Supplementary Information

Transition Metal-Free *N*-Arylation of Secondary Amides through Iodonium Salts as Aryne Precursors

Ming Wang,* Zhijian Huang
School of Chemistry and Molecular Engineering, East China Normal University, Shanghai 200062, P. R. China

General Information.

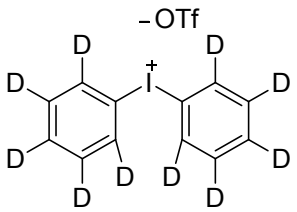
Commercially available materials purchased from Alfa Aesar or Aldrich was used as received. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a Bruker BBFO (400 MHz) spectrometer. Chemical shifts were recorded in parts per million (ppm, δ) relative to tetramethylsilane (δ 0.00) or chloroform (δ = 7.26, singlet). ^{1}H NMR splitting patterns are designated as singlet (s), doublet (d), triplet (t), quartet (q), dd (doublet of doublets); m (multiplets), and etc. All first-order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted are designated as multiplet (m) or broad (br). Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on a Bruker BBFO (400 MHz) spectrometer. GC and GC/MS analysis was conducted with Agilent J&W GC column DB-5MS-UI. High resolution mass spectral analysis (HRMS) was performed on Finnigan MAT 95 XP mass spectrometer (Thermo Electron Corporation). Analytical thin-layer chromatography (TLC) was carried out on Merck 60 F254 pre-coated silica gel plate (0.2 mm thickness).

Preparation of diaryliodonium salt substrates

The diaryliodonium salts (**2a**-**g**) were prepared and characterized adopting reported procedures[1-2] or purchased from Alfa Aesar (diphenyliodonium hexafluorophosphate).

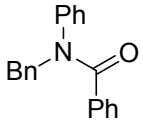
General Procedure for *N*-Arylation of Amides and Amines

To a suspension of *t*-BuOK (3.0 equiv, 672 mg, 0.6 mmol) (or 2.0 equiv) in THF (2.0 mL), was added secondary amides **1** (1.0 equiv, 0.2 mmol) at 0 °C (or RT; as specified in the manuscript). The resulting mixture was stirred at this temperature for 10 min. Diaryliodonium salt **2** (1.2 equiv, 0.24 mmol) was added in one portion and the reaction mixture was stirred at RT until TLC analysis indicated complete consumption of the amide substrate (**1**). The reaction was then quenched with H_2O at 0 °C, the organic phase was separated and the water phase was extracted with dichloromethane. The combined organic phases were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude materials were purified by SiO_2 flash chromatography to afford the product.



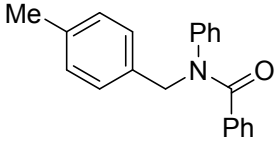
Diphenyliodonium triflate-d_{10} (2g) was prepared by adopting known procedures. [1-2] **^{13}C NMR** (100

MHz, DMSO) δ 135.5, 135.2, 135.0, 132.0, 131.7, 131.5, 122.8, 119.6, 116.6.



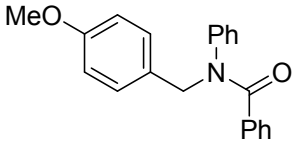
***N*-Benzyl-*N*-phenylbenzamide (3a)**[3]

White solid, 48.3 mg, 84% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38-7.04 (m, 13H), 6.95-6.82 (m, 2H), 5.13 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.6, 143.6, 137.6, 136.0, 129.7, 129.0, 128.8, 128.5, 128.4, 127.7, 127.4, 126.7, 53.9; **HRMS** for $C_{20}H_{18}NO$ $[M+1]^+$ Calculated: 288.1388, Found: 288.1383.



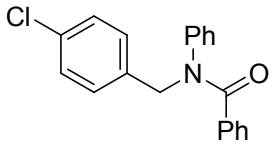
***N*-(4-Methylbenzyl)-*N*-phenylbenzamide (3b)**

White solid, 48.8 mg, 81% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40-7.28 (m, 2H), 7.24-7.02 (m, 10H), 6.90 (d, *J* = 7.2 Hz, 2H), 5.09 (s, 2H), 2.31 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.5, 143.6, 137.0, 136.1, 134.6, 129.6, 129.2, 129.0, 128.8, 128.4, 127.8, 127.7, 126.6, 53.6, 21.2; **HRMS** for $C_{21}H_{20}NO$ $[M+1]^+$ Calculated: 302.1545, Found: 302.1547.



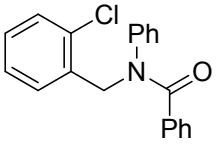
***N*-(4-Methoxybenzyl)-N-phenylbenzamide (3c)**[4]

White solid, 56.5 mg, 89% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38-7.33 (m, 2H), 7.28-7.09 (m, 8H), 6.92 (d, *J* = 7.2 Hz, 2H), 6.85 (d, *J* = 8.7 Hz, 2H), 5.10 (s, 2H), 3.81 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.5, 158.9, 143.5, 136.1, 129.9, 129.7, 129.6, 129.0, 128.8, 128.0, 127.7, 126.7, 113.8, 55.2, 53.3; **HRMS** for $C_{21}H_{20}NO_2$ $[M+1]^+$ Calculated: 318.1494, Found: 318.1497.



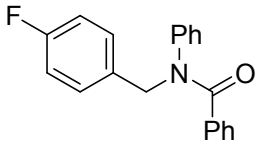
***N*-(4-Chlorobenzyl)-*N*-phenylbenzamide (3d)**

White solid, 49.6 mg, 77% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.36-7.29 (m, 2H), 7.28-7.19 (m, 5H), 7.18-7.06 (m, 5H), 6.88 (d, *J* = 7.2 Hz, 2H), 5.09 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.6, 143.3, 136.0, 135.7, 133.2, 130.0, 129.8, 129.1, 128.8, 128.6, 127.8, 127.7, 126.8, 53.3; **HRMS** for $C_{20}H_{17}ClNO$ $[M+1]^+$ Calculated: 322.0999, Found: 322.1007.



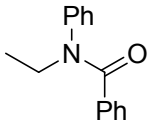
***N*-(2-Chlorobenzyl)-*N*-phenylbenzamide (3e)**[5]

Colorless oil, 47.6 mg, 74% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53-7.45 (m, 1H), 7.42-7.29 (m, 3H), 7.28-7.04 (m, 8H), 7.00-6.92 (m, 2H), 5.28 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.7, 143.4, 135.8, 134.9, 133.4, 129.8, 129.6, 129.3, 129.0, 128.8, 128.5, 127.8, 127.4, 127.0, 126.8, 51.2; **HRMS** for $C_{20}H_{17}ClNO$ $[M+1]^+$ Calculated: 322.0999, Found: 322.1007.



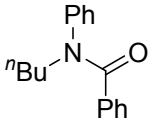
***N*-(4-Fluorobenzyl)-*N*-phenylbenzamide (3f)**

White solid, 50.1 mg, 82% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41-7.04 (m, 10H), 7.01-6.92 (m, 2H), 6.91-6.85 (m, 2H), 5.09 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.6, 162.2 (J = 245.6 Hz), 143.3, 135.8, 133.3 (J = 3.2 Hz), 130.3 (J = 8.1 Hz), 129.7, 129.1, 128.8, 127.8, 127.7, 126.8, 115.3 (J = 21.4 Hz), 53.2; **HRMS** for $C_{20}H_{17}FNO$ $[M+1]^+$ Calculated: 306.1294, Found: 306.1293.



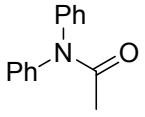
***N*-Ethyl-*N*-phenylbenzamide (3g)**[6]

Colorless oil, 28.4 mg, 63% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33-7.11 (m, 8H), 7.02 (d, J = 7.5 Hz, 2H), 3.99 (q, J = 7.1 Hz, 2H), 1.23 (t, J = 7.1 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.2, 143.3, 136.4, 129.4, 129.1, 128.7, 128.0, 127.7, 126.6, 45.4, 13.0; **HRMS** for $C_{15}H_{16}NO$ $[M+1]^+$ Calculated: 226.1232, Found: 226.1234.



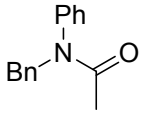
***N*-Butyl-*N*-phenylbenzamide (3h)**[7]

White solid, 32.9 mg, 65% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.29-7.10 (m, 8H), 7.02 (d, J = 7.4 Hz, 2H), 3.69-3.80 (m, 2H), 1.70-1.49 (m, 2H), 1.43-1.31 (m, 2H), 0.91 (t, J = 7.4 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.3, 143.6, 136.5, 129.4, 129.1, 128.6, 127.9, 127.7, 126.6, 50.3, 29.8, 20.2, 13.9; **HRMS** for $C_{17}H_{20}NO$ $[M+1]^+$ Calculated: 254.1545, Found: 254.1549.



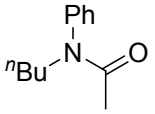
***N*,*N*-Diphenylacetanilide (3i)**[3]

White solid, 28.7 mg, 68% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.48-7.09 (m, 10H), 2.06 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.5, 23.9; **HRMS** for $C_{14}H_{14}NO$ $[M+1]^+$ Calculated: 212.1075, Found: 212.1083.



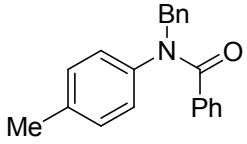
***N*-Benzyl-*N*-phenylacetamide (3j)**[8]

Colorless oil, 27.0 mg, 60% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.44-7.12 (m, 8H), 6.98 (d, *J* = 6.4 Hz, 2H), 4.89 (s, 2H), 1.89 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.4, 142.9, 137.5, 129.5, 128.8, 128.4, 128.2, 127.9, 127.3, 52.8, 22.8; **HRMS** for $C_{15}H_{16}NO$ $[M+1]^+$ Calculated: 226.1232, Found: 226.1234.



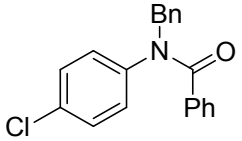
***N*-Butyl-*N*-phenylacetamide (3k)**[9]

Colorless oil, 26.0 mg, 68% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.46-7.31 (m, 3H), 7.19-7.13 (m, 2H), 3.79-3.62 (m, 2H), 1.82 (s, 3H), 1.53-1.42 (m, 2H), 1.37-1.23 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.2, 143.3, 129.6, 128.2, 127.8, 48.8, 29.9, 22.9, 20.1, 13.8; **HRMS** for $C_{12}H_{18}NO$ $[M+1]^+$ Calculated: 192.1388, Found: 192.1392.



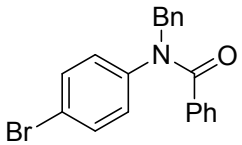
***N*-benzyl-*N*-(*p*-tolyl)benzamide (3l)**

White solid, 48.8 mg, 81% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34-7.14 (m, 10H), 6.91 (d, *J* = 8.2 Hz, 2H), 6.78 (d, *J* = 8.4 Hz, 2H), 5.11 (s, 2H), 2.17 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.6, 143.5, 137.7, 136.5, 136.1, 129.6, 128.8, 128.5, 128.4, 127.7, 127.5, 127.3, 124.9, 53.9, 21.0; **HRMS** for $C_{21}H_{20}NO$ $[M+1]^+$ Calculated: 302.1545, Found: 302.1549.



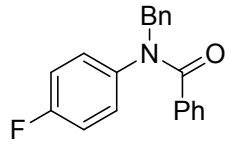
***N*-benzyl-*N*-(4-chlorophenyl)benzamide (3m)**[10]

White solid, 55.3 mg, 86% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35-7.24 (m, 10H), 7.10 (d, *J* = 8.7 Hz, 2H), 6.83 (d, *J* = 8.6 Hz, 2H), 5.10 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.5, 142.1, 137.2, 135.7, 132.3, 129.9, 129.2, 129.0, 128.7, 128.6, 128.4, 127.9, 127.6, 53.8; **HRMS** for $C_{20}H_{17}ClNO$ $[M+1]^+$ Calculated: 322.0999, Found: 322.0995.



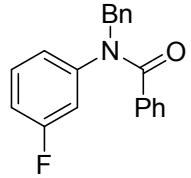
***N*-benzyl-*N*-(4-bromophenyl)benzamide (3n)**

White solid, 52.7 mg, 72% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34-7.15 (m, 12H), 6.77 (d, *J* = 8.6 Hz, 2H), 5.10 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.5, 142.6, 137.2, 135.6, 132.2, 130.0, 129.3, 128.7, 128.6, 128.4, 128.0, 127.6, 120.3, 53.7; **HRMS** for $C_{20}H_{17}BrNO$ $[M+1]^+$ Calculated: 366.0494, Found: 366.0494.



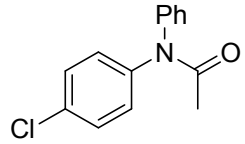
***N*-Benzyl-*N*-(4-fluorophenyl)benzamide (3o)**

White solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37-7.13 (m, 10H), 6.89-6.76 (m, 4H), 5.09 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.6, 160.9 (*J* = 247.3 Hz), 139.4 (*J* = 3.3 Hz), 137.3, 135.8, 129.7, 129.5 (*J* = 8.5 Hz), 128.6(4), 128.5(7), 128.5, 127.9, 127.5, 115.9(*J* = 22.6 Hz), 53.9; **HRMS** for $C_{20}H_{17}FNO$ $[M+1]^+$ Calculated: 306.1294, Found: 306.1296.



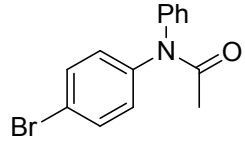
***N*-Benzyl-*N*-(3-fluorophenyl)benzamide (4o)**

White solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39-7.16 (m, 10H), 7.13-7.03 (m, 1H), 6.84-6.77 (m, 1H), 6.71- 6.62 (m, 2H), 5.12 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.5, 162.6 (*J* = 247.9 Hz), 145.1 (*J* = 9.6 Hz), 137.2, 135.6, 130.1, 130.0(0), 129.9(8), 128.6 (*J* = 8.1 Hz), 128.3, 127.9, 127.6, 123.6 (*J* = 3.1 Hz), 114.8 (*J* = 22.7 Hz), 113.7 (*J* = 21.1 Hz), 53.8; **HRMS** for $C_{20}H_{17}FNO$ $[M+1]^+$ Calculated: 306.1294, Found: 306.1292.



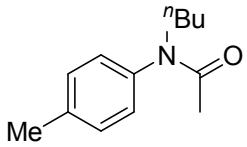
***N*-(4-chlorophenyl)-*N*-phenylacetamide (3p)**[11]

Colorless oil, 31.0 mg, 63% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54-7.03 (m, 9H), 2.06 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.4, 23.8; **HRMS** for $C_{14}H_{13}ClNO$ $[M+1]^+$ Calculated: 246.0686, Found: 246.0679.



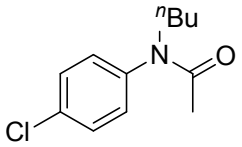
***N*-(4-bromophenyl)-*N*-phenylacetamide (3q)**[11]

Colorless oil, 37.7 mg, 65% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.56-7.10 (m, 9H), 2.06 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.4, 23.9; **HRMS** for $C_{14}H_{13}BrNO$ $[M+1]^+$ Calculated: 290.0181, Found: 290.0181.



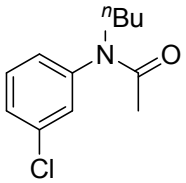
***N*-butyl-*N*-(*p*-tolyl)acetamide (3r)**[12]

Colorless oil, 29.6 mg, 72% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21 (d, *J* = 8.6 Hz, 1H), 7.15 (d, *J* = 7.6 Hz, 1H), 7.03 (d, *J* = 8.2 Hz, 1H), 6.95 (d, *J* = 8.5 Hz, 1H), 3.70-3.63 (m, 2H), 2.38 (s, 3H), 1.81 (s, 3H), 1.53-1.40 (m, 2H), 1.36-1.22 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.2, 140.6, 137.7, 130.2, 127.9, 48.8, 29.9, 22.8, 21.1, 20.1, 13.8; **HRMS** for $C_{13}H_{20}NO$ $[M+1]^+$ Calculated: 206.1545, Found: 206.1550.



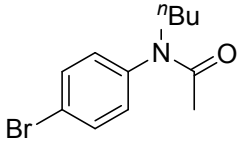
***N*-Butyl-*N*-(4-chlorophenyl)acetamide (3s)**

Colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.6 Hz, 2H), 7.11 (d, *J* = 8.6 Hz, 2H), 3.69-3.64 (m, 2H), 1.82 (s, 3H), 1.46 (dt, *J* = 15.3, 7.4 Hz, 2H), 1.30 (dq, *J* = 14.5, 7.2 Hz, 2H), 0.88 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.9, 141.8, 133.6, 129.9, 129.5, 48.8, 29.8, 22.8, 20.0, 13.8; **HRMS** for $C_{12}H_{17}ClNO$ $[M+1]^+$ Calculated: 226.0999, Found: 226.0999.



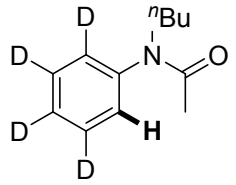
***N*-Butyl-*N*-(3-chlorophenyl)acetamide (4s)**[12]

Colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40-7.31 (m, 2H), 7.18 (s, 1H), 7.07 (d, *J* = 6.8 Hz, 1H), 3.69 (t, *J* = 7.4 Hz, 2H), 1.84 (s, 3H), 1.54-1.41 (m, 2H), 1.39-1.21 (m, 2H), 0.89 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.2, 144.5, 135.1, 130.6, 128.5, 128.1, 126.6, 48.9, 29.9, 22.9, 20.0, 13.8; **HRMS** for $C_{12}H_{17}ClNO$ $[M+1]^+$ Calculated: 226.0999, Found: 226.1005.



***N*-(4-bromophenyl)-*N*-butylacetamide (3t)**

Colorless oil, 35.1 mg, 65% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.5 Hz, 2H), 7.07 (d, *J* = 8.6 Hz, 2H), 3.74-3.59 (m, 2H), 1.83 (s, 3H), 1.59-1.37 (m, 2H), 1.37-1.23 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.8, 142.3, 132.9, 129.9, 127.0, 48.8, 29.9, 22.8, 20.0, 13.8; **HRMS** for $C_{12}H_{17}BrNO$ $[M+1]^+$ Calculated: 270.0494, Found: 270.0492.

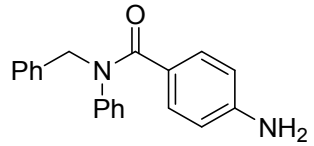


***N*-butyl-*N*-phenyl-*d*$_4$ acetamide (3u)**

Colorless oil, 25.0 mg, 64% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.16 (s, 1H), 3.69 (t, *J* = 7.6 Hz, 2H), 1.82 (s, 3H), 1.48 (dt, *J* = 15.3, 7.5 Hz, 2H), 1.30 (dq, *J* = 14.8, 7.4 Hz, 2H), 0.88 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.2, 143.2, 128.1, 48.8, 29.9, 22.9, 20.1, 13.8; **HRMS** for $C_{12}H_{13}D_4NO$ $[M+1]^+$ Calculated: 196.1639, Found: 196.1643.

General Procedure for Chemoselective *N*-Arylation of Amide and Amine

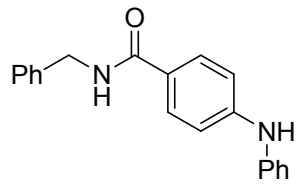
To a suspension of *t*-BuOK (5.0 equiv, 112.2 mg, 1.0 mmol) in THF (4.0 mL), was added **1l** (1.0 equiv, 45.3 mg, 0.2 mmol) at 0 °C. The resulting mixture was stirred at this temperature for 10 min. Diphenyliodonium salt **2a** (1.2 equiv, 103.2 mg, 0.24 mmol) was added in one portion and the reaction mixture was stirred at 0 °C until TLC analysis indicated complete consumption of the amide substrate (**1l)**. The reaction was then quenched with H_2O at 0 °C, the organic phase was separated and the water phase was extracted with dichloromethane. The combined organic phases were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude materials were purified by SiO_2 flash chromatography to afford the product **5a**.



4-amino-*N*-benzyl-*N*-phenylbenzamide (5a)[13]

Colorless oil, 27.2 mg, 45% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35-7.07 (m, 10H), 6.98-6.91 (m, 2H), 6.40 (d, *J* = 8.6 Hz, 2H), 5.11 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.4, 148.0, 144.5, 138.0, 131.2, 129.0, 128.4, 128.3, 127.4, 127.2, 126.2, 125.3, 113.6, 54.1; **HRMS** for $C_{20}H_{19}N_2O$ $[M+1]^+$ Calculated: 303.1497, Found: 303.1496.

To a stirred solution of **1l** (1.0 equiv, 45.3 mg, 0.2 mmol), Na_2CO_3 (2.0 equiv, 42.4 mg, 0.4 mmol) and CuBr (2.9 mg, 10 mol %) in CH_2Cl_2 (2 mL) at room temperature was added diphenyliodonium salt **2a** (1.2 equiv, 103.2 mg, 0.24 mmol). The reaction mixture was stirred for 6 h at room temperature. The mixture was filtered and evaporated *in vacuo*. The crude materials was purified by SiO_2 flash chromatography to afford the product **5b**.



***N*-benzyl-4-(phenylamino)benzamide (5b)**

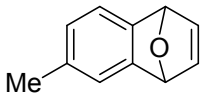
Colorless oil, 43.5 mg, 72% yield; **^{1}H NMR** (400 MHz, CDCl3) δ 7.69 (d, *J* = 8.7 Hz, 2H), 7.38-

7.27 (m, 7H), 7.14 (d, J = 8.5, 2H), 7.06-6.98 (m, 3H), 6.32 (brs, 1H), 6.00 (s, 1H), 4.63 (d, J = 5.7 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.9, 146.8, 141.3, 138.5, 129.5, 128.8, 128.7, 127.9, 127.5, 125.4, 122.7, 119.8, 115.3, 44.1; **HRMS** for $C_{20}H_{19}N_2O$ $[M+1]^+$ Calculated: 303.1497, Found: 303.1494.

General Procedure for Cycloaddition Reaction of Diaryliodinium Salt with Furan

Under a N_2 atmosphere, diaryliodonium salt **2b** (97.3 mg, 0.2 mmol), furan (68.1 mg, 1.0 mmol) and toluene (2 mL) were added to a flame-dried Schlenk tube, and *t*-BuOK (44.9 mg, 0.4 mmol) was added sequentially. The reaction mixture was stirred for 3 h at room temperature, and quenched with H_2O at 0 °C. The organic phase was separated and the water phase was extracted with dichloromethane. The combined organic phases were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude materials were purified by flash chromatography to afford the product **6** (17.7 mg) in 56% yield as a yellow oil.

Spectral data is consistent with the literature.[14]



6-methyl-1,4-dihydro-1,4-epoxynaphthalene (**6**)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (d, J = 7.2 Hz, 1H), 7.10 (s, 1H), 7.04-6.97 (m, 2H), 6.77 (d, J = 7.2 Hz, 1H), 5.68 (d, J = 5.9 Hz, 2H), 2.30 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 149.4, 146.0, 143.2, 142.7, 134.7, 125.0, 121.6, 119.9, 82.3, 82.2, 21.3.

