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Supporting Information

Catalyst-free thiolation of indoles with sulfonyl hydrazides for the synthesis of 3-sulfenylindoles in water

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General Remarks

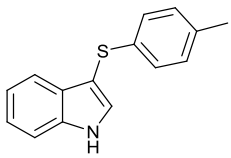
All substrates were purchased commercially without further purification. The yields were determined based on indoles.

^{1}H and ^{13}C NMR spectra were recorded on a Bruker AC-300 FT spectrometer at 400 MHz and 100 MHz, respectively, with tetramethylsilane as an internal reference. Chemical shifts (δ) and coupling constants (*J*) were expressed in ppm and Hz, respectively. High resolution mass spectra (HRMS) were recorded on a LC-TOF spectrometer (Micromass).

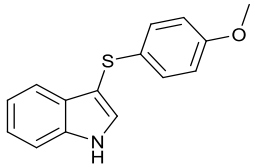
General procedures for the synthesis of Arylsulfonyl Hydrazides

Arylsulfonyl hydrazides **2b**-**2s** were prepared according to the literature procedure.[1] To a solution of an arylsulfonyl chloride (3.0 mmol) in tetrahyrdofuran (15 mL), was added hydrazine monohydrate (375 mg, 7.5 mmol) dropwise under nitrogen at 0 ℃. After vigorous stirring for 30 min at 0 ℃, the reaction mixture was added ethyl acetate (60 mL), and washed with saturated brine (3 x 10 mL). The organic layer was dried over sodium sulfate, filtered, concentrated and added to hexane (12 mL) over 5 min. The mixture was filtered, and the collected solid was dried in vacuum.

Characterization data of products

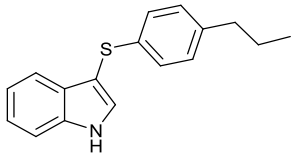


3-(p-tolylthio)-1H-indole (3aa).[1] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 87% yield; mp = 125–126 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.24 (s, 3H), 6.95 (d, *J* = 8.2 Hz, 2H), 7.01 (d, *J* = 8.2 Hz, 2H), 7.12 (t, *J* = 7.9 Hz, 1H), 7.24 (t, *J* = 7.9 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.44 (d, *J* = 2.5 Hz, 1H), 7.60 (d, *J* = 8.0 Hz, 1H), 8.37 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.5, 135.5, 134.6, 130.4, 129.5, 129.1, 126.3, 122.9, 120.8, 119.7, 111.5, 103.5, 20.8.

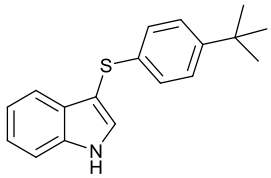


3-(4-methoxyphenylthio)-1H-indole (3ab).[1] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a

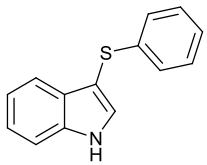
yellow solid: 93% yield, mp = 111–113 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.76 (s, 3H), 6.78 (d, *J* = 8.8 Hz, 2H), 7.18 (m, 3H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.43 (d, *J* = 8.1 Hz, 1H), 7.46 (d, *J* = 2.2 Hz, 1H), 7.68 (d, *J* = 7.8 Hz, 1H), 8.38 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 136.5, 130.1, 129.5, 129.0, 128.6, 123.0, 120.8, 119.6, 114.5, 111.6, 104.5, 55.4.



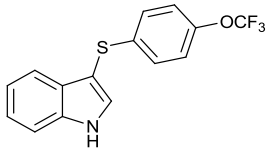
3-(4-propylphenylthio)-1H-indole (3ac). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white crystal: 89% yield; mp = 109–111 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.3 Hz, 3H), 1.56 (m, 2H), 2.47 (t, *J* = 7.3 Hz, 3H), 6.97 (d, *J* = 8.3 Hz, 2H), 7.03 (d, *J* = 8.3 Hz, 2H), 7.16 (t, *J* = 7.2 Hz, 1H), 7.24 (t, *J* = 7.2 Hz, 1H), 7.41 (d, *J* = 8.0 Hz, 1H), 7.45 (d, *J* = 2.5 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 8.39 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.5, 136.4, 135.8, 130.5, 129.2, 128.9, 126.1, 122.9, 120.8, 119.7, 111.5, 103.4, 37.4, 24.5, 13.8; HRMS $[M+H]^+$ calcd. for $C_{17}H_{18}NS$: 268.1155, found 268.1154.



