507
7.491
7.456
7.453
7.450
7.440
7.434
7.429
7.416
7.257
0.000
3a
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
1.00
4.18
4.32
2.31


146.86
137.74
136.72
133.01
129.13
128.25
127.27
77.32
77.00
76.68
N
3a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.781
8.775
8.012
8.007
8.002
7.547
7.527
7.316
7.296
7.255
2.423
0.000
3b
10.5 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 ppm
1.99
1.00
4.02
4.02
6.03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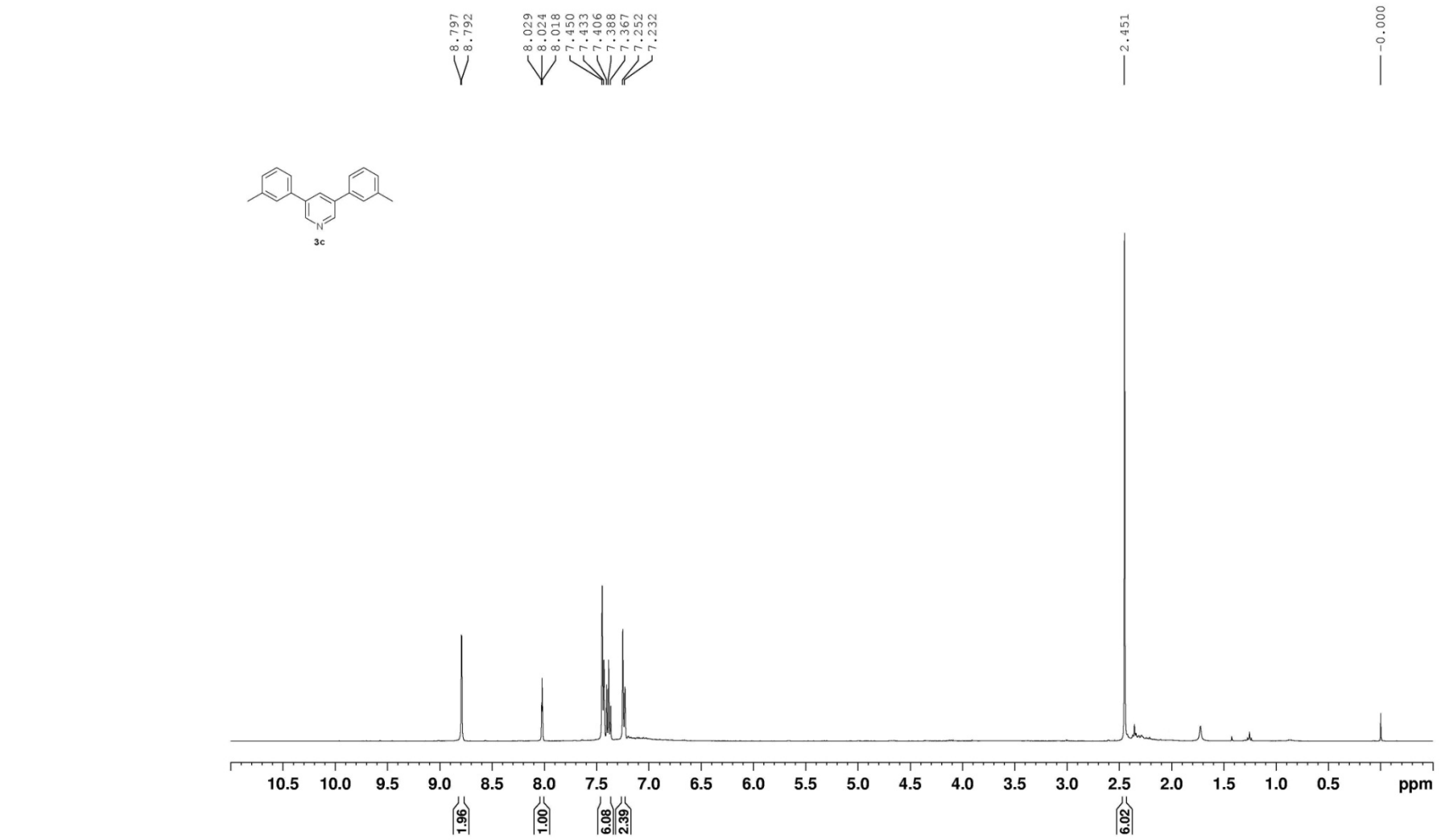
138.11
136.47
134.94
132.42
129.81
127.07
77.32
77.00
76.68
21.16
N
3b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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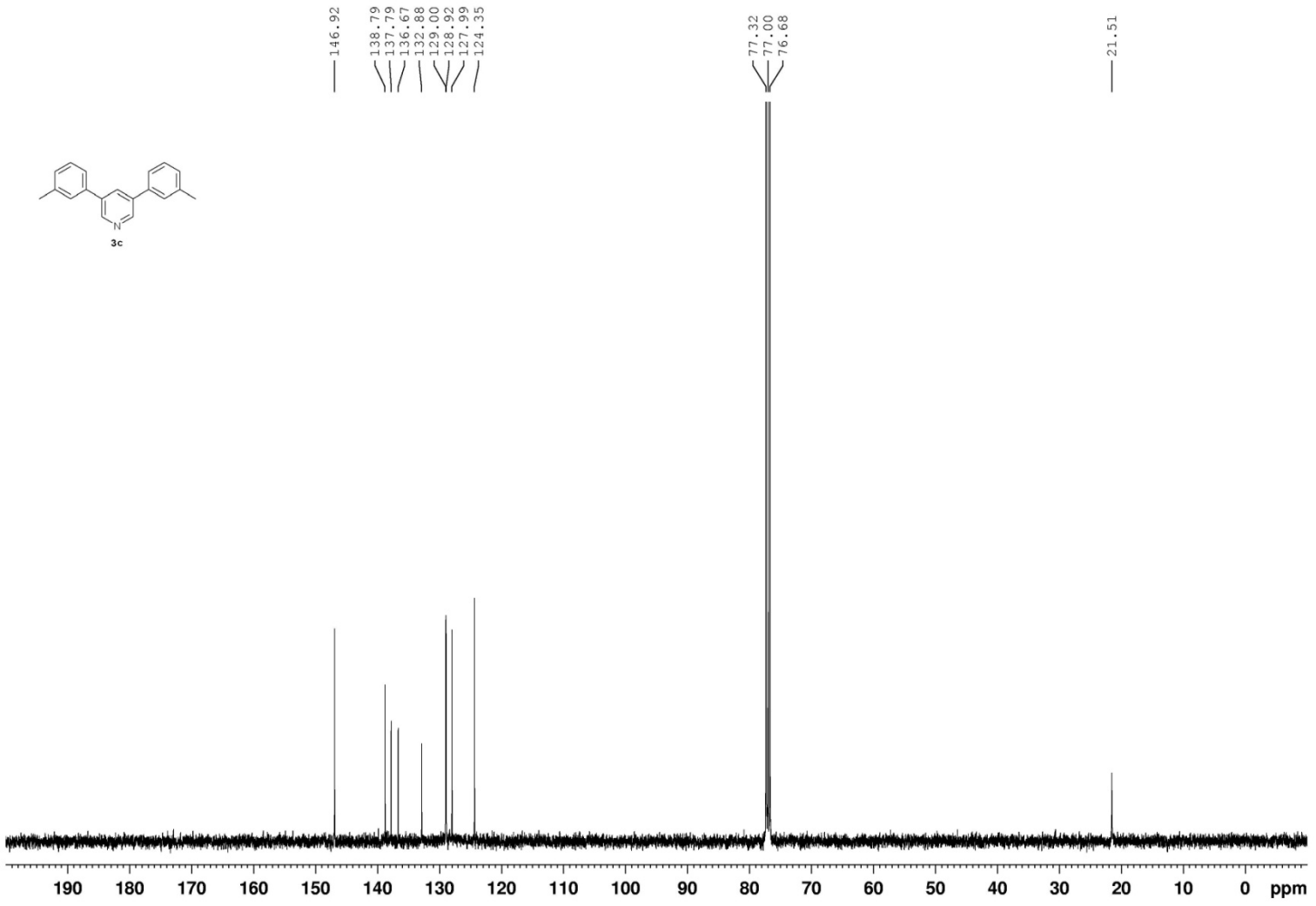
8.792
8.029
8.024
8.018
7.450
7.433
7.406
7.388
7.367
7.252
7.232
2.451
0.000
3c
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.96
1.00
6.08
2.39
6.02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132.88
129.00
128.92
127.99
124.35
77.32
77.00
76.68
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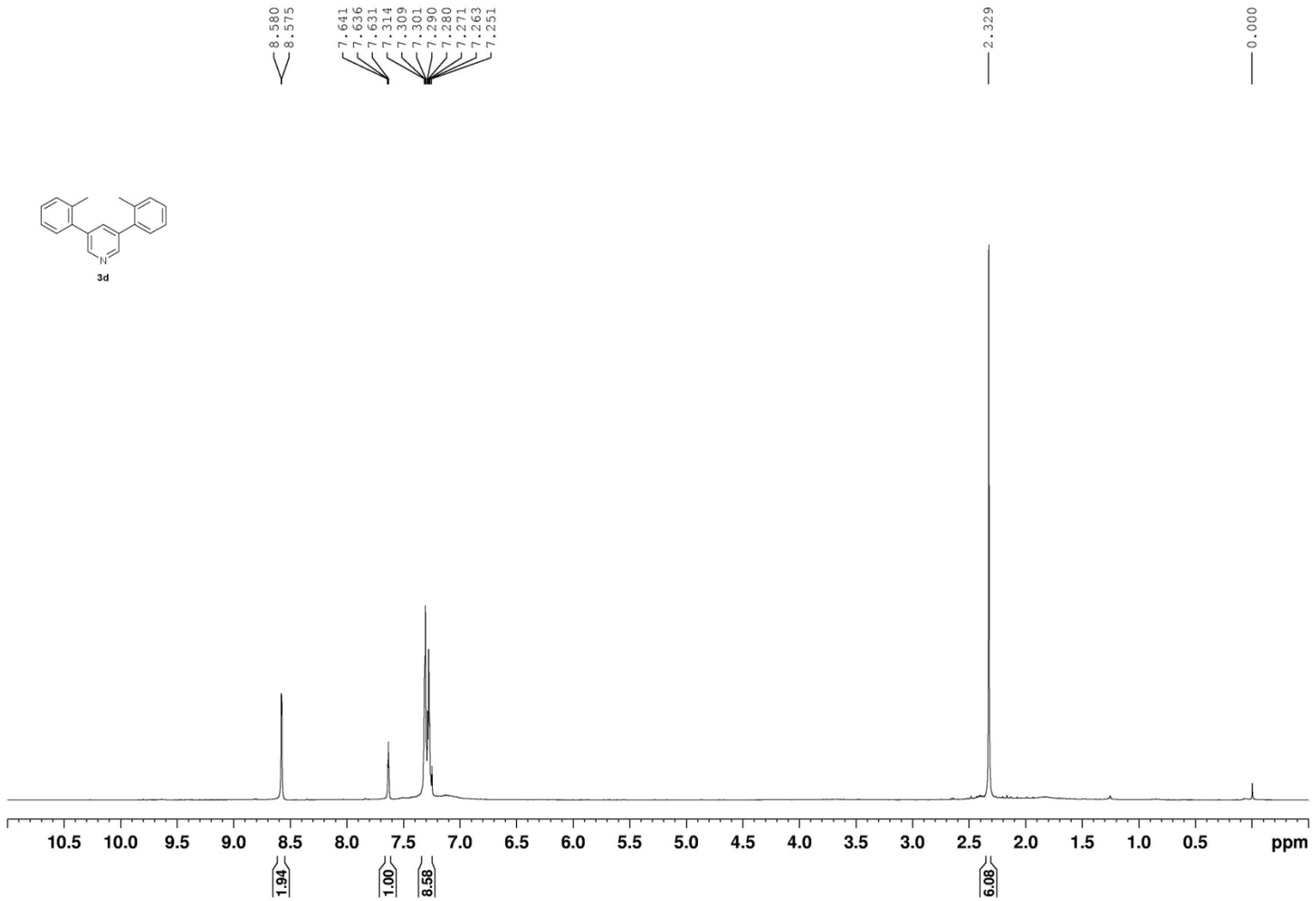
N
3c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.636
7.631
7.314
7.309
7.301
7.290
7.280
7.271
7.263
7.251
2.329
0.000
3d
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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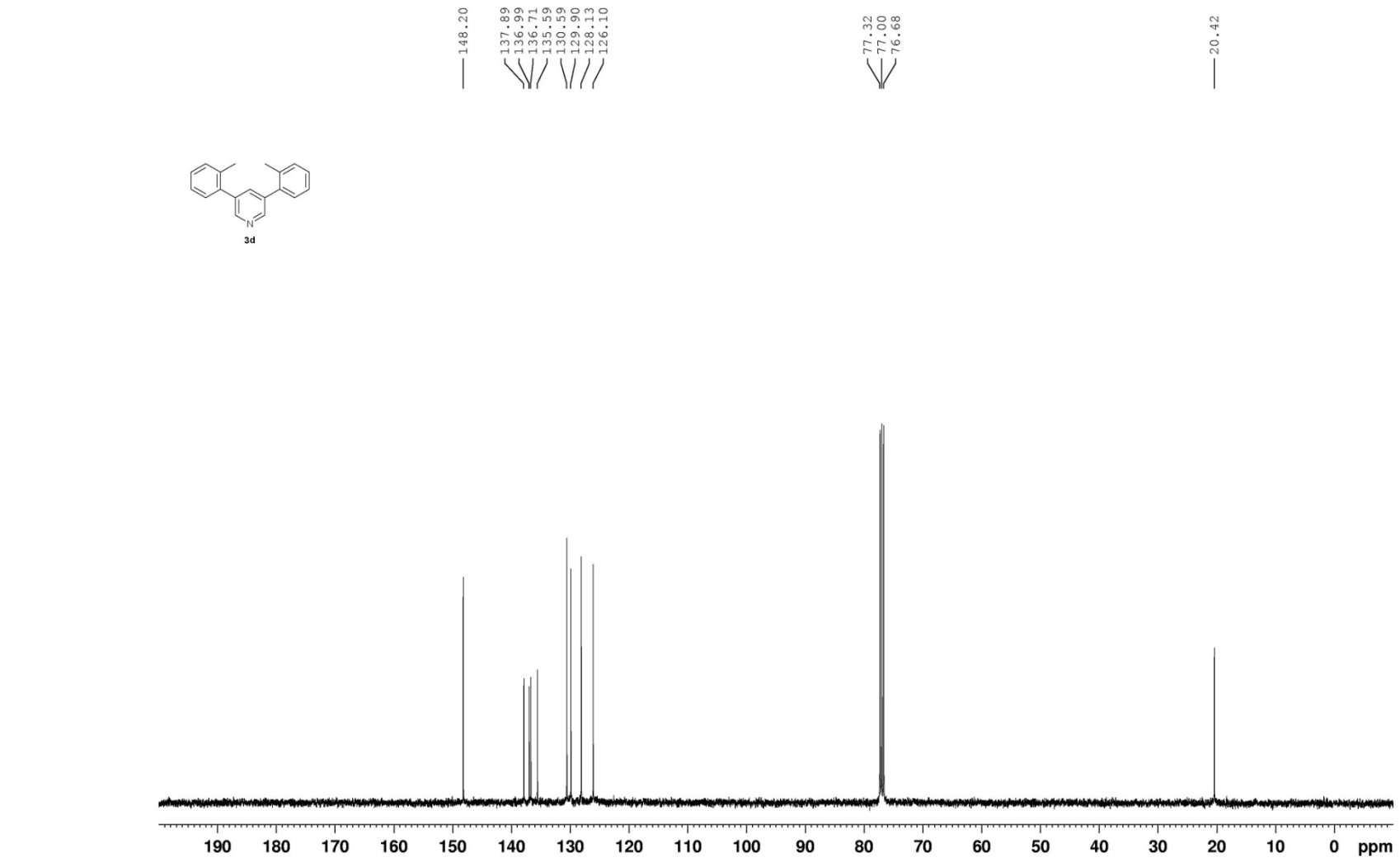
1.00
8.58
6.08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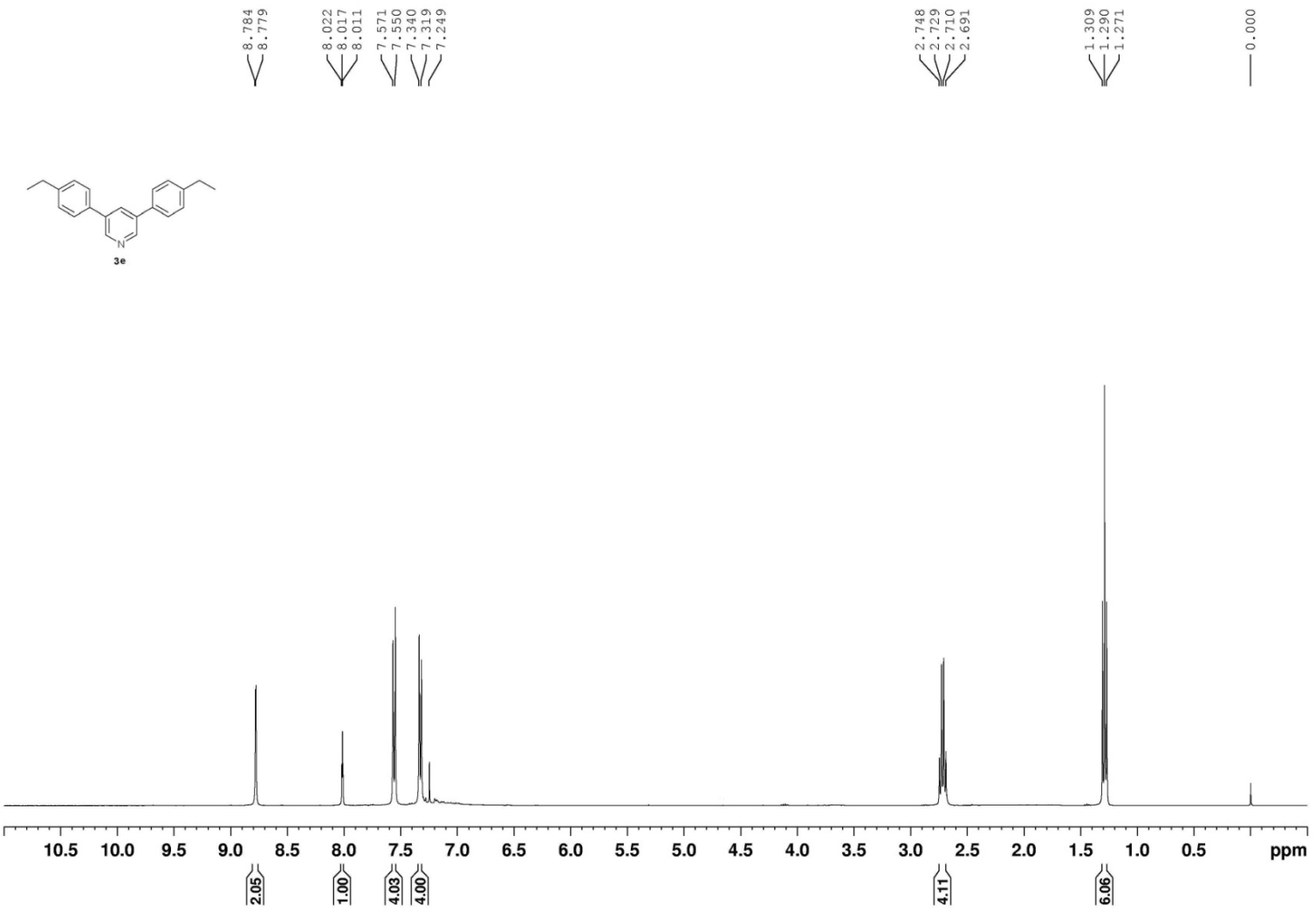
137.89
136.99
136.71
135.59
130.59
129.90
128.13
126.10
77.32
77.00
76.68
20.42
N
3d
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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8.017
8.011
7.571
7.550
7.340
7.319
7.249
2.748
2.729
2.710
2.691
1.309
1.290
1.271
0.000
3e
10.5 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 ppm
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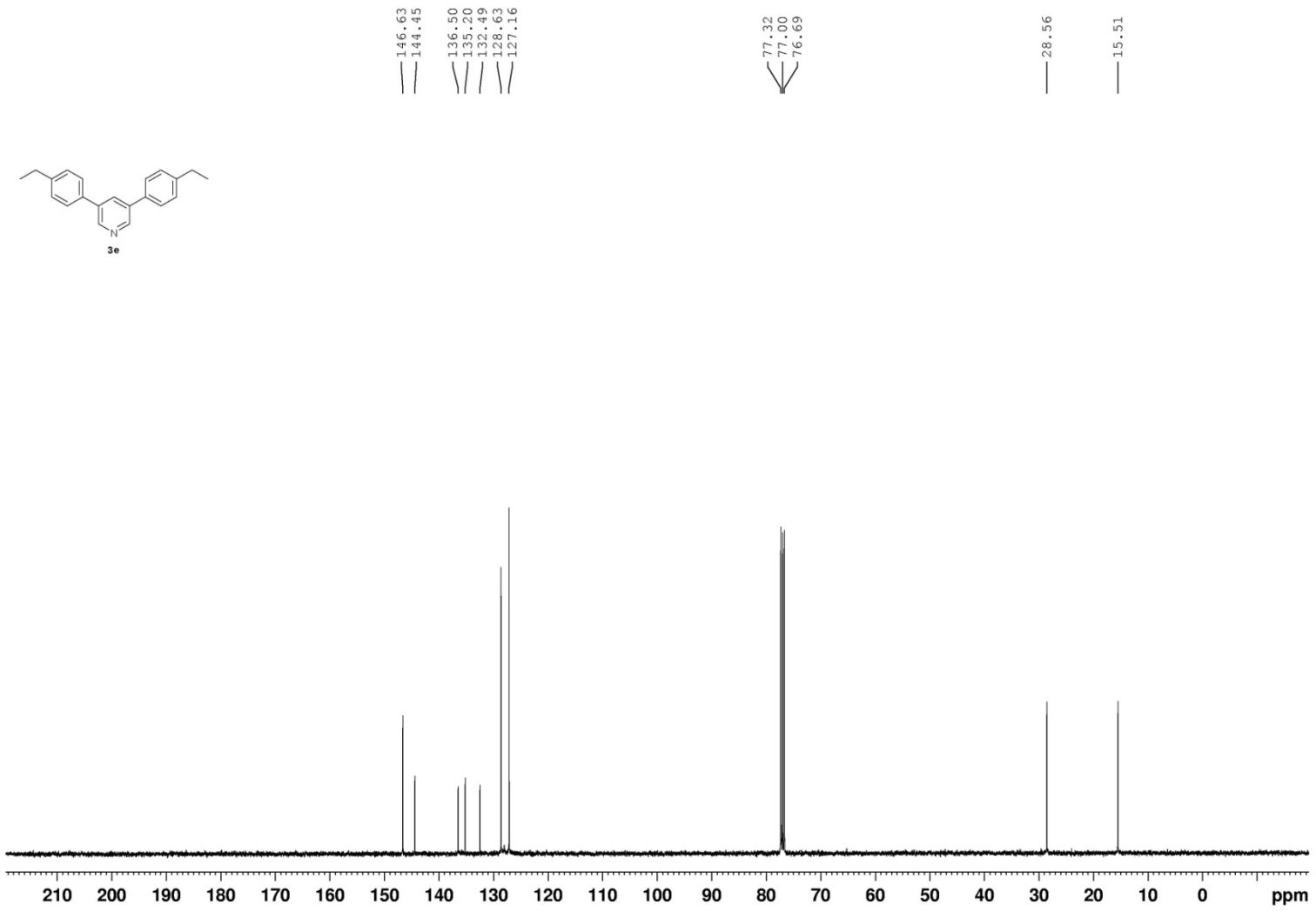
1.00
4.03
4.00
4.11
6.06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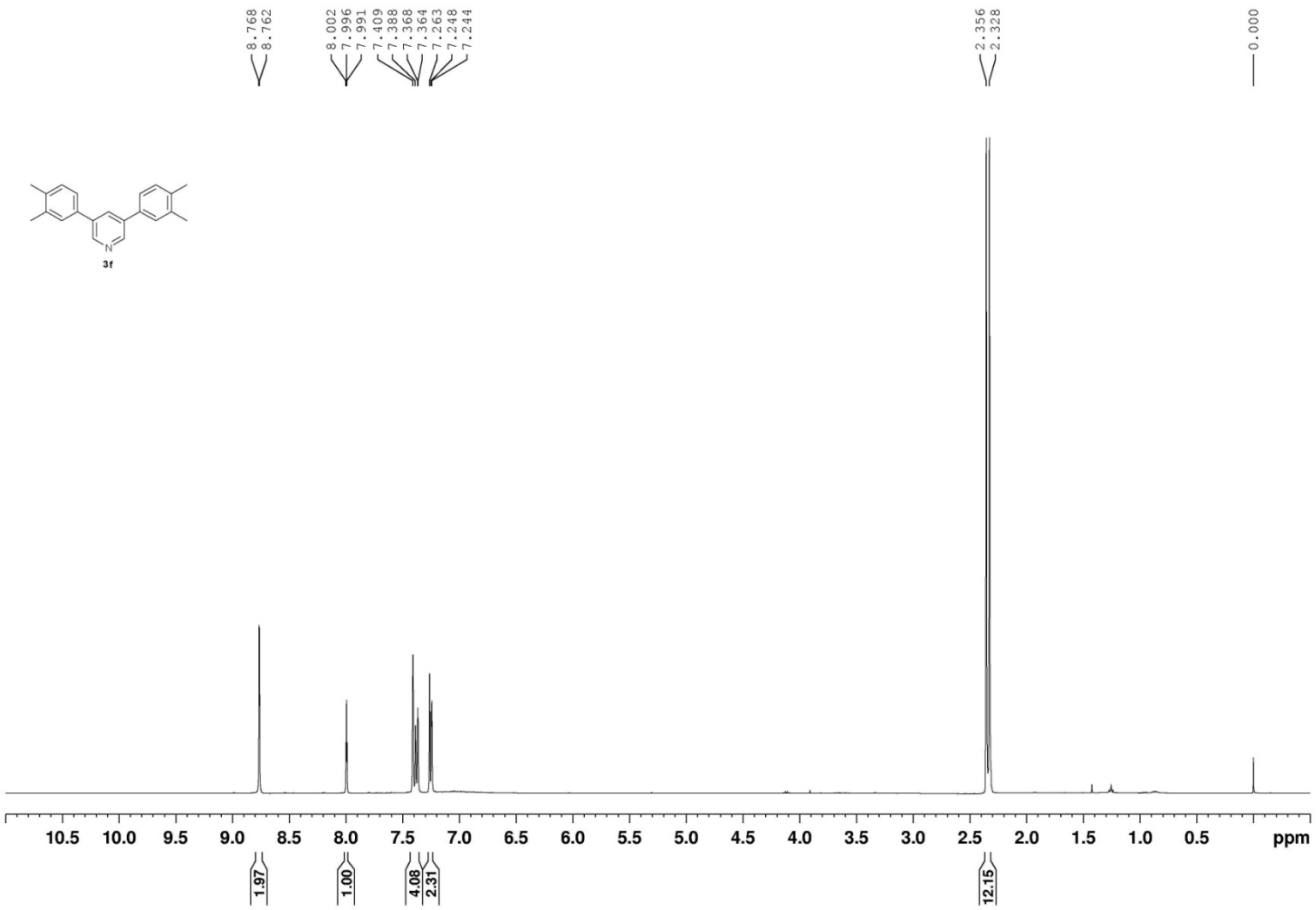
144.45
136.50
135.20
132.49
128.63
127.16
77.32
77.00
76.69
28.56
15.51
3e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.388
7.368
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7.263
