Supporting Information

Palladium-Catalyzed Synthesis of 2,3-Diaryl-*N*-methylindoles from *ortho*-Alkynylanilines and Aryl Pinacol Boronic Esters

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1. Experimental and General Procedure

General information

All commercial available chemicals were used as received. Experiments were conducted under N_2 atmosphere by using standard Schlenk techniques. Melting points were determined by Büchi 535 apparatus. ^{1}H, ^{13}C and ^{19}F NMR spectra were measured at Varian Unity Inova-600 or a Varian Mercury-400 instrument by using $CDCl_3$ solvent. Chemical shifts are indicated in δ values with reference to TMS and $CDCl_3$ internal standards. ^{19}F chemical shifts were determined in δ values by using $PhCF_3$ internal standard. Coupling constant value are measured in hertz (Hz). Standard abbreviations for multiplicity are given as s = singlet, d = doublet, t = triplet and m = multiplet. GC-MS analysis were obtained on a Agilent Technologies 5977A GC equipped with Agilent 7890B MS. High-resolution mass spectra (HRMS) were undertaken on a Jeol JMS-HX 110 spectrometer by the services given at National Chung Hsing University. Column chromatography was carried out on Merck silica gel 60 (230–400 mesh). Aminoalkyne and Aryl boronic esters were all synthesized through literature procedures.[1]

General procedure for synthesis of compound 3

Aminoalkyne (0.2 mmol, 1 equiv), arylpinacol boronic ester (0.3 mmol, 1.5 equiv), $Pd(OAc)_2$ (2.25 mg, 5 mol %), and $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv) was charged under nitrogen atmosphere and the solvent ethanol (1.5 mL) was added. The resulting mixture was stirred at 74 °C for 12 h. Then, the reaction mixture was cooled down to room temperature and diluted with ethyl acetate (20 mL), and it was filtered through a celite pad and the filtrate was concentrated under vacuum. The obtained crude residue was purified by silica gel column chromatography.

General procedure for synthesis of compound 5

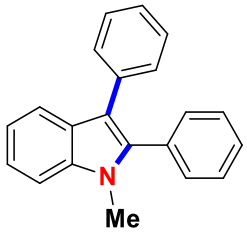
A Schlenk tube with Teflon coated magnetic stir bar was charged with B_2pin_2 (0.75 mmol), $[Ir(OMe)(cod)]_2$ (1 mol %), dtbpy (2 mol %), arenes (1.0 mmol) and THF (2 mL). The tube was heated at 66 °C for 24 h under nitrogen atmosphere. The resulting mixture was filtered through a celite pad and the filtrate was concentrated under vacuum. The obtained crude arylboronic ester was charged with aminoalkyne (0.2 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), and $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv). To this mixture ethanol (1.5 mL) was added and heated at 74 °C for 12 h under nitrogen atmosphere. Then, the reaction mixture was filtered through a celite pad and the filtrate was concentrated under vacuum and the residue was purified on column chromatography to

afford the desired product **5**. Similar reaction procedure was followed for the synthesis of **3l**.

Procedure for Sequential Coupling Reaction

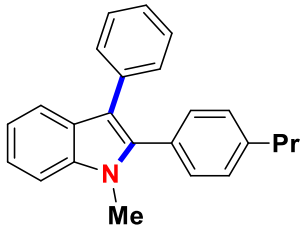
To a mixture of 2-iodo-*N*,*N*-dimethylaniline (247 mg, 1.0 mmol), $PdCl_2(PPh_3)_2$ (14 mg, 2.0 mol %), CuI (2 mg, 1.0 mol %), ethynylbenzene (1.2 equiv, 125 µL), and triethylamine (5 mL) under nitrogen atmosphere. The reaction was performed at room temperature for 2 h. After completion of the reaction, it was filtered through a pad of celite and it was concentrated under reduced pressure. Then the residue was treated with Methyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (78.6 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), and $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv) in ethanol (1.5 mL) at 74 °C under nitrogen atmosphere for 12 h. Then, the mixture was filtered through a pad of celite and was concentrated under reduced pressure. Then the residue was purified by silica gel column chromatography to afford **3j**.

1-Methyl-2,3-diphenyl-1*H*-indole (3a)[2]



Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 4,4,5,5-tetramethyl-2-phenyl-1,3,2-dioxaborolane (61.2 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3a** as the white solid (34.2 mg, 60% yield). m.p. 113-115 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.66 (s, 3H), 7.14 – 7.24 (m, 2H), 7.26 – 7.41 (m, 11H), 7.80 (d, J = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.9, 109.6, 115.0, 119.5, 120.1, 122.1, 125.4, 126.9, 128.0, 128.1, 128.3, 129.8, 131.1, 131.8, 135.1, 137.2, 137.7.

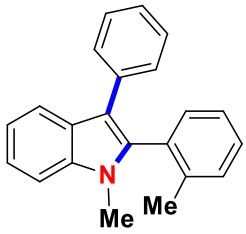
1-Methyl-3-phenyl-2-(4-propylphenyl)-1*H*-indole (3b)



Following the general procedure, using *N*,*N*-dimethyl-2-((4-propylphenyl)ethynyl)aniline (52.6 mg, 0.2 mmol), 4,4,5,5-tetramethyl-2-phenyl-1,3,2-dioxaborolane (61.2 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl

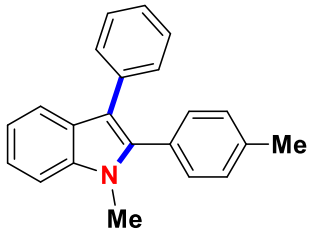
ether in hexane) to provide **3b** as the white solid (36.0 mg, 55% yield). m.p. 94-96 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.94 (t, J = 7.2 Hz, 3H), 1.62 – 1.68 (m, 2H), 2.59 (t, J = 7.6 Hz, 2H), 3.61 (s, 3H), 7.13 – 7.36 (m, 12H), 7.79 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.8, 24.3, 30.8, 37.8, 109.5, 114.8, 119.5, 120.1, 122.0, 125.3, 127.0, 128.1, 128.4, 129.0, 129.8, 130.9, 135.4, 137.2, 137.9, 142.5. HRMS (EI) calcd for $C_{24}H_{23}N$ $[M]^+$ 325.1830, found 325.1825.

1-Methyl-3-phenyl-2-(*o*-tolyl)-1*H*-indole (3c)



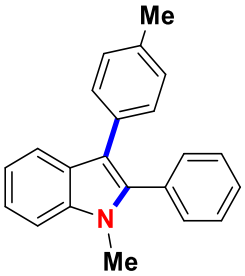
Following the general procedure, using *N*,*N*-dimethyl-2-(*o*-tolylethynyl)aniline (47 mg, 0.2 mmol), 4,4,5,5-tetramethyl-2-phenyl-1,3,2-dioxaborolane (61.2 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3c** as the colorless oil (42.2 mg, 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.99 (s, 3H), 3.49 (s, 3H), 7.11 (t, J = 6.0 Hz, 1H), 7.16 – 7.31 (m, 10H), 7.39 (d, J = 8.0 Hz, 1H), 7.86 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.8, 30.2, 109.4, 114.9, 119.6, 120.0, 121.8, 125.3, 125.7, 126.7, 128.1, 128.7, 128.9, 130.1, 131.6, 131.7, 135.4, 137.05, 137.1, 138.6. HRMS (EI) calcd for $C_{22}H_{19}N$ $[M]^+$ 297.1517, found 297.1511.

1-Methyl-3-phenyl-2-(*p*-tolyl)-1*H*-indole (3d)



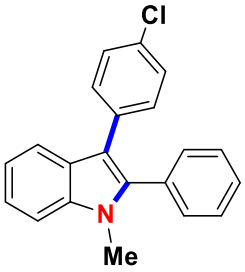
Following the general procedure, using *N*,*N*-dimethyl-2-(*p*-tolylethynyl)aniline (47 mg, 0.2 mmol), 4,4,5,5-tetramethyl-2-phenyl-1,3,2-dioxaborolane (61.2 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3d** as the white solid (26.4 mg, 44% yield). m.p. 114-117 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.42 (s, 3H), 3.70 (s, 3H), 7.20 – 7.35 (m, 11H), 7.43 (d, J = 8.0 Hz, 1H), 7.82 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.4, 30.9, 109.5, 114.8, 119.5, 120.1, 122.0, 125.4, 127.0, 128.1, 128.8, 129.1, 129.8, 131.0, 135.4, 137.2, 137.8. HRMS (EI) calcd for $C_{22}H_{19}N$ $[M]^+$ 297.1517, found 297.1523.

1-Methyl-2-phenyl-3-(*p*-tolyl)-1*H*-indole (3e)[2]



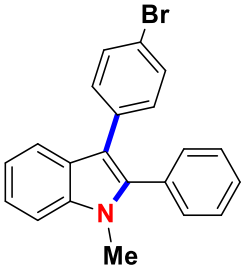
Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 4,4,5,5-tetramethyl-2-(*p*-tolyl)-1,3,2-dioxaborolane (65.4 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3e** as the white solid (27.4 mg, 46% yield). m.p. 155-156 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.32 (s, 3H), 3.66 (s, 3H), 7.07 (d, J = 8.0 Hz, 2H), 7.16 – 7.21 (m, 3H), 7.27 – 7.41 (m, 7H), 7.79 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.2, 30.9, 109.5, 114.9, 119.6, 120.0, 122.1, 126.9, 127.9, 128.3, 128.9, 129.6, 131.1, 131.9, 132.1, 134.9, 137.2, 137.5.

3-(4-Chlorophenyl)-1-methyl-2-phenyl-1*H*-indole (3f)[3]



Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 2-(4-chlorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (71.4 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3f** as the white solid (44.2 mg, 70% yield). m.p. 164-165 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.67 (s, 3H), 7.18 – 7.22 (m, 5H), 7.29 – 7.33 (m, 3H), 7.38 – 7.43 (m, 4H), 7.74 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.9, 109.7, 113.8, 119.2, 120.4, 122.3, 126.6, 128.2, 128.4, 128.5, 130.9, 131.0, 131.1, 131.5, 133.7, 137.2, 137.9.

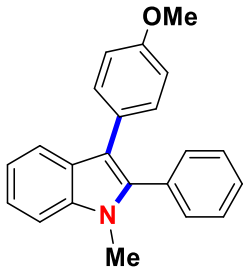
3-(4-Bromophenyl)-1-methyl-2-phenyl-1*H*-indole (3g)



Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 2-

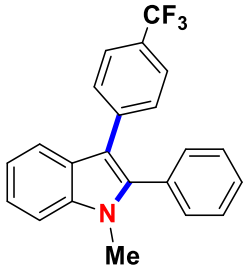
(4-bromophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (84.6 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3g** as the white solid (41.3 mg, 57% yield). m.p. 164-166 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.67 (s, 3H), 7.15 – 7.24 (m, 3H), 7.29 – 7.33 (m, 3H), 7.36 – 7.42 (m, 6H), 7.74 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.9, 109.7, 113.8, 119.2, 120.4, 122.3, 126.6, 128.2, 128.5, 131.0, 131.3, 131.5, 134.2, 137.2, 137.9. HRMS (EI) calcd for $C_{21}H_{16}BrN$ $[M]^+$ 361.0466, found 361.0467.

3-(4-Methoxyphenyl)-1-methyl-2-phenyl-1*H*-indole (3h)[4]



Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (70.2 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3h** as the white solid (41.9 mg, 67% yield). m.p. 128-129 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.66 (s, 3H), 3.78 (s, 3H), 6.82 (d, J = 8.4 Hz, 2H), 7.16 – 7.23 (m, 3H), 7.28 – 7.33 (m, 3H), 7.35 – 7.41 (m, 4H), 7.76 (d, J = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.9, 55.1, 109.5, 113.6, 114.6, 119.5, 120.0, 122.0, 127.0, 127.5, 127.9, 128.3, 130.8, 131.1, 131.9, 137.2, 137.3, 157.5.

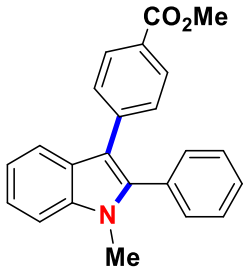
1-Methyl-2-phenyl-3-(4-(trifluoromethyl)phenyl)-1*H*-indole (3i)[2]



Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 4,4,5,5-tetramethyl-2-(4-(trifluoromethyl)phenyl)-1,3,2-dioxaborolane (81.6 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3i** as the white solid (50.4 mg, 72% yield). m.p. 134-136 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.66 (s, 3H), 7.20 – 7.23 (m, 1H), 7.29 – 7.34 (m, 3H), 7.38 – 7.43 (m, 6H), 7.49 (d, J = 8.0 Hz, 2H), 7.78 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.9, 109.8, 113.7, 119.2, 120.6, 122.5, 125.08, 125.1 126.5, 127.0, 128.4, 128.6, 129.7, 131.0, 131.3, 137.3, 138.5, 139.2.

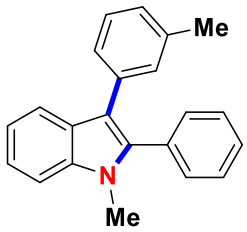
^{19}F NMR (376 MHz, $CDCl_3$): δ -63.7.

Methyl 4-(1-methyl-2-phenyl-1*H*-indol-3-yl)benzoate (3j)



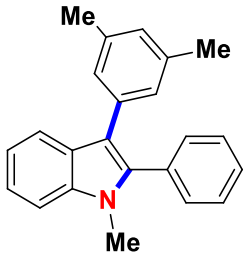
Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), Methyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (78.6 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3j** as the white solid (58.0 mg, 85% yield). m.p. 158-160 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.67 (s, 3H), 3.88 (s, 3H), 7.22 – 7.24 (m, 1H), 7.29 – 7.44 (m, 9H), 7.81 (d, J = 7.6 Hz, 1H), 7.92 (d, J = 6.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.9, 51.9, 109.7, 114.0, 119.3, 120.6, 122.4, 126.4, 126.7, 128.3, 128.5, 129.4, 129.5, 131.0, 131.4, 137.3, 138.5, 140.4, 167.2. HRMS (EI) calcd for $C_{23}H_{19}NO_2$ $[M]^+$ 341.1416, found 341.1413.

1-Methyl-2-phenyl-3-(*m*-tolyl)-1*H*-indole (3k)



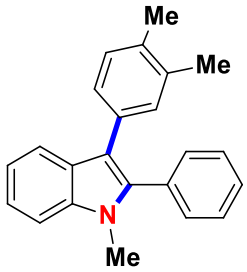
Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 4,4,5,5-tetramethyl-2-(*m*-tolyl)-1,3,2-dioxaborolane (65.4 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3k** as the white solid (24.0 mg, 40% yield). m.p. 105-107 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.01 (s, 3H), 3.52 (s, 3H), 7.13 (t, J = 7.2 Hz, 1H), 7.19 – 7.32 (m, 10H), 7.41 (d, J = 8.4 Hz, 1H), 7.87 (d, J = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.8, 30.3, 109.4, 114.9, 119.6, 120.0, 121.8, 125.3, 125.7, 126.7, 128.2, 128.7, 128.9, 130.1, 131.6, 131.7, 135.4, 137.0, 137.1, 138.6. HRMS (EI) calcd for $C_{22}H_{19}N$ $[M]^+$ 297.1517, found 297.1514.

3-(3,5-Dimethylphenyl)-1-methyl-2-phenyl-1*H*-indole (3l)



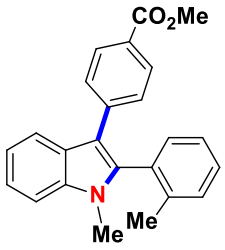
Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 2-(3,5-dimethylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (69.6 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3l** as the white solid (16.5 mg, 27% yield). m.p. 92-100 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.22 (s, 6H), 3.67 (s, 3H), 6.82 (s, 1H), 6.92 (s, 2H), 7.18 – 7.19 (m, 1H), 7.29 – 7.41 (m, 7H), 7.80 (d, J = 7.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.4, 30.9, 109.5, 115.0, 119.7, 120.0, 122.0, 126.9, 127.2, 127.6, 127.9, 128.3, 131.1, 132.0, 134.9, 137.2, 137.4, 137.6. HRMS (EI) calcd for $C_{23}H_{21}N$ $[M]^+$ 311.1674, found 311.1677.

3-(3,4-Dimethylphenyl)-1-methyl-2-phenyl-1*H*-indole (3m)



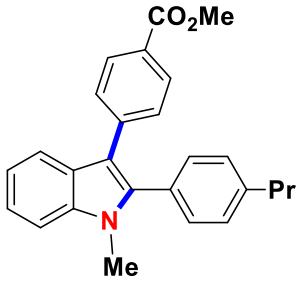
Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 2-(3,4-dimethylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (69.6 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3m** as the white solid (22.1 mg, 36% yield). m.p. 110-112 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.19 (s, 3H), 2.23 (s, 3H), 3.67 (s, 3H), 6.99-7.02 (m, 2H), 7.14-7.20 (m, 2H), 7.25 –7.41 (m, 7H), 7.80 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.4, 19.8, 30.9, 109.5, 115.0, 119.8, 120.0, 122.0, 127.0, 127.2, 127.9, 128.3, 129.5, 131.0, 131.1, 132.1, 132.5, 133.6, 136.1, 137.3, 137.4. HRMS (EI) calcd for $C_{23}H_{21}N$ $[M]^+$ 311.1674, found 311.1678.