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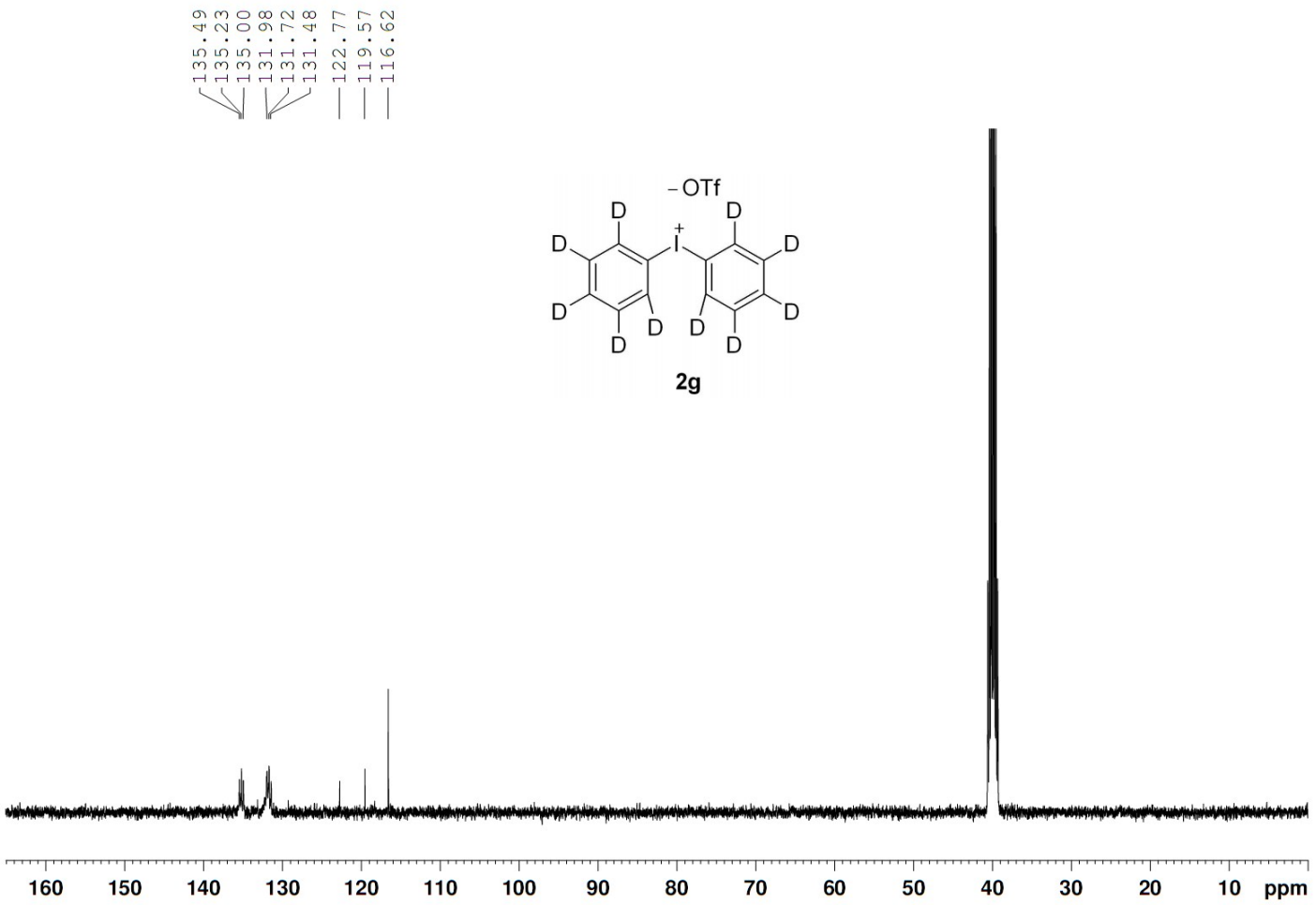
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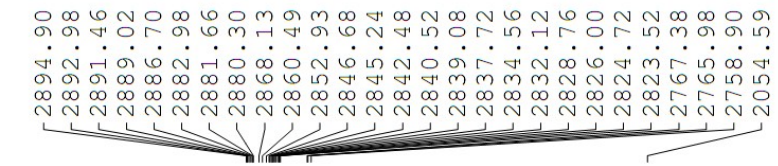
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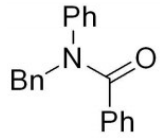
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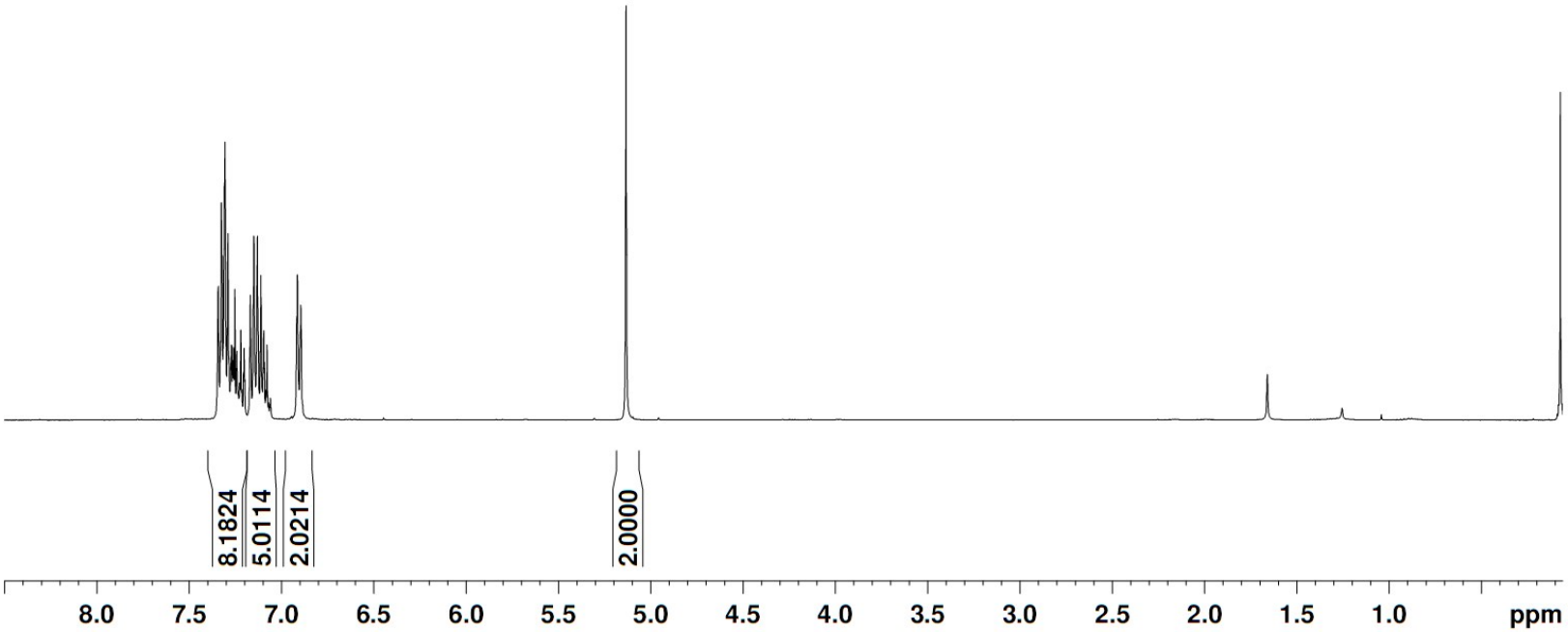
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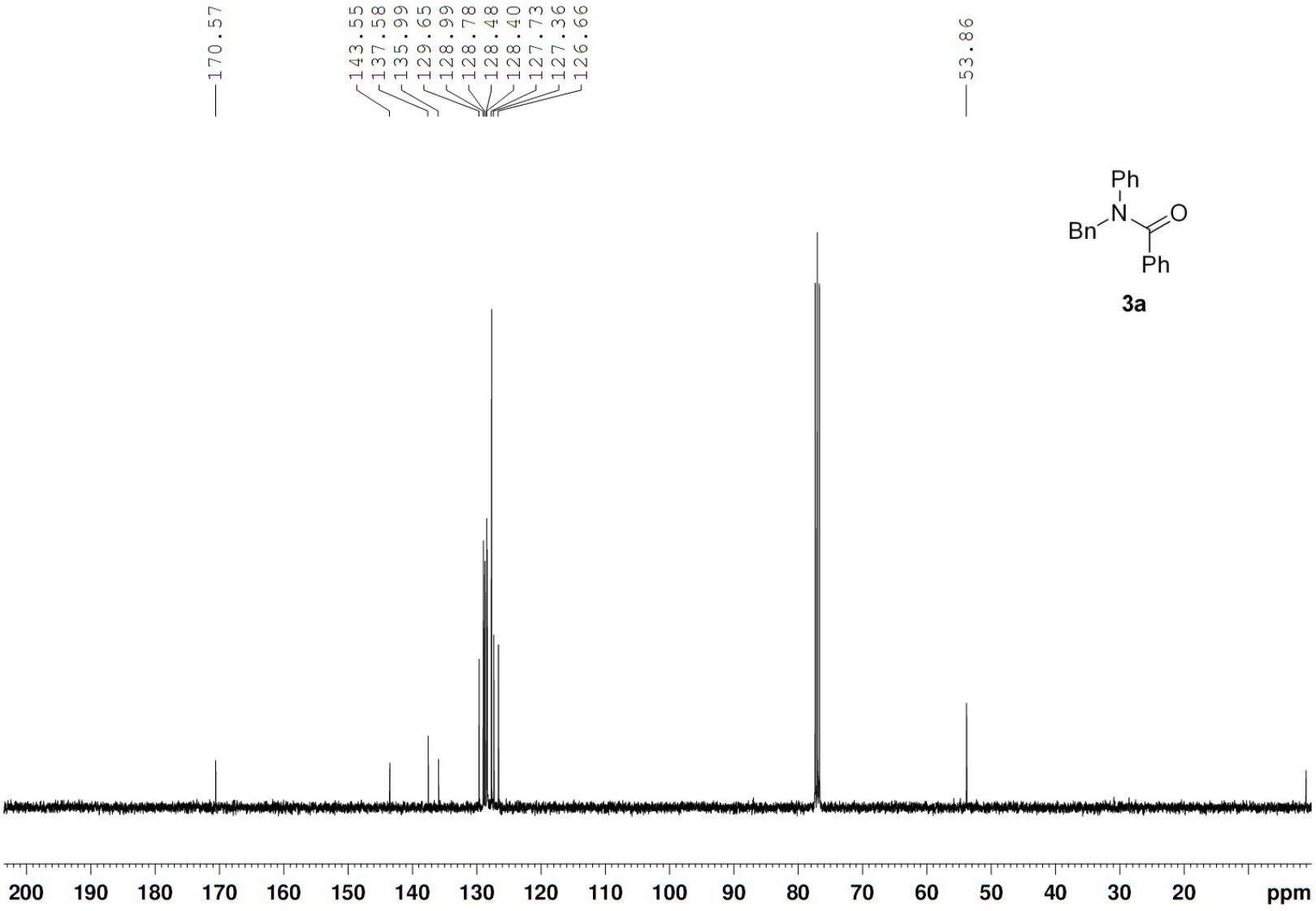
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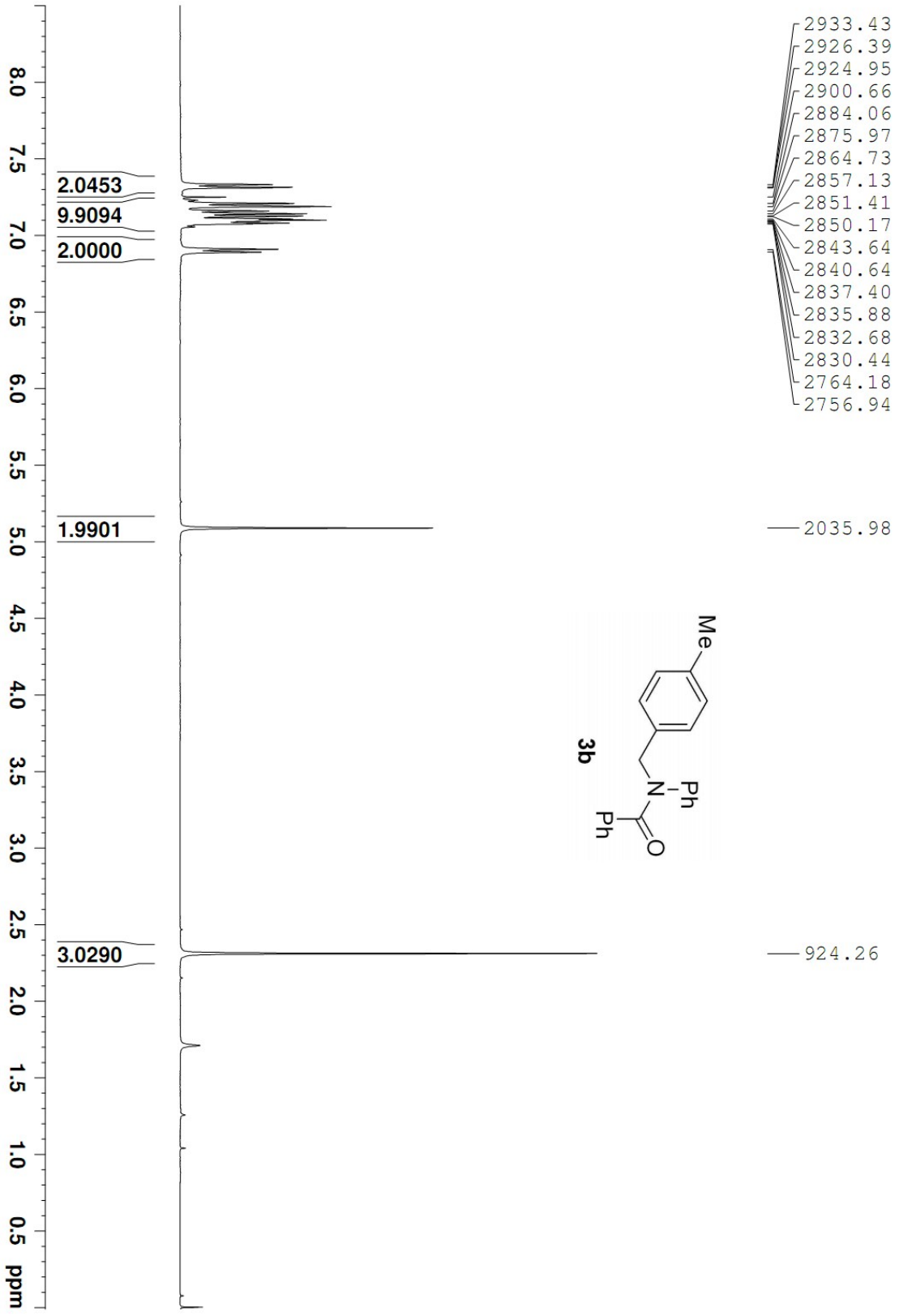
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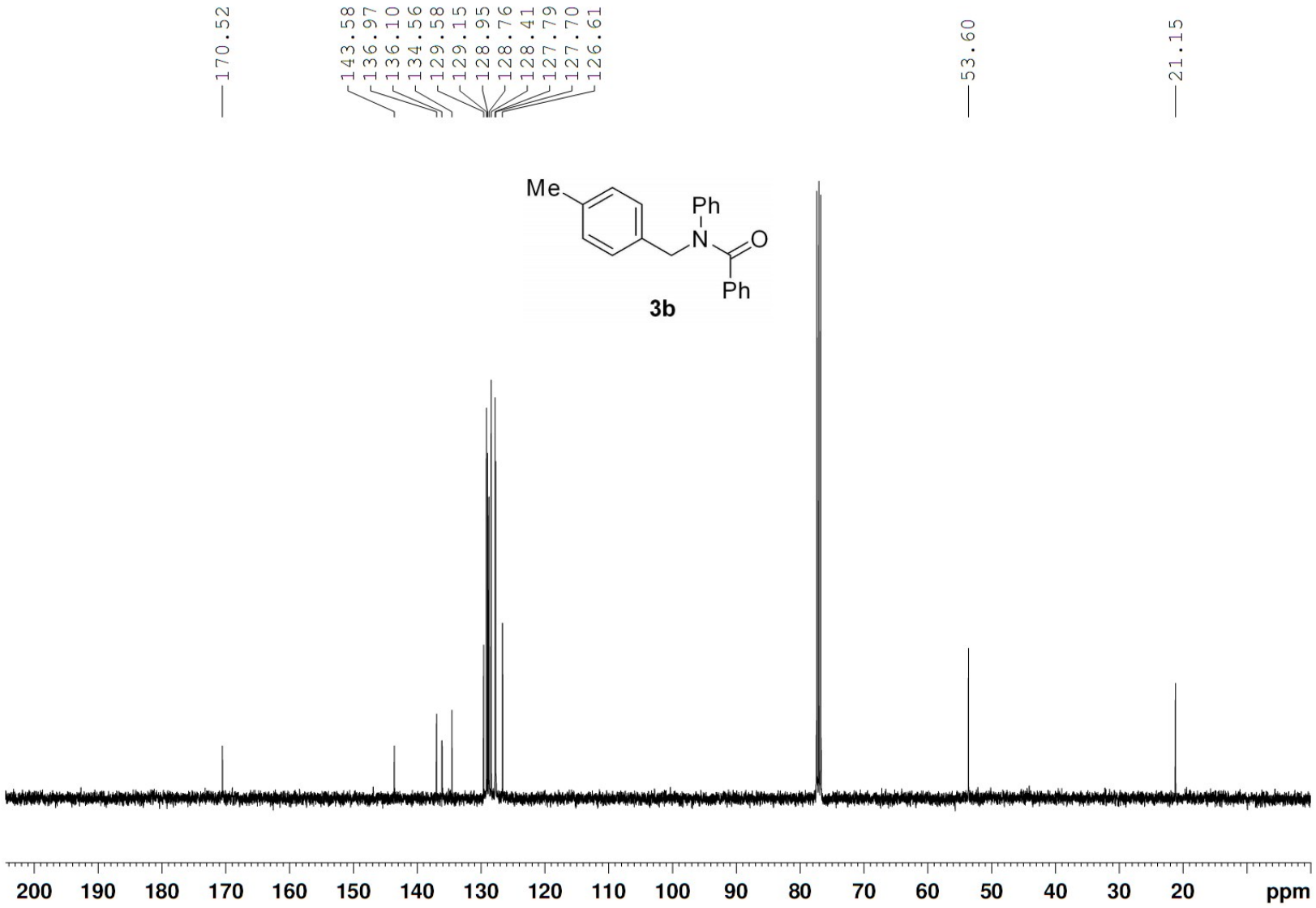
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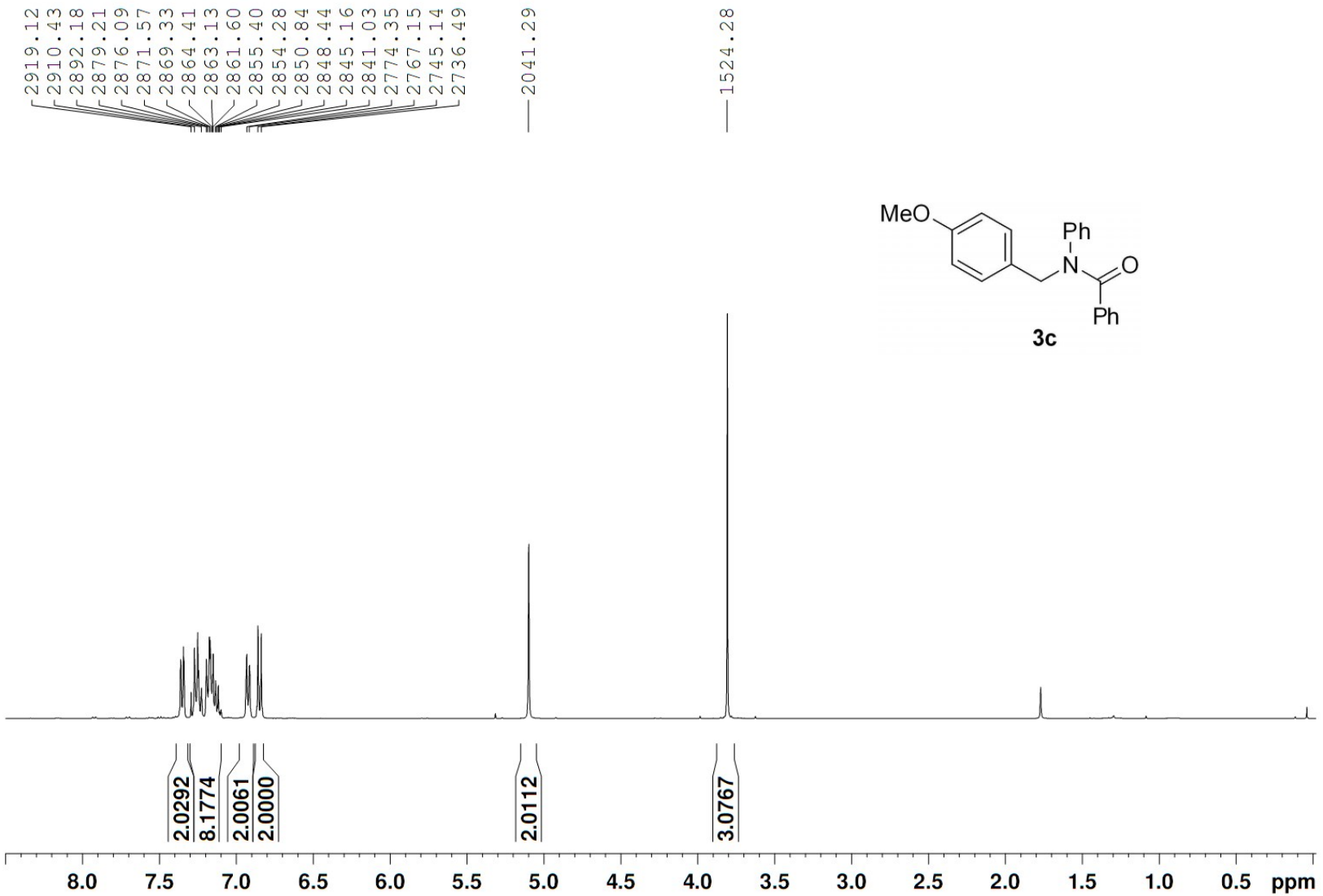
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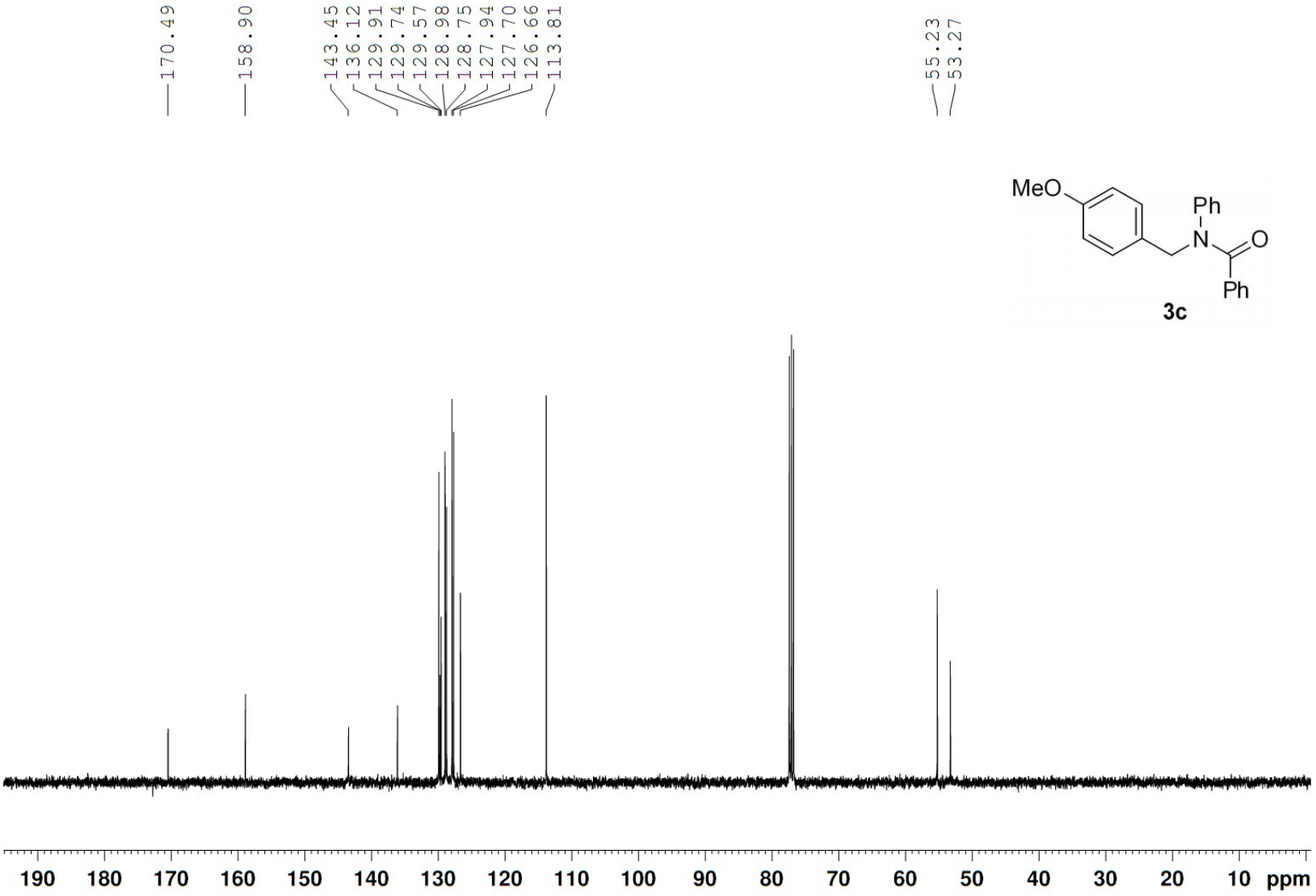
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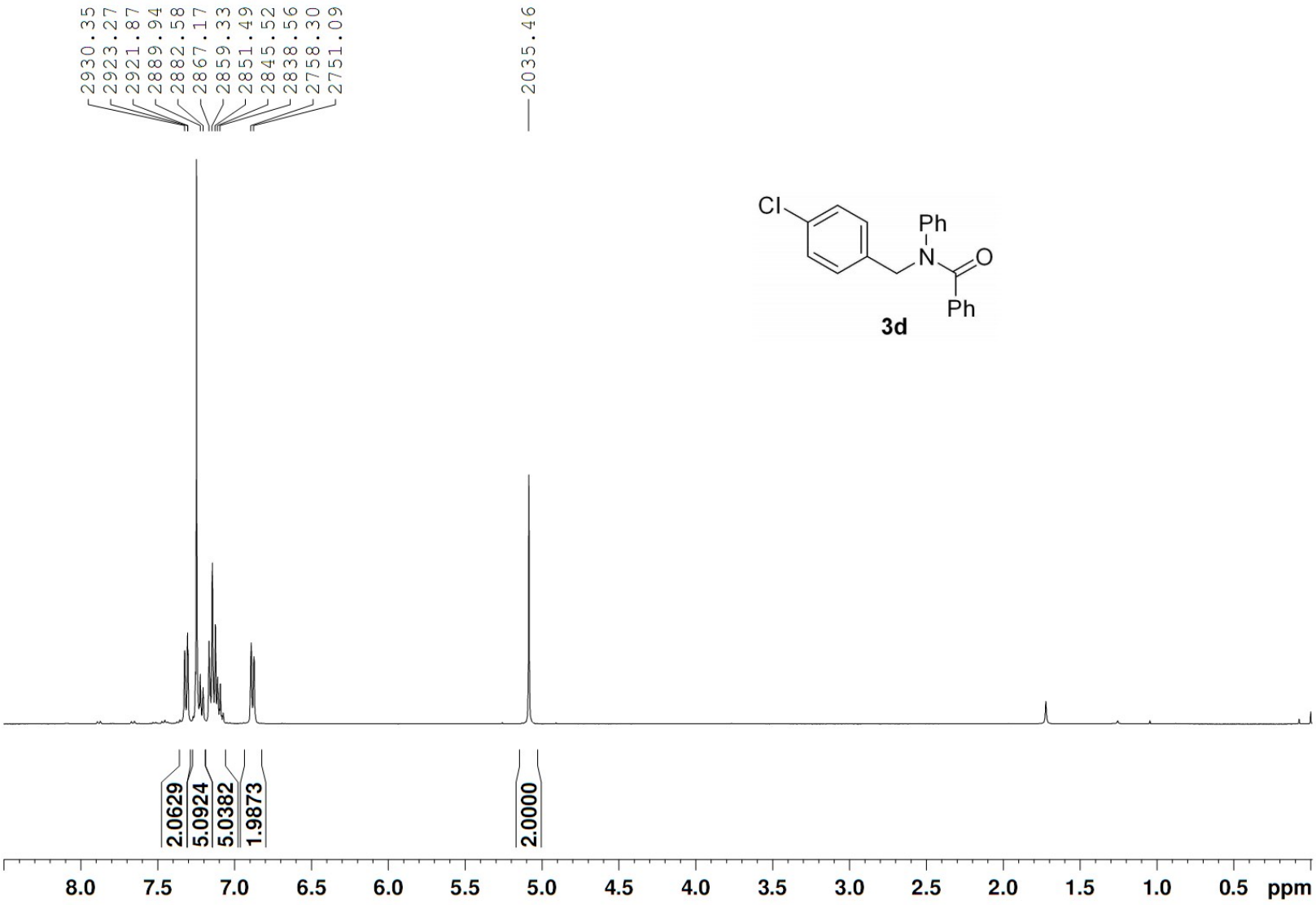
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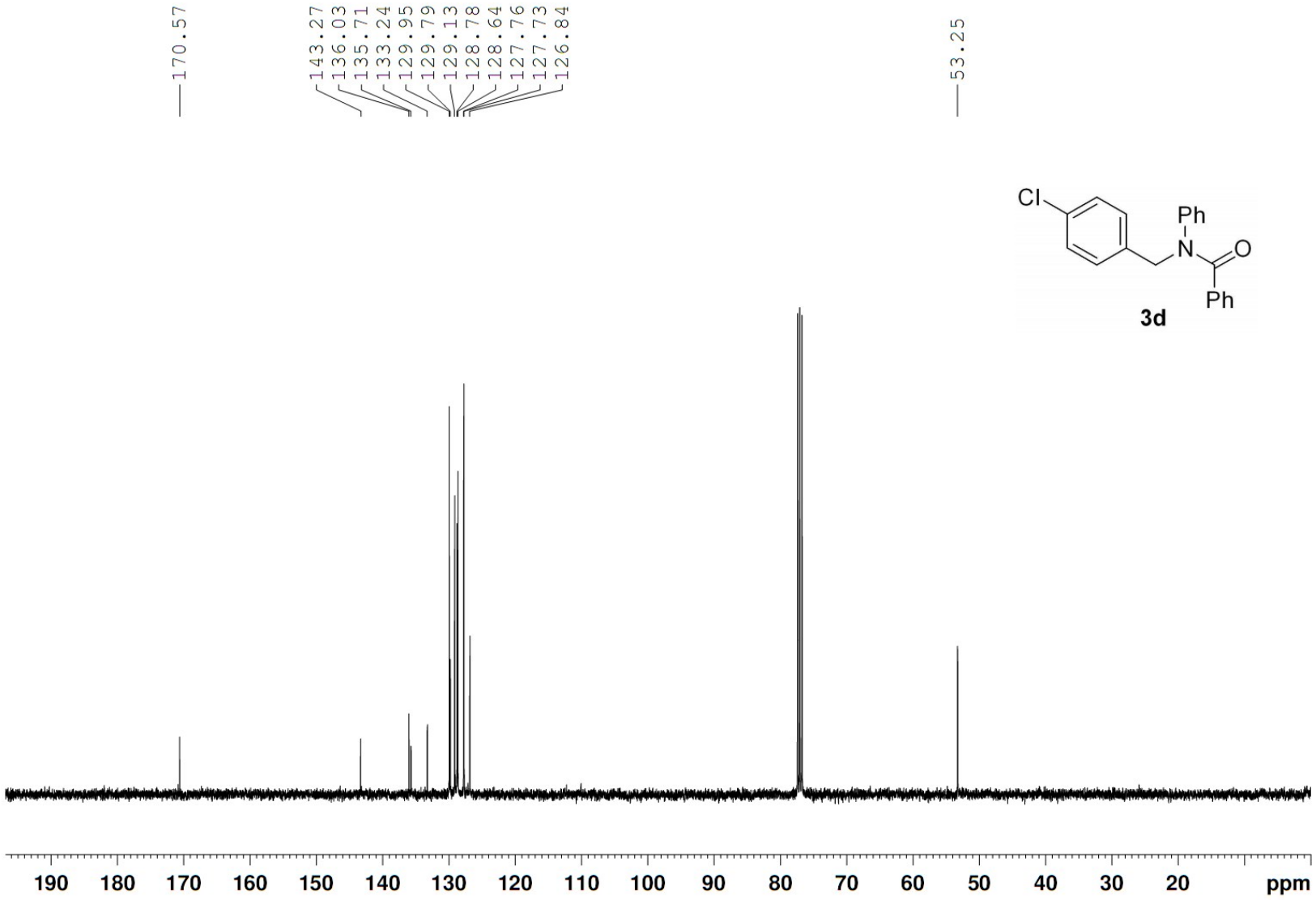
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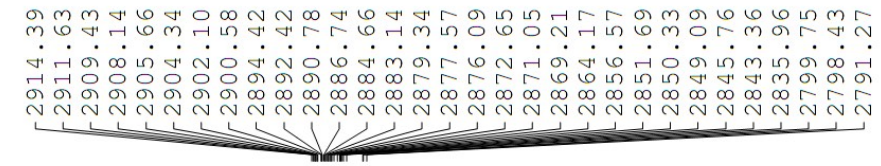
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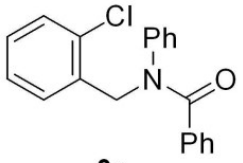
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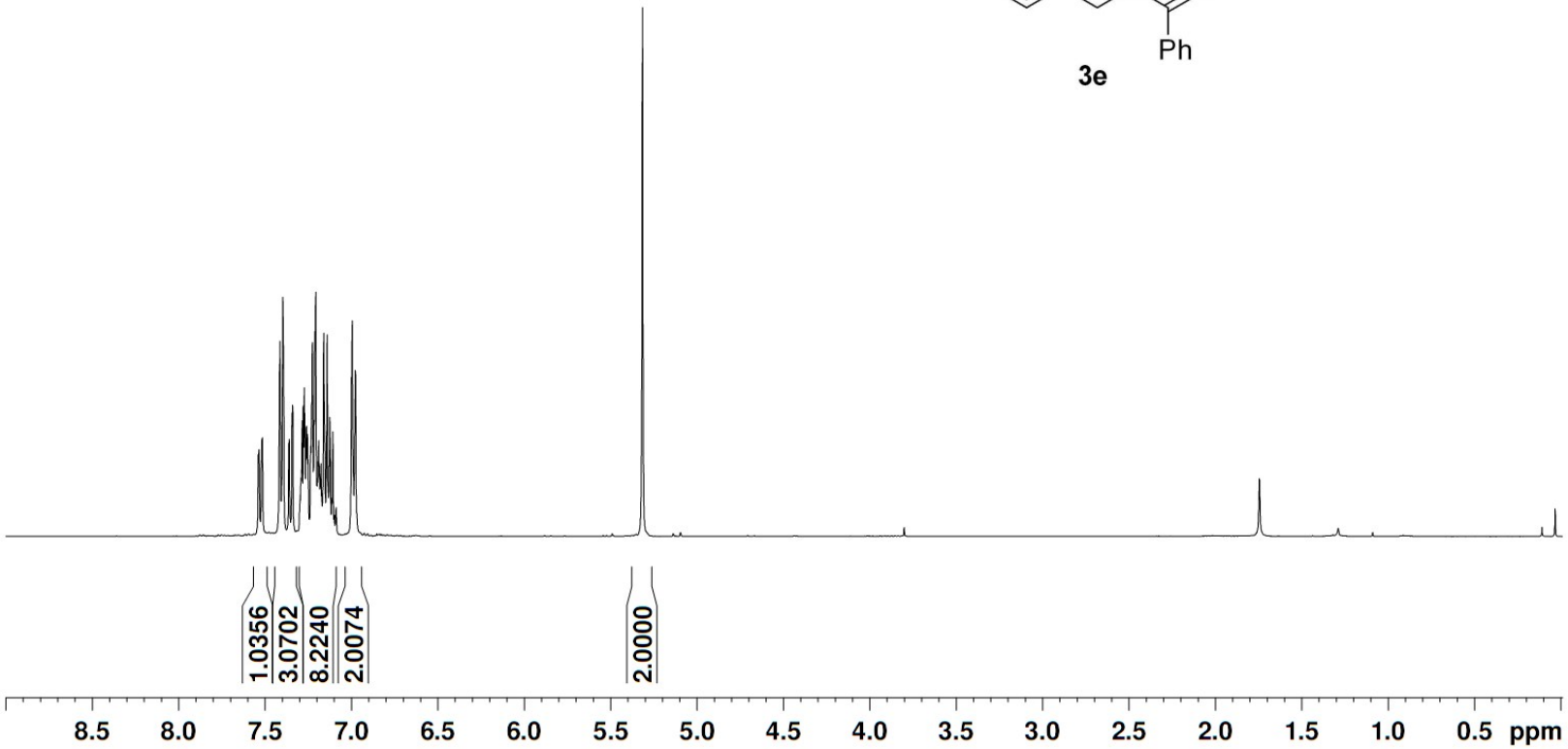
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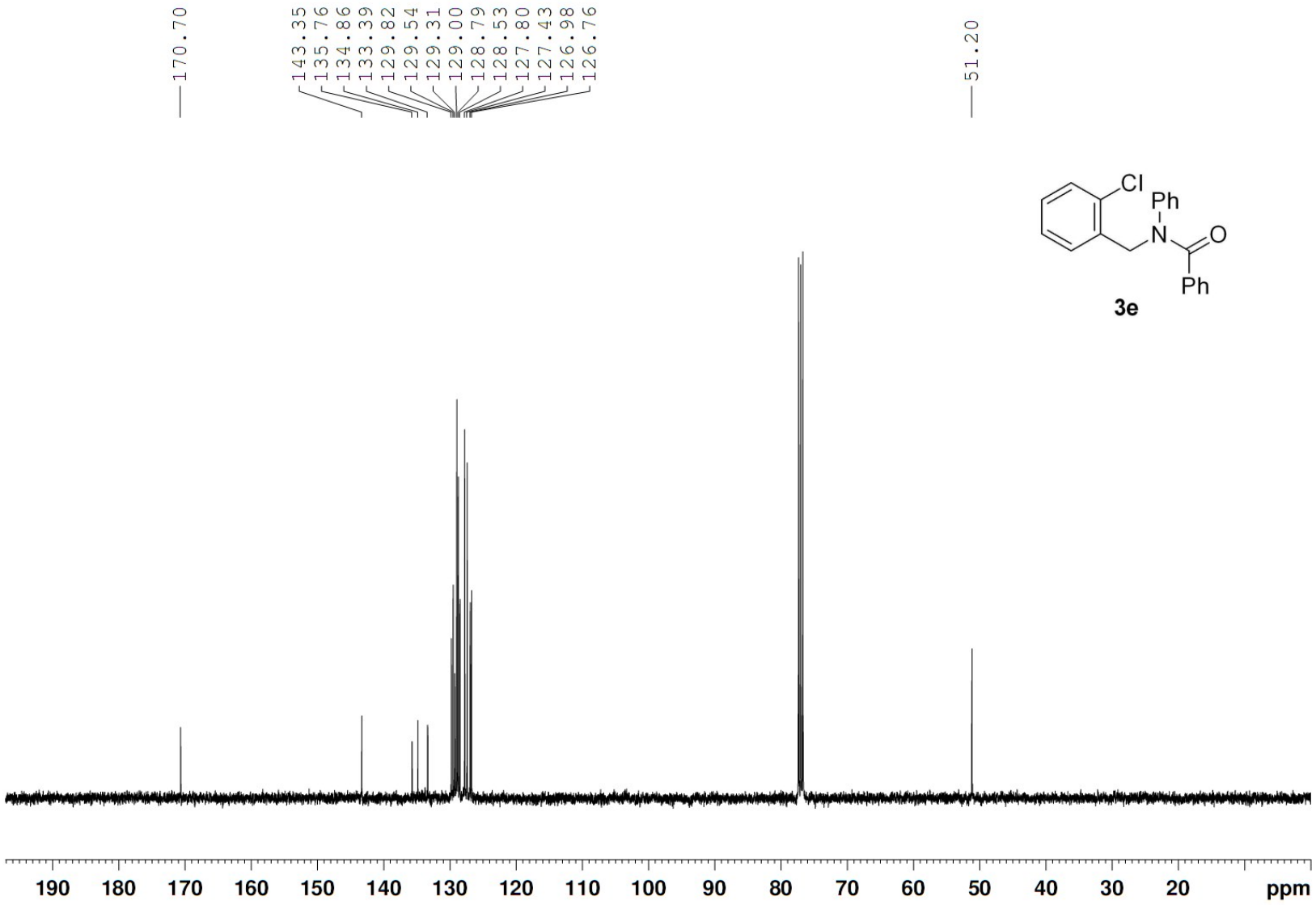
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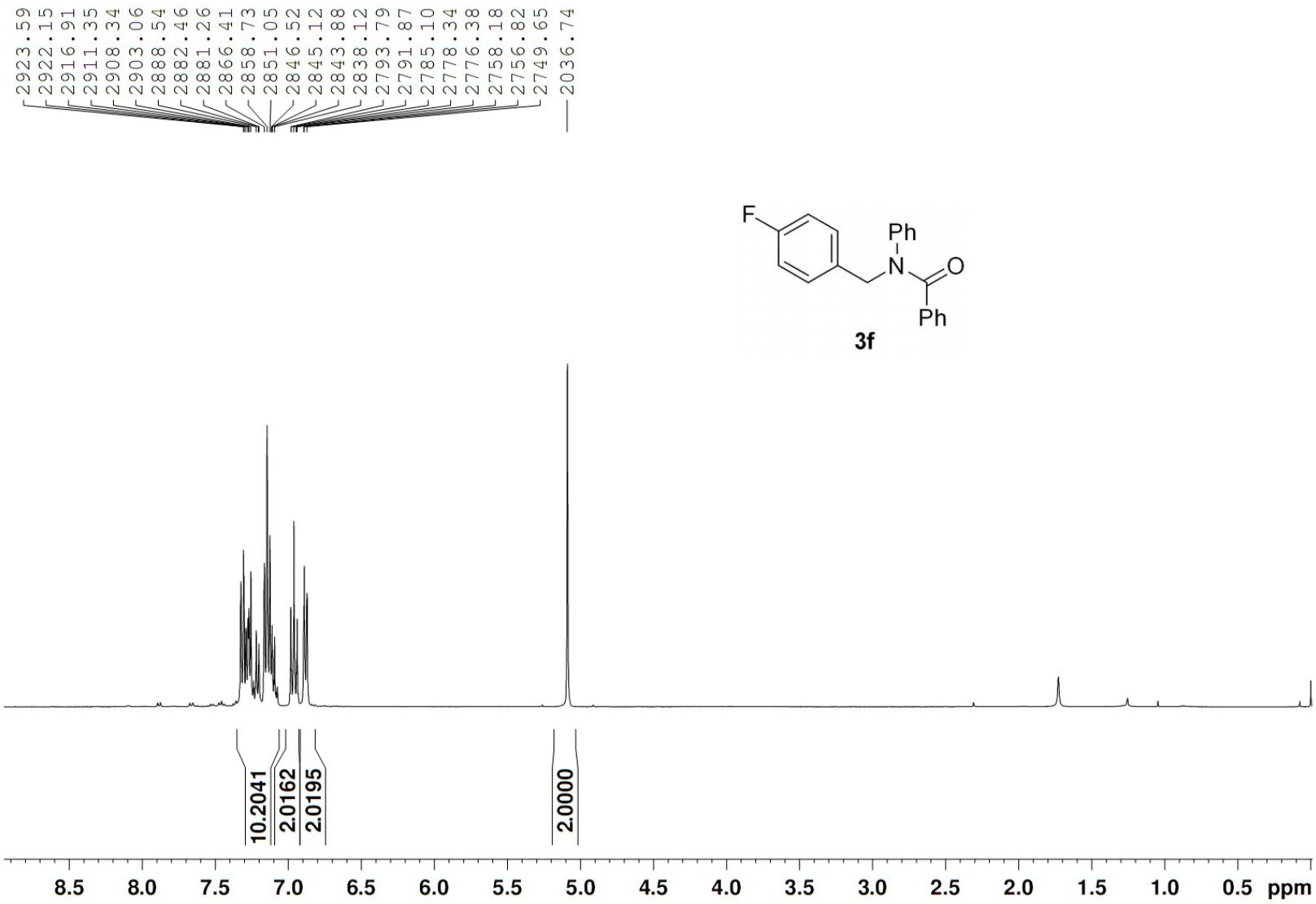
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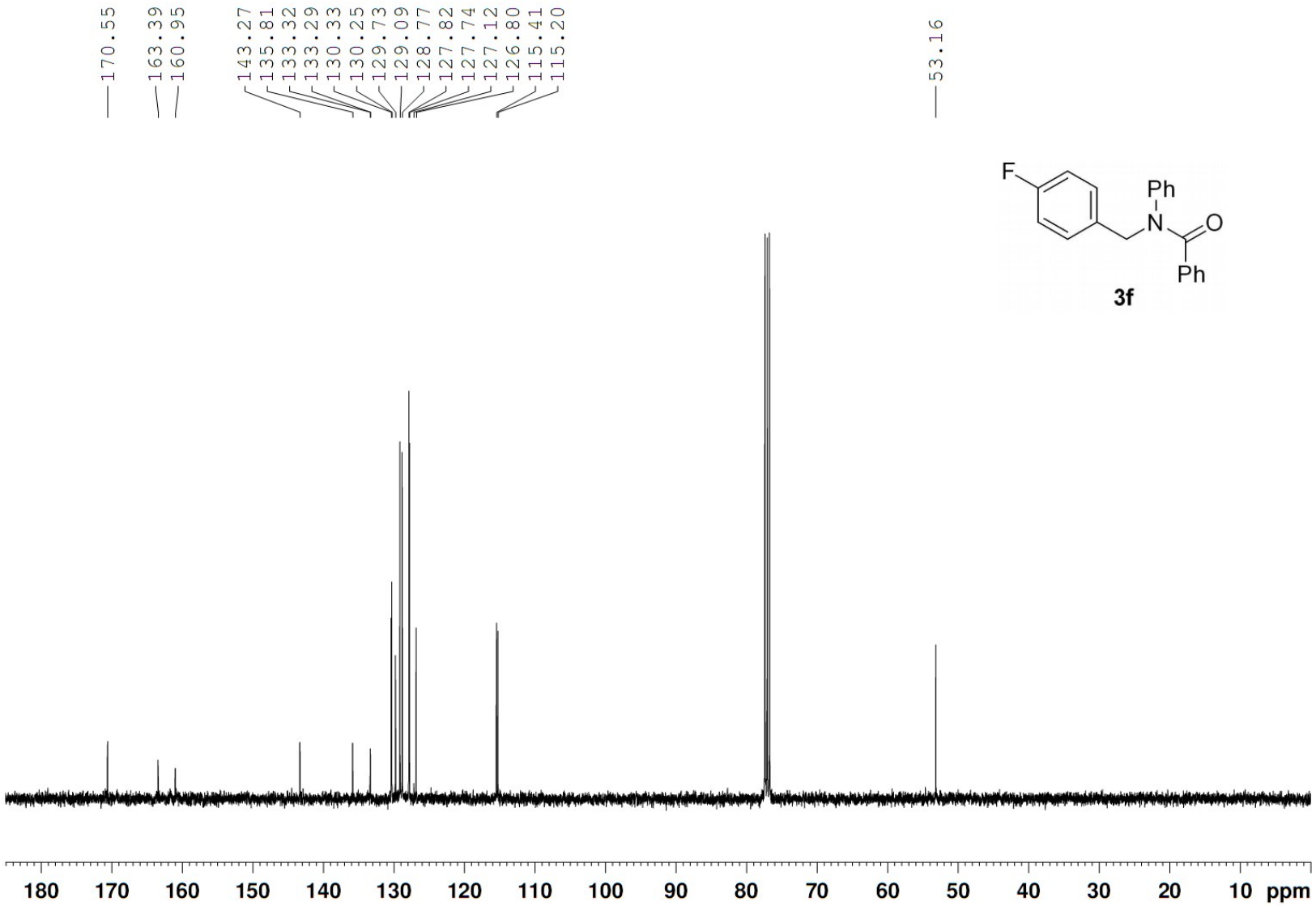
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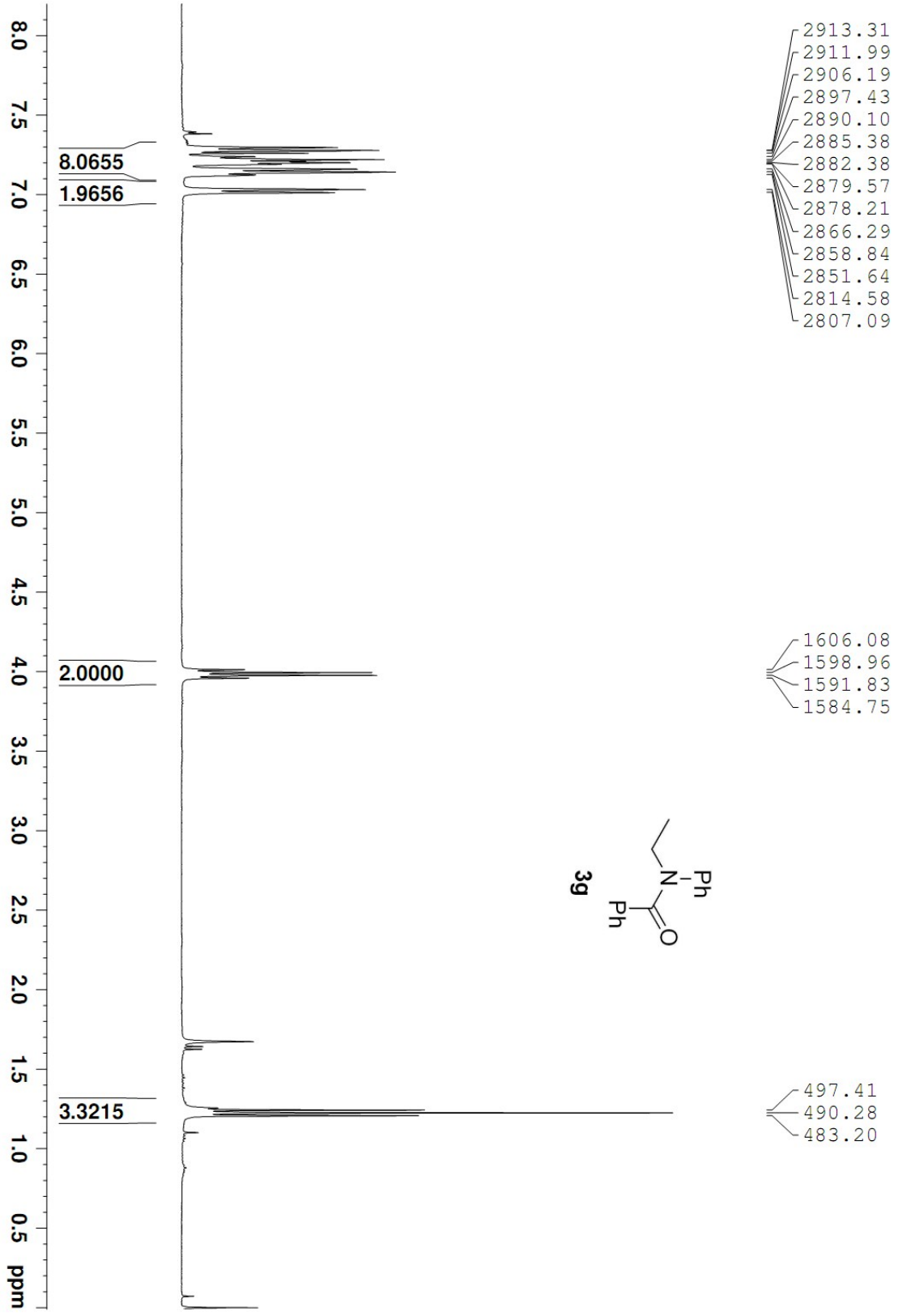
163.39
160.95
143.27
135.81
133.32
133.29
130.33
130.25
129.73
129.09
128.77
127.82
127.74
127.12
126.80
115.41
115.20
53.16
F
Ph
N
O
Ph
3f
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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2.0000
3.3215
2913.31
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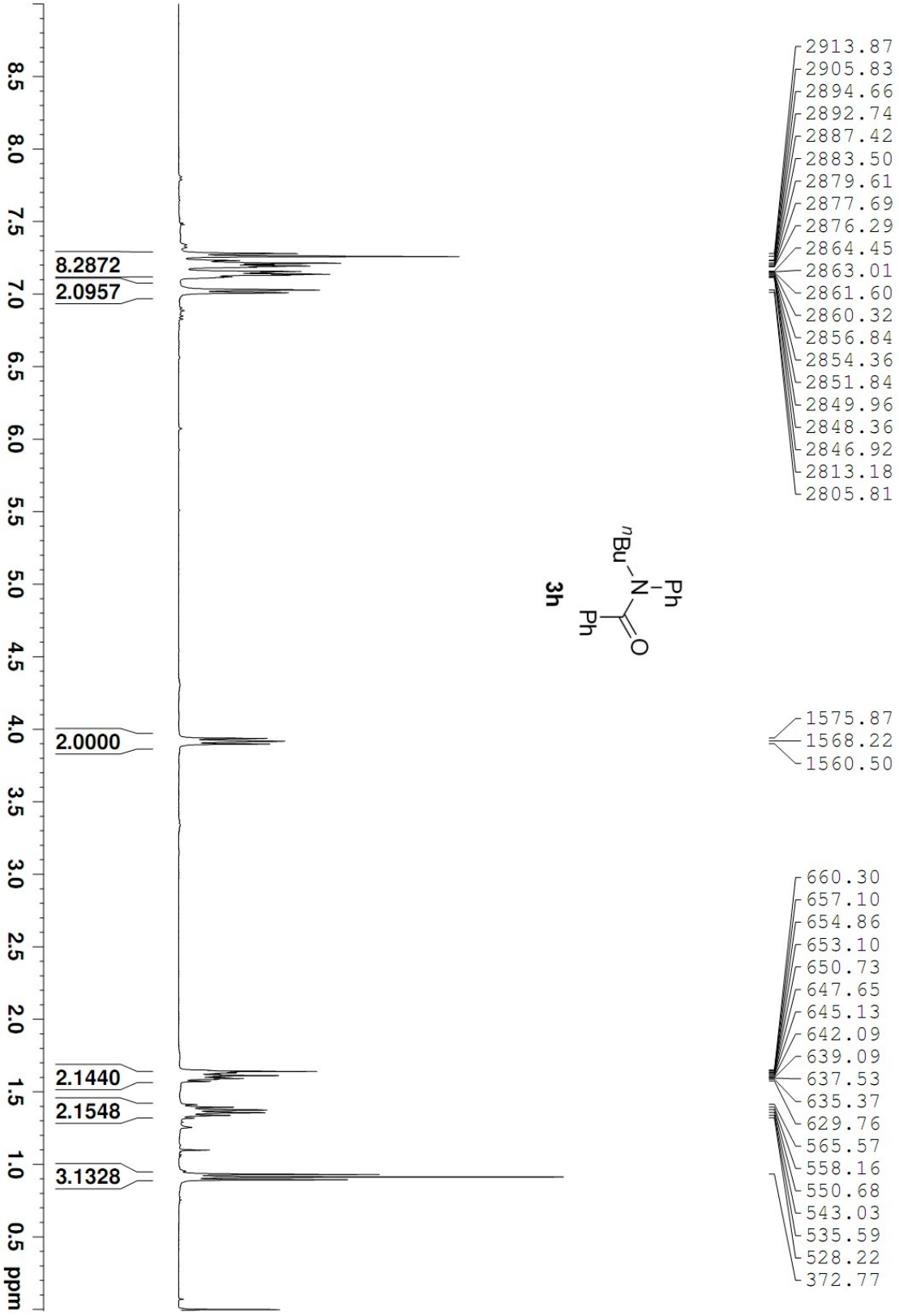
2906.19
2897.43
2890.10
2885.38
2882.38
2879.57
2878.21
2866.29
2858.84
2851.64
2814.58
2807.09
1606.08
1598.96
1591.83
1584.75
497.41
490.28
483.20
Ph
N
Ph
O
3g
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