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7.244
2.356
2.328
0.000
3f
10.5
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
ppm
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1.00
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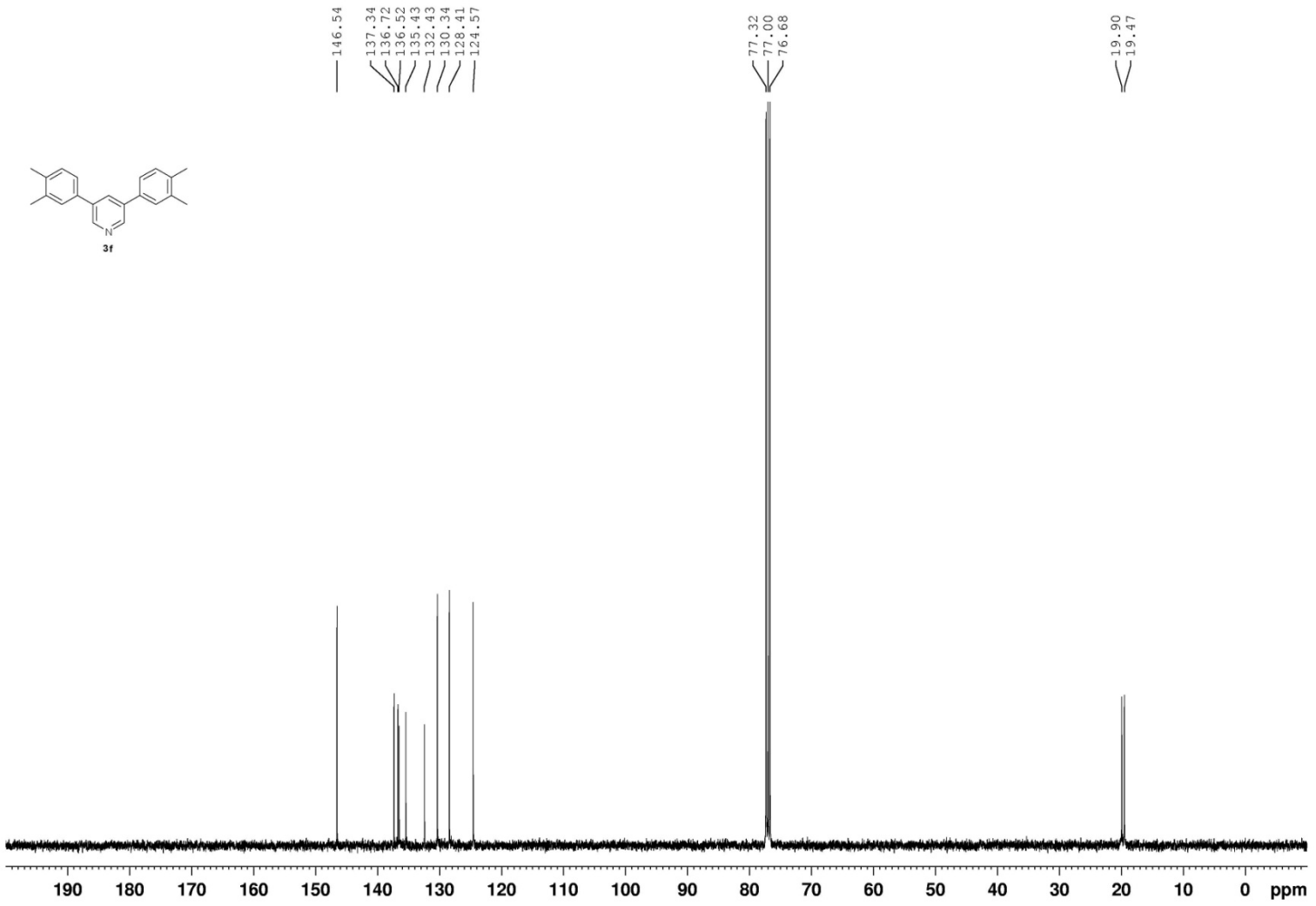
2.31
12.15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77.32
77.00
76.68
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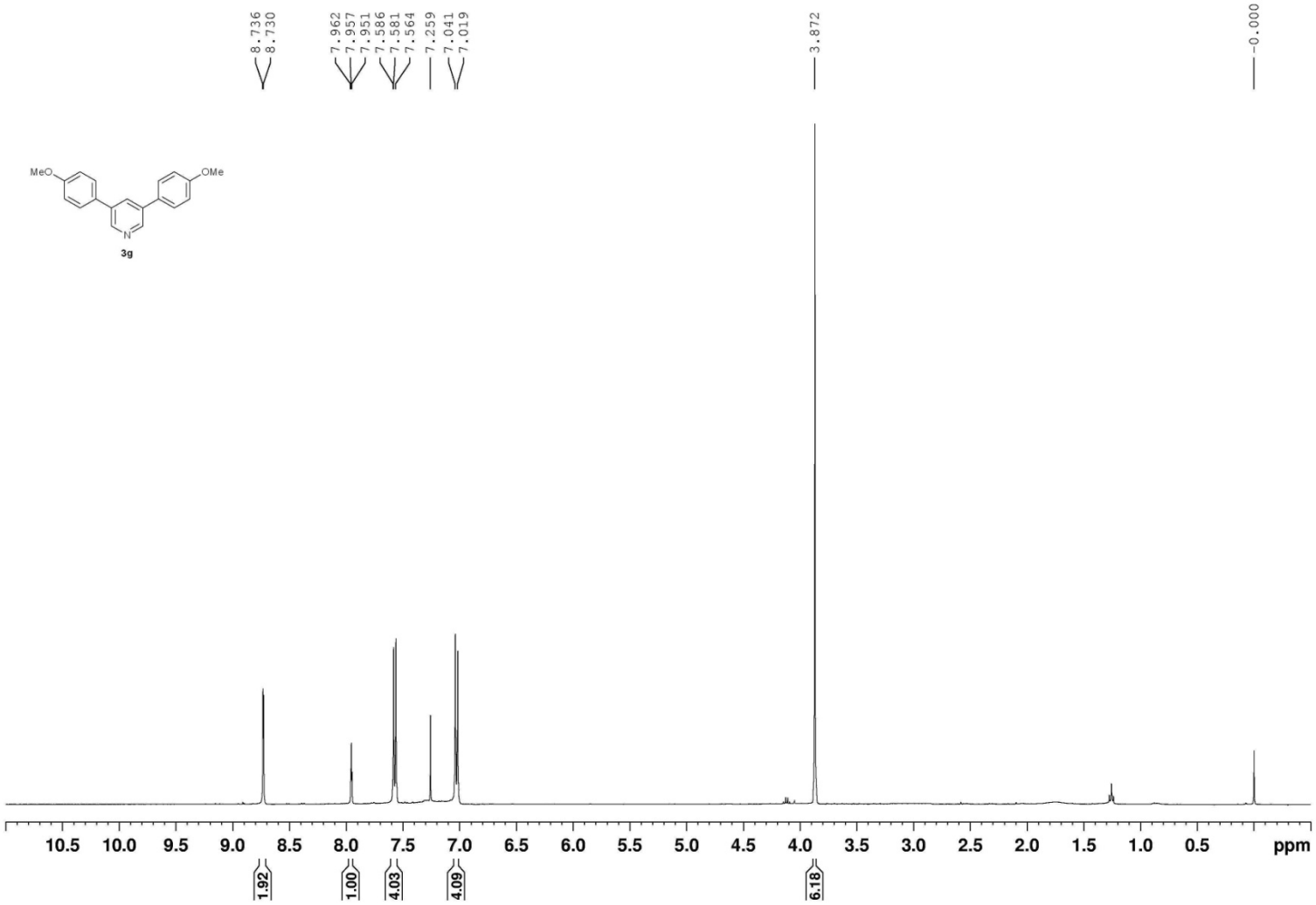
19.47
3f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.951
7.586
7.581
7.564
7.259
7.041
7.019
3.872
0.000
MeO
OMe
N
3g
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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2.0
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ppm
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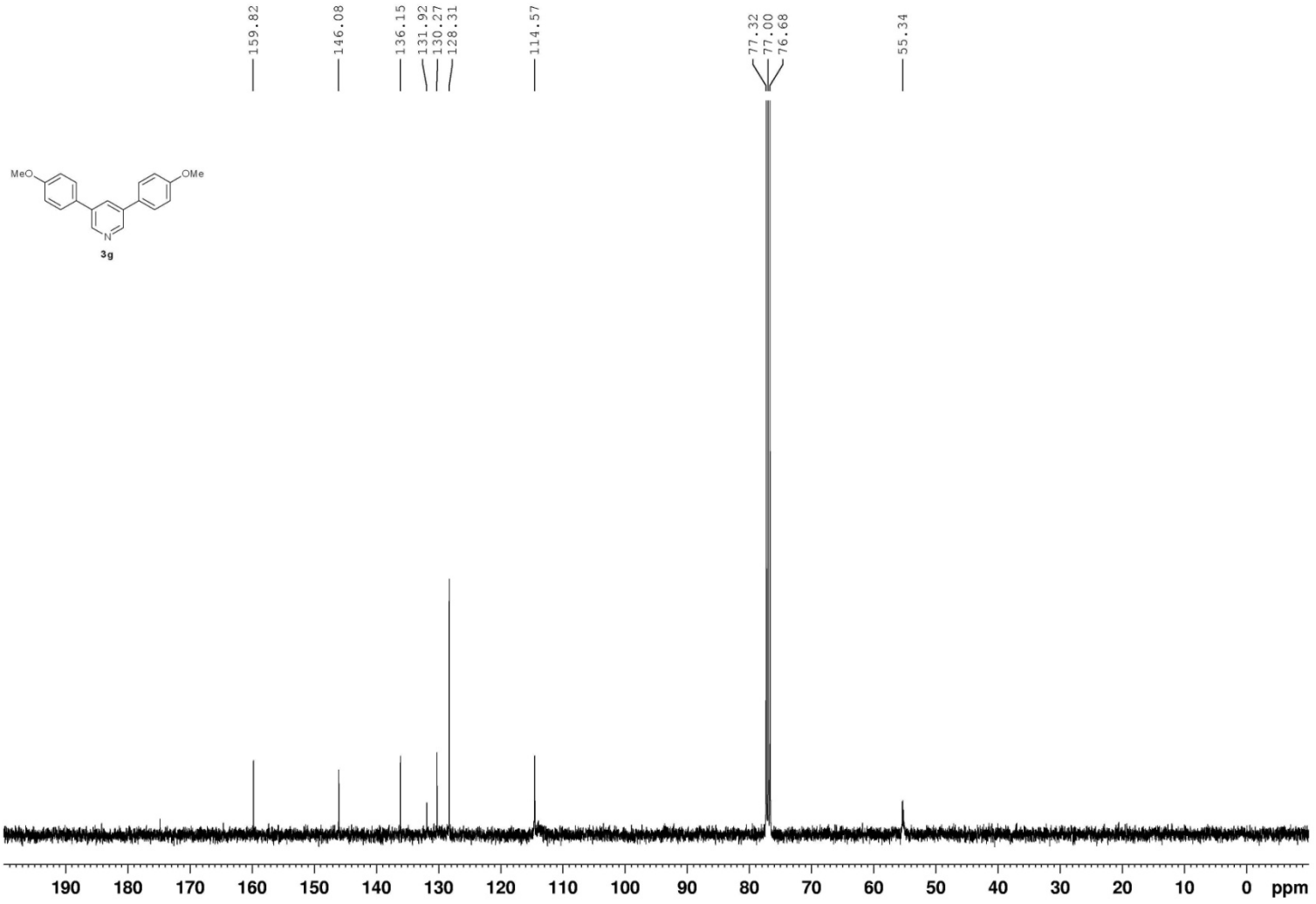
4.09
6.18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3g
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110
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90
80
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60
50
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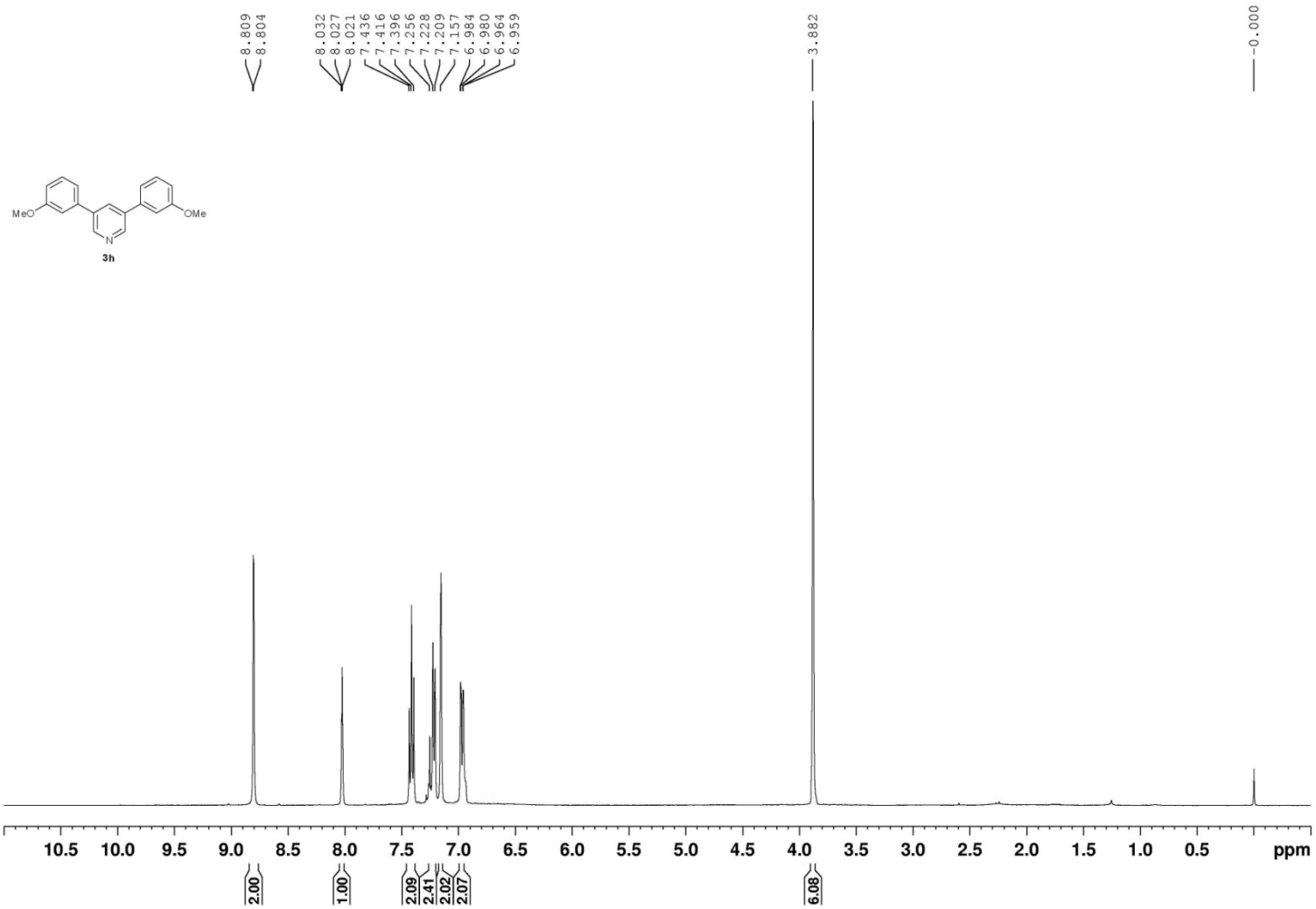
30
20
10
0
ppm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7.228
7.209
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6.984
6.980
6.964
6.959
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-0.000
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OMe
N
3h
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7.0
6.5
6.0
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5.0
4.5
4.0
3.5
3.0
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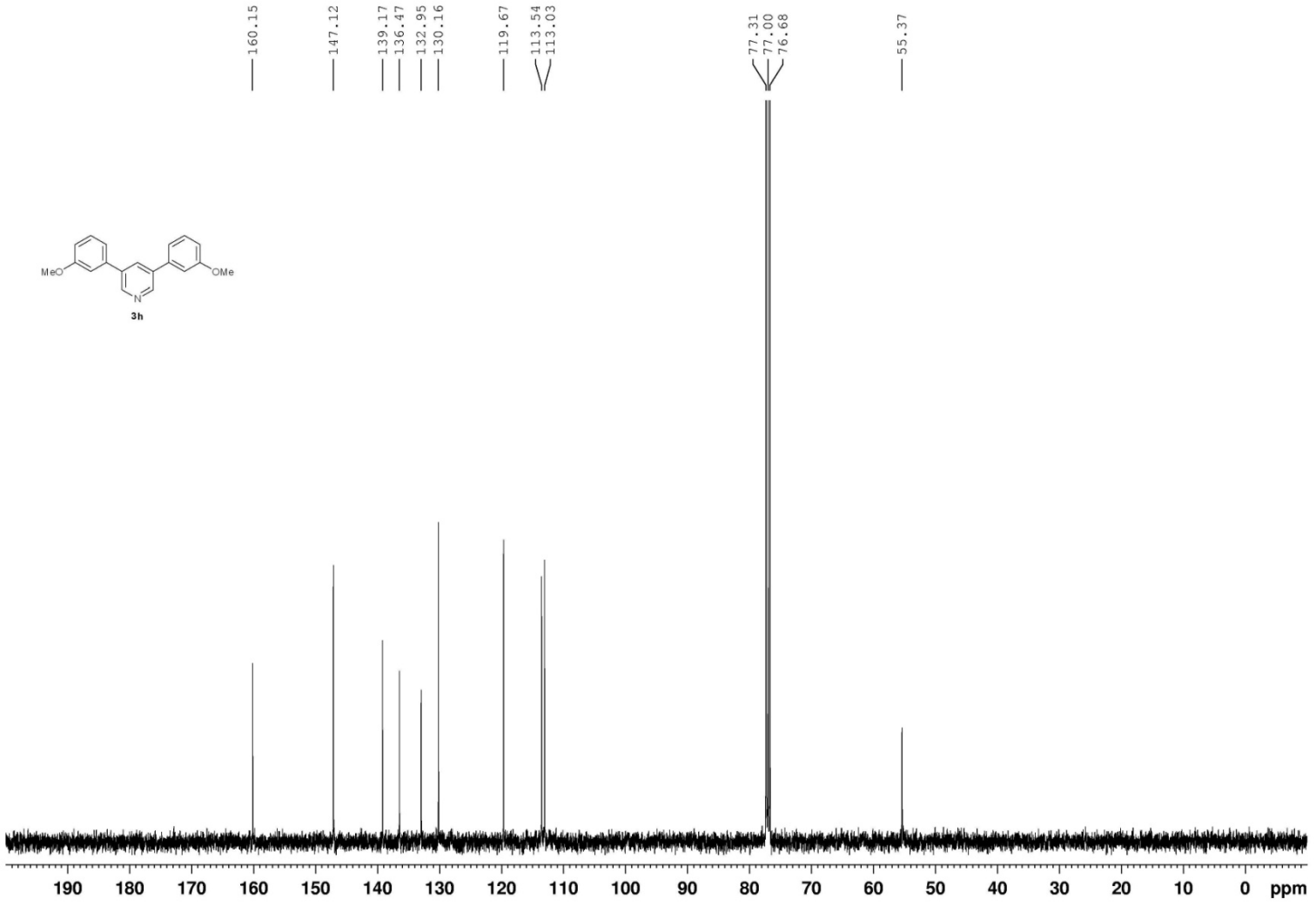
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55.37
MeO
OMe
N
3h
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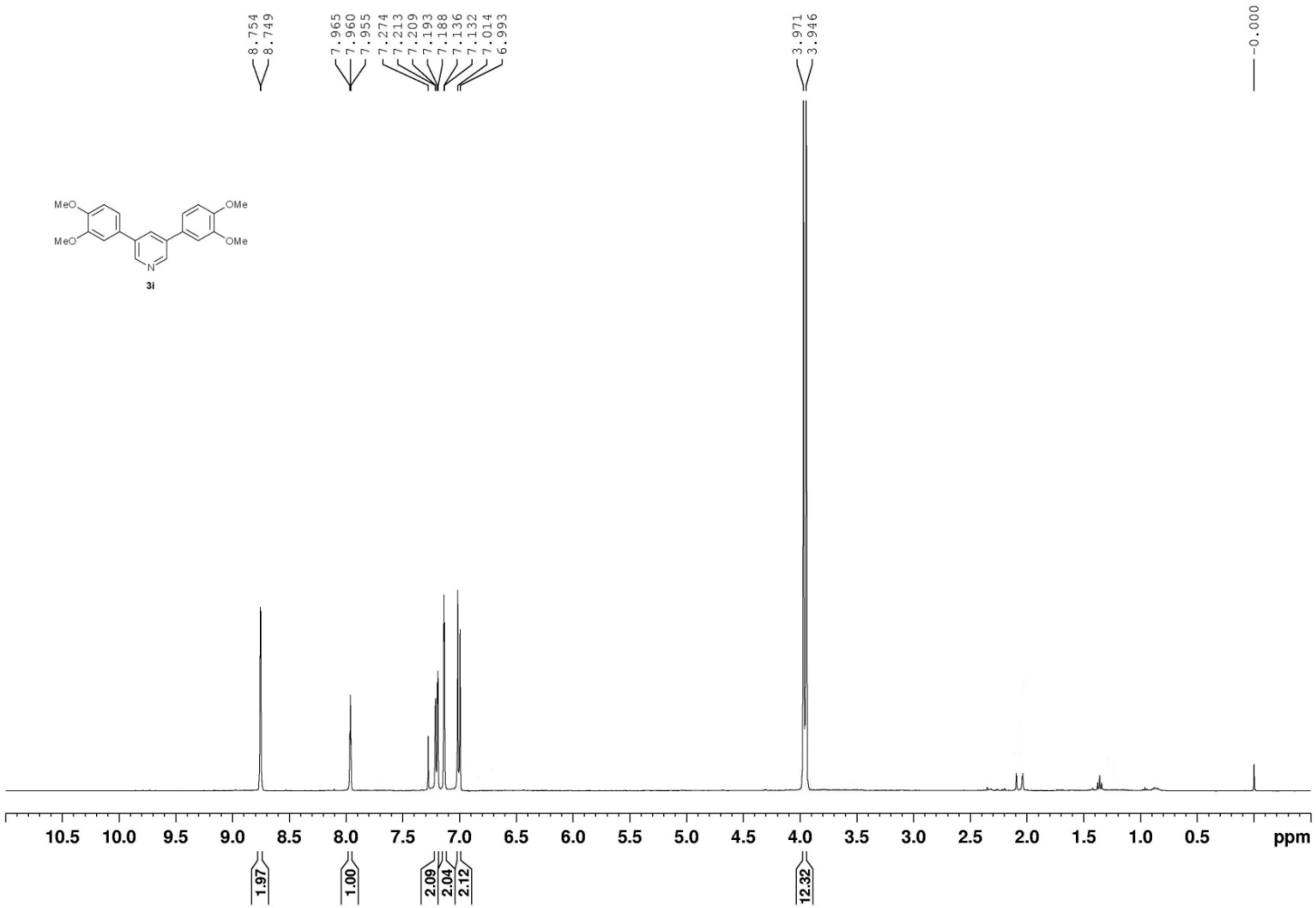
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7.209
7.193
7.188
7.136
7.132
7.014
6.993
3.971
3.946
-0.000
MeO
MeO
OMe
OMe
N
3i
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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1.0
0.5
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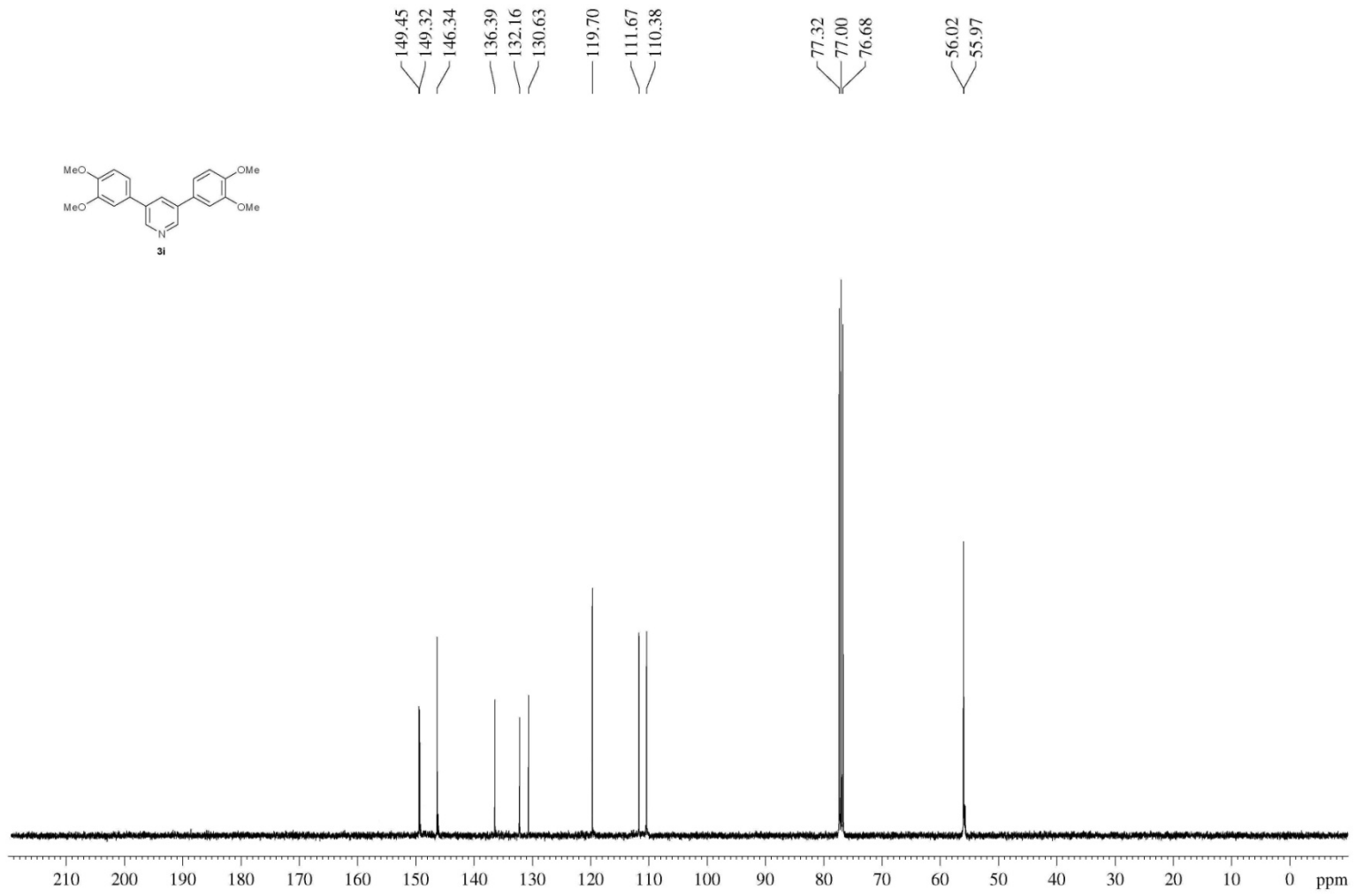
2.04
2.12