Methyl 4-(1-methyl-2-(*o*-tolyl)-1*H*-indol-3-yl)benzoate (3n)



Following the general procedure, using *N*,*N*-dimethyl-2-(*o*-tolylethynyl)aniline (47 mg, 0.2 mmol), Methyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (78.6 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3n** as the white solid (50.1 mg, 71% yield). m.p. 128-132 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.00 (s, 3H), 3.54 (s, 3H), 3.88 (s, 3H), 7.24 – 7.28 (m, 4H), 7.33-7.35 (m, 4H), 7.43 (d, J = 8.0 Hz, 1H), 7.89 (t, J = 8.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.8, 30.3, 51.9, 109.7, 114.0, 119.4, 120.5, 122.2, 125.9, 126.3, 126.7, 128.4, 129.1, 129.6, 130.3, 131.2, 131.5, 137.2, 138.1, 138.4, 140.6, 167.2. HRMS (EI) calcd for $C_{24}H_{21}NO_2$ $[M]^+$ 355.1572, found 355.1563.

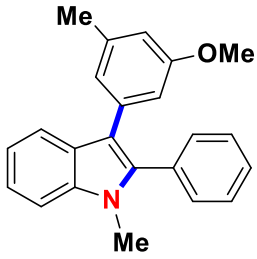
Methyl 4-(1-methyl-2-(4-propylphenyl)-1*H*-indol-3-yl)benzoate (3o)



Following the general procedure, using *N*,*N*-dimethyl-2-((4-propylphenyl)ethynyl)aniline (52.6 mg, 0.2 mmol), Methyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (78.6 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3o** as the white solid (46.2 mg, 60% yield). m.p. 116-119 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.97 (t, J = 7.2 Hz, 3H), 1.63 – 1.71 (m, 2H), 2.63 (t, J = 7.6 Hz, 2H), 3.67 (s, 3H), 3.89 (s, 3H), 7.20 – 7.23 (m, 5H), 7.30 – 7.37 (m, 3H), 7.41 (d, J = 8.0 Hz, 1H), 7.81 (d, J = 8.0 Hz, 1H), 7.92 (d, J = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.9, 24.3, 30.9, 37.8, 51.9, 109.7, 113.8, 119.2, 120.5, 122.3, 126.5, 126.6, 128.5, 128.7, 129.3, 129.5, 130.8, 137.3, 138.8, 140.6, 143.0, 167.3. HRMS (EI) calcd for $C_{26}H_{25}NO_2$ $[M]^+$ 383.1885, found 383.1892.

Following the general procedure, using *N*,*N*-dimethyl-2-((4-propylphenyl)ethynyl)aniline (263 mg, 1.0 mmol), Methyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoate (393.2 mg, 1.5 mmol), $Pd(OAc)_2$ (11.2 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (299.5 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3o** as the white solid (237.6 mg, 62% yield).

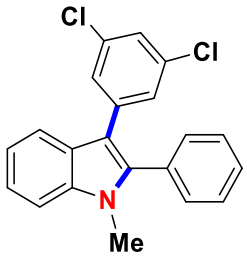
2-(3-Methoxy-5-methylphenyl)-1-methyl-3-phenyl-1*H*-indole (3p)



Following the general procedure, using *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 2-

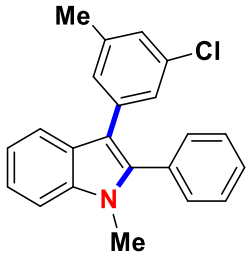
(3-methoxy-5-methylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (74.4 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **3p** as the colorless oil (40.2 mg, 61% yield). ^1H NMR (400 MHz, $CDCl_3$): δ 2.25 (s, 3H), 3.58 (s, 3H), 3.66 (s, 3H), 6.56 (d, J = 12.0 Hz, 2H), 6.79 (s, 1H), 7.17 – 7.20 (m, 1H), 7.27-7.40 (m, 7H), 7.83 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.6, 30.9, 54.9, 109.5, 111.9, 112.5, 115.0, 119.7, 120.1, 122.1, 123.1, 126.8, 128.0, 128.3, 131.1, 132.0, 136.2, 137.3, 137.8, 138.9, 159.3. HRMS (EI) calcd for $C_{23}H_{21}NO$ $[M]^+$ 327.1623, found 327.1628.

3-(3,5-Dichlorophenyl)-1-methyl-2-phenyl-1*H*-indole (5a)



Following the Sequential procedure, using 1,3-dichlorobenzene (147 µL, 1 mmol), B_2pin_2 (190.5 mg, 0.75 mmol), $[Ir(OMe)(cod)_2]_2$ (6.6 mg, 1 mol %), dtbpy (5.4 mg, 2 mol %). *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 2-(3,5-dichlorophenyl)-4,4,5,5-tetra methyl-1,3,2-dioxaborolane (excess, 1 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **5a** as the white solid (51.5 mg, 73% yield). m.p. 112-114 °C; ^1H NMR (400 MHz, $CDCl_3$): δ 3.64 (s, 3H), 7.13-7.15 (m, 3H), 7.20-7.24 (m, 1H), 7.28 – 7.33 (m, 3H), 7.39 – 7.43 (m, 4H), 7.75 (d, J = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.9, 109.8, 112.4, 119.0, 120.7, 122.6, 125.4, 126.3, 127.8, 128.7, 130.9, 134.5, 137.2, 138.5, 138.6. HRMS (EI) calcd for $C_{21}H_{15}Cl_2N$ $[M]^+$ 351.0582, found 351.0588.

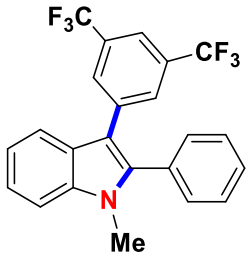
3-(3-Chloro-5-methylphenyl)-1-methyl-2-phenyl-1*H*-indole (5b)



Following the Sequential procedure, using 3-chlorotoluene (126.6 µL, 1 mmol), B_2pin_2 (190.5 mg, 0.75 mmol), $[Ir(OMe)(cod)_2]_2$ (6.6 mg, 1 mol %), dtbpy (5.4 mg, 2 mol %). *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 2-(3-chloro-5-methylphenyl)-4,4,5,5-tetra methyl-1,3,2-dioxaborolane (75.6 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **5b** as the white solid (26.6 mg, 40% yield). m.p. 101-103 °C; ^1H NMR (400 MHz, $CDCl_3$): δ 2.20 (s, 3H), 3.64 (s, 3H),

6.96 (s, 2H), 7.08 (s, 1H), 7.19 (t, J = 7.2 Hz, 1H), 7.28-7.31 (m, 3H), 7.36-7.39 (m, 4H), 7.76 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.2, 30.9, 109.6, 113.8, 119.3, 120.4, 122.3, 126.2, 126.7, 128.2, 128.4, 128.7, 131.0, 131.5, 133.6, 136.9, 137.2, 138.1, 139.3. HRMS (EI) calcd for $C_{22}H_{18}ClN$ $[M]^+$ 331.1128, found 331.1126.

3-(3,5-Bis(trifluoromethyl)phenyl)-1-methyl-2-phenyl-1*H*-indole (5c)

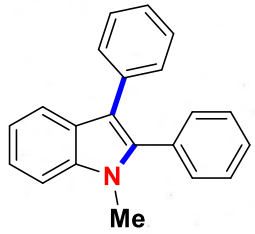


Following the Sequential procedure, using 1,3-bis(trifluoromethyl)benzene (214 mg, 1 mmol), B_2pin_2 (190.5 mg, 0.75 mmol), $[Ir(OMe)(cod)_2]_2$ (6.6 mg, 1 mol %), dtbpy (5.4 mg, 2 mol %). *N*,*N*-dimethyl-2-(phenylethynyl)aniline (44 mg, 0.2 mmol), 2-(3,5-bis(trifluoromethyl)phenyl) -4,4,5,5-tetramethyl-1,3,2-dioxaborolane (102 mg, 0.3 mmol), $Pd(OAc)_2$ (2.25 mg, 5 mol %), $Cu(OAc)_2 \cdot H_2O$ (60 mg, 1.5 equiv), then purified by column chromatography (SiO_2, 6% diethyl ether in hexane) to provide **5c** as the white solid (46.1 mg, 55% yield). m.p. 111-115 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.71 (s, 3H), 7.25 – 7.39 (m, 4H), 7.44 – 7.47 (m, 4H), 7.63 (s, 1H), 7.70 (s, 2H), 7.76 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.9, 110.0, 112.2, 118.6, 121.1, 121.7, 122.1, 122.8, 124.8, 126.0, 128.9, 129.2, 130.7, 130.9, 131.1, 131.5, 137.3, 137.6, 139.1. ^{19}F NMR (376 MHz, $CDCl_3$): δ -64.6. HRMS (EI) calcd for $C_{23}H_{15}F_6N$ $[M]^+$ 419.1109, found 419.1107.

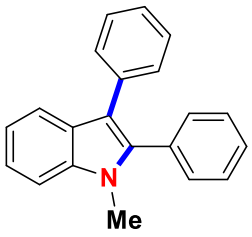
2. References

1. (a) Yue, D.; Yao, T.; Larock, R. C. *J. Org. Chem.* **2006**, *71*, 62–69. (b) Xia, X.-F.; Wang, N.; Zhang, L.-L.; Song, X.-R.; Liu, X.-Y.; Liang, Y.-M. *J. Org. Chem*. **2012**, *77*, 9163−9170. (c) Ratniyom, J.; Dechnarong, N.; Yotphan, S.; Kiatisevi, S. *Eur. J. Org. Chem.*, **2014**, 1381–1385. (d) Partridge, B. M.; Hartwig, J. F. *Org. Lett.*, **2013**, *15*, 140–143.
2. Miyasaka, M.; Fukushima, A.; Satoh, T.; Hirano, K.; Miura, M. *Chem. Eur. J.* **2009**, *15*, 3674–3677.
3. Li, J.; An, Y.; Li, J.; Yang, S.; Wu, W.; Jiang, H. *Org. Chem. Front.*, **2017**, *4*, 1590–1594.
4. Worlikar, S. A.; Neuenswander, B.; Lushington, G. H.; Larock, R. C. *J. Comb. Chem.*, **2009**, *11*, 875–879.

3. NMR spectra of compounds



3a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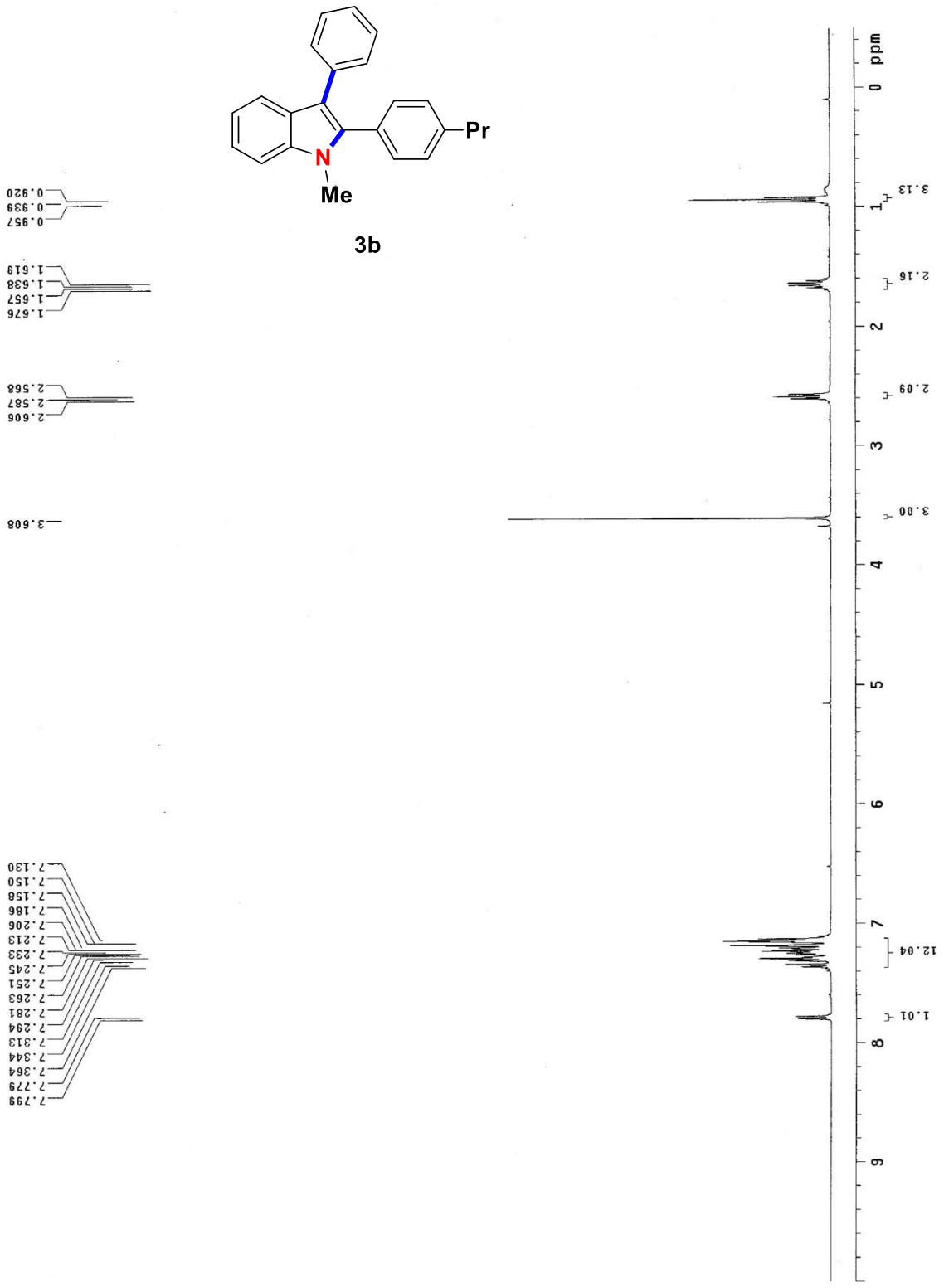
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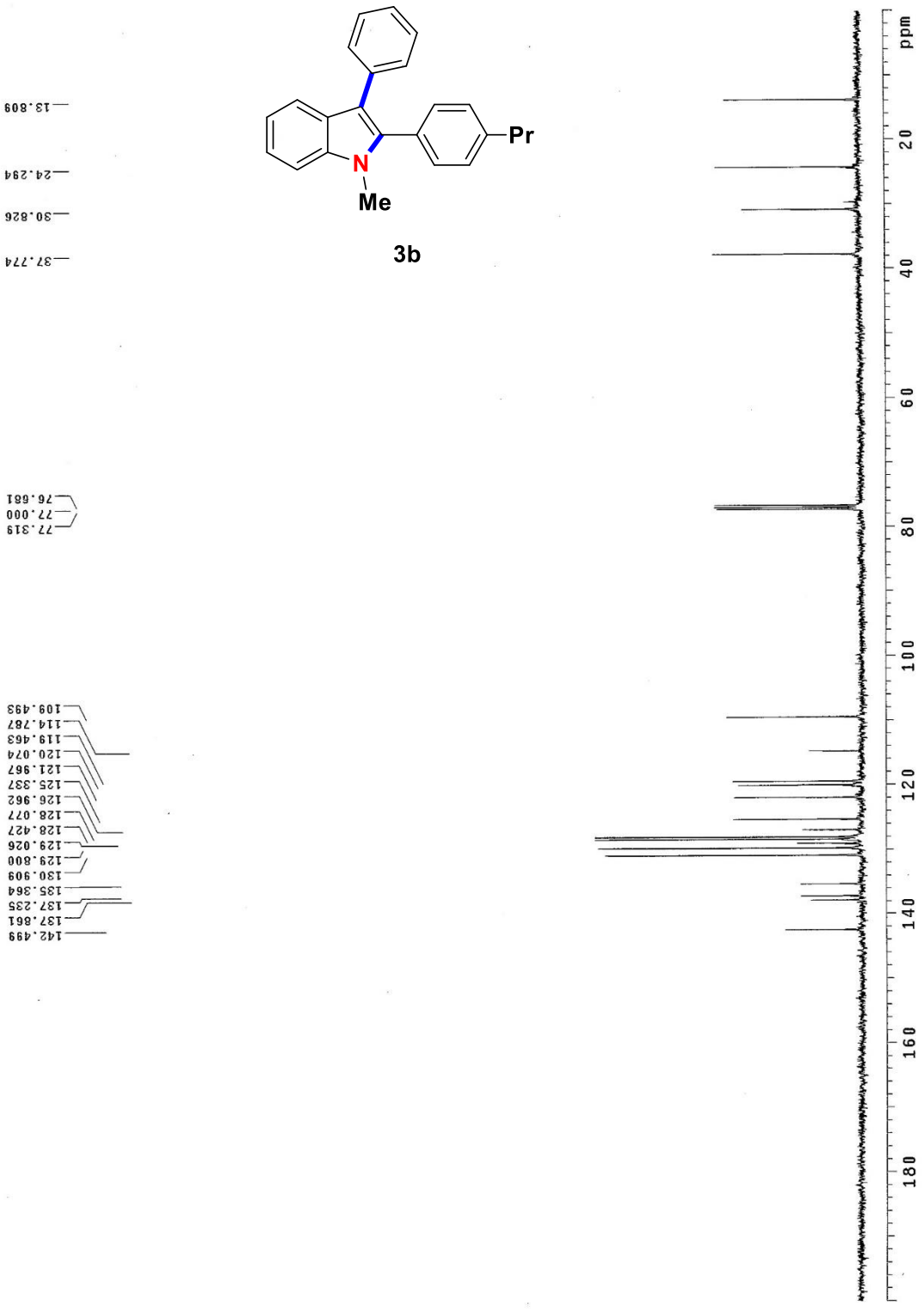
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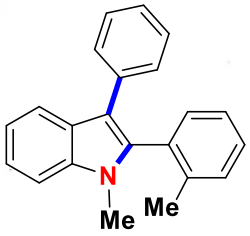
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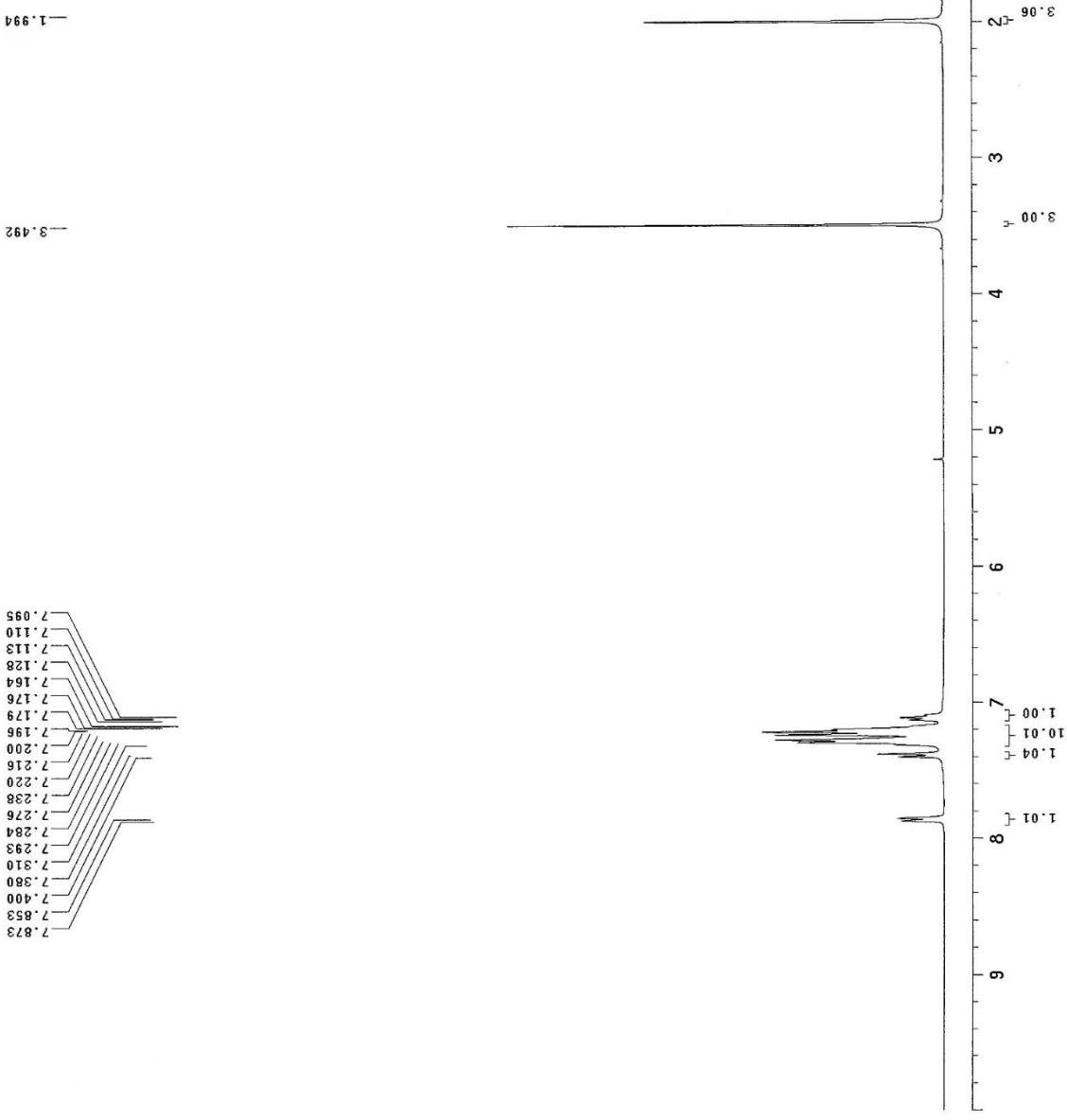
Me