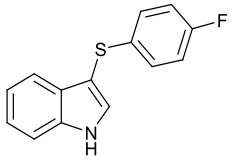
3-(4-tert-butylphenylthio)-1H-indole (3ad).[2] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a a white solid: 90% yield; mp =185-187 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.24 (s, 9H), 7.04 (d, *J* = 8.4 Hz, 2H), 7.17 (m, 3H), 7.24 (t, *J* = 7.2 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 1H), 7.44 (d, *J* = 2.5 Hz, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 8.37 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.0, 136.5, 135.7, 130.6, 129.3, 125.9, 125.8, 123.0, 120.8, 119.7, 111.5, 103.4, 34.3, 31.3.



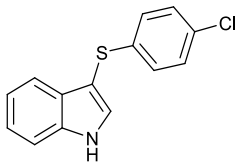
3-(phenylthio)-1H-indole (3ae).[1] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 91% yield; mp =151–152 ℃; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 7.11 (m, 7H), 7.55 (m, 2H), 7.68 (m, 1H), 10.84 (s, 1H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 139.8, 137.2, 131.9, 129.2, 128.7, 125.6, 124.6, 122.4, 120.3, 118.7, 112.2, 100.9.



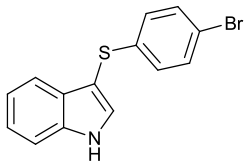
3-(4-(trifluoromethoxy)phenylthio)-1H-indole (3af).[2] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.92 (d, *J* = 8.3 Hz, 2H), 7.01 (d, *J* = 8.3 Hz, 2H), 7.11 (t, *J* = 7.4 Hz, 1H), 7.21 (t, *J* = 7.4 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.42 (s, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 8.48 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.6, 138.2, 136.5, 130.9, 128.8, 126.9, 123.3, 121.7 (q, *J* = 255.1 Hz), 121.5, 121.1, 119.5, 111.7, 102.3.



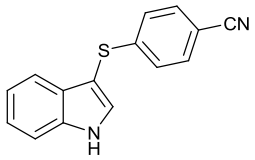
3-(4-fluorophenylthio)-1H-indole (3ag).[1] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 83% yield; mp = 132-133 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.86 (t, *J* = 8.4 Hz, 2H), 7.08 (m, 2H), 7.17 (t, *J* = 7.5 Hz, 1H), 7.27 (t, *J* = 7.5 Hz, 1H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.47 (d, *J* = 2.5 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 1H), 8.40 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ162.1 (d, J_{CF} = 242.5 Hz), 136.5, 134.0 (d, J_{CF} = 2.9 Hz), 130.5, 128.9, 127.9 (d, J_{CF} = 7.7 Hz), 123.1, 121.0, 119.5, 115.8 (d, J_{CF} = 21.8 Hz), 111.6, 102.5.



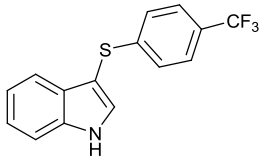
3-(4-chlorophenylthio)-1H-indole (3ah).[1] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white crystal: 66% yield; mp = 130-131 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.01 (d, *J* = 8.6 Hz, 2H), 7.11 (d, *J* = 8.6 Hz, 2H), 7.17 (t, *J* = 7.5 Hz, 1H), 7.27 (t, *J* = 7.5 Hz, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.47 (d, *J* = 2.5 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 8.43 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.8, 136.5, 130.7, 130.6, 128.8, 128.7, 127.1, 123.2, 121.1, 119.5, 111.7, 102.5.



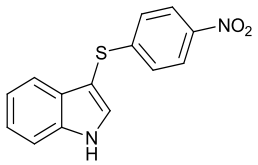
3-(4-bromophenylthio)-1H-indole (3ai).[1] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 65% yield; mp = 140-142 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.95 (d, *J* = 8.2 Hz, 2H), 7.17 (d, *J* = 8.2 Hz, 1H), 7.27 (m, 3H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.49 (s, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 8.44 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.6, 136.5, 131.7, 130.8, 128.8, 127.4, 123.3, 121.1, 119.5, 118.3, 111.7, 102.3.