12.32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130.63
119.70
111.67
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77.00
76.68
56.02
55.97
MeO
MeO
OMe
OMe
N
3i
210
200
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10
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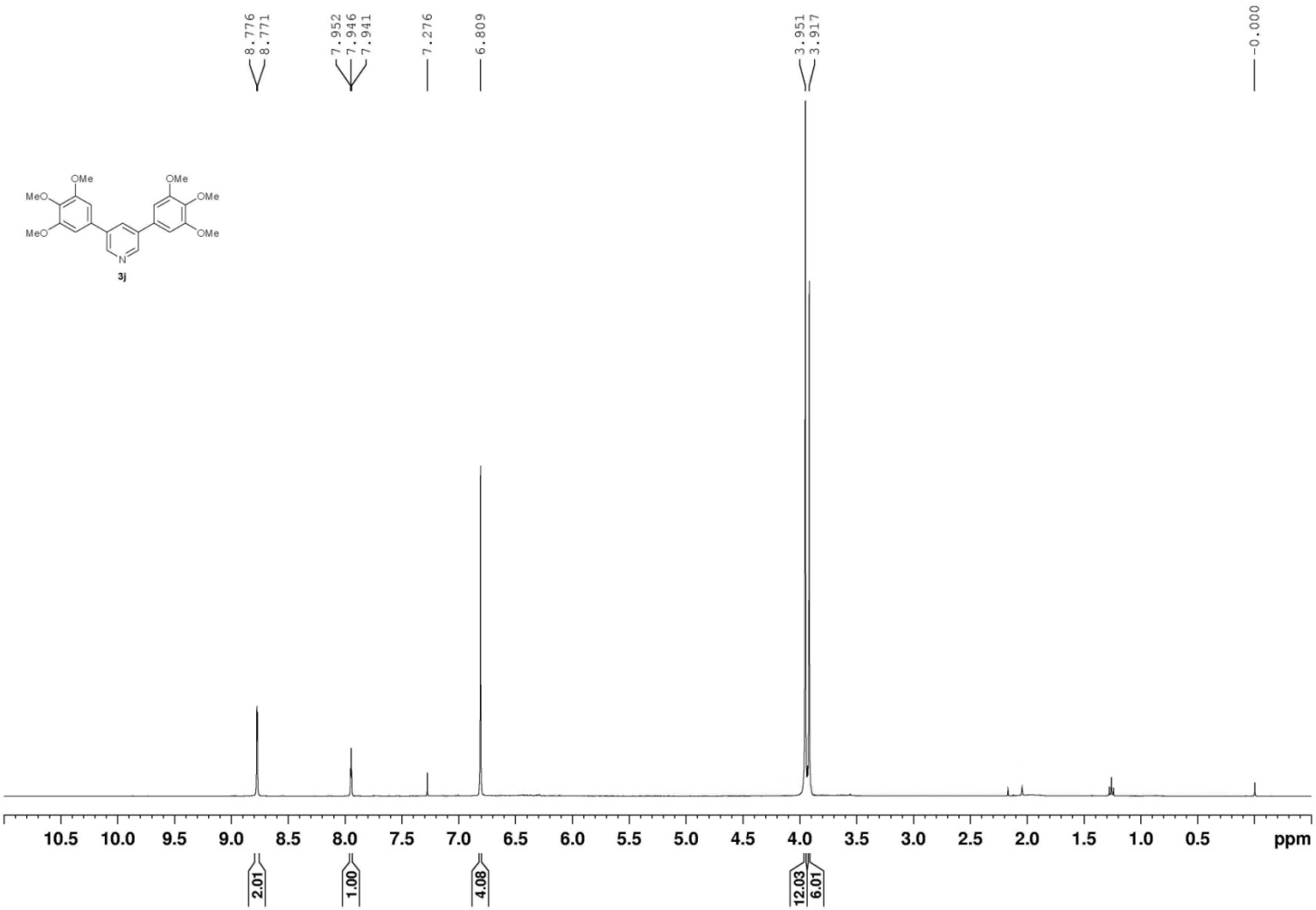
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7.276
6.809
3.951
3.917
0.000
OMe
MeO
MeO
OMe
OMe
OMe
N
3j
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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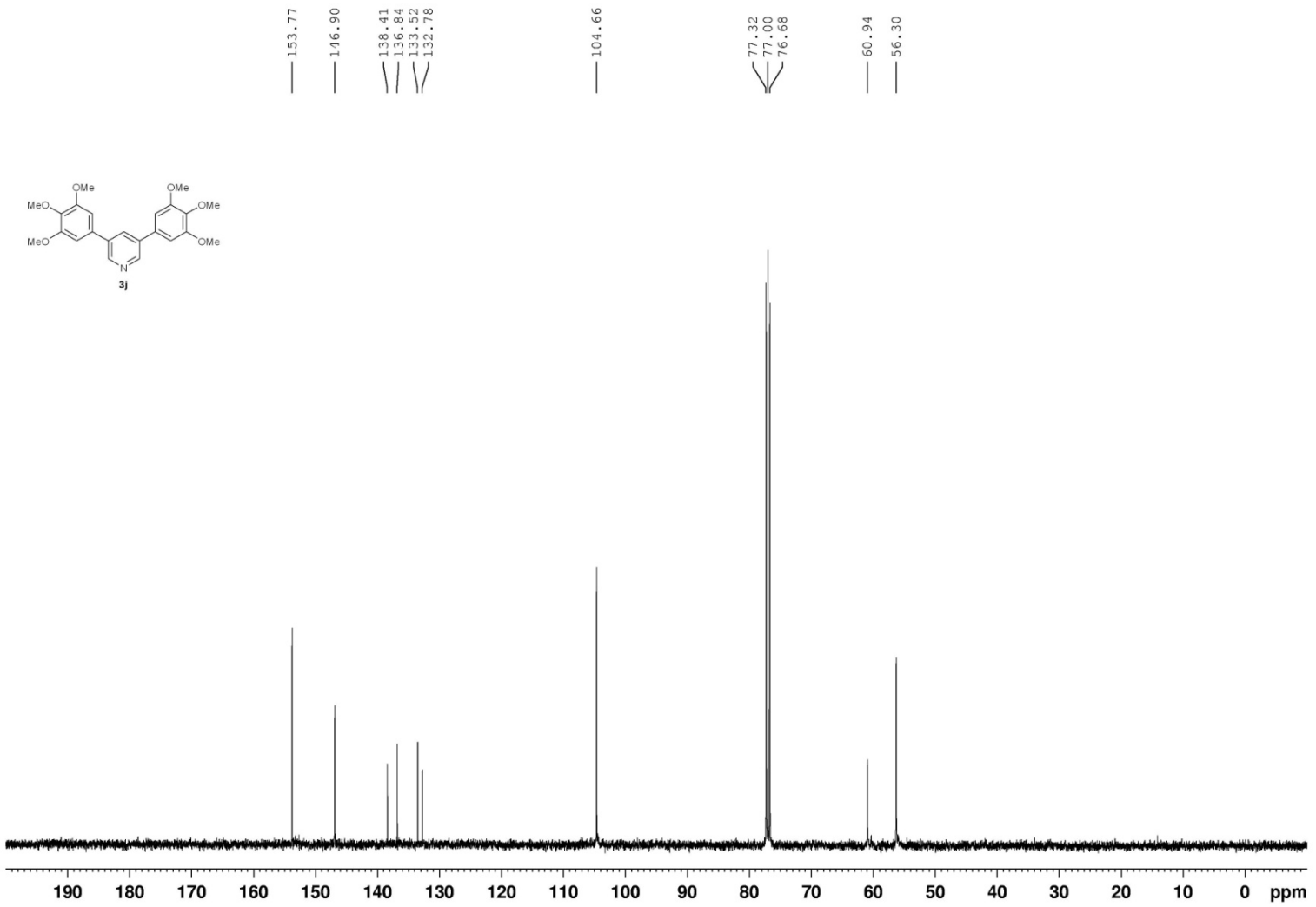
2.0
1.5
1.0
0.5
ppm
2.01
1.00
4.08
12.03
6.01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136.84
133.52
132.78
104.66
77.32
77.00
76.68
60.94
56.30
OMe
MeO
MeO
OMe
OMe
OMe
N
3j
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
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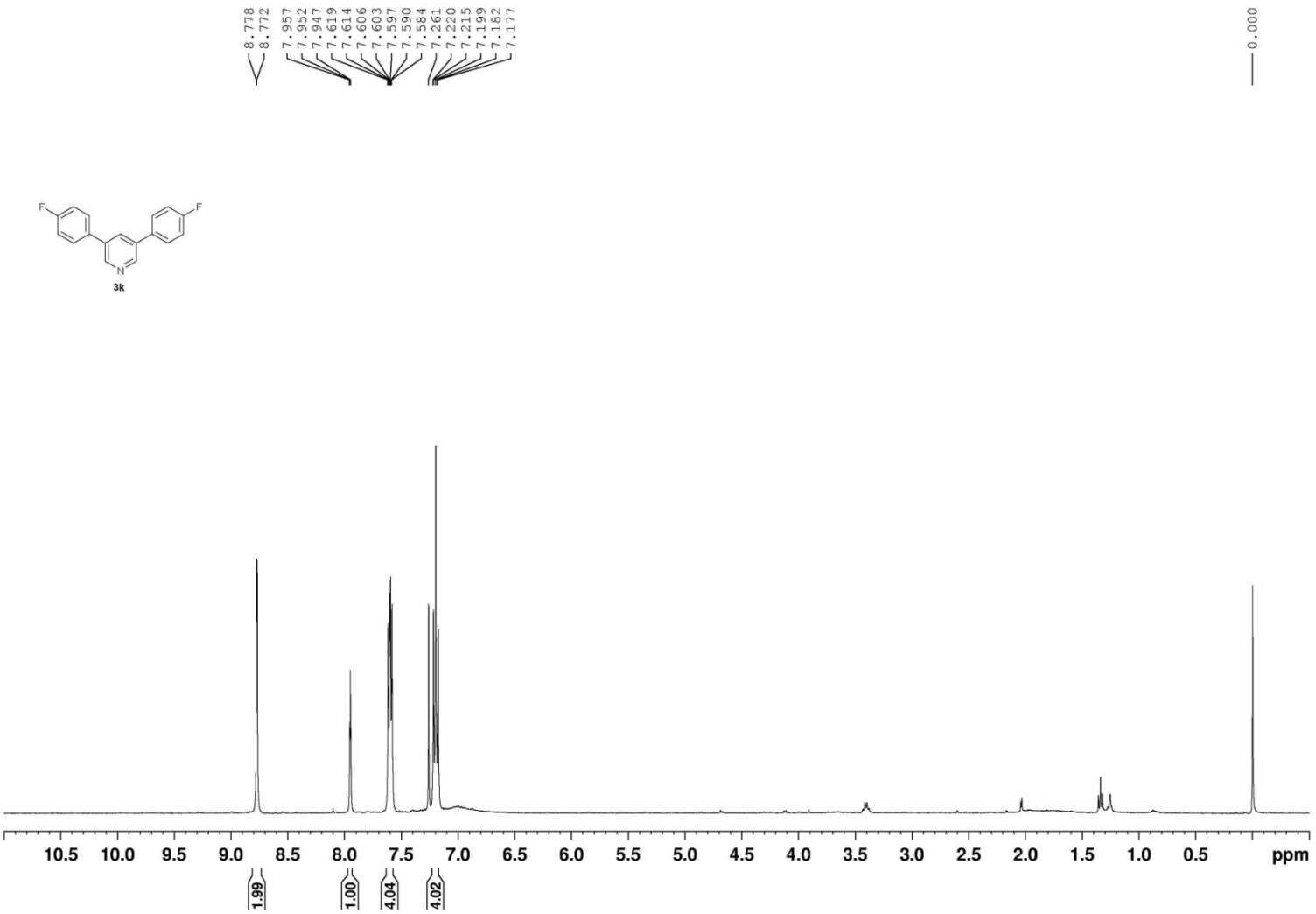
30
20
10
0
ppm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7.619
7.614
7.606
7.603
7.597
7.590
7.584
7.261
7.220
7.215
7.199
7.182
7.177
0.000
F
F
N
3k
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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2.5
2.0
1.5
1.0
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ppm
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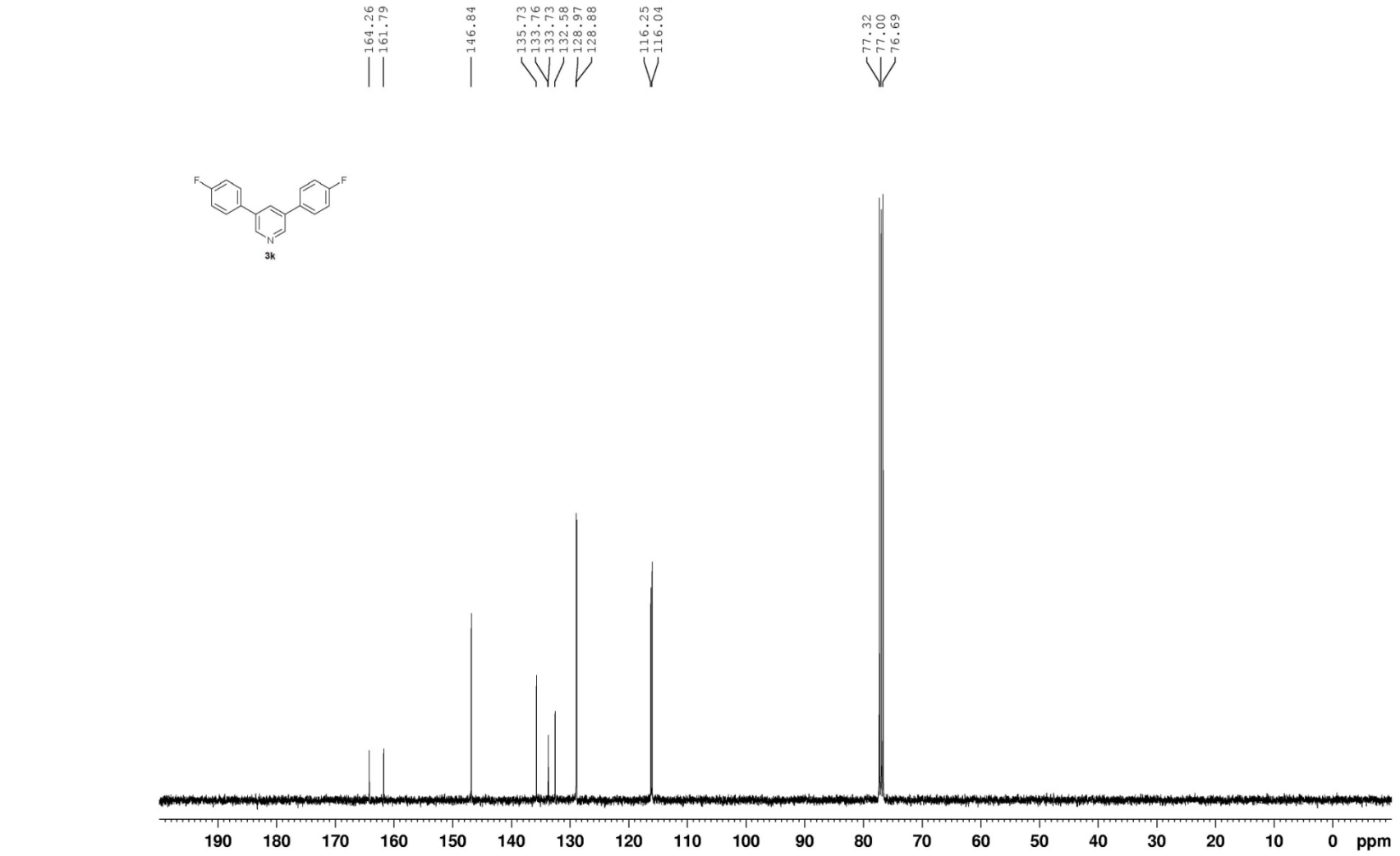
1.00
4.04
4.02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161.79
146.84
135.73
133.76
133.73
132.58
128.97
128.88
116.25
116.04
77.32
77.00
76.69
F
F
N
3k
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180
170
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150
140
130
120
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100
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80
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60
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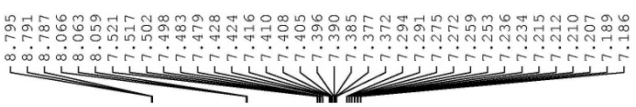
ppm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8.059
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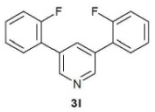
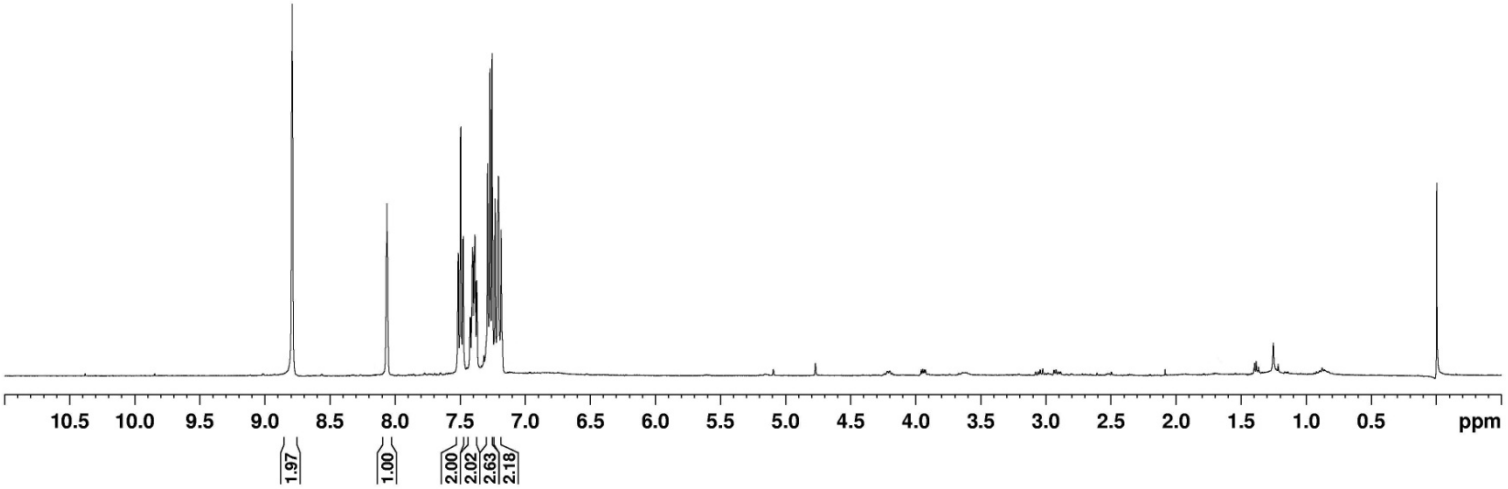
7.517
7.502
7.498
7.483
7.479
7.428
7.424
7.416
7.410
7.408
7.405
7.396
7.390
7.385
7.377
7.372
7.294
7.291
7.275
7.272
7.259
