Copper-Catalyzed Synthesis of *N*-Aryl and *N*-Sulfonyl Indoles from 2-Vinylanilines with O_2 as the Terminal Oxidant and TEMPO as Co-Catalyst

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Supporting Information

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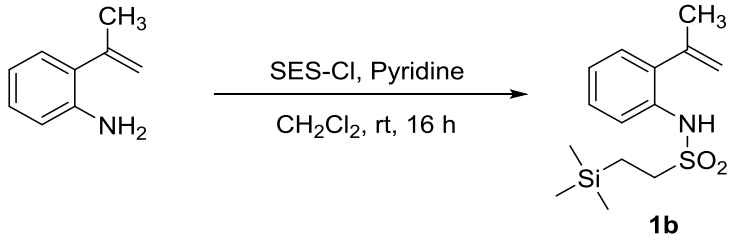
General Information:

All reagents were used out of the bottle as purchased from the supplier without further purification unless otherwise noted. Substrates **1a,e,g** were synthesized according to literature procedures.[1] Achiral bis(oxazoline) ligand **3** was synthesized according to the procedure reported by Miao et. al.[2] ^{1}H NMR spectra were recorded in $CDCl_3$ (using 7.26 ppm as internal reference) at 300, 400 or 500 MHz. ^{13}C NMR spectra were recorded in $CDCl_3$ (using 77.0 ppm as internal reference) at 75 MHz unless otherwise noted. ^{19}F NMR spectra were recorded in $CDCl_3$ with α,α,α-trifluorotoluene as an internal reference. IR spectra were taken neat using a Nicolet-Impact 420 FTIR. Wave numbers in cm^{-1} are reported for characteristic peaks. High resolution mass spectra were obtained at SUNY Buffalo's mass spec. facility on a ThermoFinnigan MAT XL spectrometer. Melting points are reported as uncorrected.

Synthesis of Aniline Substrates:
Substrates 1b-d,f
Substrates **1b-d,f** were synthesized by sulfonylation of the corresponding aniline.



1b

N-(2-(Prop-1-en-2-yl)phenyl)-2-(trimethylsilyl)ethane-1-sulfonamide (1b)
The aniline (0.5 g, 3.75 mmol, 1 equiv) was dissolved in dry CH_2Cl_2 (7.5 mL, 0.5 M), and the solution was treated with sulfonyl chloride (0.79 g, 4.13 mmol, 1.1 equiv) and pyridine (0.91 mL, 11.3 mmol, 3 equiv). The mixture was stirred at room temperature for 24 h, diluted with H_2O (25 mL) and extracted with CH_2Cl_2 (3 x 25 mL). The combined organic layers were washed with 1M HCl, brine, dried over Na_2SO_4, and concentrated in vacuo. Flash chromatography of the resulting crude mixture on SiO_2 (0-30% EtOAc in hexanes gradient) afforded the sulfonamides in good yields. Substrate **1b** was synthesized according to the above procedure using 2-isopropenylaniline and 2-(trimethylsilyl)ethanesulfonyl chloride. Sulfonamide **1b** was obtained (0.33 g, 97%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (d, J = 8.0 Hz, 1H), 7.25 (t, J = 7.2 Hz, 1H), 7.16 (d, J = 8.0 Hz, 1H), 7.08 (t, J = 7.2 Hz, 1H), 6.83 (bs, 1H), 5.42 (t, J = 1.6 Hz, 1H), 5.02 (t, J = 0.8 Hz, 1H), 3.06-3.02 (m, 2H), 2.08 (d, J = 1.6 Hz, 3H), 1.01-0.98 (m, 2H), -0.20 (s, 9H). ^{13}C NMR (75 Hz, $CDCl_3$) δ 142.0, 133.4, 133.3, 128.3, 123.8, 118.2, 117.6, 48.1, 24.6, 10.4, -2.1; IR (neat): 3343, 3288, 2953, 1493, 1396, 1335, 1251, 1169, 1149, 911, 861, 841, 761 cm^{-1}; HRMS (EI) calcd for $[M]^+$ $C_{14}H_{23}O_2NSSi$: 297.1204, found: 297.1213.

3-Nitro-4-((2-(prop-1-en-2-yl)phenyl)amino)benzenesulfonic acid (1c)[3]
Substrate **1c** was synthesized according to the above procedure using 2-isopropenylaniline and *p*-nitrobenzenesulfonyl chloride. The known sulfonamide **1c** was obtained (0.48 g, 99%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.27 (d, J = 8 Hz, 2H), 7.94-7.91 (m, 2H), 7.63 (d, J = 8.4 Hz, 1H), 7.26 (t, J = 7.2 Hz, 1 H), 7.13-7.04 (m, 2H), 5.28 (t, J = 1.6 Hz, 1H), 4.67 (s, 1H), 1.70 (s, 3H).

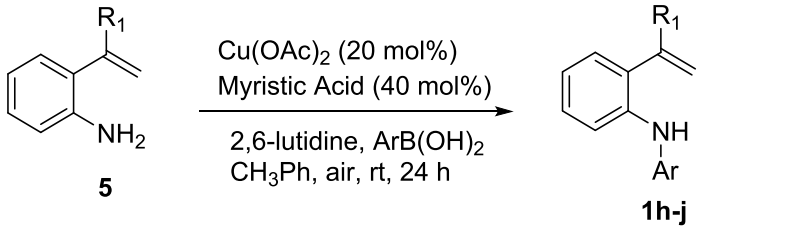
N-(2-(Prop-1-en-2-yl)phenyl)methanesulfonamide (1d)[4]

Substrate **1d** was synthesized according to the above procedure using 2-isopropenylaniline and methanesulfonyl chloride. The known sulfonamide **1d** was obtained (0.3 g, 99%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, J = 8.0 Hz, 1H), 7.28 (t, J = 8.0 Hz, 1H), 7.18 (d, J = 7.6 Hz, 1H), 7.12 (t, J = 7.6 Hz, 1H), 6.85 (bs, 1H), 5.42-5.41 (m, 1H), 5.01-5.00 (m, 1H), 3.01 (s, 3H), 2.08-2.07 (m, 3H).

(E,Z)-4-Methyl-N-(2-(1-phenylprop-1-en-1-yl)phenyl)benzenesulfonamide (1f)

Substrate **1f** was synthesized according to the above procedure using 2-(1-phenylprop-1-en-1-yl)aniline[5] and p-toluenesulfonyl chloride. Sulfonamide **1f** was obtained (0.14 g, 88%) as a colorless oil in a 3:2 Z:E ratio. Product characterized as a mixture of isomers. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (d, J = 7.6 Hz, 1H), 7.57 (d, J = 8.4 Hz, 0.6H), 7.46 (d, J = 8.8 Hz, 2H), 7.37 (d, J = 8.4 Hz, 1.3H), 7.32-7.07 (m, 13H), 7.00-6.93 (m, 4H), 6.54 (bs, 1H), 6.42 (bs, 0.6H), 6.38 (q, J = 7.2 Hz, 1H), 5.42 (q, J = 7.2 Hz, 0.6H), 2.38 (s, 2H), 2.36 (s, 3H), 1.77 (d, J = 6.8 Hz, 2H), 1.47 (d, J = 7.2 Hz, 3H); ^{13}C NMR (75 Hz, $CDCl_3$) δ 143.6, 143.5, 139.6, 138.1, 137.0, 136.1, 134.6, 134.0, 130.8, 129.5, 129.4, 129.0, 128.9, 128.7, 128.6, 128.3, 127.7, 127.7, 127.4, 127.2, 127.2, 125.9, 124.9, 124.2, 121.7, 119.0, 21.5, 15.4, 15.3; IR (neat): 3326. 3057, 1491, 1396, 1336, 1166, 1090, 918, 760, 667 cm^{-1}; HRMS (ESI) calcd for $[M+Na]^+$ $C_{22}H_{21}O_2NNaS$: 386.1199, found: 386.1185.

Substrates 1h-j



R_1, Ar =-Me, *p*-FC_6H_4 (**1h**)
R_1, Ar =-Me, *p*-$OMeC_6H_4$ (**1i**)
R_1, Ar =-H, *p*-$OMeC_6H_4$ (**1j**)

Substrates **1h-j** were synthesized using a procedure reported by Buchwald et. al.[6] The $ArB(OH)_2$ (0.28 g, 2.26 mmol, 1.2 equiv), $Cu(OAc)_2$ (0.07 g, 0.38 mmol, 0.2 equiv) and myristic acid (0.172 g, 0.75 mmol, 0.4 equiv) were dissolved in toluene (3.8 mL, 0.5M) in a 100 mL round bottom flask. The reaction was stirred and 2,6-lutidine (0.22 mL, 1.88 mmol, 1 equiv) and the corresponding aniline (0.26 mL, 1.88 mmol, 1 equiv) were added. The reaction was stirred at room temperature with the flask uncapped and open to air for 24 h. The reaction was filtered through a SiO_2 plug with ether (100 mL) and the filtrate was condensed. Crude mixtures were purified via flash chromatography on SiO_2 (0-10% Et_2O in hexanes gradient) to afford the anilines in high yields.

N-(4-Fluorophenyl)-2-(prop-1-en-2-yl)aniline (1h)

Substrate **1h** was synthesized according to the above procedure using 2-isopropenylaniline and 4-fluorophenylboronic acid. Aniline **1h** was obtained (0.22 g, 65%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18-7.15 (m, 3H), 7.07-6.97 (m, 4H), 6.92-6.87 (m, 1H), 5.80 (bs, 1H), 5.32 (q, J = 2.2 Hz, 1H), 5.08 (t, J = 1.6 Hz, 1H), 2.09 (d, J = 2.4 Hz, 3H); ^{13}C NMR (75 Hz,

$CDCl_3$) δ 158.1 (d, J_{CF} = 239.3), 143.8, 140.2, 139.3, 139.3, 132.8, 128.7, 127.8, 120.7, 120.5 (d, J_{CF} = 45.8), 116.1, 116.0 (d, J_{CF} = 5.7 Hz), 115.7, 24.0; ^{19}F NMR (282 MHz, $CDCl_3$): δ -123.1; IR (neat): 3407, 3076, 2968, 1575, 1508, 1450, 1310, 1218, 821, 757 cm^{-1}; HRMS (EI) calcd for $[M]^+$ $C_{15}H_{14}FN$: 227.1098, found: 227.1105.