3c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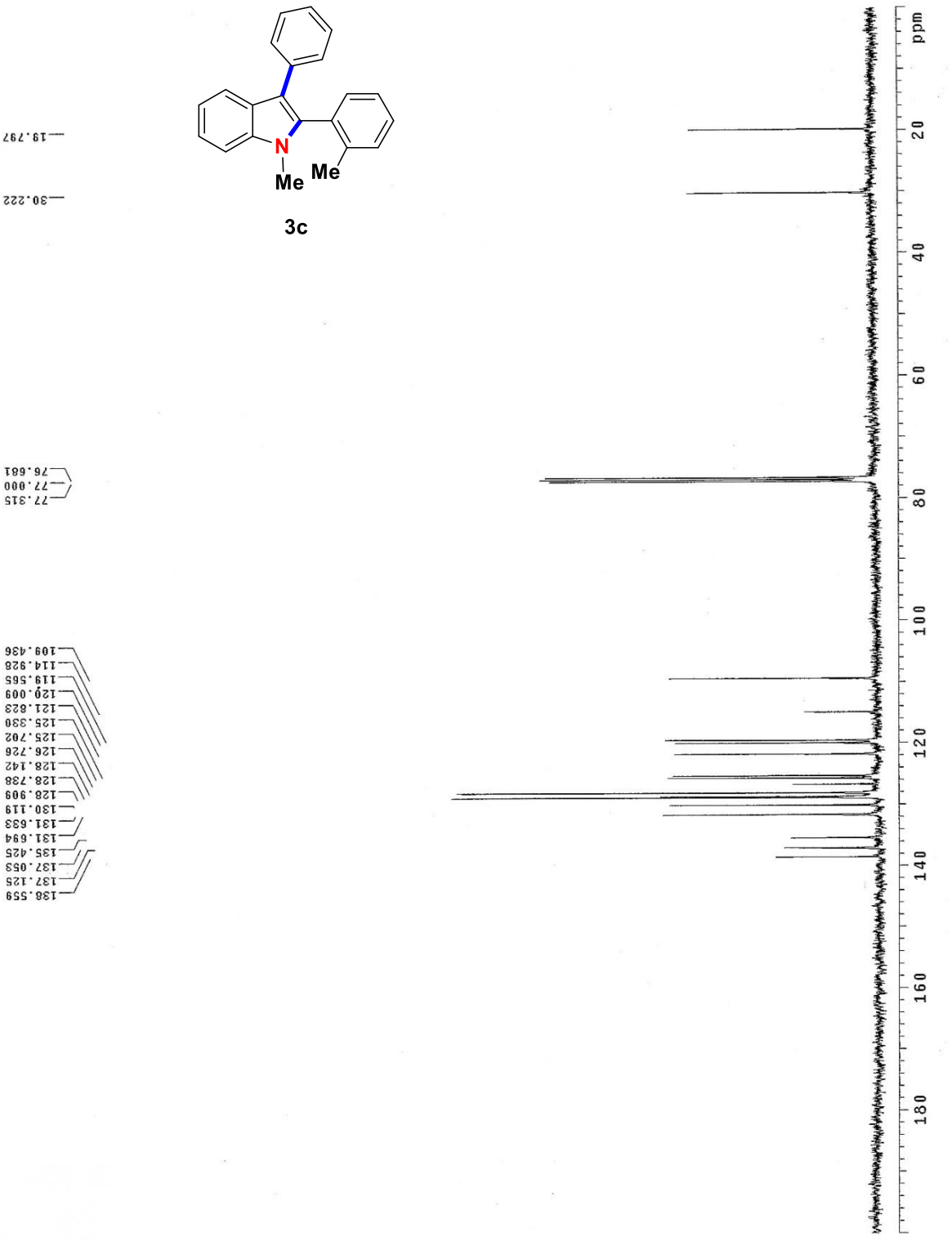
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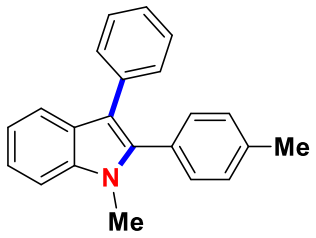
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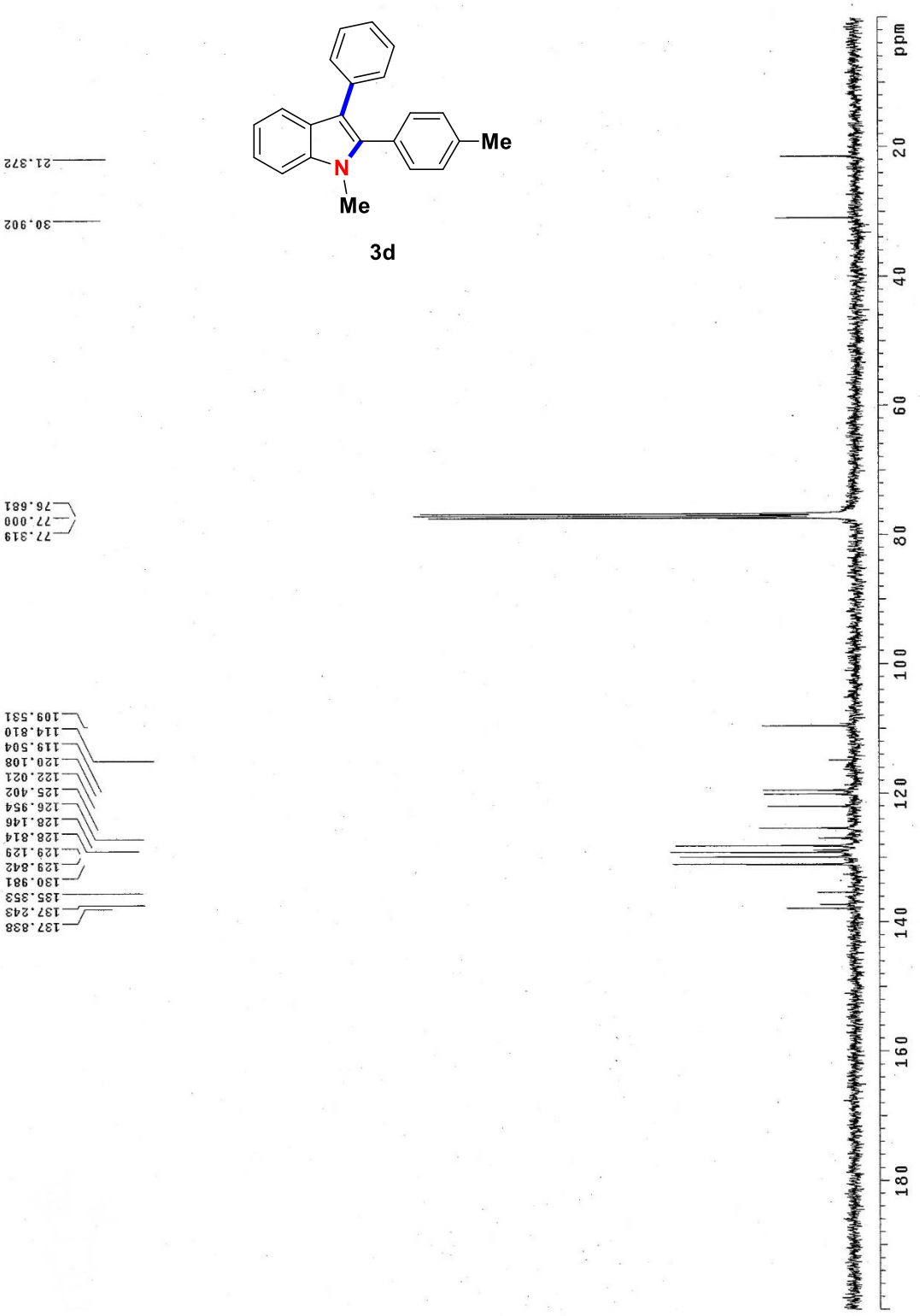
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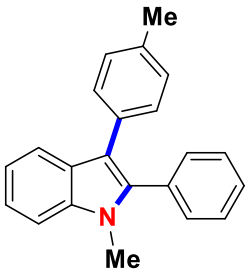
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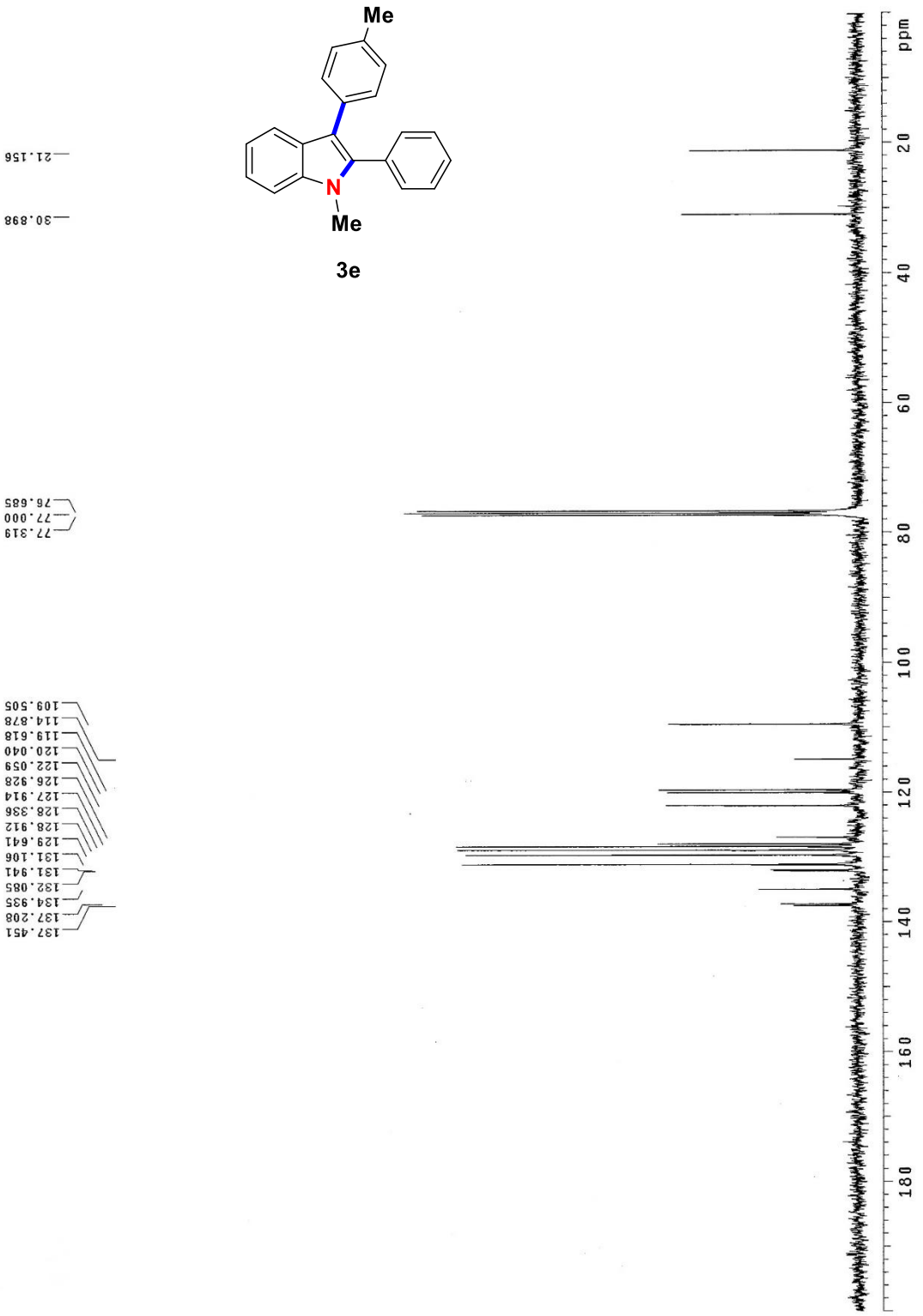
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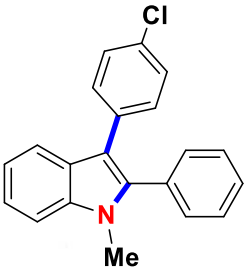
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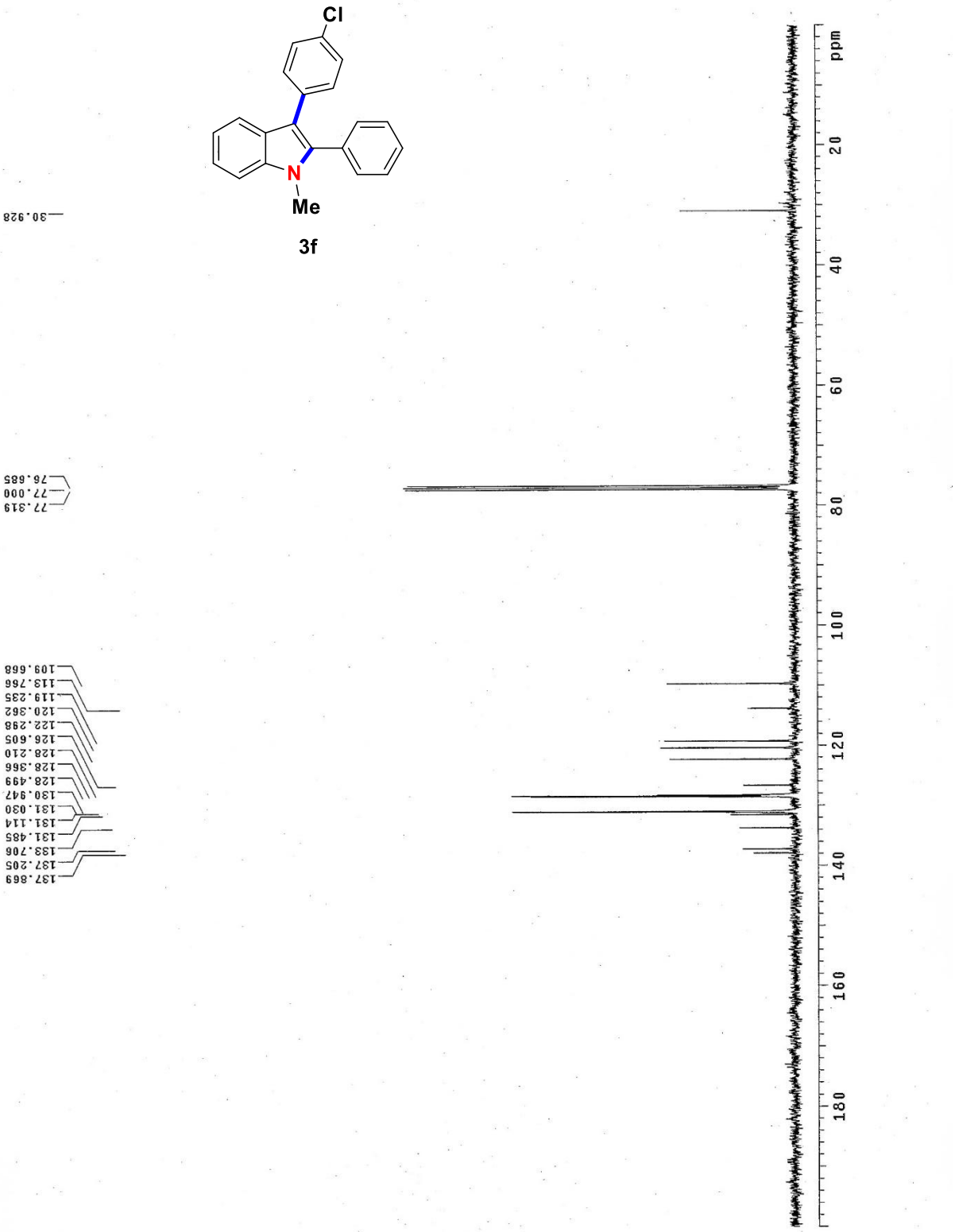
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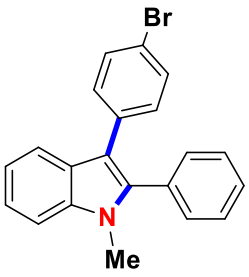
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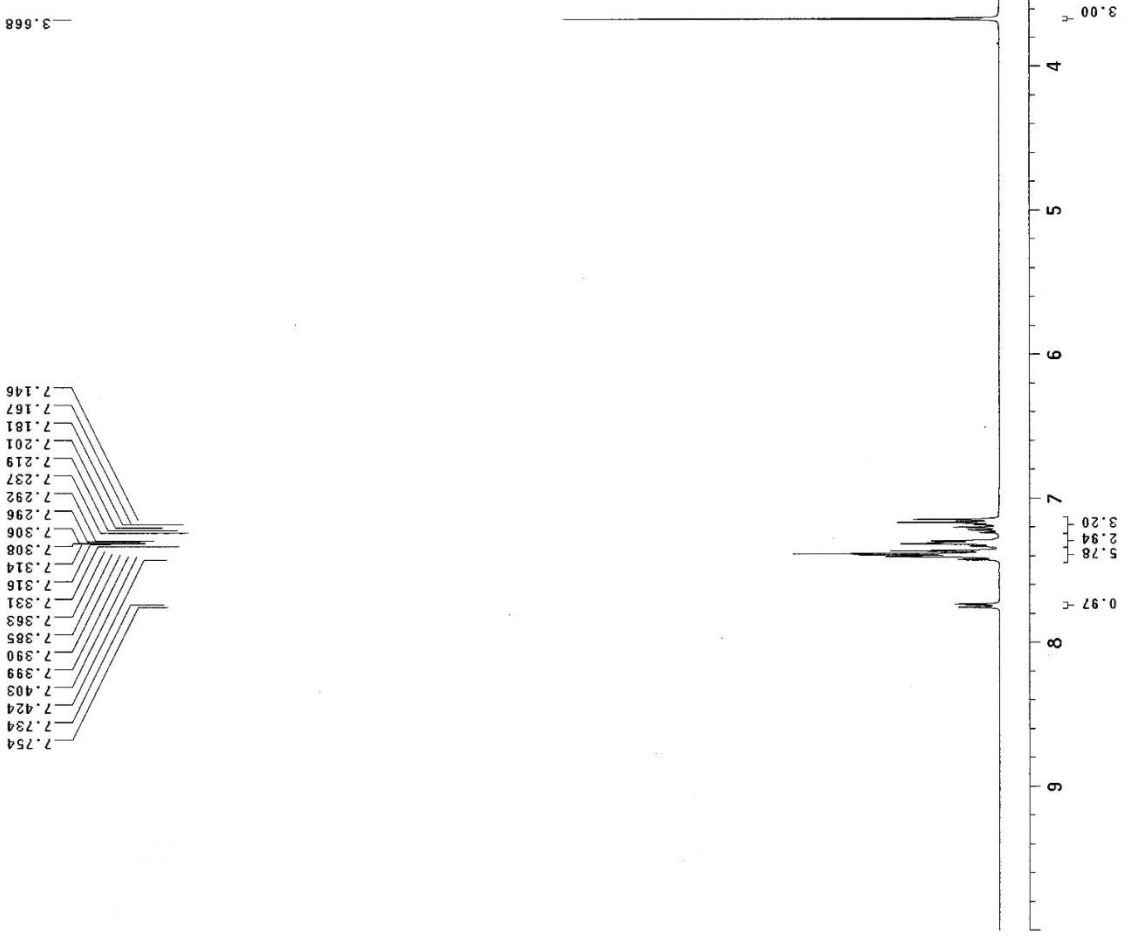
Me