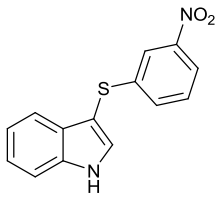
4-(1H-indol-3-ylthio)benzonitrile (3aj). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light green solid: 74% yield; mp = 184-186 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (d, *J* = 8.4 Hz, 2H), 7.12 (t, *J* = 7.2 Hz, 1H), 7.23 (m, 1H), 7.32 (d, *J* = 8.4 Hz, 1H), 7.44 (m, 3H), 8.58 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 136.6, 132.2, 131.2, 128.5, 125.4, 123.5, 121.3, 119.3, 119.2, 111.9, 107.6, 100.2. HRMS $[M]^+$ calcd. for $C_{15}H_{10}N_2S$: 250.0565, found 250.0564.



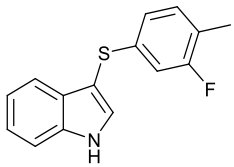
3-(4-(trifluoromethyl)phenylthio)-1H-indole (3ak).[2] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 52% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, *J* = 8.2 Hz, 2H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.35 (t, *J* = 7.6 Hz, 1H), 7.43 (d, *J* = 8.2 Hz, 2H), 7.53 (m, 2H), 7.63 (d, *J* = 8.0 Hz, 1H), 8.48 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.7, 136.6, 131.1, 128.8-120.3 (q, *J* = 271.2 Hz), 127.2-126.3 (q, *J* = 32.2 Hz), 125.5 (q, *J* = 3.6 Hz), 125.3, 123.4, 123.0, 121.3, 120.3, 119.4, 111.78, 101.2.



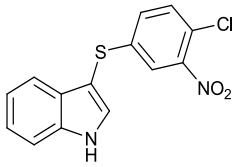
3-(4-nitrophenylthio)-1H-indole (3al). [1] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a yellow solid: 82% yield; mp = 174–175 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (m, 2H), 7.17 (m, 1H), 7.30 (m, 1H), 7.50 (m, 3H), 7.98 (m, 2H), 8.75 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.0, 144.8, 136.6, 131.4, 128.4, 125.1, 123.9, 123.5, 121.4, 119.2, 112.1, 100.0.



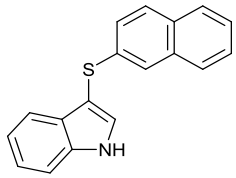
3-(3-nitrophenylthio)-1H-indole (3am). [1] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 78% yield; mp = 135–136 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (t, *J* = 7.9 Hz, 2H), 7.29 (m, 1H), 7.35 (m, 1H), 7.48 (d, *J* = 8.4 Hz, 1H), 7.55 (m, 2H), 7.88 (m, 1H), 7.91 (m, 1H), 8.62 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.6, 142.6, 136.6, 131.3, 129.3, 128.5, 123.5, 121.3, 120.4, 119.7, 119.2, 111.9, 100.8.



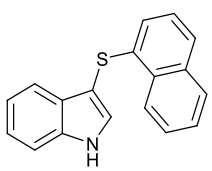
3-(3-fluoro-4-methylphenylthio)-1H-indole (3ao). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 82% yield; mp = 138-140 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.15 (s, 3H), 6.72 (m, 1H), 6.82 (m, 1H), 6.96 (t, *J* = 8.0 Hz, 1H), 7.17 (t, *J* = 7.8 Hz, 1H), 7.27 (d, *J* = 7.2 Hz, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.47 (d, *J* = 2.5 Hz, 1H), 7.6 (d, *J* = 7.8 Hz, 1H), 8.45 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6 (d, J_{CF} = 244.8 Hz), 138.3(d, J_{CF} = 7.6 Hz), 136.5, 131.4 (d, J_{CF} = 5.5 Hz), 130.7, 128.9, 123.1, 121.3 (d, J_{CF} = 3.5 Hz), 121.2, 121.0, 119.5, 112.7 (d, J_{CF} = 24.9 Hz), 111.6, 102.5,14.0 (d, J_{CF} = 3.5 Hz); HRMS $[M+H]^+$ calcd. for $C_{15}H_{13}FNS$: 258.0753, found 258.0749.