170.20
143.26
136.36
129.43
129.09
128.66
127.95
127.68
126.62
45.37
12.96
Ph
N
Ph
O
3g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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2.0000
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2.1548
3.1328
8.5
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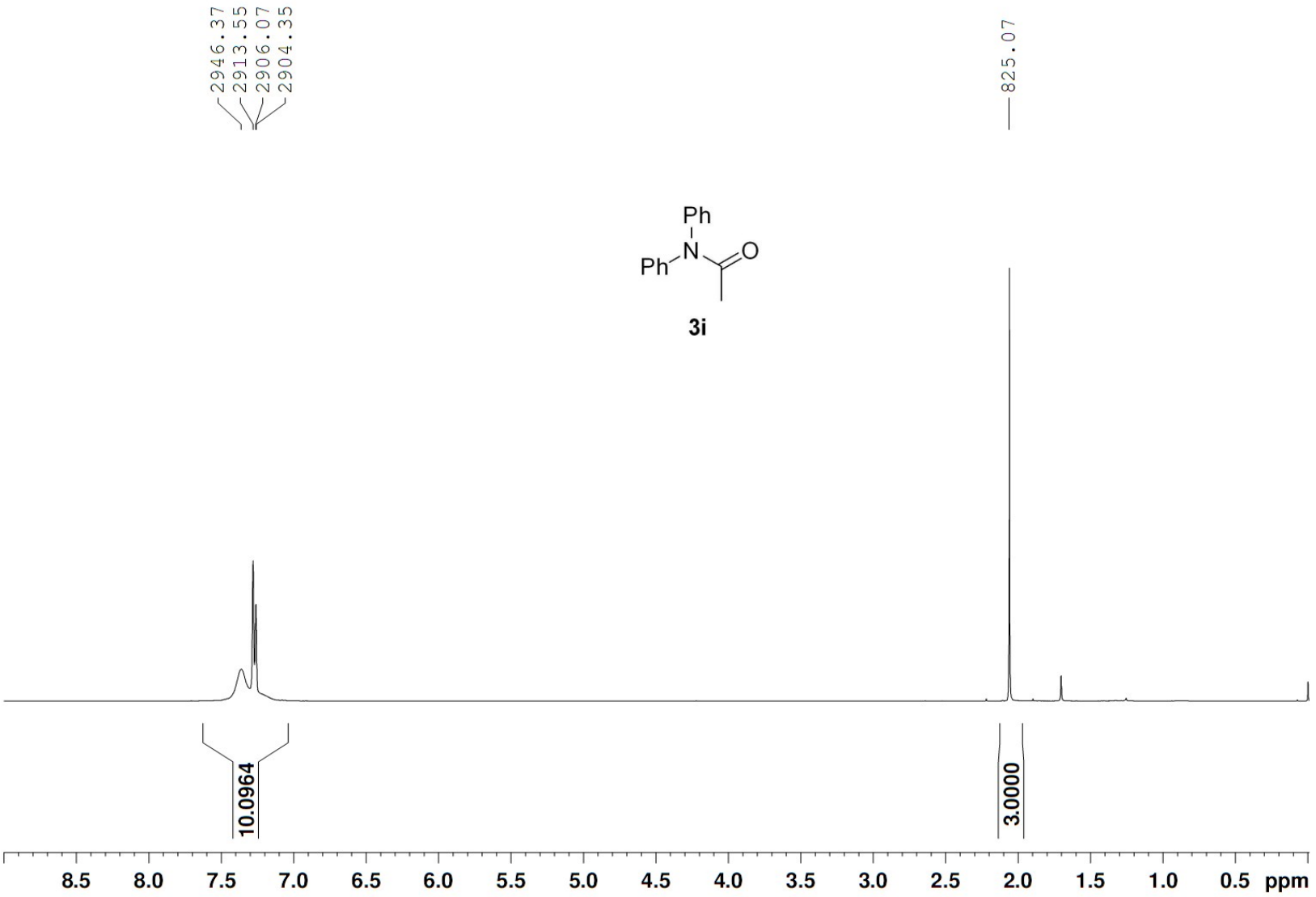
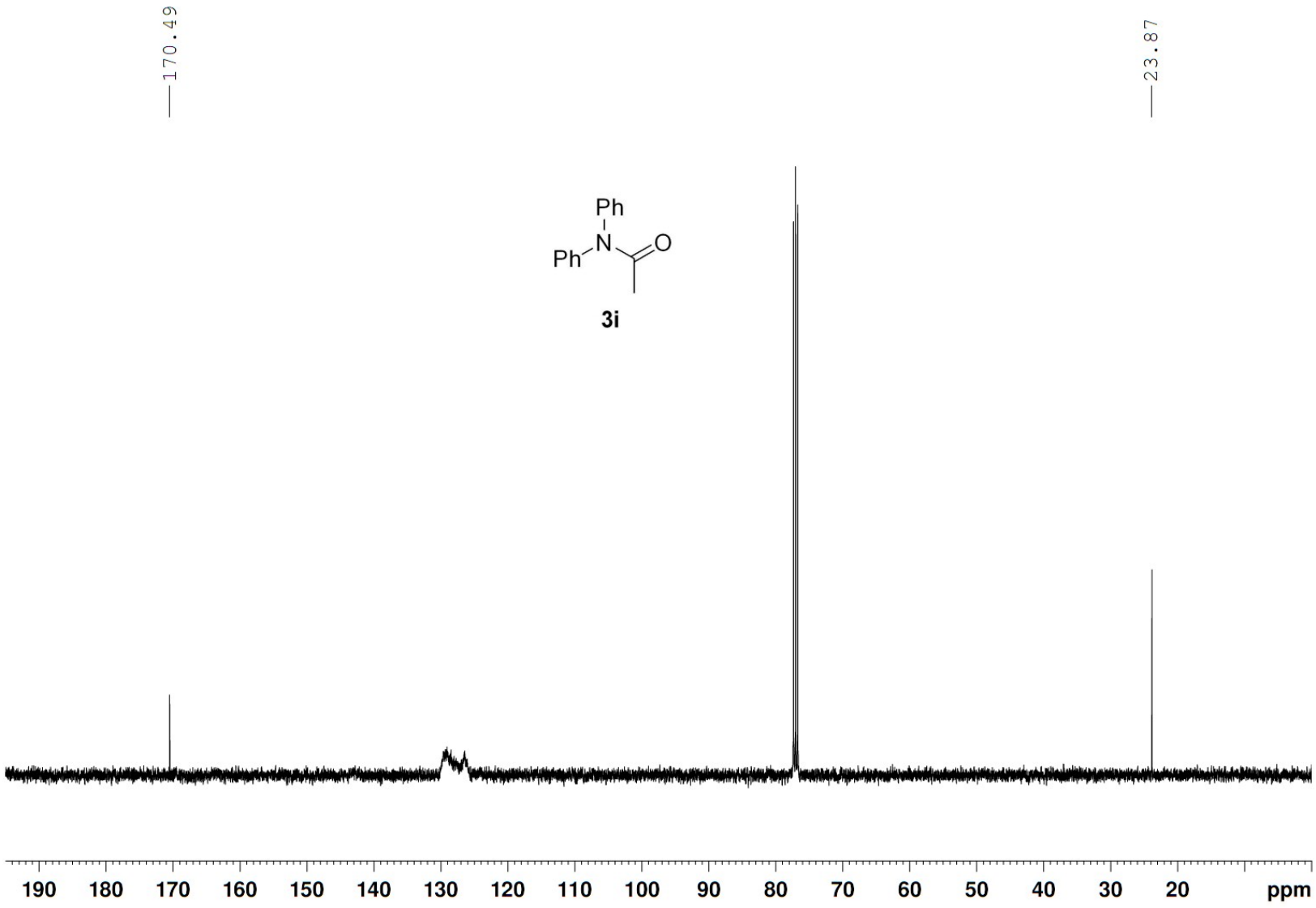
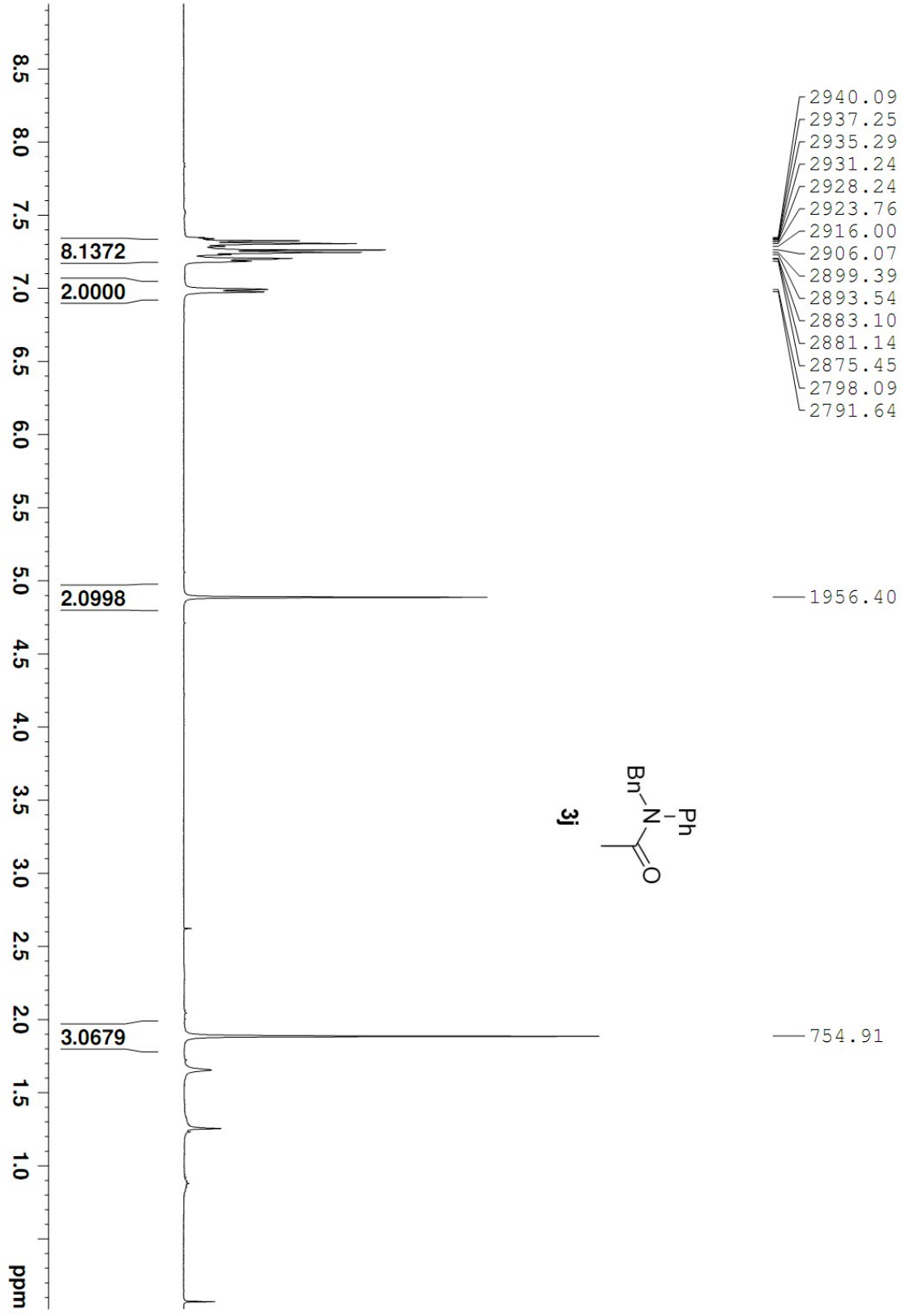
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2913.87
2905.83
2894.66
2892.74
2887.42
2883.50
2879.61
2877.69
2876.29
2864.45
2863.01
2861.60
2860.32
2856.84
2854.36
2851.84
2849.96
2848.36
2846.92
2813.18
2805.81
1575.87
1568.22
1560.50
660.30
657.10
654.86
653.10
650.73
647.65
645.13
642.09
639.09
637.53
635.37
629.76
565.57
558.16
550.68
543.03
535.59
528.22
372.77