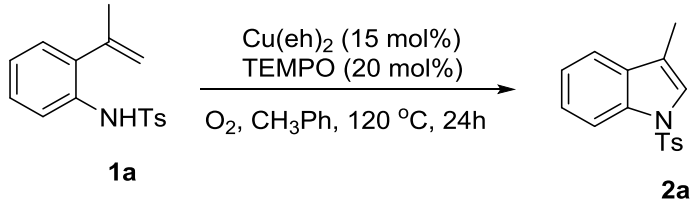
N-(4-Methoxyphenyl)-2-(prop-1-en-2-yl)aniline (1i)[7]

Substrate **1i** was synthesized according to the above procedure using 2-isopropenylaniline and 4-methoxyphenylboronic acid. The known aniline **1i** was obtained (0.18 g, 79%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.12-7.01 (m, 5H), 6.88-6.79 (m, 3H), 5.74 (bs, 1H), 5.32 (s, 1H), 5.09 (s, 1H), 3.80 (s, 3H), 2.09 (s, 3H).

N-(4-methoxyphenyl)-2-vinylaniline (1j)[7]

Substrate **1j** was synthesized according to the above procedure using 2-vinylaniline and 4-methoxyphenylboronic acid. The known aniline **1j** was obtained (0.24 g, 62%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 (d, J = 8 Hz, 1H), 7.15 (t, J = 6.8 Hz, 1H), 7.04 (d, J = 7.6 Hz, 1H), 7.00-6.97 (m, 2H), 6.92-6.85 (m, 4H), 5.68 (dd, J = 17.6, 1.6 Hz, 1H), 5.41 (bs, 1H), 5.33 (dd, J = 10.4, 1.6 Hz, 1H), 3.80 (s, 3H).

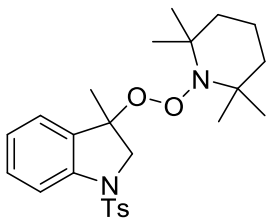
Representative Procedure for the $Cu(eh)_2$ / TEMPO-Catalyzed Aerobic Intramolecular C-H Amination



1a **2a**

3-Methyl-1-tosyl-1H-indole (2a)[1]

To an oven dried 100 mL round bottom flask was charge with **1a** (50 mg, 0.174 mmol, 1 equiv), $Cu(eh)_2$ (9 mg, 0.026 mmol, 0.15 equiv), TEMPO (5 mg, 0.035 mmol, 0.2 equiv) and toluene (1.74 mL, 0.1M). The flask was purged with O_2, put under an O_2 atmosphere using an O_2 balloon and stirred for 24 h at 120 °C. Filtration of the cooled reaction mixture through a pad of silica gel with ethyl acetate (100 mL), and subsequent evaporation of the solvent in vacuo afforded crude mixture. Flash chromatography of the resulting crude mixture on silica gel (0-20% ethyl acetate in hexanes gradient) afforded the known indole **2a** (36 mg, 71% yield) as an off-white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, J = 8.4 Hz, 1H), 7.74 (d, J = 8.0 Hz, 2H), 7.45 (d, J = 7.2 Hz, 1H), 7.34-7.29 (m, 2H), 7.26-7.22 (m, 1H), 7.19 (d, J = 8.4 Hz, 2H), 2.32 (s, 3H), 2.24 (d, J = 0.8 Hz, 3H).



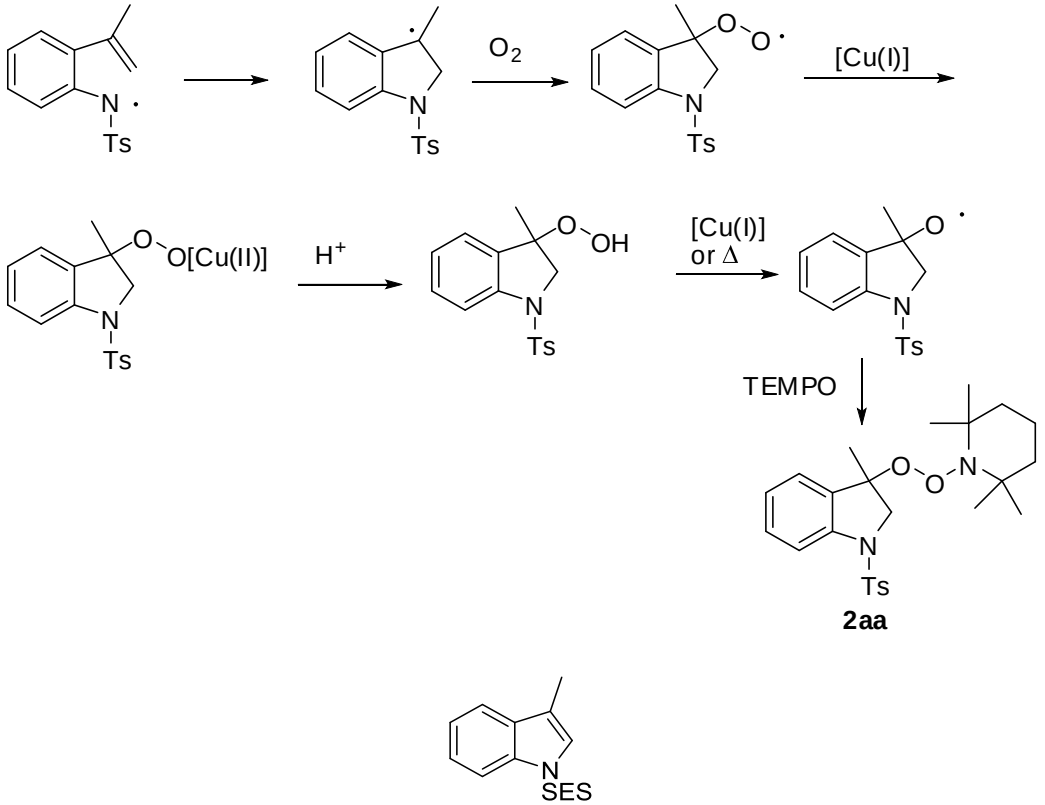
3-Methyl-3-((2,2,6,6-tetramethylpiperidin-1-yl)peroxy)-1-tosylindoline (2aa)

The TEMPO-peroxide product **2aa** was obtained (15 mg, 20% yield) as an off-white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ7.66-7.61 (m, 3H), 7.40 (t, J = 7.2 Hz, 1H), 7.32-7.22 (m, 5H), 6.98

(d, J = 8.1 Hz, 1H), 5.73 (d, J = 9.6 Hz, 1H), 4.81 (d, J = 9.6 Hz, 1H), 2.60 (s, 3H), 2.43 (s, 3H), 1.58-1.25 (m, 9H), 1.13 (s, 3H), 0.96 (s, 3H), 0.67 (s, 3H); ^{13}C NMR (75 Hz, $CDCl_3$) δ 143.6, 141.4, 137.1, 136.1, 132.1, 130.8, 129.2, 128.9, 128.8, 128.2, 84.9, 59.9, 39.8, 33.0, 32.0, 30.0, 29.7, 21.6, 20.3, 20.2, 16.9; IR (neat): 2973, 2928, 2871, 1697, 1347, 1165, 1141, 1024, 869, 814, 686, 660 cm^{-1}; HRMS (ESI) calcd for $[M+H]^+$ $C_{25}H_{35}O_4N_2S$: 459.2312, found: 459.2301.

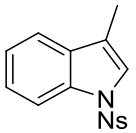
Possible Mechanistic Pathway to 2aa

The following scheme shows a possible rationale for product **2aa**. After *N*-radical addition to the alkene (see **Scheme 2** in manuscript), oxygen can trap the benzylic radical. Reduction by [Cu(I)] and protonation can give a peroxide intermediate that undergoes either Cu-catalyzed or thermal homolysis to an alkoxy radical that can then be trapped by TEMPO to yield **2aa**.



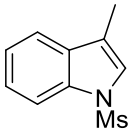
3-Methyl-1-((2-(trimethylsilyl)ethyl)sulfonyl)-1H-indole (2b)

The indole **2b** was obtained (27 mg, 55% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, J = 7.6 Hz, 1H), 7.56 (d, J = 7.6 Hz, 1H), 7.35-7.30 (m, 2H), 7.21 (s, 1H), 3.15-3.10 (m, 2H), 2.30 (d, J = 1.2 Hz, 3H), 0.90-0.84 (m, 2H), -0.05 (s, 9H); ^{13}C NMR (75 Hz, $CDCl_3$) δ 135.4, 131.5, 124.6, 123.6, 122.8, 119.5, 117.1, 113.1, 50.3, 9.9, 9.6, -2.2; IR (neat): 2954, 2922, 1448, 1365, 1273, 1252, 1174, 1158, 1121, 1001, 972, 860, 841, 756, 680 cm^{-1}; HRMS (EI) calcd for $[M]^+$ $C_{14}H_{21}O_2NSSi$: 295.1057, found: 295.1057.



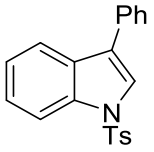
3-Methyl-1-((4-nitrophenyl)sulfonyl)-1H-indole (2c)[3]

The known indole **2c** was obtained (25 mg, 51% yield) as an yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.24 (d, J = 9.0 Hz, 2H), 8.03-7.96 (m, 3H), 7.46 (d, J = 8.1 Hz, 1H), 7.37-7.28 (m, 3H), 2.25 (s, 3H).