7.253
7.236
7.234
7.215
7.212
7.210
7.207
7.189
7.186

0.000


F
F
N

3l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9.5
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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
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1.5
1.0
0.5
ppm
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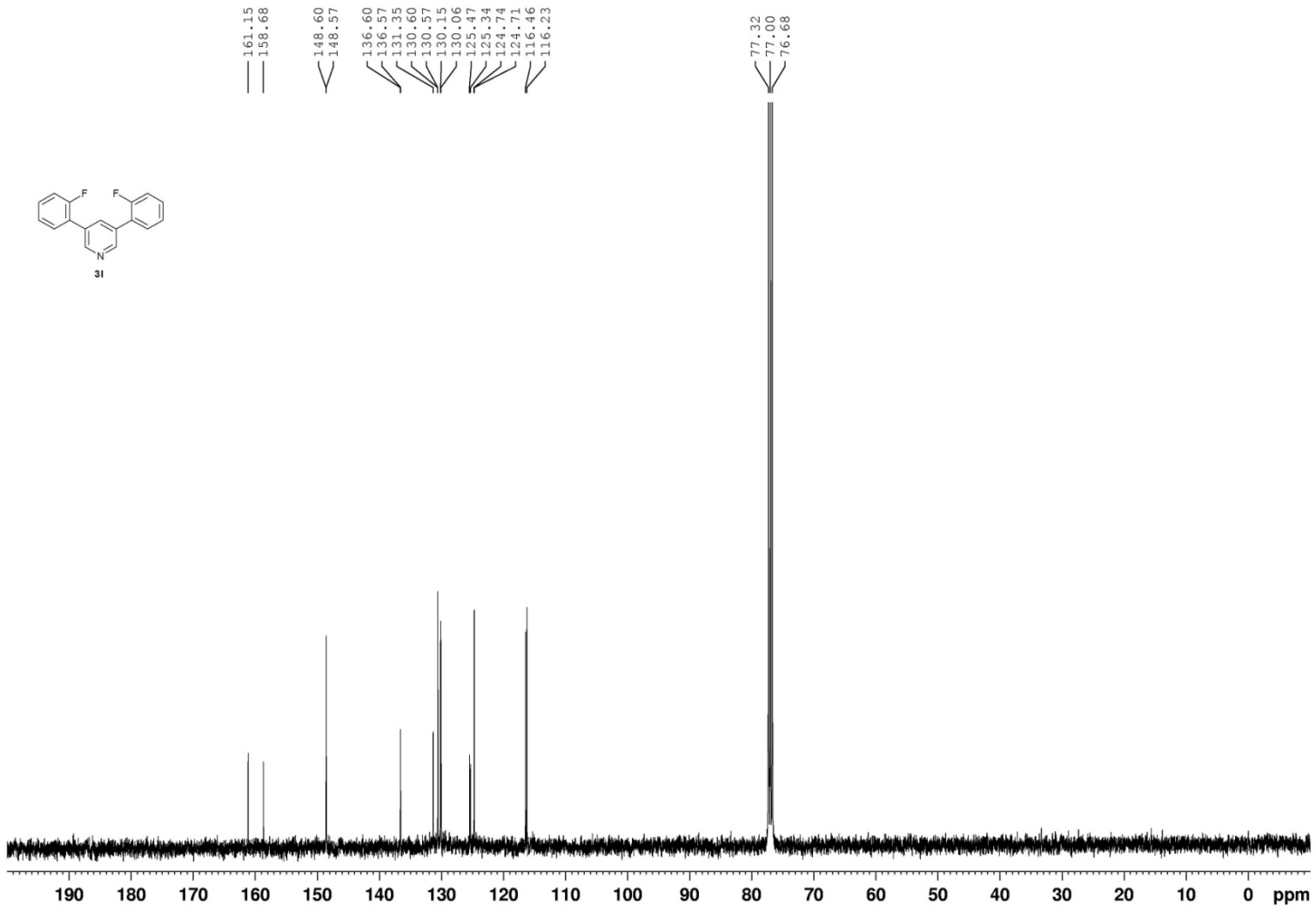
1.00
2.00
2.02
2.63
2.18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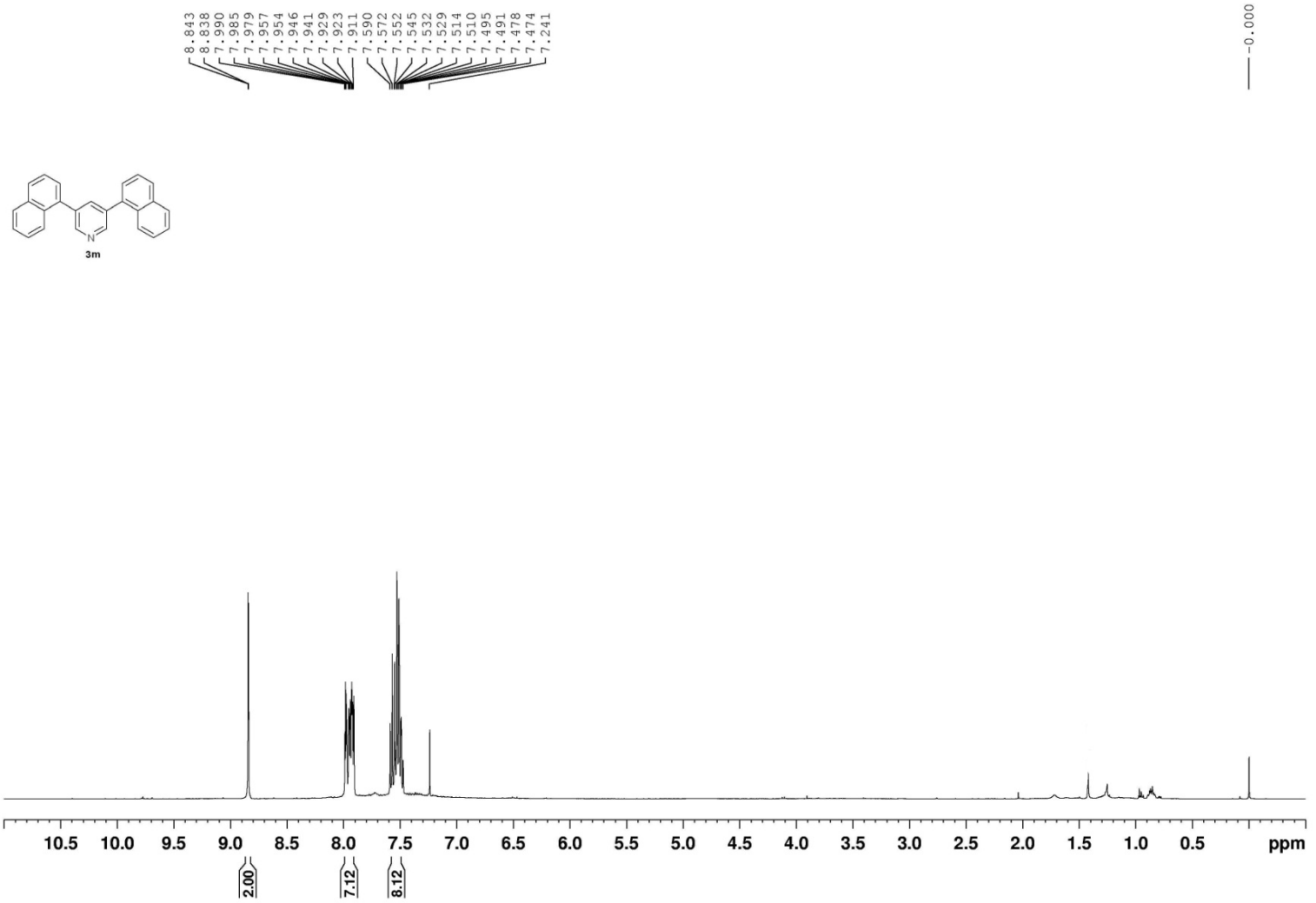
148.57
136.60
136.57
131.35
130.60
130.57
130.15
130.06
125.47
125.34
124.74
124.71
116.46
116.23
77.32
77.00
76.68
F
F
N
3l
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.941
7.929
7.923
7.911
7.590
7.572
7.552
7.545
7.532
7.529
7.514
7.510
7.495
7.491
7.478
7.474
7.241
-0.000
3m
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.0
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2.0
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0.5
ppm
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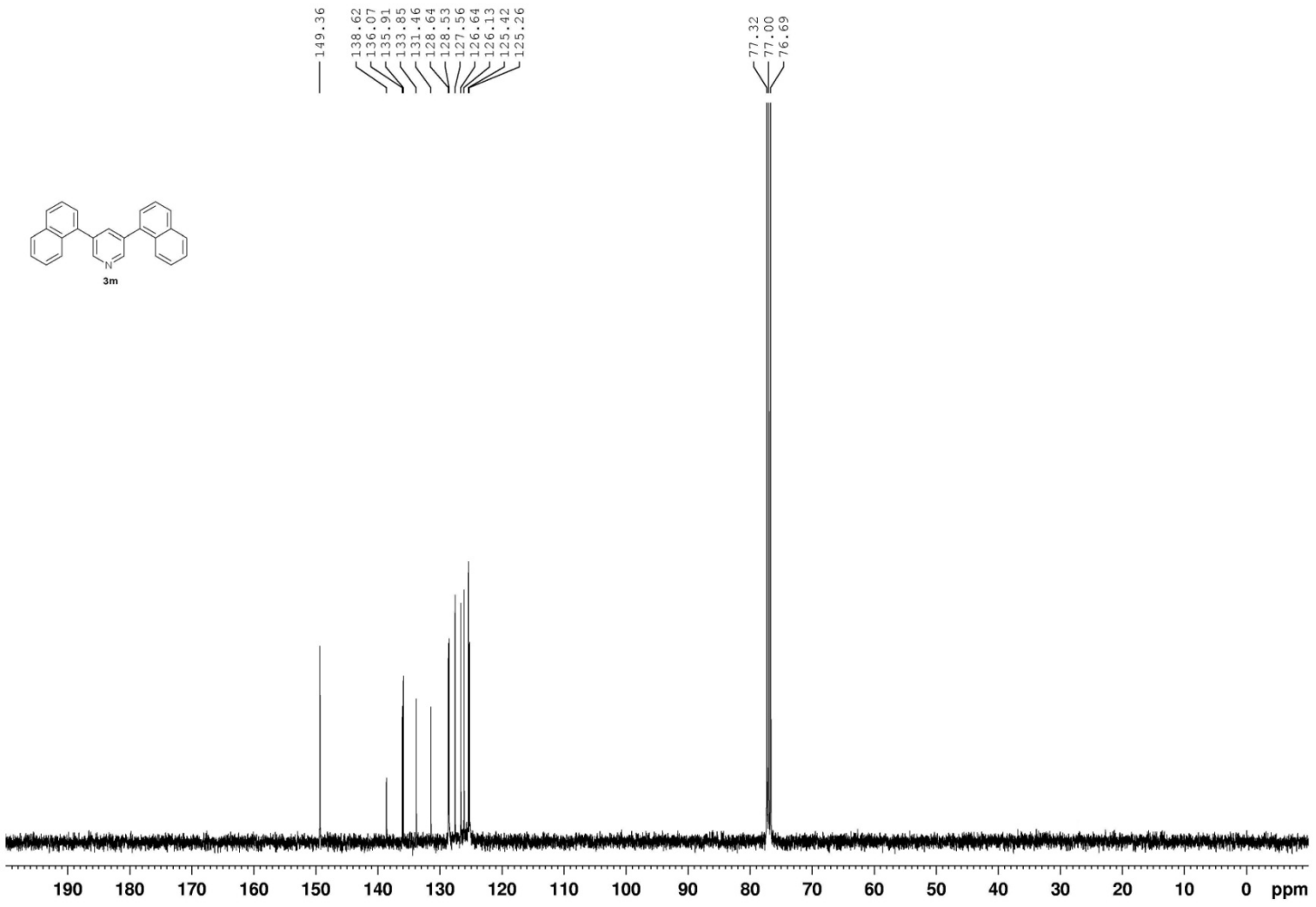
7.12
8.12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131.46
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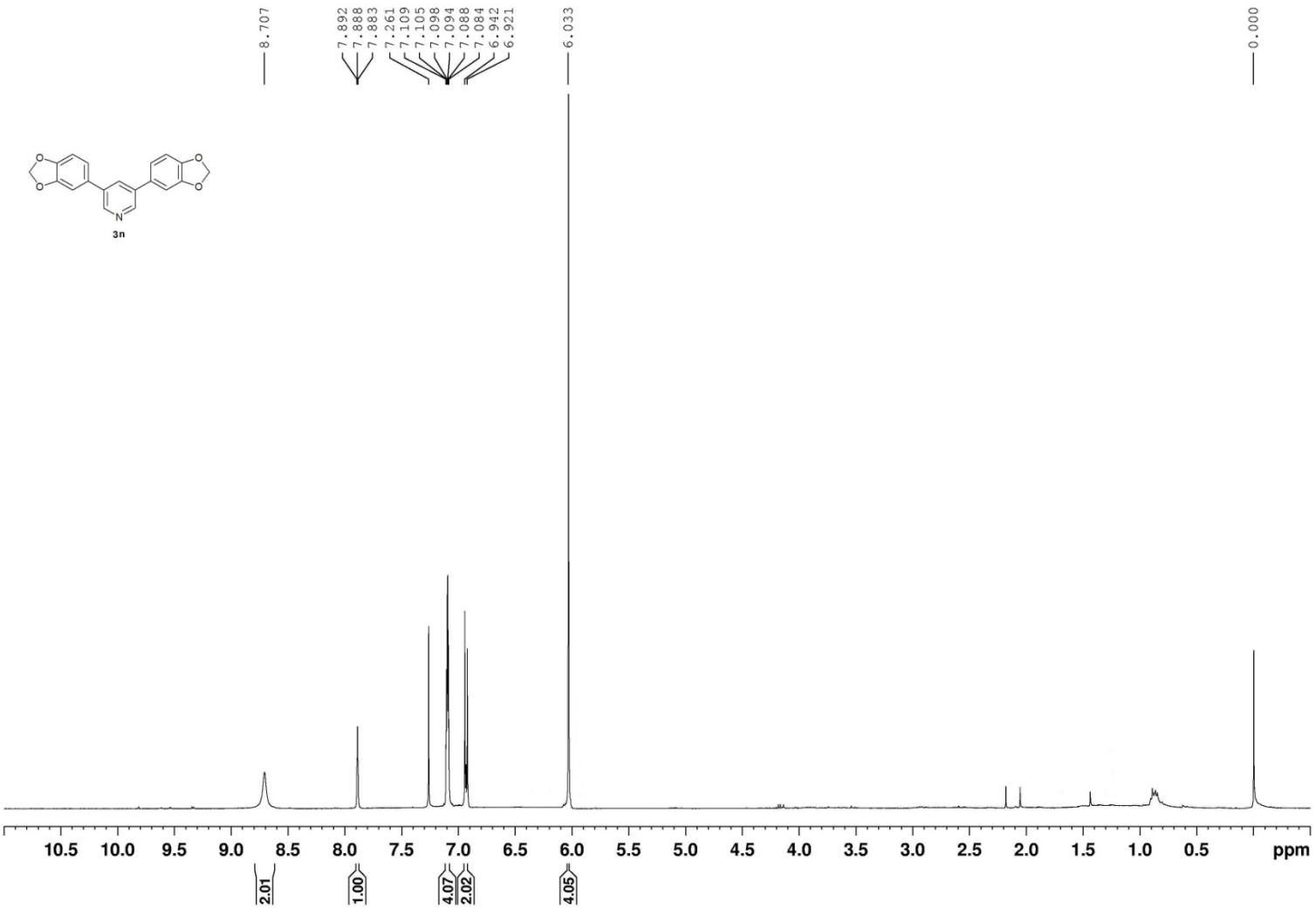
128.53
127.56
126.64
126.13
125.42
125.26
77.32
77.00
76.69
N
3m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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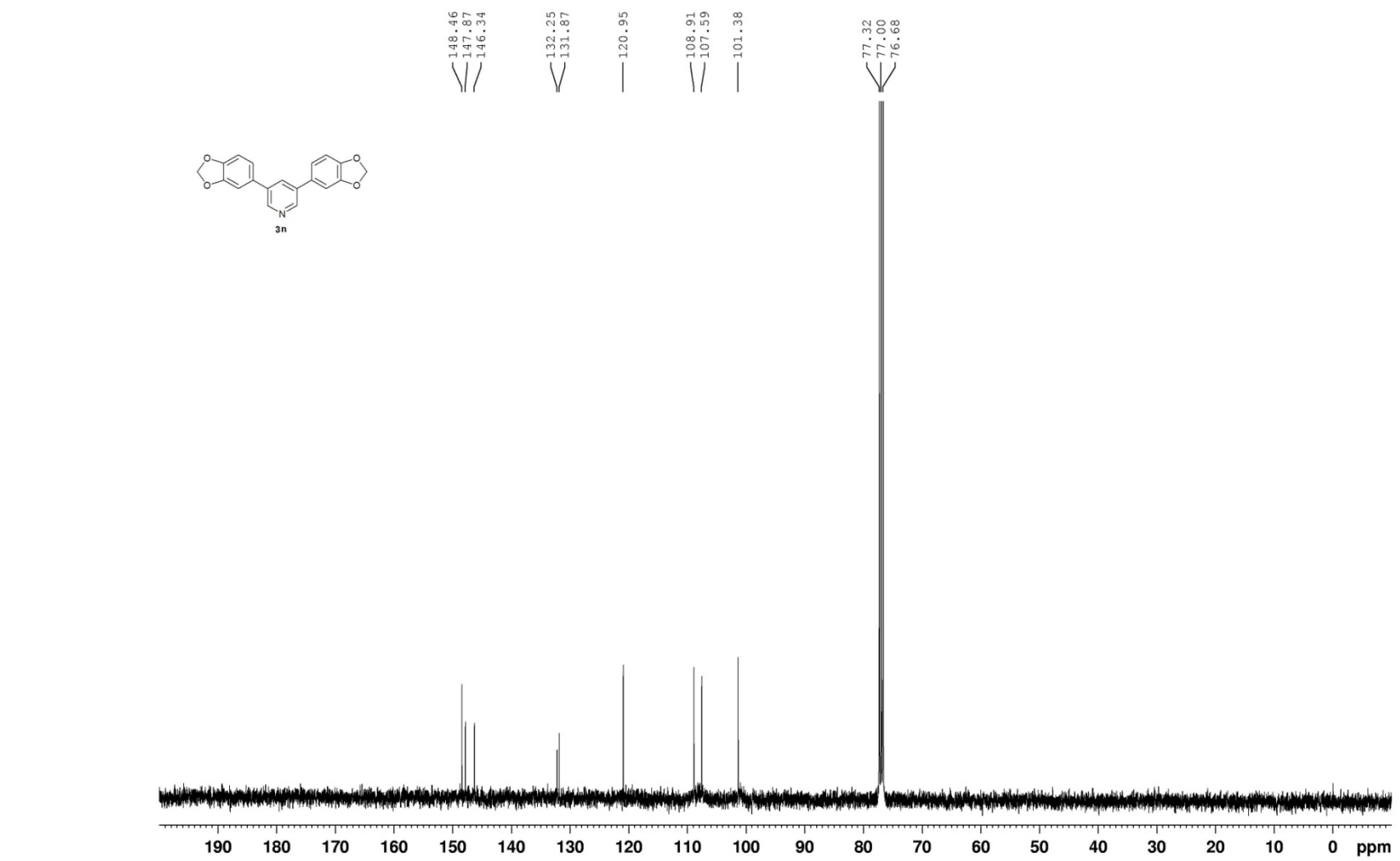
7.105
7.098
7.094
7.088
7.084
6.942
6.921
6.033
0.000
3n
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.01
1.00
4.07
2.02
4.05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132.25
131.87
120.95
108.91
107.59
101.38
77.32
77.00
76.68
3n
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180
170
160
150
140
130
120
110
100
90
80
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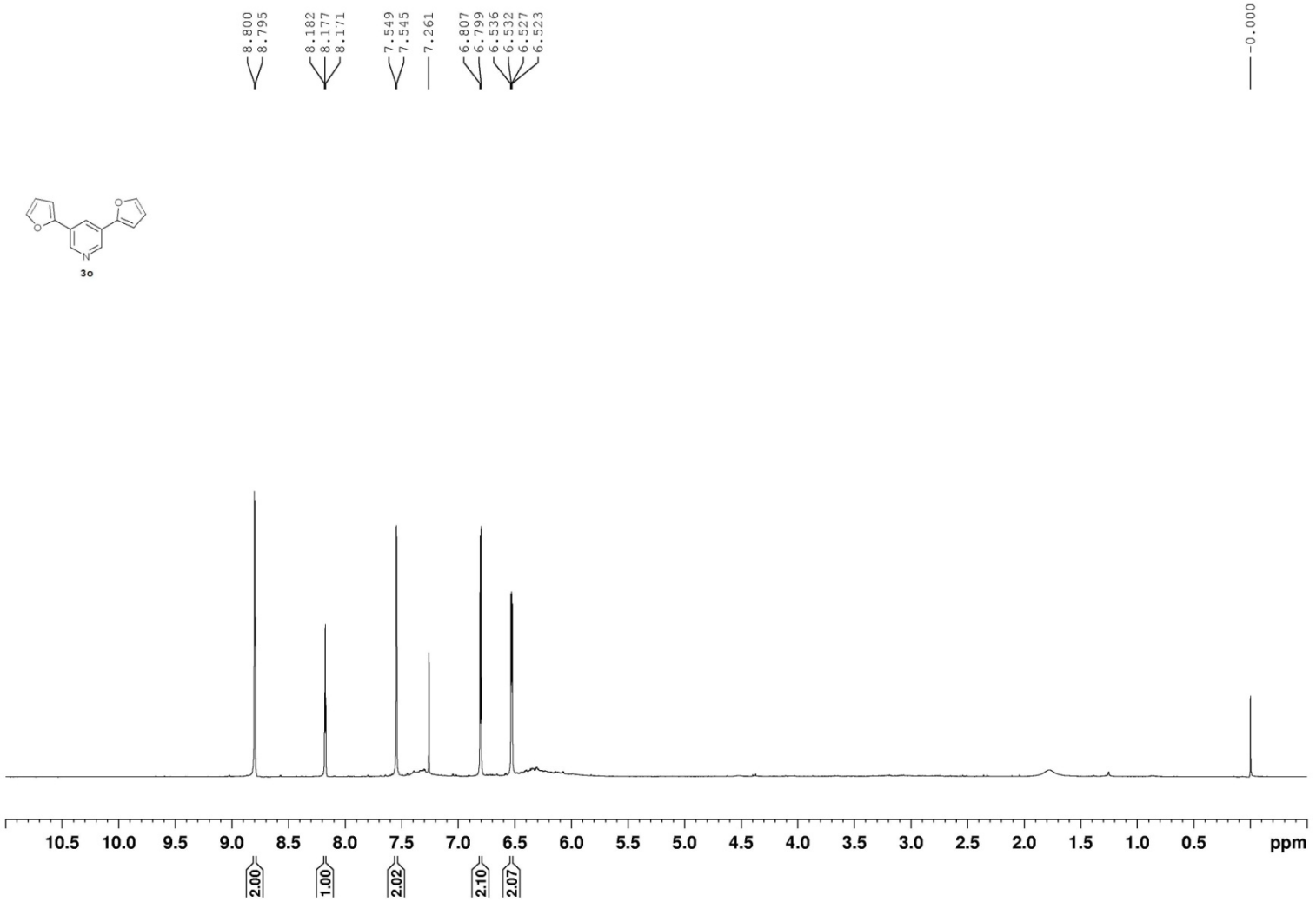
60
50
40
30
20
10
0
ppm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8.171
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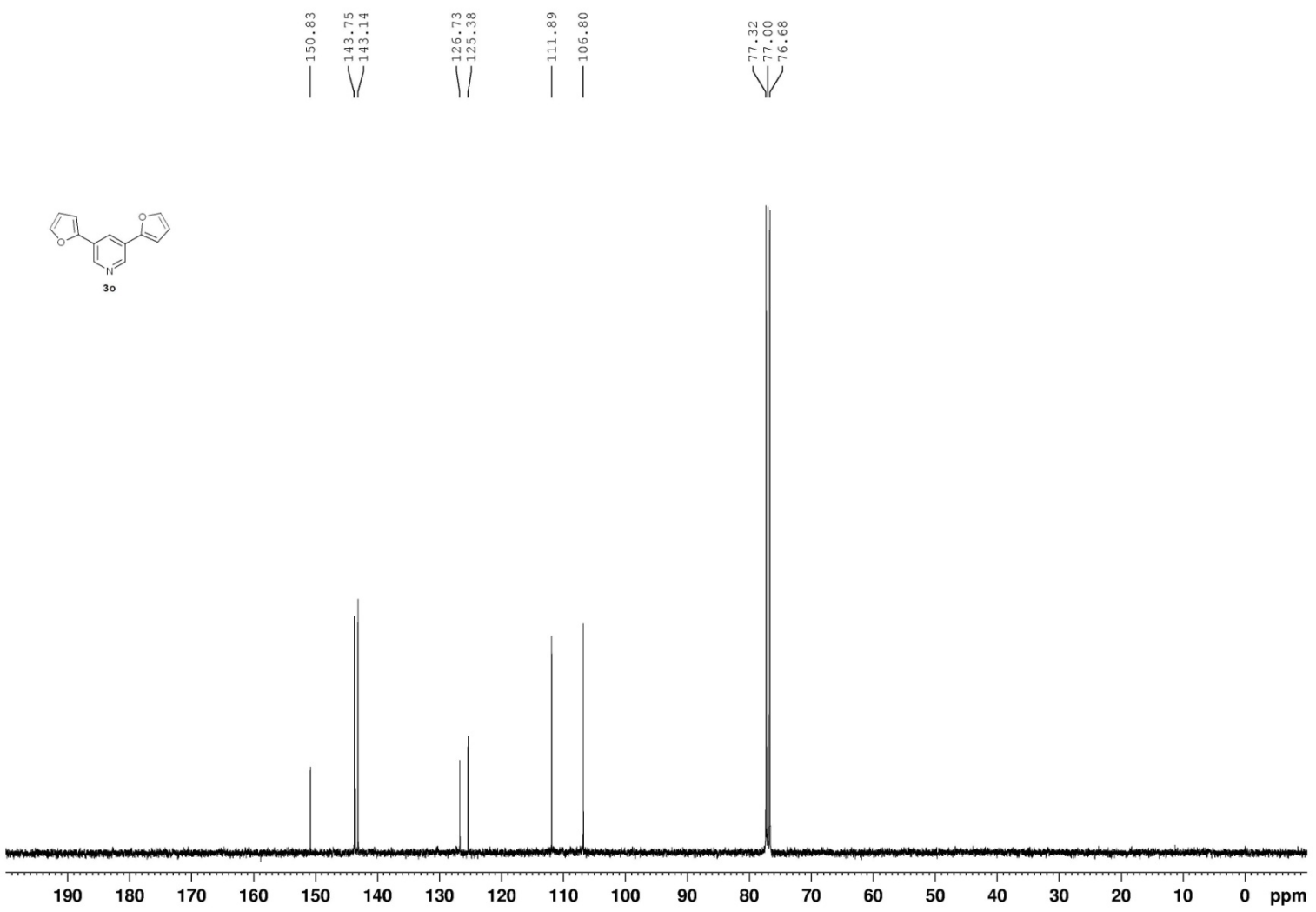
7.545
7.261
6.807
6.799
6.536
6.532
6.527
6.523
0.000
3o
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.00
1.00
2.02
2.10