3h

170.34
143.55
136.44
129.37
129.05
128.63
127.85
127.66
126.55
50.26
29.83
20.22
13.86
nBu
N
Ph
Ph
O
3h
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


2946.37
2913.55
2906.07
2904.35
825.07
Ph
N
Ph
O
3i
10.0964
3.0000
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


170.49
23.87
Ph
N
Ph
O
3i
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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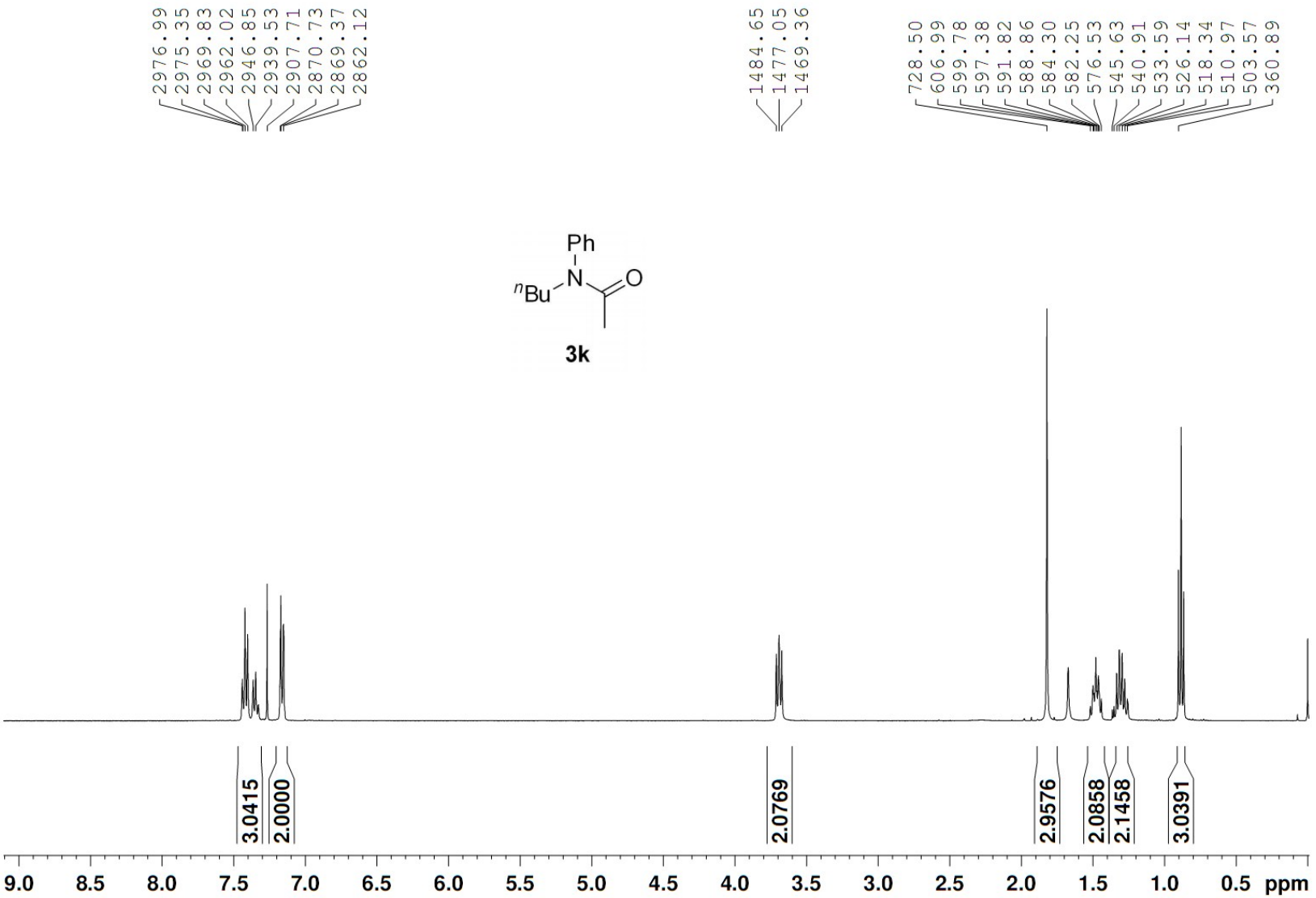
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
8.1372
2.0000
2.0998
3.0679
2940.09
2937.25
2935.29
2931.24
2928.24
2923.76
2916.00
2906.07
2899.39
2893.54
2883.10
2881.14
2875.45
2798.09
2791.64
1956.40
754.91
Bn
N
Ph
O
3j