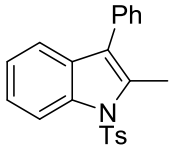
3-Methyl-1-(methylsulfonyl)-1H-indole (2d)[4]

The known indole **2d** was obtained (25 mg, 50% yield) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, J = 8.0 Hz, 1H), 7.56 (d, J = 7.6 Hz, 1H), 7.39-7.31 (m, 2H), 7.20 (s, 1H), 3.03 (s, 3H), 2.30 (s, 3H).



3-Phenyl-1-tosyl-1H-indole (2e)[1]

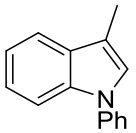
The known indole **2e** was obtained (36 mg, 73% yield) as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (d, J = 8.0 Hz, 1H), 7.82-7.77 (m, 3H), 7.70 (s, 1H), 7.61 (d, J = 8.0 Hz, 2H), 7.47 (t, J = 7.4 Hz, 2H), 7.39-7.35 (m, 2H), 7.30-7.22 (m, 3H), 2.34 (s, 3H



2-Methyl-3-phenyl-1-tosyl-1H-indole (2f)[8]

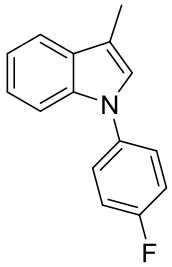
The known indole **2f** was obtained (11 mg, 22% yield) from a 3:2 Z:E ratio of alkene isomers as an off-white solid. Obtained a 22% yield of unreacted (Z)-isomer after reaction. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.26 (d, J = 8.1 Hz, 1H), 7.72 (d, J = 8.1 Hz, 2H), 7.46-7.19 (m, 10H), 2.60 (s, 3H), 2.37 (s, 3H).

When reaction was run with exclusively the (E)-isomer the product was obtained in a 43% yield. When the (Z)-isomer was run it was determined to be unreactive under the reaction conditions.



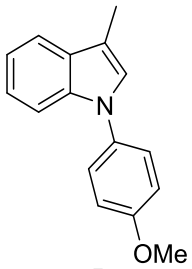
3-Methyl-1-phenyl-1H-indole (2g)[1]

The known indole **2g** was obtained (41 mg, 82% yield) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, J = 8.0 Hz, 1H), 7.56 (d, J = 7.6 Hz, 1H), 7.50-7.47 (m, 4H), 7.32-7.29 (m, 1H), 7.25-7.14 (m, 3H), 2.40 (d, J = 1.2 Hz, 3H)



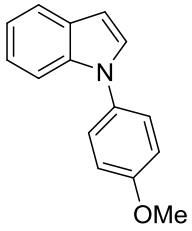
1-(4-Fluorophenyl)-3-methyl-1H-indole (2h)[9]

The known indole **2h** was obtained (40 mg, 81% yield) as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.63 (d, J = 6.6 Hz, 1H), 7.47-7.41 (m, 3H), 7.25-7.16 (m, 4H), 7.08 (s, 1H), 2.40 (s, 3H).



1-(4-Methoxyphenyl)-3-methyl-1H-indole (2i)[7]

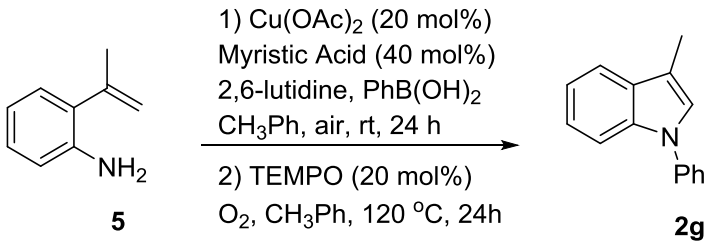
The known indole **2i** was obtained (42 mg, 85% yield) as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.62 (d, J = 7.2 Hz, 1H), 7.44 (d, J = 6.6 Hz, 1H), 7.39 (d, J = 7.2 Hz, 2H), 7.21-7.13 (m, 2H), 7.07 (s, 1H), 7.02 (d, J = 7.2 Hz, 2H), 3.88 (s, 3H), 2.39 (d, J = 1.2 Hz, 3H).



1-(4-Methoxyphenyl)-1H-indole (2j)[7]

The known indole **2j** was obtained (27 mg, 55% yield) as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.70 (d, J = 8.4 Hz, 1H), 7.47 (d, J = 8.4 Hz, 1H), 7.41 (d, J = 6.6 Hz, 2H), 7.29 (d, J = 3 Hz, 1H), 7.22-7.15 (m, 2H), 7.04 (d, J = 6.9 Hz, 2H), 6.67 (d, J = 2.4 Hz, 1H), 3.89 (s, 3H).

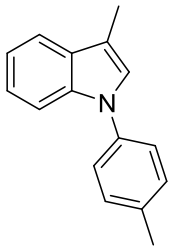
Representative Procedure for the One-Pot Chan-Lam Coupling / C-H Amination



5 **2g**

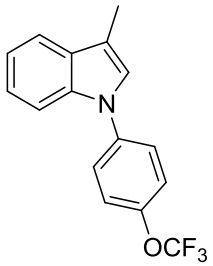
3-Methyl-1-phenyl-1H-indole (2g)[1]

$PhB(OH)_2$ (0.15 g, 1.2 mmol, 1.2 equiv), $Cu(OAc)_2$ (0.036 g, 0.2 mmol, 0.2 equiv) and myristic acid (0.091 g, 0.4 mmol, 0.4 equiv) were dissolved in toluene (2 mL, 0.5M). The reaction was stirred, 2,6-lutidine (0.12 mL, 1 mmol, 1 equiv) and 2-isopropenylaniline (0.14 mL, 1 mmol, 1 equiv) were added. The reaction was stirred at room temperature exposed to air for 24 h. TEMPO (0.031 g, 0.2 mmol, 0.2 equiv) and toluene (8 mL, 0.1M overall) were then added. The flask was purged with O_2, put under an O_2 atmosphere using an O_2 balloon and stirred for 24 h at 120 ^oC. Filtration of the cooled reaction mixture through a pad of silica gel with ethyl acetate (100 mL), and subsequent evaporation of the solvent in vacuo afforded crude mixtures. Flash chromatography of the resulting crude mixture on silica gel (0-5% diethyl ether in hexanes gradient) afforded the known indole **2g** (0.11 g, 53% yield) as a yellow liquid.



3-Methyl-1-(p-tolyl)-1H-indole (1k)[10]

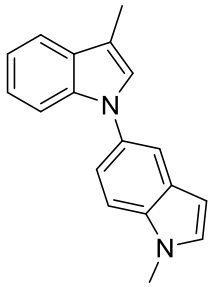
The known indole **2k** was obtained (0.12 g, 54% yield) as a yellow oil from the one-pot reaction using *p*-tolylboronic acid. ^1H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, J = 7.2 Hz, 1H), 7.52 (d, J = 8.0 Hz, 1H), 7.37 (d, J = 8.4 Hz, 2H), 7.30 (d, J = 8.4 Hz, 2H), 7.24-7.15 (m, 2H), 7.12 (s, 1H), 2.43 (s, 3H), 2.39 (d, J = 1.2 Hz, 3H).



3-Methyl-1-(4-(trifluoromethoxy)phenyl)-1H-indole (2l)

The indole **2l** was obtained (0.12 g, 42% yield) as a yellow oil from the one-pot reaction using 4-(trifluoromethoxy)phenyl boronic acid. ^1H NMR (300 MHz, $CDCl_3$): δ 7.63 (d, J = 7.8 Hz, 1H),

7.54-7.49 (m, 3H), 7.35 (d, J = 8.7 Hz, 2H), 7.27-7.17 (m, 2H), 7.10 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (75 Hz, $CDCl_3$) δ 146.7, 138.6, 135.9, 129.9, 125.2, 125.0, 122.7, 122.3, 120.1, 119.3, 118.8, 113.4, 110.1, 110.0, 9.5; ^{19}F NMR (282 MHz, $CDCl_3$): δ -59.0; IR (neat):2922, 1607, 1513, 1457, 1260, 1222, 1167, 740 cm^{-1}; HRMS (EI) calcd for $[M]^+$ $C_{16}H_{12}F_3ON$: 291.0856, found: 291.0866.



1',3-Dimethyl-1'H-1,5'-biindole (2m)

The indole **2m** was obtained (0.1 g, 39% yield) as a brownish solid from the one-pot reaction using N-methylindole-5-boronic acid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, J = 2.0 Hz, 1H), 7.66-7.63 (m, 1H), 7.51-7.49 (m, 1H), 7.42 (d, J = 8.8 Hz, 1H), 7.35-7.32 (m, 1H), 7.20-7.15 (m, 4H), 6.54 (dd, J = 2.8, 0.8 Hz, 1H), 3.86 (s, 3H), 2.42 (d, J = 1.2 Hz, 3H); ^{13}C NMR (75 Hz, $CDCl_3$) δ 136.9, 135.2, 132.3, 130.2, 129.2, 128.8, 126.6, 121.9, 119.2, 119.2, 118.9, 116.8, 111.5, 110.4, 109.8, 101.2, 33.0, 9.6; IR (neat): 3048, 2914, 1572, 1497, 1458, 1330, 1236, 801, 757, 740, 722 cm^{-1}; HRMS (EI) calcd for $[M]^+$ $C_{18}H_{16}N_2$: 260.1305, found: 260.1308.

