Supplementary Material (ESI) for Chemical Communications

Supporting Information for

N-Heterocyclic Carbene-Catalyzed Tandem Aza-Benzoin/Michael Reactions: *On Site* Reversal of the Reactivity of *N*-Boc Imines

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General Method: All manipulations were carried out under the argon atmosphere using standard Schlenk techniques. All glassware was oven or flame dried immediately prior to use. All solvents were purified and dried according to standard methods prior to use, unless stated otherwise.

All reagents were obtained from commercial sources and used without further purification. ^{1}H NMR spectra were obtained at 300 MHz or 400 MHz and recorded relative to tetramethylsilane signal (0 ppm) or DMSO-d_6 (2.50 ppm). ^{13}C NMR spectra were obtained at 75 MHz or 100 MHz and chemical shifts were recorded relative to the solvent resonance ($CDCl_3$, 77.0 ppm, DMSO-d_6, 39.6 ppm). Data for ^{1}H NMR are recorded as follows: chemical shift (B, ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quadruplet, m = multiplet or unresolved, br = broad singlet, coupling constant(s) in Hz, integration). Data for ^{13}C NMR are reported in terms of chemical shift (B, ppm).

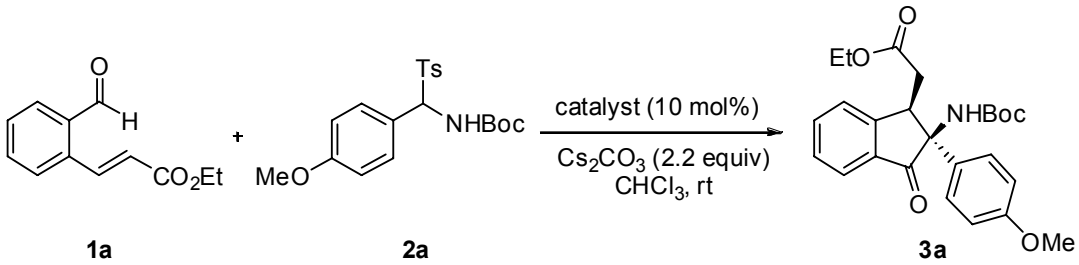
Arylacrylates **1a-1e**[1] and aryl(tosyl)methylcarbamates **2a-2j**[2] were prepared according to the literature.

References:

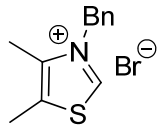
[1] D. Enders, A. A. Narine, F. Toulgoat, T. Bisschops. *Angew. Chem., Int. Ed.* **2008**, *47,* 5661.

[2] A. G. Wenzel, E. N. Jacobsen. *J. Am. Chem. Soc.* **2002**, *124,* 12964.

Table 1. Screening the catalysts[a]



1a 2a 3a



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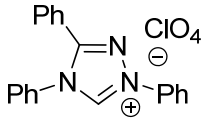

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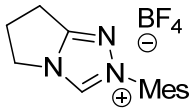

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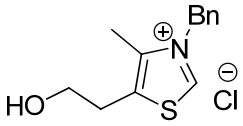

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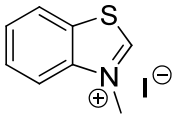

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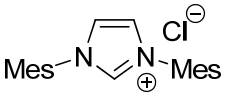
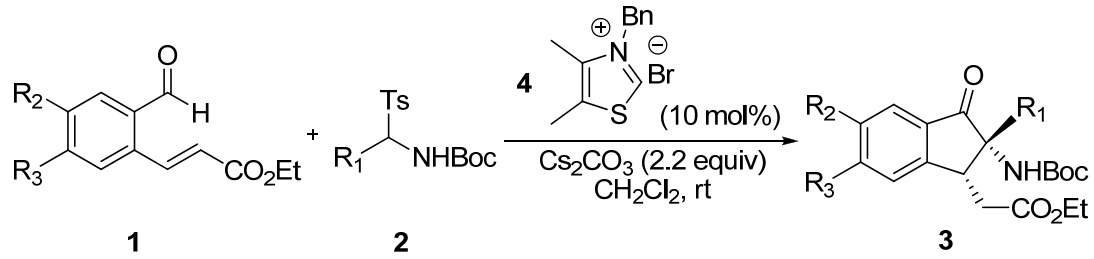


10

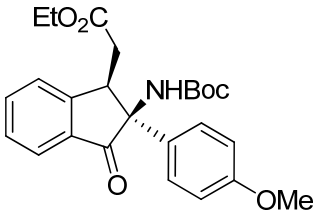
entry	catalyst	time (h)	yield (%)[b]	dr
1	**4**	5	68	> 20:1
2	**6**	5	N.R.	/
3	**7**	5	N.R.	/
4	**8**	5	43	> 20:1
5	**9**	5	trace	/
6	**10**	5	N.R.	/

[a] Reaction conditions: **1a**/**2a**/Cs_2CO_3 = 2.0/1.0/2.2, 0.1 M of **2a** in $CHCl_3$ at 25 °C. [b] Isolated yields.

General Procedure for the Synthesis of 3:

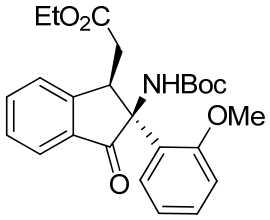


To a 25 mL Schlenk flask was added *tert*-butyl aryl(tosyl)methylcarbamate **2** (0.50 mmol), 3-benzyl-4,5-dimethylthiazol-3-ium bromide **4** (14.2 mg, 0.05 mmol), Cs_2CO_3 (358.4 mg, 1.10 mmol), CH_2Cl_2 (5.0 mL) and (*E*)-ethyl 3-(2-formylphenyl) acrylate **1** (1.0 mmol). The reaction mixture was stirred at room temperature for 5 h and then was filtered through a pad of celite, washed with EtOAc. The filtrate was evaporated and the residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate: 8/1) to afford product **3**.



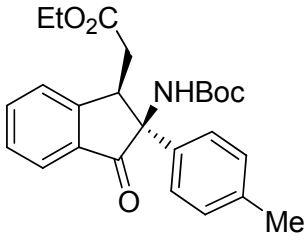
3aa

3aa was obtained as a pale yellow solid (162.9 mg, 74% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.74 (t, J = 7.2 Hz, 1H), 7.68-7.61 (m, 2H), 7.47 (t, J = 7.5 Hz, 1H), 7.35 (s, 1H), 7.21 (d, J = 9.0 Hz, 2H), 6.83 (d, J = 8.7 Hz, 2H), 4.35 (t, J = 6.9 Hz, 1H), 4.10 (q, J = 6.9 Hz, 2H), 3.68 (s, 3H), 2.85 (dd, J = 7.5, 16.2 Hz, 1H), 2.69 (dd, J = 6.9, 15.9 Hz, 1H), 1.36, 1.07 (s, 9H), 1.18 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 199.9, 172.0, 158.6, 155.5, 153.8, 135.7, 133.5, 131.5, 128.7, 127.4, 126.0, 124.5, 113.8, 78.7, 68.5, 60.3, 55.2, 47.5, 36.6, 28.2, 14.2; IR (film): v_{max} (cm^{-1}) = 3375, 2977, 2928, 1720, 1608, 1511, 1468, 1465, 1368, 1254, 1165, 1032; EI-MS (m/z): 323 (78), 322 (74), 265 (38), 264 (37), 252 (43), 251 (100), 134 (26), 57 (54); HRMS (EI): Exact mass calcd. for $C_{25}H_{29}NO_6$ $[M]^+$: 439.1995. Found: 439.1992.



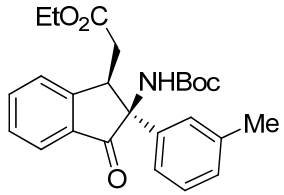
3ab

3ab was obtained as a pale yellow solid (182.6 mg, 83% yield). ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.76 (d, J = 7.6 Hz, 1H), 7.67 (t, J = 7.6 Hz, 1H), 7.47 (t, J = 7.6 Hz, 1H), 7.38 (d, J = 7.6 Hz, 1H), 7.29-7.25 (m, 2H), 7.07-7.02 (m, 2H), 6.83 (t, J = 7.6 Hz, 1H), 4.18 (dq, J = 1.6, 7.2 Hz, 2H), 3.96 (dd, J = 3.6, 9.6 Hz, 1H), 3.66 (s, 3H), 3.12 (dd, J = 3.6, 16.0 Hz, 1H), 2.62 (dd, J = 9.6, 16.4 Hz, 1H), 1.30 (s, 9H), 1.22 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 202.4, 172.9, 157.6, 155.2, 153.7, 135.4, 135.2, 129.3, 129.2, 128.0, 126.7, 125.3, 123.2, 119.9, 112.5, 78.8, 67.5, 60.4, 55.4, 48.3, 35.3, 28.6, 14.2; IR (film): ν_{max} (cm^{-1}) = 3452, 2977, 2929, 2854, 1728, 1605, 1489, 1464, 1369, 1283, 1250, 1167, 1028, 913, 755, 733; ESI-MS (m/z): 446 (M+Na^{+}, 100); HRMS (ESI): Exact mass calcd. for $C_{25}H_{29}NO_6Na$ [M+Na]$^{+}$: 462.1887. Found: 462.1884.



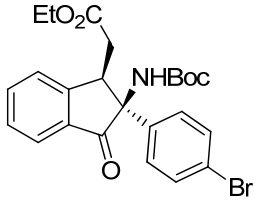
3ac

3ac was obtained as a white solid (180.5 mg, 85% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.77-7.61 (m, 3H), 7.47 (t, J = 7.2 Hz, 1H), 7.31 (s, 1H), 7.18 (d, J = 7.8 Hz, 2H), 7.07 (d, J = 8.1 Hz, 2H), 4.36 (m, 1H), 4.10 (q, J = 6.9 Hz, 2H), 2.87 (dd, J = 7.2, 16.5 Hz, 1H), 2.68 (dd, J = 6.6, 15.9 Hz, 1H), 2.21 (s, 3H), 1.36, 1.06 (s, 9H), 1.17 (t, J = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 199.8, 171.9, 155.4, 153.9, 136.8, 136.7, 135.7, 133.5, 129.0, 128.7, 126.0, 125.9, 124.4, 78.8, 68.8, 60.2, 47.5, 36.6, 28.1, 20.5, 14.1; IR (film): ν_{max} (cm^{-1}) = 3406, 2978, 2928, 1721, 1607, 1485, 1368, 1276, 1246, 1165, 1072, 1029, 891; ESI-MS (m/z): 446 (M+Na^{+}, 100); HRMS (ESI): Exact mass calcd. for $C_{25}H_{29}NO_6Na$ [M+Na]$^{+}$: 446.1938. Found: 446.1934.



3ad

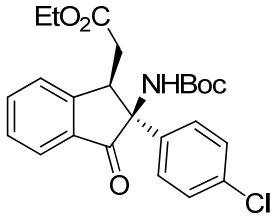
3ad was obtained as a white solid (197.2 mg, 93% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.78-7.63 (m, 3H), 7.48 (t, J = 7.2 Hz, 1H), 7.33 (s, 1H), 7.17-7.11 (m, 2H), 7.04 (m, 2H), 4.37 (t, J = 6.9 Hz, 1H), 4.11 (q, J = 6.9 Hz, 2H), 2.87 (dd, J = 6.9, 15.9 Hz, 1H), 2.71 (dd, J = 6.0, 16.2 Hz, 1H), 2.23 (s, 3H), 1.37, 1.05 (s, 9H), 1.18 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 199.8, 172.0, 155.5, 154.0, 139.8, 137.5, 135.8, 133.6, 128.7, 128.3, 128.1, 126.6, 126.0, 124.5, 123.0, 78.8, 68.9, 60.2, 47.8, 36.5, 28.1, 21.3, 14.2; IR (film): v_{max} (cm^{-1}) = 3388, 2978, 2929, 1721, 1606, 1485, 1392, 1368, 1280, 1248, 1166, 1056, 1029, 909, 891, 732, 701; ESI-MS (m/z): 446 (M+Na^{+}, 100); HRMS (ESI): Exact mass calcd. for $C_{25}H_{29}NO_5Na$ [M+Na]$^{+}$: 446.1938. Found: 446.1943.



3ae

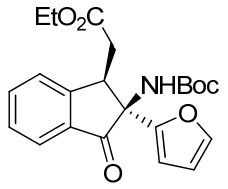
3ae was obtained as a white solid (200.5 mg, 82% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.77 (t, J = 7.2 Hz, 1H), 7.69 (d, J = 7.5 Hz, 1H), 7.64 (d, J = 7.5 Hz, 1H), 7.55 (s, 1H), 7.51-7.47 (m, 3H), 7.25 (d, J = 9.0 Hz, 2H), 4.36 (t, J = 7.2 Hz, 1H), 4.10 (q, J = 6.9 Hz, 2H), 2.83 (dd, J = 6.9, 16.2 Hz, 1H), 2.71 (dd, J = 6.3, 16.2 Hz, 1H), 1.36, 1.06 (s, 9H), 1.17 (t, J = 7.2 Hz, 3H); ^{1}H NMR at 80 ^{o}C (400 MHz, DMSO-d_6) δ 7.75 (t, J = 6.8 Hz, 1H), 7.69 (d, J = 7.6 Hz, 1H), 7.64 (d, J = 7.6 Hz, 1H), 7.50-7.44 (m, 3H), 7.27 (d, J = 8.8 Hz, 2H), 6.92 (br s, 1H), 4.41 (t, J = 6.8 Hz, 1H), 4.10 (q, J = 7.2 Hz, 2H), 2.90 (dd, J = 6.0, 16.4 Hz, 1H), 2.62 (dd, J = 6.8, 16.0 Hz, 1H), 1.32 (s, 9H), 1.17 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 199.4, 171.9, 155.5, 153.7, 139.2, 135.9, 133.2, 131.3, 128.9, 128.4, 126.1, 124.5, 120.9, 79.0, 68.7, 60.3, 47.3, 36.5, 28.1, 14.2; IR (film): v_{max} (cm^{-1}) = 3400, 2978, 2930, 1720, 1607, 1487, 1393, 1368, 1246, 1163, 1077, 1029, 1009, 911, 890, 779, 735; EI-MS (m/z): 433 (10), 388 (30), 372 (100), 344 (14), 314 (27), 299 (67), 271

(13), 233 (67), 218 (29), 103 (17), 57 (96); HRMS (EI): Exact mass calcd. for $C_{24}H_{26}NO_5Br$ $[M]^+$: 487.0994. Found: 487.1000.



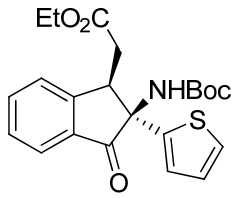
3af

3af was obtained as a white solid (178.8 mg, 81% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.79-7.63 (m, 3H), 7.53-7.46 (m, 2H), 7.34 (m, 4H), 4.38 (t, J = 6.0 Hz, 1H), 4.10 (q, J = 6.6 Hz, 2H), 2.85 (dd, J = 7.5, 16.8 Hz, 1H), 2.72 (dd, J = 6.0, 15.9 Hz, 1H), 1.36, 1.07 (s, 9H), 1.17 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 199.5, 171.9, 155.6, 153.8, 138.8, 136.0, 133.3, 132.3, 128.9, 128.4, 128.1, 126.2, 124.6, 79.0, 68.7, 60.4, 47.4, 36.6, 28.1, 14.2; IR (film): v_{max} (cm^{-1}) = 3160, 3060, 2925, 2840, 1709, 1687, 1604, 1494, 1464, 1286, 1259, 1093, 1014, 815, 727, 675; EI-MS (m/z): 268 (13), 255 (100), 234 (18), 191 (18), 102 (14), 77 (12); HRMS (EI): Exact mass calcd. for $C_{24}H_{26}NO_5Cl$ $[M]^+$: 443.1500. Found: 443.1504.



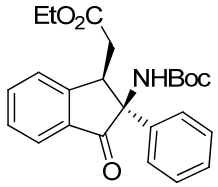
3ag

3ag was obtained as a pale-yellow solid (124.8 mg, 62% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.75-7.64 (m, 3H), 7.53-7.45 (m, 3H), 6.53 (d, J = 3.0 Hz, 1H), 6.37 (dd, J = 1.8, 3.3 Hz, 1H), 4.37 (t, J = 7.2 Hz, 1H), 4.11 (dd, J = 2.7, 7.2 Hz, 2H), 2.82-2.77 (m, 2H), 1.35, 1.09 (s, 9H), 1.18 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 198.1, 171.8, 155.5, 153.7, 151.7, 143.0, 135.7, 133.4, 128.5, 126.0, 124.2, 110.8, 107.5, 79.1, 65.5, 60.3, 45.7, 35.6, 28.1, 14.2; IR (film): v_{max} (cm^{-1}) = 3365, 3133, 3114, 2981, 2934, 1734, 1716, 1601, 1493, 1464, 1392, 1368, 1288, 1249, 1163, 1016, 959, 892, 755, 626; EI-MS (m/z): 343 (13), 299 (15), 283 (15), 282 (20), 256 (20), 254 (13), 212 (100), 211 (87), 196 (17), 183 (11), 57 (83), 41 (30); HRMS (EI): Exact mass calcd. for $C_{22}H_{25}NO_6$ $[M]^+$: 399.1682. Found: 399.1687.



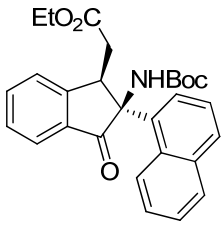
3ah

3ah was obtained as a pale-yellow solid (164.6 mg, 79% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.74 (t, J = 7.5 Hz, 1H), 7.67 (d, J = 8.7 Hz, 2H), 7.50-7.46 (m, 2H), 7.40 (d, J = 4.5 Hz, 1H), 7.05 (d, J = 2.7 Hz, 1H), 6.90 (t, J = 4.5 Hz, 1H), 4.49 (t, J = 6.9 Hz, 1H), 4.12 (m, 2H), 2.84-2.78 (m, 2H), 1.36, 1.12 (s, 9H), 1.19 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 198.4, 171.8, 155.3, 153.3, 143.6, 135.8, 132.6, 128.7, 126.9, 126.3, 126.1, 125.9, 124.6, 79.0, 66.8, 60.3, 48.6, 36.4, 28.1, 14.2; IR (film): ν_{max} (cm^{-1}) = 3388, 3338, 2975, 2926, 2847, 1720, 1602, 1485, 1360, 1286, 1247, 1164, 1026, 884, 699; EI-MS (m/z): 415 (1), 314 (35), 298 (32), 240 (56), 228 (87), 227 (100), 225 (21), 199 (20), 57 (63); HRMS (EI): Exact mass calcd. for $C_{22}H_{25}NO_5S$ $[M]^+$: 415.1453. Found: 415.1448.



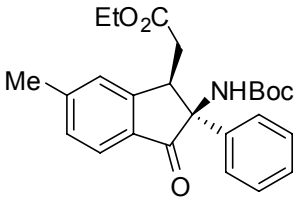
3ai

3ai was obtained as a white solid (158.0 mg, 77% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.78-7.62 (m, 3H), 7.48 (t, J = 7.2 Hz, 1H), 7.39 (s, 1H), 7.29-7.24 (m, 5H), 4.38 (t, J = 6.6 Hz, 1H), 4.10 (q, J = 6.9 Hz, 2H), 2.87 (dd, J = 6.6, 15.9 Hz, 1H), 2.70 (dd, J = 5.7, 15.9 Hz, 1H), 1.37, 1.05 (s, 9H), 1.18 (t, J = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 199.8, 172.0, 155.5, 154.0, 139.8, 135.8, 133.5, 128.8, 128.5, 127.5, 126.0, 125.9, 124.5, 78.9, 69.0, 60.3, 47.7, 36.6, 28.1, 14.2; IR (film): ν_{max} (cm^{-1}) = 3367, 2984, 1724, 1701, 1609, 1493, 1364, 1331, 1248, 1160, 1072, 1055, 1035, 888, 754, 698, 649; EI-MS (m/z): 353 (9), 308 (28), 292 (100), 264 (16), 234 (67), 221 (56), 193 (16), 104 (22), 77 (14), 57 (88), 41 (28); HRMS (EI): Exact mass calcd. for $C_{24}H_{27}NO_5$ $[M]^+$: 409.1889. Found: 409.1888.



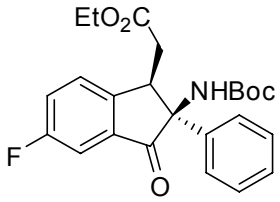
3aj

3aj was obtained as a white solid (206.4 mg, 90% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.25 (br s, 1H), 7.94 (d, J = 8.1 Hz, 2H), 7.82 (d, J = 7.8 Hz, 2H), 7.71-7.48 (m, 4H), 7.37 (br s, 1H), 7.28 (t, J = 7.8 Hz, 1H), 7.15 (m, 1H), 4.35 (m, 1H), 4.17-4.11 (m, 2H), 3.39 (m, 1H), 2.75 (m, 1H), 1.33 (s, 9H), 1.20 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 202.3, 172.5, 155.8, 155.7, 153.8, 136.8, 136.0, 134.6, 133.8, 131.2, 129.2, 128.7, 126.2, 125.9, 125.8, 125.0, 124.6, 124.4, 124.0, 78.8, 70.5, 60.5, 48.4, 36.6, 28.1, 14.2; IR (film): ν_{max} (cm^{-1}) = 3363, 2979, 1731, 1702, 1604, 1503, 1464, 1392, 1368, 1285, 1252, 1167, 1096, 1027, 945, 862, 784, 776, 707, 673; EI-MS (m/z): 459 (2), 403 (74), 342 (70), 284 (57), 271 (92), 259 (18), 243 (24), 127 (25), 57 (100), 41 (43); HRMS (EI): Exact mass calcd. for $C_{28}H_{29}NO_5$ $[M]^+$: 459.2046. Found: 459.2048.



3bi

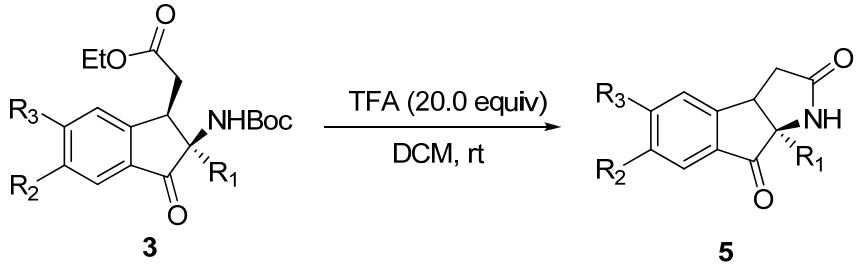
3bi was obtained as a pale yellow solid (111.3 mg, 52% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.52 (d, J = 7.5 Hz, 1H), 7.48 (s, 1H), 7.32 (d, J = 6.0 Hz, 2H), 7.28-7.20 (m, 5H), 4.32 (t, J = 6.9 Hz, 1H), 4.10 (q, J = 7.2 Hz, 2H), 2.84 (dd, J = 6.9, 16.5 Hz, 1H), 2.65 (dd, J = 7.2, 16.5 Hz, 1H), 2.43 (s, 3H), 1.36, 1.04 (s, 9H), 1.17 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 199.1, 172.0, 155.4, 154.4, 146.6, 140.0, 131.1, 129.9, 128.4, 127.4, 126.2, 125.9, 124.4, 78.8, 69.2, 60.3, 47.5, 36.6, 28.1, 21.9, 14.2; IR (film): ν_{max} (cm^{-1}) = 3352, 2983, 1720, 1609, 1493, 1446, 1367, 1326, 1278, 1239, 1164, 1109, 1053, 1021, 955, 896, 874, 787, 729, 702, 642, 614, 594, 492, 441; EI-MS (m/z): 367 (7), 322 (46), 306 (95), 278 (18), 248 (80), 235 (58), 104 (15), 57 (100), 41 (23); HRMS (EI): Exact mass calcd. for $C_{21}H_{20}NO_4$ $[M\text{-}OC_4H_9]^+$: 350.1392. Found: 350.1393.



3ci

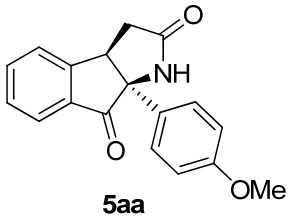
3ci was obtained as a pale yellow solid (190.2 mg, 89% yield). ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.76 (dd, J = 4.8, 8.4 Hz, 1H), 7.63 (dt, J = 2.1, 9.0 Hz, 1H), 7.49 (s, 1H), 7.43 (dd, J = 2.1, 7.5 Hz, 1H), 7.33-7.23 (m, 5H), 4.37 (t, J = 6.6 Hz, 1H), 4.10 (q, J = 6.9 Hz, 2H), 2.87 (dd, J = 7.8, 16.5 Hz, 1H), 2.76 (dd, J = 6.3, 16.8 Hz, 1H), 1.37, 1.05 (s, 9H), 1.18 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 199.2, 172.0, 162.4 (d, J = 245.6 Hz), 155.7, 149.8, 139.6, 135.6 (d, J = 7.5 Hz), 128.6, 128.3 (d, J = 7.5 Hz), 127.7, 126.1, 123.3 (d, J = 23.4 Hz), 110.3 (d, J = 21.8 Hz), 79.1, 69.6, 60.4, 47.4, 36.4, 28.1, 14.2; ^{19}F NMR (282 MHz, DMSO-d_6) δ -113.5 (m); IR (film): ν_{max} (cm^{-1}) = 3408, 3062, 2979, 2933, 1732, 1613, 1484, 1446, 1392, 1368, 1268, 1166, 1057, 1031, 840, 763, 736, 701, 540; EI-MS (m/z): 371 (7), 326 (7), 310 (100), 282 (11), 252 (29), 239 (38), 237 (14), 211 (15), 104 (14), 57 (79), 41 (20); HRMS (EI): Exact mass calcd. for $C_{24}H_{26}NO_5F$ $[M]^+$: 427.1795. Found: 427.1798.

General Procedure for the Synthesis of 5:



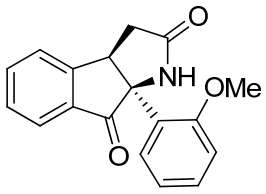
3 **5**

To a flask containing **3** (0.24 mmol) in CH_2Cl_2 (8.0 mL) was added TFA (0.36 mL, 4.8 mmol) in one portion. The mixture was stirred at room temperature for 24 h (monitored by TLC until the full conversion of substrate **3**) and then quenched with sodium carbonate saturated aqueous solution. The reaction mixture was extracted with CH_2Cl_2 (10 mLX3) and the combined organic layers were dried over $MgSO_4$, filtered and evaporated under reduced pressure. The residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate: 2/1) to afford product **5**.



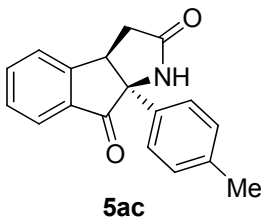
5aa

5aa was isolated as white solid (57.7 mg, 82% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.8 Hz, 1H), 7.76 (t, *J* = 7.2 Hz, 1H), 7.58 (br s, 1H, NH), 7.52 (t, *J* = 6.6 Hz, 2H), 7.15 (d, *J* = 8.7 Hz, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 3.94 (d, *J* = 9.0 Hz, 1H), 3.78 (s, 3H), 3.12 (dd, *J* = 10.8, 18.0 Hz, 1H), 2.51 (dd, *J* = 2.4, 17.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.8, 175.5, 159.4, 154.7, 136.7, 134.0, 131.2, 129.0, 126.5, 126.1, 125.4, 114.3, 71.6, 55.3, 48.2, 36.3; IR (film): ν_{max} (cm^{-1}) = 3210, 2925, 2854, 1697, 1606, 1514, 1463, 1249, 1181, 1033, 774; EI-MS (m/z): 293 (16), 265 (47), 251 (100), 234 (29), 222 (16); HRMS (EI): Exact mass calcd. for $C_{18}H_{15}NO_3$ $[M]^+$: 293.1052. Found: 293.1051.



5ab

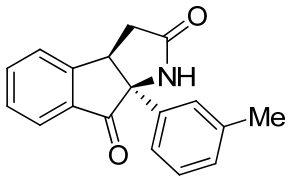
5ab was isolated as white solid (54.2 mg, 77% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.2 Hz, 1H), 7.73 (t, *J* = 7.5 Hz, 1H), 7.53-7.44 (m, 3H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.04 (t, *J* = 7.8 Hz, 1H), 6.86 (d, *J* = 8.1 Hz, 1H), 6.45 (br s, 1H, NH), 3.97 (d, *J* = 9.9 Hz, 1H), 3.48 (s, 3H), 2.98 (dd, *J* = 10.8, 17.7 Hz, 1H), 2.54 (d, *J* = 17.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.0, 175.5, 155.8, 153.4, 135.8, 134.0, 129.8, 128.6, 127.8, 126.5, 125.6, 124.8, 121.1, 111.2, 70.6, 55.2, 46.3, 35.5; IR (film): ν_{max} (cm^{-1}) = 3174, 2923, 2853, 1721, 1695, 1604, 1460, 1342, 1154, 1093, 1024, 746; EI-MS (m/z): 293 (9), 264 (37), 251 (100), 234 (22), 221 (15); HRMS (EI): Exact mass calcd. for $C_{18}H_{15}NO_3$ $[M]^+$: 293.1052. Found: 293.1055.



5ac

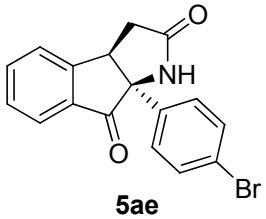
5ac was isolated as white solid (57.2 mg, 86% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.8 Hz, 1H), 7.76 (t, *J* = 6.9 Hz, 1H), 7.53 (t, *J* = 6.6 Hz, 2H), 7.42 (br s, 1H, NH),

7.17-7.10 (m, 4H), 3.96 (dd, *J* = 2.1, 10.2 Hz, 1H), 3.12 (dd, *J* = 10.8, 17.7 Hz, 1H), 2.51 (dd, *J* = 2.7, 17.7 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.7, 175.6, 154.8, 138.0, 136.7, 136.3, 134.1, 129.6, 129.0, 126.0, 125.4, 125.1, 71.8, 48.3, 36.2, 21.0; IR (film): v_{max} (cm^{-1}) = 3181, 2924, 2840, 1697, 1604, 1509, 1464, 1339, 1283, 913, 804, 774, 731, 676; EI-MS (m/z): 277 (9), 248 (16), 235 (100), 220 (11), 191 (15); HRMS (EI): Exact mass calcd. for $C_{18}H_{15}NO_2$ $[M]^+$: 277.1103. Found: 277.1097.