3g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7.424
7.403
7.399
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7.385
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7.331
7.316
7.314
7.308
7.306
7.296
7.292
7.237
7.219
7.201
7.181
7.167
7.146
ppm
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2
3
4
5
6
7
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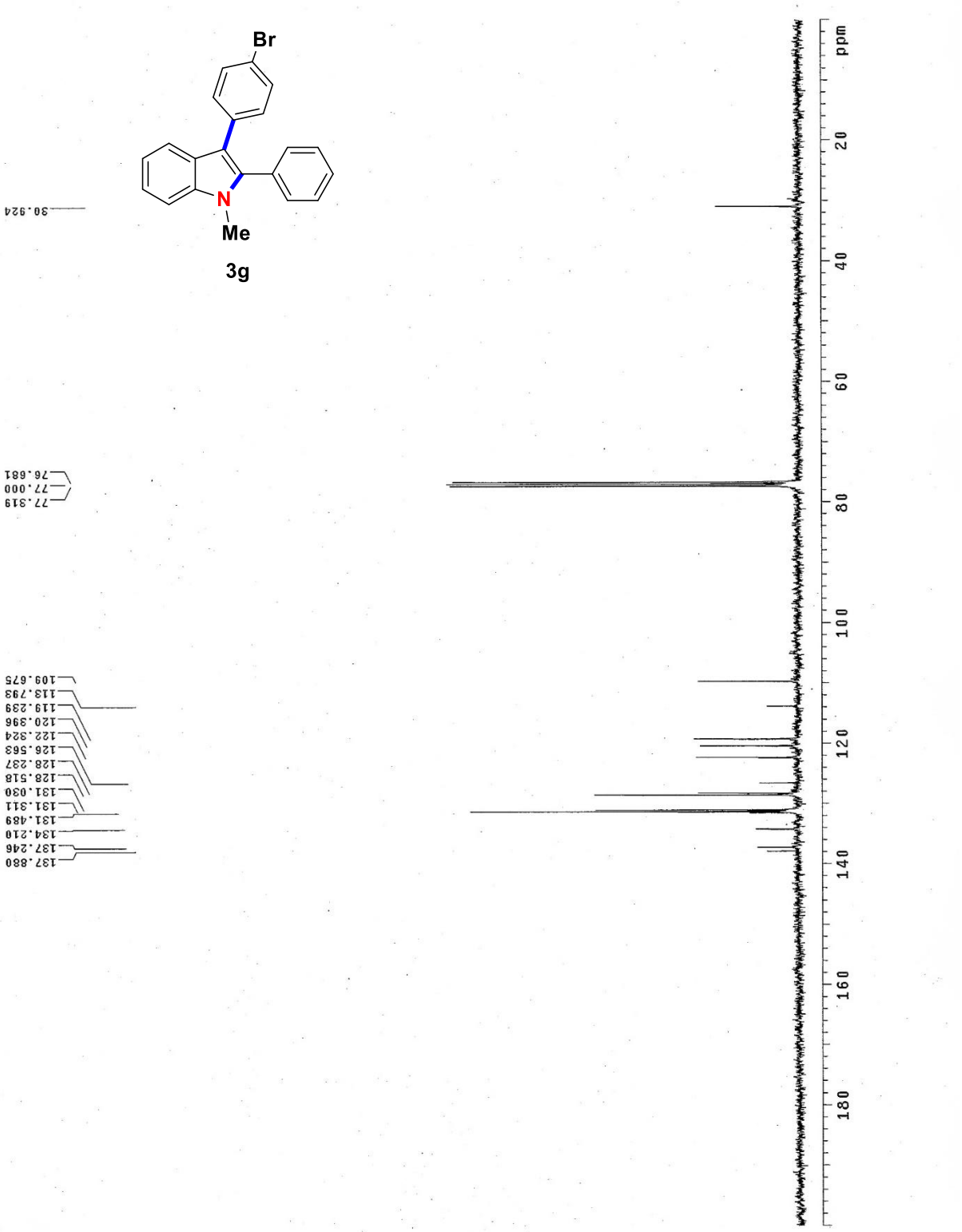
3.00
0.97
3.20
2.94
5.78


Br
N
Me
3g
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128.237
128.518
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131.489
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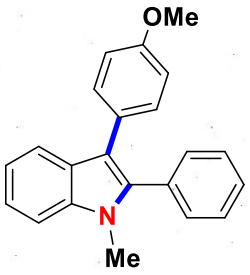
100
120
140
160
180


OMe
N
Me

3h

3.779
3.663
7.771
7.752
7.409
7.401
7.388
7.380
7.362
7.348
7.330
7.325
7.313
7.295
7.275
7.230
7.221
7.209
7.197
7.178
7.177
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6.834
6.813
ppm
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2.04
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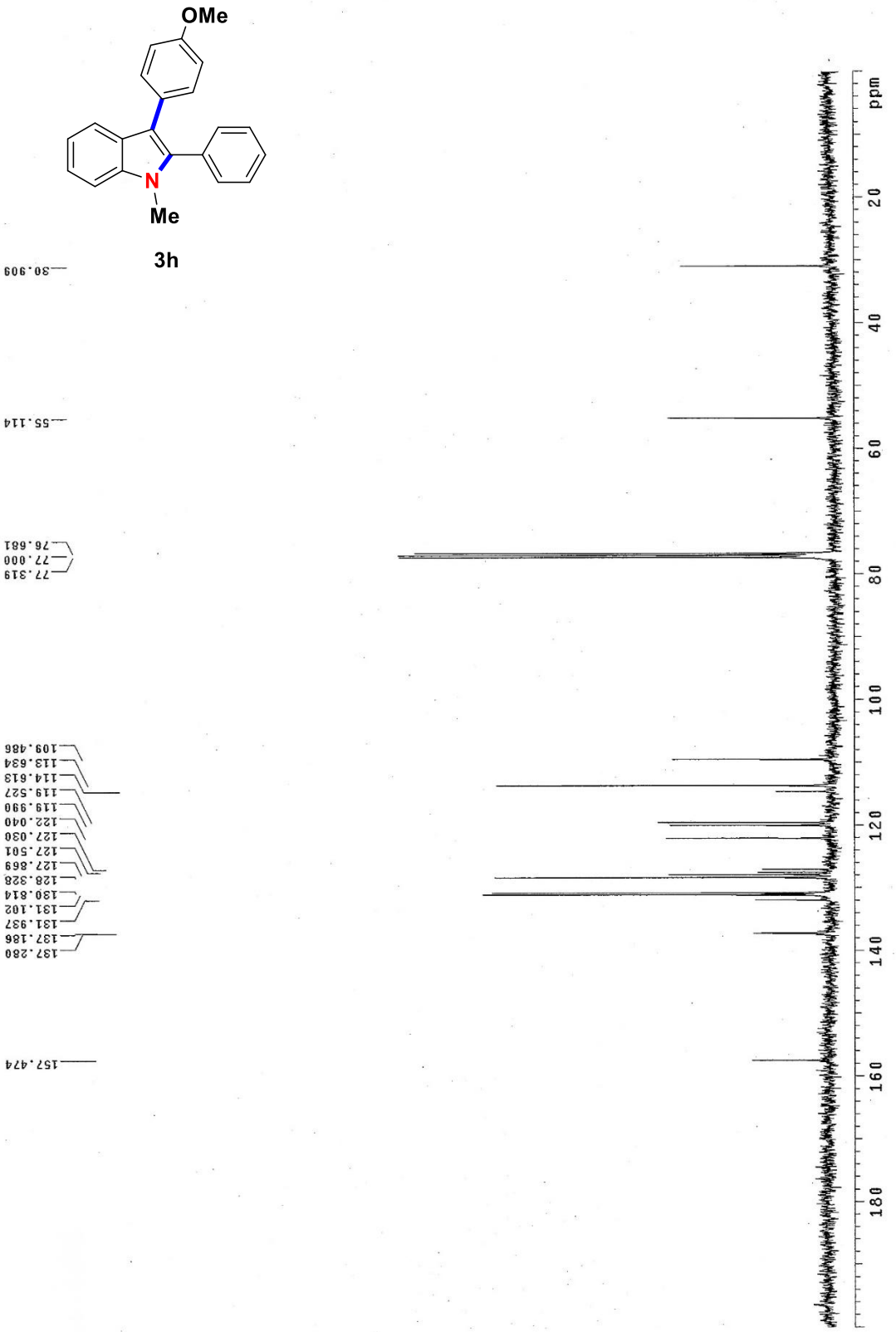
3.10
0.99


OMe
N
Me
3h
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77.000
77.319
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113.634
114.613
119.527
119.990
122.040
127.030
127.501
127.869
128.328
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131.937
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ppm
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160
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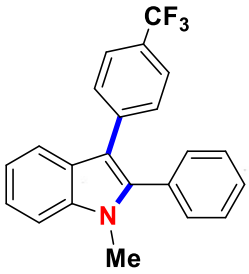
CF3
N
Me