References

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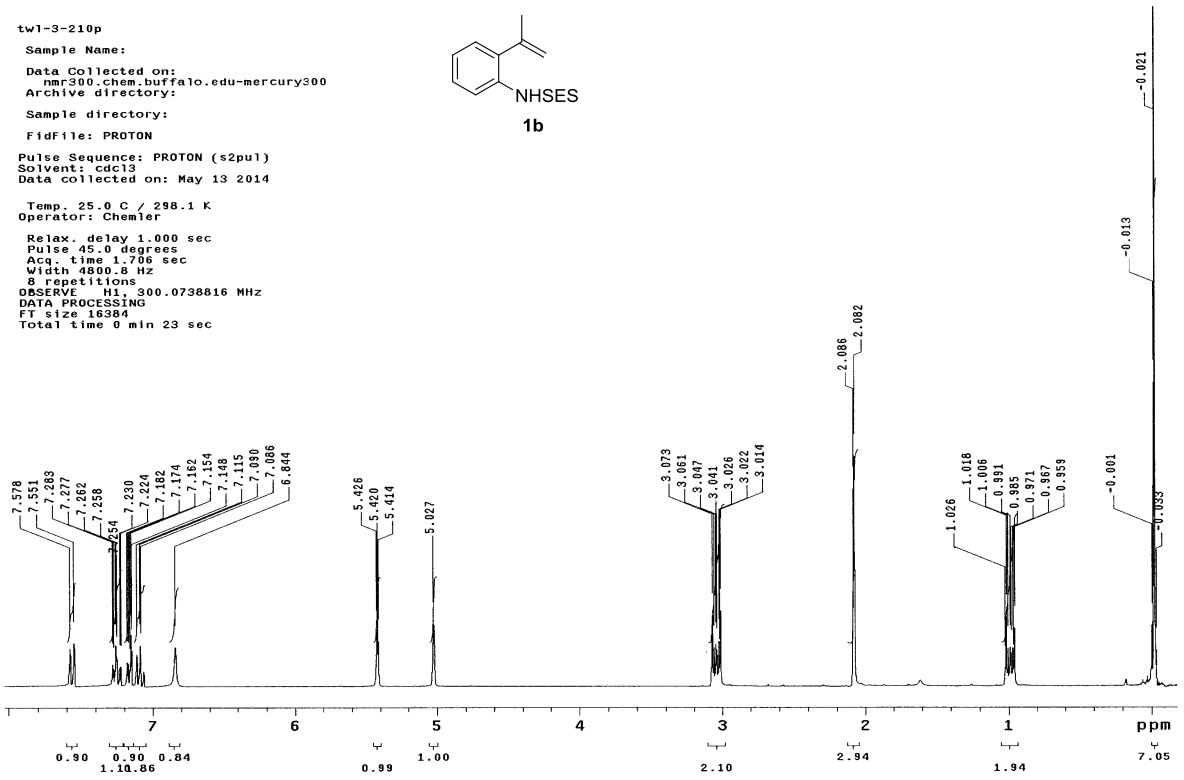
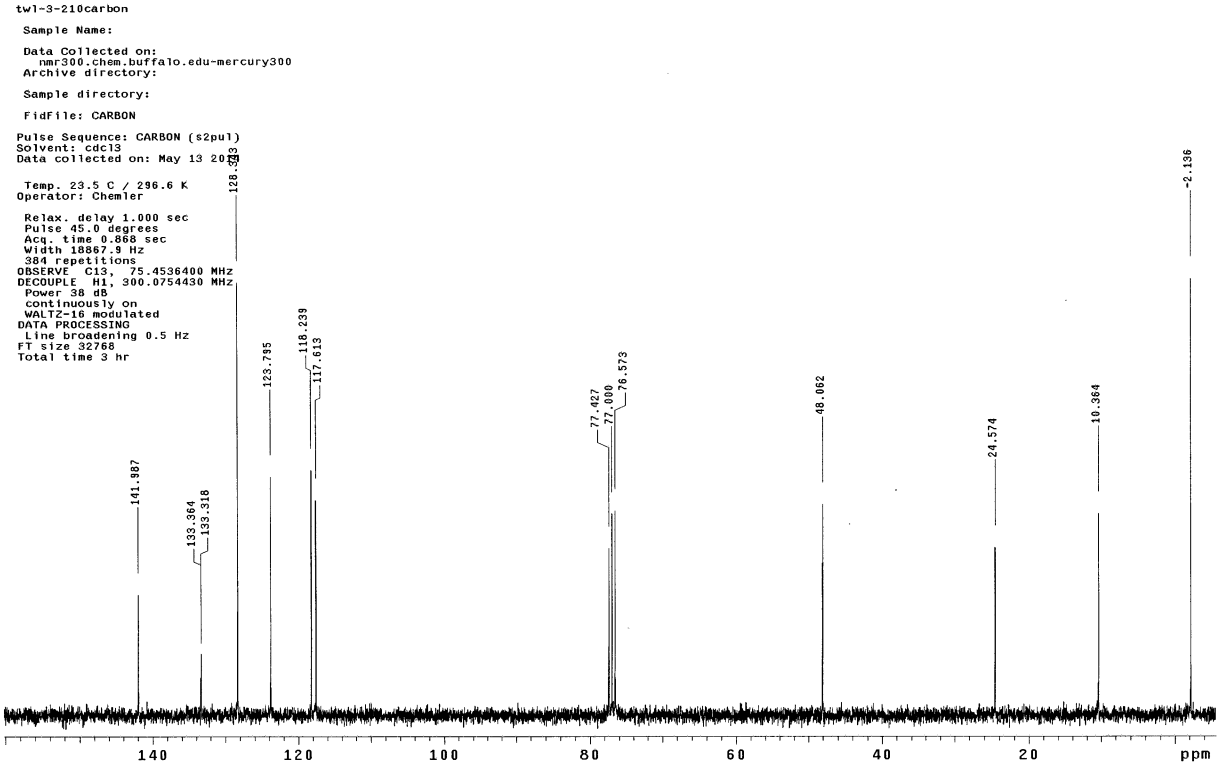
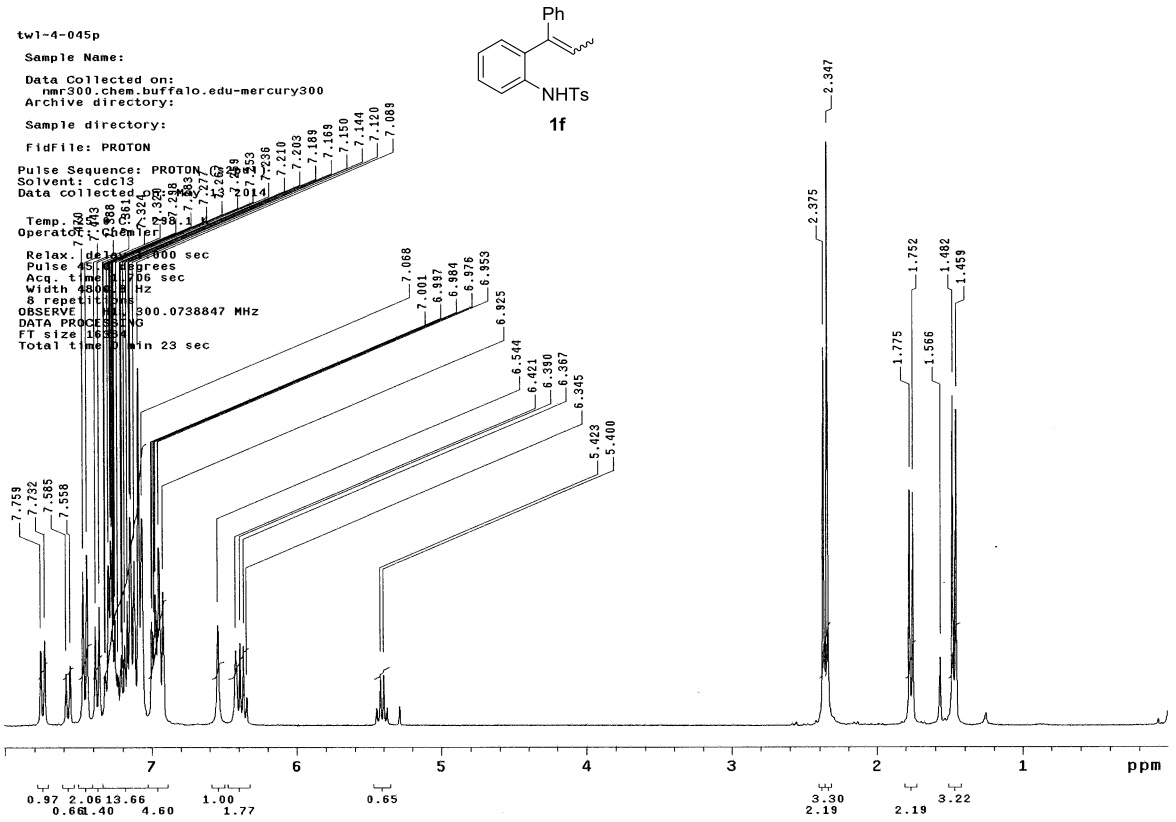
^{1}H and ^{13}C Spectra