5ad

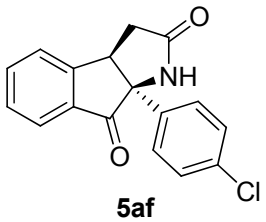
5ad was isolated as white solid (52.6 mg, 79% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.1 Hz, 1H), 7.79 (t, *J* = 7.8 Hz, 1H), 7.55 (t, *J* = 9.0 Hz, 2H), 7.29-7.24 (m, 1H), 7.15 (d, *J* = 7.2 Hz, 1H), 7.04-7.02 (m, 2H), 6.56-6.46 (m, 1H), 3.98 (d, *J* = 10.2 Hz, 1H), 3.14 (dd, *J* = 10.5, 17.4 Hz, 1H), 2.52 (dd, *J* = 2.4, 17.4 Hz, 1H), 2.34 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.7, 175.5, 154.9, 139.2, 138.8, 136.8, 134.1, 129.2, 129.1, 128.9, 126.1, 125.7, 125.5, 122.2, 71.8, 48.4, 36.2, 21.4; IR (film): v_{max} (cm^{-1}) = 3212, 3071, 1717, 1685, 1603, 1463, 1354, 1282, 1238, 1187, 1099, 1022, 941, 790, 766, 687, 672; EI-MS (m/z): 277 (4), 248 (16), 235 (100), 220 (11), 206 (8), 191 (15), 178 (8), 165 (6), 118 (7), 103 (6), 91 (7), 77 (6), 65 (4); HRMS (EI): Exact mass calcd. for $C_{18}H_{15}NO_2$ (M^+): 277.1103. Found: 277.1101.



5ae

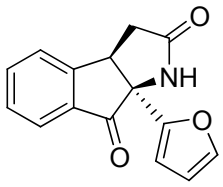
5ae was isolated as white solid (69.8 mg, 85% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.2 Hz, 1H), 7.78 (t, *J* = 7.5 Hz, 1H), 7.66 (br s, 1H, NH), 7.55 (t, *J* = 8.1 Hz, 2H), 7.47 (d, *J* = 7.8 Hz, 2H), 7.11 (d, *J* = 8.4 Hz, 2H), 3.94 (d, *J* = 9.9 Hz, 1H), 3.12 (dd, *J* = 10.8, 18.0 Hz, 1H), 2.52 (d, *J* = 17.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.0, 175.6, 154.6, 138.4, 137.0, 133.8, 132.0, 129.3, 127.0, 126.1, 125.6, 122.3, 71.7, 48.2, 36.2; IR (film): v_{max} (cm^{-1}) = 3163, 3058, 2887, 1720, 1683, 1604, 1364, 1286, 1076, 1010, 813,

707, 675, 504; EI-MS (m/z): 314 (9), 301 (100), 233 (22), 191 (13), 165 (14), 103 (16), 77 (11); HRMS (EI): Exact mass calcd. for $C_{17}H_{12}NO_2Br$ $[M]^+$: 341.0051. Found: 341.0059.



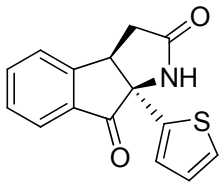
5af

5af was isolated as white solid (64.3 mg, 90% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.89-7.84 (m, 2H), 7.78 (t, *J* = 7.5 Hz, 1H), 7.54 (t, *J* = 7.5 Hz, 2H), 7.30 (d, *J* = 8.7 Hz, 2H), 7.16 (d, *J* = 8.7 Hz, 2H), 3.94 (dd, *J* = 2.1, 10.5 Hz, 1H), 3.12 (dd, *J* = 10.8, 17.7 Hz, 1H), 2.52 (dd, *J* = 3.0, 18.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.2, 175.6, 154.6, 137.9, 137.0, 134.1, 133.8, 129.3, 129.0, 126.7, 126.1, 125.5, 71.7, 48.2, 36.2; IR (film): ν_{max} (cm^{-1}) = 3166, 3059, 2886, 1720, 1683, 1602, 1499, 1460, 1406, 1365, 1286, 1202, 1092, 1014, 927, 875, 838, 817, 754, 727, 676, 511, 468; EI-MS (m/z): 268 (13), 255 (100), 240 (9), 234 (16), 191 (16), 102 (14), 71 (12), 57 (16), 43 (13); HRMS (EI): Exact mass calcd. for $C_{17}H_{12}NO_2Cl$ $[M]^+$: 297.0557. Found: 297.0559.



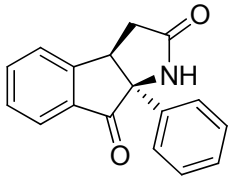
5ag

5ag was isolated as white solid (55.3 mg, 91% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.8 Hz, 1H), 7.76 (t, *J* = 7.5 Hz, 1H), 7.54-7.49 (m, 2H), 7.41 (t, *J* = 0.9 Hz, 1H), 7.16 (br s, 1H, NH), 6.48 (d, *J* = 3.3 Hz, 1H), 6.36-6.35 (m, 1H), 4.23 (dd, *J* = 2.4, 10.8 Hz, 1H), 3.20 (dd, *J* = 10.8, 17.7 Hz, 1H), 2.54 (dd, *J* = 2.7, 17.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 199.4, 175.1, 154.2, 150.5, 143.5, 136.9, 133.3, 129.1, 126.0, 125.5, 110.5, 108.6, 67.5, 44.4, 36.2; IR (film): ν_{max} (cm^{-1}) = 3129, 3062, 2831, 1720, 1687, 1651, 1602, 1463, 1418, 1348, 1293, 1255, 1224, 1193, 1167, 1152, 1124, 1004, 930, 847, 797, 760, 684, 671, 653, 597, 538, 475, 447; EI-MS (m/z): 224 (18), 211 (100), 196 (22), 168 (10), 77 (15); HRMS (EI): Exact mass calcd. for $C_{15}H_{11}NO_3$ $[M]^+$: 253.0739. Found: 253.0730.



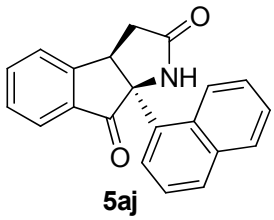
5ah

5ah was isolated as white solid (55.6 mg, 86% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.5 Hz, 1H), 7.79-7.74 (m, 1H), 7.52 (m, 2H), 7.47 (br s, 1H, NH), 7.29 (dd, *J* = 1.2, 10.8 Hz, 1H), 7.04 (dd, *J* = 0.9, 3.6 Hz, 1H), 6.98 (dd, *J* = 3.6, 4.8 Hz, 1H), 4.13 (dd, *J* = 2.7, 10.5 Hz, 1H), 3.23 (dd, *J* = 10.5, 17.7 Hz, 1H), 2.54 (dd, *J* = 3.0, 17.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.4, 174.8, 153.9, 142.3, 136.9, 133.1, 129.2, 127.3, 126.0, 125.7, 125.6, 125.3, 69.2, 48.2, 36.2; IR (film): v_{max} (cm^{-1}) = 3196, 3075, 2940, 2840, 1694, 1605, 1460, 1337, 1286, 1246, 831, 770, 727, 702; EI-MS (m/z): 269 (5), 240 (27), 227 (100), 212 (23), 197 (17), 110 (13), 77 (12); HRMS (EI): Exact mass calcd. for $C_{15}H_{11}NO_2S$ $[M]^+$: 269.0511. Found: 269.0515.



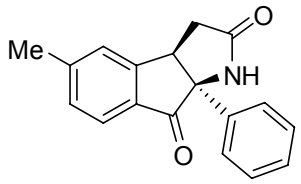
5ai

5ai was isolated as white solid (50.6 mg, 80% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 (d, *J* = 7.8 Hz, 1H), 7.79 (t, *J* = 7.5 Hz, 1H), 7.59-7.53 (m, 2H), 7.40-7.35 (m, 3H), 7.24 (m, 2H), 6.36 (br s, 1H, NH), 4.00 (dd, *J* = 2.1, 9.9 Hz, 1H), 3.15 (dd, *J* = 10.5, 17.7 Hz, 1H), 2.53 (dd, *J* = 2.4, 17.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.6, 175.6, 154.8, 139.3, 136.8, 134.0, 129.1, 128.9, 128.2, 126.1, 125.5, 125.1, 72.0, 48.3, 36.2; IR (film): v_{max} (cm^{-1}) = 3423, 3153, 3064, 2865, 1718, 1701, 1604, 1445, 1347, 1287, 1259, 1221, 1192, 1169, 1154, 969, 920, 821, 759, 704, 692, 678, 640, 500, 448; EI-MS (m/z): 263 (4), 234 (35), 221 (100), 206 (15), 191 (21), 165 (11), 104 (12), 77 (22), 51 (9); HRMS (EI): Exact mass calcd. for $C_{17}H_{13}NO_2$ $[M]^+$: 263.0946. Found: 263.0947.



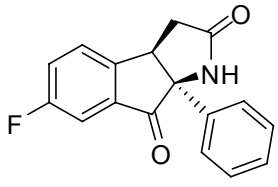
5aj

5aj was isolated as white solid (60.9 mg, 81% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (d, *J* = 7.8 Hz, 1H), 7.90-7.81 (m, 3H), 7.70 (d, *J* = 7.2 Hz, 1H), 7.64-7.58 (m, 2H), 7.47-7.39 (m, 2H), 7.25-7.20 (m, 2H), 7.01 (d, *J* = 8.7, 1H), 4.23 (d, *J* = 9.6 Hz, 1H), 2.97 (dd, *J* = 9.6, 17.7 Hz, 1H), 2.62 (d, *J* = 17.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.4, 175.4, 153.1, 136.7, 134.3, 134.1, 129.7, 129.6, 129.5, 126.8, 126.4, 125.8, 125.2, 125.0, 123.2, 72.7, 46.6, 35.7; IR (film): ν_{max} (cm^{-1}) = 3416, 3166, 3064, 1716, 1689, 1605, 1512, 1466, 1416, 1341, 1288, 1245, 1160, 796, 772, 687, 667, 640, 449; EI-MS (m/z): 248 (26), 235 (100), 220 (10), 205 (10), 191 (16), 115 (11), 104 (14), 85 (17), 71 (22), 57 (33), 43 (17); HRMS (EI): Exact mass calcd. for $C_{21}H_{15}NO_2$ $[M]^+$: 313.1103. Found: 313.1100.



5bi

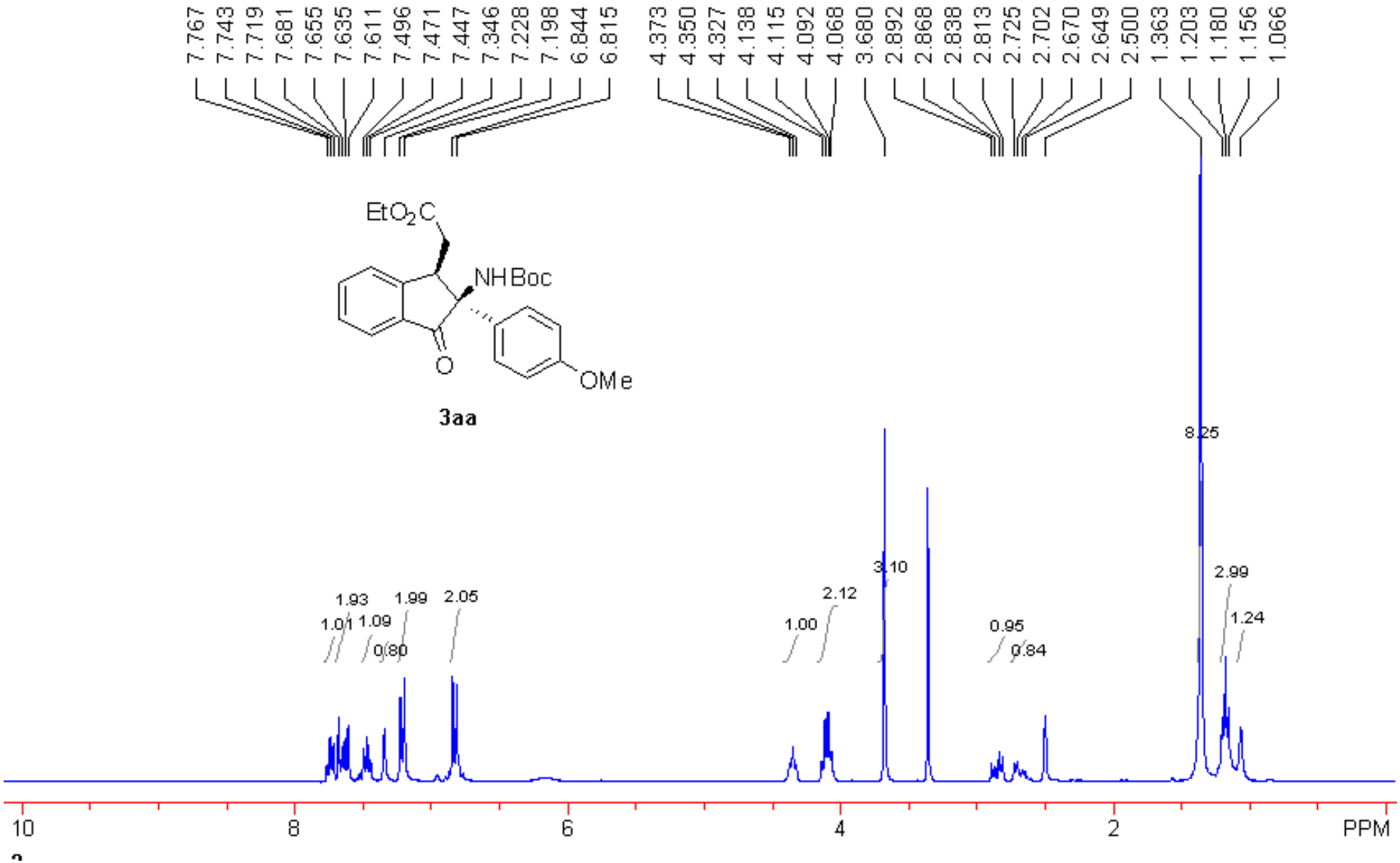
5bi was isolated as white solid (57.9 mg, 87% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.5 Hz, 1H), 7.35-7.32 (m, 6H), 7.24-7.21 (m, 2H), 3.93 (dd, *J* = 2.1, 10.5 Hz, 1H), 3.11 (dd, *J* = 10.8, 17.4 Hz, 1H), 2.51 (s, 3H), 2.50 (d, *J* = 2.7, 17.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.1, 175.5, 155.4, 148.5, 139.6, 131.8, 130.4, 128.9, 128.1, 126.3, 125.3, 125.1, 72.2, 48.2, 36.2, 22.3; IR (film): ν_{max} (cm^{-1}) = 3420, 3166, 3077, 2935, 2853, 1698, 1604, 1496, 1446, 1346, 1279, 1255, 1106, 919, 838, 817, 786, 754, 734, 703, 695, 486; EI-MS (m/z): 248 (26), 235 (100), 220 (10), 205 (11), 115 (10), 104 (11), 84 (13), 77 (12), 57 (7), 49 (15); HRMS (EI): Exact mass calcd. for $C_{18}H_{15}NO_2$ $[M]^+$: 277.1103. Found: 277.1102.



5ci

5ci was isolated as white solid (54.7 mg, 81% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54-7.50 (m, 3H), 7.39-7.35 (m, 3H), 7.26-7.21 (m, 2H), 6.93 (br s, 1H, NH), 3.96 (d, *J* = 10.5 Hz, 1H), 3.13 (dd, *J* = 10.5, 17.7 Hz, 1H), 2.50 (dd, *J* = 2.7, 18.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.7, 175.1, 163.1 (d, *J* = 250 Hz), 150.3, 138.9, 136.0 (d, *J* = 7.3 Hz), 129.1, 128.5, 127.7 (d, *J* = 8.1 Hz), 125.1, 124.7 (d, *J* = 24.0 Hz), 111.2 (d, *J* = 22.1 Hz),

72.5, 47.9, 36.2; ^{19}F NMR (282 MHz, $CDCl_3$) δ -111.4 (m); IR (film): ν_{max} (cm^{-1}) = 3437, 3158, 3069, 2872, 1724, 1691, 1609, 1481, 1431, 1357, 1296, 1262, 1228, 1186, 1020, 879, 835, 818, 733, 695, 675, 492, 469; EI-MS (m/z): 281 (11), 252 (43), 239 (100), 224 (20), 221 (11), 209 (27), 183 (17), 104 (17), 84 (37), 77 (19), 57 (26), 49 (49); HRMS (EI): Exact mass calcd. for $C_{17}H_{12}NO_2F$ $[M]^+$: 281.0852. Found: 281.0855.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OMe
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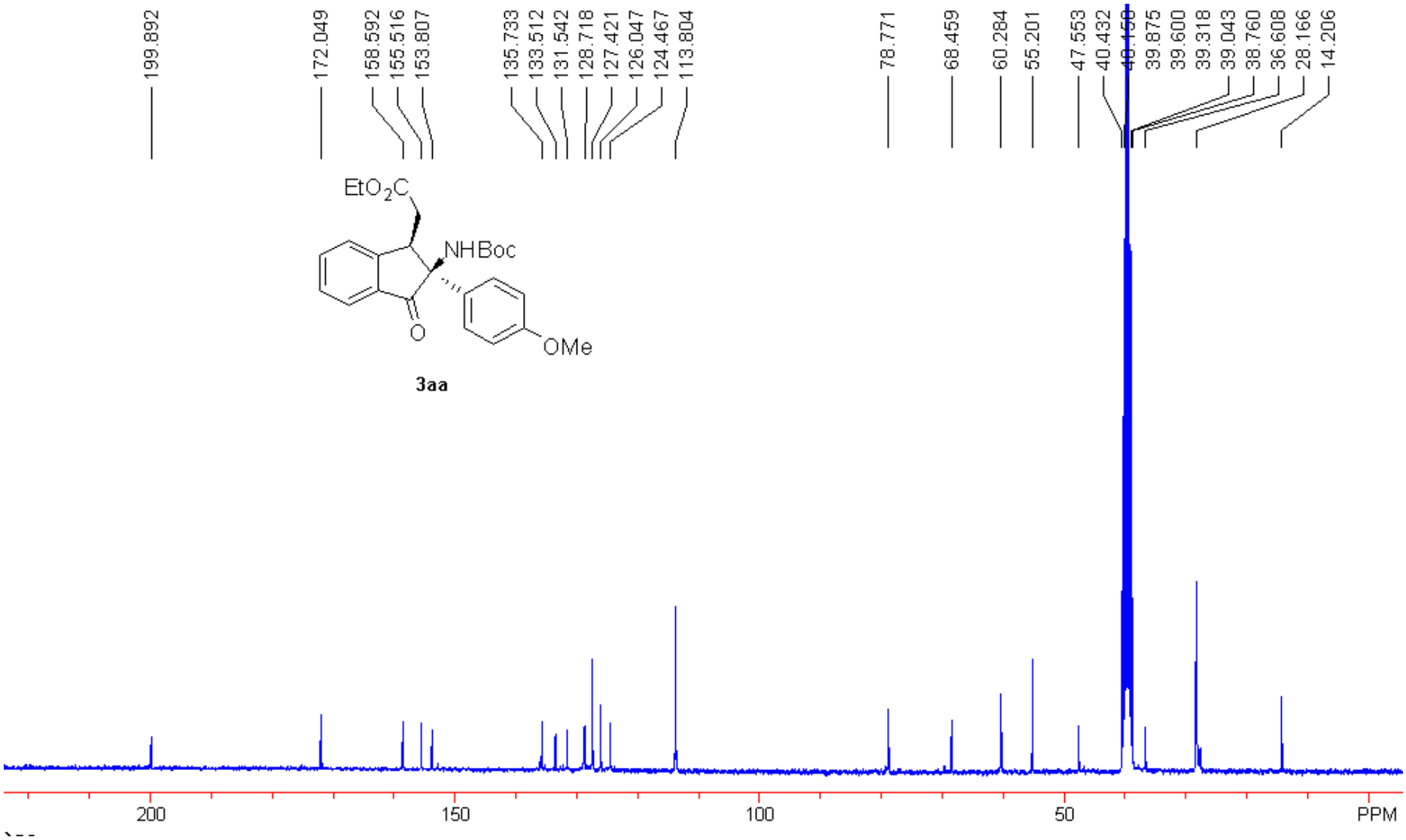
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OMe
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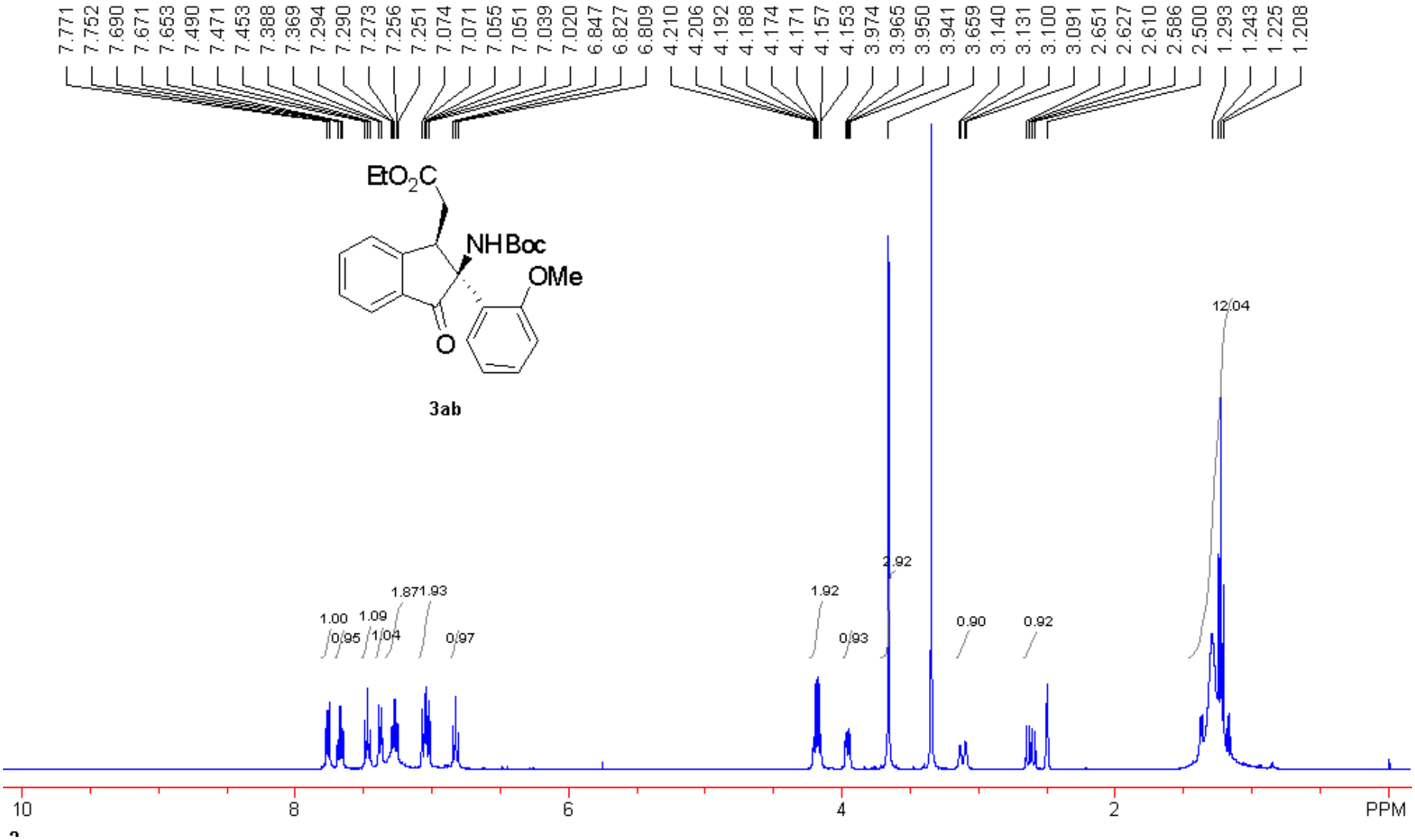
PPM