2.07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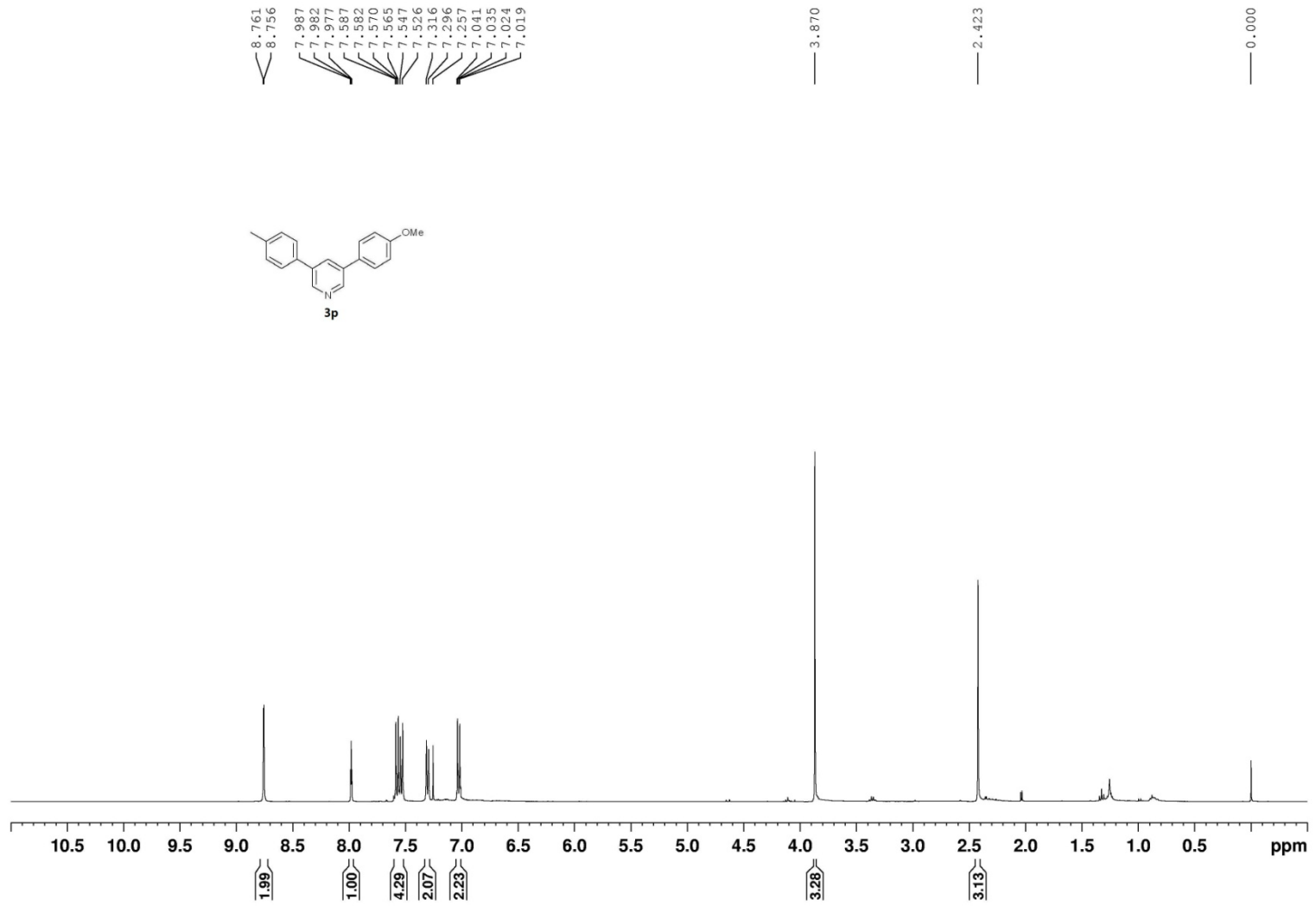
106.80
77.32
77.00
76.68
3o
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.570
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7.547
7.526
7.316
7.296
7.257
7.041
7.035
7.024
7.019
3.870
2.423
0.000
OMe
N
3p
10.5
10.0
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
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1.0
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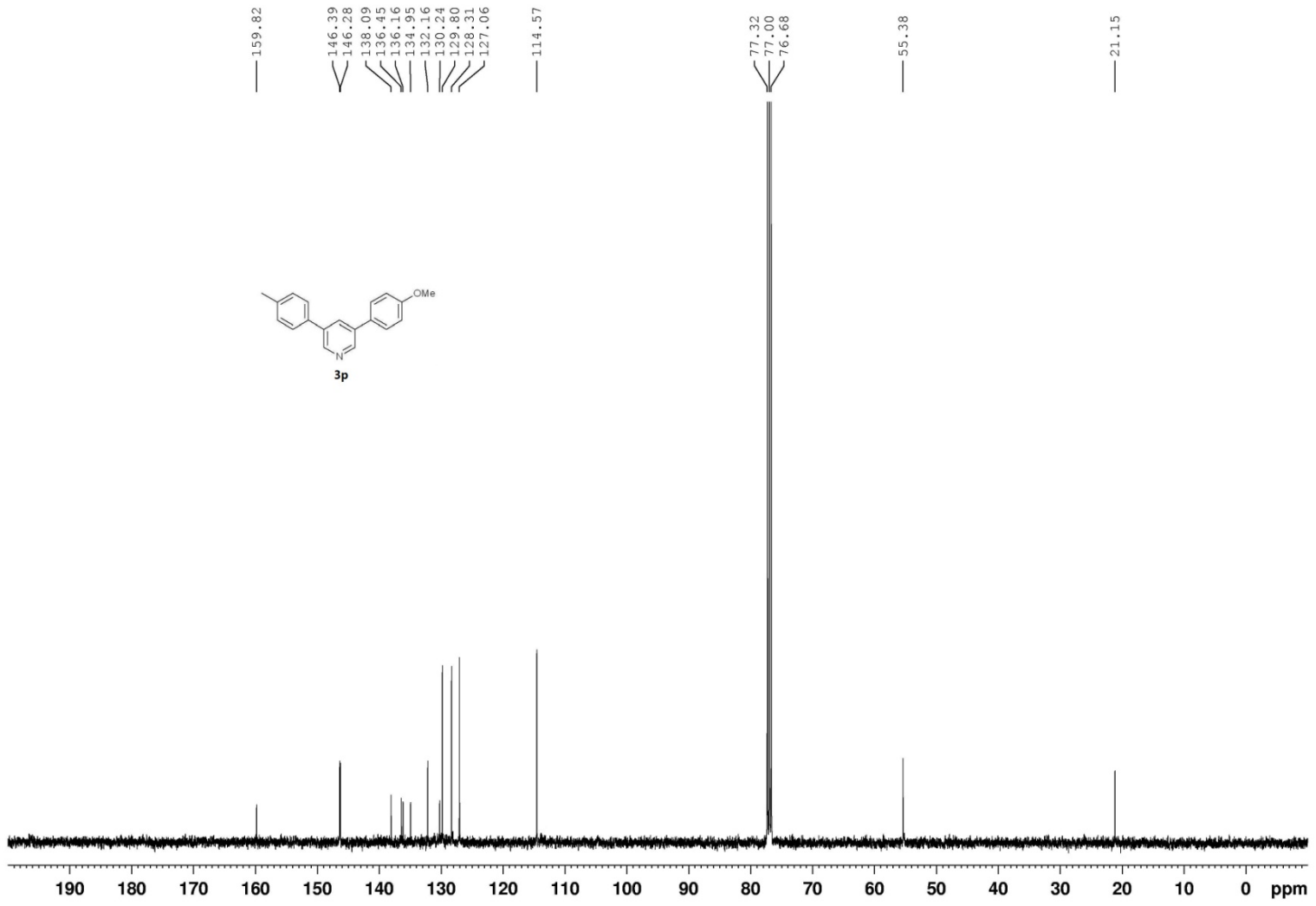
3.13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132.16
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129.80
128.31
127.06
114.57
77.32
77.00
76.68
55.38
21.15
OMe
N
3p
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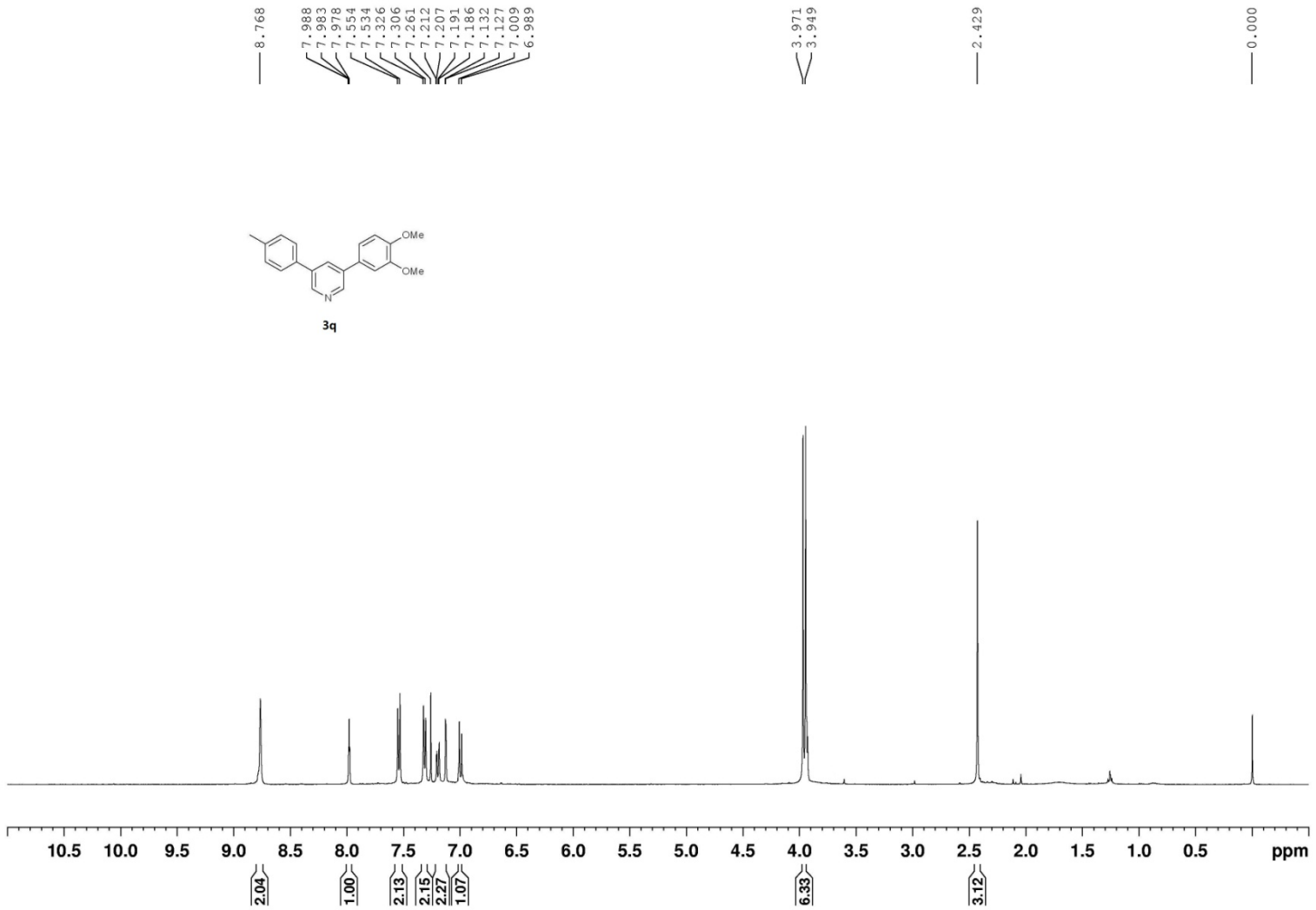
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7.207
7.191
7.186
7.132
7.127
7.009
6.989
3.971
3.949
2.429
0.000
OMe
OMe
N
3q
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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ppm
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2.15
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1.07
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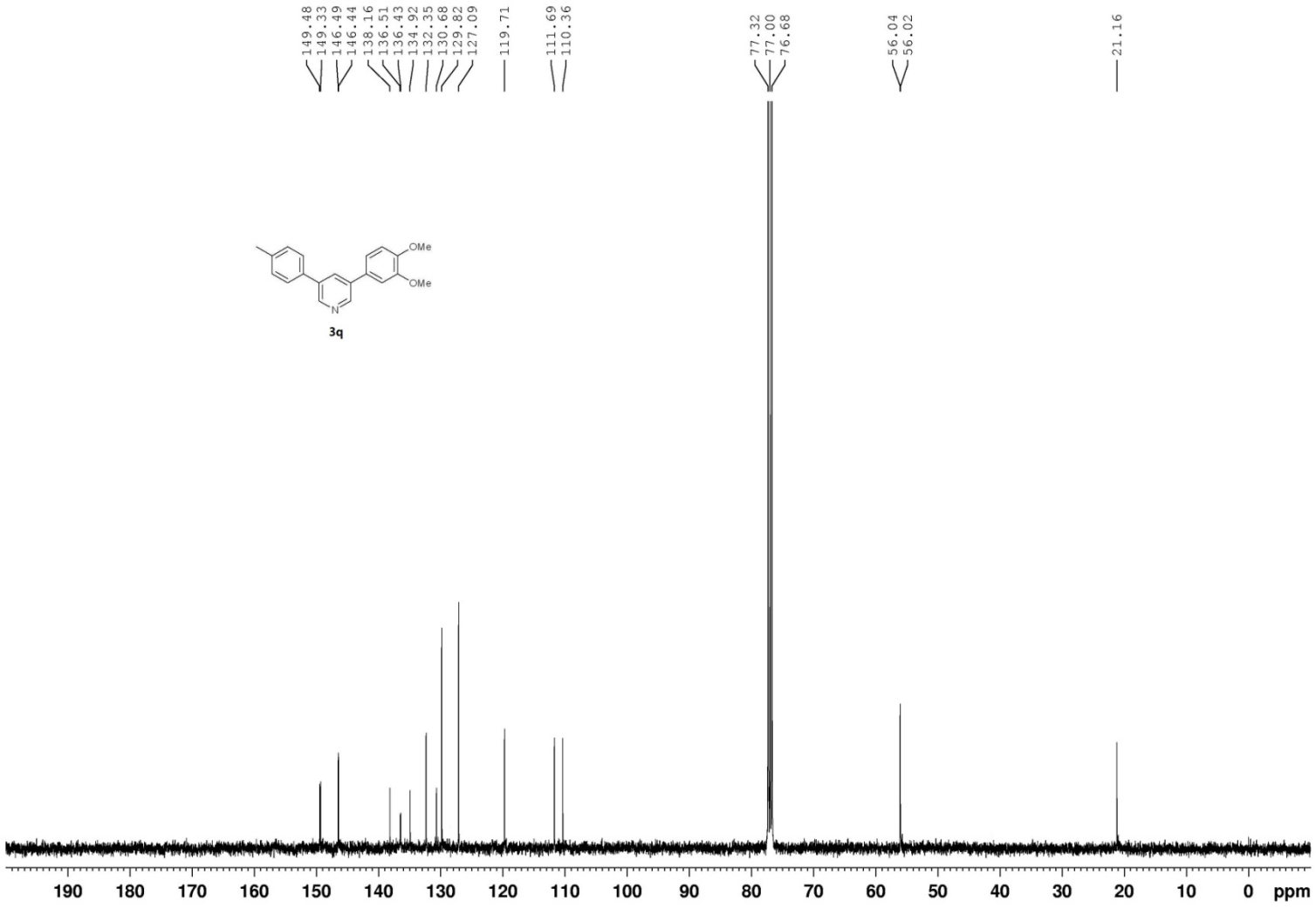
3.12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134.92
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130.68
129.82
127.09
119.71
111.69
110.36
77.32
77.00
76.68
56.04
56.02
21.16
OMe
OMe
N
3q
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140
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120
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100
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80
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60
50
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20
10
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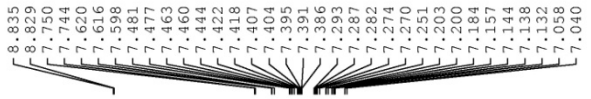
ppm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7.418
7.407
7.404
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7.391
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7.287
7.282
7.274
7.270
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7.132
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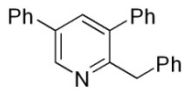