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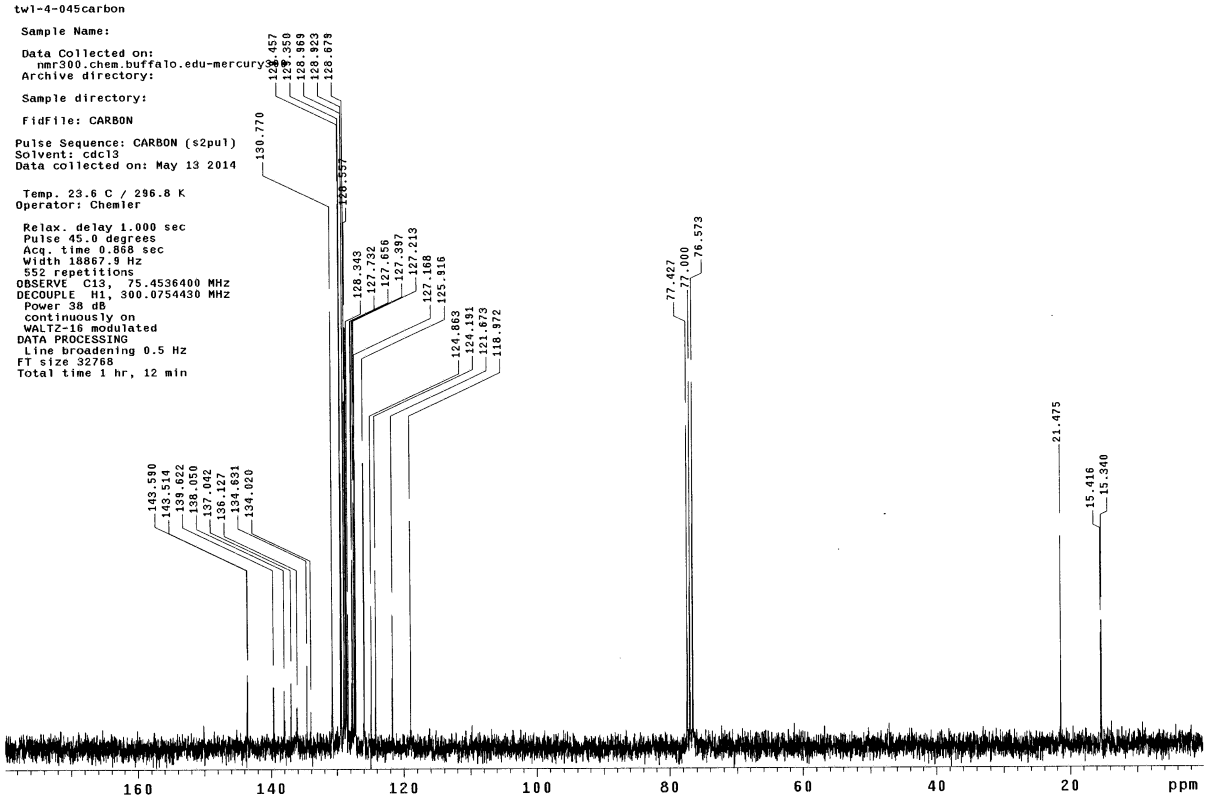
Archive directory:
Sample directory:
fidFile: PROTON
Pulse Sequence: PROTON (
Solvent: cdcl3
Data collected on:
Temp.
Operator: Chemier
Relax. delay
Pulse 45 degrees
Acq. time
Width
8 repetitions
OBSERVE
DATA PROCESSING
FT size
Total time 0 min 23 sec
7.759
7.732
7.585
7.558
7.068
7.001
6.997
6.984
6.976
6.953
6.925
6.544
6.421
6.390
6.367
6.345
5.423
5.400
2.347
2.375
1.775
1.752
1.566
1.482
1.459
ppm
0.97 2.06 13.66 1.00 0.65 3.30 2.19 3.22
0.66 1.40 4.60 1.77 2.19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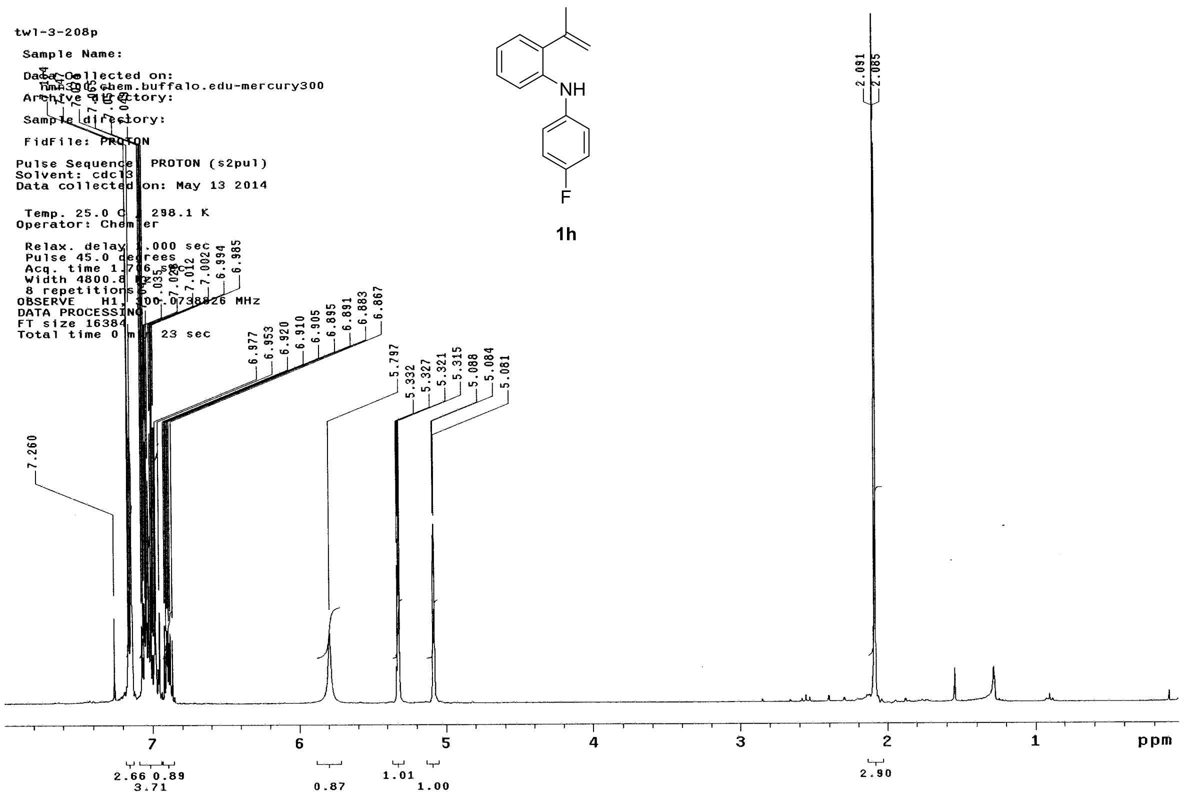
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 13 2014
Temp. 23.6 C / 296.8 K
Operator: Chemier
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.868 sec
Width 18867.9 Hz
552 repetitions
OBSERVE C13, 75.4536400 MHz
DECOUPLE H1, 300.0754430 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 1 hr, 12 min
130.770
128.457
128.350
128.969
128.923
128.679
126.557
128.343
127.732
127.656
127.397
127.213
127.168
125.916
124.863
124.191
121.673
118.972
143.590
143.514
139.622
139.056
137.402
136.127
134.631
134.020
77.427
77.000
76.573
21.475
15.416
15.340
ppm