3aa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OMe
O
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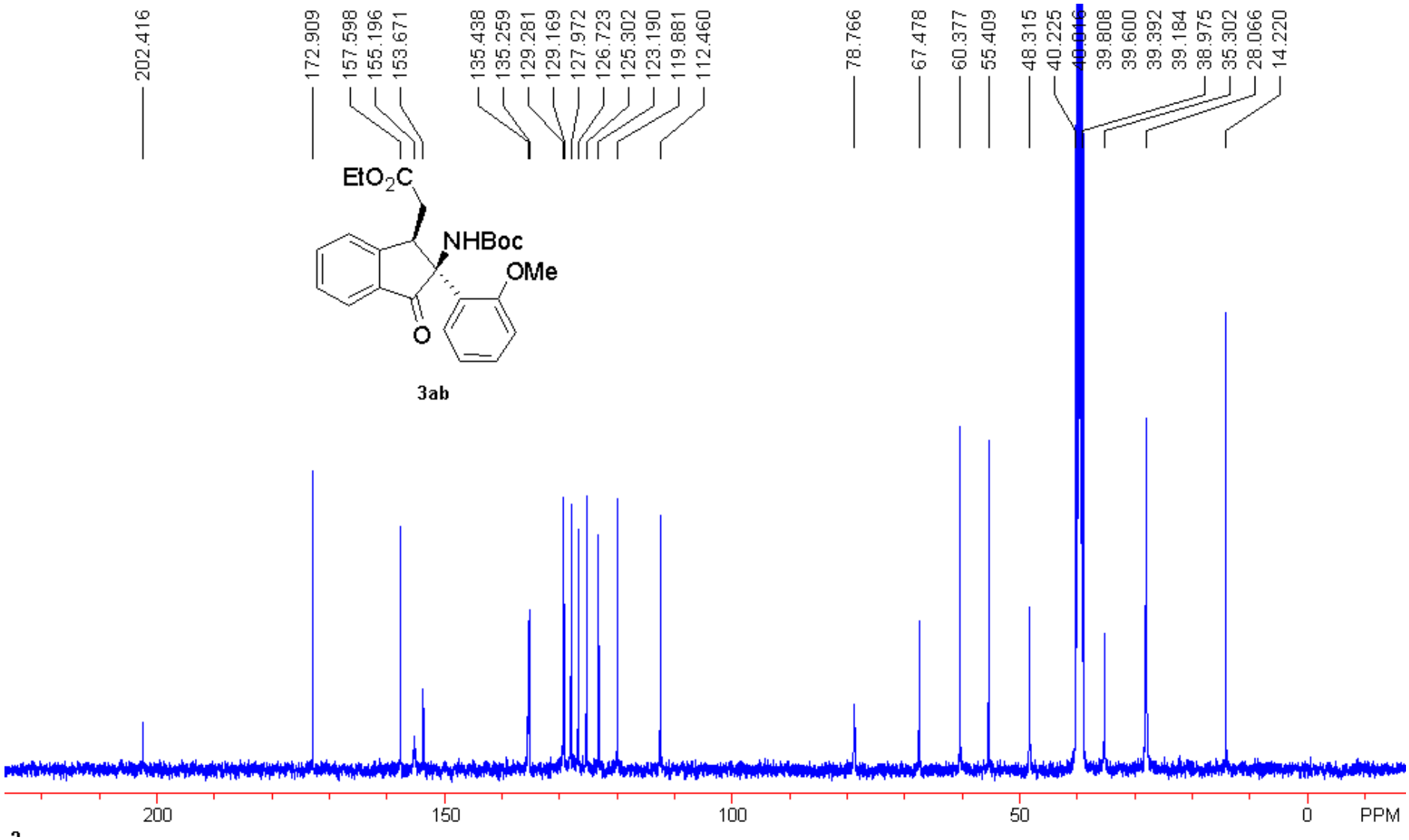
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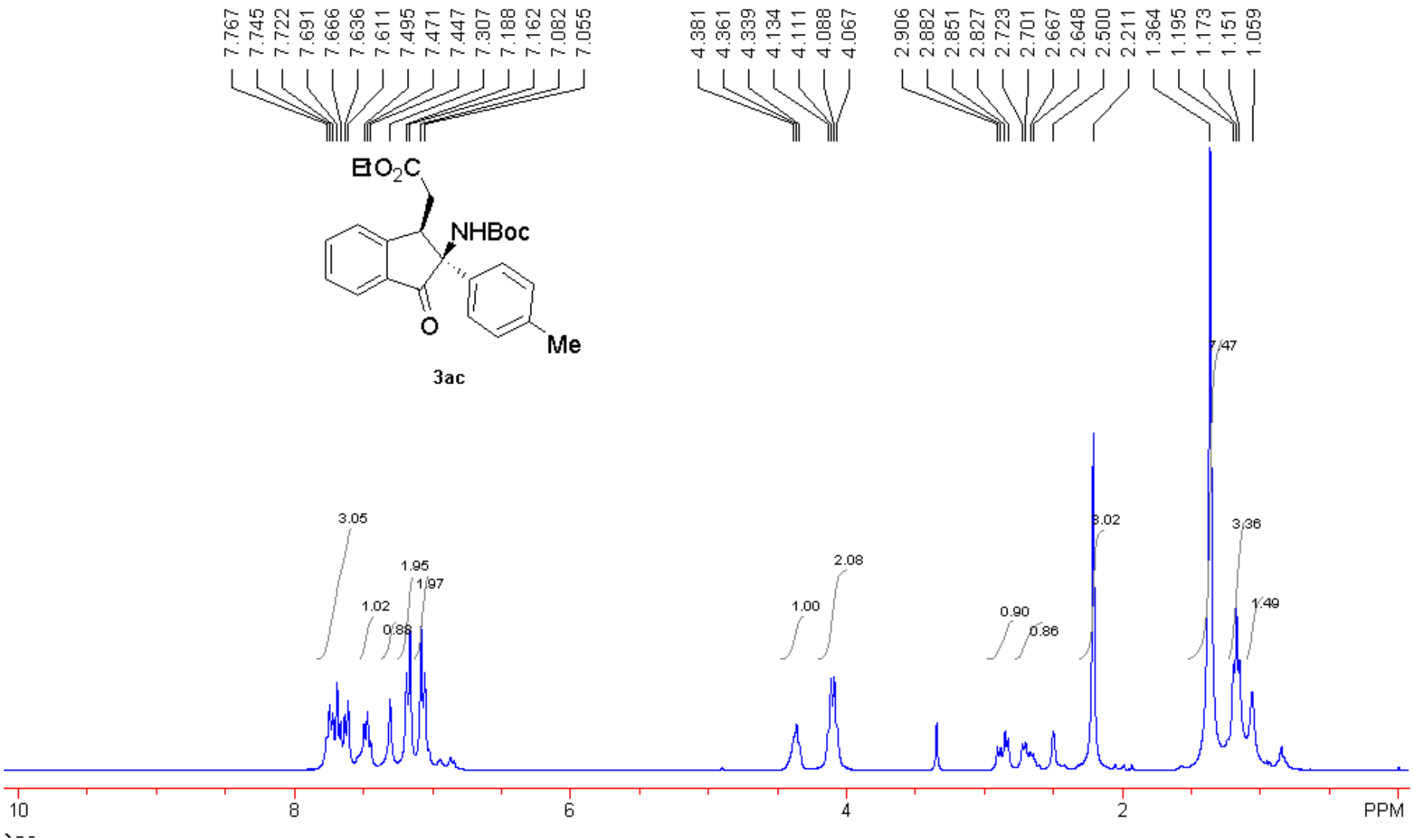
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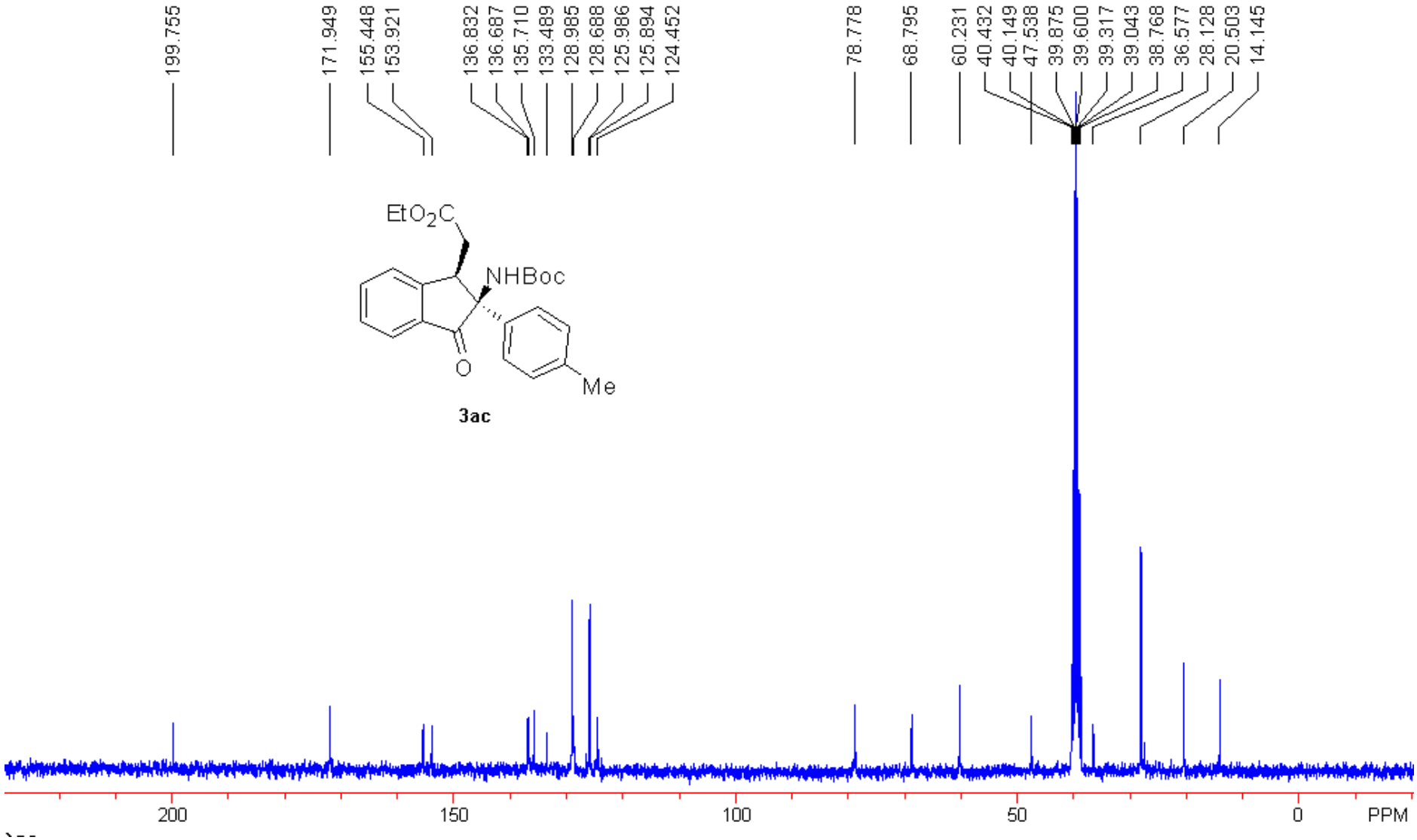
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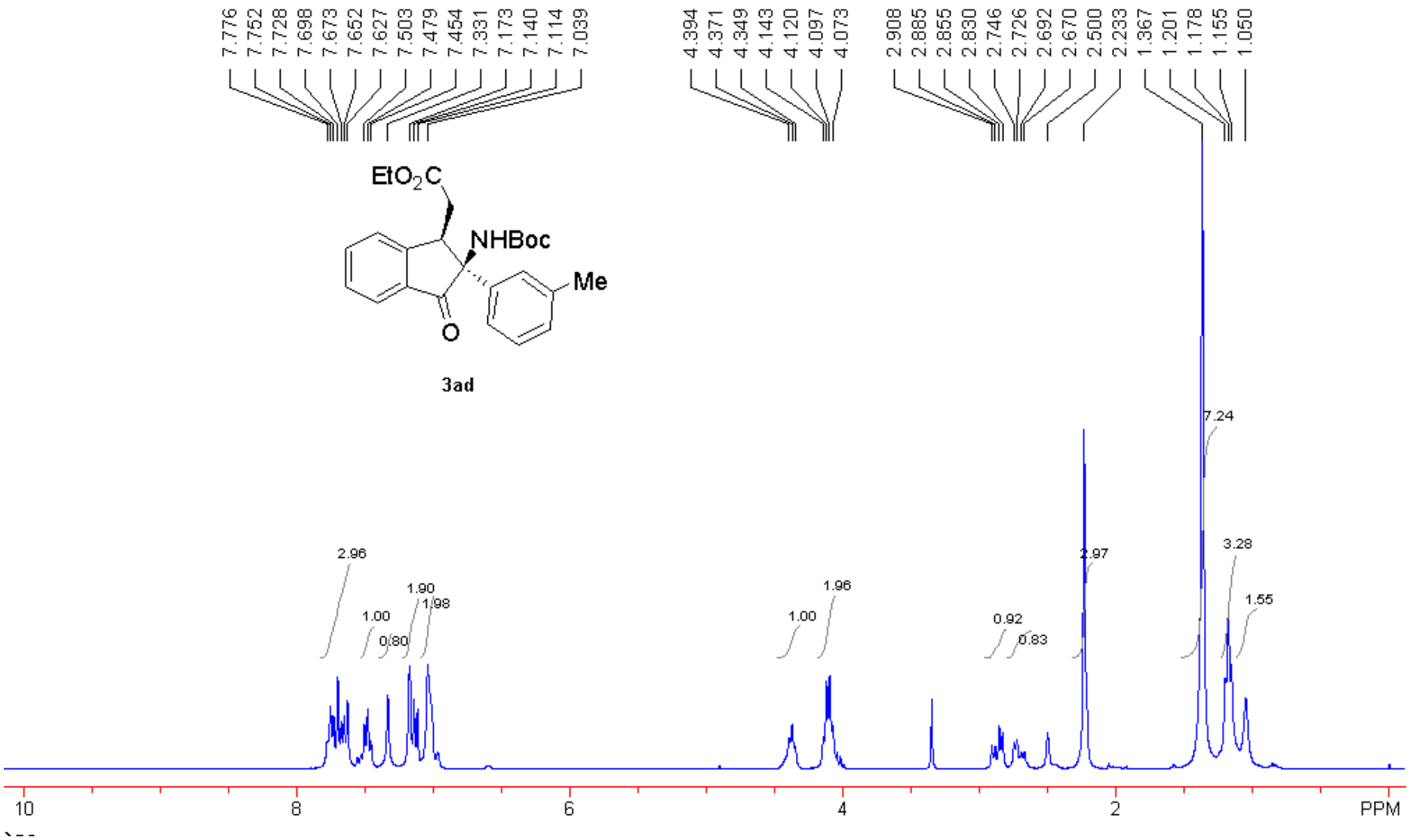
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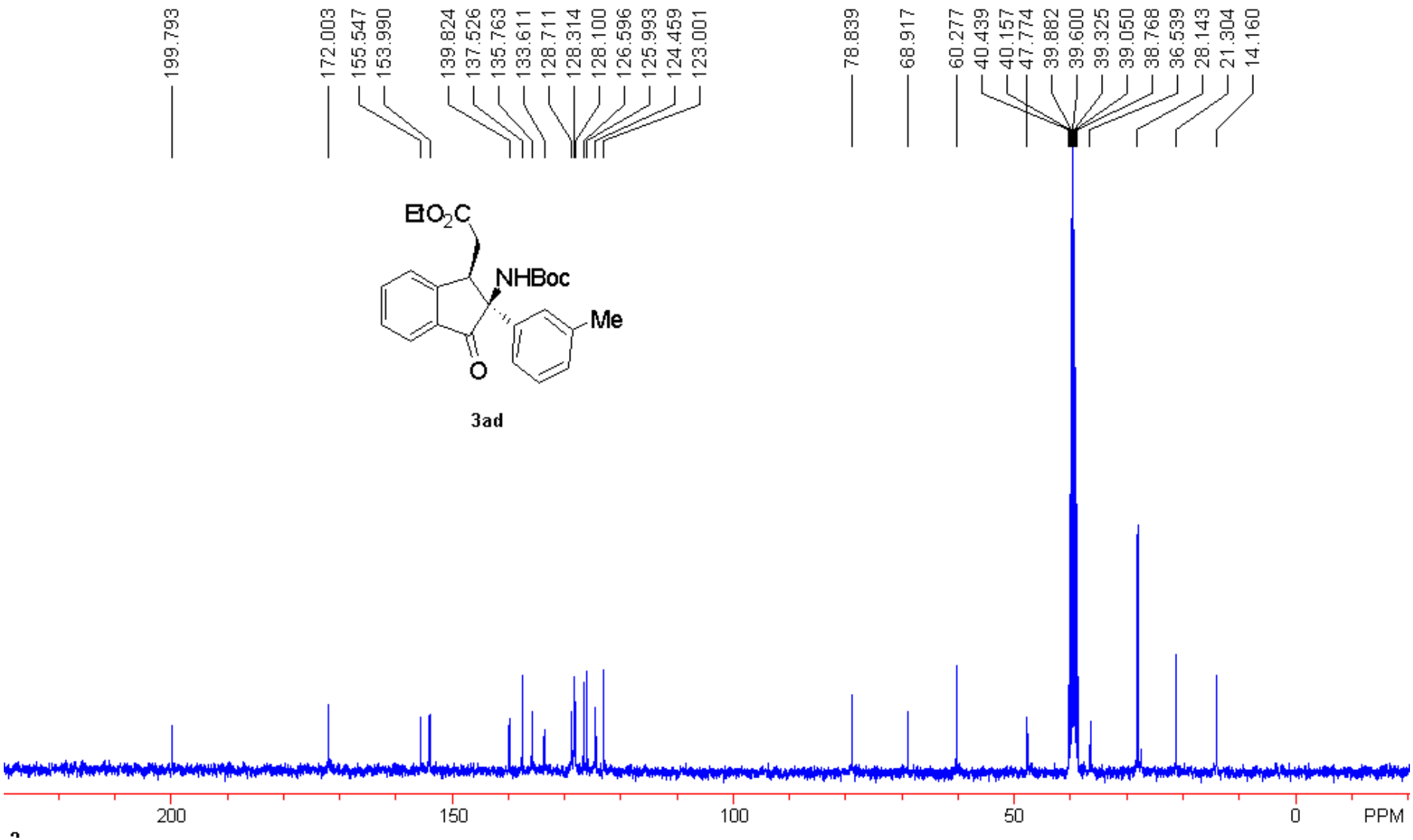
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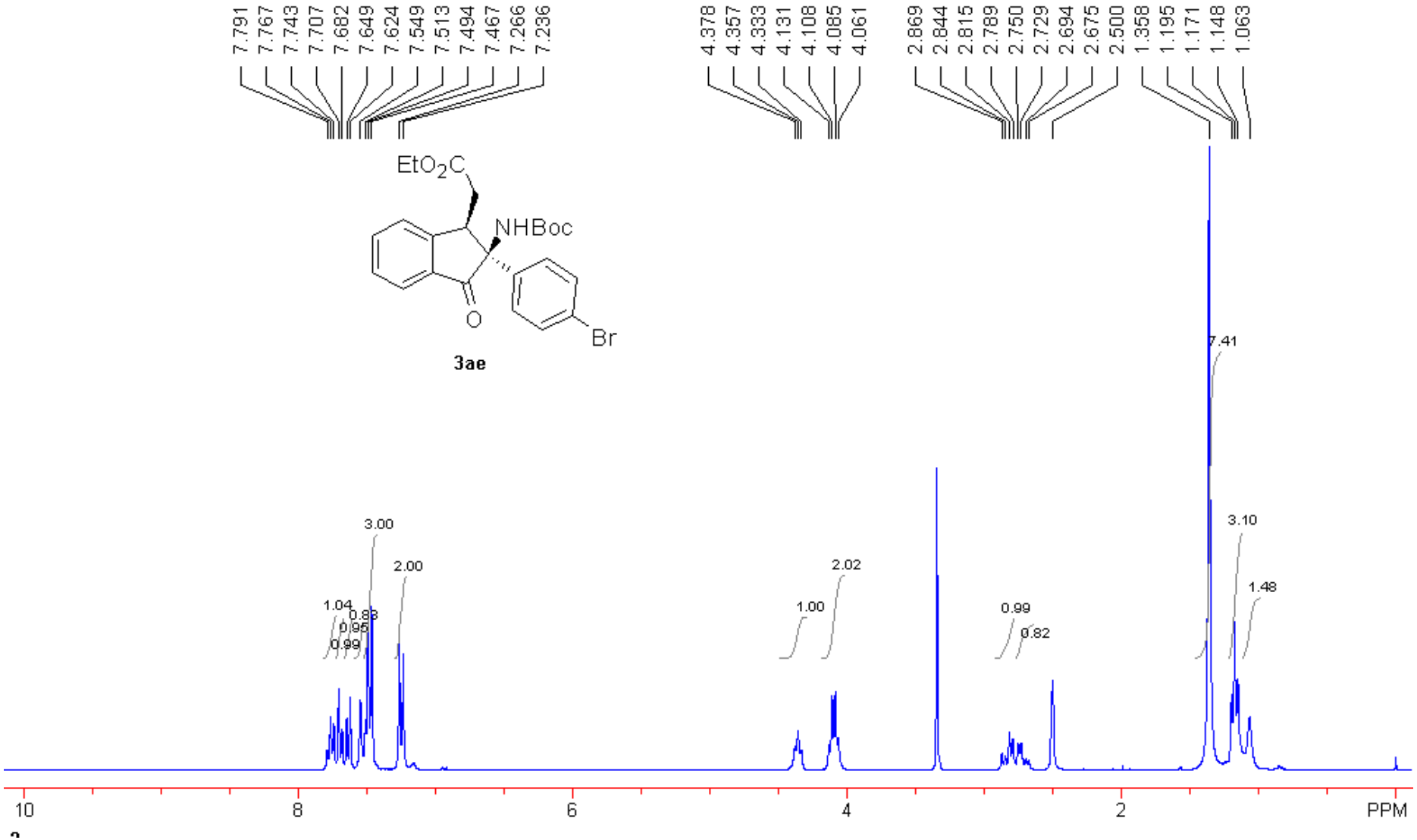
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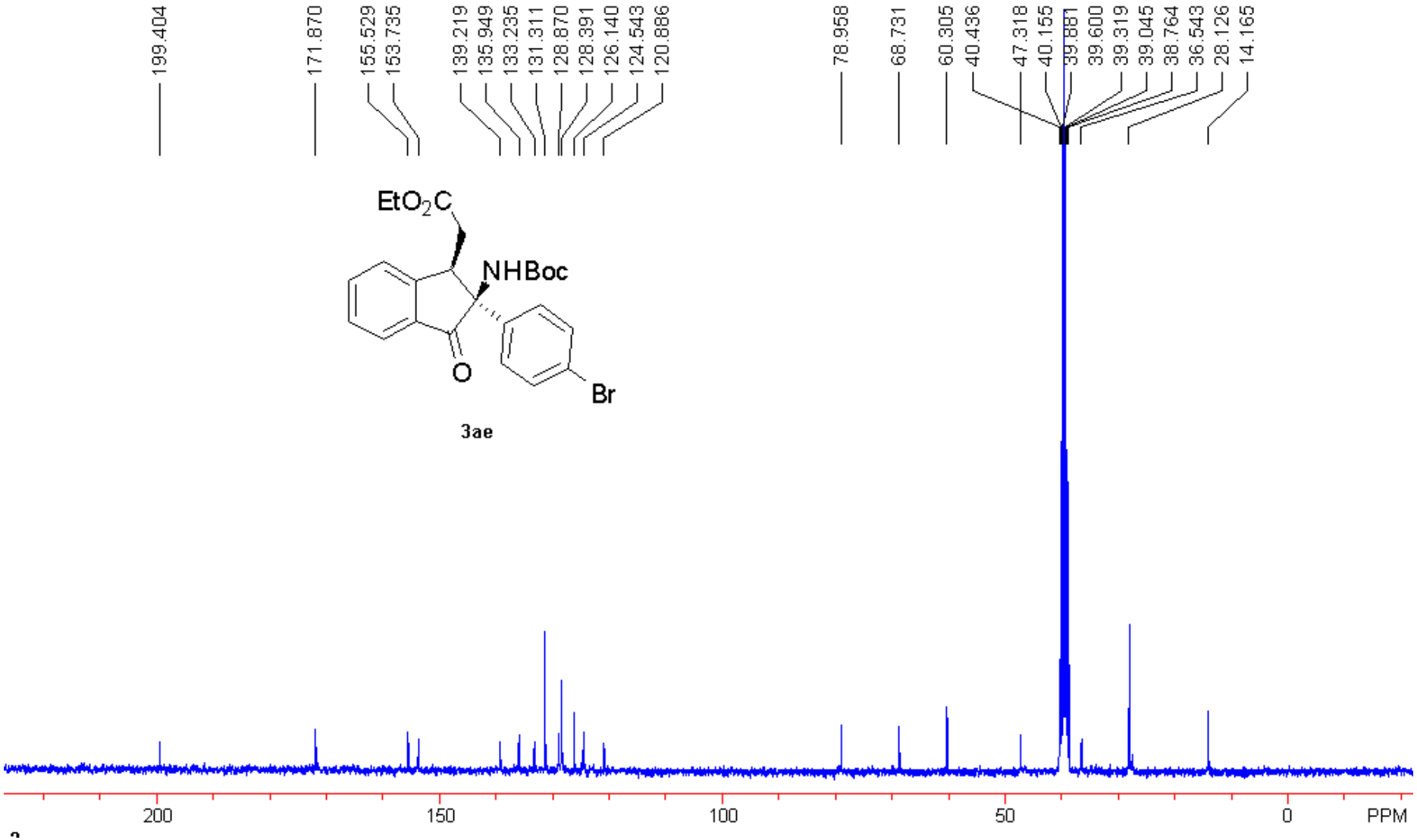
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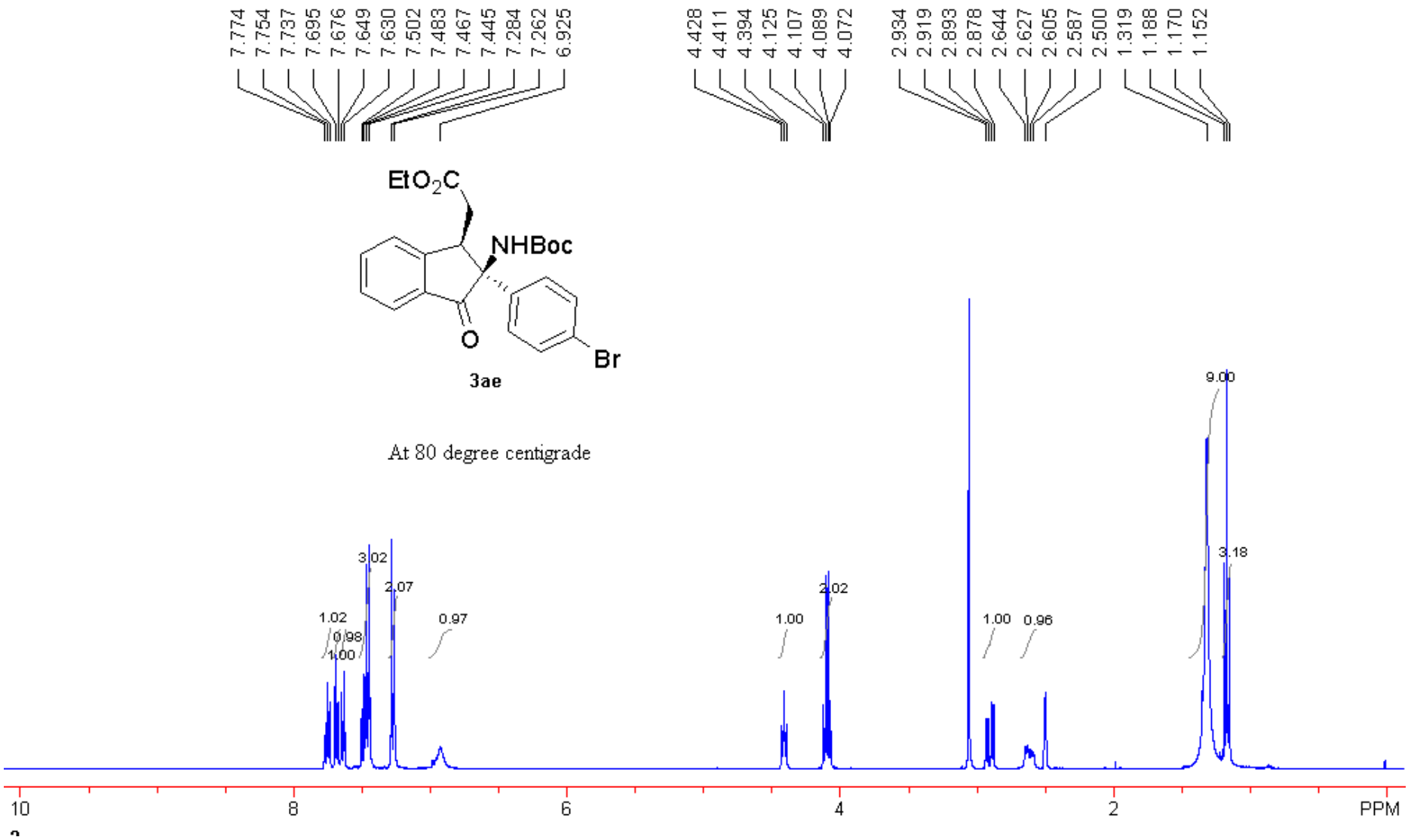
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NHBoc
O
Br
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At 80 degree centigrade
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1.00
3.02
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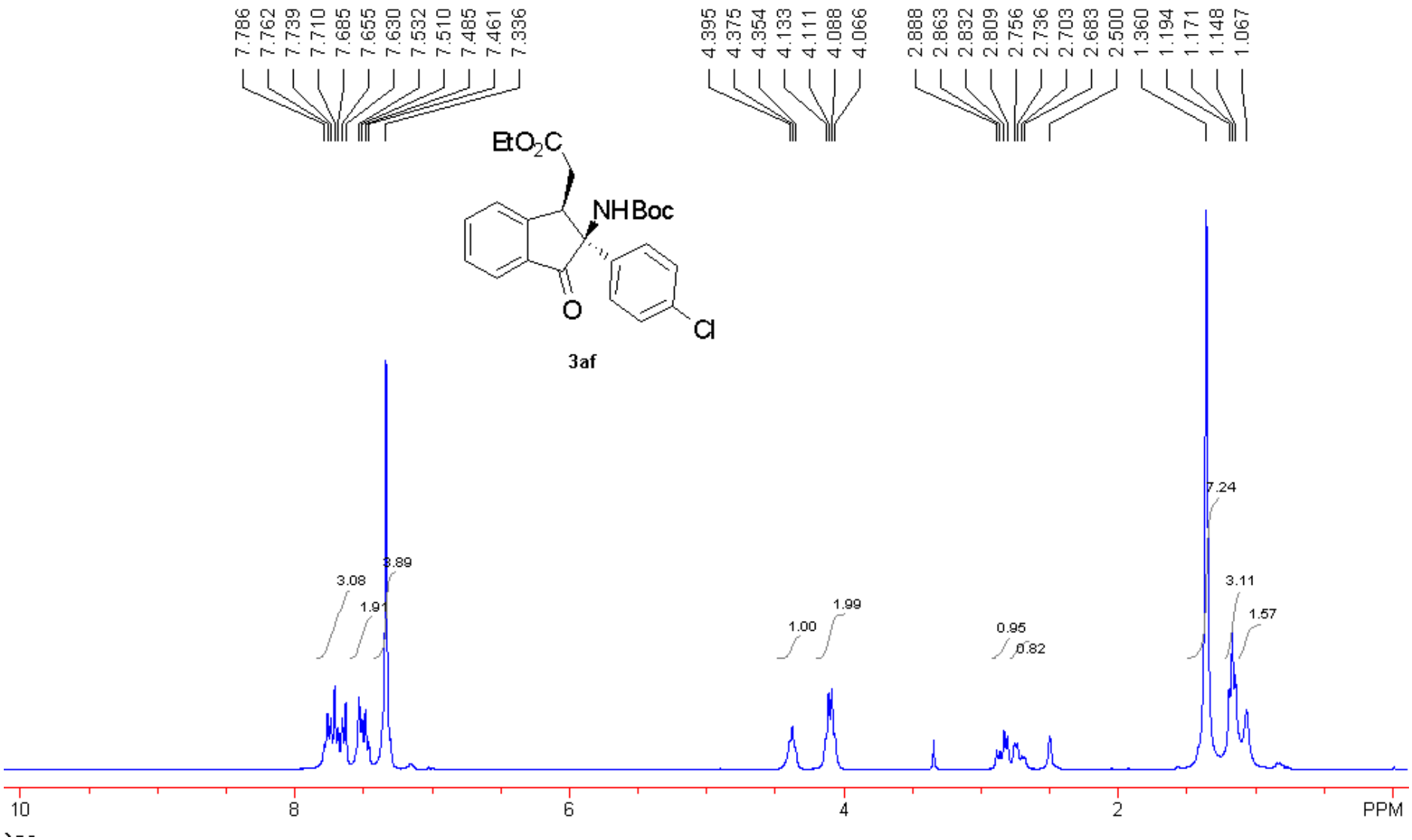
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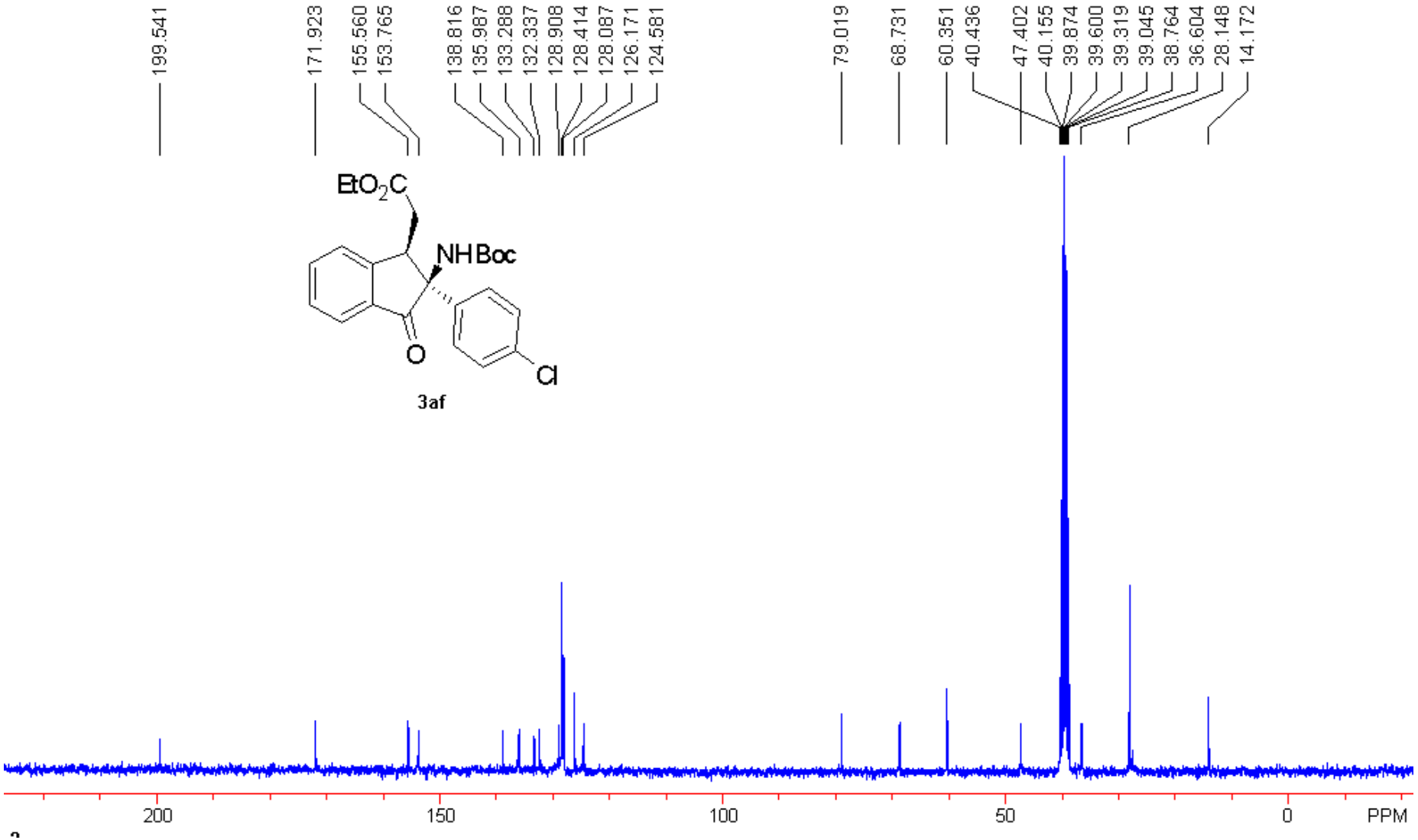