3i

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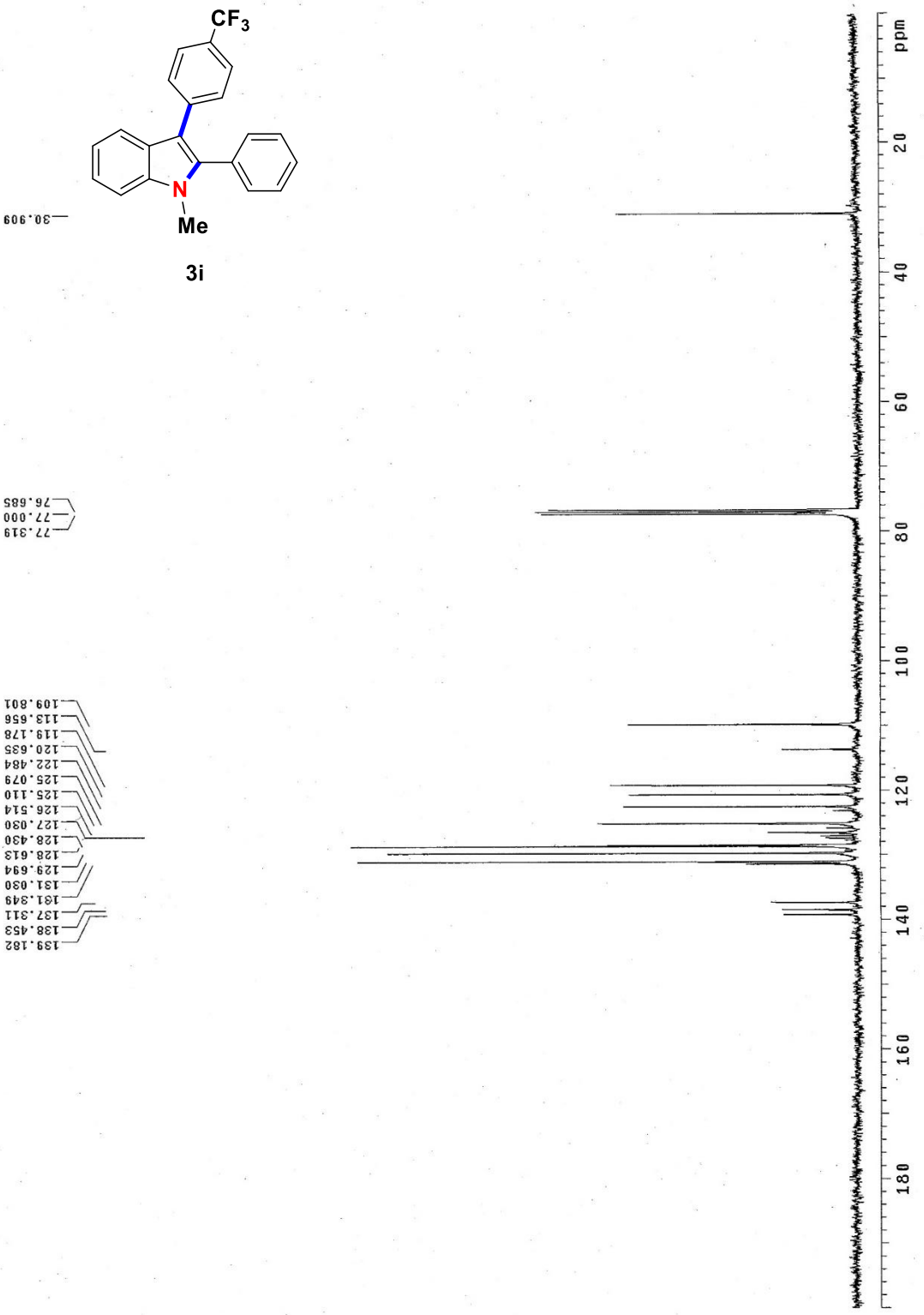
7.791
7.771
7.501
7.481
7.428
7.408
7.391
7.385
7.375
7.340
7.339
7.321
7.311
7.302
7.294
7.287
7.232
7.214
7.195
3.00
1.02
1.95
5.95
3.06
1.05
ppm


CF3
N
Me
3i
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120.635
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125.079
125.110
126.514
127.030
128.430
128.613
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ppm
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140
160
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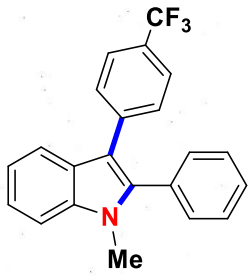
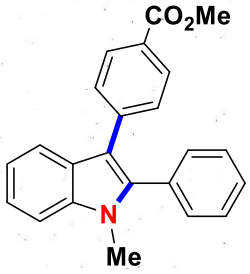
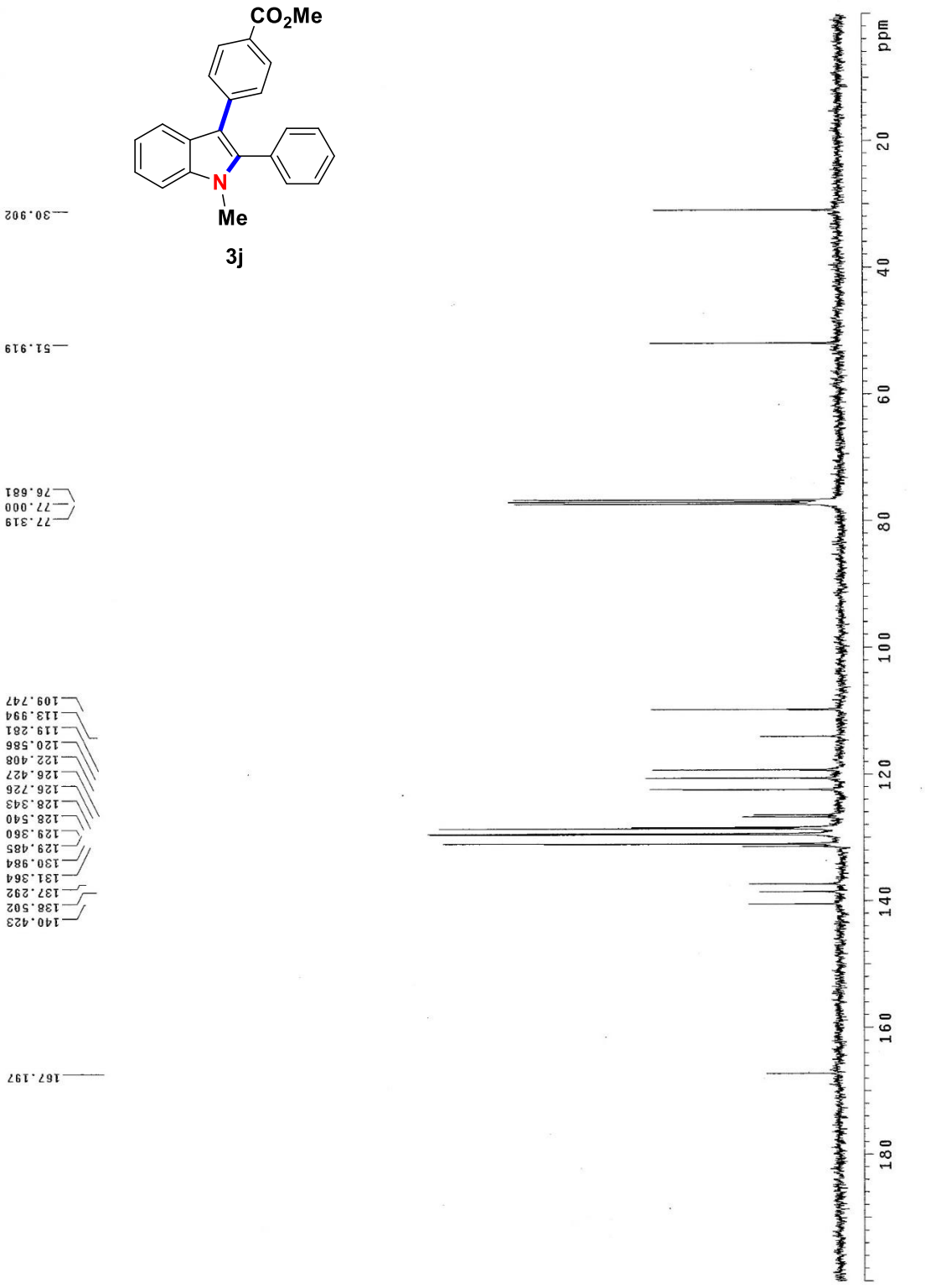


3i


CO2Me
N
Me

3j

3.674
3.883
7.938
7.921
7.916
7.826
7.825
7.806
7.805
7.436
7.415
7.403
7.400
7.394
7.391
7.387
7.384
7.366
7.349
7.346
7.344
7.329
7.326
7.316
7.312
7.309
7.306
7.303
7.299
7.296
7.292
7.244
7.241
7.225
7.224
7.221
3.03
3.00
1.02
9.15
1.02
2.00
ppm


CO2Me
N
Me
3j
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77.000
76.681
109.747
113.994
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120.586
122.408
126.427
126.726
128.343
128.540
129.360
129.485
130.984
131.364
137.292
138.502
140.423
167.197
ppm
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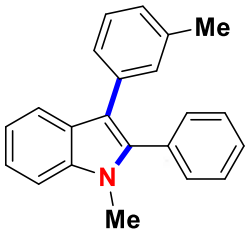
40
60
80
100
120
140
160
180


Me
N
Me

3k

2.009
3.523
7.115
7.133
7.150
7.191
7.209
7.216
7.228
7.237
7.258
7.280
7.292
7.301
7.308
7.321
7.325
7.405
7.426
7.864
7.883
ppm
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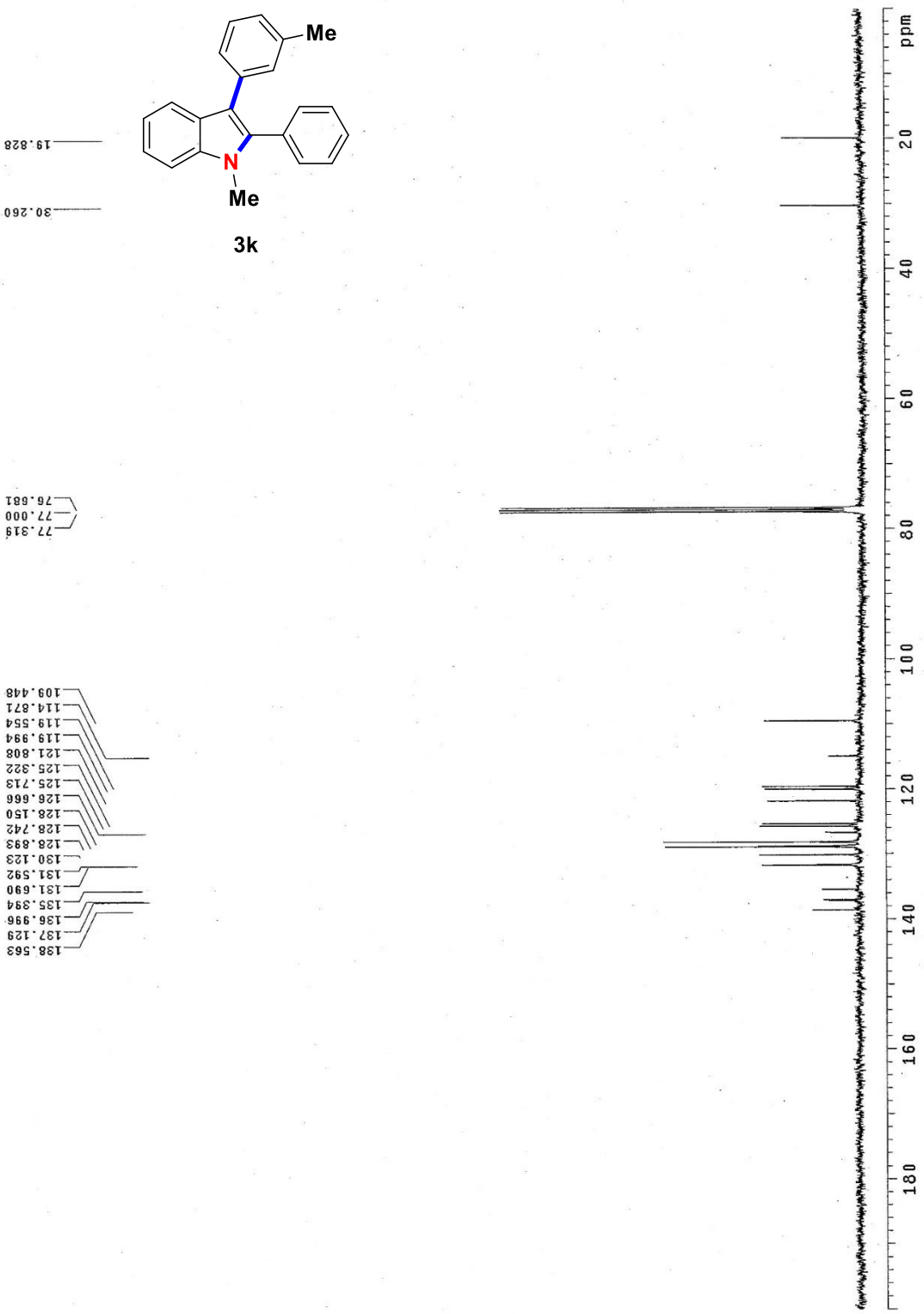
4
5
6
7
8
9
2.80
3.00
0.99
10.05
1.02
1.01


Me
N
Me
3k
19.828
30.260
77.819
77.000
76.681
109.448
114.871
119.554
119.994
121.808
125.322
125.713
126.666
128.150
128.742
128.893
130.123
131.592
131.690
135.894
136.996
137.129
138.563
ppm
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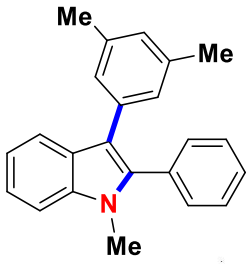
40
60
80
100
120
140
160
180


Me
Me
N
Me

3l

2.222
3.666
6.818
6.917
7.183
7.185
7.293
7.296
7.298
7.313
7.317
7.322
7.331
7.337
7.340
7.352
7.364
7.366
7.377
7.382
7.388
7.390
7.411
7.796
7.798
7.816
7.818
6.07
3.00
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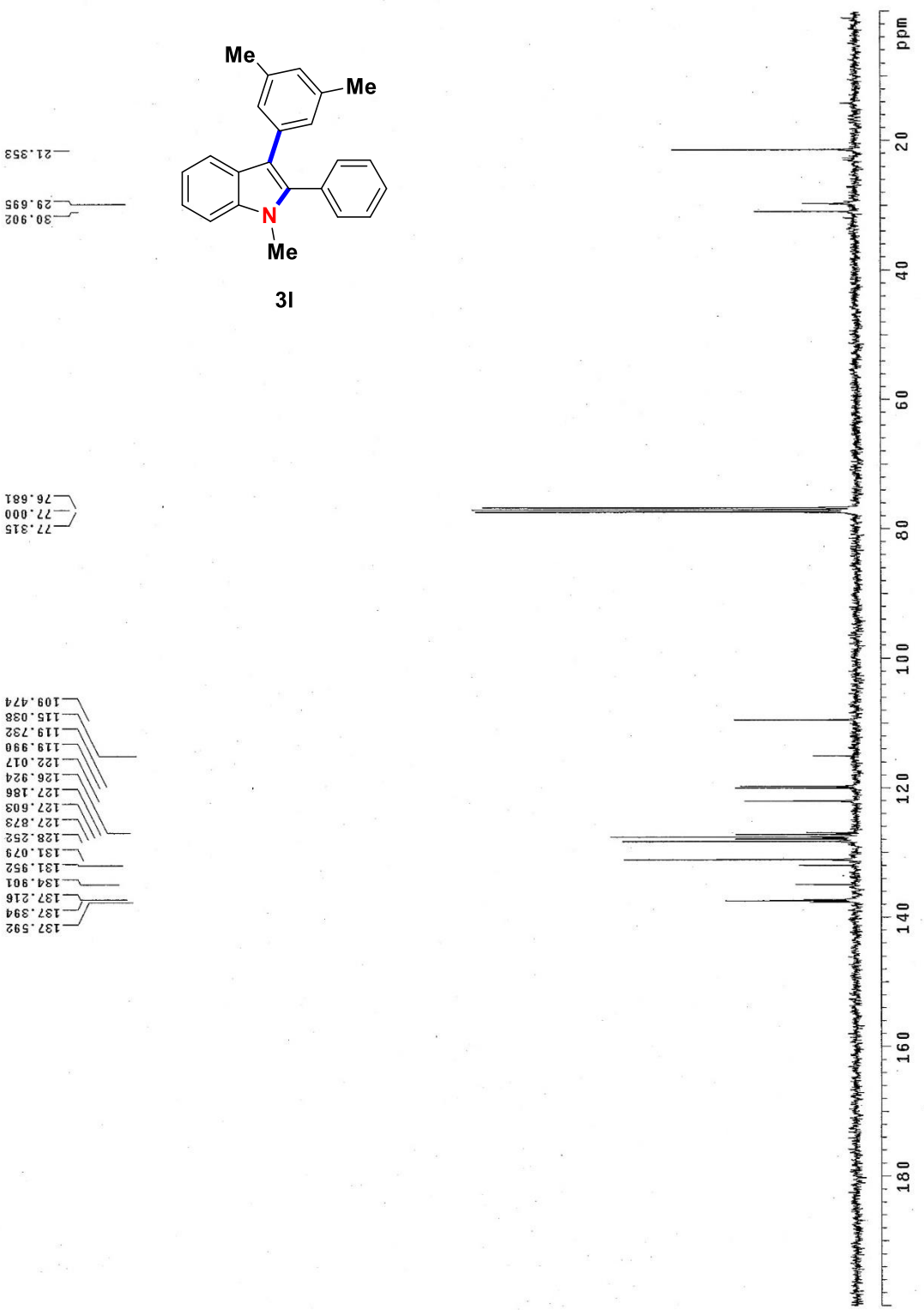
1.87
1.17
7.03
1.05
ppm