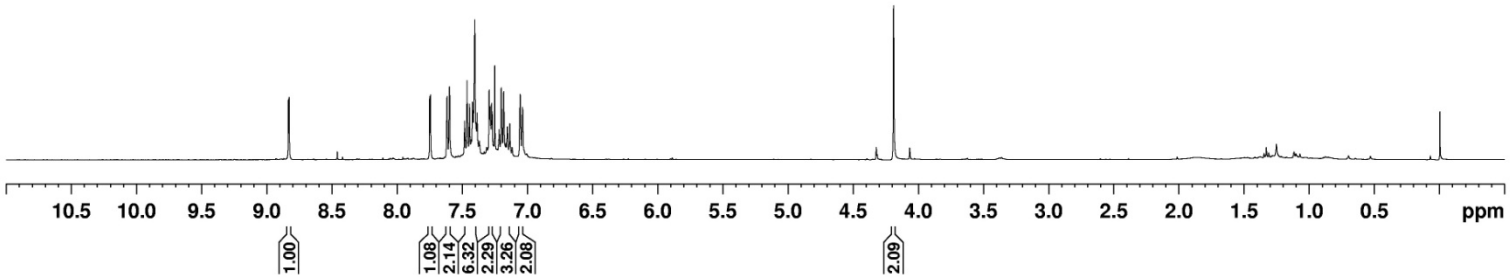
4.192

0.000


Ph
Ph
Ph
N

3aa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5.0
4.5
4.0
3.5
3.0
2.5
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6.32
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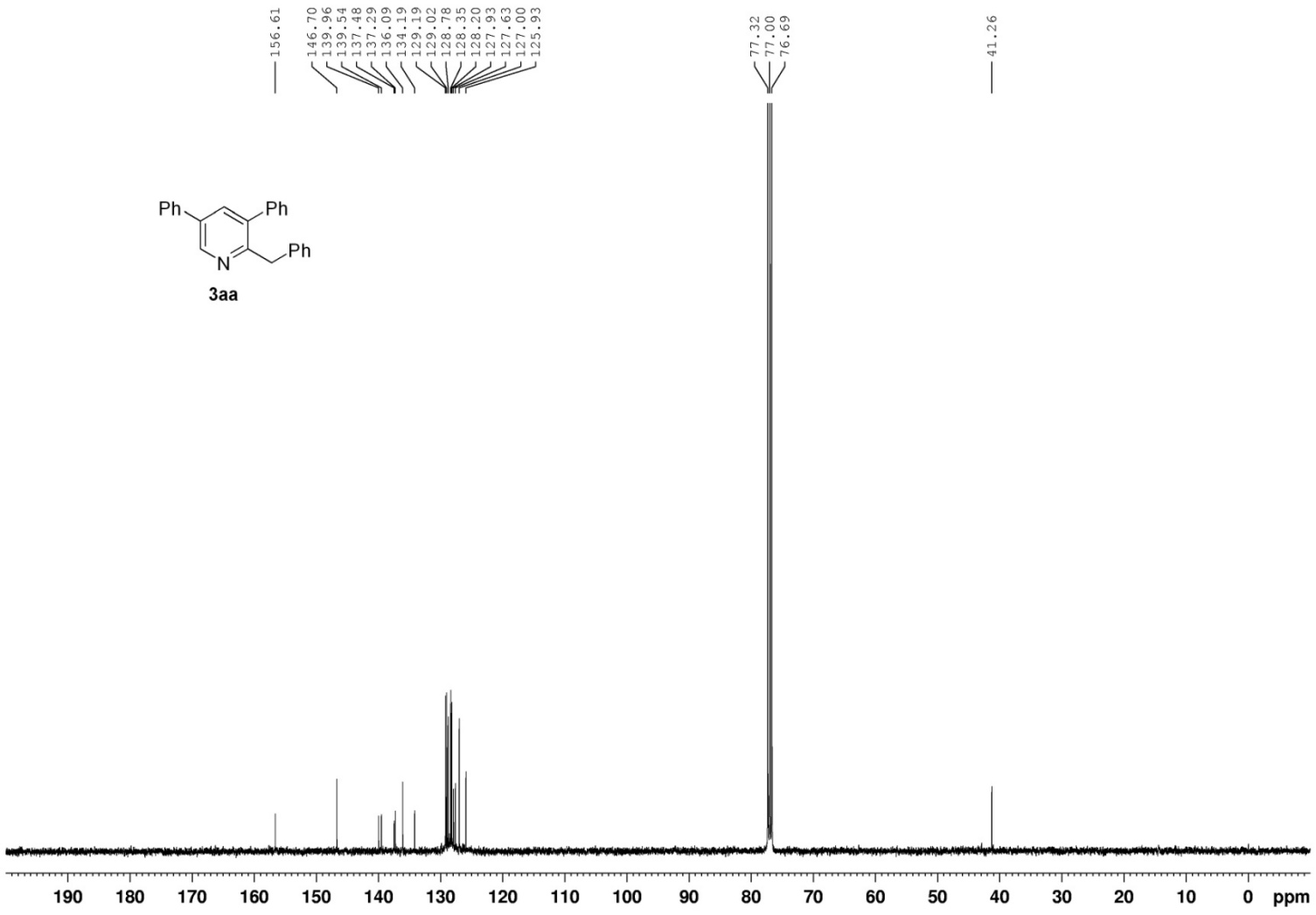
3.26
2.08
2.09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128.20
127.93
127.63
127.00
125.93
77.32
77.00
76.69
41.26
Ph
Ph
Ph
N
3aa
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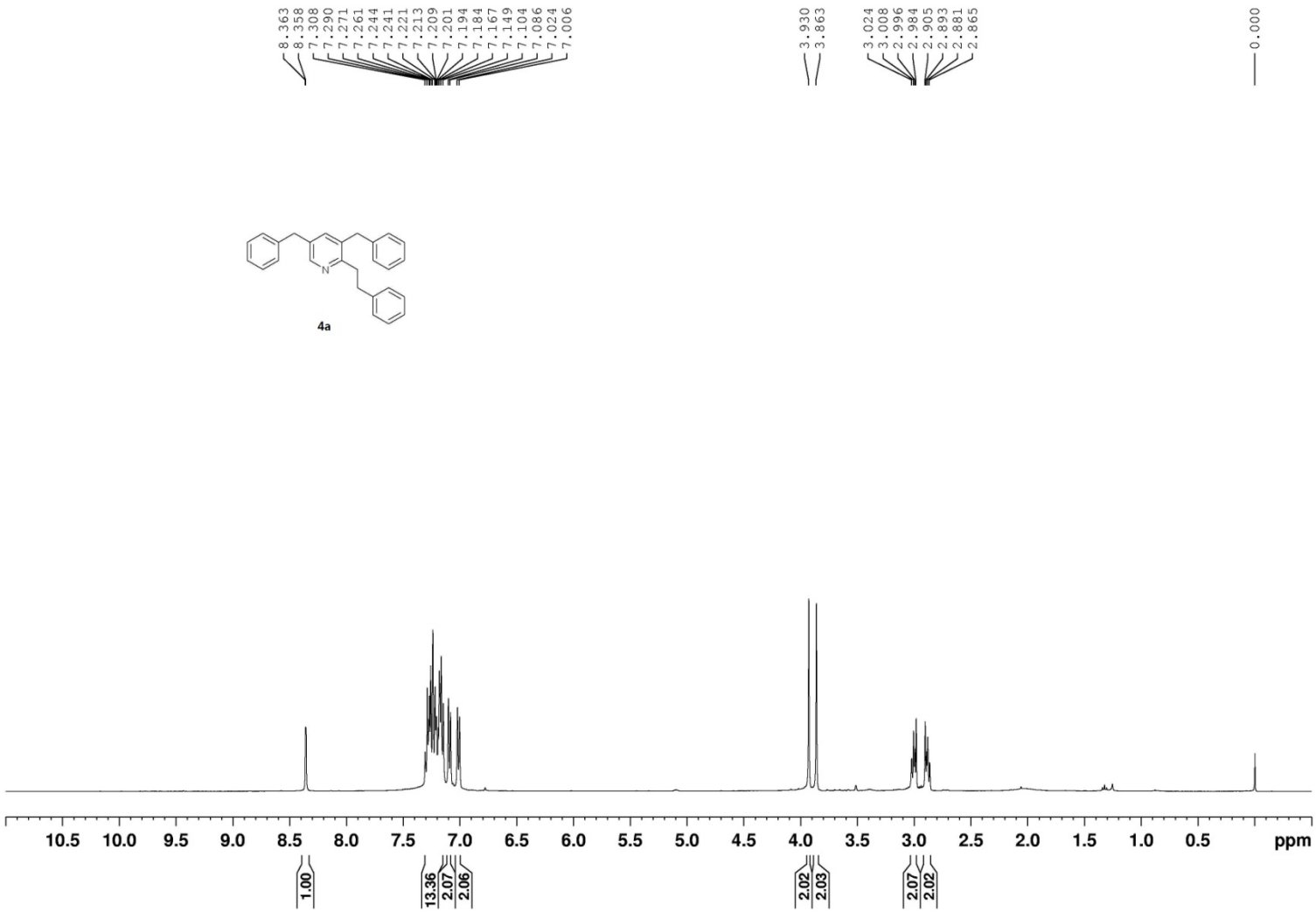
0
ppm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7.167
7.149
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7.086
7.024
7.006
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3.863
3.024
3.008
2.996
2.984
2.905
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2.881
2.865
0.000
4a
10.5 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 ppm
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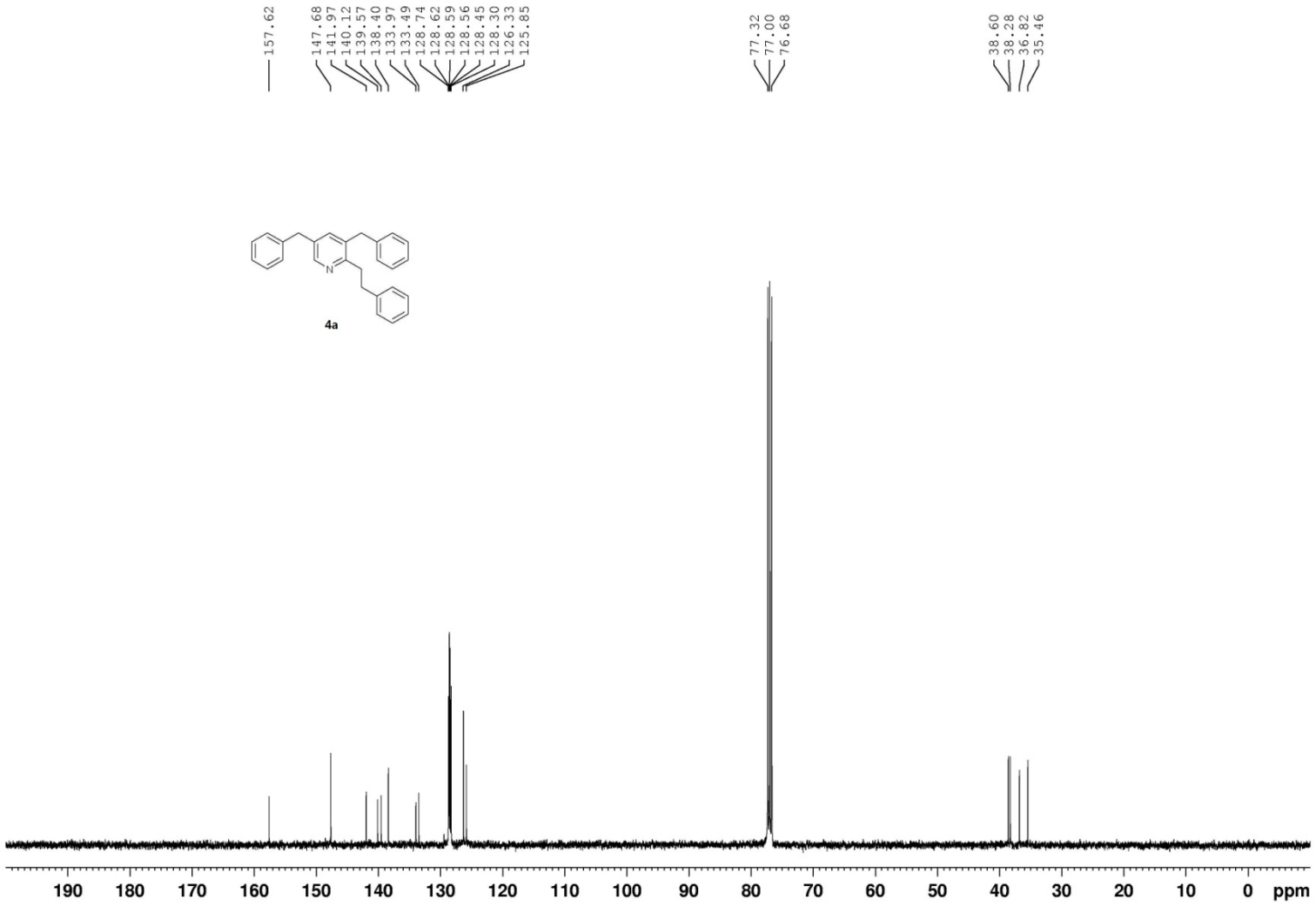
2.06
2.02
2.03
2.07
2.02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128.30
126.33
125.85
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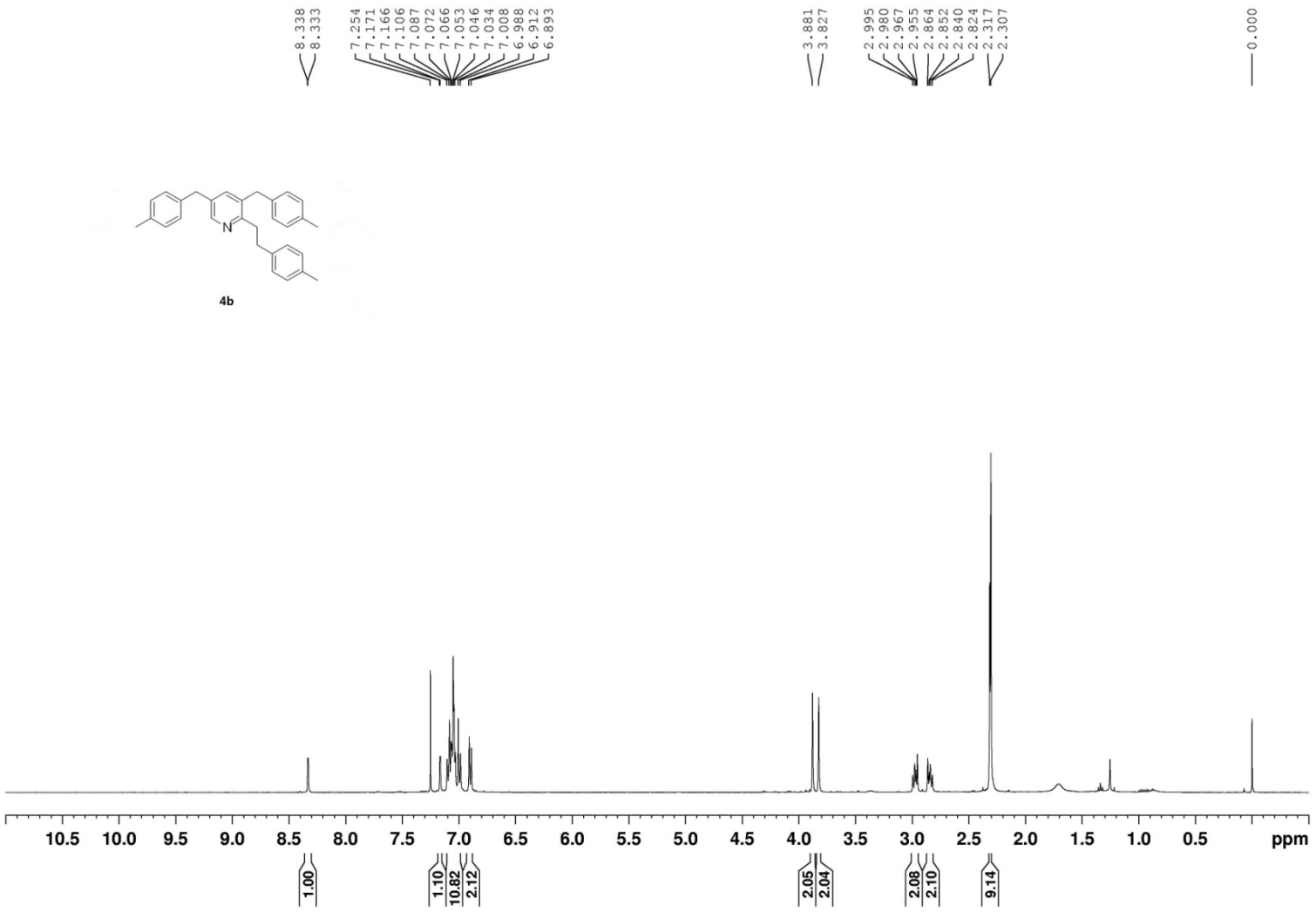
77.00
76.68
38.60
38.28
36.82
35.46
N
4a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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6.988
6.912
6.893
3.881
3.827
2.995
2.980
2.967
2.955
2.864
2.852
2.840
2.824
2.317
2.307
0.000
N
4b
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 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 ppm
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2.08
2.10
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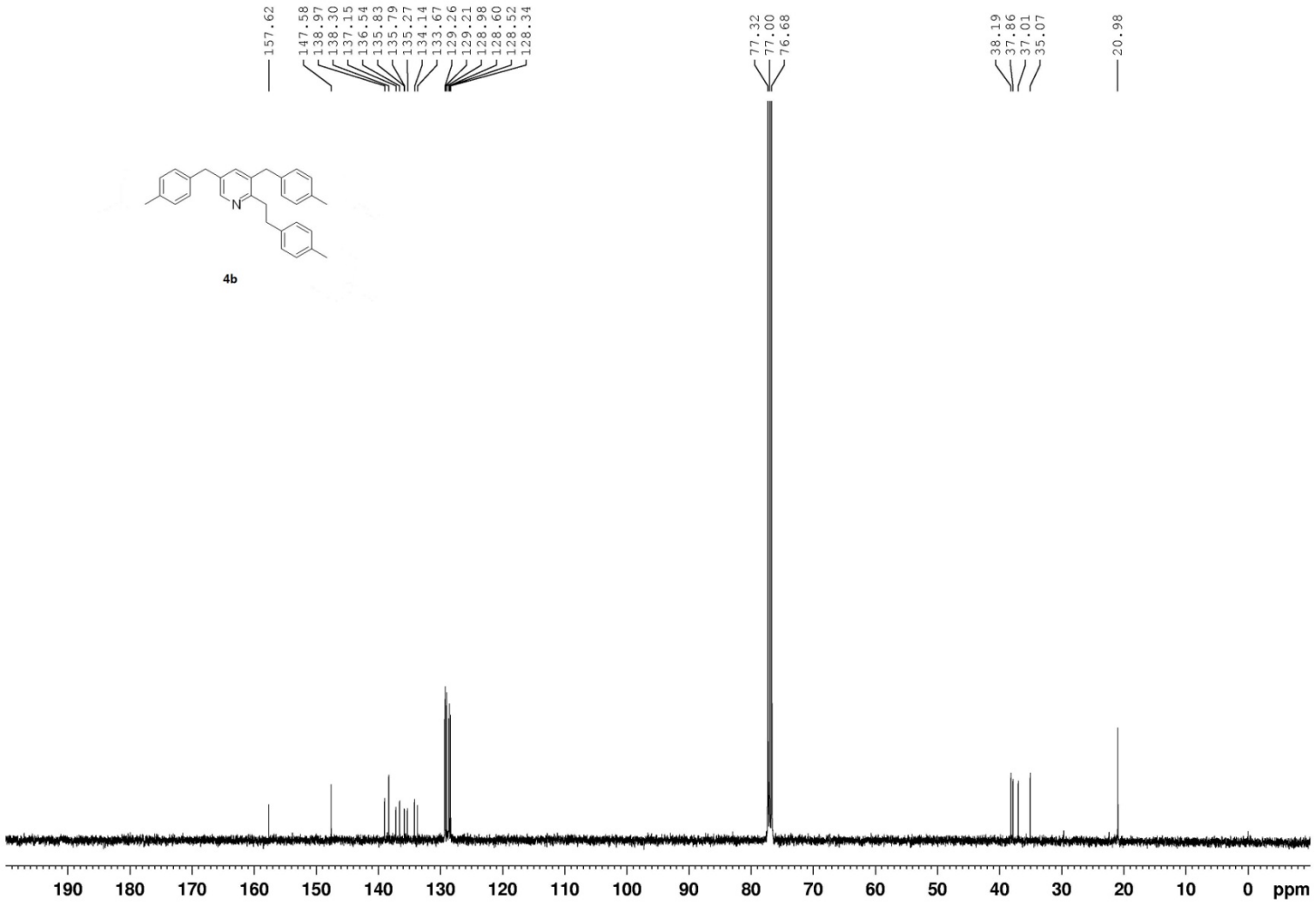
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128.98
128.60
128.52
128.34
77.32
77.00
76.68
38.19
37.86
37.01
35.07
20.98
N
4b
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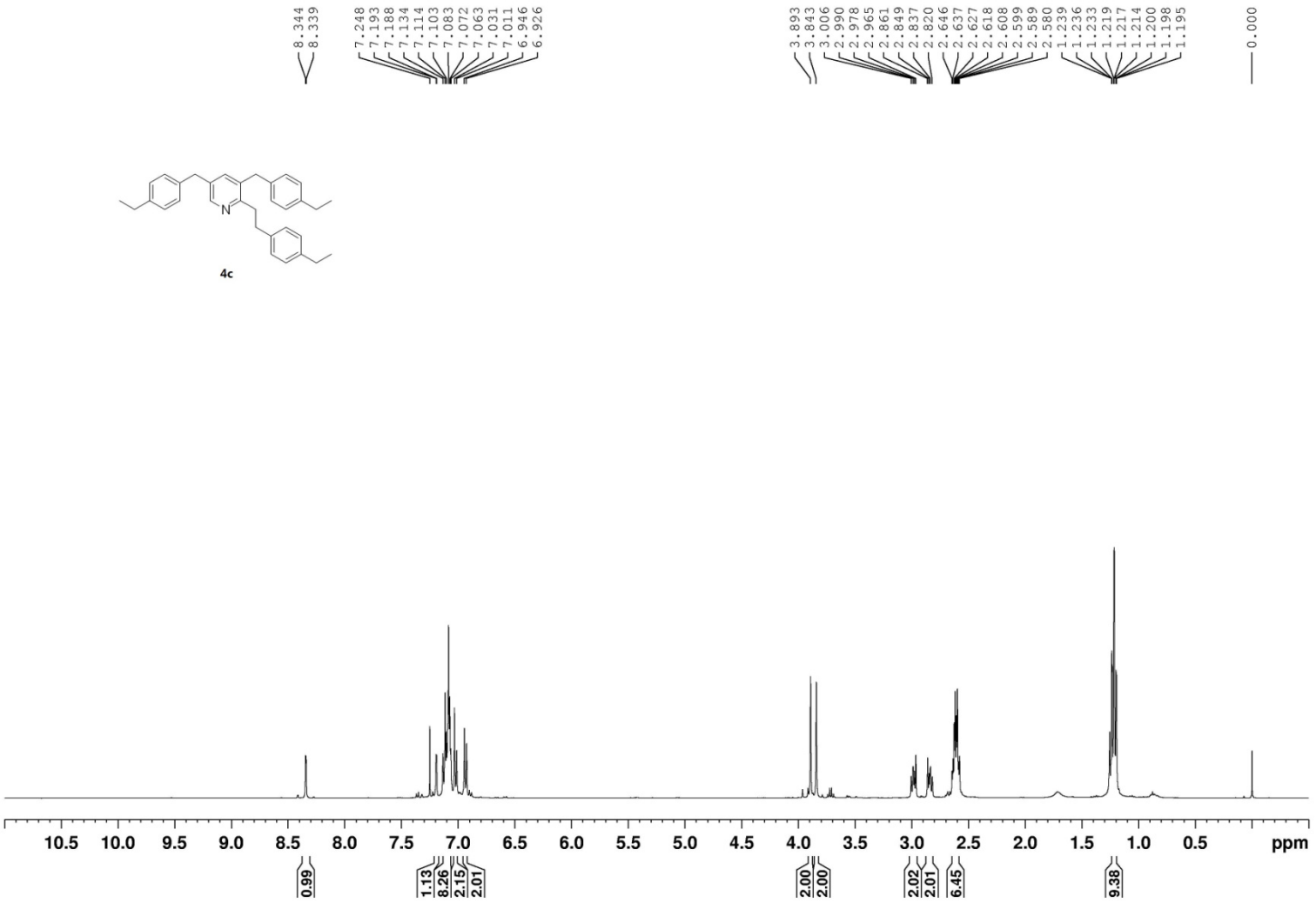
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7.011
6.946
6.926
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3.006
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2.978
2.965
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2.849
2.837
2.820
2.646
2.637
2.627
2.618
2.608