170.40
142.92
137.52
129.53
128.80
128.36
128.24
127.90
127.33
52.82
22.75
3j
Bn
N
Ph
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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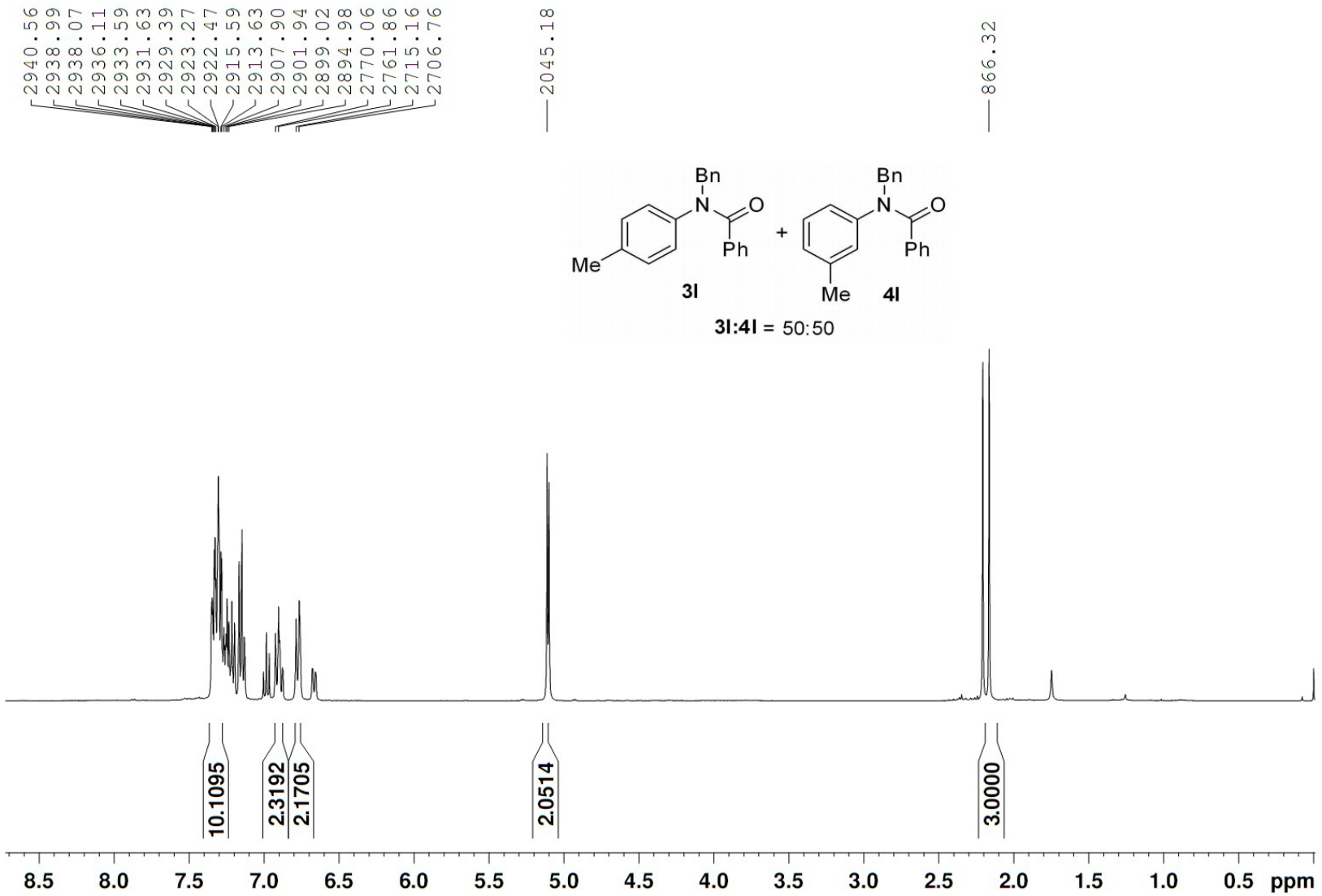
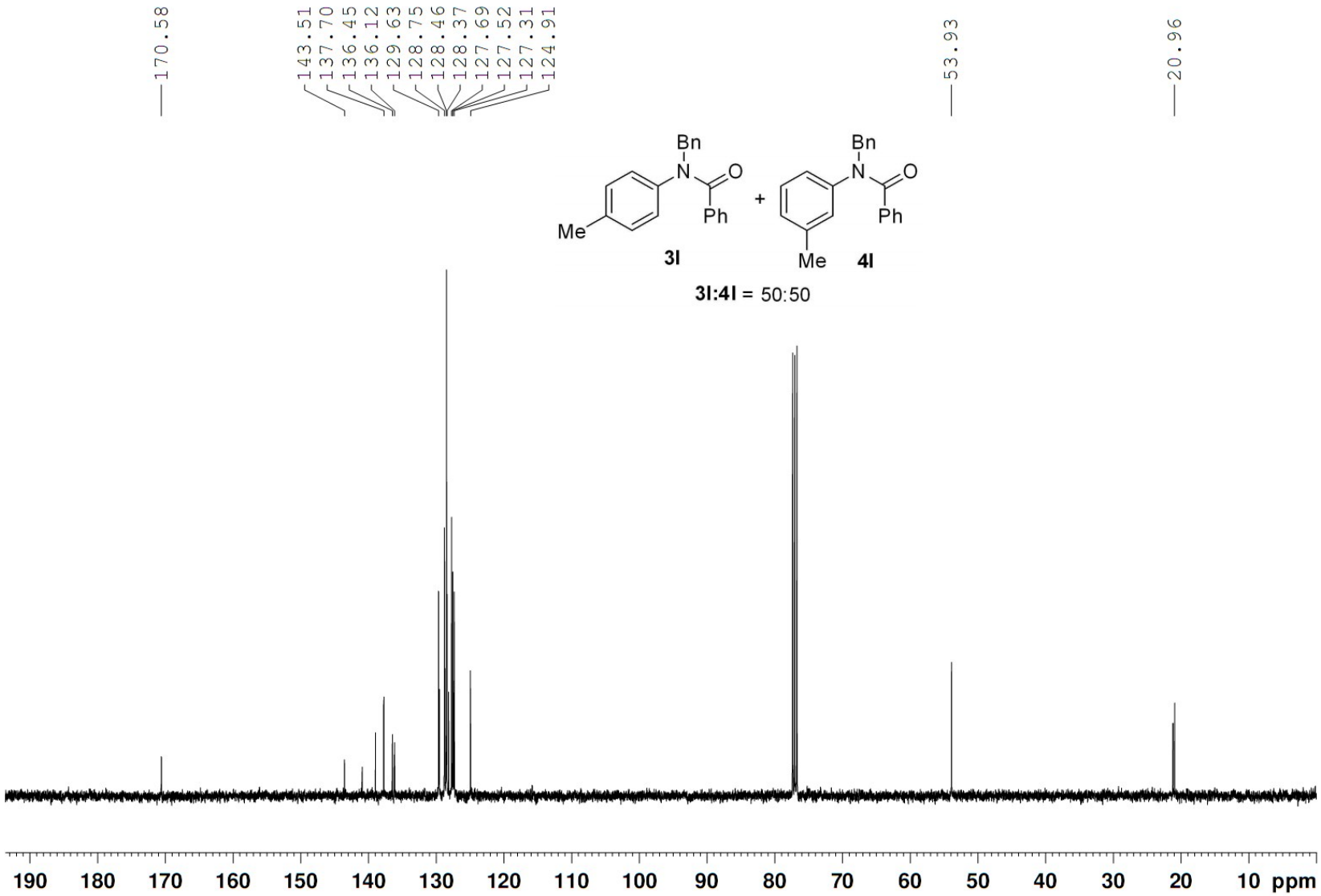
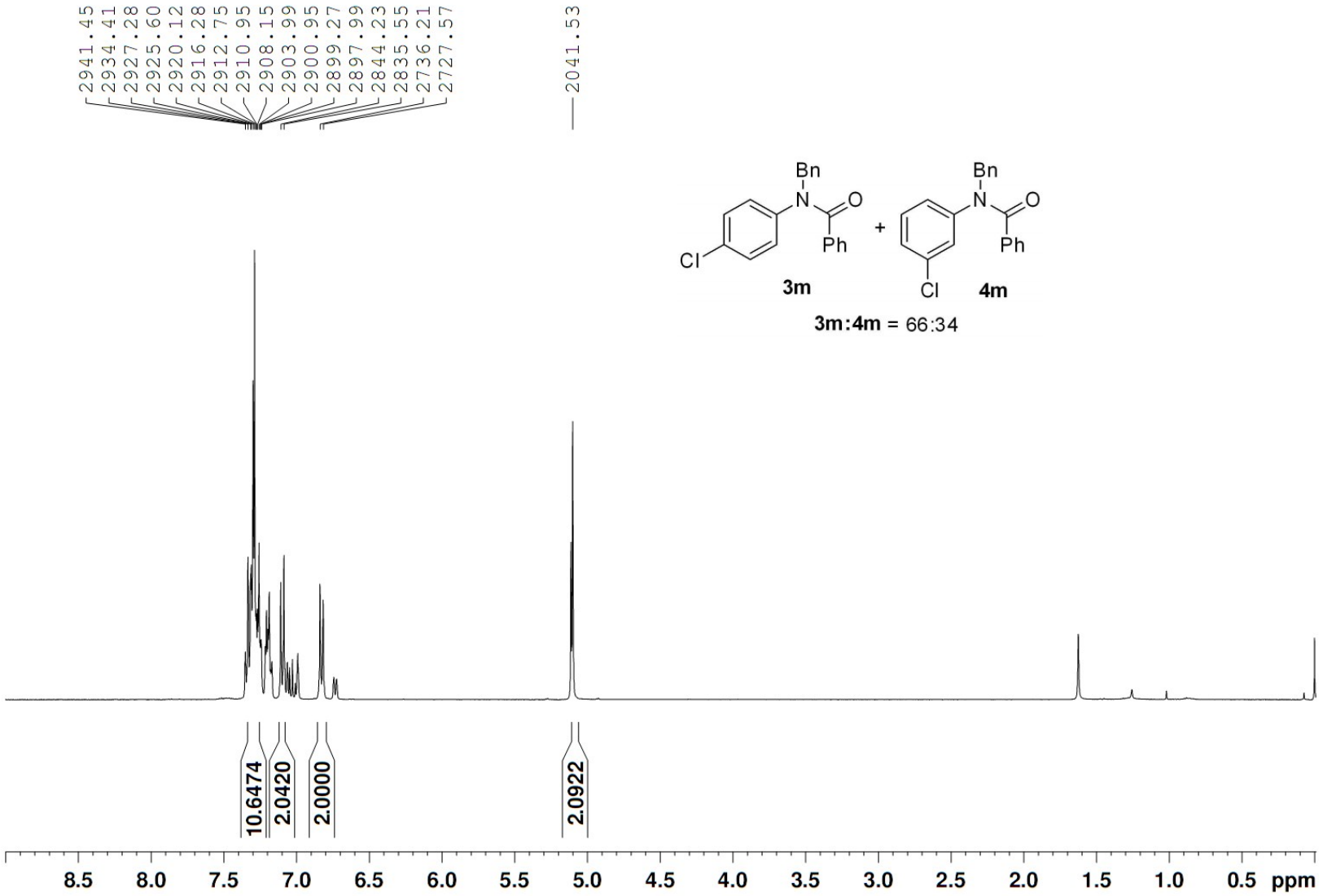
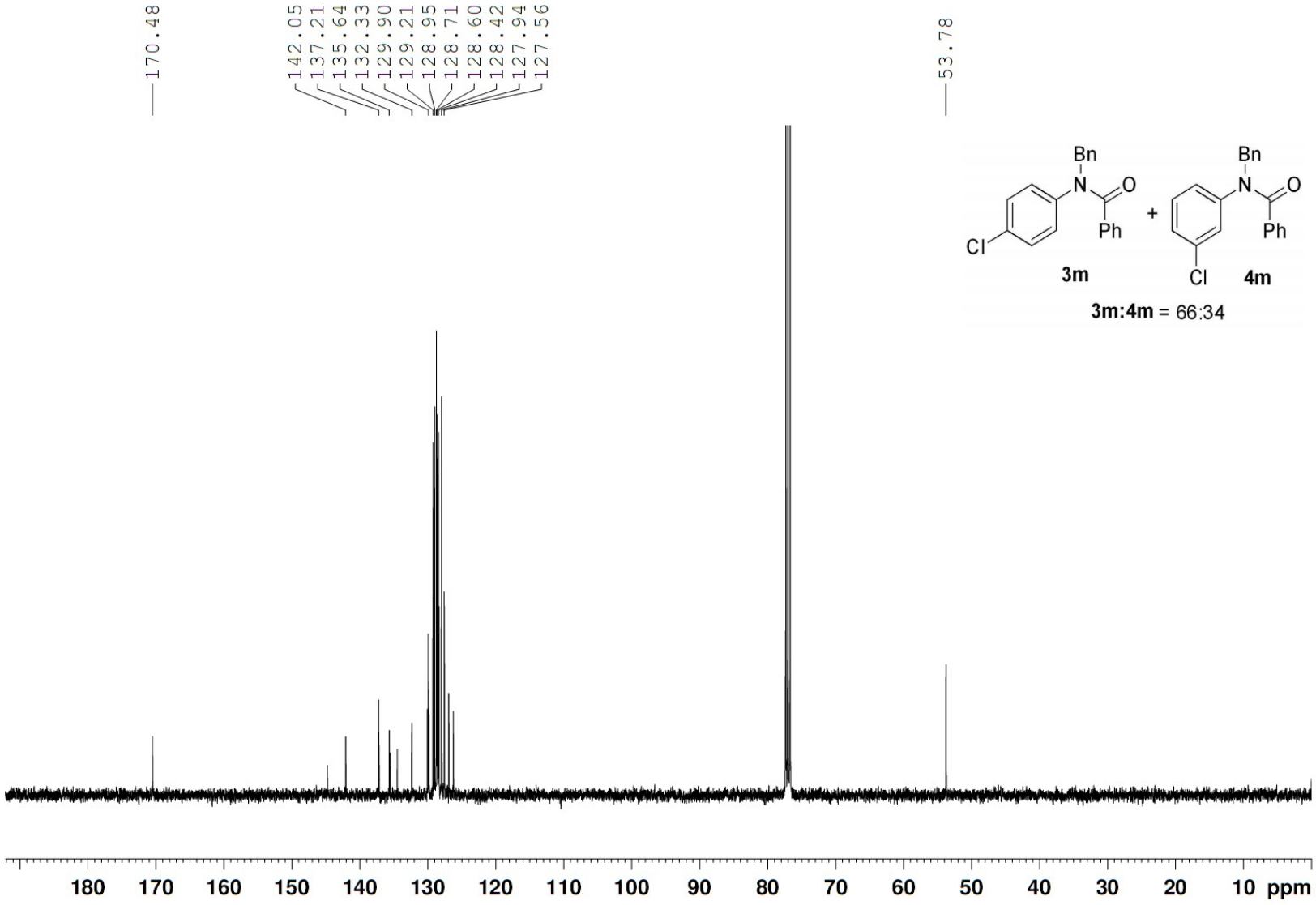
2975.35
2969.83
2962.02
2946.85
2939.53
2907.71
2870.73
2869.37
2862.12
1484.65
1477.05
1469.36
728.50
606.99
599.78
597.38
591.82
588.86
584.30
582.25
576.53
545.63
540.91
533.59
526.14
518.34
510.97
503.57
360.89
Ph
N
O
nBu
3k
3.0415
2.0000
2.0769
2.9576
2.0858
2.1458
3.0391
9.0
8.5
8.0
7.5
7.0
6.5
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

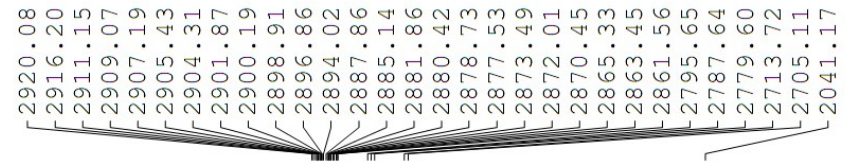
170.16
143.27
129.63
128.18
127.77
48.82
29.90
22.86
20.06
13.83
3k
Ph
nBu
N
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

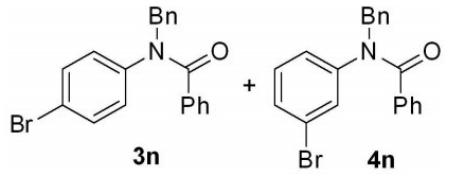

2940.56
2938.99
2938.07
2936.11
2933.59
2931.63
2929.39
2923.27
2922.47
2915.59
2913.63
2907.90
2901.94
2899.02
2894.98
2770.06
2761.86
2715.16
2706.76
2045.18
866.32
Bn
N
O
Ph
Me
3l
+
Bn
N
O
Ph
Me
4l
3l:4l = 50:50
10.1095
2.3192
2.1705
2.0514
3.0000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


170.58
143.51
137.70
136.45
136.12
129.63
128.75
128.46
128.37
127.69
127.52
127.31
124.91
53.93
20.96
Bn
N
O
Ph
Me
3l
+
Bn
N
O
Ph
Me
4l
3l:4l = 50:50
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm


2941.45
2934.41
2927.28
2925.60
2920.12
2916.28
2912.75
2910.95
2908.15
2903.99
2900.95
2899.27
2897.99
2844.23
2835.55
2736.21
2727.57
2041.53
Bn
N
O
Ph
Cl
3m
+
Bn
N
O
Ph
Cl
4m
3m:4m = 66:34
10.6474
2.0420
2.0000
2.0922
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

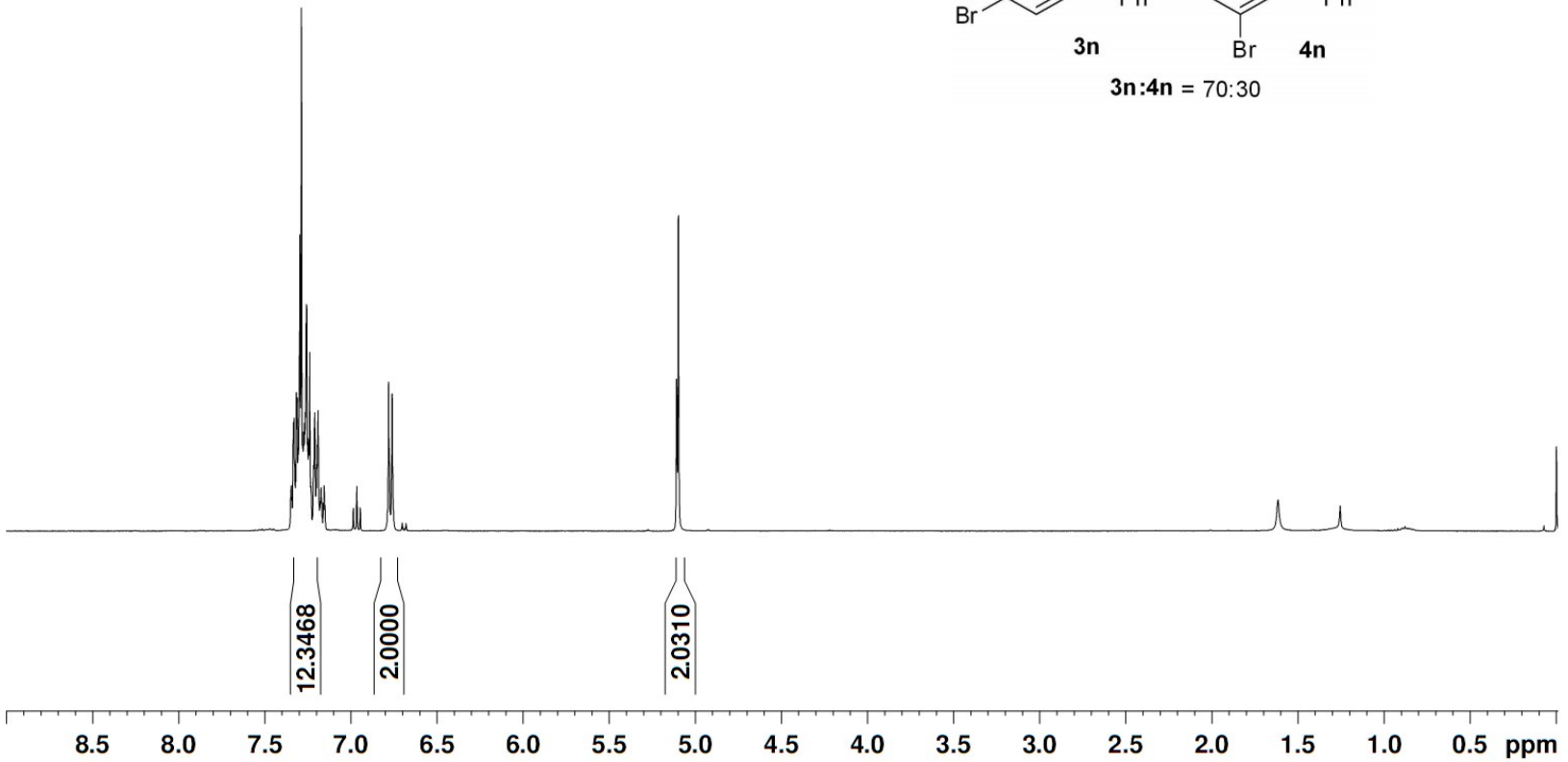

170.48
142.05
137.21
135.64
132.33
129.90
129.21
128.95
128.71
128.60
128.42
127.94
127.56
53.78
Bn
N
O
Ph
Cl
3m
+
Bn
N
O
Ph
Cl
4m
3m:4m = 66:34
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm

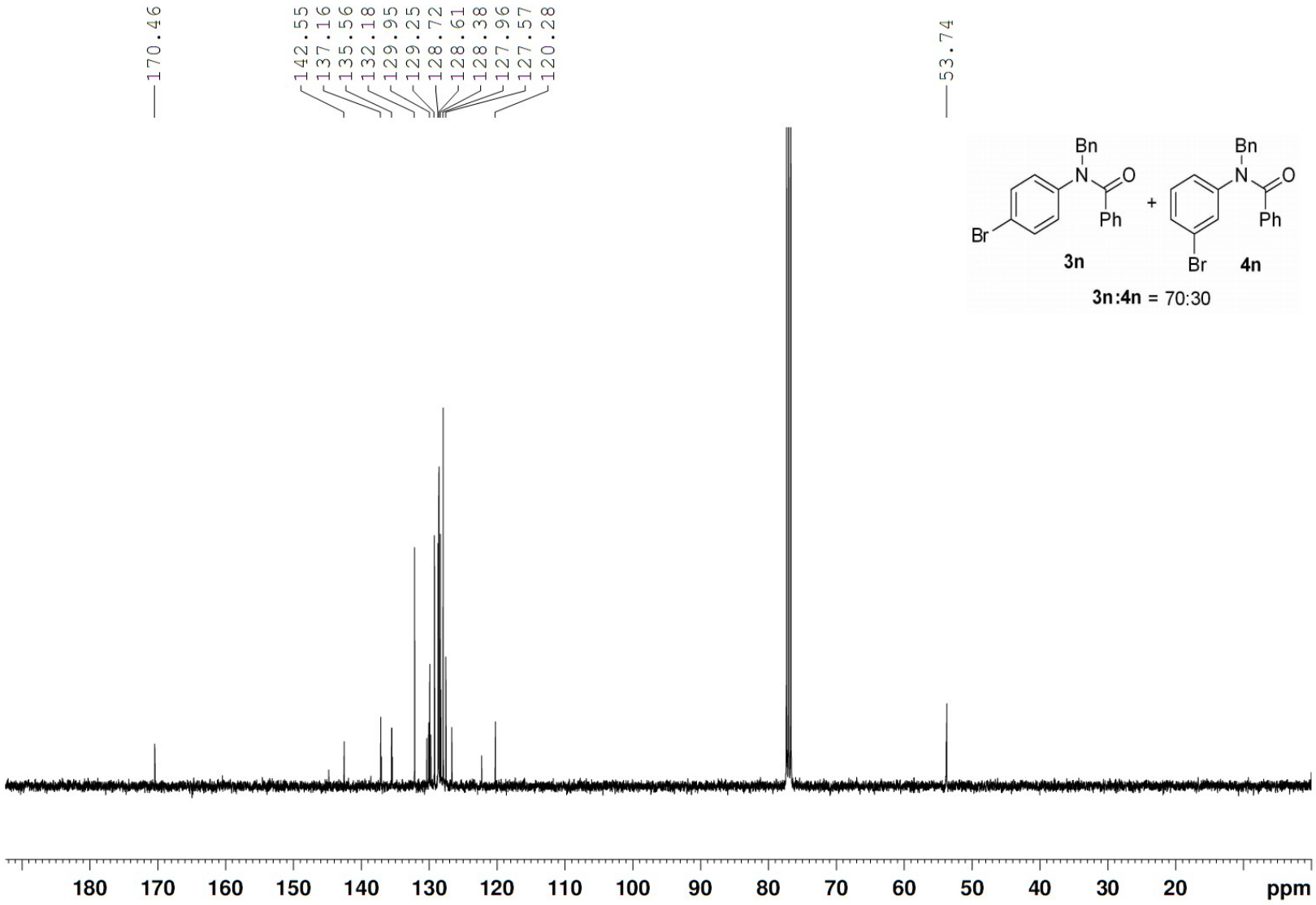


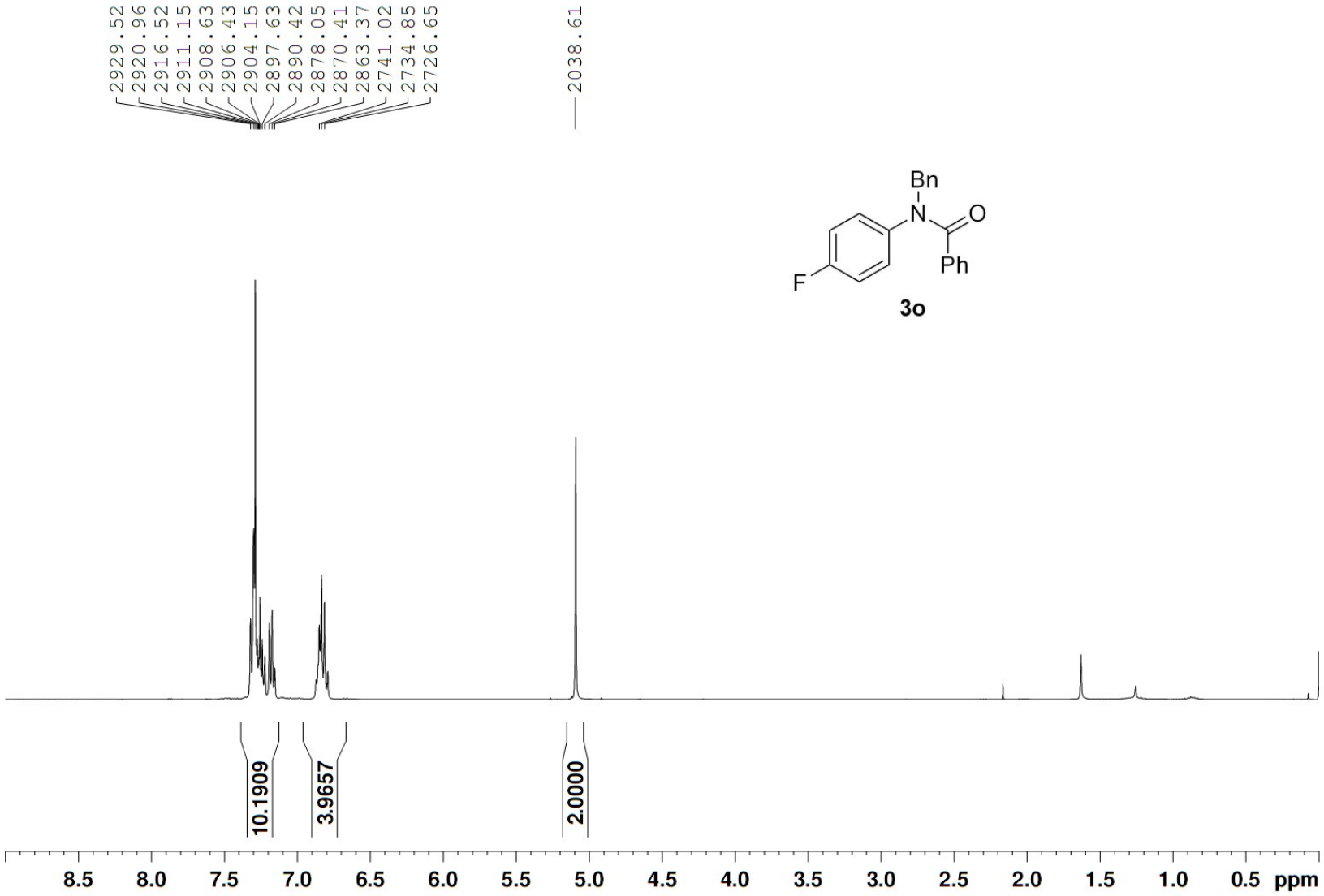


3n **4n**

3n:4n = 70:30




170.46
142.55
137.16
135.56
132.18
129.95
129.25
128.72
128.61
128.38
127.96
127.57
120.28
53.74
Bn
N
O
Ph
Br
3n
+
Bn
N
O
Ph
Br
4n
3n:4n = 70:30
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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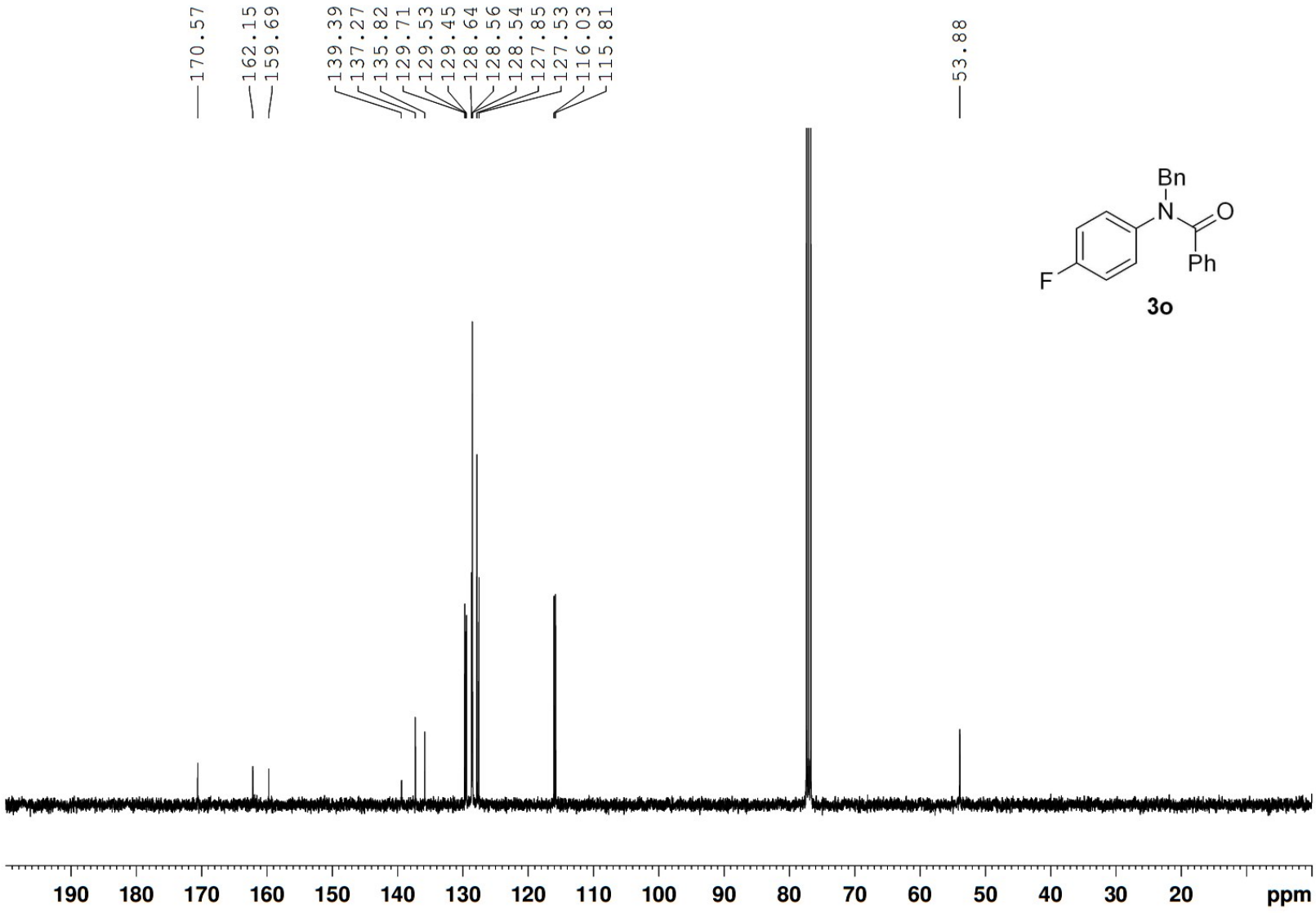
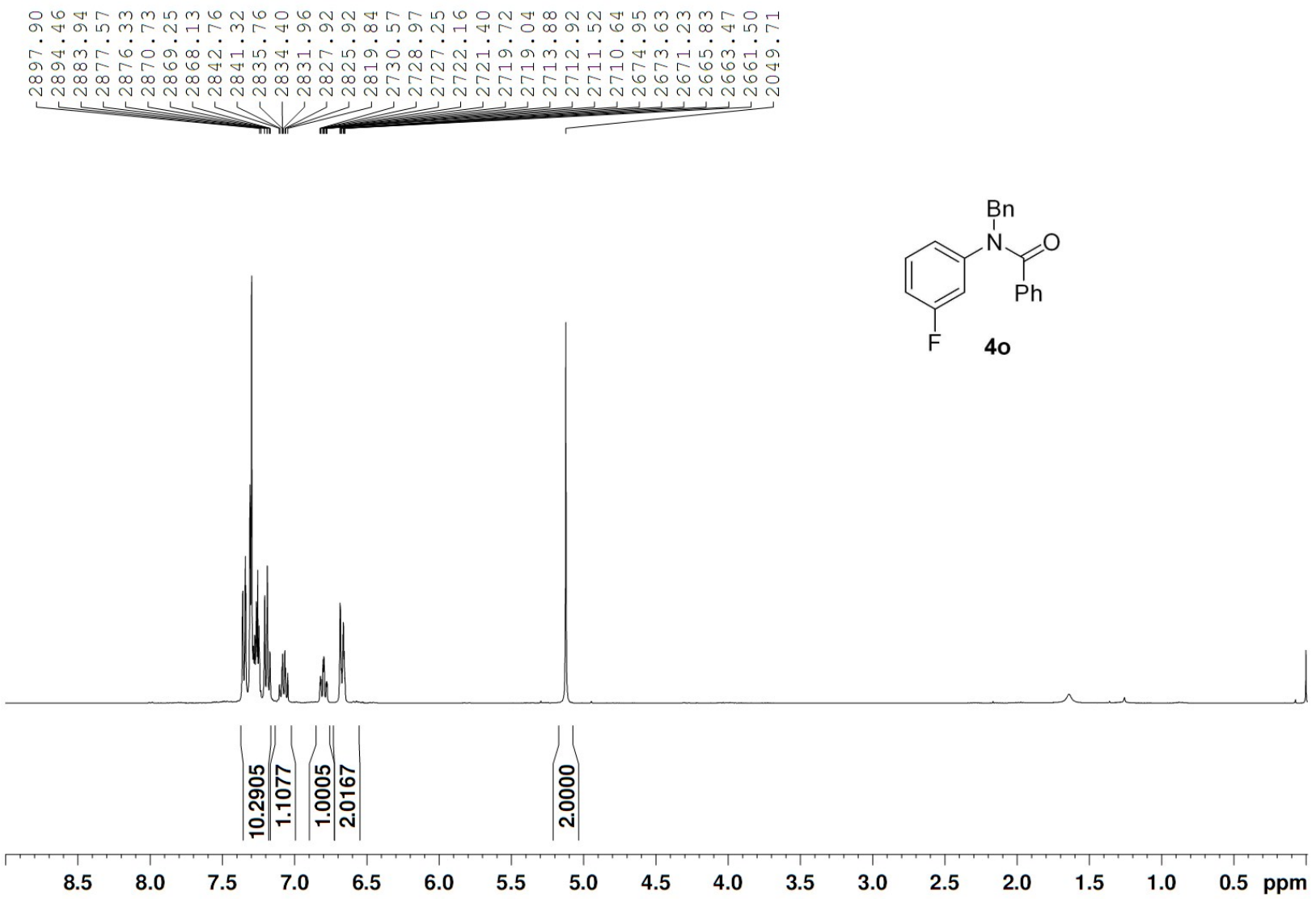
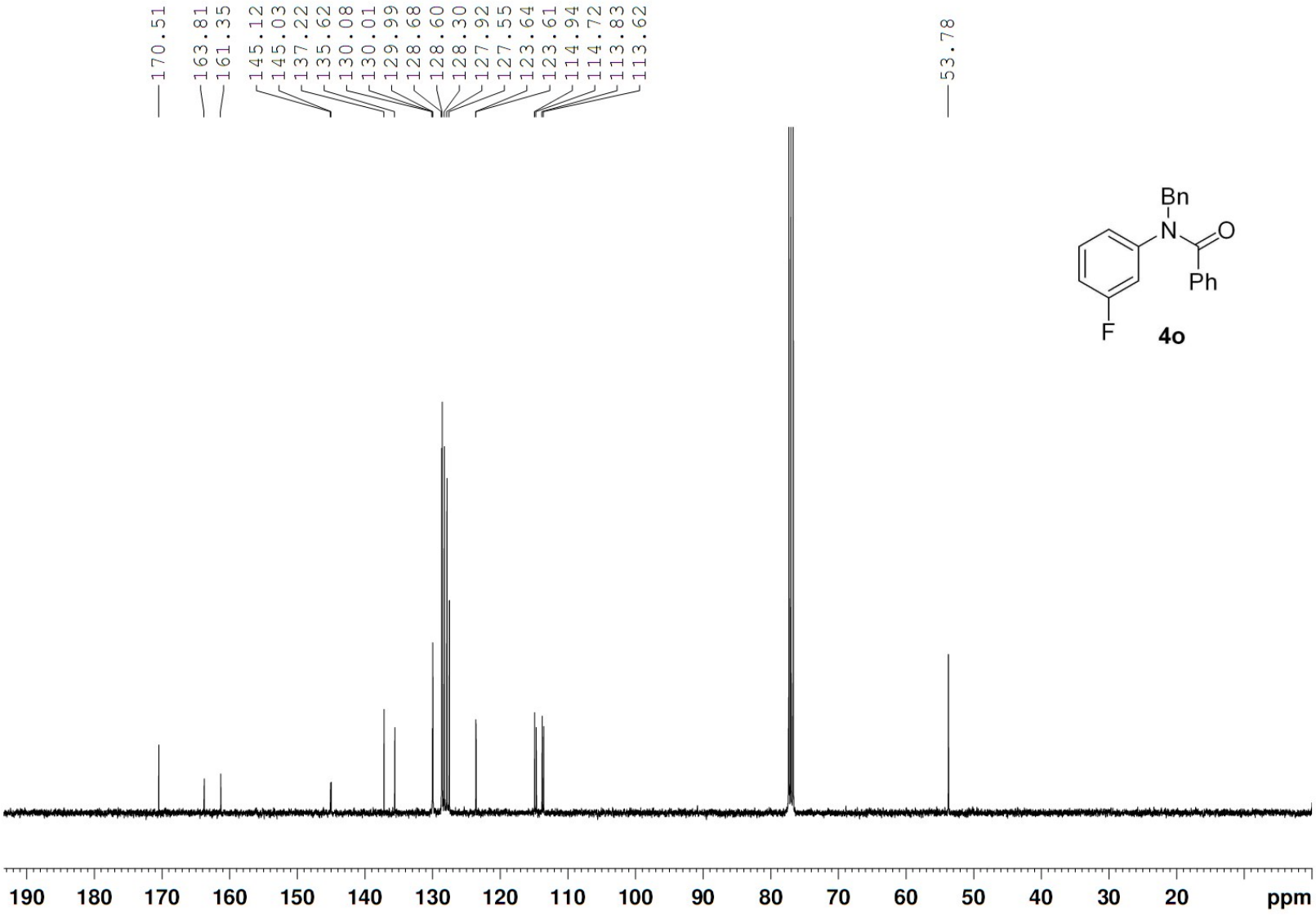
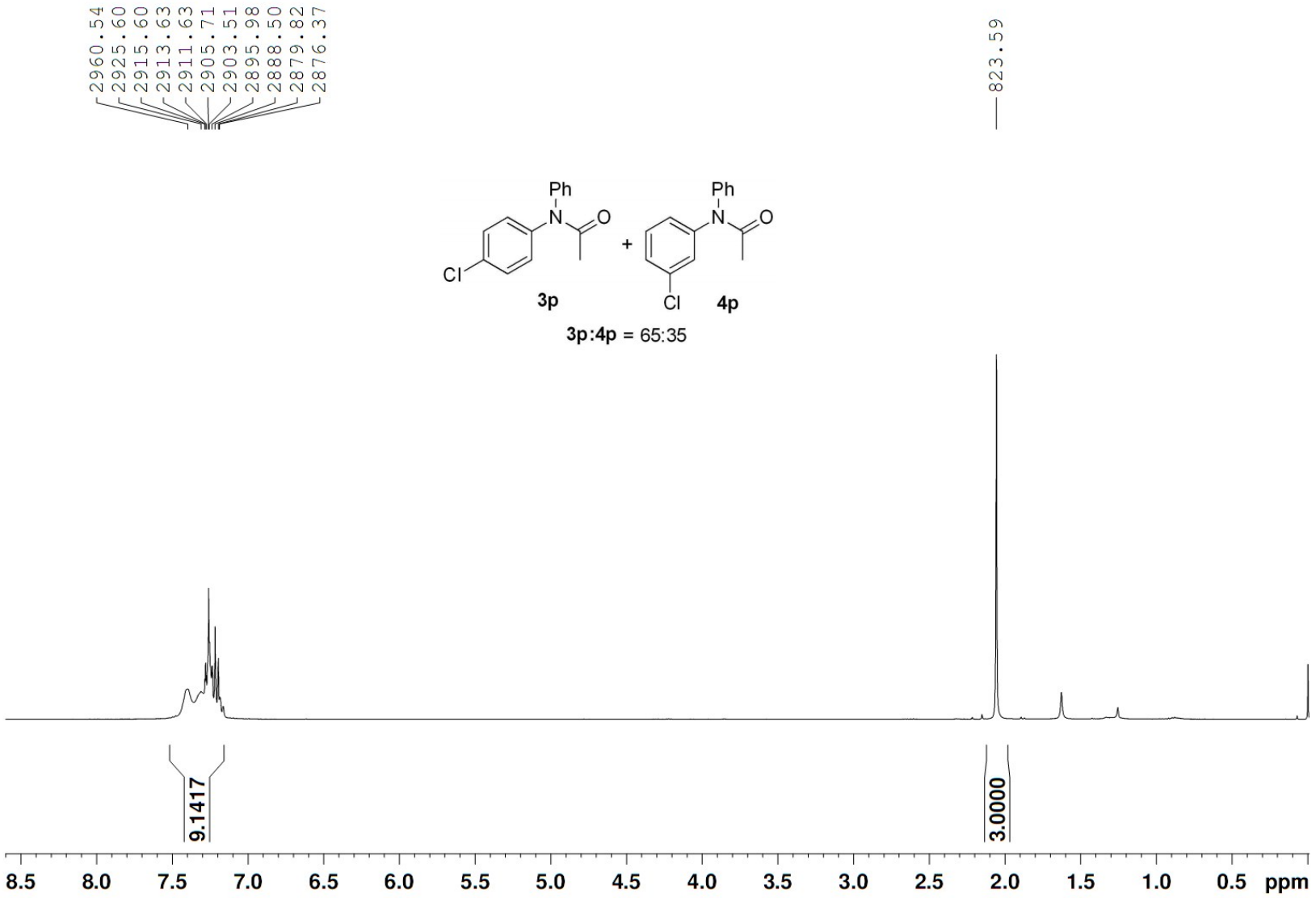
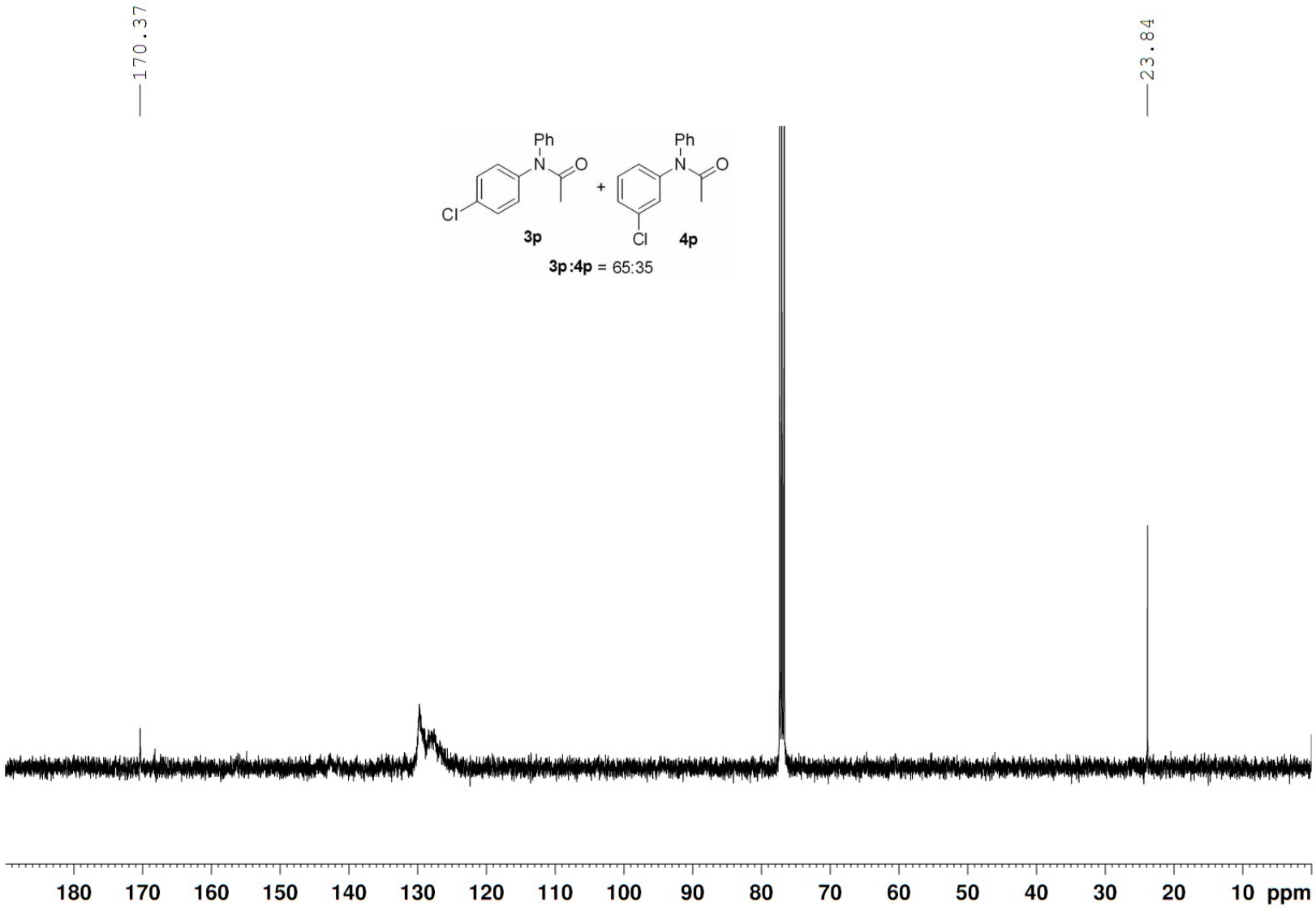
2920.96
2916.52
2911.15
2908.63
2906.43
2904.15
2897.63
2890.42
2878.05
2870.41
2863.37
2741.02
2734.85
2726.65
2038.61
Bn
N
O
Ph
F
3o
10.1909
3.9657
2.0000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

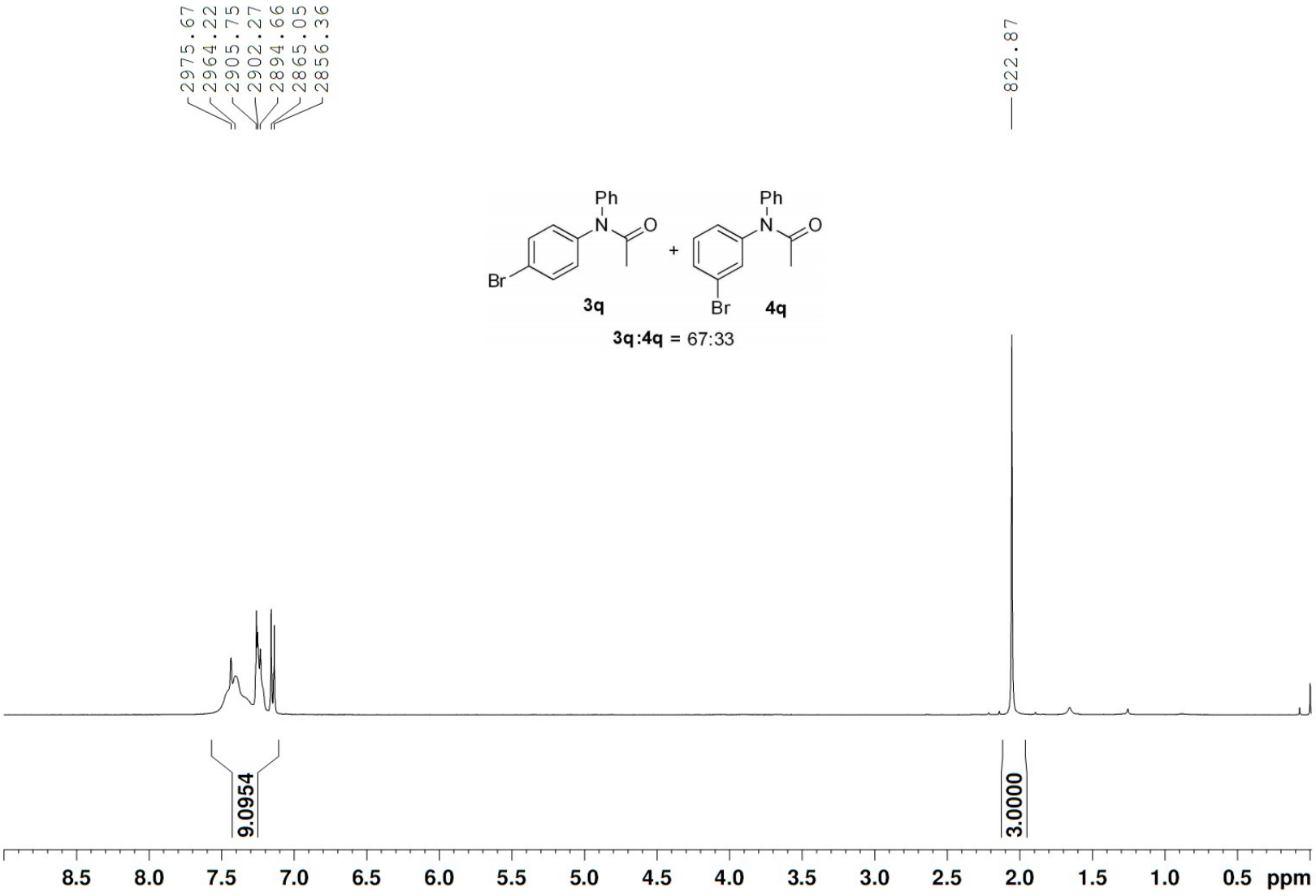

170.57
162.15
159.69
139.39
137.27
135.82
129.71
129.53
129.45
128.64
128.56
128.54
127.85
127.53
116.03
115.81
53.88
Bn
N
O
Ph
F
3o
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