Me
Me
N
Me
3l
21.353
29.695
80.902
76.681
77.000
77.315
109.474
115.038
119.732
119.990
122.017
126.924
127.186
127.603
127.873
128.252
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131.952
134.901
137.216
137.394
137.592
ppm
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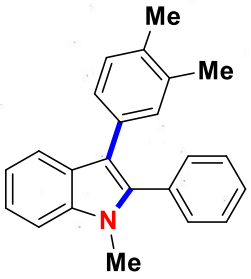
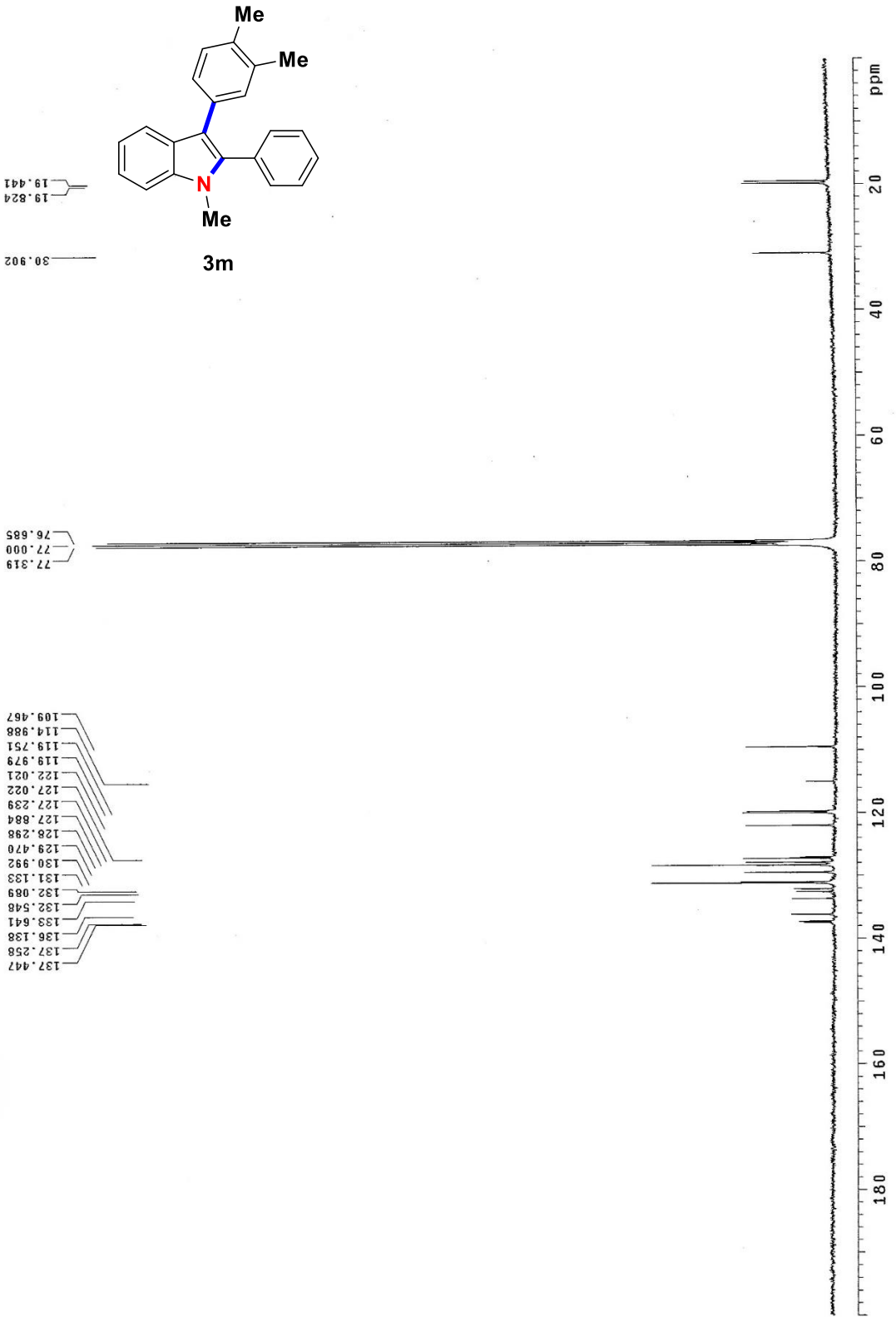
40
60
80
100
120
140
160
180


Me
Me
N
Me

3m

2.189
2.284
3.668
6.987
7.004
7.023
7.135
7.161
7.179
7.197
7.199
7.252
7.274
7.276
7.294
7.312
7.315
7.322
7.327
7.384
7.340
7.346
7.358
7.370
7.389
7.391
7.412
7.769
7.809
ppm
0
1
2
3
4
5
6
7
8
9
3.00
3.01
3.00
2.03
2.01
7.00
1.00


Me
Me
N
Me
3m
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19.824
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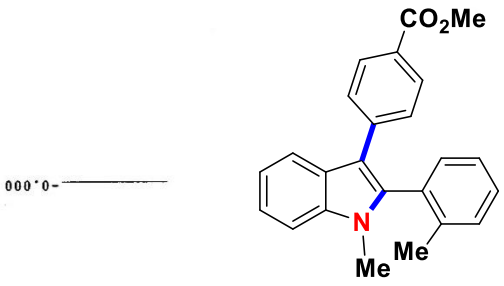
76.685
77.000
77.319
109.467
114.988
119.751
119.979
122.021
127.022
127.239
127.884
128.298
129.470
130.992
131.133
132.089
132.548
133.641
136.138
137.258
137.447
ppm
20
40
60
80
100
120
140
160
180


CO2Me
N
Me
Me

3n

0.000
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3.877
7.244
7.254
7.259
7.279
7.331
7.352
7.425
7.445
7.872
7.889
7.910
ppm
0
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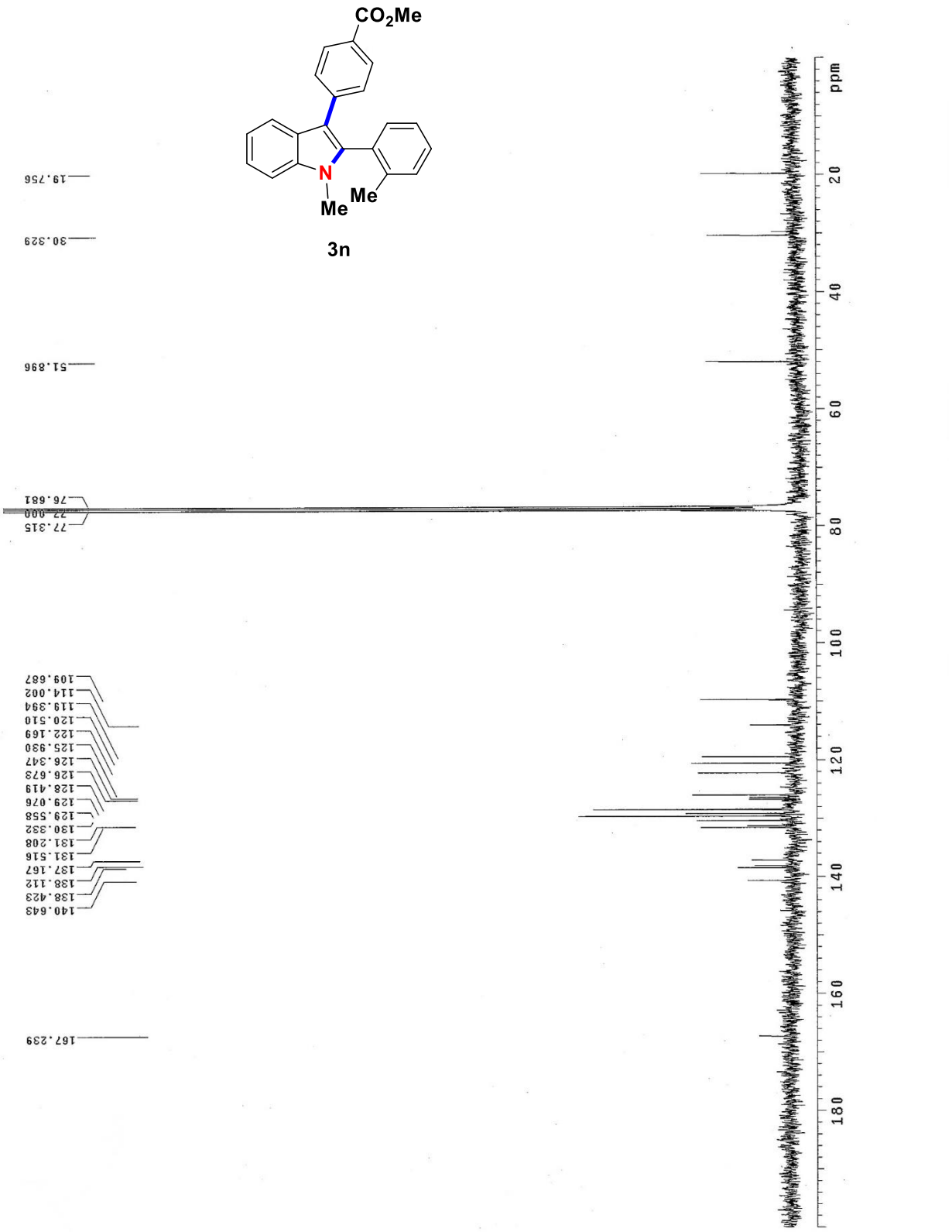
3
4
5
6
7
8
9
3.00
3.07
3.00
1.05
4.03
4.11
3.03


CO2Me
N
Me
Me
3n
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77.315
109.687
114.002
119.394
120.510
122.169
125.930
126.347
126.673
128.419
129.076
129.558
130.332
131.208
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138.112
138.423
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167.239
ppm
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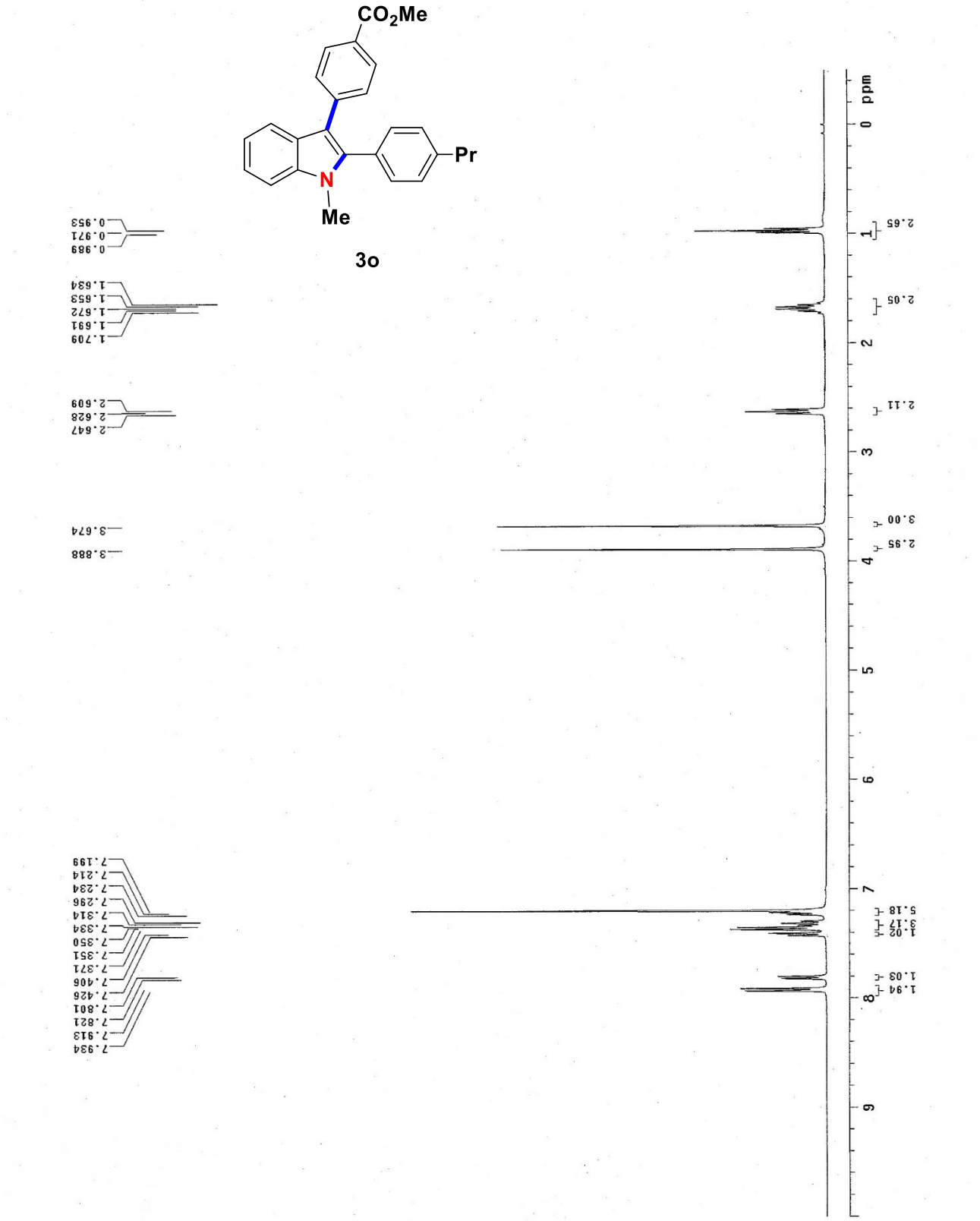
40
60
80
100
120
140
160
180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Pr
N
Me
3o
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1.691
1.709
2.609
2.628
2.647
3.674
3.888
7.199
7.214
7.234
7.296
7.314
7.334
7.350
7.351
7.371
7.406
7.426
7.801
7.821
7.913
7.934
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2.05
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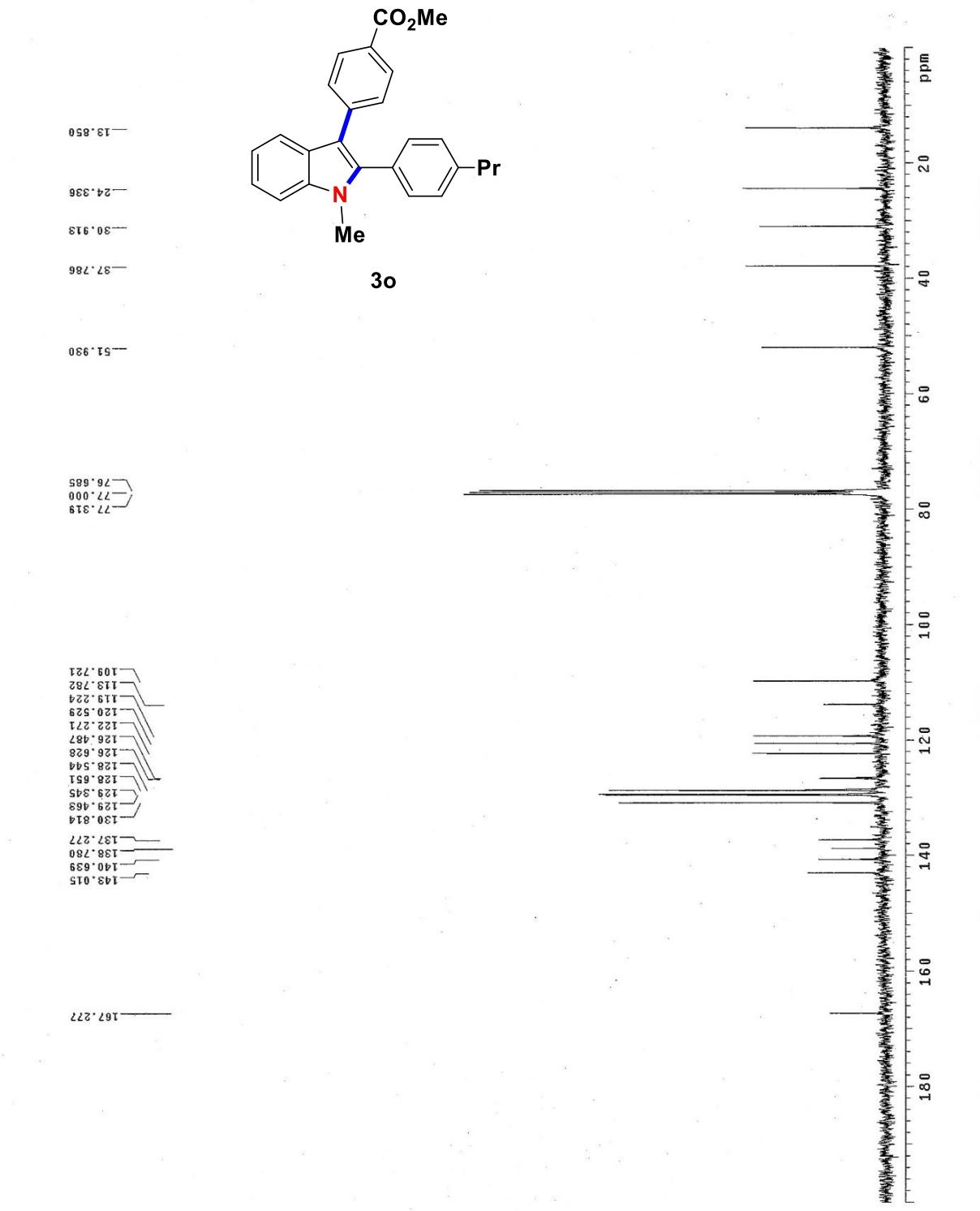
3.00
2.95
1.02
3.17
5.18
1.03
1.94