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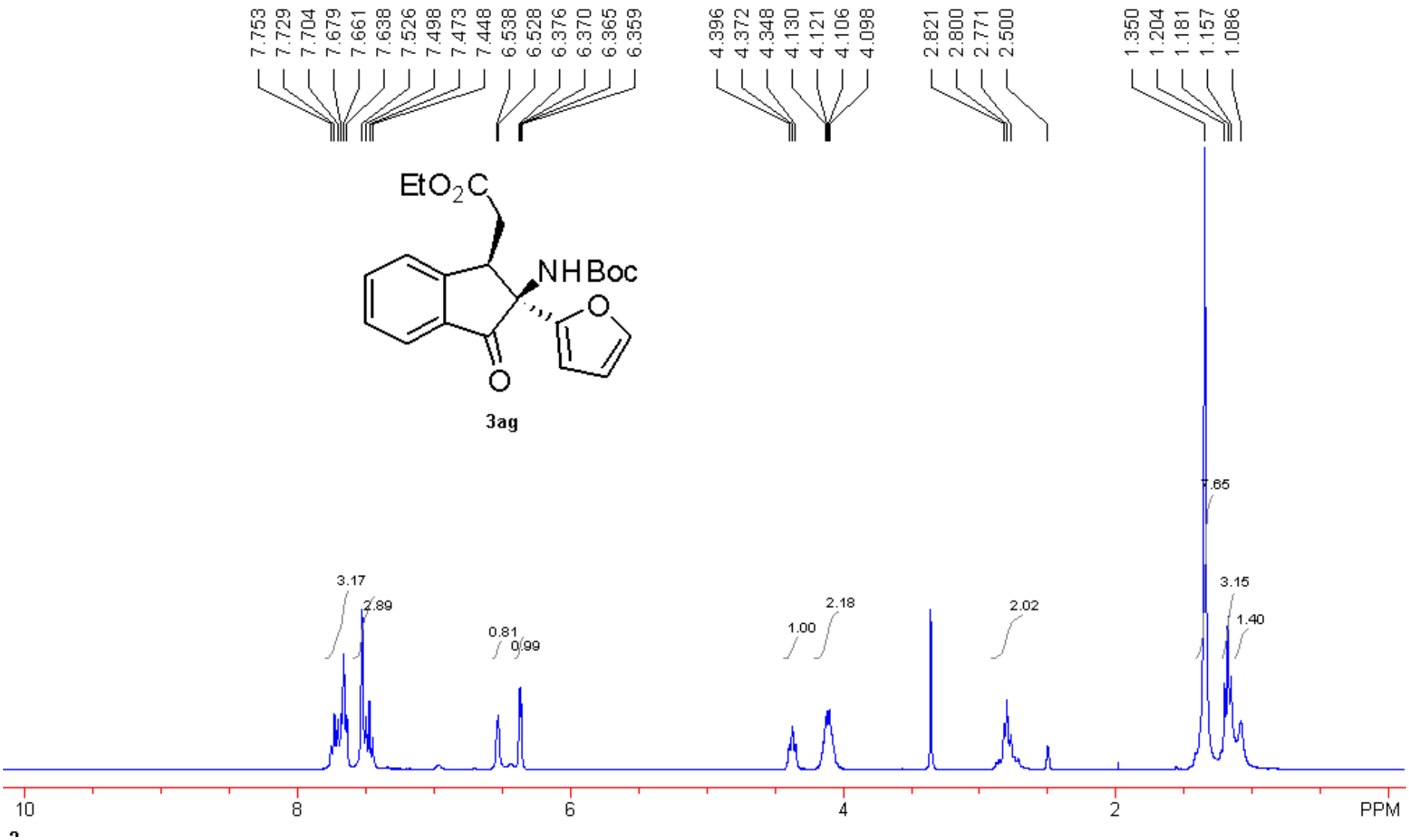
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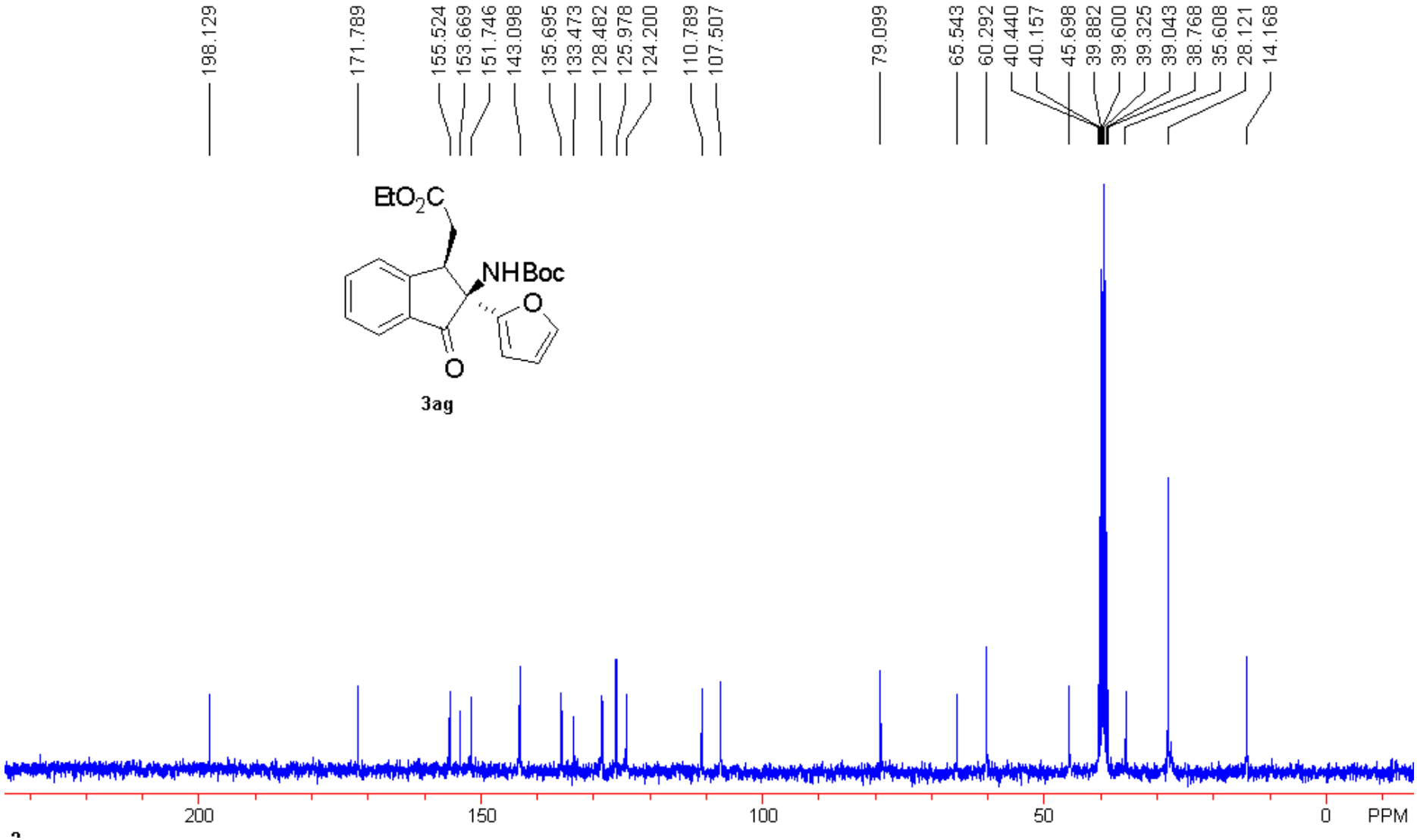
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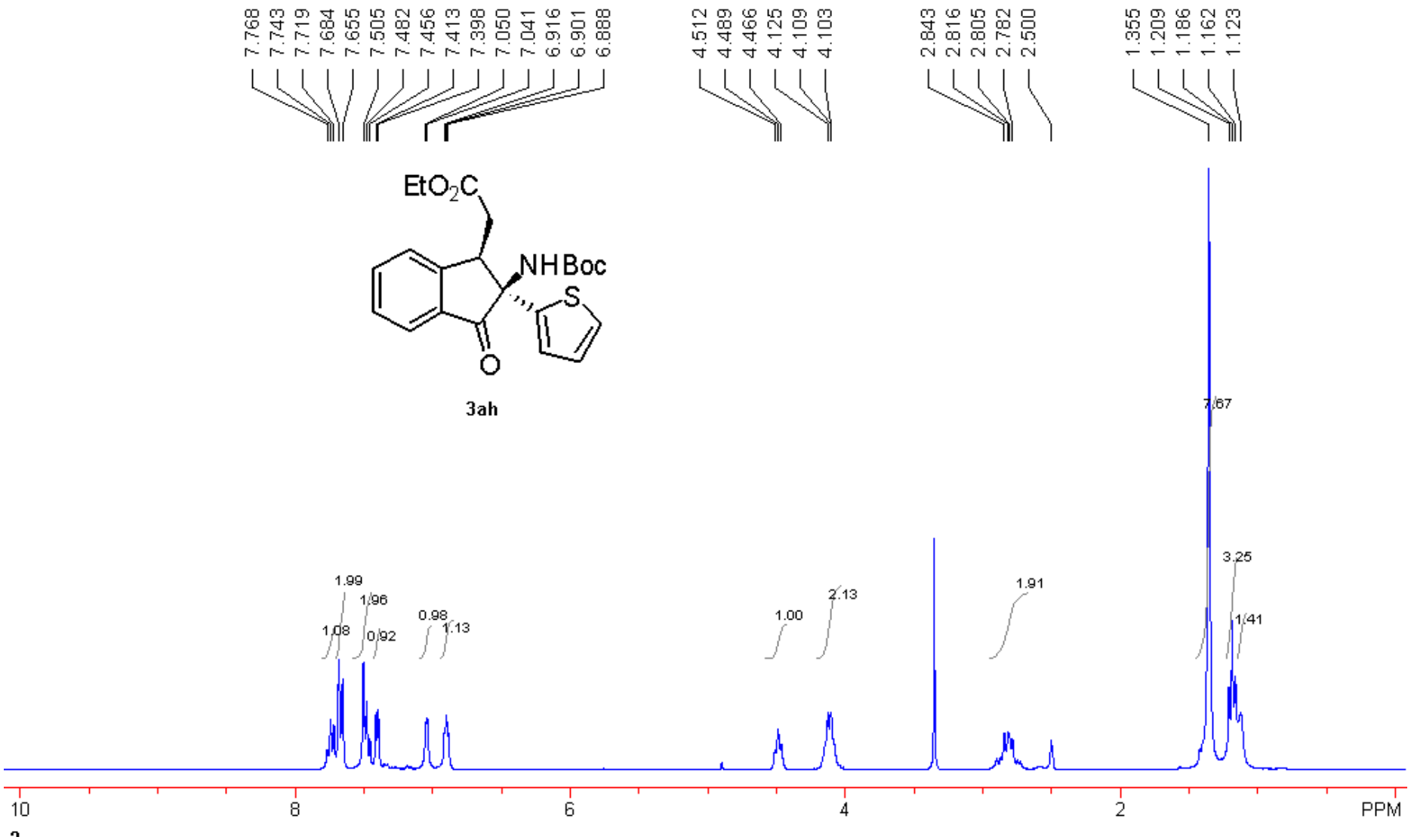
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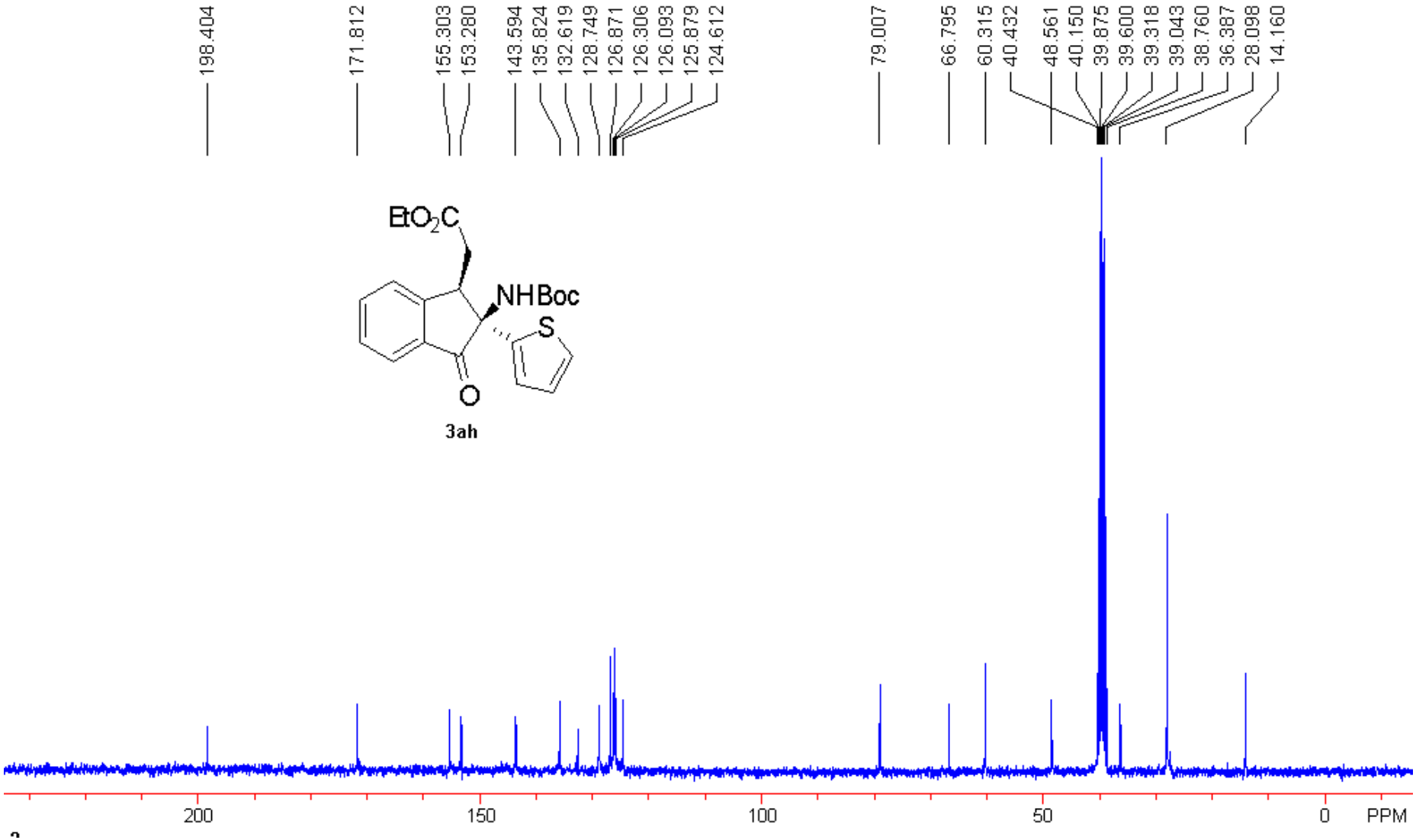
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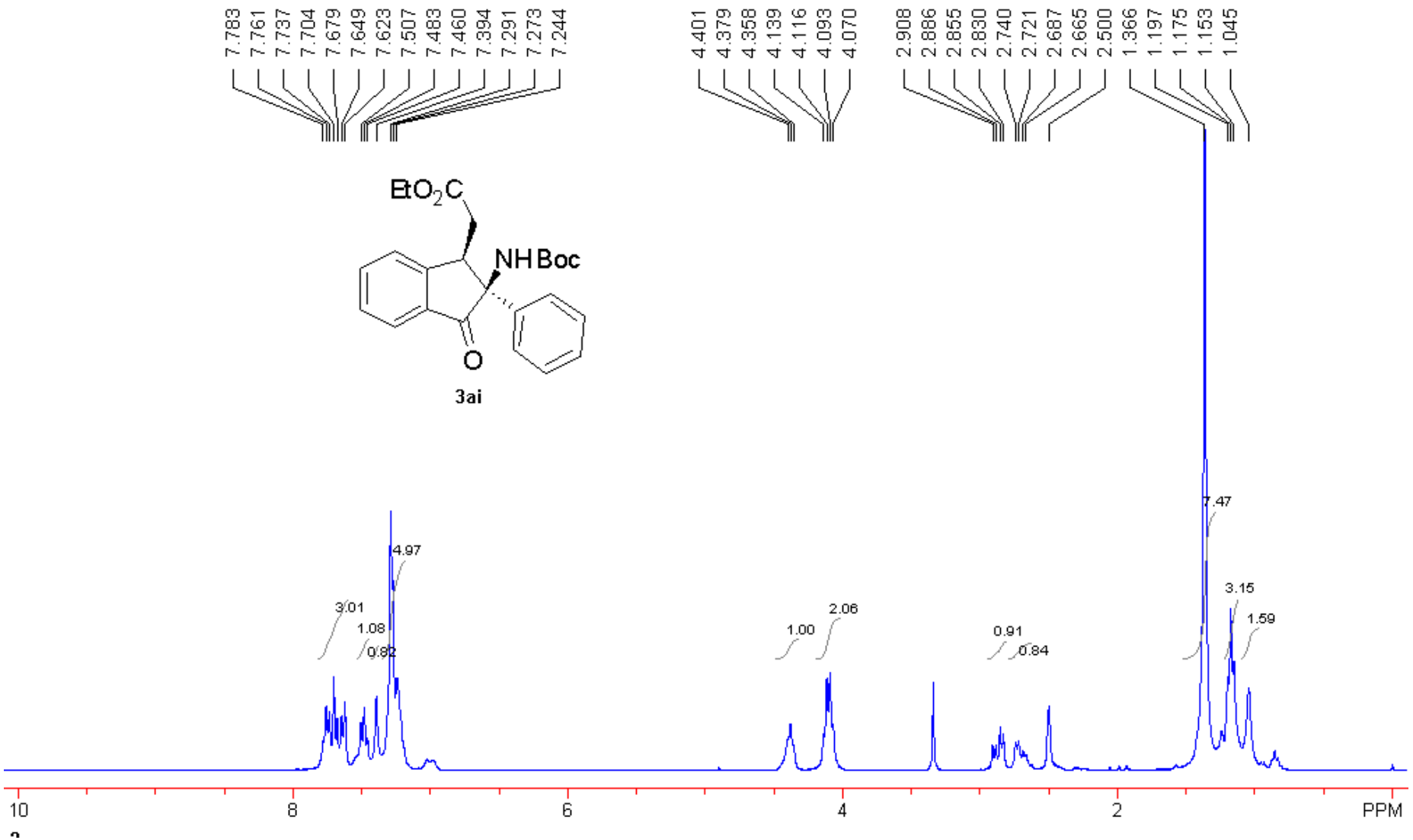
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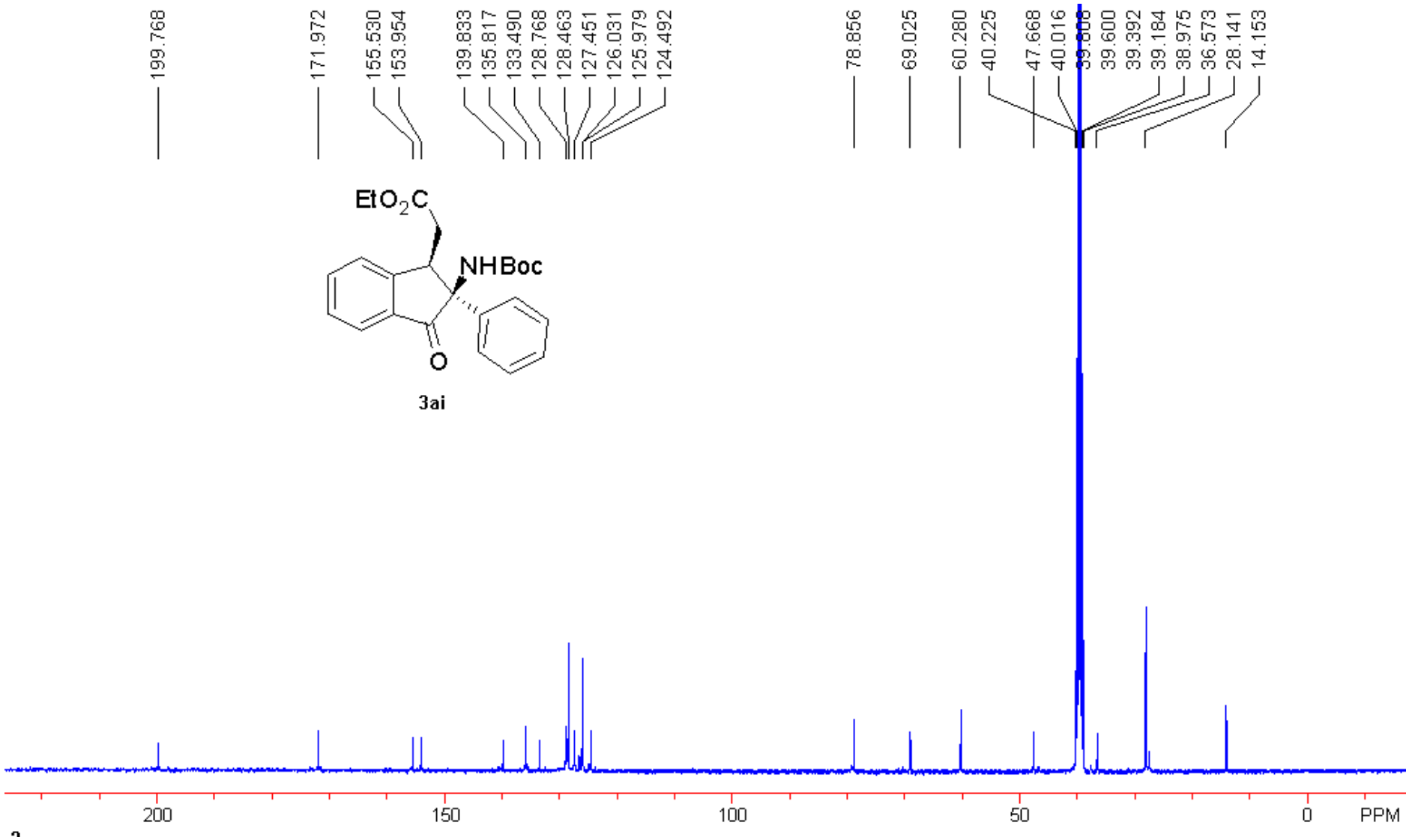
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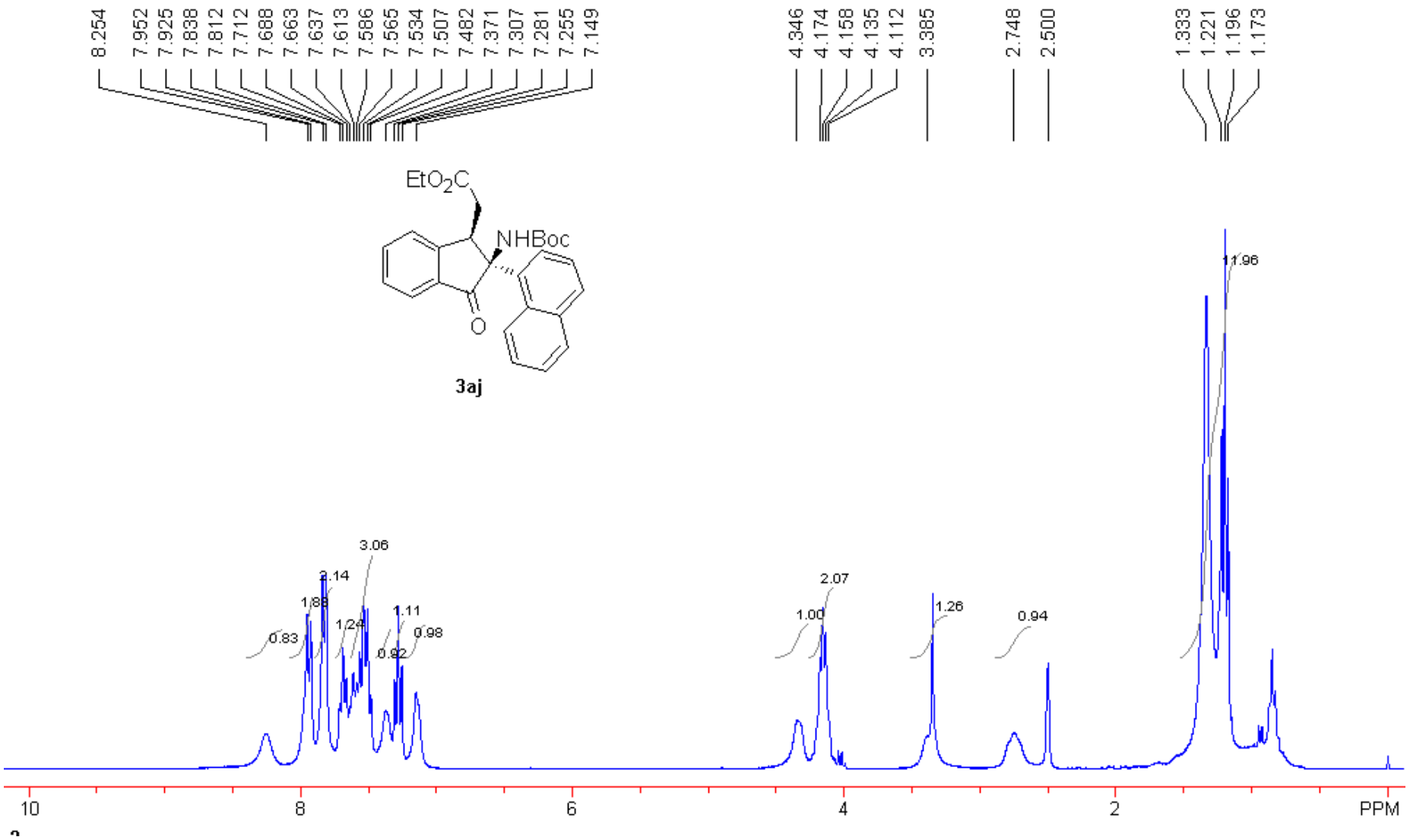
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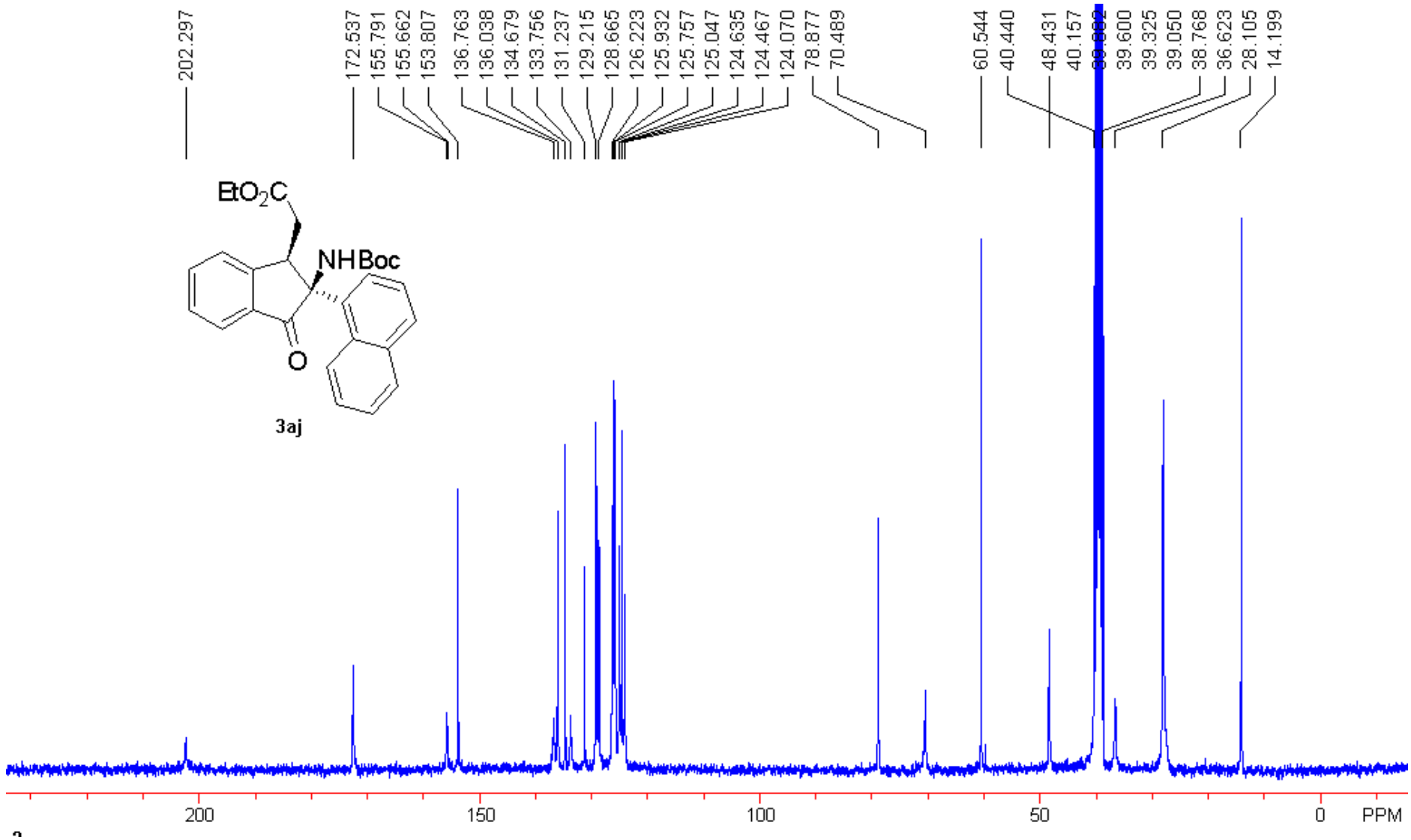
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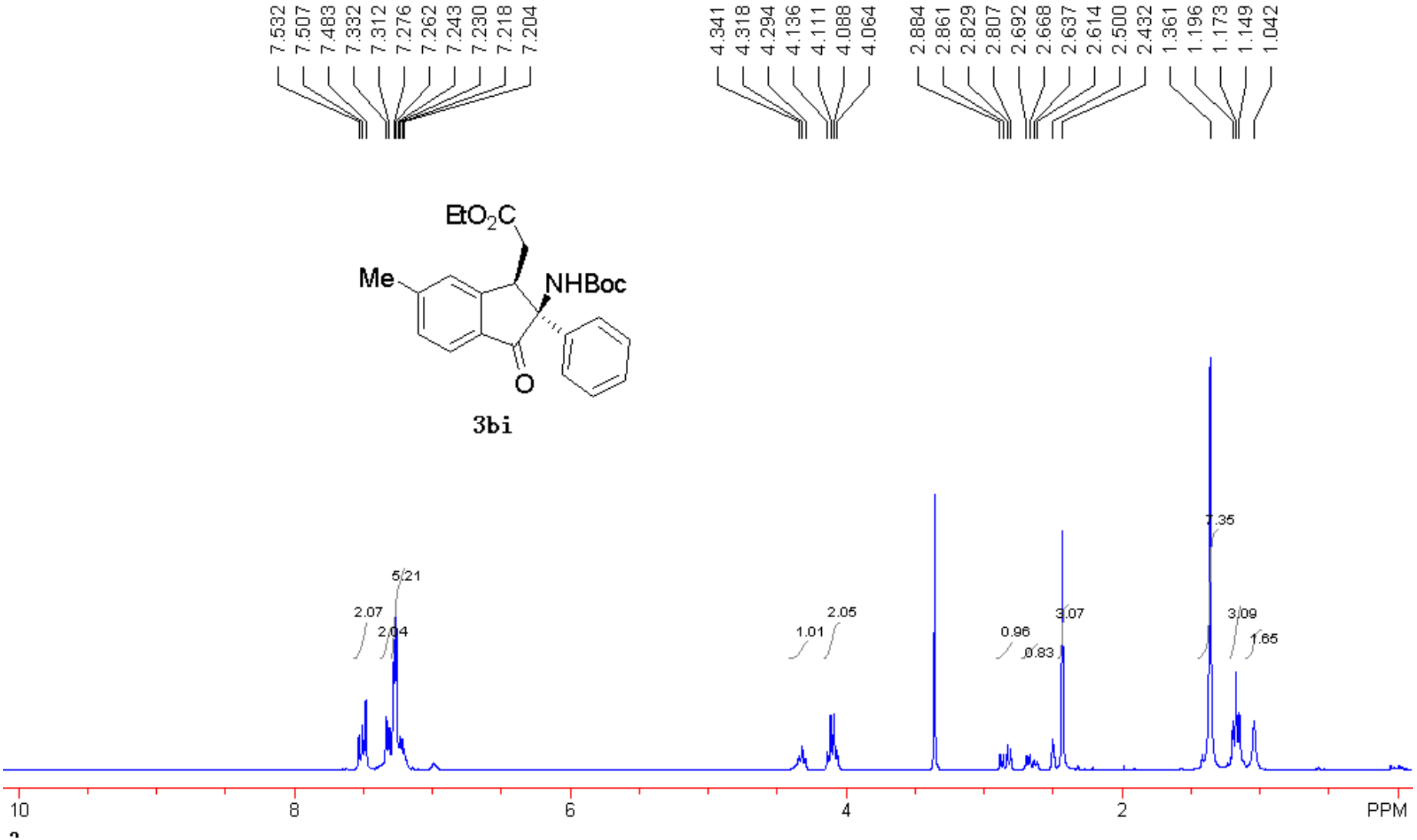
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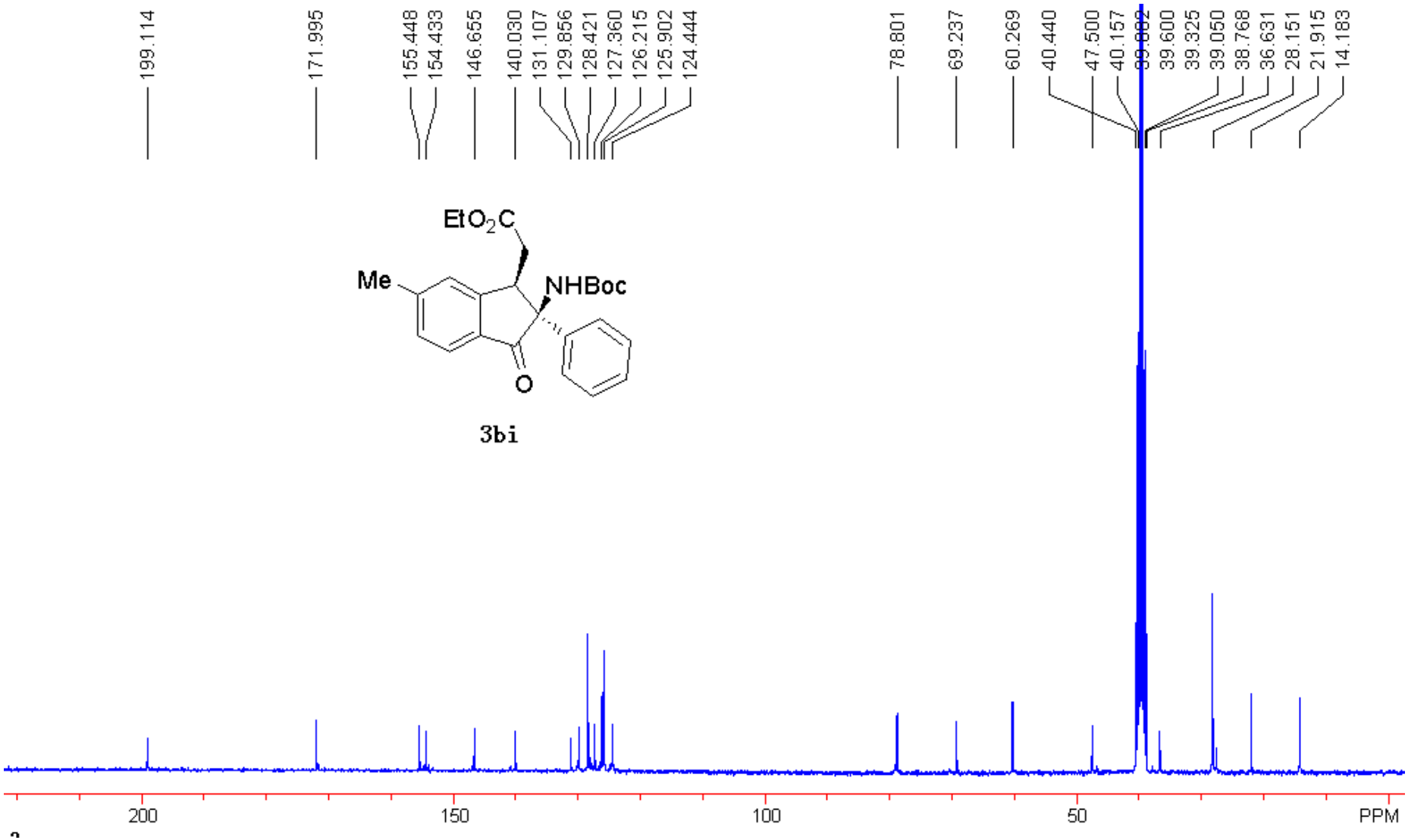
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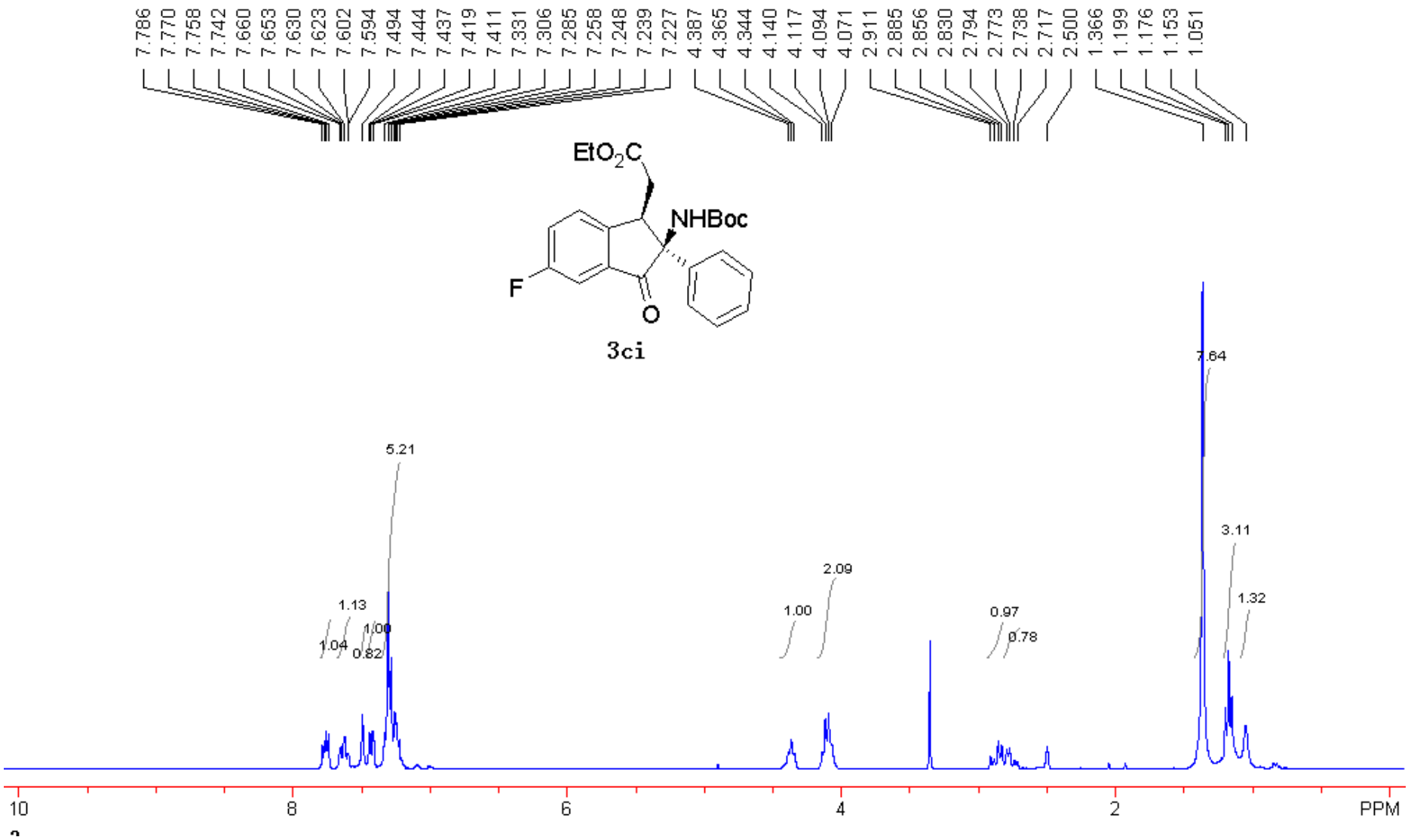
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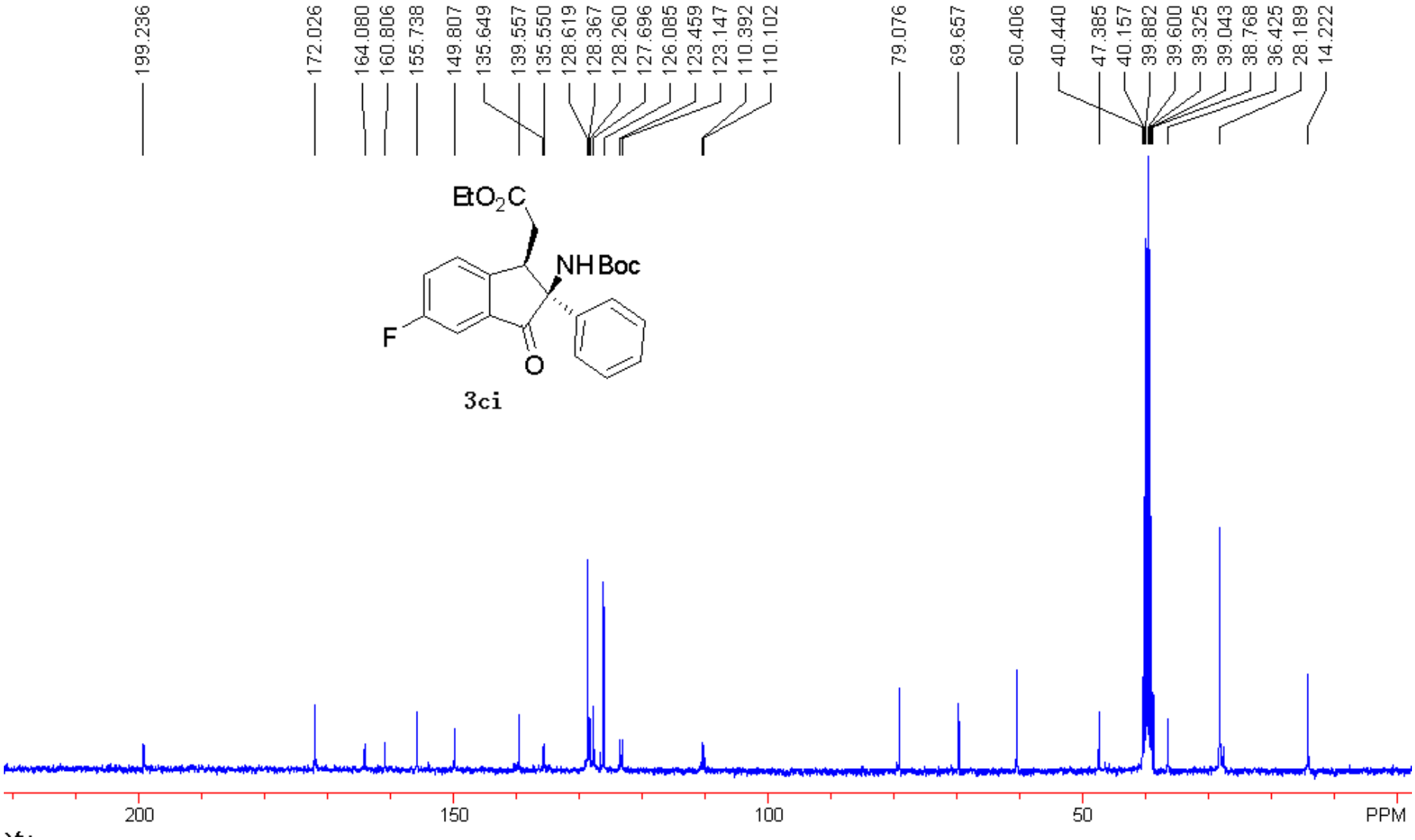
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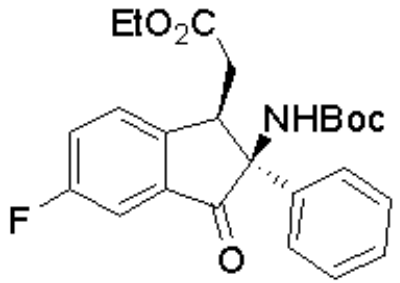