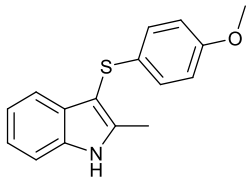
3-(4-chloro-3-nitrophenylthio)-1H-indole (3ap). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a yellow solid: 50% yield; mp = 196-198 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.65 (d, *J* = 8.7 Hz, 1H), 7.23 (m, 4H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.51 (d, *J* = 2.5 Hz, 1H), 7.61 (d, *J* = 7.9 Hz, 1H), 8.01 (d, *J* = 2.1 Hz, 1H), 8.42 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.8, 136.5, 135.6, 132.3, 130.3, 128.7, 126.5, 124.6, 123.2, 121.0, 119.4, 119.3, 111.7, 103.6; HRMS $[M+H]^+$ calcd. for $C_{14}H_{10}ClN_2O_2S$: 305.0151, found 305.0141.



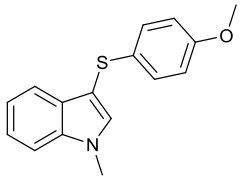
3-(naphthalen-2-ylthio)-1H-indole (3aq). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white crystal: 99% yield; mp = 142-143 ℃; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 7.09 (t, *J* = 7.5 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.28 (d, *J* = 8.6 Hz, 1H), 7.39 (m, 2H), 7.57 (m, 4H), 7.77 (m, 3H), 10.88 (s, 1H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 137.4, 137.2, 133.9, 132.1, 131.4, 129.2, 128.2, 127.7, 126.7, 126.5, 125.1, 124.5, 123.1, 122.5, 120.3, 118.8, 112.2, 100.8.



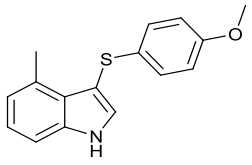
3-(naphthalen-1-ylthio)-1H-indole (3ar). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 67% yield; mp = 166-168 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.95 (dd, *J* = 7.4 Hz, 1.0Hz, 1H), 7.13 (m, 1H), 7.15 (d, *J* = 7.6, 1H),7.27 (m, 1H), 7.43 (d, *J* = 8.0, 1H), 7.55 (m, 5H), 7.82 (m, 1H), 8.41 (s, 1H), 8.48 (d, *J* = 8.0, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.6, 136.2, 133.7, 130.9, 130.8, 129.1, 128.5, 126.1, 126.0, 125.7, 125.2, 124.0, 123.4, 123.1, 120.9, 119.7, 116.7, 102.3; HRMS $[M+H]^+$ calcd. for $C_{18}H_{14}NS$: 276.0841, found 276.0849.



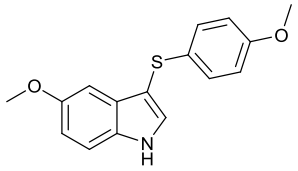
3-(4-methoxyphenylthio)-2-methyl-1H-indole (3bb).[3] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a colorless solid: 68% yield; mp = 115-116 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.52 (s, 3H), 3.72 (s, 3H), 6.70 (m, 2H), 7.03 (m, 2H), 7.09 (m, 2H), 7.30 (m, 1H), 7.57 (d, *J* = 7.8 Hz, 1H), 8.19 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.5, 140.6, 135.4, 132.7, 129.9, 127.9, 122.1, 120.6, 119.0, 114.5, 110.6, 101.0, 55.3, 12.2.



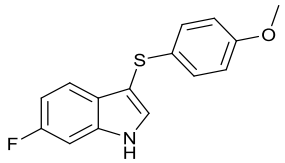
3-(4-methoxyphenylthio)-1-methyl-1H-indole (3cb).[4] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 99% yield; mp = 60-62 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.72 (s, 3H), 3.81 (s, 3H), 6.71 (d, *J* = 8.8 Hz, 2H), 7.13 (m, 3H), 7.29 (m, 2H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.82 - 7.85 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.7, 137.5, 134.5, 130.0, 129.7, 128.4, 122.5, 120.4, 119.7, 114.5, 109.7, 102.3, 55.3, 33.1.



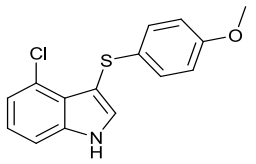
3-(4-methoxyphenylthio)-4-methyl-1H-indole (3db). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 56% yield; mp = 117-119 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.67 (s, 3H), 3.74 (s, 3H), 6.75 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 7.1 Hz, 1H), 7.03 (d, *J* = 8.8 Hz, 2H), 7.13 (t, *J* = 7.8 Hz, 1H), 7.25 (d, *J* = 7.1 Hz, 1H), 7.42 (d, *J* = 2.5 Hz, 1H), 8.37 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.4, 137.0, 132.1, 132.0, 131.5, 127.6, 126.9, 123.1, 122.4, 114.6, 109.4, 103.8, 55.4, 18.8; HRMS $[M+H]^+$ calcd. for $C_{16}H_{16}NOS$: 270.0953, found 270.0944.