CO2Me
Pr
N
Me
3o
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87.786
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77.000
77.319
109.721
113.782
119.224
120.529
122.271
126.487
126.628
128.544
128.651
129.345
129.463
130.814
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140.639
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ppm
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40
60
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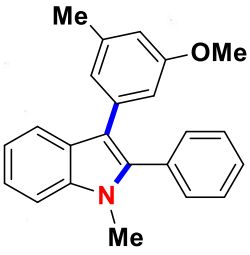
120
140
160
180


Me
OMe
N
Me

3p

2.254
3.580
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6.547
6.577
6.794
7.168
7.187
7.205
7.273
7.275
7.294
7.312
7.314
7.322
7.326
7.333
7.340
7.345
7.358
7.363
7.367
7.377
7.385
7.405
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7.842
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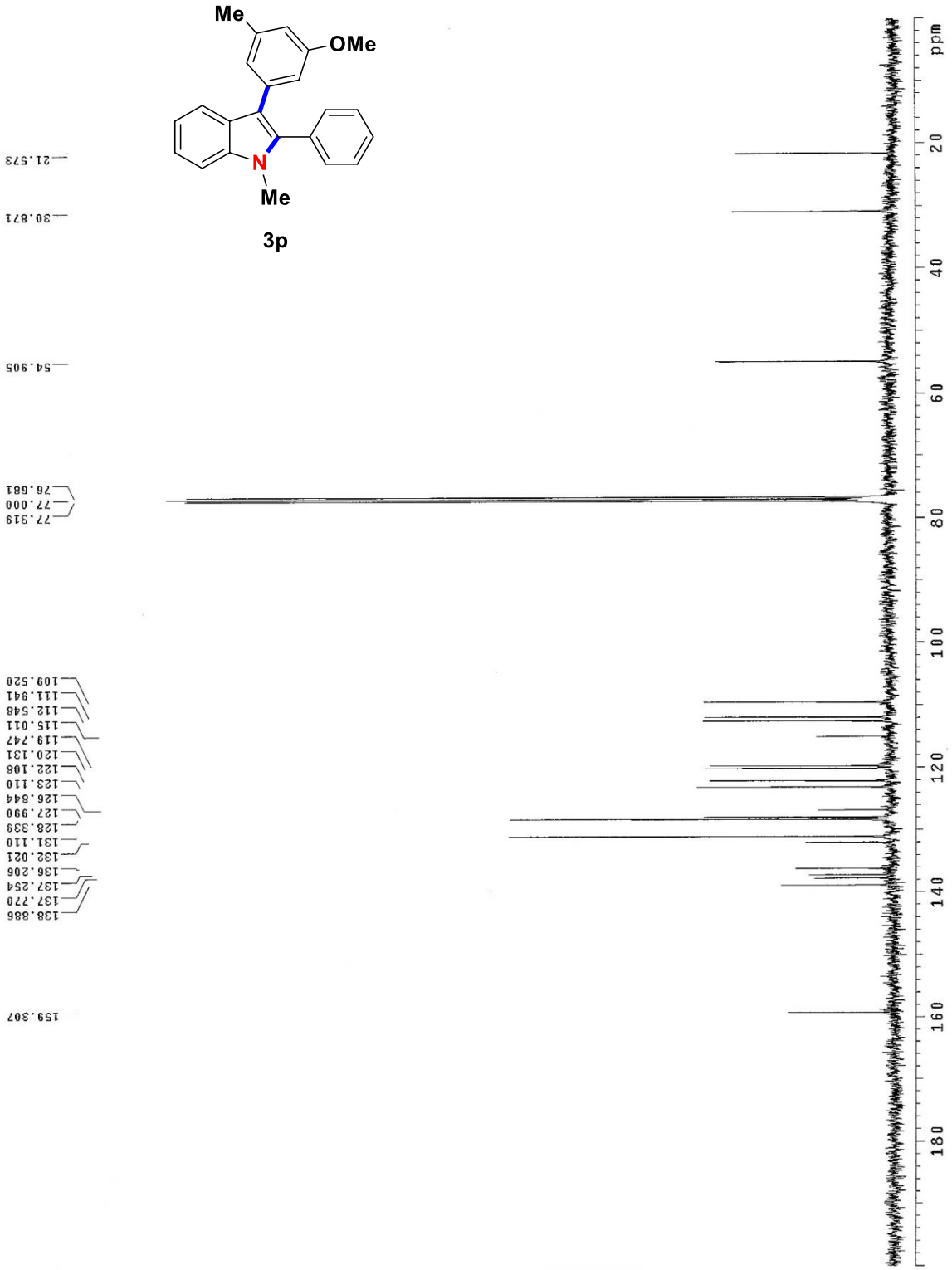
1.90
1.00
1.08
7.05
1.05
ppm


Me
OMe
N
Me
3p
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77.000
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111.941
112.548
115.011
119.747
120.131
122.108
123.110
126.844
127.990
128.339
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137.770
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40
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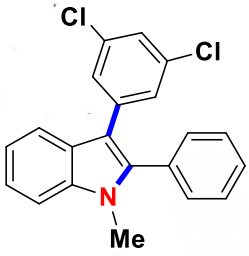
Cl
Cl
N
Me

5a

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7.426
7.417
7.415
7.408
7.401
7.387
7.332
7.330
7.315
7.312
7.303
7.294
7.285
7.279
7.237
7.234
7.217
7.199
7.197
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7.142
7.135
7.132
7.127
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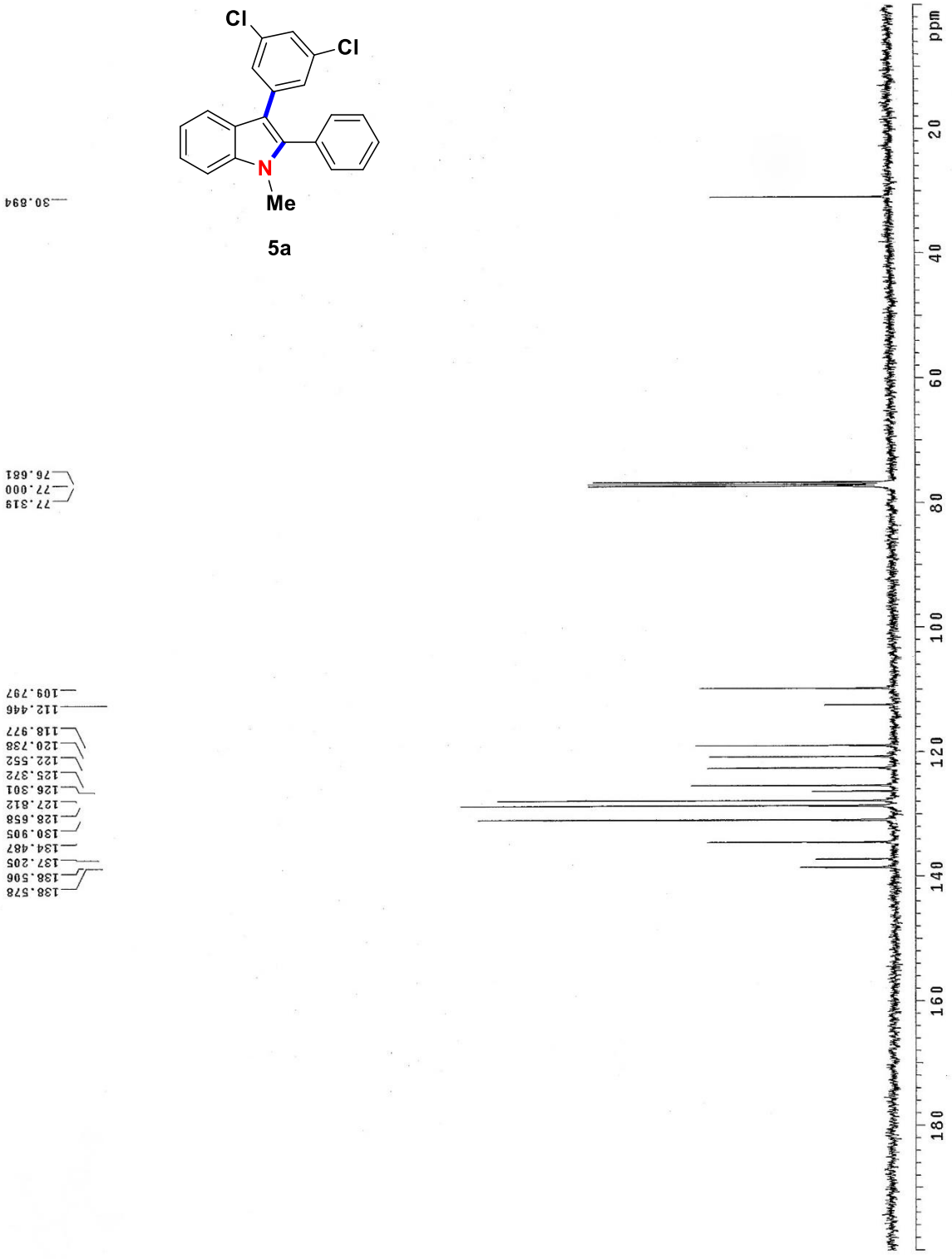
4.02
3.03
1.09
2.68
0 ppm


Cl
Cl
N
Me
5a
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109.797
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118.977
120.738
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127.812
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130.905
134.487
137.205
138.506
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ppm
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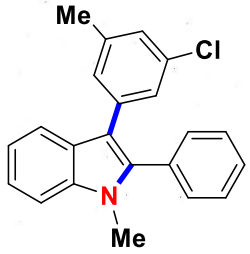
40
60
80
100
120
140
160
180


Me
Cl
N
Me

5b

2.202
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6.958
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7.173
7.175
7.193
7.210
7.275
7.283
7.289
7.296
7.307
7.364
7.369
7.376
7.381
7.393
7.756
7.776
ppm
0
1
2
3
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7
8
9
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1.00
4.05
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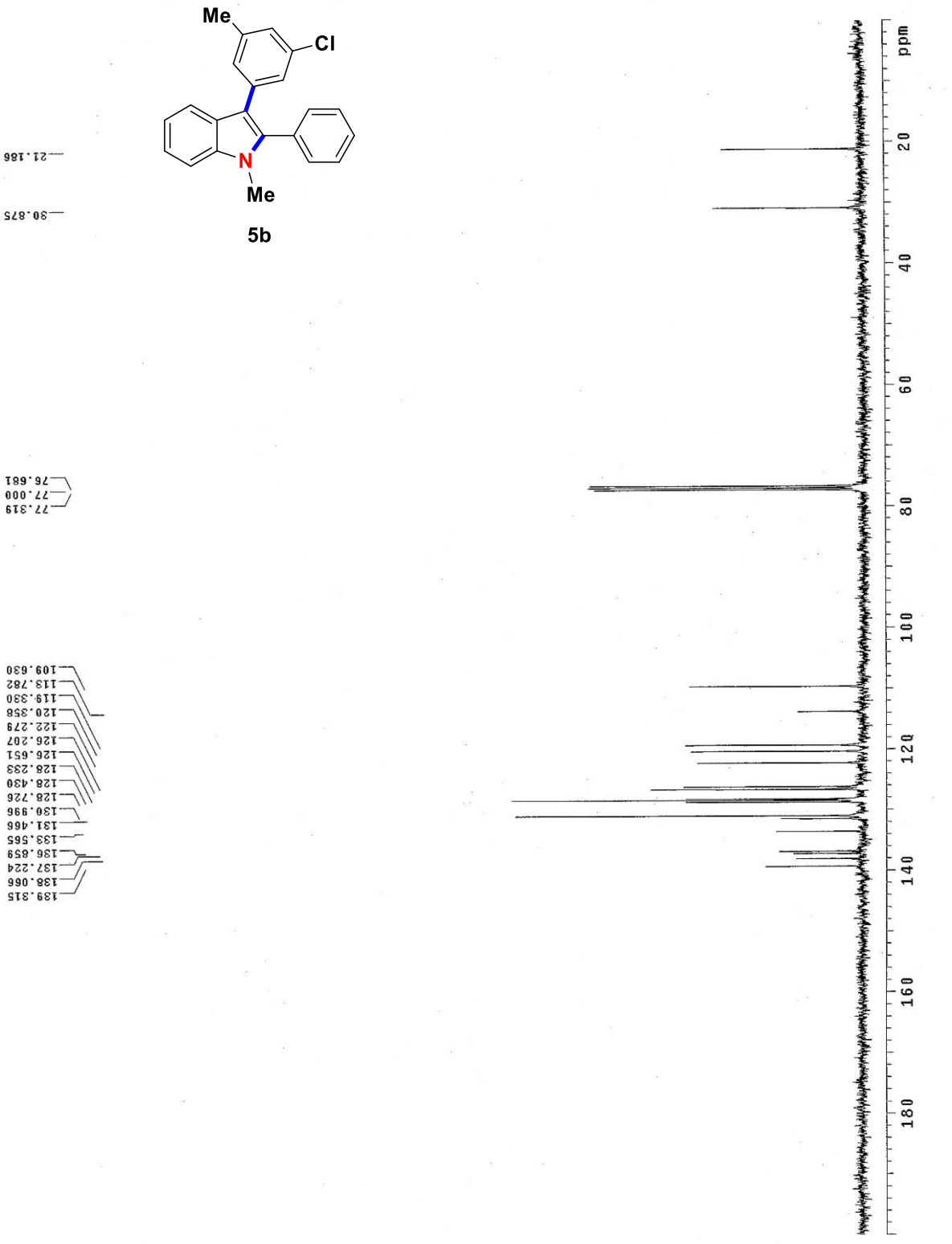
1.86


Me
Cl
N
Me
5b
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120.356
122.279
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126.651
128.283
128.430
128.726
130.996
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137.224
138.066
139.315
ppm
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100
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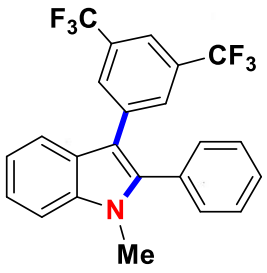
F3C
CF3
N
Me