1h
twl-3-208p
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Data Collected on:
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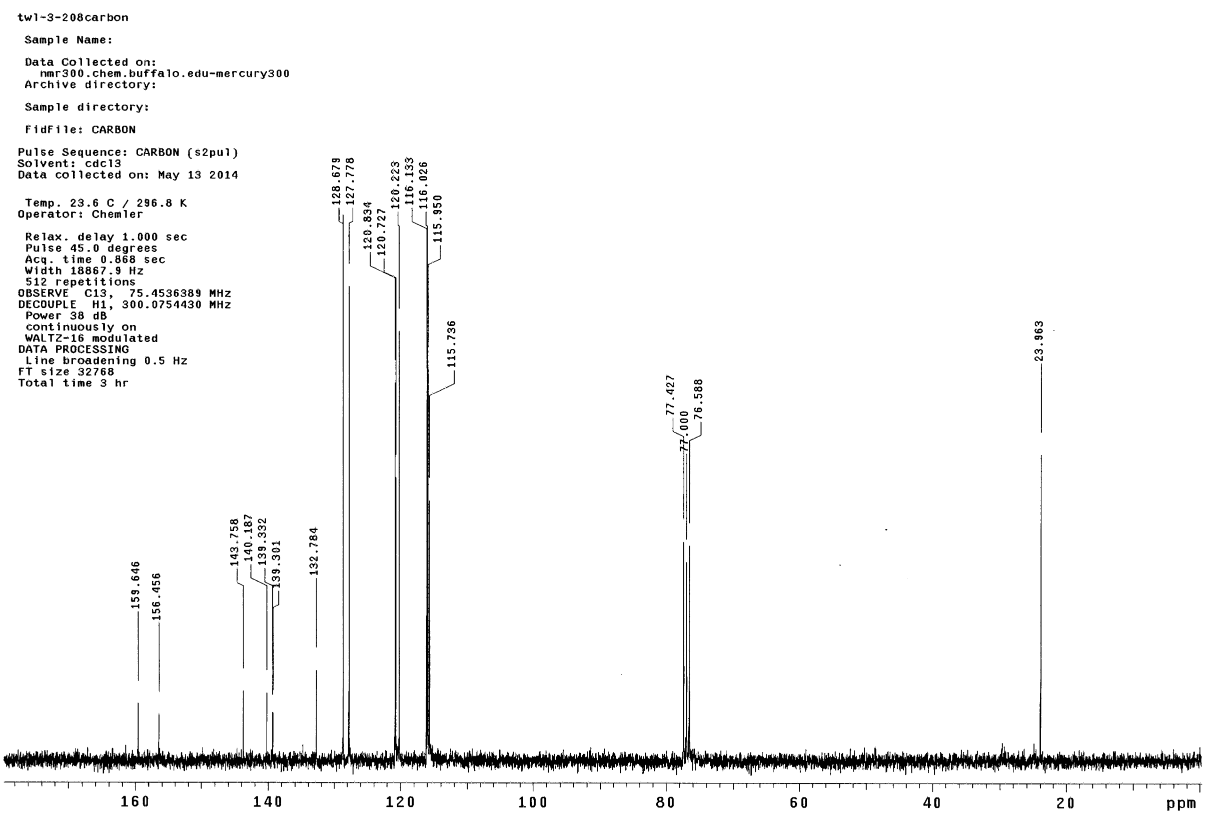
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 13 2014
Temp. 25.0 C / 298.1 K
Operator: Chemler
Total time 0 min, 23 sec
7.260
6.977
6.953
6.920
6.910
6.905
6.895
6.891
6.883
6.867
5.797
5.332
5.327
5.321
5.315
5.088
5.084
5.081
2.091
2.085
ppm
2.66
0.89
3.71
0.87
1.01
1.00
2.90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nmr300.chem.buffalo.edu-mercury300
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Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 13 2014
Temp. 23.6 C / 296.8 K
Operator: Chemler
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.868 sec
Width 18867.9 Hz
512 repetitions
OBSERVE C13, 75.4536389 MHz
DECOUPLE H1, 300.0754430 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 3 hr
159.646
156.456
143.758
140.187
139.332
139.301
132.784
128.679
127.778
120.834
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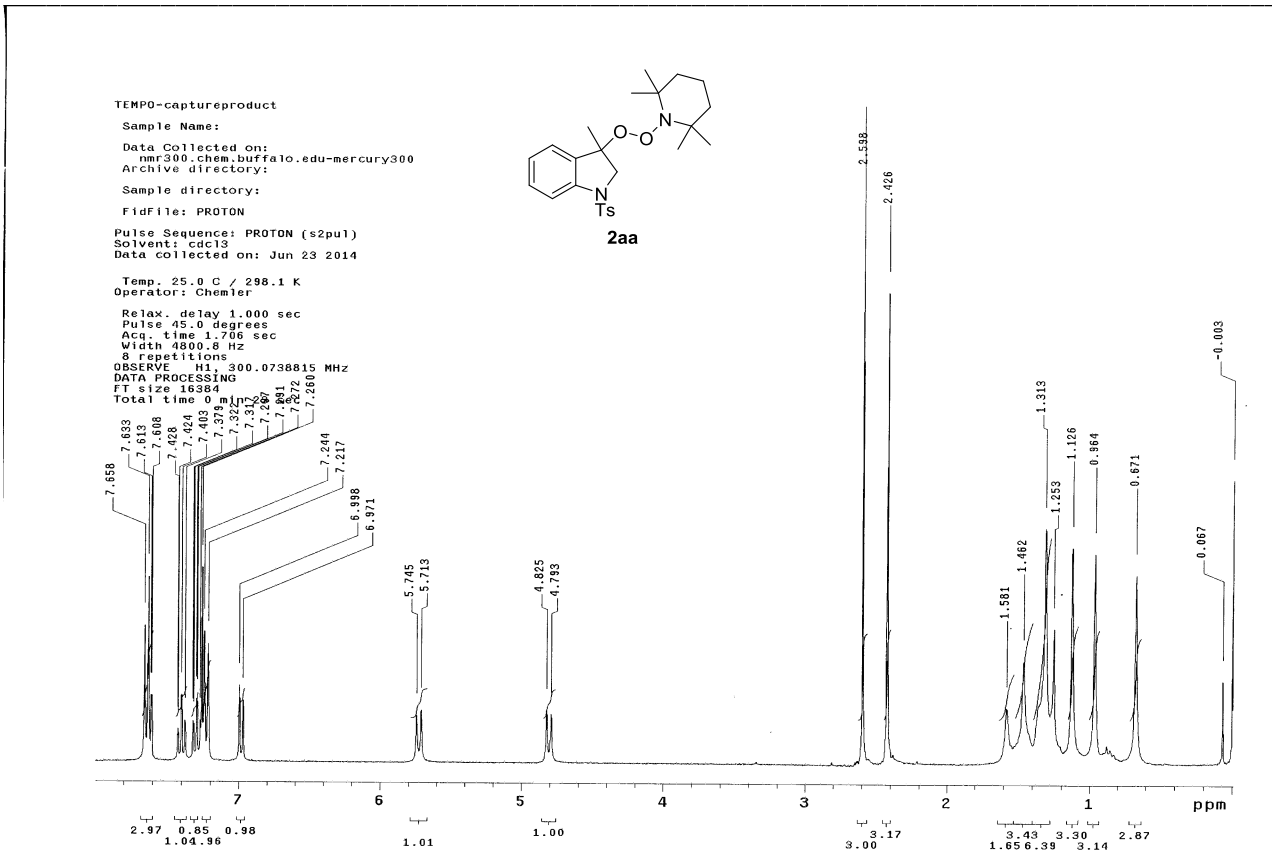
115.736
77.427
77.000
76.588
23.963
ppm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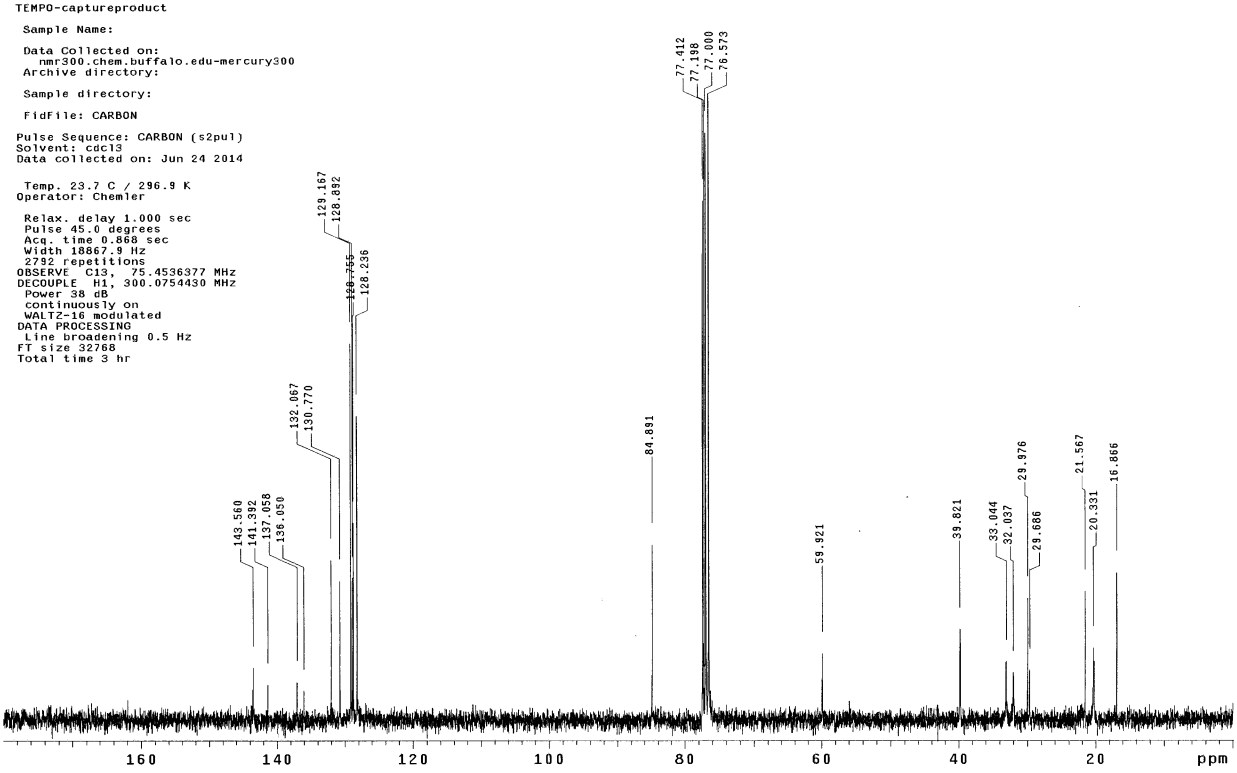
Data Collected on:
nmr300.chem.buffalo.edu-mercury300
Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jun 23 2014
Temp. 25.0 C / 298.1 K
Operator: Chemler
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4800.8 Hz
8 repetitions
OBSERVE H1, 300.0738815 MHz
DATA PROCESSING
FT size 16384
Total time 0 min, 27 sec
2aa
ppm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Archive directory:
Sample directory:
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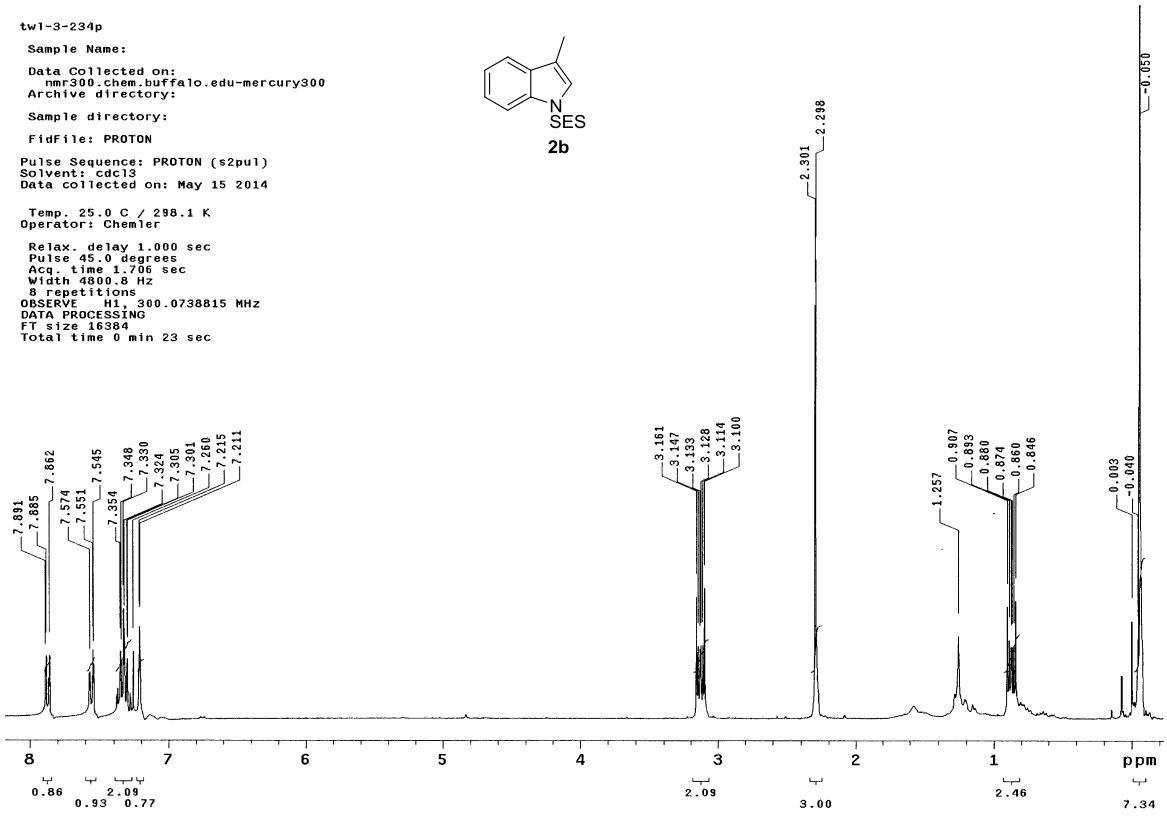
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jun 24 2014
Temp. 23.7 C / 296.9 K
Operator: Chemler
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.868 sec
Width 18867.9 Hz
2792 repetitions
OBSERVE C13, 75.4536377 MHz
DECOUPLE H1, 300.0754430 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 3 hr
ppm