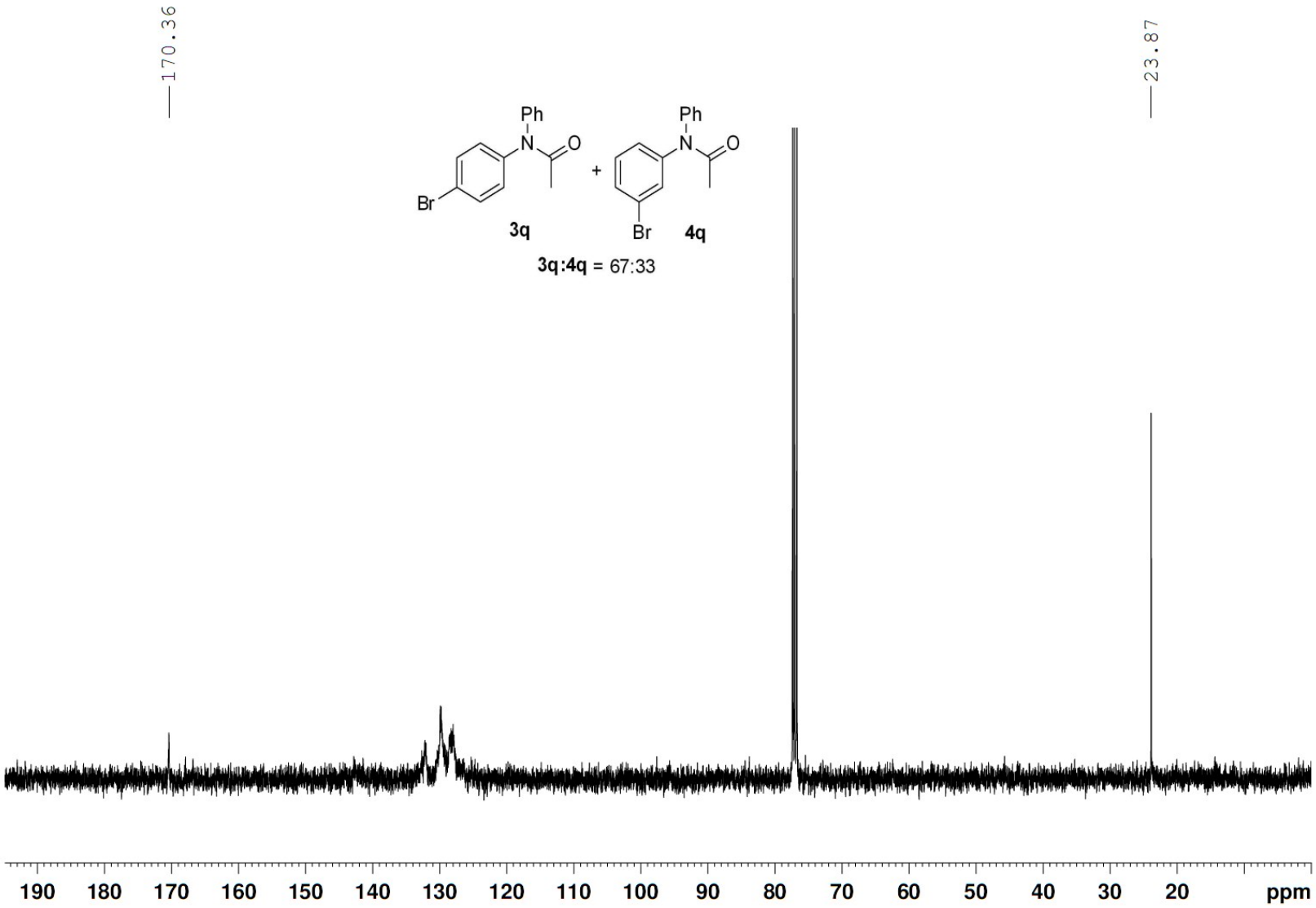

2897.90
2894.46
2883.94
2877.57
2876.33
2870.73
2869.25
2868.13
2842.76
2841.32
2835.76
2834.40
2831.96
2827.92
2825.92
2819.84
2730.57
2728.97
2727.25
2722.16
2721.40
2719.72
2719.04
2713.88
2712.92
2711.52
2710.64
2674.95
2673.63
2671.23
2665.83
2663.47
2661.50
2049.71
Bn
N
O
Ph
F
4o
10.2905
1.1077
1.0005
2.0167
2.0000
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

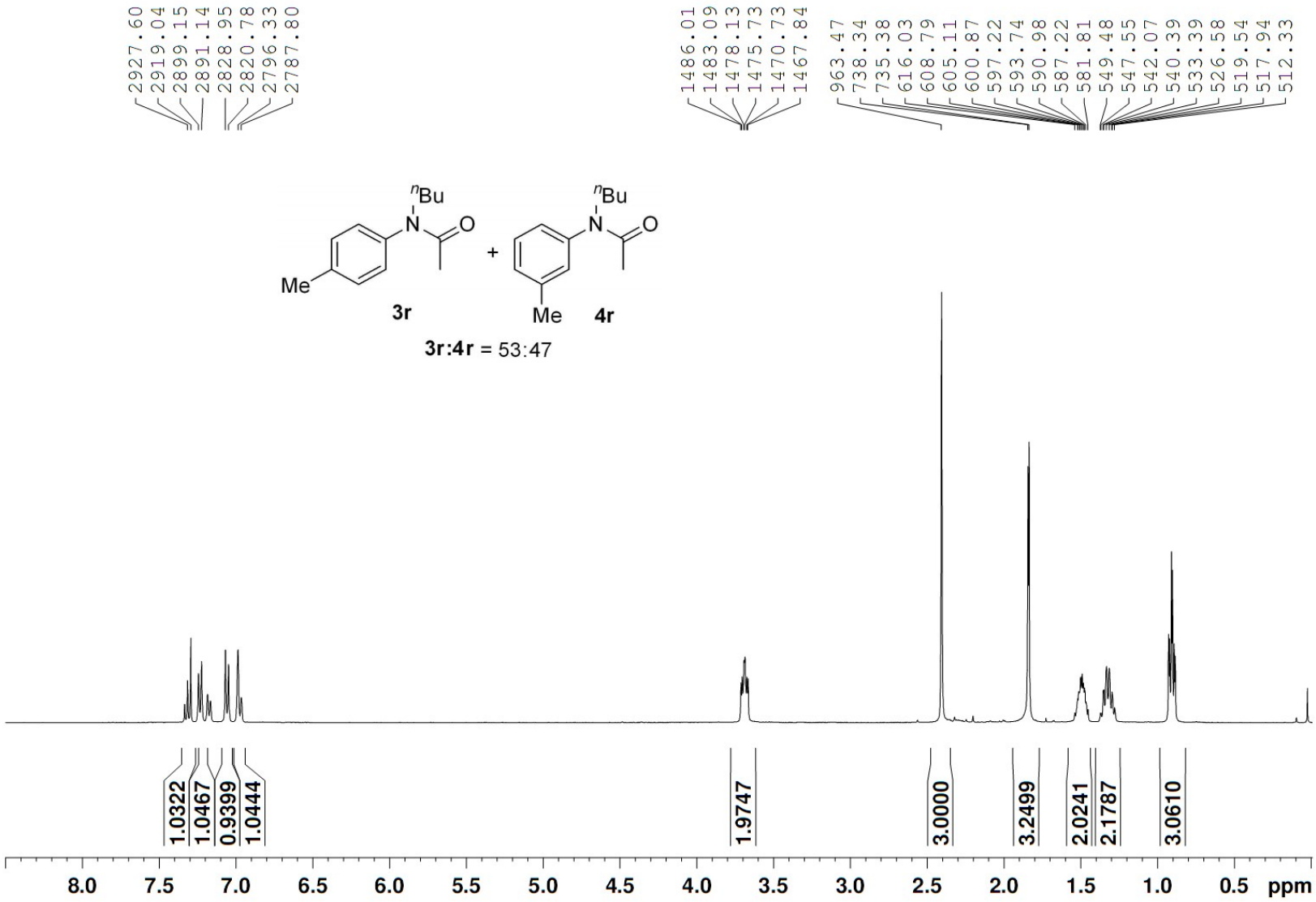

170.51
163.81
161.35
145.12
145.03
137.22
135.62
130.08
130.01
129.99
128.68
128.60
128.30
127.92
127.55
123.64
123.61
114.94
114.72
113.83
113.62
53.78
Bn
N
O
Ph
F
4o
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

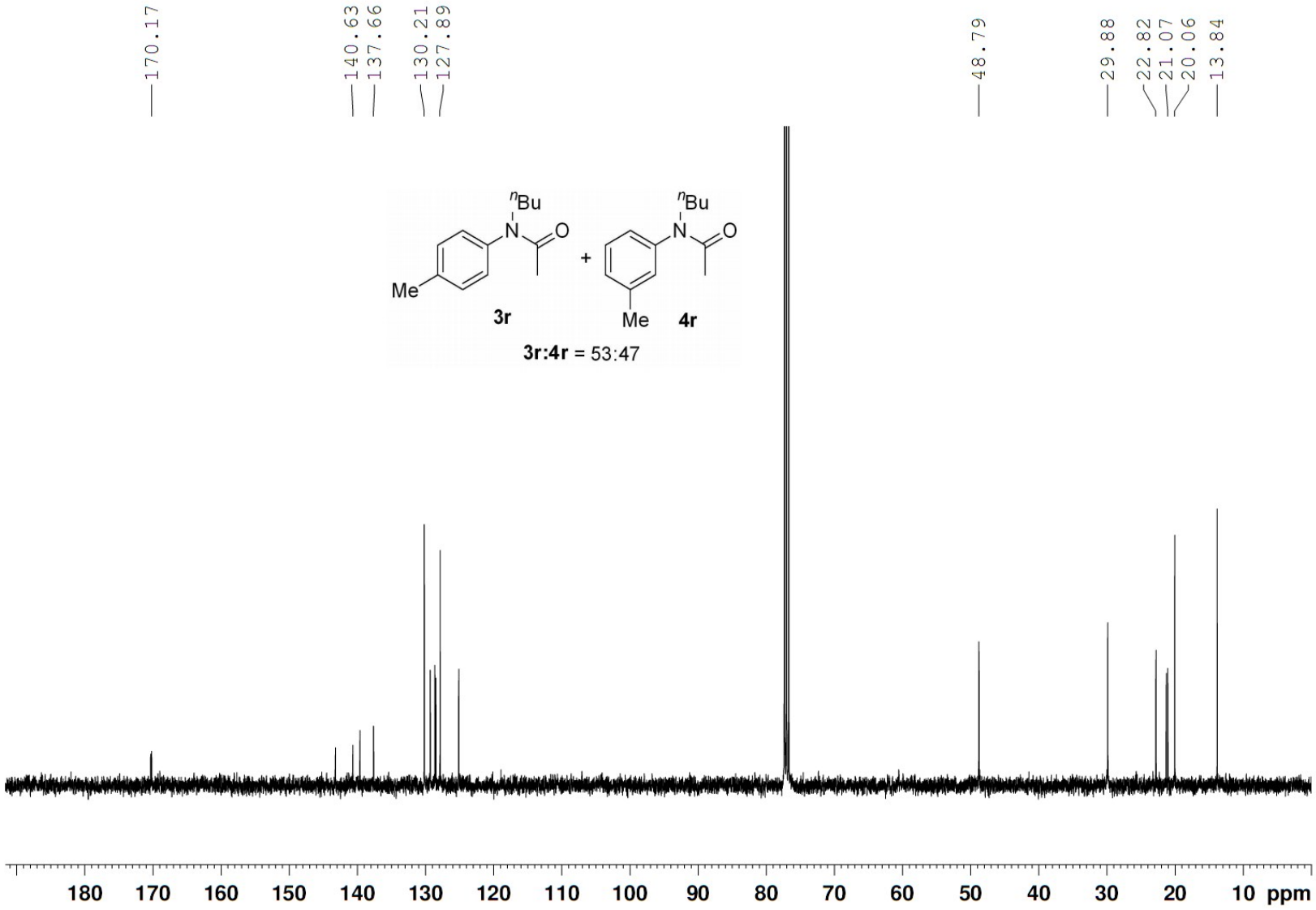

2960.54
2925.60
2915.60
2913.63
2911.63
2905.71
2903.51
2895.98
2888.50
2879.82
2876.37
823.59
Ph
N
O
Cl
3p
+
Ph
N
O
Cl
4p
3p:4p = 65:35
9.1417
3.0000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


170.37
23.84
Ph
N
O
Cl
3p
+
Ph
N
O
Cl
4p
3p:4p = 65:35
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


2975.67
2964.22
2905.75
2902.27
2894.66
2865.05
2856.36
822.87
Ph
N
O
Br
3q
+
Ph
N
O
Br
4q
3q:4q = 67:33
9.0954
3.0000
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


170.36
23.87
Ph
N
O
Br
3q
+
Ph
N
O
Br
4q
3q:4q = 67:33
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


2927.60
2919.04
2899.15
2891.14
2828.95
2820.78
2796.33
2787.80
1486.01
1483.09
1478.13
1475.73
1470.73
1467.84
963.47
738.34
735.38
616.03
608.79
605.11
600.87
597.22
593.74
590.98
587.22
581.81
549.48
547.55
542.07
540.39
533.39
526.58
519.54
517.94
512.33
nBu
N
O
Me
3r
+
nBu
N
O
Me
4r
3r:4r = 53:47
1.0322
1.0467
0.9399
1.0444
1.9747
3.0000
3.2499
2.0241
2.1787
3.0610
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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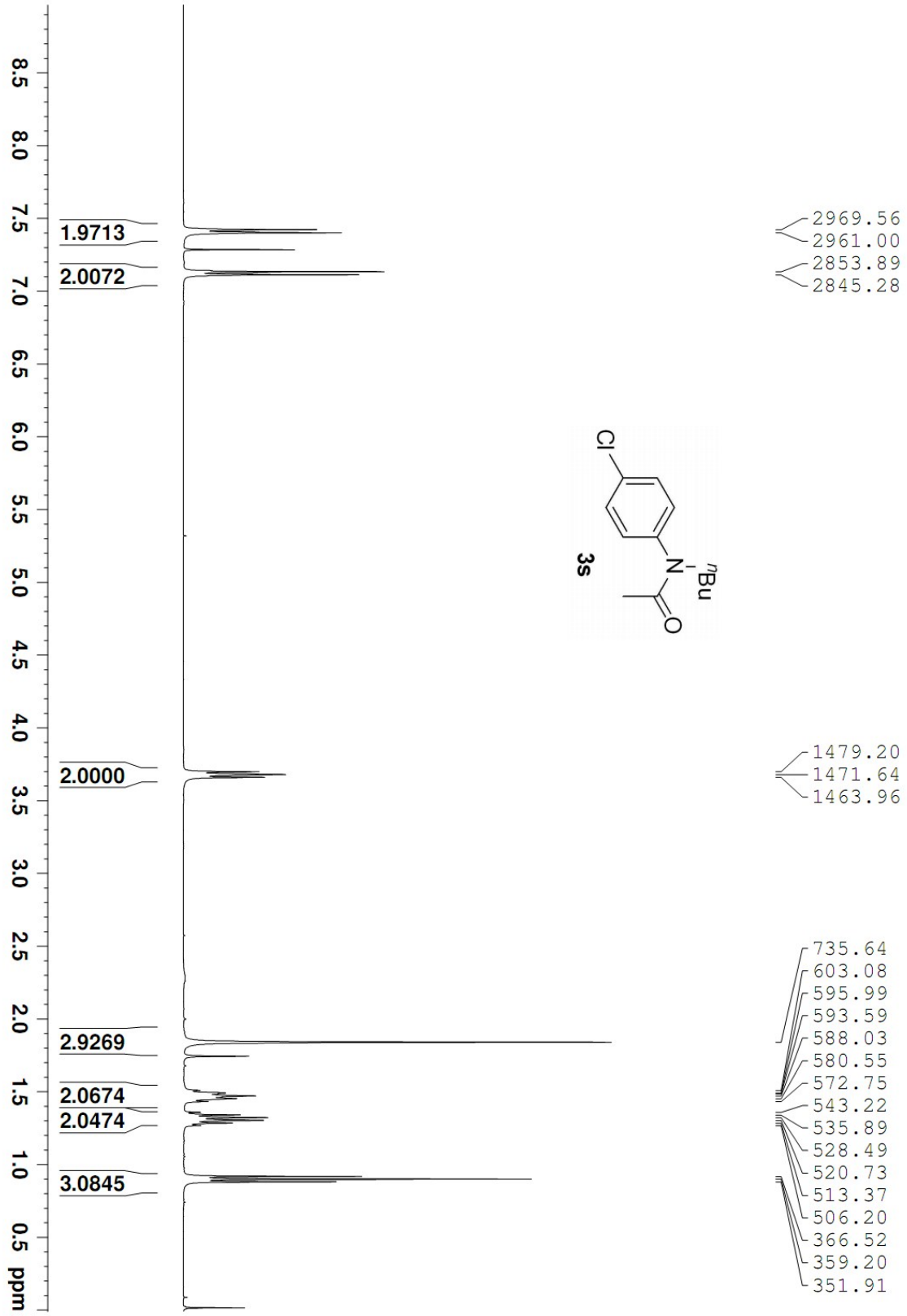
140.63
137.66
130.21
127.89
48.79
29.88
22.82
21.07
20.06
13.84
nBu
N
O
Me
3r
+
nBu
N
O
Me
4r
3r:4r = 53:47
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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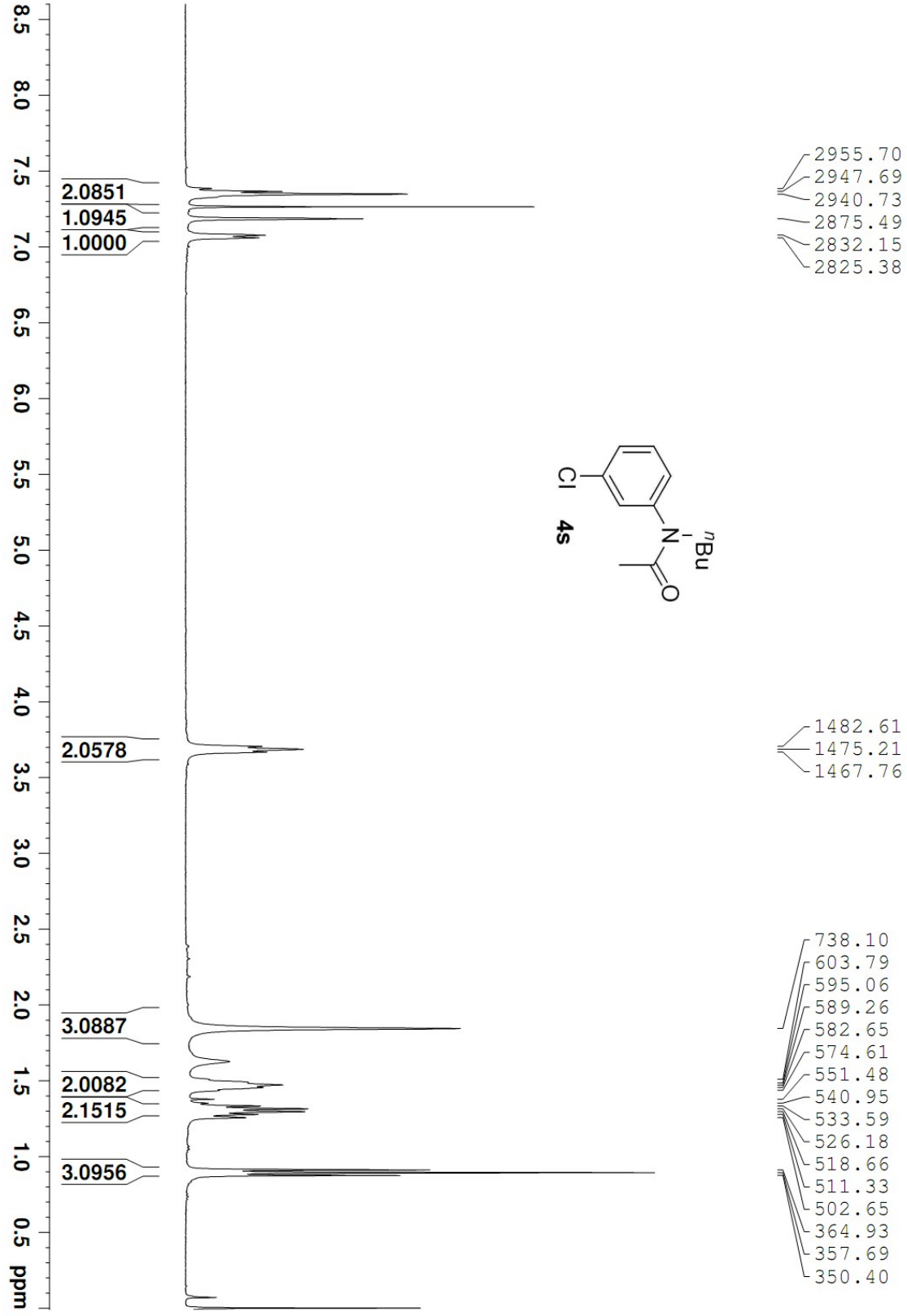
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.9713
2.0072
2.0000
2.9269
2.0674
2.0474
3.0845
Cl
N
nBu
O
3s
2969.56
2961.00
2853.89
2845.28
1479.20
1471.64
1463.96
735.64
603.08
595.99
593.59
588.03
580.55
572.75
543.22
535.89
528.49
520.73
513.37
506.20
366.52
359.20
351.91

169.90
141.75
133.64
129.88
129.50
48.80
29.84
22.84
20.00
13.80
Cl
N
nBu
O
3s
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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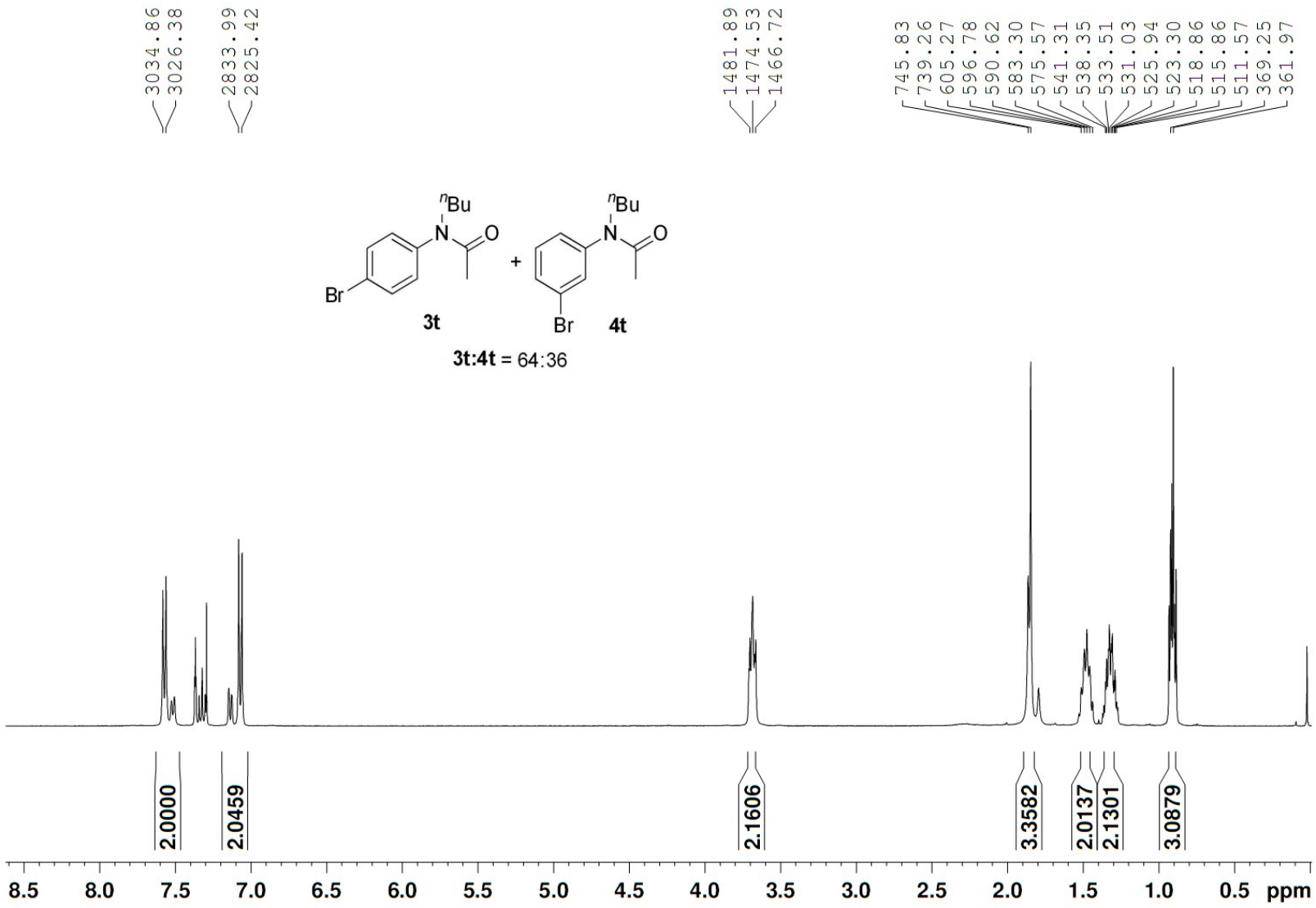
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.0851
1.0945
1.0000
2.0578
3.0887
2.0082
2.1515
3.0956
Cl
4s
N
nBu
O
2955.70
2947.69
2940.73
2875.49
2832.15
2825.38
1482.61
1475.21
1467.76
738.10
603.79
595.06
589.26
582.65
574.61
551.48
540.95
533.59
526.18
518.66
511.33
502.65
364.93
357.69
350.40