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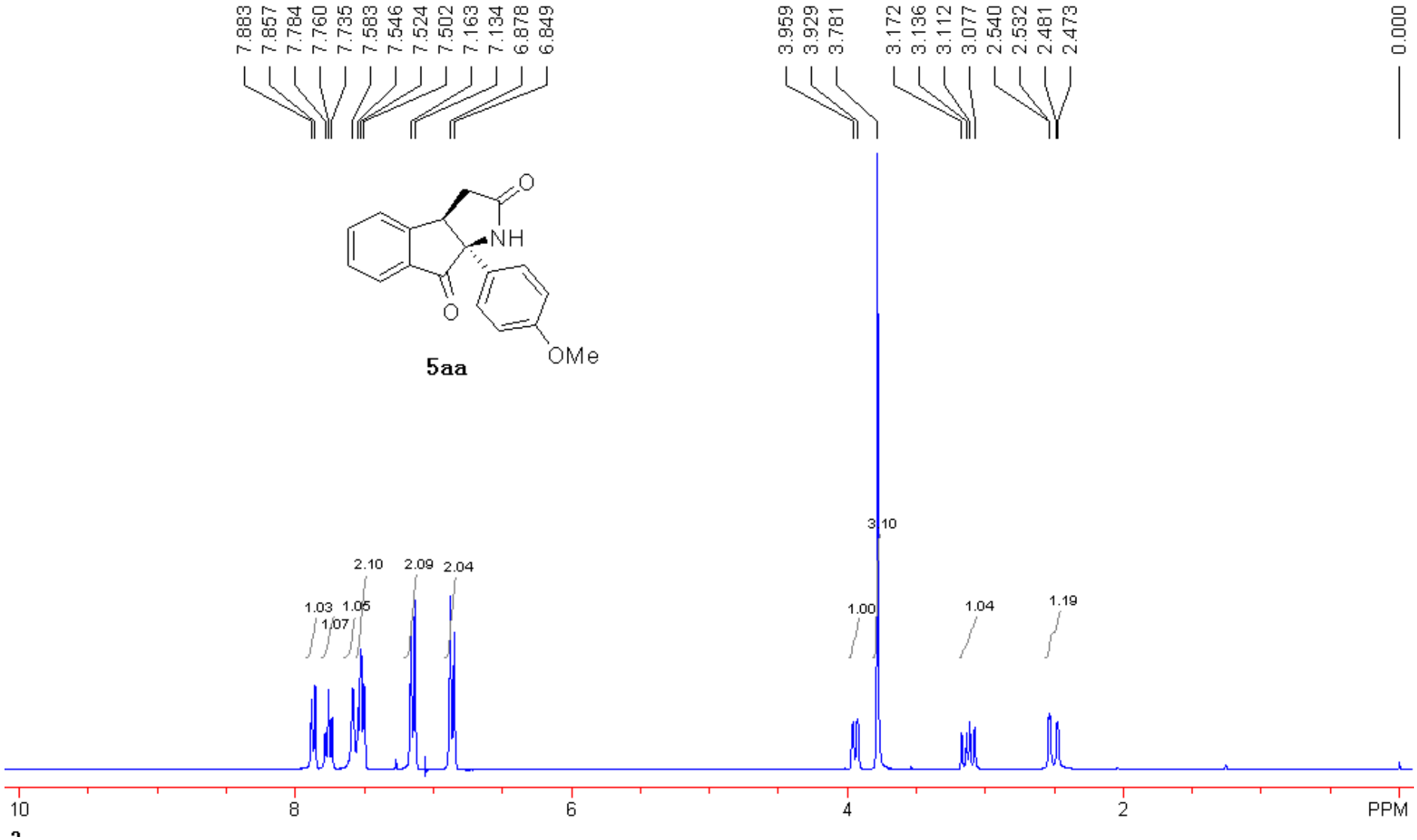
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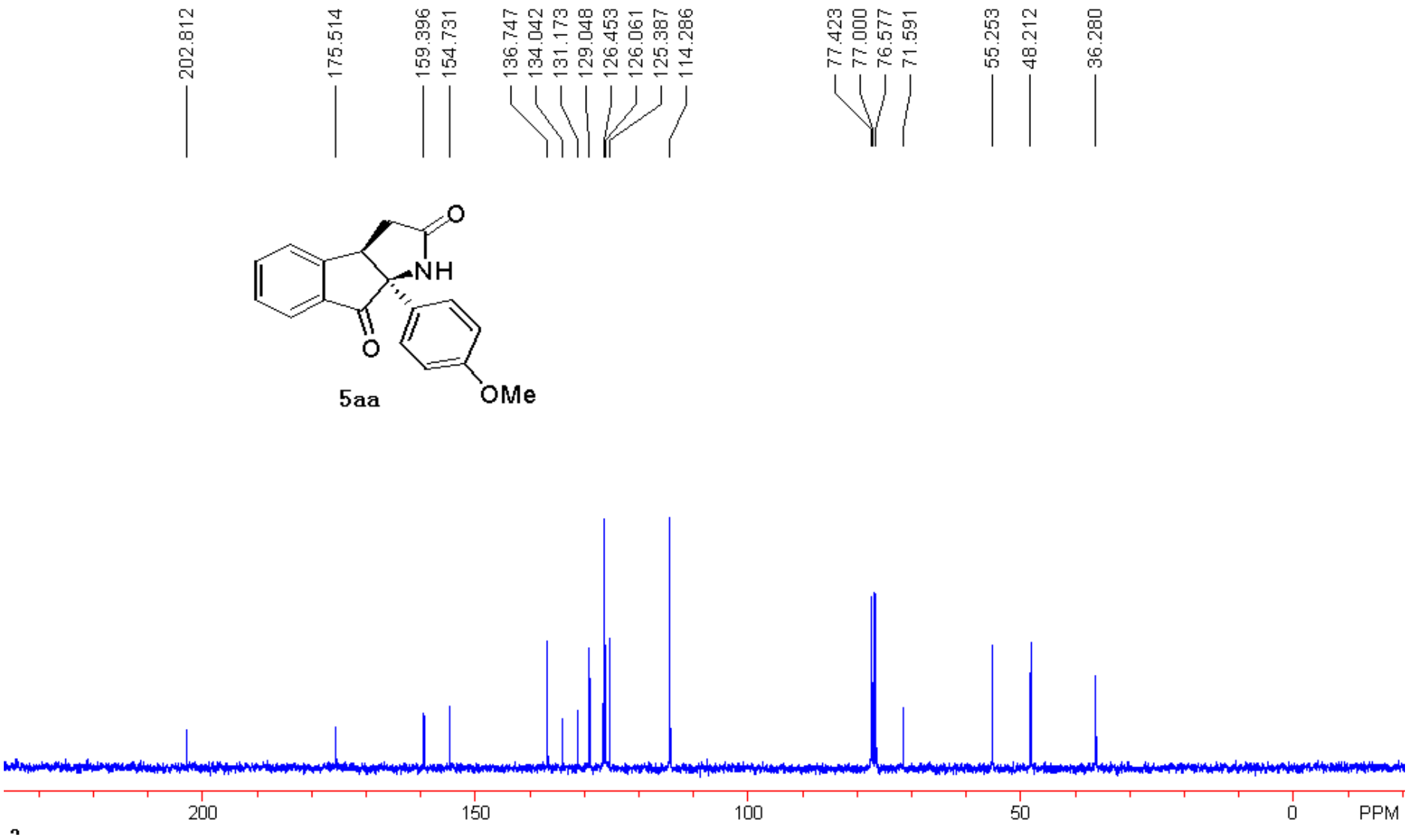
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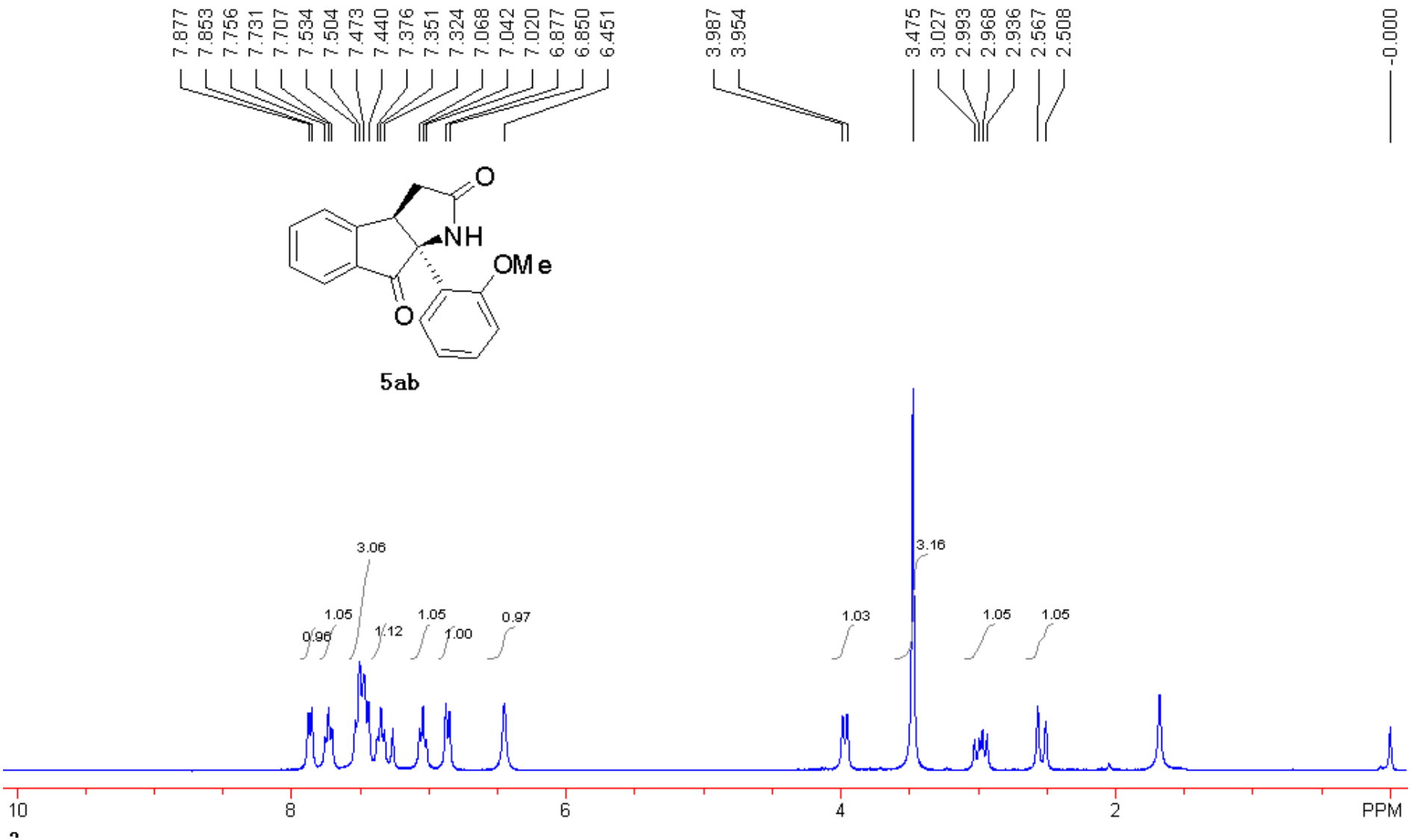
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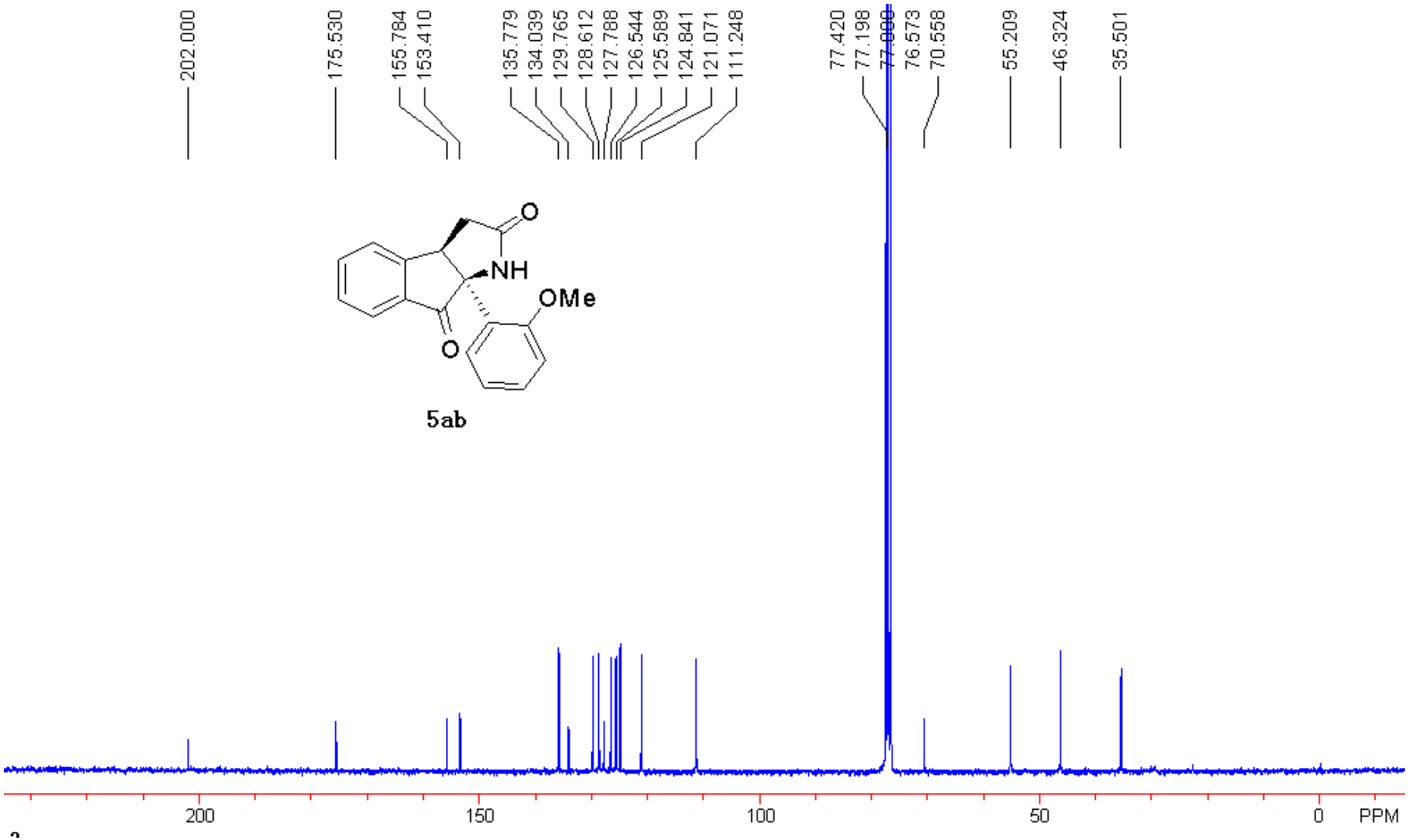
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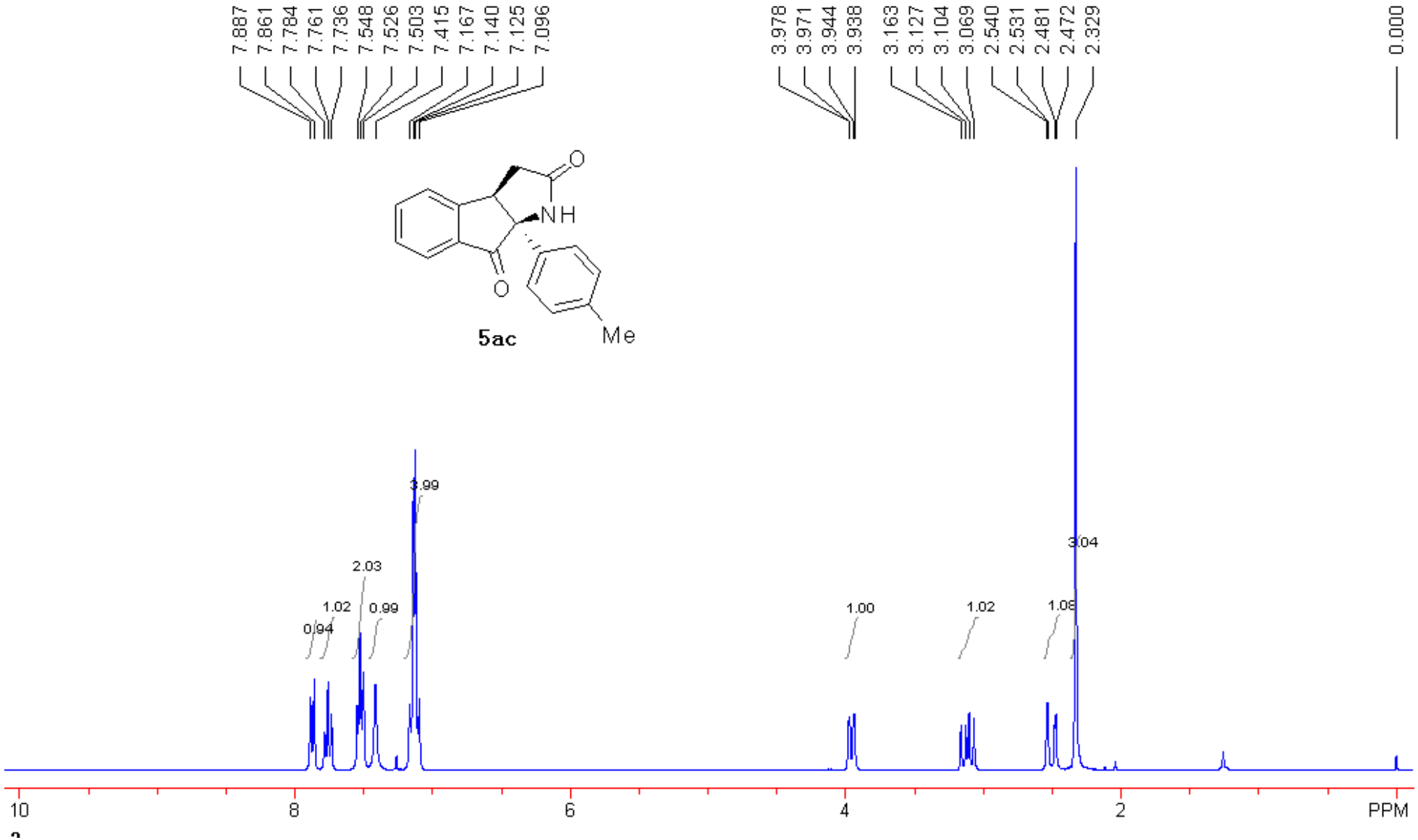
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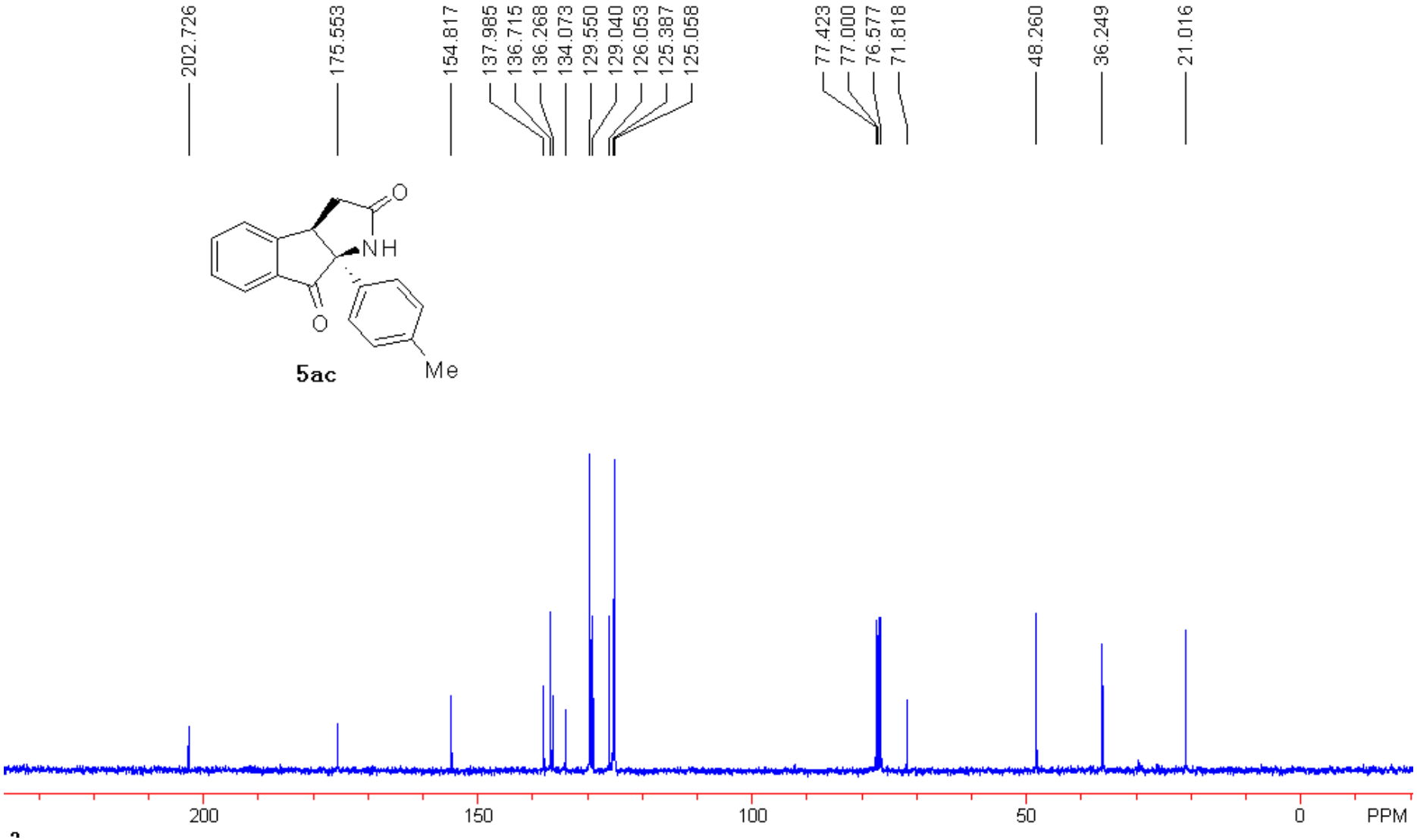
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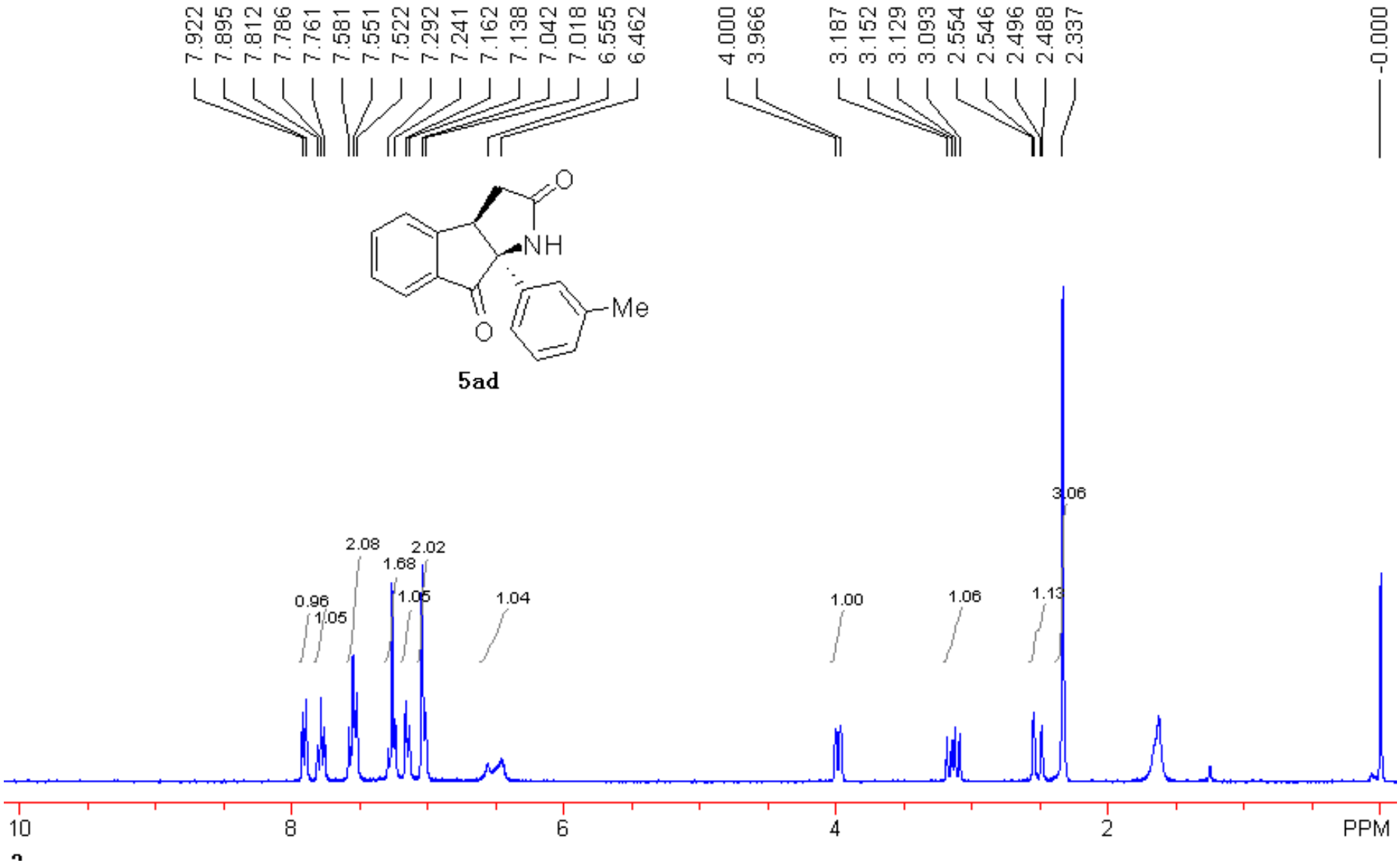
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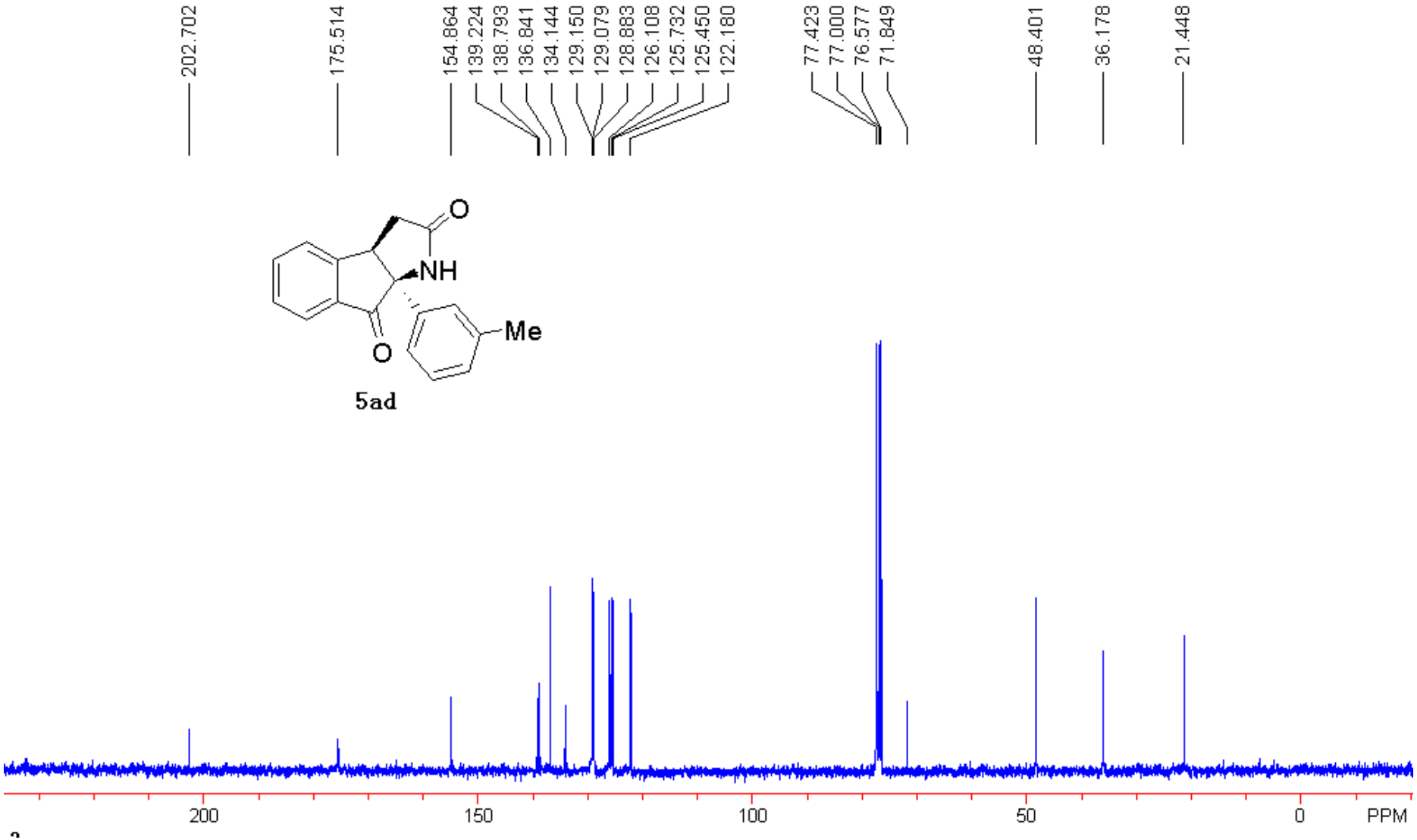
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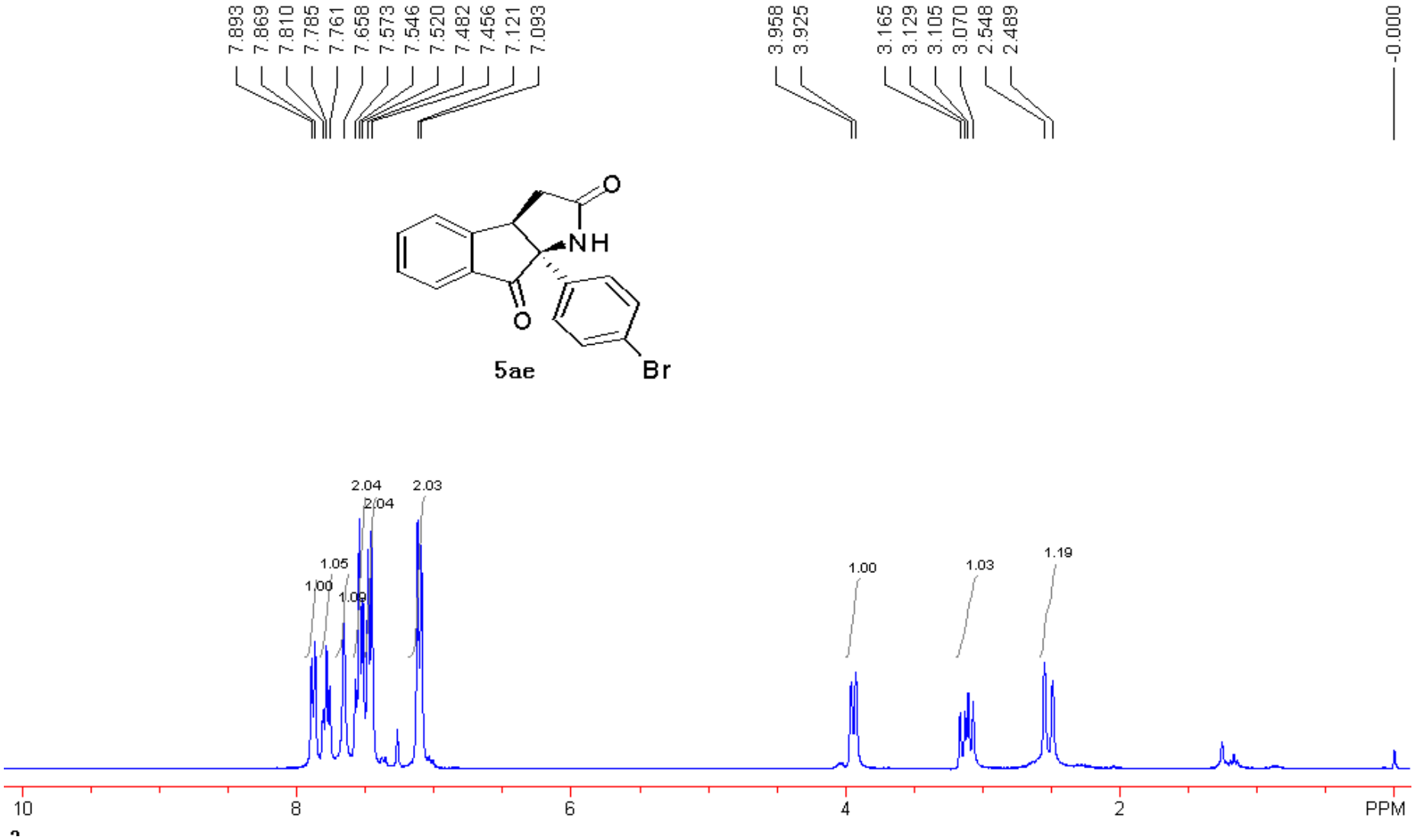
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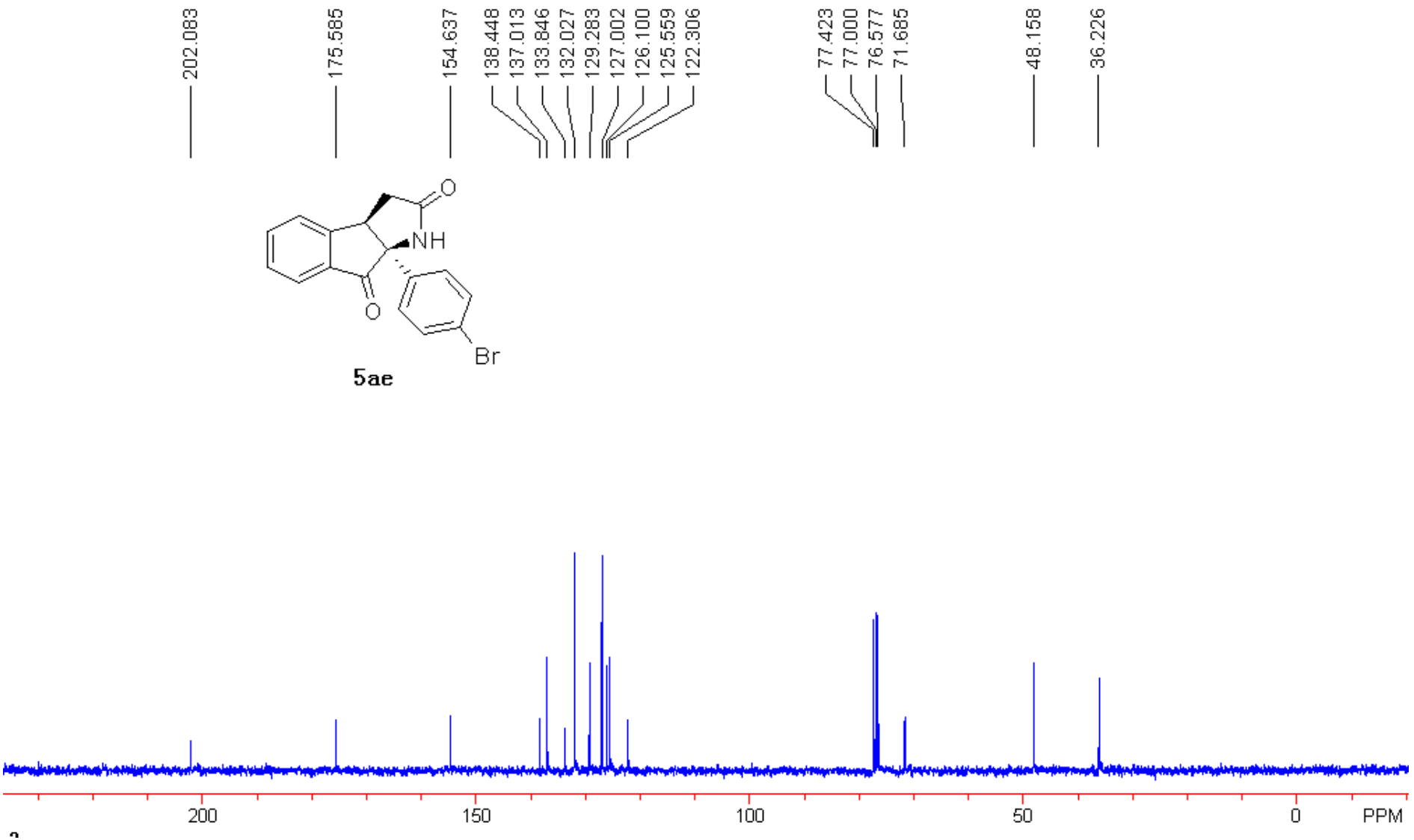
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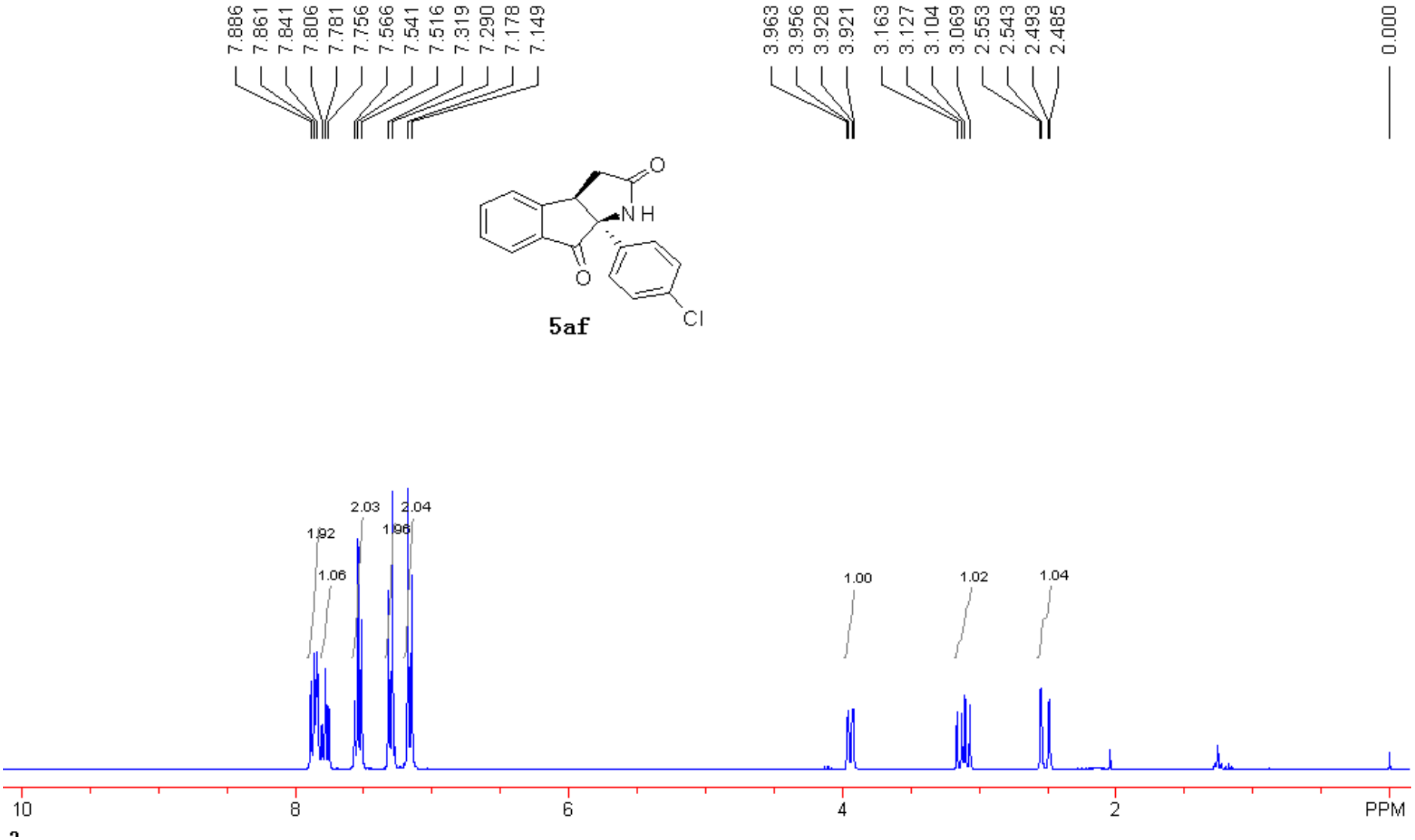
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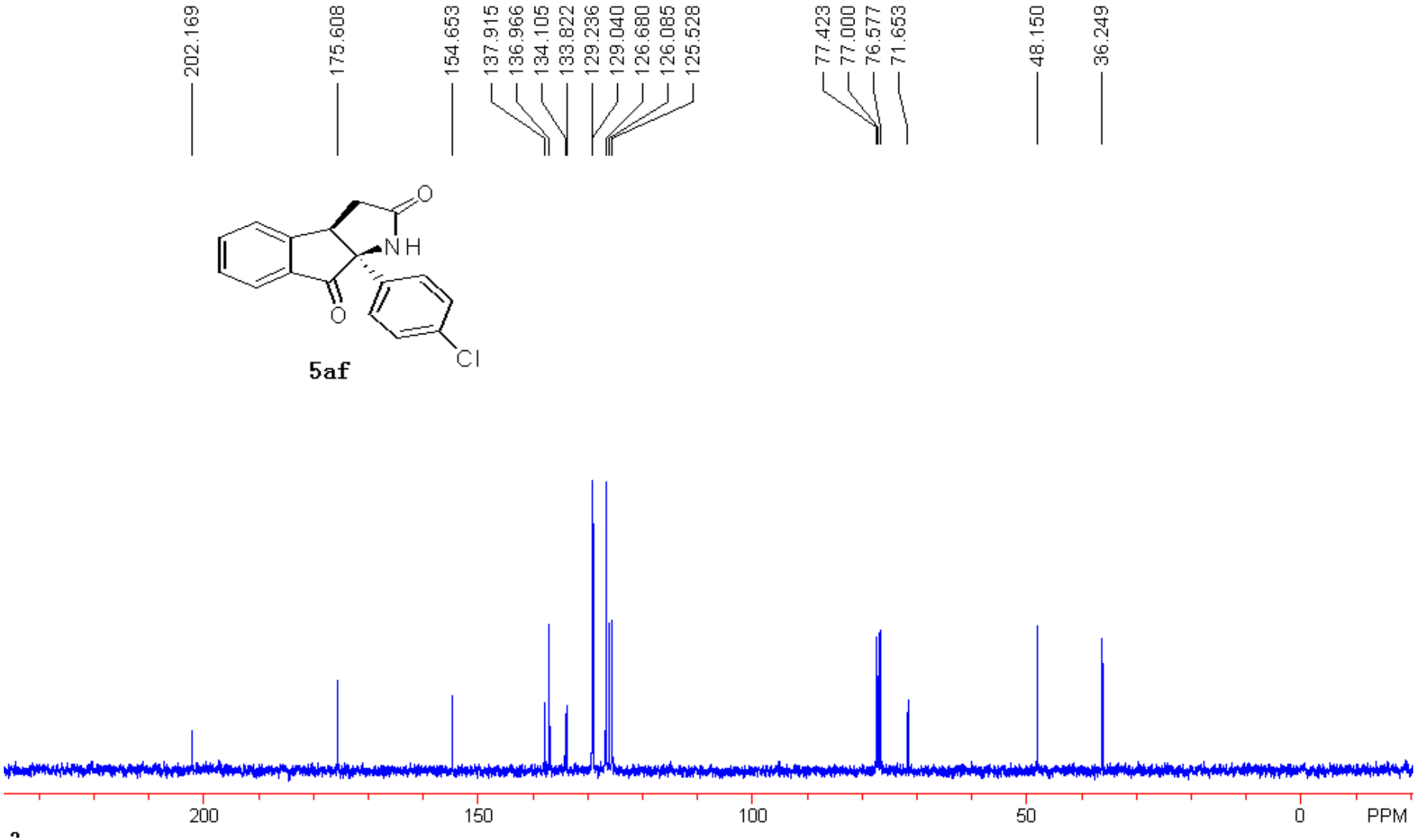
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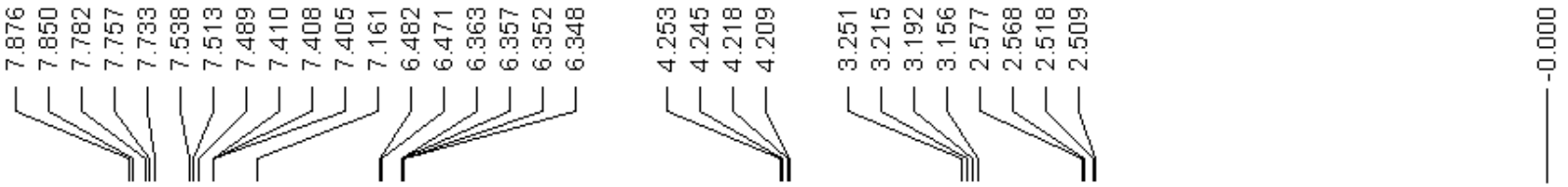