twl-3-234p
Sample Name:
Data Collected on:
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Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2014
Temp. 25.0 C / 298.1 K
Operator: Chemler
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4800.8 Hz
8 repetitions
OBSERVE H1, 300.0738815 MHz
DATA PROCESSING
FT size 16384
Total time 0 min 23 sec
N
SES
2b
7.891
7.885
7.862
7.574
7.551
7.545
7.354
7.348
7.330
7.324
7.305
7.301
7.260
7.215
7.211
3.161
3.147
3.133
3.128
3.114
3.100
2.301
2.298
1.257
0.907
0.883
0.880
0.874
0.860
0.846
0.003
-0.040
-0.050
8
7
6
5
4
3
2
1
ppm
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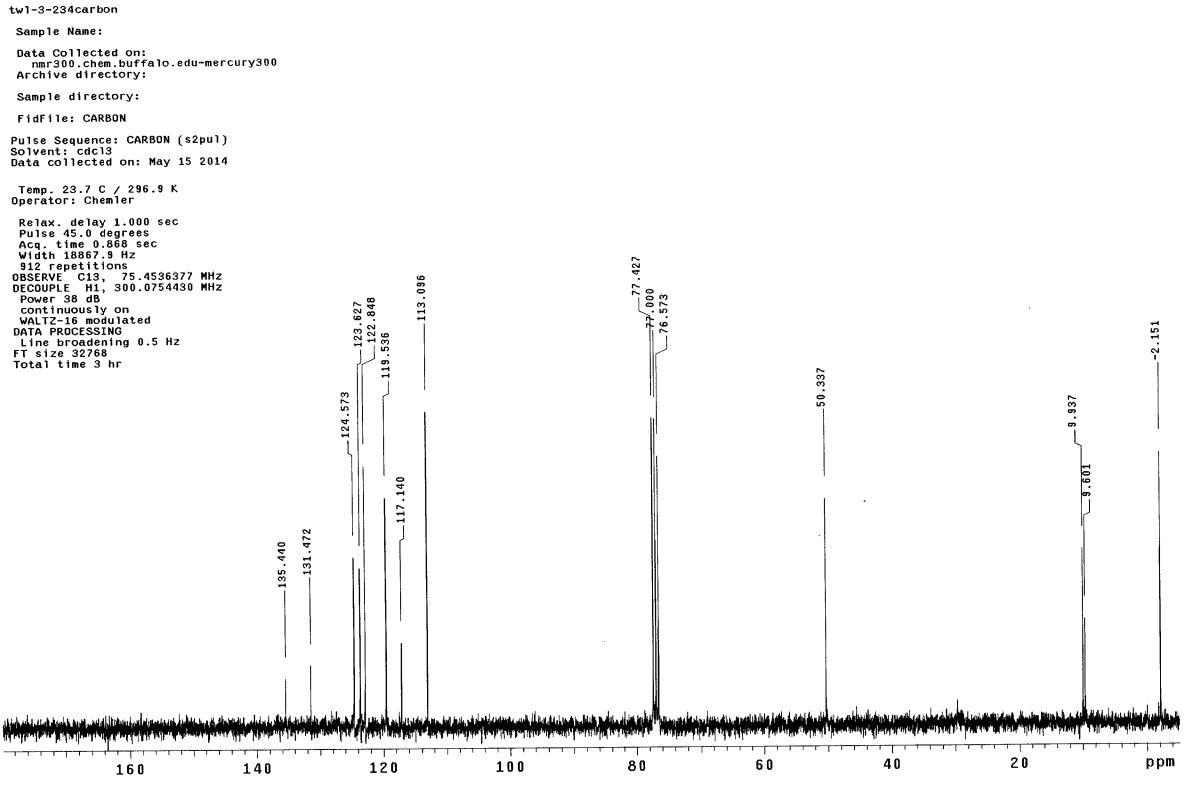
0.93
2.09
0.77
2.09
3.00
2.46
7.34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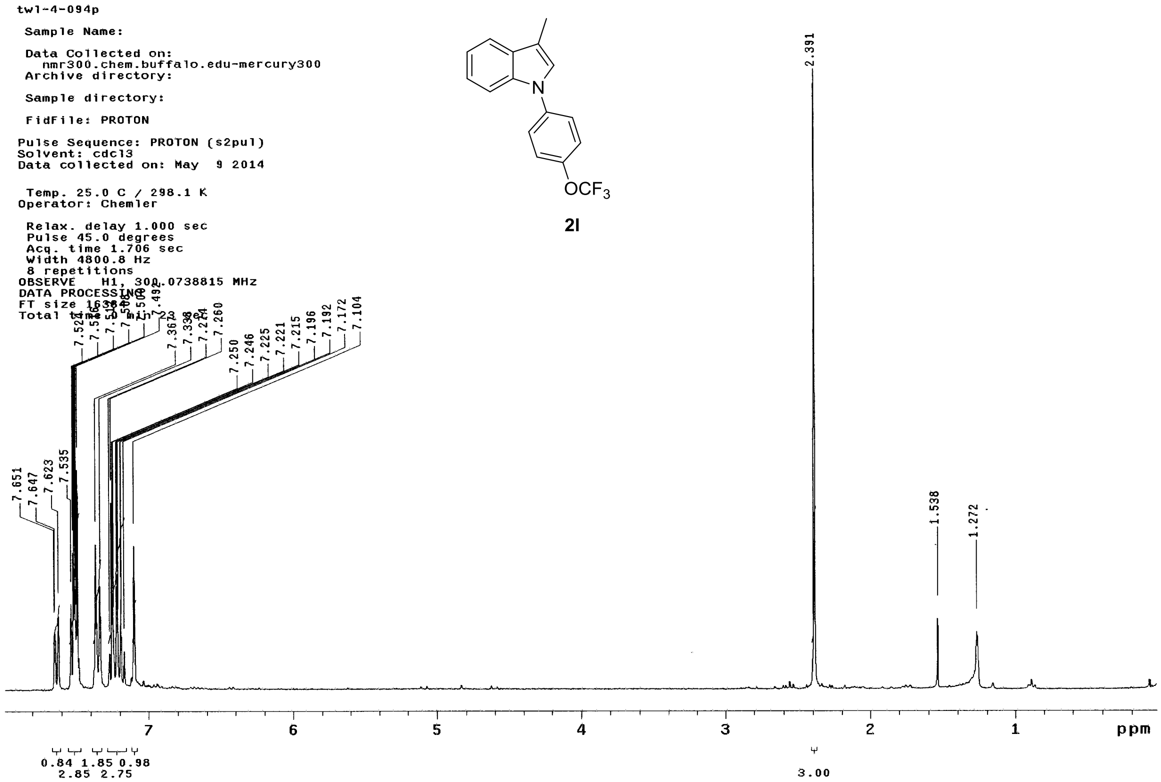
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2014
Temp. 23.7 C / 296.9 K
Operator: Chemler
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.868 sec
Width 18867.9 Hz
912 repetitions
OBSERVE C13, 75.4536377 MHz
DECOUPLE H1, 300.0754430 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 3 hr
135.440
131.472
124.573
123.627
122.848
119.536
117.140
113.096
77.427
77.000
76.573
50.937
9.937
9.601
-2.151
160
140
120
100
80
60
40
20
ppm