2.599
2.589
2.580
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1.236
1.233
1.219
1.217
1.214
1.200
1.198
1.195
0.000
N
4c
10.5 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 ppm
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2.00
2.02
2.01
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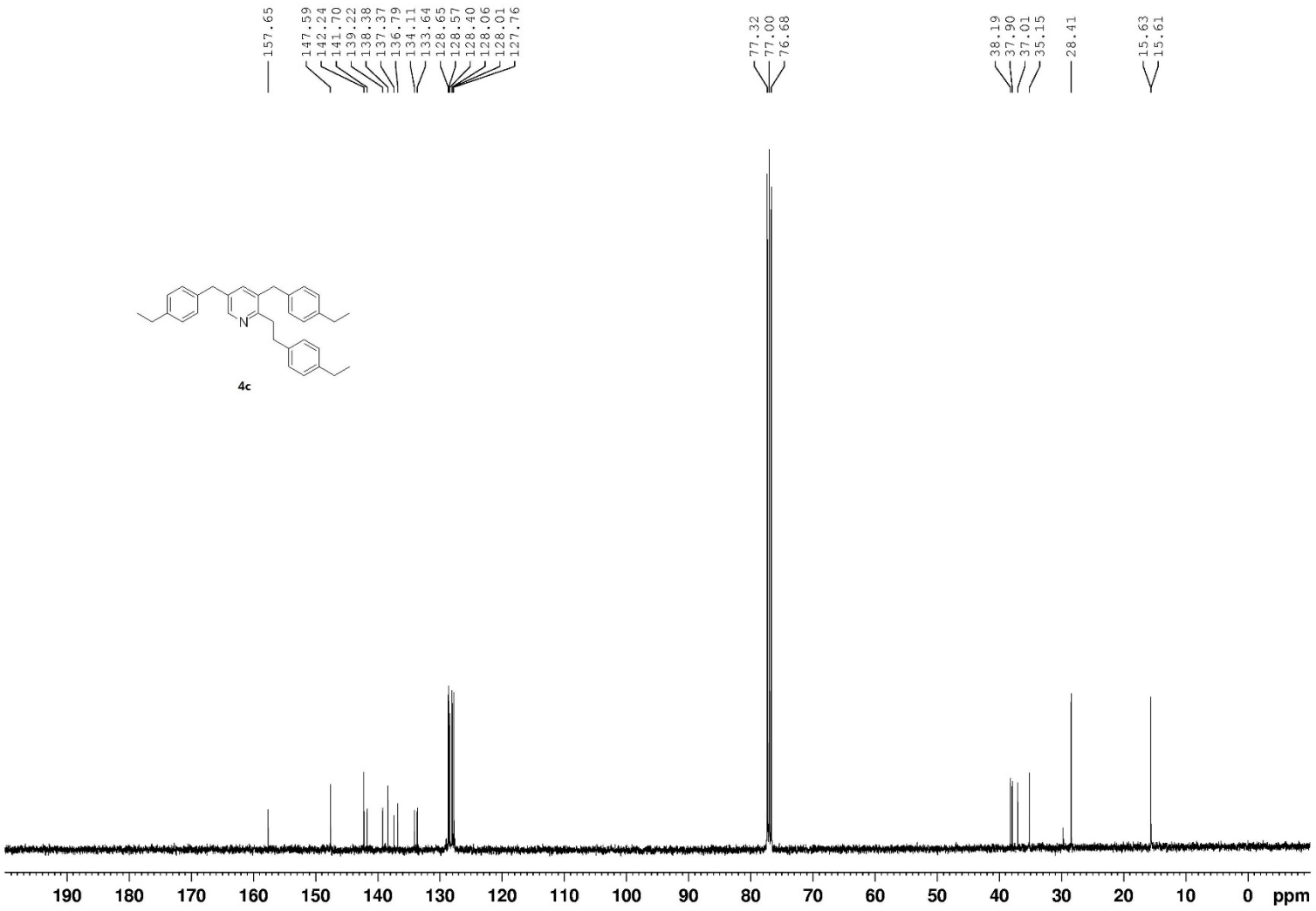
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128.40
128.06
128.01
127.76
77.32
77.00
76.68
38.19
37.90
37.01
35.15
28.41
15.63
15.61
N
4c
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180
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140
130
120
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60
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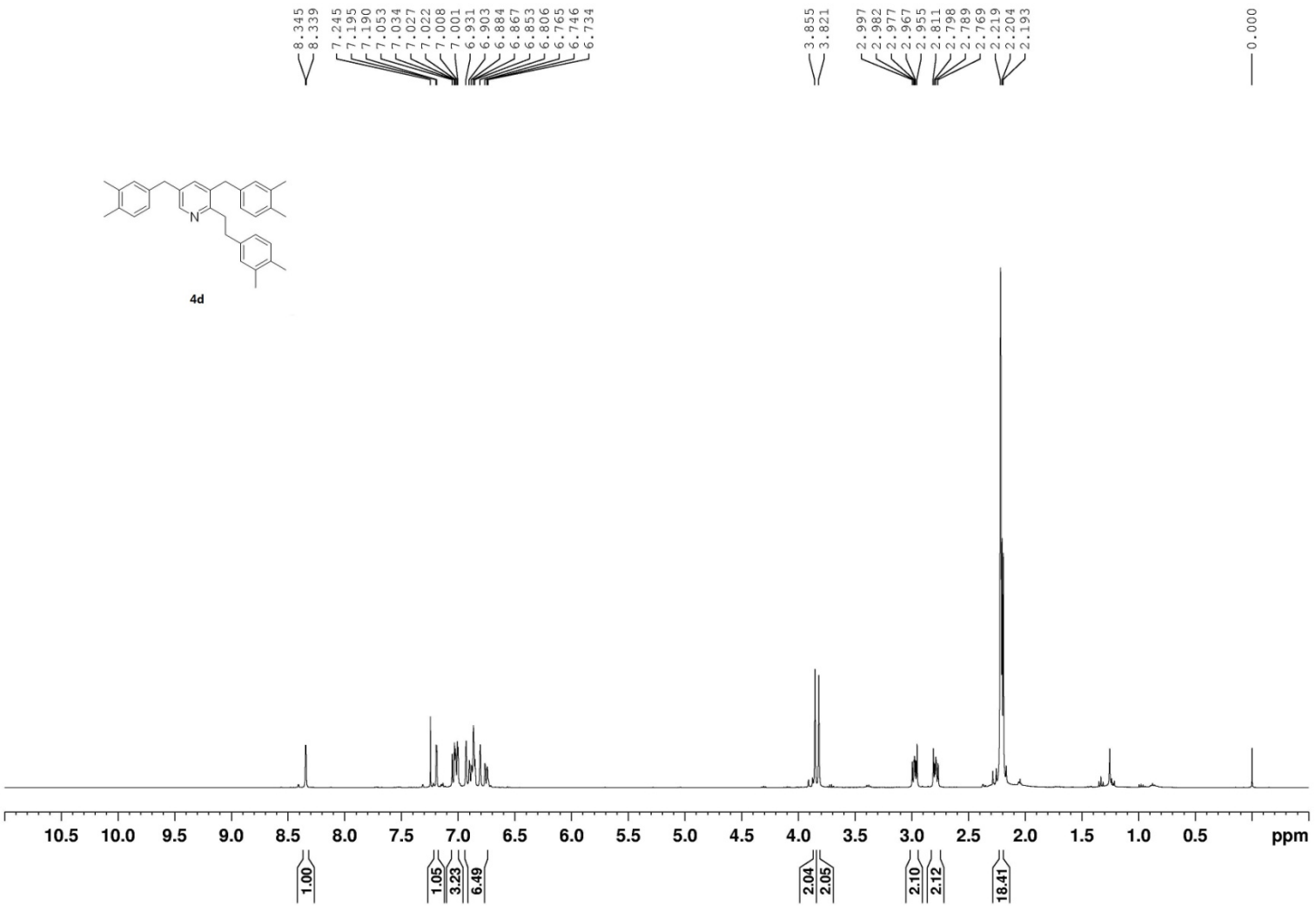
30
20
10
0
ppm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7.008
7.001
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6.903
6.884
6.867
6.853
6.806
6.765
6.746
6.734
3.855
3.821
2.997
2.982
2.977
2.967
2.955
2.811
2.798
2.789
2.769
2.219
2.204
2.193
0.000
N
4d
10.5 10.0 9.5 9.0 8.5 8.0 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 ppm
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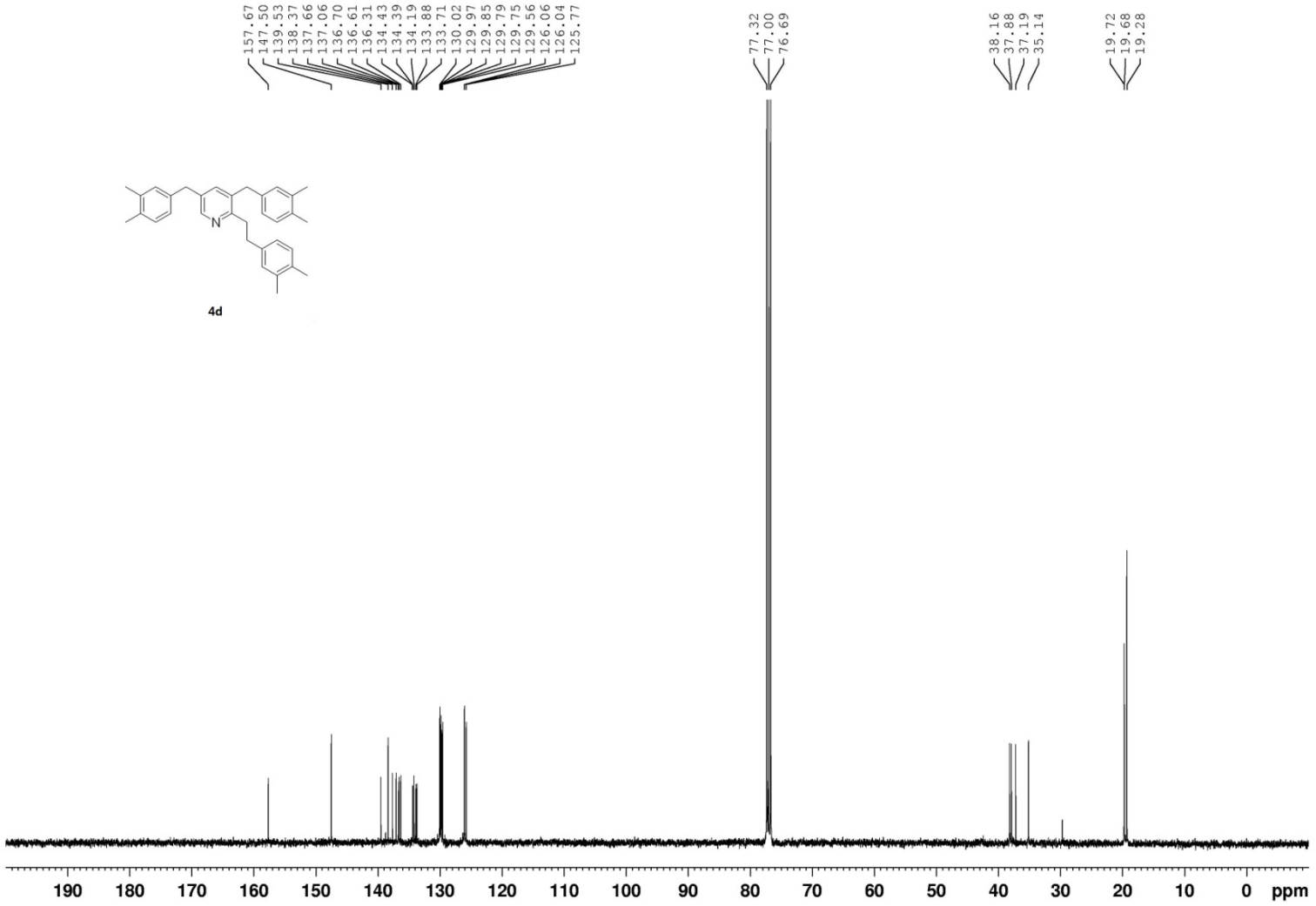
18.41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134.39
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133.71
130.02
129.97
129.85
129.79
129.75
129.56
126.06
126.04
125.77
77.32
77.00
76.69
38.16
37.88
37.19
35.14
19.72
19.68
19.28
N
4d
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180
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160
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140
130
120
110
100
90
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70
60
50
40
30
20
10
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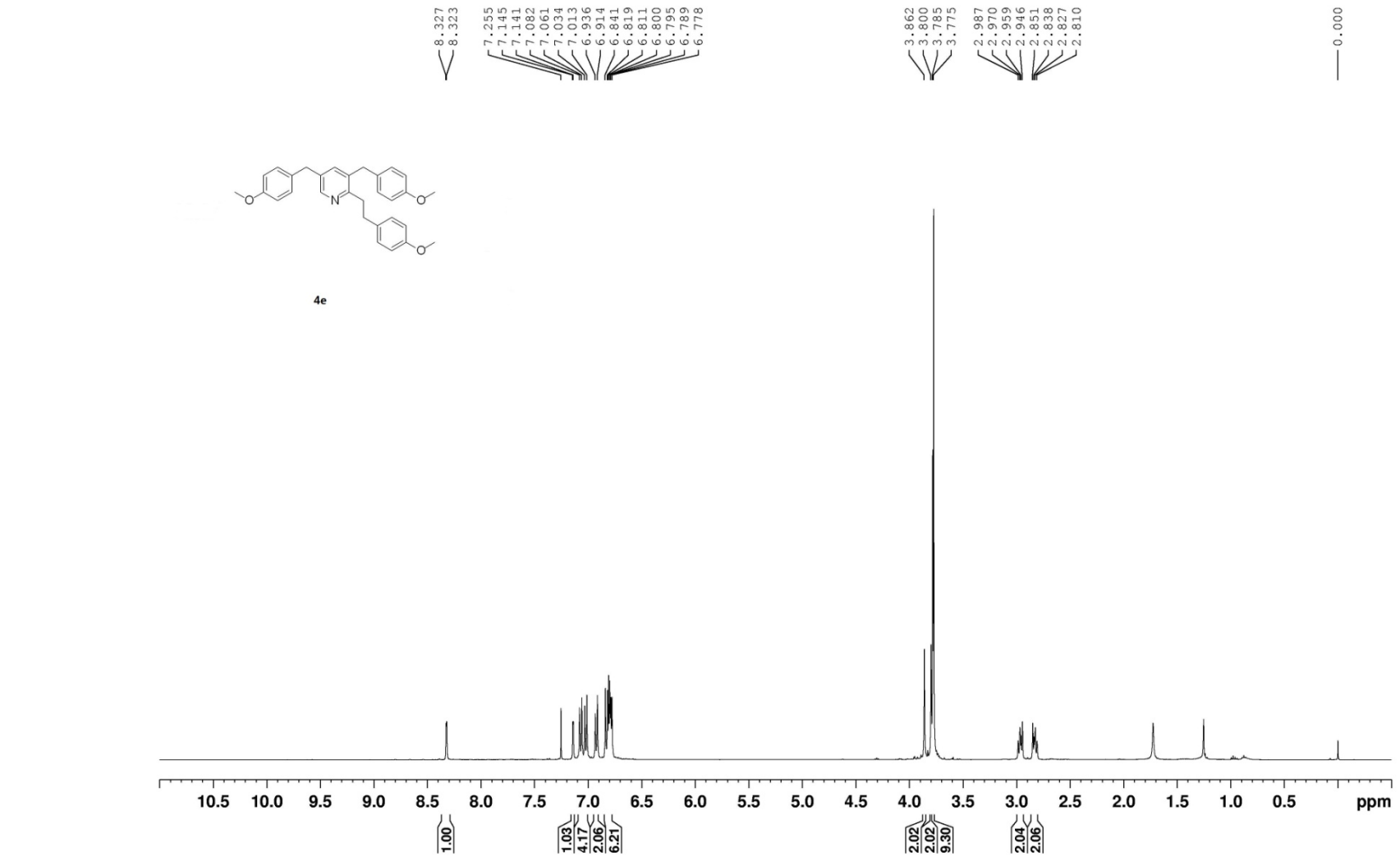
ppm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6.819
6.811
6.800
6.795
6.789
6.778
3.862
3.800
3.785
3.775
2.987
2.970
2.959
2.946
2.851
2.838
2.827
2.810
0.000
4e
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10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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2.0
1.5
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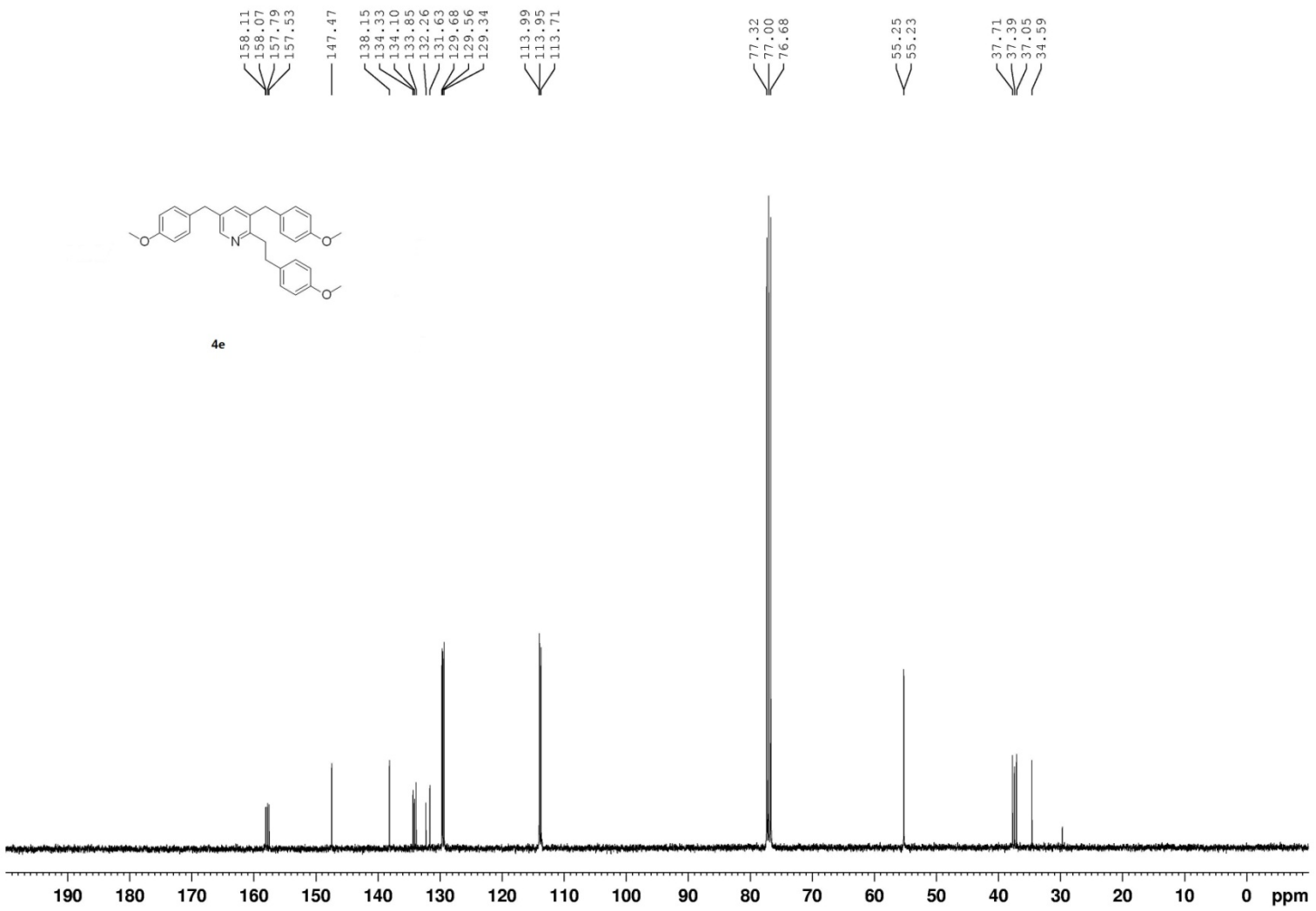