170.25
144.48
135.08
130.60
128.50
128.12
126.60
48.89
29.90
22.92
20.02
13.80
Cl
4s
N
nBu
O
ppm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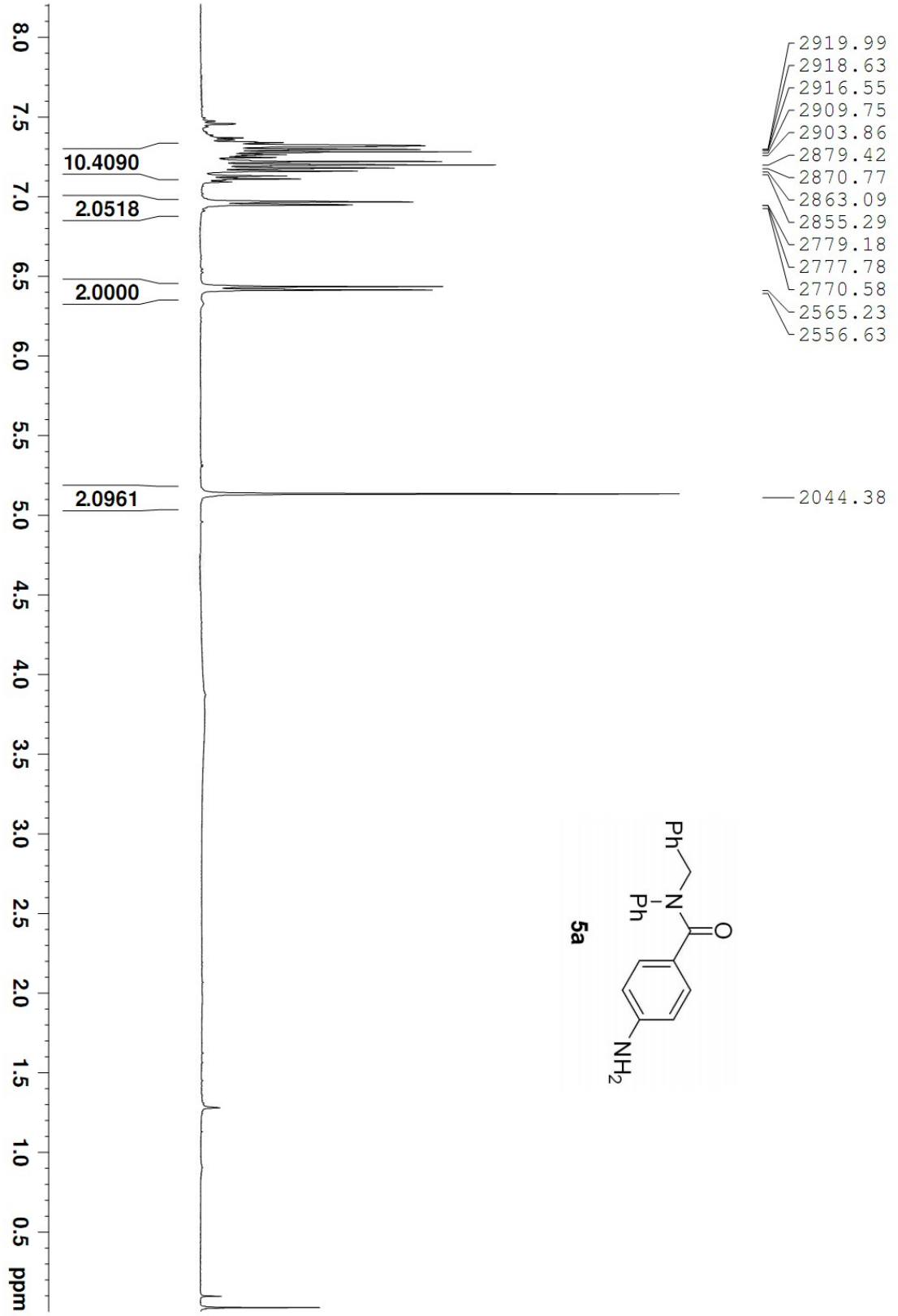
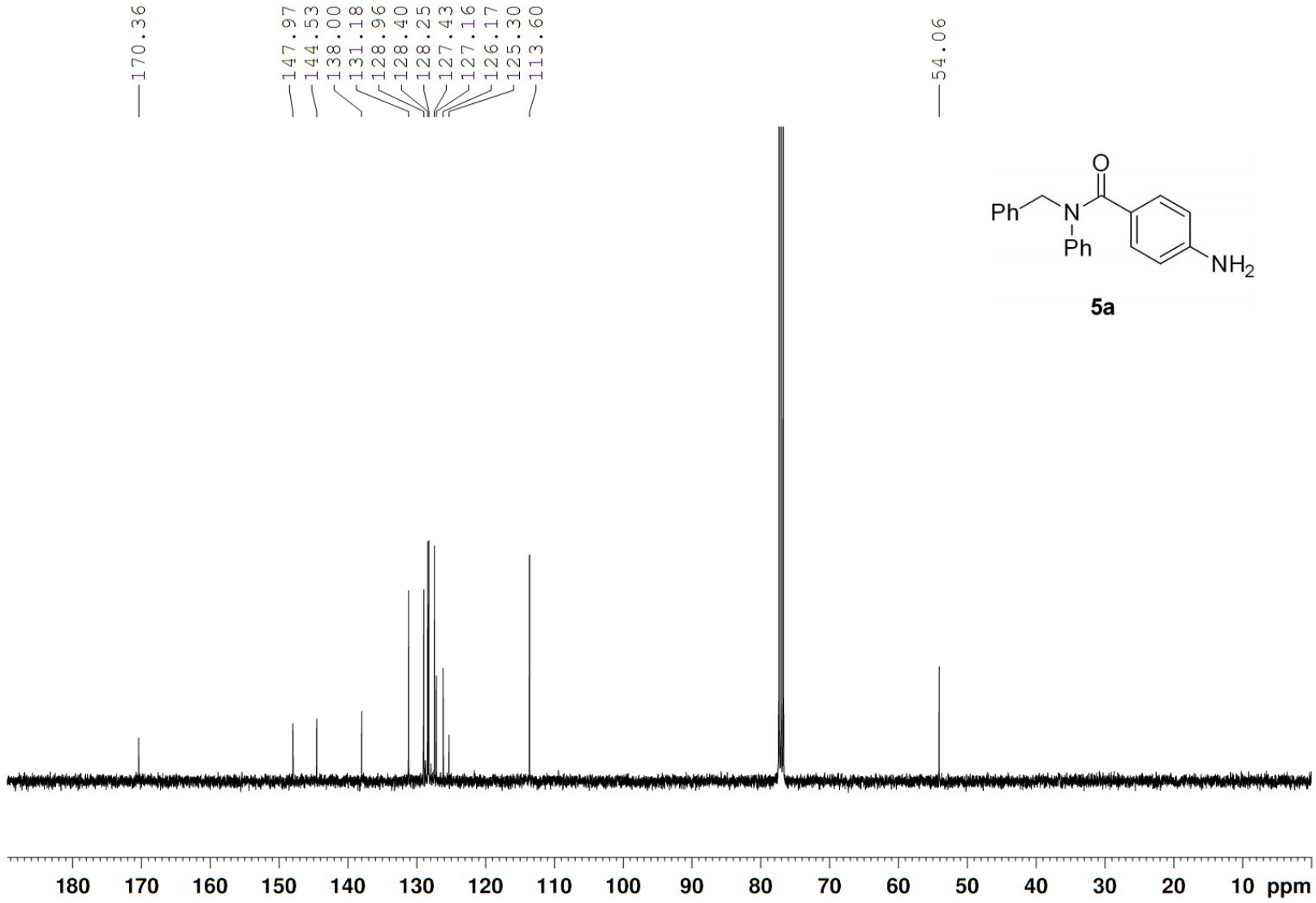
3026.38
2833.99
2825.42
1481.89
1474.53
1466.72
745.83
739.26
605.27
596.78
590.62
583.30
575.57
541.31
538.35
533.51
531.03
525.94
523.30
518.86
515.86
511.57
369.25
361.97
nBu
N
O
Br
3t
+
nBu
N
O
Br
4t
3t:4t = 64:36
2.0000
2.0459
2.1606
3.3582
2.0137
2.1301
3.0879
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

169.83
142.29
132.89
129.86
127.02
48.79
29.85
22.85
20.00
13.79
3t
3t:4t = 64:36
4t
Br
N
nBu
O
+
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

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
0.9210
2.0000
2.8251
2.1493
2.0609
3.1586
2866.29
1484.85
1477.25
1469.56
728.26
606.83
599.62
597.26
591.66
588.74
584.14
576.37
540.79
533.47
526.02
518.26
510.89
503.49
360.77
353.44
346.12
3u
D
D
D
D
H
N
nBu
O

170.16
143.20
128.05
48.82
29.91
22.87
20.06
13.84
3u
D
D
D
D
H
N
nBu
O
ppm


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
10.4090
2.0518
2.0000
2.0961
2919.99
2918.63
2916.55
2909.75
2903.86
2879.42
2870.77
2863.09
2855.29
2779.18
2777.78
2770.58
2565.23
2556.63
2044.38
Ph
N
Ph
O
NH2
5a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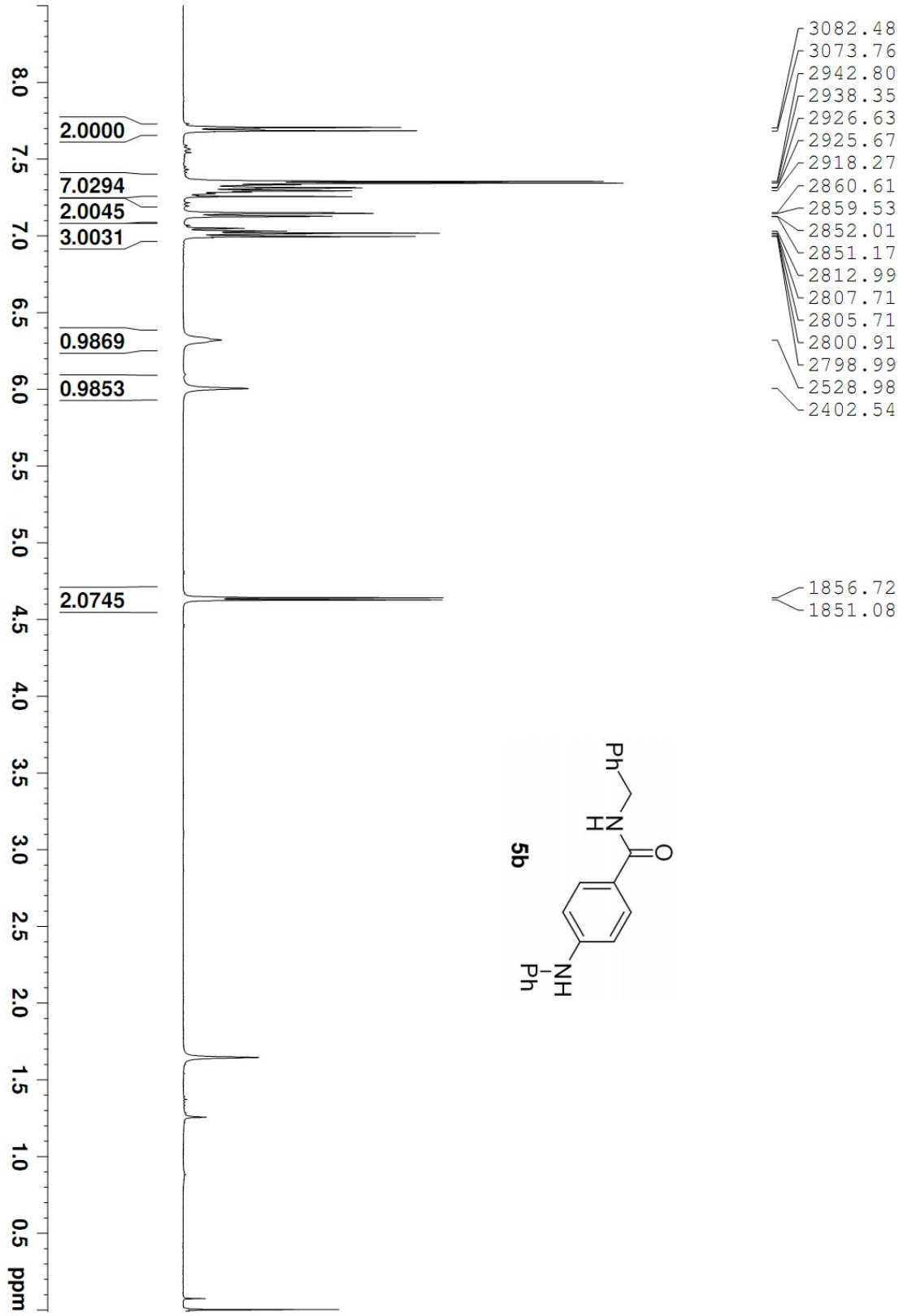
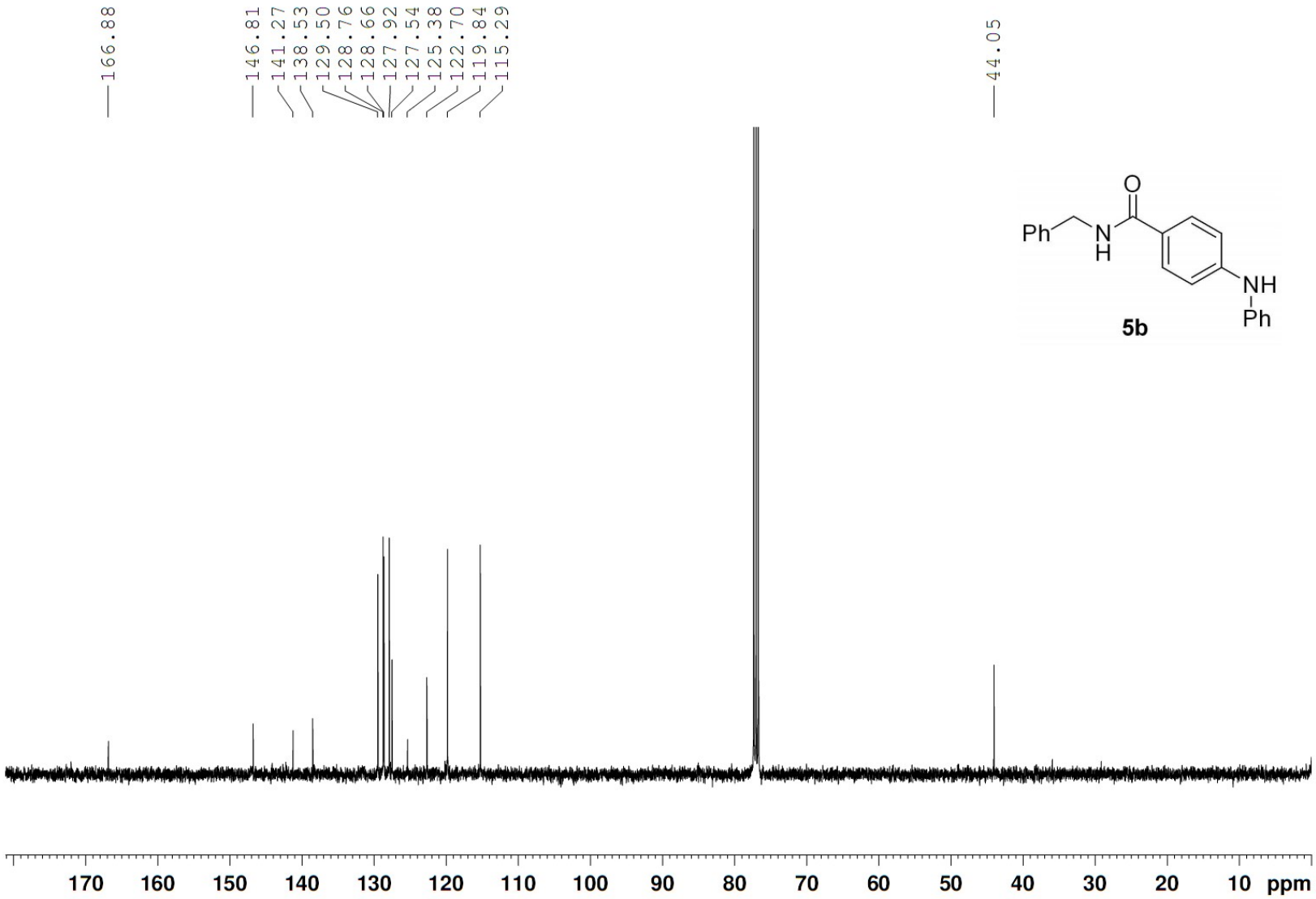
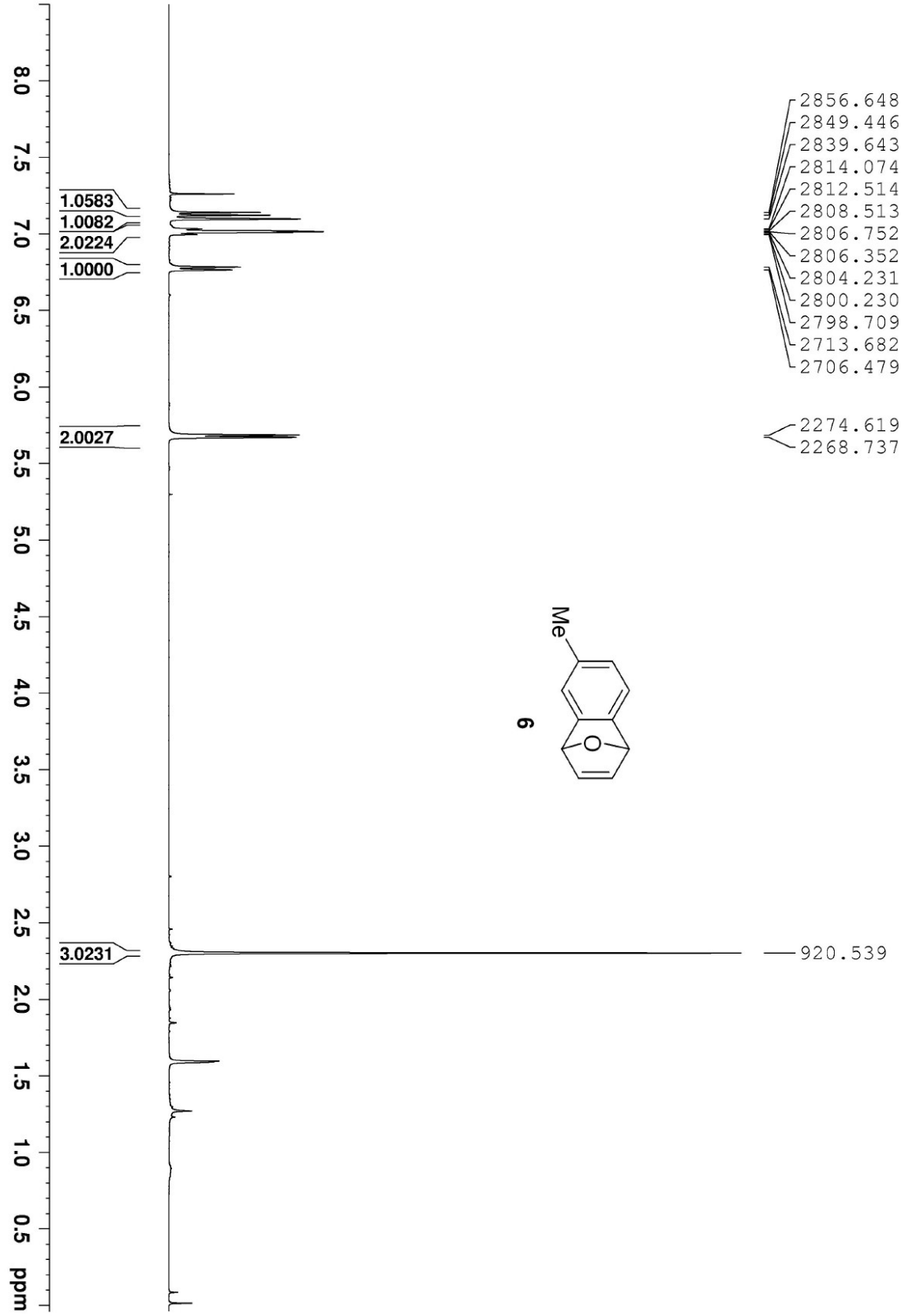
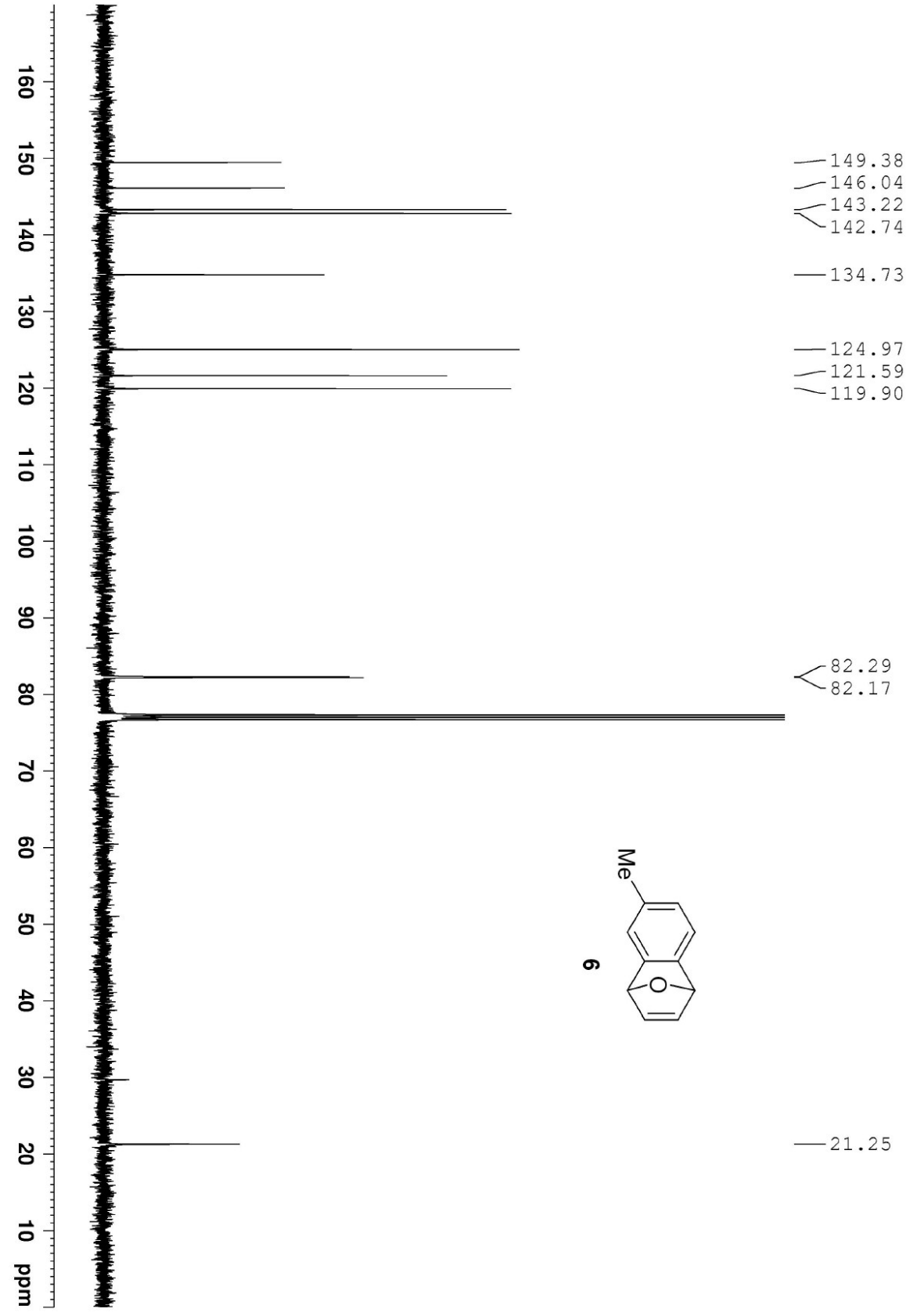
147.97
144.53
138.00
131.18
128.96
128.40
128.25
127.43
127.16
126.17
125.30
113.60
54.06
Ph
N
Ph
O
NH2
5a
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


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
2.0000
7.0294
2.0045
3.0031
0.9869
0.9853
2.0745
3082.48
3073.76
2942.80
2938.35
2926.63
2925.67
2918.27
2860.61
2859.53
2852.01
2851.17
2812.99
2807.71
2805.71
2800.91
2798.99
2528.98
2402.54
1856.72
1851.08
Ph
N
H
O
NH
Ph
5b


166.88
146.81
141.27
138.53
129.50
128.76
128.66
127.92
127.54
125.38
122.70
119.84
115.29
44.05
Ph
N
H
O
NH
Ph
5b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


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
1.0583
1.0082
2.0224
1.0000
2.0027
3.0231
2856.648
2849.446
2839.643
2814.074
2812.514
2808.513
2806.752
2806.352
2804.231
2800.230
2798.709
2713.682
2706.479
2274.619
2268.737
920.539
Me
O
6


160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
149.38
146.04
143.22
142.74
134.73
124.97
121.59
119.90
82.29
82.17
21.25
Me
O
6