2l

twl-4-094p
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Data Collected on:
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Archive directory:
Sample directory:
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 9 2014
Temp. 25.0 C / 298.1 K
Operator: Chemler
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4800.8 Hz
8 repetitions
OBSERVE H1, 300.0738815 MHz
DATA PROCESSING
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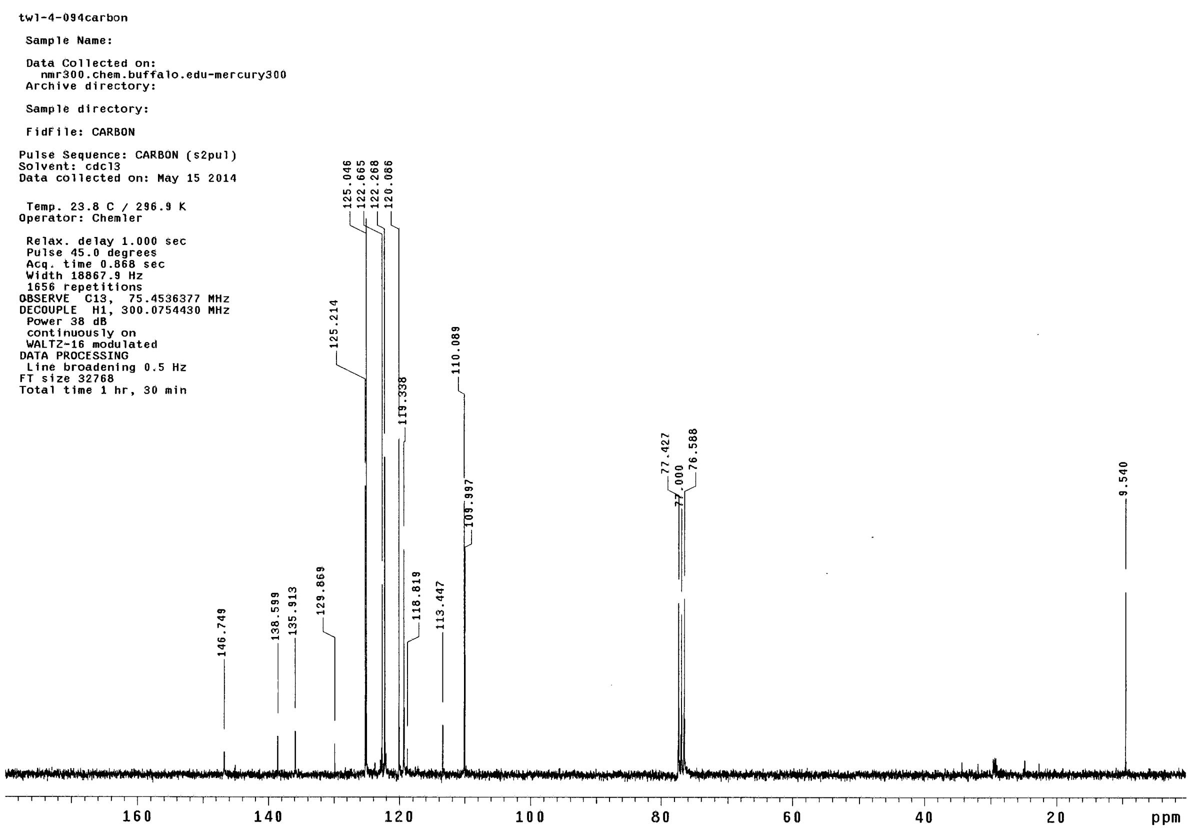
1.538
1.272
ppm
0.84 1.85 0.98
2.85 2.75
3.00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Data Collected on:
nmr300.chem.buffalo.edu-mercury300
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 15 2014
Temp. 23.8 C / 296.9 K
Operator: Chemler
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.868 sec
Width 18867.9 Hz
1656 repetitions
OBSERVE C13, 75.4536377 MHz
DECOUPLE H1, 300.0754430 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 1 hr, 30 min
146.749
138.599
135.913
129.869
125.214
125.046
122.665
122.268
120.086
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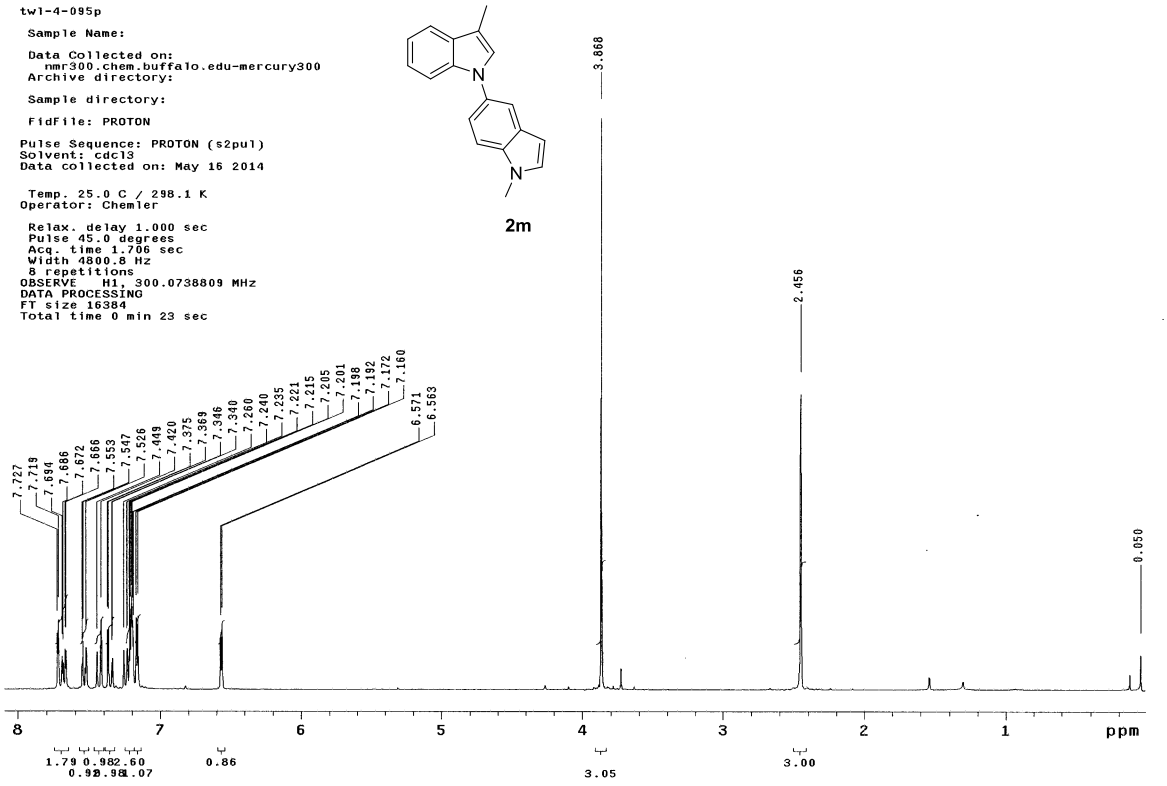
109.997
77.427
77.000
76.588
9.540
ppm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Archive directory:
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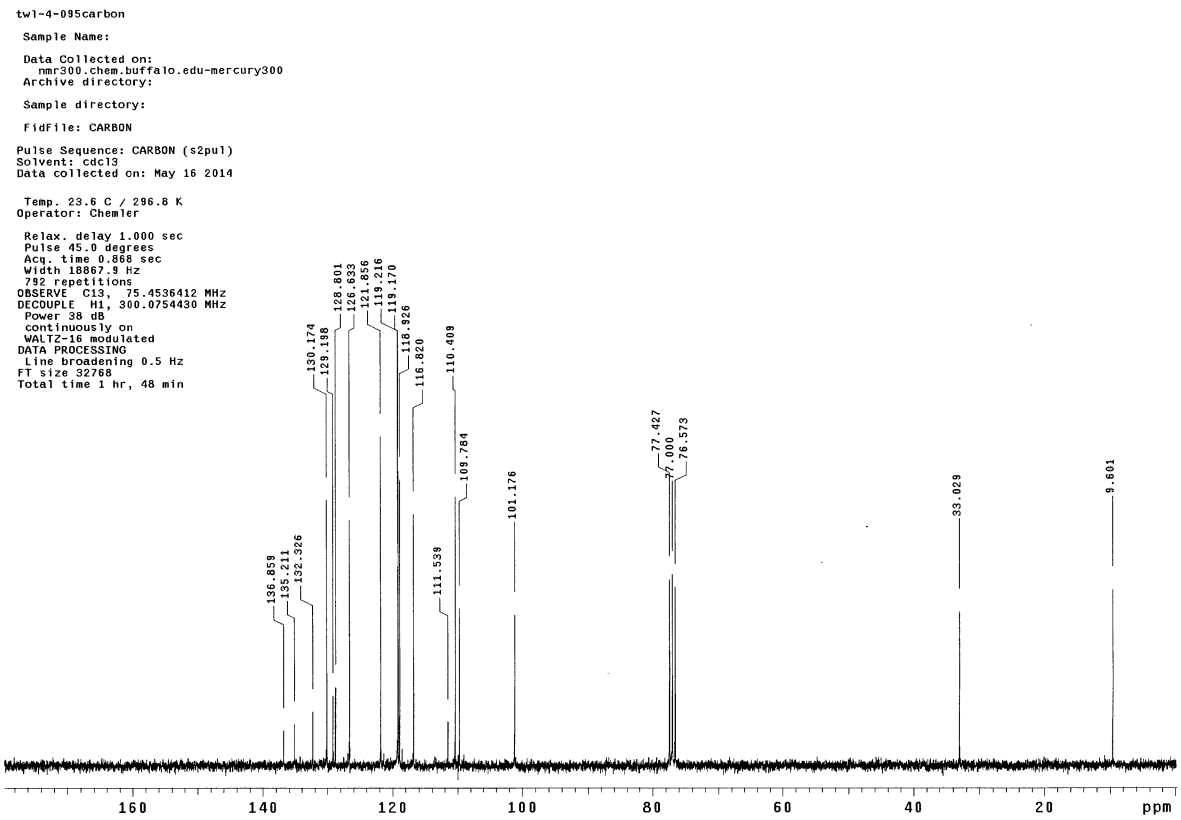
FidFile: PROTON
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 16 2014
Temp. 25.0 C / 298.1 K
Operator: Chemler
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4800.8 Hz
8 repetitions
OBSERVE H1, 300.0738809 MHz
DATA PROCESSING
FT size 16384
Total time 0 min 23 sec
2m
3.868
2.456
0.050
7.727
7.719
7.694
7.686
7.672
7.666
7.553
7.547
7.526
7.449
7.420
7.375
7.369
7.346
7.340
7.260
7.240
7.235
7.221
7.215
7.205
7.201
7.198
7.192
7.172
7.160
6.571
6.563
8
7
6
5
4
3
2
1
ppm
1.79
0.98
2.60
0.86
3.05
3.00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Sample directory:
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Solvent: cdcl3
Data collected on: May 16 2014
Temp. 23.6 C / 296.8 K
Operator: Chemler
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.868 sec
Width 18867.9 Hz
792 repetitions
OBSERVE C13, 75.4536412 MHz
DECOUPLE H1, 300.0754430 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 1 hr, 48 min
136.859
135.211
132.326
130.174
129.198
128.801
126.633
121.656
119.216
119.170
118.926
116.820
111.539
110.409
109.784
101.176
77.427
77.000
76.573
33.029
9.601
160
140
120
100
80
60
40
20
ppm