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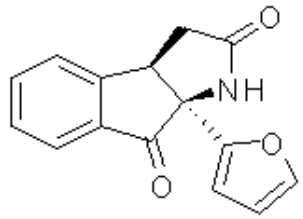
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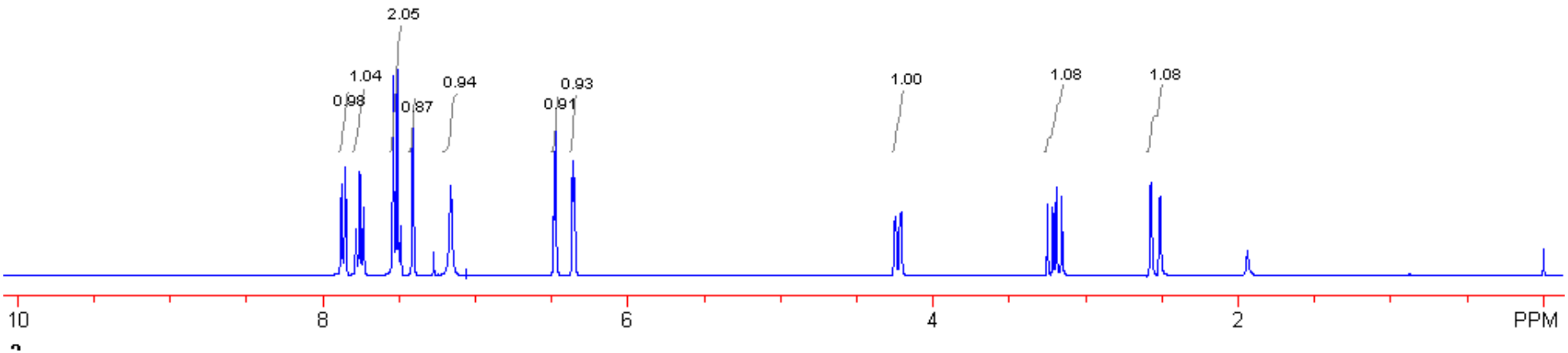
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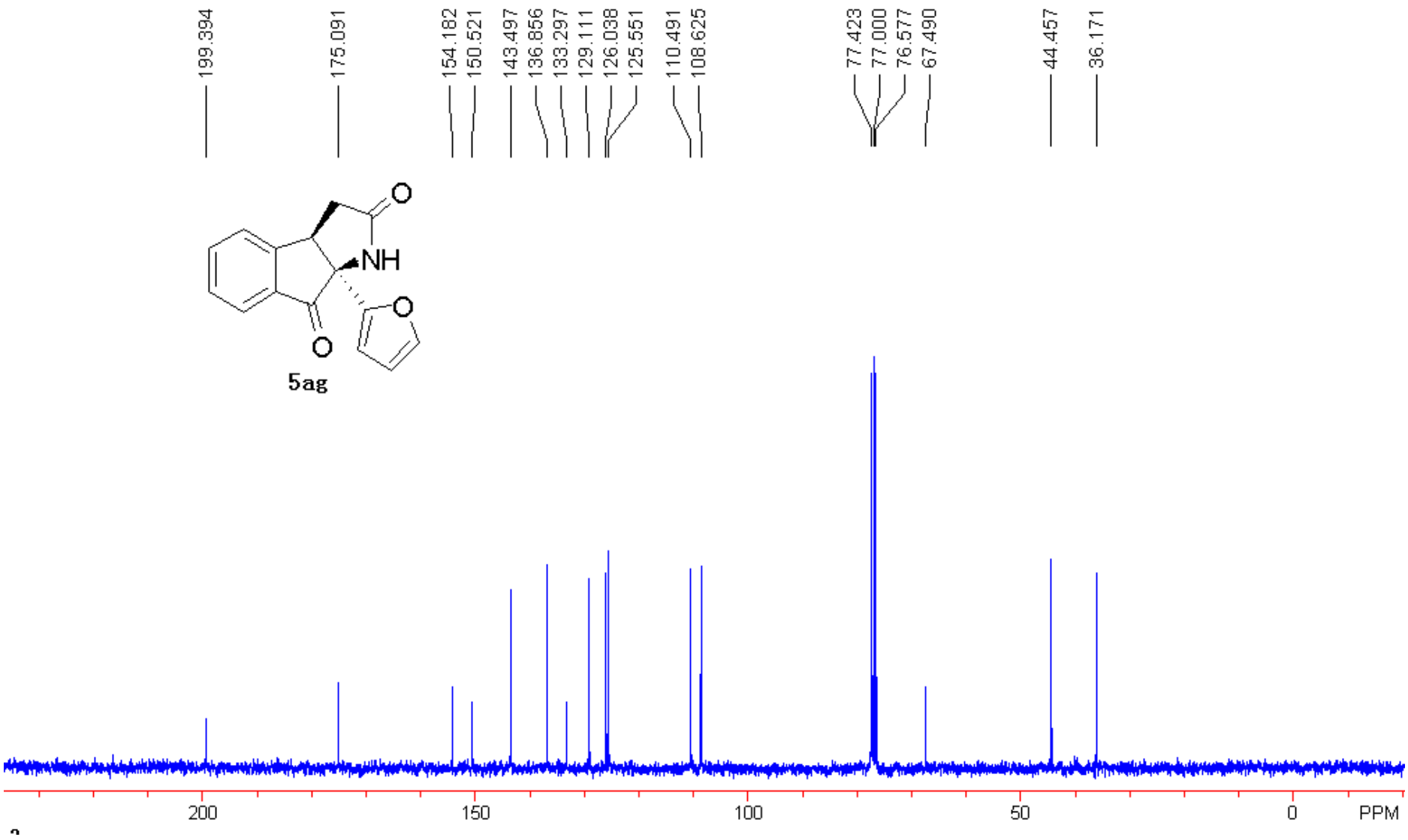
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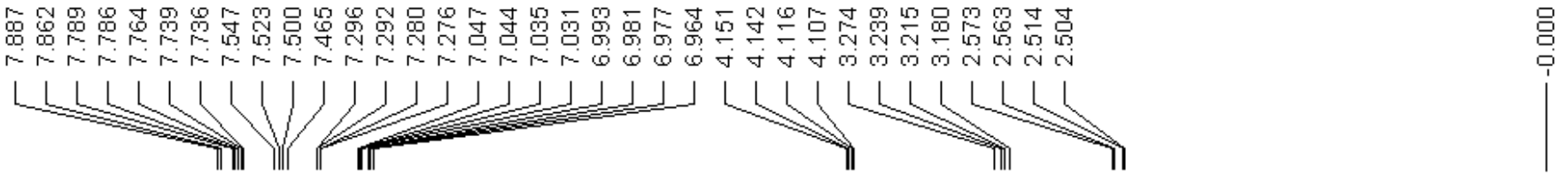
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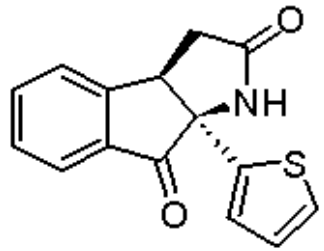
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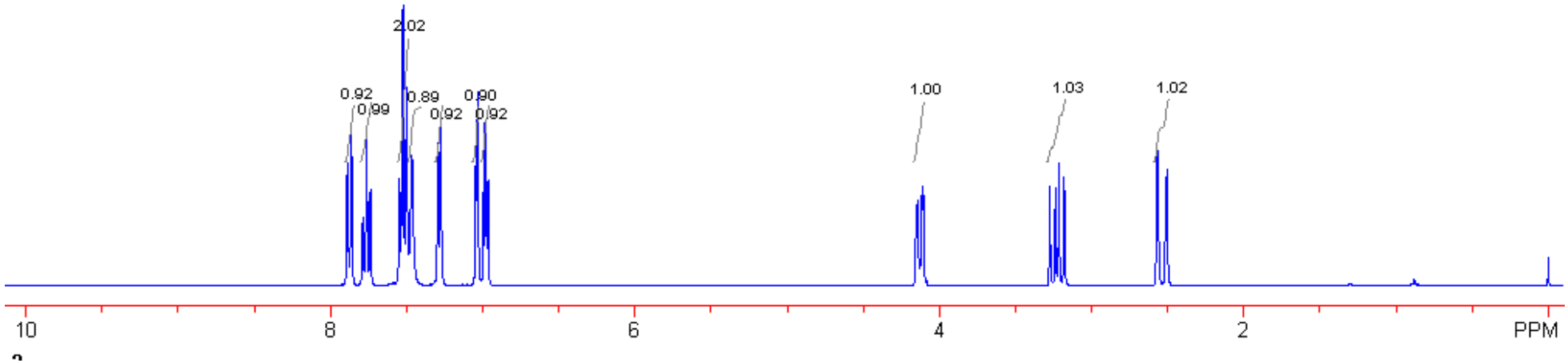
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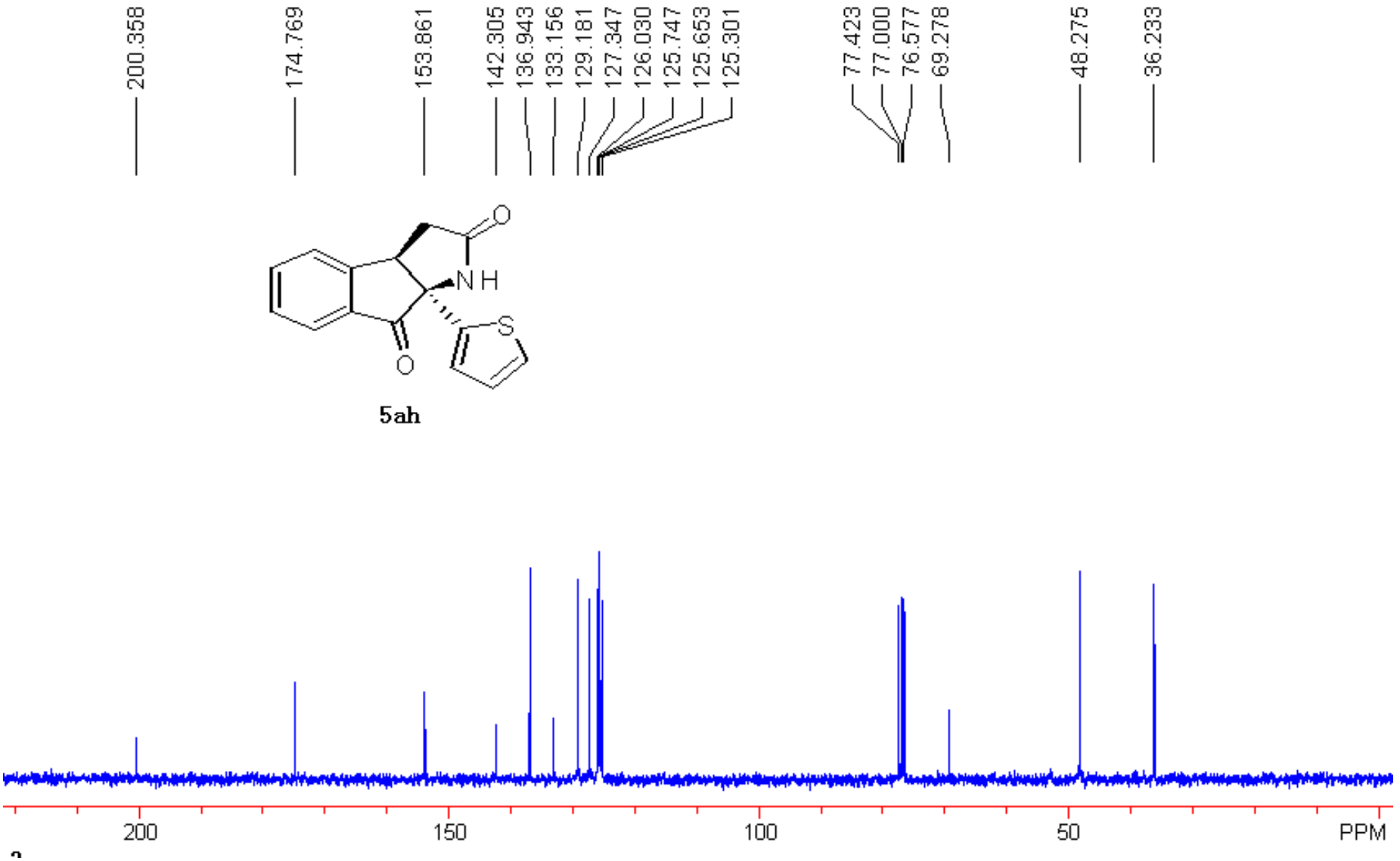
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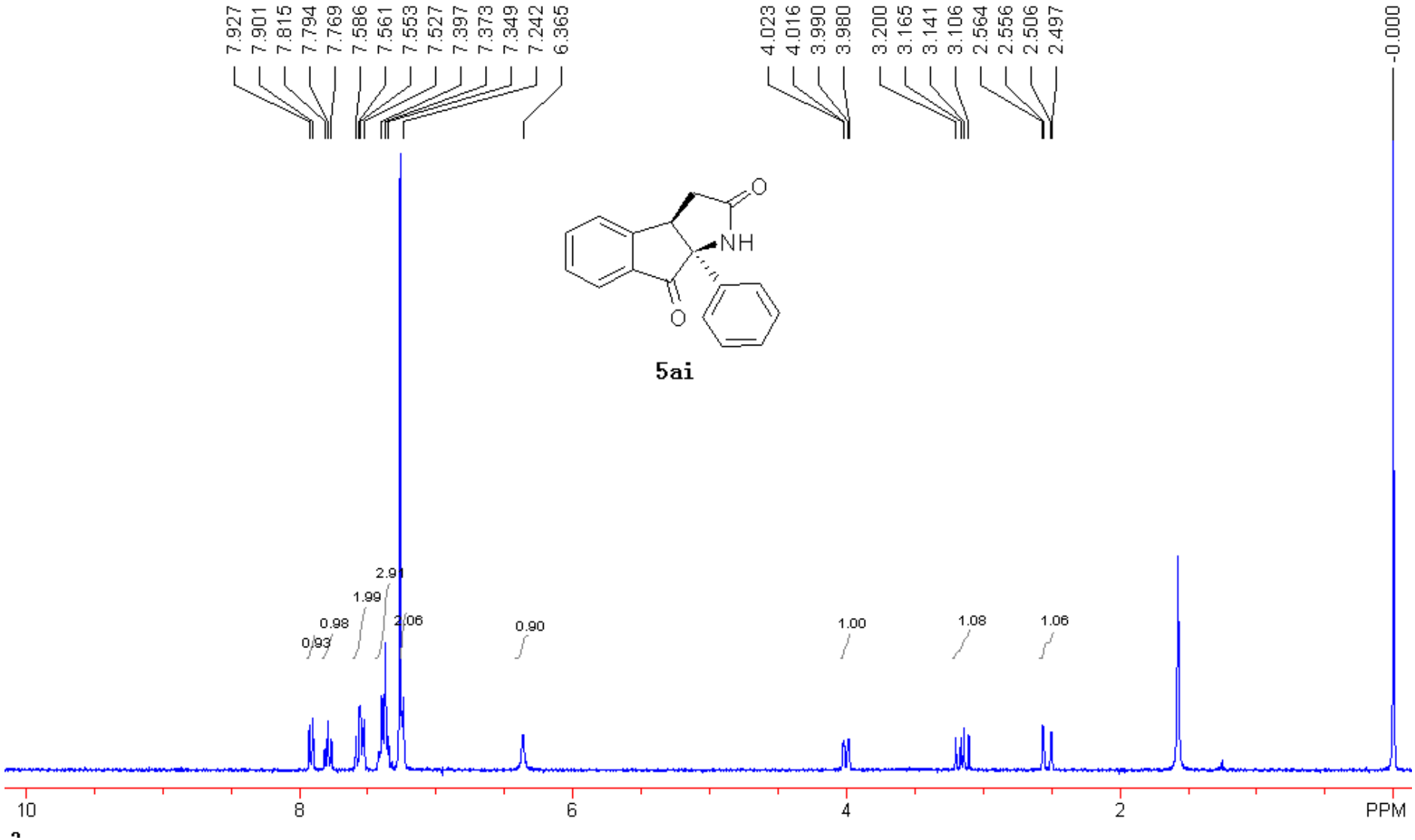
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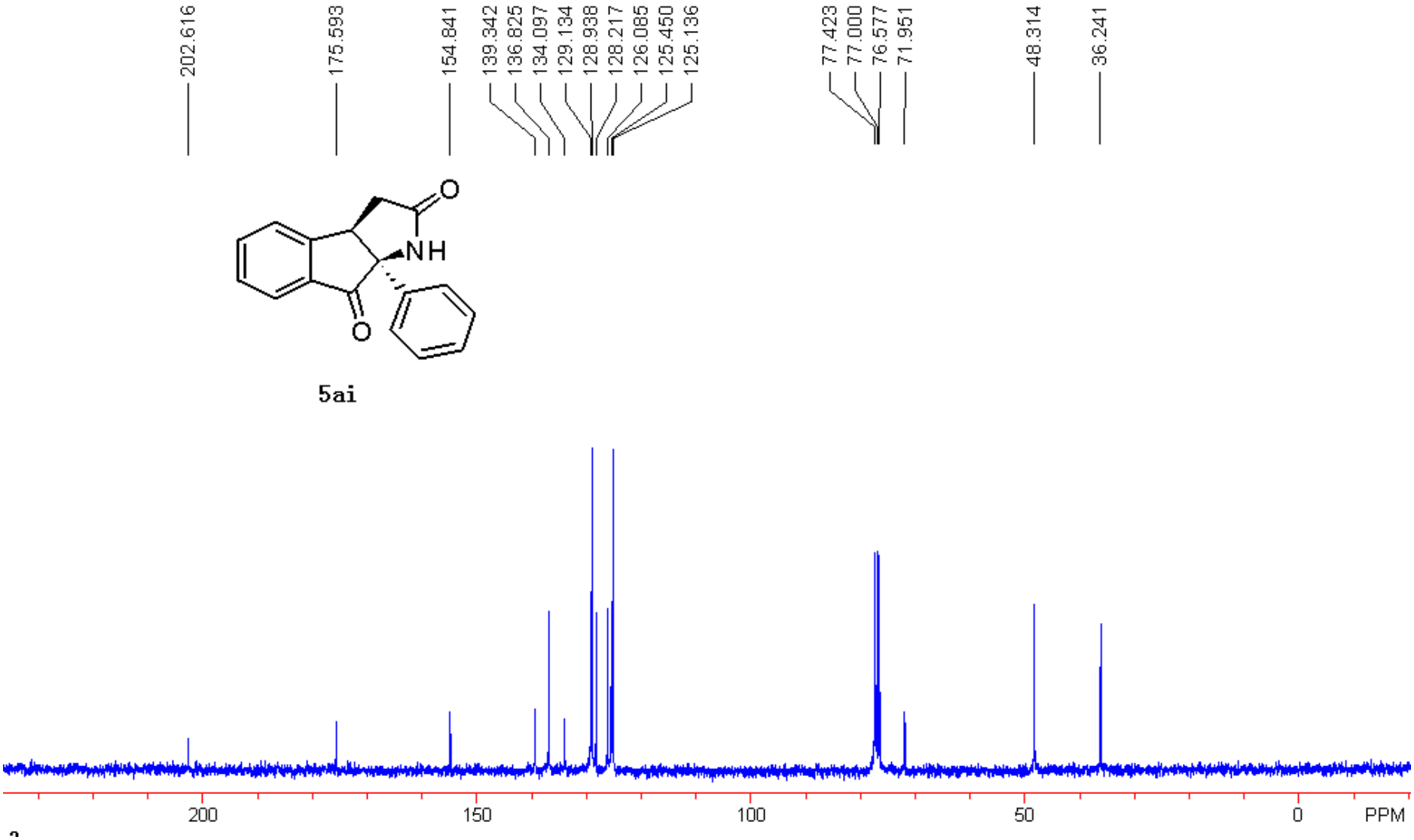
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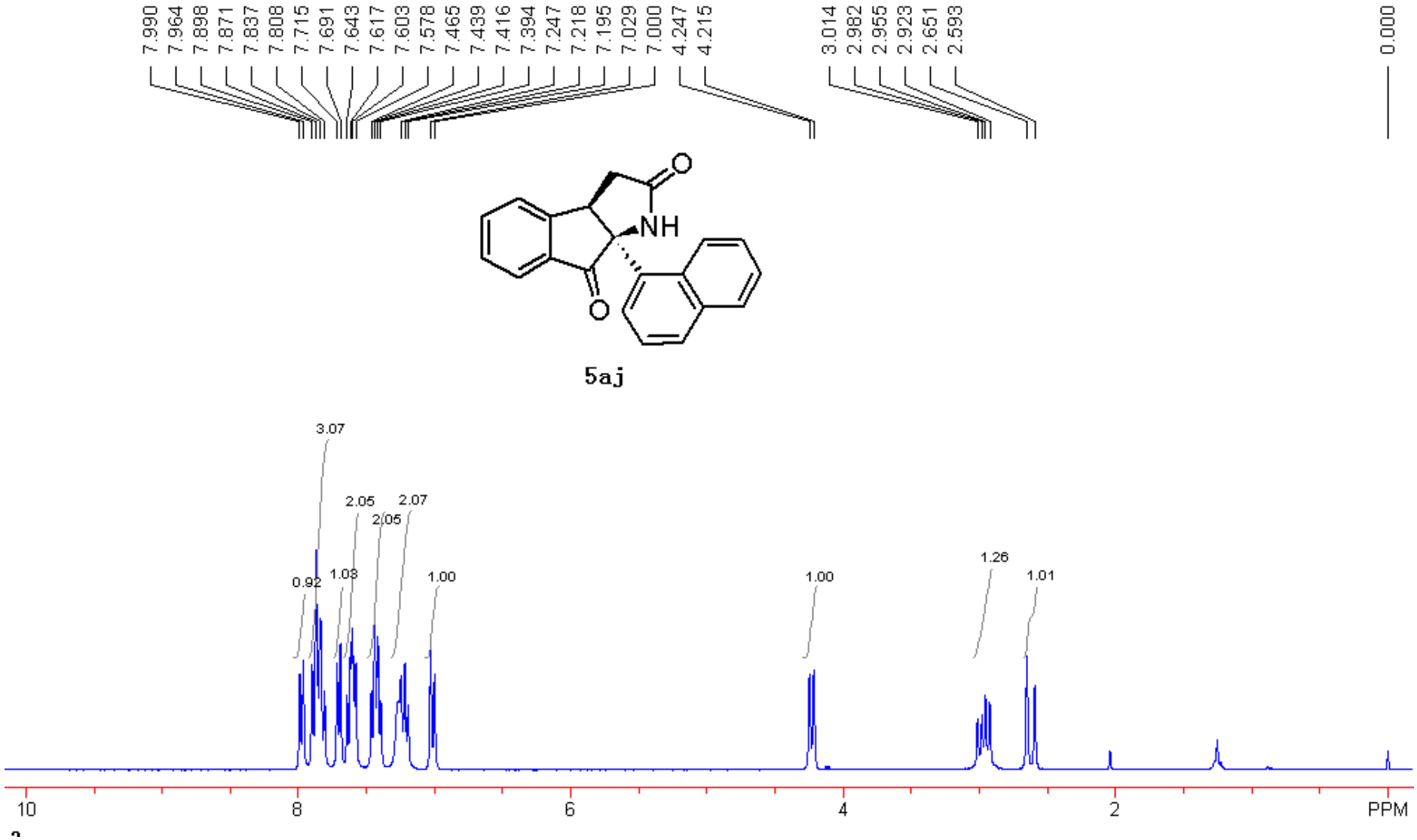
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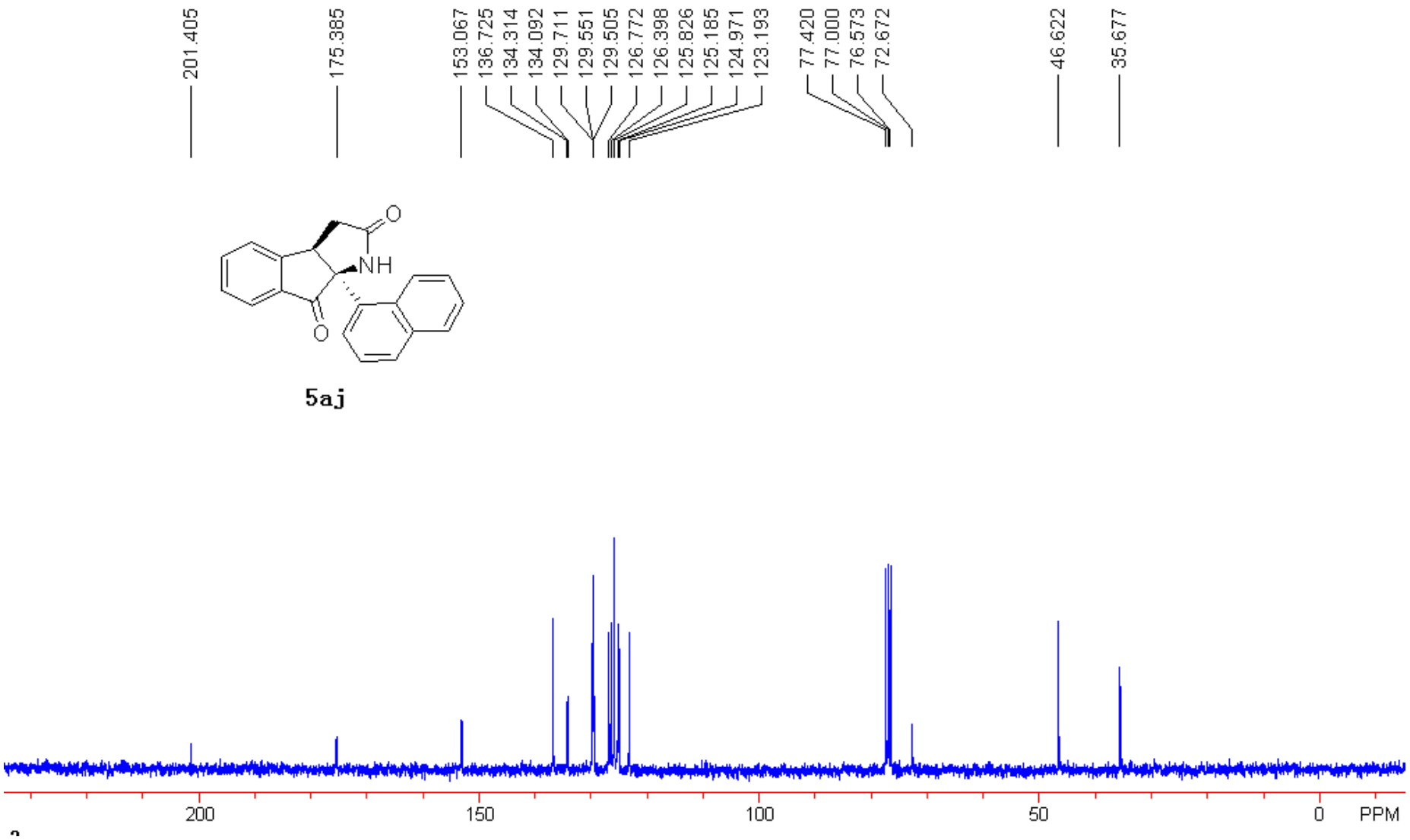
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125.826
125.185
124.971
123.193
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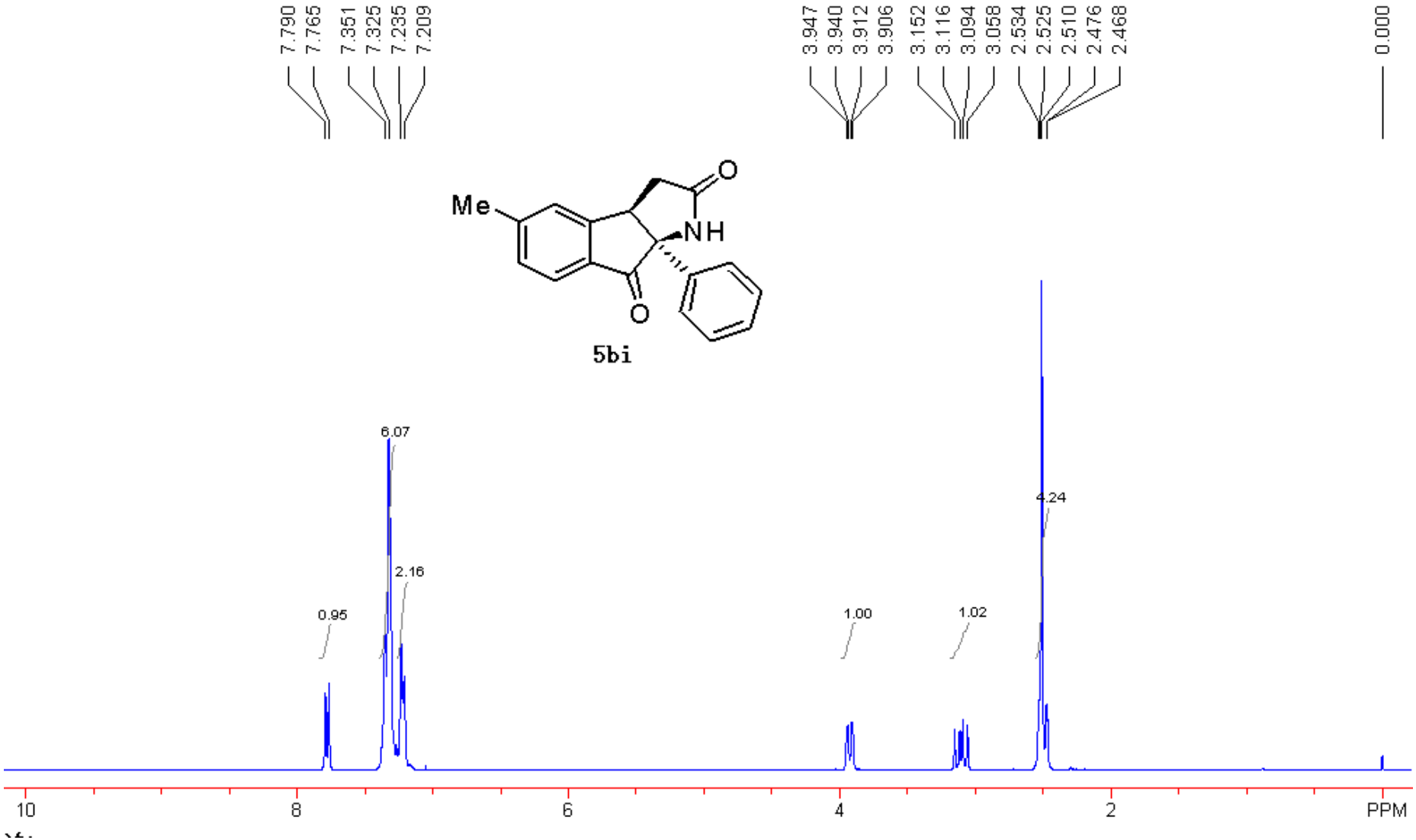
PPM