5c

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7.296
7.300
7.306
7.309
7.315
7.318
7.347
7.365
7.367
7.385
7.435
7.443
7.445
7.449
7.451
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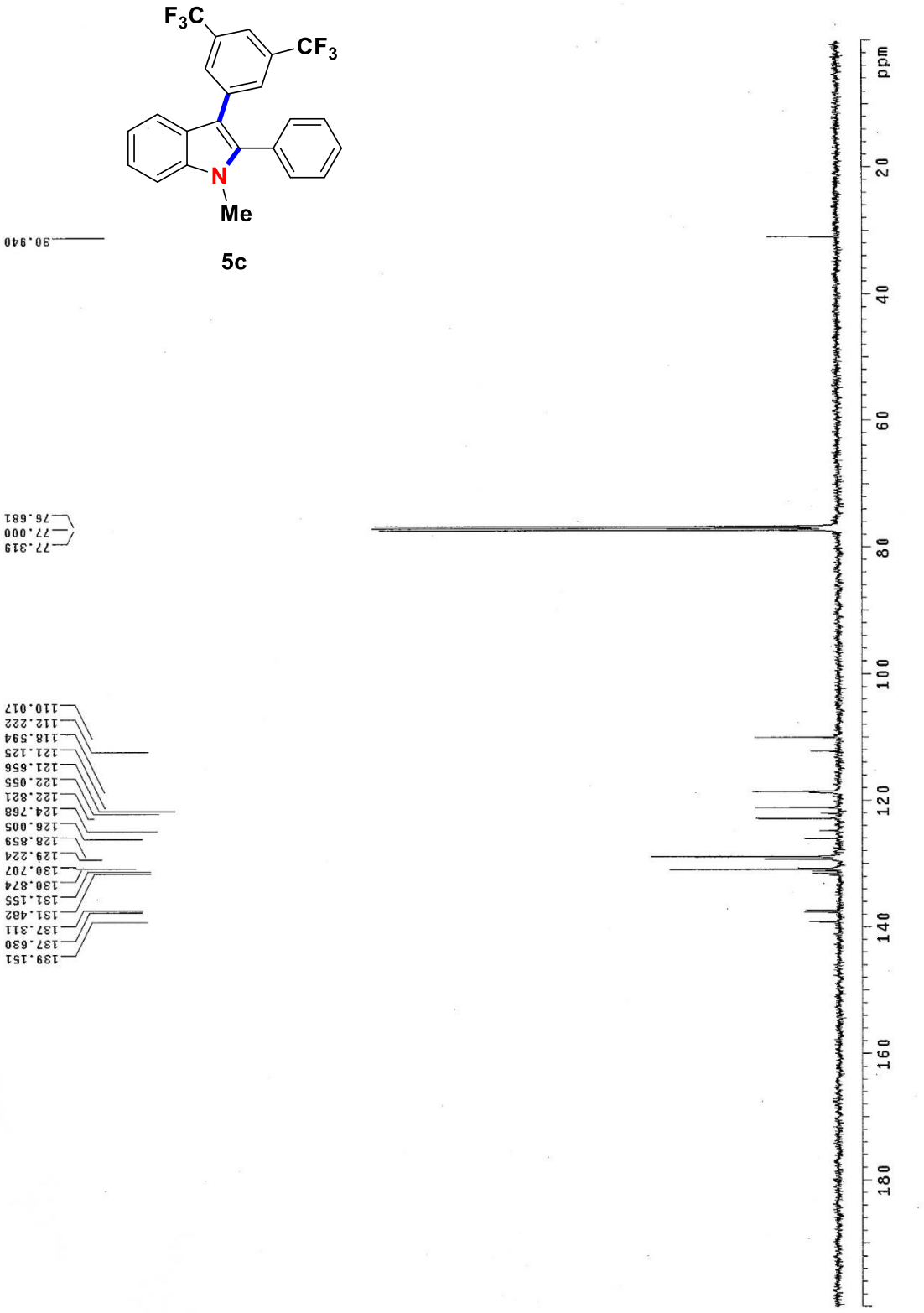
ppm


F3C
CF3
N
Me
5c
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77.319
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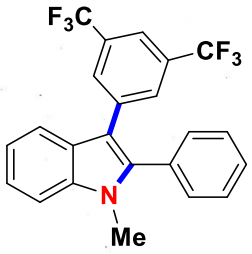
112.222
118.594
121.125
121.656
122.055
122.821
124.768
126.005
128.859
129.224
130.707
130.874
131.155
131.482
137.311
137.630
139.151
ppm
20
40
60
80
100
120
140
160
180



5c

4. Crystal description and data

Table 1. Crystal data and structure refinement for **3o**.

Identification code	**3o**	
Empirical formula	C26 H25 N O2	
Formula weight	383.47	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.0443(9) Å	α= 114.161(3)°.
	b = 10.5766(11) Å	β= 98.162(3)°.
	c = 12.7178(11) Å	γ = 104.309(3)°.
Volume	1034.08(18) $Å^3$	
Z	2	
Density (calculated)	1.232 Mg/m^3	
Absorption coefficient	0.077 mm^{-1}	
F(000)	408	
Crystal size	0.430 x 0.360 x 0.290 mm^3	
Theta range for data collection	3.283 to 26.438°.	
Index ranges	-11<=h<=11, -13<=k<=13, -15<=l<=15	
Reflections collected	30990	
Independent reflections	4168 [R(int) = 0.0481]	
Completeness to theta = 25.242°	98.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9281 and 0.8394	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4168 / 0 / 262	
Goodness-of-fit on F^2	1.111	
Final R indices [I>2sigma(I)]	R1 = 0.0551, wR2 = 0.1207	
R indices (all data)	R1 = 0.0762, wR2 = 0.1320	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.290 and -0.270 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) For **3o**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3789(2)	12191(2)	5037(1)	31(1)
O(2)	2086(2)	11206(2)	5842(1)	41(1)
N	2188(2)	3415(2)	-234(1)	17(1)
C(1)	2028(2)	2894(2)	591(2)	18(1)
C(2)	1856(2)	1488(2)	461(2)	23(1)
C(3)	1808(2)	1277(2)	1459(2)	26(1)
C(4)	1916(2)	2433(2)	2555(2)	27(1)
C(5)	2059(2)	3822(2)	2677(2)	23(1)
C(6)	2120(2)	4072(2)	1682(2)	18(1)
C(7)	2346(2)	5344(2)	1488(2)	17(1)
C(8)	2400(2)	4907(2)	315(2)	16(1)
C(9)	2057(3)	2483(2)	-1484(2)	26(1)
C(10)	2488(2)	6830(2)	2392(2)	17(1)
C(11)	1414(2)	6983(2)	3084(2)	22(1)
C(12)	1529(2)	8354(2)	3952(2)	24(1)
C(13)	2715(2)	9606(2)	4142(2)	20(1)
C(14)	3789(2)	9472(2)	3454(2)	22(1)
C(15)	3678(2)	8099(2)	2590(2)	21(1)
C(16)	2806(2)	11053(2)	5095(2)	24(1)
C(17)	3917(3)	13629(2)	5935(2)	40(1)
C(18)	2657(2)	5808(2)	-316(2)	16(1)
C(19)	1756(2)	6720(2)	-278(2)	18(1)
C(20)	2052(2)	7639(2)	-802(2)	21(1)
C(21)	3253(2)	7693(2)	-1375(2)	21(1)
C(22)	4119(2)	6753(2)	-1439(2)	20(1)
C(23)	3831(2)	5828(2)	-915(2)	19(1)
C(24)	3602(3)	8720(2)	-1930(2)	29(1)
C(25)	2241(3)	8376(3)	-2961(2)	36(1)
C(26)	1800(5)	6839(3)	-3981(2)	76(1)

Table 3. Bond lengths [Å] and angles [°] for **3o**.

O(1)-C(16)	1.344(3)
O(1)-C(17)	1.444(2)
O(2)-C(16)	1.207(2)
N-C(1)	1.380(2)
N-C(8)	1.388(2)
N-C(9)	1.456(2)
C(1)-C(2)	1.393(3)
C(1)-C(6)	1.409(3)
C(2)-C(3)	1.380(3)
C(2)-H(2A)	0.9500
C(3)-C(4)	1.398(3)
C(3)-H(3A)	0.9500
C(4)-C(5)	1.383(3)
C(4)-H(4A)	0.9500
C(5)-C(6)	1.401(3)
C(5)-H(5A)	0.9500
C(6)-C(7)	1.436(2)
C(7)-C(8)	1.382(3)
C(7)-C(10)	1.479(2)
C(8)-C(18)	1.473(2)
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-C(11)	1.397(3)
C(10)-C(15)	1.398(3)
C(11)-C(12)	1.383(3)
C(11)-H(11A)	0.9500
C(12)-C(13)	1.387(3)
C(12)-H(12A)	0.9500
C(13)-C(14)	1.392(3)
C(13)-C(16)	1.487(3)
C(14)-C(15)	1.384(3)
C(14)-H(14A)	0.9500
C(15)-H(15A)	0.9500
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800

C(17)-H(17C)	0.9800
C(18)-C(23)	1.394(3)
C(18)-C(19)	1.400(2)
C(19)-C(20)	1.384(3)
C(19)-H(19A)	0.9500
C(20)-C(21)	1.394(3)
C(20)-H(20A)	0.9500
C(21)-C(22)	1.394(3)
C(21)-C(24)	1.516(3)
C(22)-C(23)	1.387(3)
C(22)-H(22A)	0.9500
C(23)-H(23A)	0.9500
C(24)-C(25)	1.515(3)
C(24)-H(24A)	0.9900
C(24)-H(24B)	0.9900
C(25)-C(26)	1.514(4)
C(25)-H(25A)	0.9900
C(25)-H(25B)	0.9900
C(26)-H(26A)	0.9800
C(26)-H(26B)	0.9800
C(26)-H(26C)	0.9800

C(16)-O(1)-C(17)	115.06(17)
C(1)-N-C(8)	108.80(15)
C(1)-N-C(9)	123.53(15)
C(8)-N-C(9)	127.59(16)
N-C(1)-C(2)	129.23(17)
N-C(1)-C(6)	108.20(15)
C(2)-C(1)-C(6)	122.53(17)
C(3)-C(2)-C(1)	117.53(18)
C(3)-C(2)-H(2A)	121.2
C(1)-C(2)-H(2A)	121.2
C(2)-C(3)-C(4)	120.98(18)
C(2)-C(3)-H(3A)	119.5
C(4)-C(3)-H(3A)	119.5
C(5)-C(4)-C(3)	121.42(18)
C(5)-C(4)-H(4A)	119.3
C(3)-C(4)-H(4A)	119.3

C(4)-C(5)-C(6)	118.94(18)
C(4)-C(5)-H(5A)	120.5
C(6)-C(5)-H(5A)	120.5
C(5)-C(6)-C(1)	118.58(17)
C(5)-C(6)-C(7)	134.55(17)
C(1)-C(6)-C(7)	106.79(16)
C(8)-C(7)-C(6)	107.14(15)
C(8)-C(7)-C(10)	127.43(16)
C(6)-C(7)-C(10)	125.43(16)
C(7)-C(8)-N	109.07(16)
C(7)-C(8)-C(18)	128.49(16)
N-C(8)-C(18)	122.43(16)
N-C(9)-H(9A)	109.5
N-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
N-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(11)-C(10)-C(15)	118.34(16)
C(11)-C(10)-C(7)	119.37(17)
C(15)-C(10)-C(7)	122.29(16)
C(12)-C(11)-C(10)	120.86(18)
C(12)-C(11)-H(11A)	119.6
C(10)-C(11)-H(11A)	119.6
C(13)-C(12)-C(11)	120.31(18)
C(13)-C(12)-H(12A)	119.8
C(11)-C(12)-H(12A)	119.8
C(12)-C(13)-C(14)	119.47(17)
C(12)-C(13)-C(16)	118.35(17)
C(14)-C(13)-C(16)	122.18(18)
C(15)-C(14)-C(13)	120.21(18)
C(15)-C(14)-H(14A)	119.9
C(13)-C(14)-H(14A)	119.9
C(14)-C(15)-C(10)	120.81(17)
C(14)-C(15)-H(15A)	119.6
C(10)-C(15)-H(15A)	119.6
O(2)-C(16)-O(1)	123.12(18)
O(2)-C(16)-C(13)	124.46(19)

O(1)-C(16)-C(13)	112.42(17)
O(1)-C(17)-H(17A)	109.5
O(1)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
O(1)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(23)-C(18)-C(19)	118.27(16)
C(23)-C(18)-C(8)	121.67(16)
C(19)-C(18)-C(8)	119.99(16)
C(20)-C(19)-C(18)	120.56(17)
C(20)-C(19)-H(19A)	119.7
C(18)-C(19)-H(19A)	119.7
C(19)-C(20)-C(21)	121.37(17)
C(19)-C(20)-H(20A)	119.3
C(21)-C(20)-H(20A)	119.3
C(22)-C(21)-C(20)	117.86(17)
C(22)-C(21)-C(24)	120.63(17)
C(20)-C(21)-C(24)	121.51(17)
C(23)-C(22)-C(21)	121.15(17)
C(23)-C(22)-H(22A)	119.4
C(21)-C(22)-H(22A)	119.4
C(22)-C(23)-C(18)	120.74(17)
C(22)-C(23)-H(23A)	119.6
C(18)-C(23)-H(23A)	119.6
C(25)-C(24)-C(21)	113.41(17)
C(25)-C(24)-H(24A)	108.9
C(21)-C(24)-H(24A)	108.9
C(25)-C(24)-H(24B)	108.9
C(21)-C(24)-H(24B)	108.9
H(24A)-C(24)-H(24B)	107.7
C(24)-C(25)-C(26)	112.8(2)
C(24)-C(25)-H(25A)	109.0
C(26)-C(25)-H(25A)	109.0
C(24)-C(25)-H(25B)	109.0
C(26)-C(25)-H(25B)	109.0
H(25A)-C(25)-H(25B)	107.8
C(25)-C(26)-H(26A)	109.5

C(25)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
C(25)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3o**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	46(1)	17(1)	23(1)	5(1)	7(1)	11(1)
O(2)	46(1)	34(1)	32(1)	1(1)	21(1)	13(1)
N	20(1)	15(1)	18(1)	7(1)	6(1)	8(1)
C(1)	14(1)	20(1)	21(1)	11(1)	4(1)	6(1)
C(2)	21(1)	17(1)	29(1)	11(1)	5(1)	6(1)
C(3)	24(1)	19(1)	38(1)	18(1)	4(1)	5(1)
C(4)	25(1)	32(1)	30(1)	23(1)	4(1)	6(1)
C(5)	24(1)	25(1)	21(1)	13(1)	5(1)	6(1)
C(6)	16(1)	18(1)	20(1)	9(1)	3(1)	6(1)
C(7)	15(1)	18(1)	20(1)	9(1)	6(1)	6(1)
C(8)	13(1)	16(1)	20(1)	8(1)	5(1)	6(1)
C(9)	34(1)	19(1)	21(1)	5(1)	8(1)	9(1)
C(10)	22(1)	17(1)	14(1)	8(1)	5(1)	9(1)
C(11)	22(1)	21(1)	21(1)	9(1)	8(1)	4(1)
C(12)	26(1)	27(1)	20(1)	10(1)	11(1)	12(1)
C(13)	27(1)	20(1)	15(1)	8(1)	5(1)	11(1)
C(14)	25(1)	18(1)	22(1)	9(1)	7(1)	4(1)
C(15)	24(1)	21(1)	20(1)	10(1)	10(1)	8(1)
C(16)	27(1)	24(1)	20(1)	7(1)	2(1)	11(1)
C(17)	52(2)	19(1)	35(1)	1(1)	4(1)	13(1)
C(18)	17(1)	14(1)	14(1)	6(1)	2(1)	5(1)
C(19)	19(1)	18(1)	19(1)	9(1)	8(1)	8(1)
C(20)	24(1)	18(1)	25(1)	12(1)	10(1)	12(1)
C(21)	22(1)	18(1)	22(1)	10(1)	6(1)	5(1)
C(22)	17(1)	26(1)	21(1)	12(1)	9(1)	8(1)
C(23)	19(1)	22(1)	20(1)	10(1)	6(1)	11(1)
C(24)	30(1)	29(1)	39(1)	24(1)	16(1)	10(1)
C(25)	43(1)	39(1)	37(1)	29(1)	12(1)	10(1)
C(26)	138(3)	42(2)	32(2)	21(1)	2(2)	12(2)

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

	x	y	z	U(eq)
H(2A)	1775	703	-287	27
H(3A)	1700	332	1400	32
H(4A)	1892	2261	3230	33
H(5A)	2114	4594	3424	28
H(9A)	2211	3084	-1902	39
H(9B)	2868	2011	-1528	39
H(9C)	1002	1725	-1864	39
H(11A)	593	6134	2959	26
H(12A)	793	8439	4419	28
H(14A)	4602	10325	3577	26
H(15A)	4418	8018	2127	25
H(17A)	4654	14388	5826	61
H(17B)	2870	13744	5855	61
H(17C)	4314	13733	6735	61
H(19A)	934	6708	111	22
H(20A)	1423	8246	-772	25
H(22A)	4920	6746	-1849	24
H(23A)	4441	5202	-966	23
H(24A)	3852	9744	-1304	35
H(24B)	4553	8659	-2222	35
H(25A)	2540	9100	-3265	43
H(25B)	1303	8482	-2662	43
H(26A)	918	6673	-4622	114
H(26B)	2717	6733	-4293	114
H(26C)	1480	6115	-3689	114