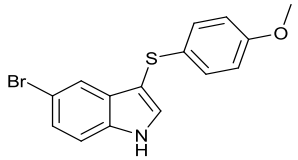
5-methoxy-3-(4-methoxyphenylthio)-1H-indole (3eb). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 72% yield; mp = 57-59 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.73 (s, 3H), 3.80 (s, 3H), 6.88 (dd, *J* = 8.8 Hz,2.3 Hz, 2H), 7.06 (d, *J* = 2.3 Hz, 1H), 7.10 (d, *J* = 8.8 Hz, 2H), 7.31 (d, *J* = 8.8 Hz, 1H), 7.43 (d, *J* = 2.5 Hz, 1H), 8.31 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.4, 154.0, 130.3, 129.7, 128.8, 128.6, 127.2, 113.4, 112.4, 111.3, 102.8, 99.8, 54.8, 54.3; HRMS $[M+H]^+$ calcd. for $C_{16}H_{16}NO_2S$: 286.0902, found 286.0896.



6-fluoro-3-(4-methoxyphenylthio)-1H-indole (3fb). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 85% yield; mp = 86-88 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.73 (s, 3H), 6.75 (d, *J* = 8.6 Hz, 2H), 6.89 (m, 1H), 7.10 (m, 3H), 7.43 (s, 1H), 7.51 (m, 1H), 8.39 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ161.6 (d, J_{CF} = 237.7 Hz), 157.9, 136.4 (d, J_{CF} = 12.7 Hz), 130.2 (d, J_{CF} = 3.1Hz), 139.0, 128.7, 125.4, 120.6 (d, J_{CF} = 10.1 Hz), 114.5, 109.7 (d, J_{CF} = 24.6 Hz), 105.1, 98.0 (d, J_{CF} = 26.2 Hz), 55.3; HRMS $[M+H]^+$ calcd. for $C_{15}H_{13}FNOS$: 274.0702, found 274.0701.

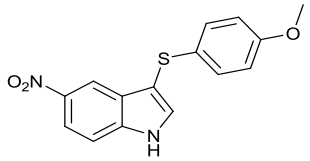


4-chloro-3-(4-methoxyphenylthio)-1H-indole (3gb). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 74% yield; mp = 77-79 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.74 (s, 3H), 6.75 (m, 2H), 7.13 (m, 4H), 7.30 (m, 1H), 7.41 (s, 1H), 8.47 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 138.0, 131.8, 130.8, 128.8, 126.9, 125.0, 123.5, 122.1, 114.5, 110.4, 105.0, 55.3; HRMS $[M+H]^+$ calcd. for $C_{15}H_{13}ClNOS$: 290.0406, found 290.0408.

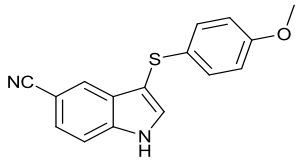


5-bromo-3-(4-methoxyphenylthio)-1H-indole (3hb). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 81% yield; mp = 106-108 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.74 (s, 3H), 6.75 (m, 2H), 7.11 (m, 2H), 7.28 (d, *J* = 8.6 Hz, 1H), 7.31 (dd, *J* = 8.6 Hz, 1.8 Hz, 1H), 7.45 (d, *J*

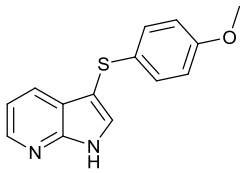
= 2.5 Hz, 1H), 7.76 (d, *J* = 1.8 Hz, 1H), 8.40 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 135.1, 131.2, 130.9, 128.9, 128.7, 126.0, 122.2, 114.6, 114.3, 113.0, 104.6, 55.4; HRMS $[M+H]^+$ calcd. for $C_{15}H_{13}BrNOS$:333.9901, found 333.9904.