5aj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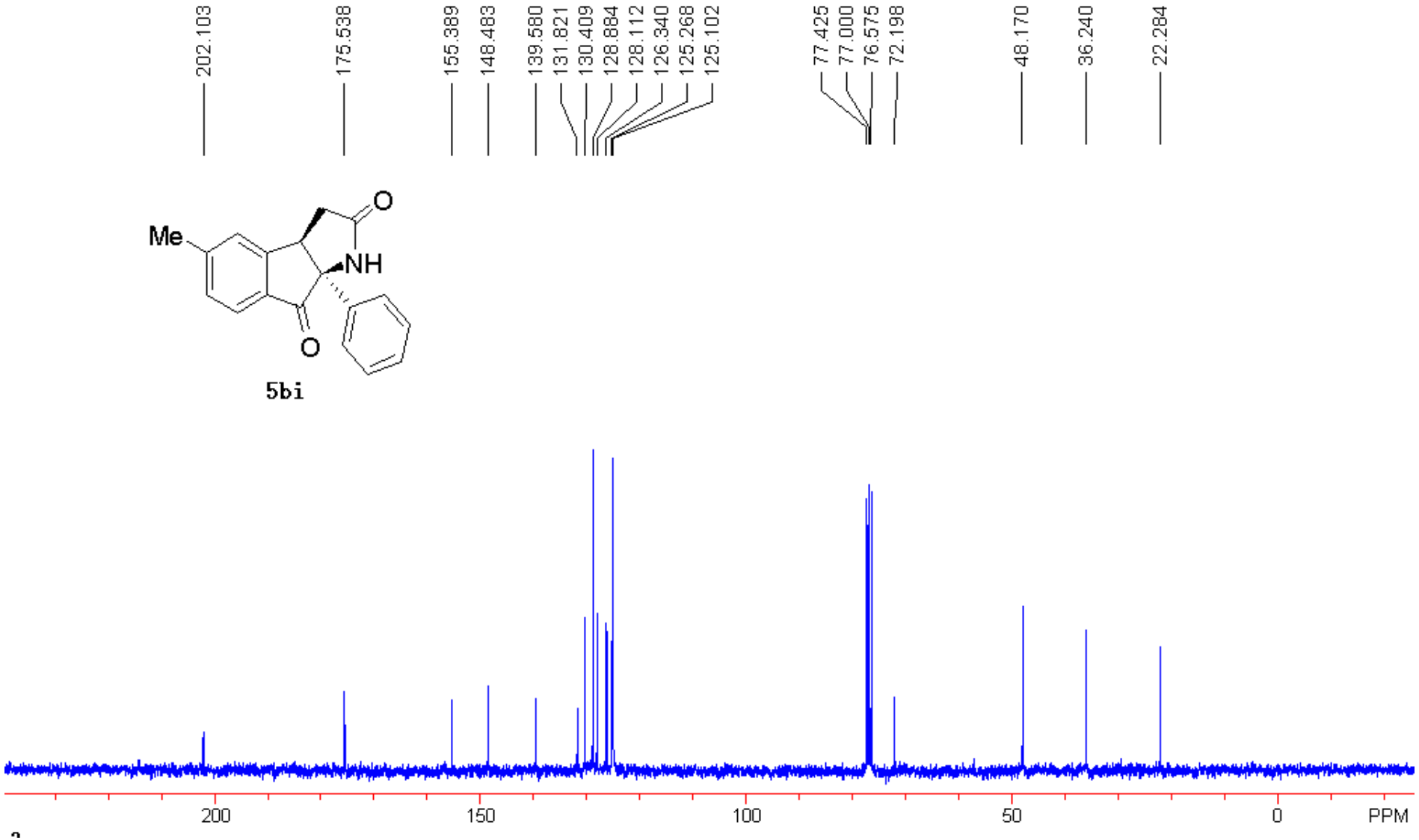
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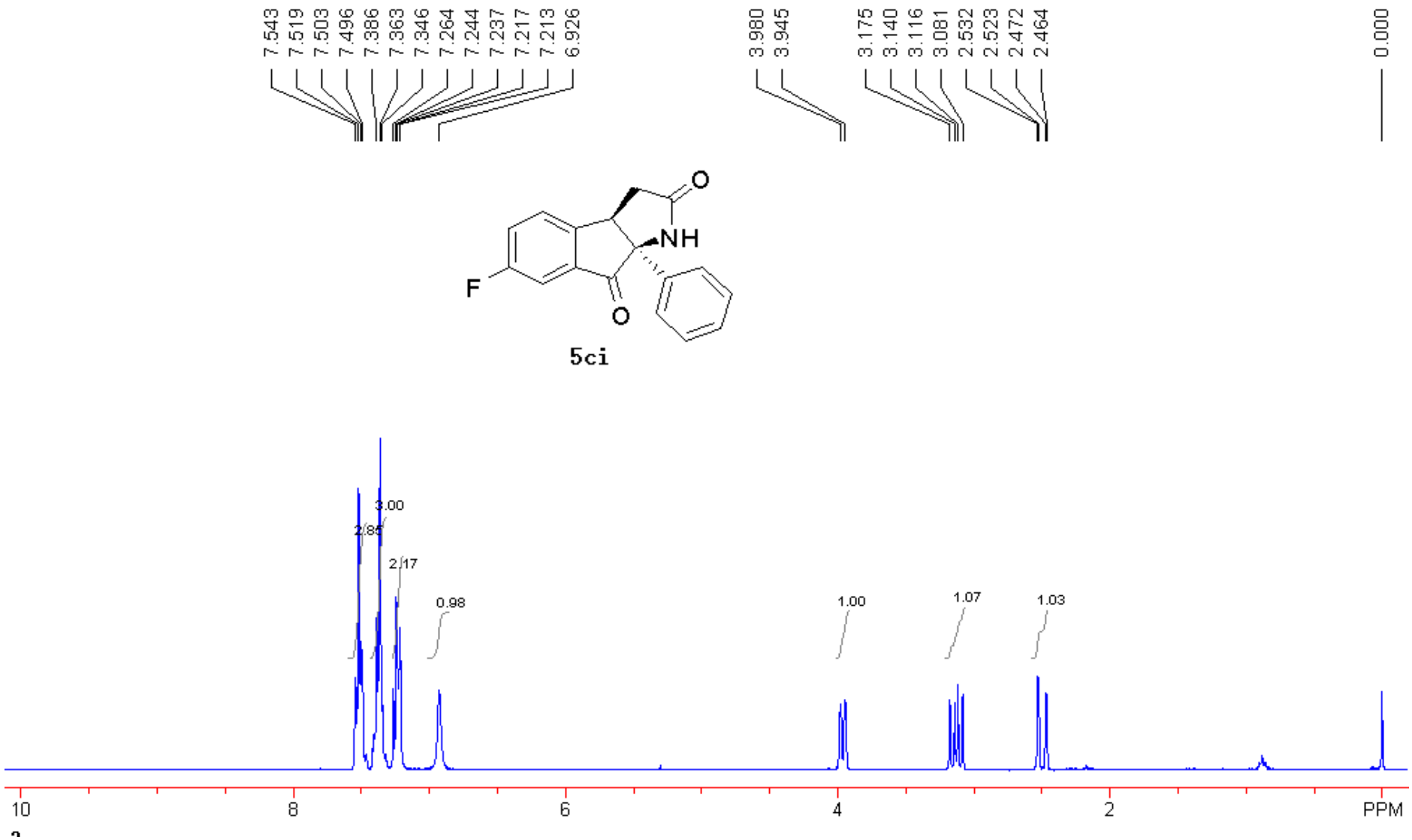
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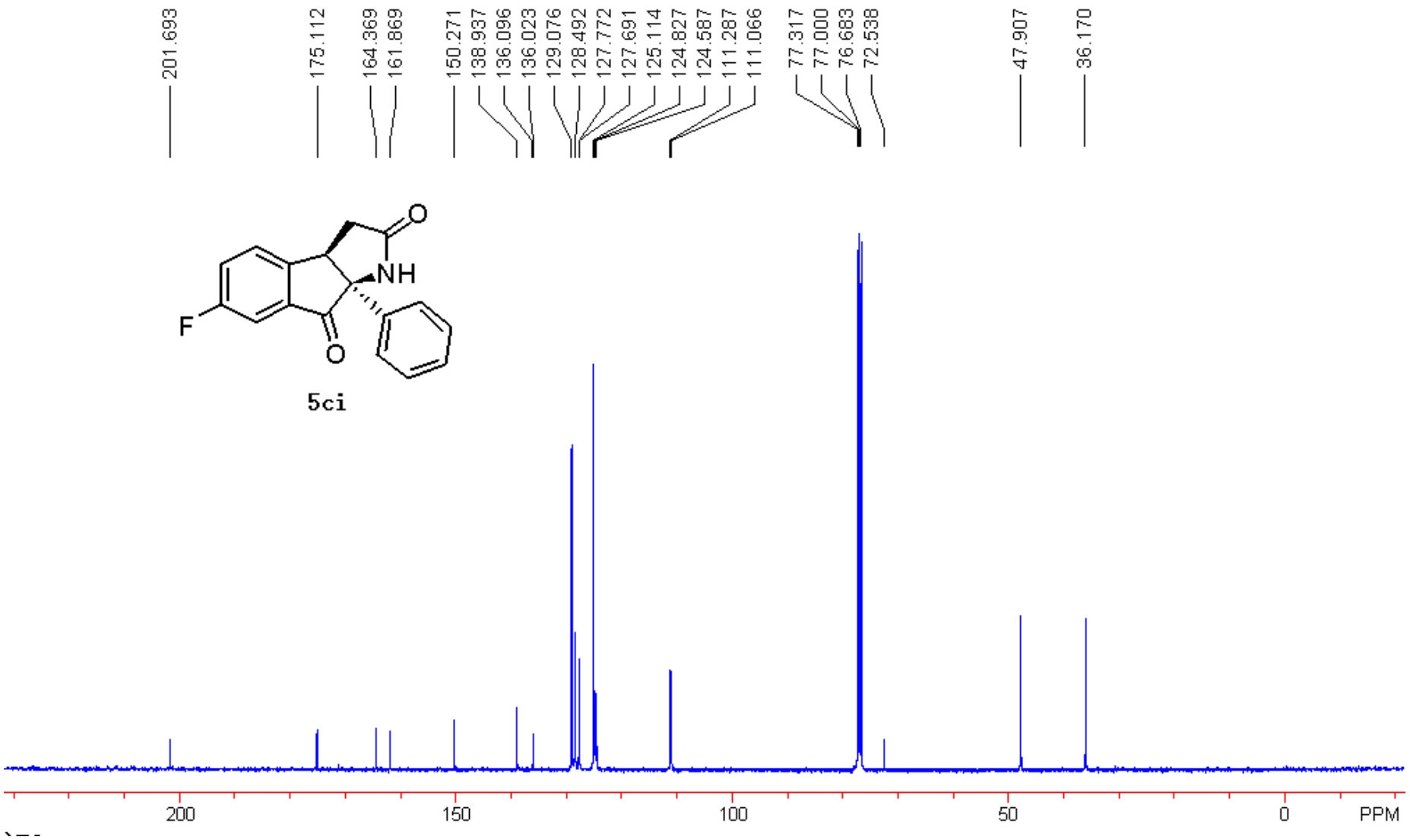
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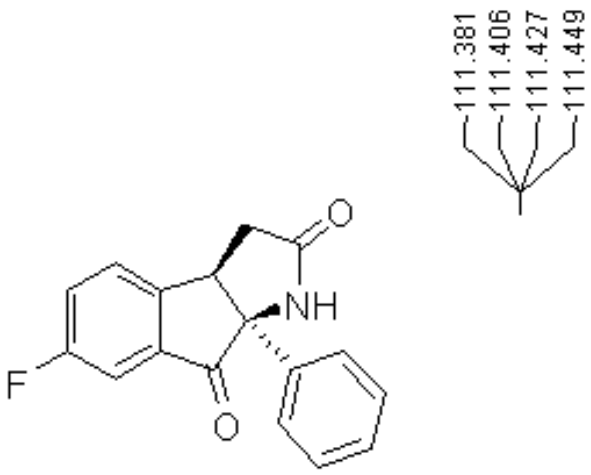
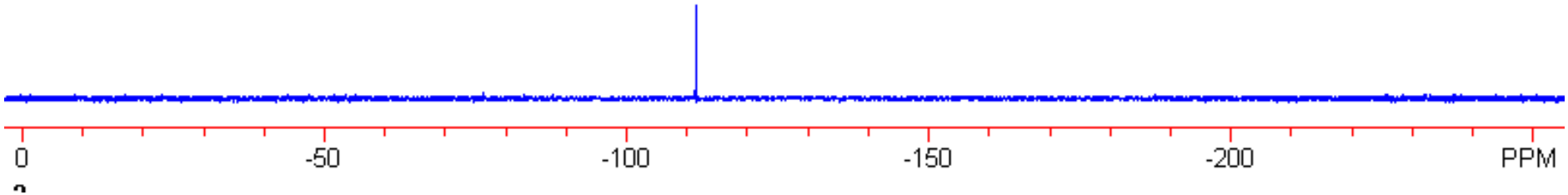
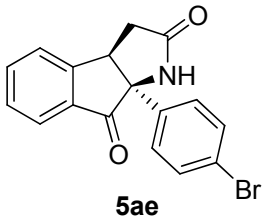