2.06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113.99
113.95
113.71
77.32
77.00
76.68
55.25
55.23
37.71
37.39
37.05
34.59
4e
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130
120
110
100
90
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60
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20
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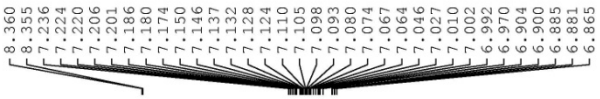
ppm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7.027
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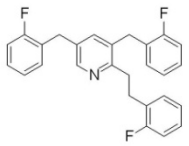
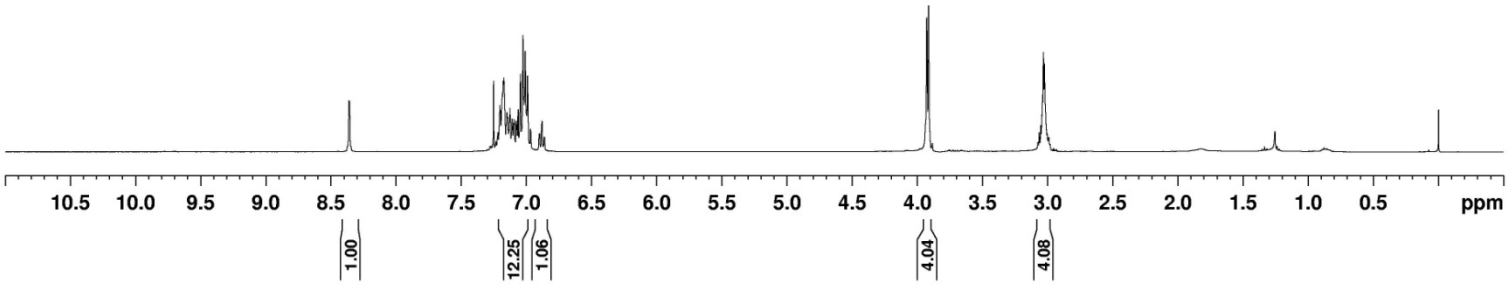

F
F
N
F

4f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6.0
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4.5
4.0
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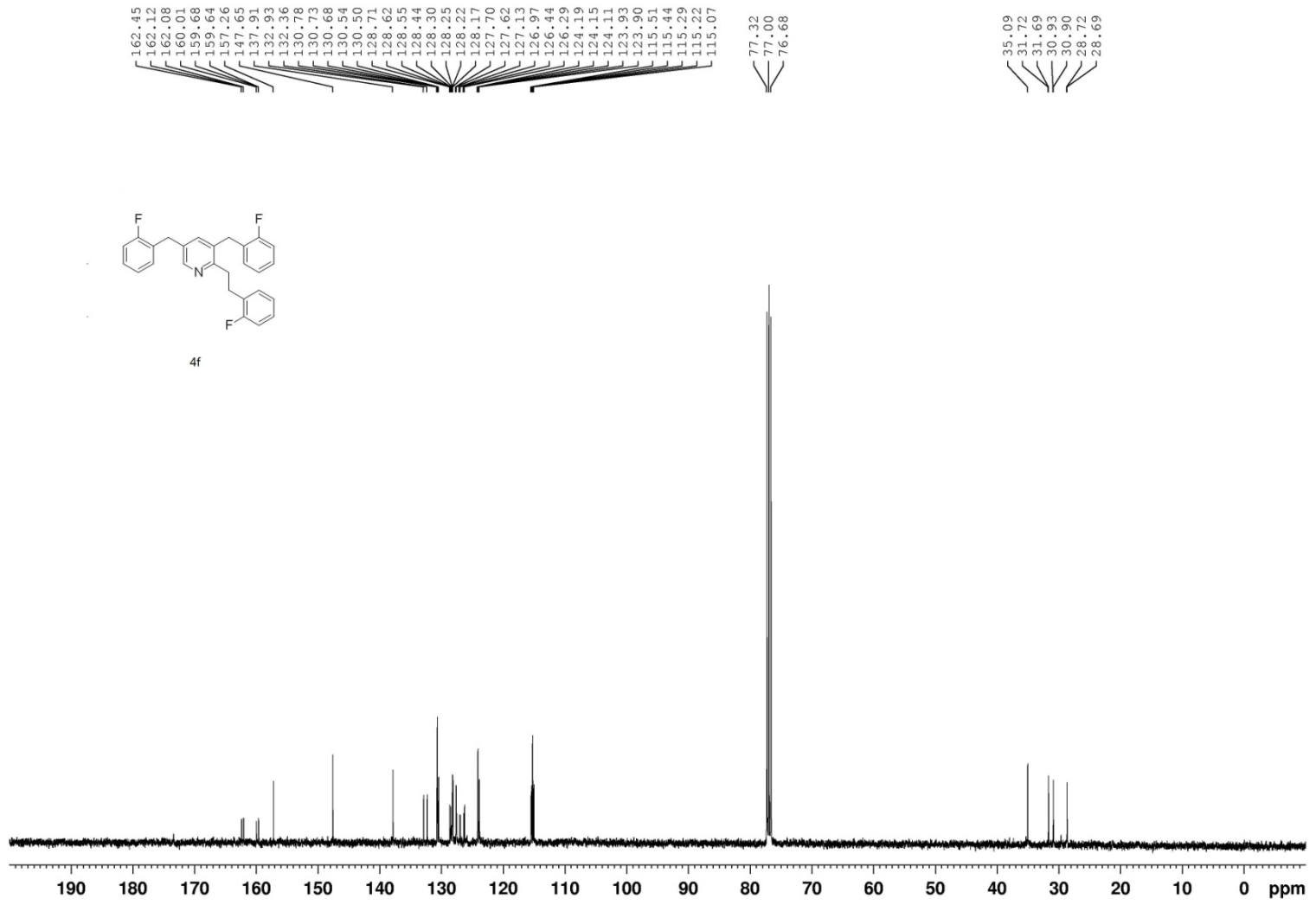
4.04
4.08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28.69
F
F
N
F
4f
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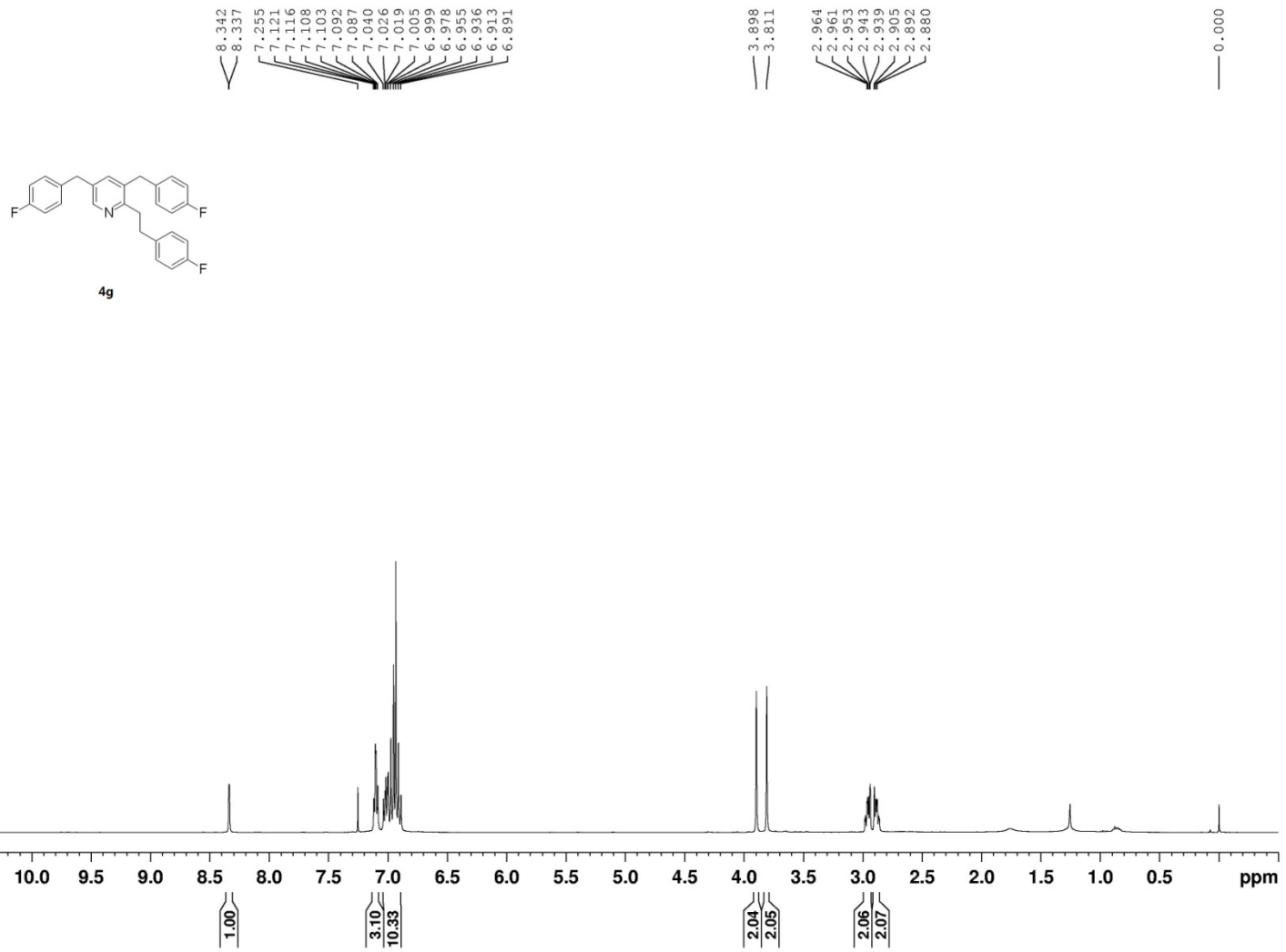
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7.026
7.019
7.005
6.999
6.978
6.955
6.936
6.913
6.891
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3.811
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2.961
2.953
2.943
2.939
2.905
2.892
2.880
0.000
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N
F
F
4g
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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
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ppm
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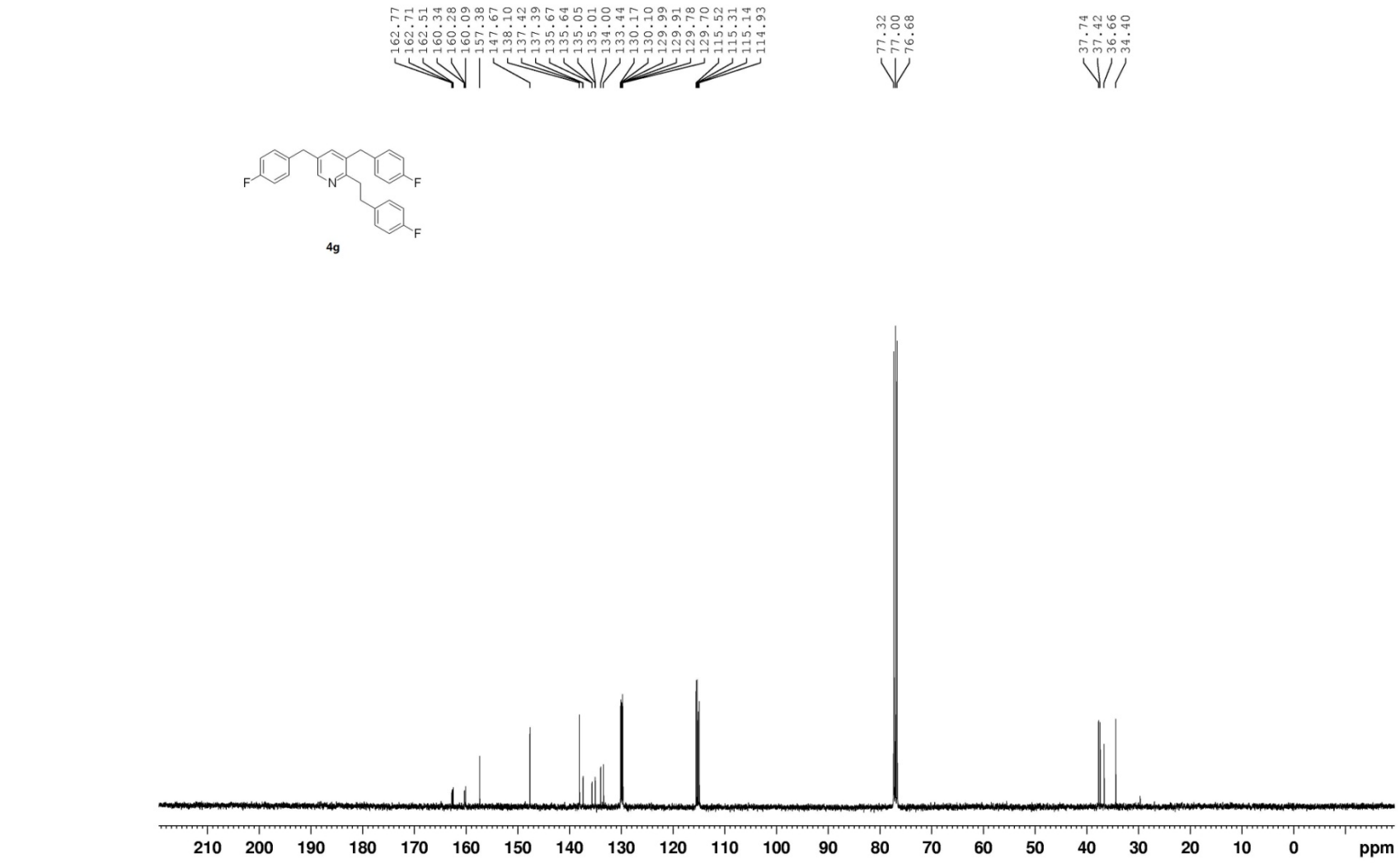
2.07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134.00
133.44
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130.10
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115.14
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77.32
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F
4g
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120
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100
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80
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40
30
20
10
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ppm