5ci



ORTEP Representation of **5ae**



5ae

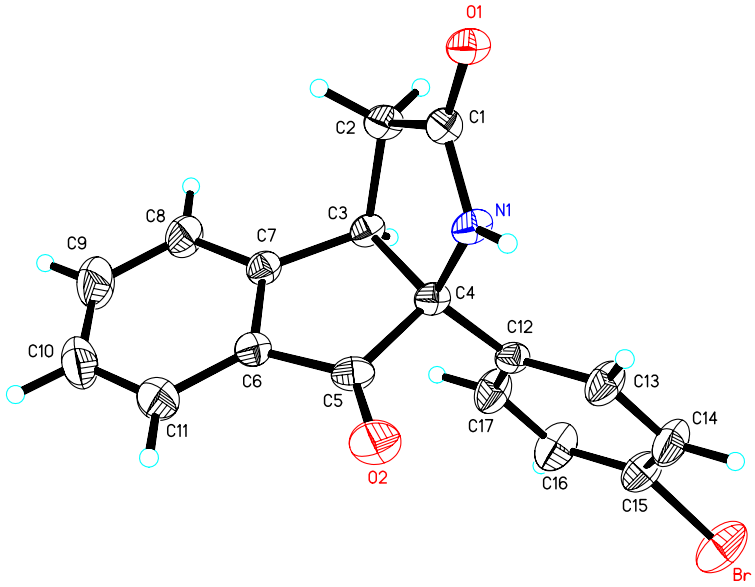


Table 1. Crystal data and structure refinement for cd29258.

Identification code	cd29258
Empirical formula	C17 H12 Br N O2
Formula weight	342.19
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 11.978(2) Å alpha = 90 deg.
	b = 11.514(2) Å beta = 113.623(3) deg.
	c = 12.103(2) Å gamma = 90 deg.
Volume	1529.4(4) $Å^3$
Z, Calculated density	4, 1.486 Mg/m^3
Absorption coefficient	2.691 mm^{-1}
F(000)	688
Crystal size	0.452 x 0.380 x 0.287 mm
Theta range for data collection	2.02 to 27.50 deg.
Limiting indices	-12<=h<=15, -13<=k<=14, -13<=l<=15
Reflections collected / unique	9025 / 3484 [R(int) = 0.0528]
Completeness to theta = 27.50	99.1 %
Absorption correction	Empirical
Max. and min. transmission	1.0000 and 0.4856
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3484 / 1 / 195
Goodness-of-fit on F^2	0.853
Final R indices [I>2sigma(I)]	R1 = 0.0480, wR2 = 0.1108
R indices (all data)	R1 = 0.0901, wR2 = 0.1200
Extinction coefficient	0.0289(17)
Largest diff. peak and hole	0.548 and -0.541 e. $Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for cd29258. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Br	8852(1)	9007(1)	-2405(1)	89(1)
O(1)	5727(2)	4591(2)	1612(2)	40(1)
O(2)	4532(2)	8523(2)	-202(2)	63(1)
N(1)	5821(2)	6286(2)	687(2)	34(1)
C(1)	6137(2)	5574(3)	1632(2)	33(1)
C(2)	7065(3)	6161(2)	2708(3)	40(1)
C(3)	7157(2)	7410(2)	2317(2)	32(1)
C(4)	6376(2)	7429(2)	925(2)	33(1)
C(5)	5359(3)	8327(2)	756(3)	40(1)
C(6)	5578(3)	8827(2)	1931(3)	40(1)
C(7)	6582(3)	8303(2)	2819(3)	36(1)
C(8)	6940(3)	8650(3)	4000(3)	57(1)
C(9)	6304(4)	9520(4)	4267(4)	83(1)
C(10)	5289(4)	10031(4)	3372(4)	83(1)
C(11)	4917(3)	9690(3)	2201(3)	63(1)
C(12)	7035(2)	7740(2)	135(3)	34(1)
C(13)	6693(3)	7282(3)	-1005(3)	53(1)
C(14)	7232(3)	7646(3)	-1764(3)	62(1)
C(15)	8126(3)	8468(3)	-1370(3)	53(1)
C(16)	8486(3)	8932(3)	-246(3)	64(1)
C(17)	7939(3)	8558(3)	507(3)	54(1)

Table 3. Bond lengths [Å] and angles [deg] for cd29258.

Br-C(15)	1.893(3)
O(1)-C(1)	1.231(3)
O(2)-C(5)	1.207(3)
N(1)-C(1)	1.332(4)
N(1)-C(4)	1.449(3)
N(1)-H(1)	0.846(17)
C(1)-C(2)	1.492(4)
C(2)-C(3)	1.532(4)
C(2)-H(2A)	0.9700
C(2)-H(2B)	0.9700
C(3)-C(7)	1.495(4)
C(3)-C(4)	1.566(4)
C(3)-H(3)	0.9800
C(4)-C(12)	1.507(4)
C(4)-C(5)	1.547(4)
C(5)-C(6)	1.458(4)
C(6)-C(11)	1.388(4)
C(6)-C(7)	1.390(4)
C(7)-C(8)	1.377(4)
C(8)-C(9)	1.373(5)
C(8)-H(8)	0.9300
C(9)-C(10)	1.394(5)
C(9)-H(9)	0.9300
C(10)-C(11)	1.362(5)
C(10)-H(10)	0.9300
C(11)-H(11)	0.9300
C(12)-C(17)	1.368(4)
C(12)-C(13)	1.378(4)
C(13)-C(14)	1.382(4)
C(13)-H(13)	0.9300
C(14)-C(15)	1.364(5)
C(14)-H(14)	0.9300
C(15)-C(16)	1.361(5)
C(16)-C(17)	1.387(4)
C(16)-H(16)	0.9300
C(17)-H(17)	0.9300

C(1)-N(1)-C(4)	116.0(2)
C(1)-N(1)-H(1)	122.4(19)
C(4)-N(1)-H(1)	121.6(19)
O(1)-C(1)-N(1)	125.1(2)
O(1)-C(1)-C(2)	125.8(3)
N(1)-C(1)-C(2)	109.1(2)

C(1)-C(2)-C(3)	106.0(2)
C(1)-C(2)-H(2A)	110.5
C(3)-C(2)-H(2A)	110.5
C(1)-C(2)-H(2B)	110.5
C(3)-C(2)-H(2B)	110.5
H(2A)-C(2)-H(2B)	108.7
C(7)-C(3)-C(2)	114.7(2)
C(7)-C(3)-C(4)	104.7(2)
C(2)-C(3)-C(4)	105.0(2)
C(7)-C(3)-H(3)	110.7
C(2)-C(3)-H(3)	110.7
C(4)-C(3)-H(3)	110.7
N(1)-C(4)-C(12)	113.9(2)
N(1)-C(4)-C(5)	107.8(2)
C(12)-C(4)-C(5)	110.0(2)
N(1)-C(4)-C(3)	103.3(2)
C(12)-C(4)-C(3)	116.5(2)
C(5)-C(4)-C(3)	104.5(2)
O(2)-C(5)-C(6)	128.3(3)
O(2)-C(5)-C(4)	123.5(3)
C(6)-C(5)-C(4)	108.1(2)
C(11)-C(6)-C(7)	121.9(3)
C(11)-C(6)-C(5)	128.0(3)
C(7)-C(6)-C(5)	110.1(3)
C(8)-C(7)-C(6)	119.3(3)
C(8)-C(7)-C(3)	128.3(3)
C(6)-C(7)-C(3)	112.4(3)
C(9)-C(8)-C(7)	119.0(3)
C(9)-C(8)-H(8)	120.5
C(7)-C(8)-H(8)	120.5
C(8)-C(9)-C(10)	121.2(4)
C(8)-C(9)-H(9)	119.4
C(10)-C(9)-H(9)	119.4
C(11)-C(10)-C(9)	120.5(3)
C(11)-C(10)-H(10)	119.8
C(9)-C(10)-H(10)	119.8
C(10)-C(11)-C(6)	118.1(3)
C(10)-C(11)-H(11)	120.9
C(6)-C(11)-H(11)	120.9
C(17)-C(12)-C(13)	118.1(3)
C(17)-C(12)-C(4)	120.1(3)
C(13)-C(12)-C(4)	121.6(3)
C(12)-C(13)-C(14)	121.3(3)
C(12)-C(13)-H(13)	119.4
C(14)-C(13)-H(13)	119.4
C(15)-C(14)-C(13)	119.2(3)

C(15)-C(14)-H(14)	120.4
C(13)-C(14)-H(14)	120.4
C(16)-C(15)-C(14)	120.8(3)
C(16)-C(15)-Br	119.2(3)
C(14)-C(15)-Br	120.0(3)
C(15)-C(16)-C(17)	119.4(3)
C(15)-C(16)-H(16)	120.3
C(17)-C(16)-H(16)	120.3
C(12)-C(17)-C(16)	121.2(3)
C(12)-C(17)-H(17)	119.4
C(16)-C(17)-H(17)	119.4

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for cd29258. The anisotropic displacement factor exponent takes the form: $-2\ pi^2\ [\ h^2\ a^{*2}\ U11 + ... + 2\ h\ k\ a^*\ b^*\ U12\ ]$

	U11	U22	U33	U23	U13	U12
Br	79(1)	137(1)	66(1)	17(1)	45(1)	-12(1)
O(1)	43(1)	37(1)	27(1)	0(1)	1(1)	-9(1)
O(2)	51(1)	77(2)	38(1)	5(1)	-7(1)	18(1)
N(1)	34(1)	38(1)	17(1)	-2(1)	-4(1)	-12(1)
C(1)	27(2)	38(2)	27(2)	-2(1)	2(1)	-1(1)
C(2)	41(2)	40(2)	24(2)	-1(1)	-3(1)	0(1)
C(3)	26(2)	40(2)	22(1)	-2(1)	1(1)	-5(1)
C(4)	31(2)	33(2)	24(2)	-2(1)	1(1)	-5(1)
C(5)	33(2)	42(2)	36(2)	7(1)	5(1)	-1(1)
C(6)	42(2)	40(2)	38(2)	4(2)	16(2)	3(1)
C(7)	38(2)	38(2)	30(2)	-1(1)	11(1)	-5(1)
C(8)	66(2)	69(2)	29(2)	-5(2)	12(2)	8(2)
C(9)	111(4)	92(3)	48(2)	-15(2)	35(3)	21(3)
C(10)	103(3)	91(3)	65(3)	-4(2)	43(3)	39(3)
C(11)	64(2)	69(2)	58(2)	10(2)	26(2)	22(2)
C(12)	33(2)	34(2)	27(2)	1(1)	5(1)	-2(1)
C(13)	62(2)	59(2)	33(2)	-10(2)	16(2)	-22(2)
C(14)	79(3)	74(2)	35(2)	-10(2)	25(2)	-14(2)
C(15)	52(2)	68(2)	45(2)	5(2)	25(2)	-11(2)
C(16)	59(2)	80(3)	54(2)	-11(2)	25(2)	-33(2)
C(17)	60(2)	61(2)	39(2)	-15(2)	19(2)	-24(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for cd29258.

	x	y	z	U(eq)
H(2A)	7846	5771	2958	48
H(2B)	6811	6153	3375	48
H(3)	8007	7613	2495	39
H(8)	7603	8301	4608	68
H(9)	6555	9773	5060	99
H(10)	4863	10609	3575	100
H(11)	4239	10025	1599	75
H(13)	6088	6717	-1269	63
H(14)	6988	7334	-2534	74
H(16)	9093	9495	15	76
H(17)	8192	8869	1279	65
H(1)	5330(20)	6090(20)	-11(18)	41(9)

Table 6. Torsion angles [deg] for cd29258.

C(4)-N(1)-C(1)-O(1)	177.2(3)
C(4)-N(1)-C(1)-C(2)	-2.9(3)
O(1)-C(1)-C(2)-C(3)	-173.2(3)
N(1)-C(1)-C(2)-C(3)	6.9(3)
C(1)-C(2)-C(3)-C(7)	106.4(3)
C(1)-C(2)-C(3)-C(4)	-7.9(3)
C(1)-N(1)-C(4)-C(12)	125.1(3)
C(1)-N(1)-C(4)-C(5)	-112.5(3)
C(1)-N(1)-C(4)-C(3)	-2.3(3)
C(7)-C(3)-C(4)-N(1)	-114.9(2)
C(2)-C(3)-C(4)-N(1)	6.2(3)
C(7)-C(3)-C(4)-C(12)	119.4(2)
C(2)-C(3)-C(4)-C(12)	-119.4(3)
C(7)-C(3)-C(4)-C(5)	-2.2(3)
C(2)-C(3)-C(4)-C(5)	119.0(2)
N(1)-C(4)-C(5)-O(2)	-65.9(4)
C(12)-C(4)-C(5)-O(2)	58.8(4)
C(3)-C(4)-C(5)-O(2)	-175.4(3)
N(1)-C(4)-C(5)-C(6)	112.5(2)
C(12)-C(4)-C(5)-C(6)	-122.8(2)
C(3)-C(4)-C(5)-C(6)	3.0(3)
O(2)-C(5)-C(6)-C(11)	-3.7(5)
C(4)-C(5)-C(6)-C(11)	178.0(3)
O(2)-C(5)-C(6)-C(7)	175.5(3)
C(4)-C(5)-C(6)-C(7)	-2.8(3)
C(11)-C(6)-C(7)-C(8)	-0.4(5)
C(5)-C(6)-C(7)-C(8)	-179.7(3)
C(11)-C(6)-C(7)-C(3)	-179.4(3)
C(5)-C(6)-C(7)-C(3)	1.3(3)
C(2)-C(3)-C(7)-C(8)	67.3(4)
C(4)-C(3)-C(7)-C(8)	-178.2(3)
C(2)-C(3)-C(7)-C(6)	-113.9(3)
C(4)-C(3)-C(7)-C(6)	0.6(3)
C(6)-C(7)-C(8)-C(9)	-1.0(5)
C(3)-C(7)-C(8)-C(9)	177.8(3)
C(7)-C(8)-C(9)-C(10)	1.8(6)
C(8)-C(9)-C(10)-C(11)	-1.2(7)
C(9)-C(10)-C(11)-C(6)	-0.2(6)
C(7)-C(6)-C(11)-C(10)	1.0(5)
C(5)-C(6)-C(11)-C(10)	-179.8(3)
N(1)-C(4)-C(12)-C(17)	-156.6(3)
C(5)-C(4)-C(12)-C(17)	82.2(3)

C(3)-C(4)-C(12)-C(17)	-36.5(4)
N(1)-C(4)-C(12)-C(13)	28.1(4)
C(5)-C(4)-C(12)-C(13)	-93.1(3)
C(3)-C(4)-C(12)-C(13)	148.3(3)
C(17)-C(12)-C(13)-C(14)	-0.9(5)
C(4)-C(12)-C(13)-C(14)	174.4(3)
C(12)-C(13)-C(14)-C(15)	0.5(5)
C(13)-C(14)-C(15)-C(16)	-0.2(6)
C(13)-C(14)-C(15)-Br	-178.5(3)
C(14)-C(15)-C(16)-C(17)	0.3(6)
Br-C(15)-C(16)-C(17)	178.6(3)
C(13)-C(12)-C(17)-C(16)	1.0(5)
C(4)-C(12)-C(17)-C(16)	-174.4(3)
C(15)-C(16)-C(17)-C(12)	-0.7(6)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd29258 [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(1)#1	0.846(17)	2.000(18)	2.840(3)	172(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z