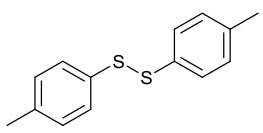
3-(4-methoxyphenylthio)-5-nitro-1H-indole (3ib). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a yellow solid: 35% yield; mp = 181-183 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.75 (s, 3H), 6.76 (m, 2H), 7.18 (m, 2H), 7.47 (d, *J* = 8.9 Hz, 1H), 7.61 (d, *J* = 8.6 Hz, 1H), 8.14 (dd, *J* = 8.9 Hz, 2.4 Hz, 1H), 8.58 (d, *J* = 2.0 Hz, 1H), 8.78 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.4, 141.7, 138.4, 131.5, 128.8, 127.6, 126.8, 117.6, 115.9, 113.7, 110.7, 107.9, 55.3; HRMS $[M+H]^+$ calcd. for $C_{15}H_{13}N_2O_3S$: 301.0642, found 301.0637.



3-(4-methoxyphenylthio)-1H-indole-5-carbonitrile (3jb). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 56% yield; mp = 236-238 ℃; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 3.73 (s, 3H), 6.83 (m, 2H), 7.20 (m, 2H), 7.51 (dd, *J* = 8.5 Hz, 1.6 Hz, 1H), 7.71 (m, 1H), 7.88 (d, *J* = 2.5 Hz, 1H), 7.92 (m, 1H), 11.21 (s, 1H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 158.5, 138.8, 133.6, 129.5, 129.0, 128.4, 125.1, 124.1, 119.8, 114.6, 113.5, 105.1, 103.4, 54.7; HRMS $[M]^+$ calcd. for $C_{16}H_{12}N_2OS$: 280.0670, found 280.0671.

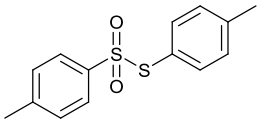


3-(4-methoxyphenylthio)-1H-pyrrolo[2,3-b]pyridine (3kb). The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 65% yield; mp = 173-175 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.12 (s, 3H), 6.78 (d, *J* = 8.7 Hz, 2H), 7.15 (m, 3H), 7.66 (s, 1H), 7.97 (d, *J* = 7.8 Hz, 1H), 8.38 (s, 1H), 10.94 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.1, 148.4, 142.9, 130.9, 129.1, 128.8, 128.6, 122.2, 116.7, 114.6, 103.8, 55.3; HRMS $[M]^+$ calcd. for $C_{14}H_{12}N_2OS$: 256.0665, found 256.0656.



1,2-dip-tolyldisulfane (4). [1] The title compound was prepared according to the general working

procedure and purified by column chromatography to give the product as a white solid: 25% yield; mp = 45–46 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.31 (s, 6H), 7.10 (d, *J* = 8.0 Hz, 4H), 7.38 (d, *J* = 8.0 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.5, 133.9, 129.8, 128.5, 21.1.

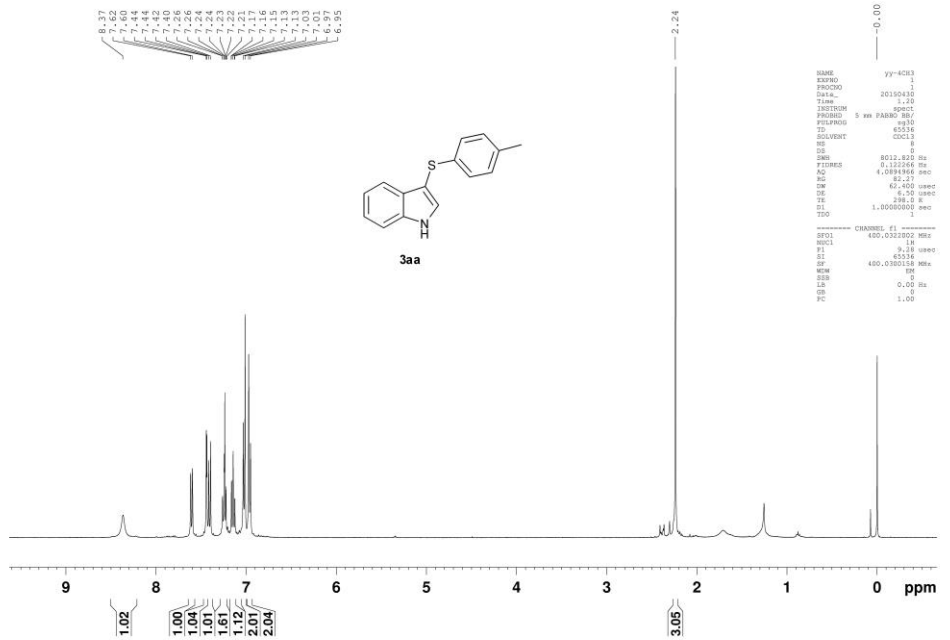


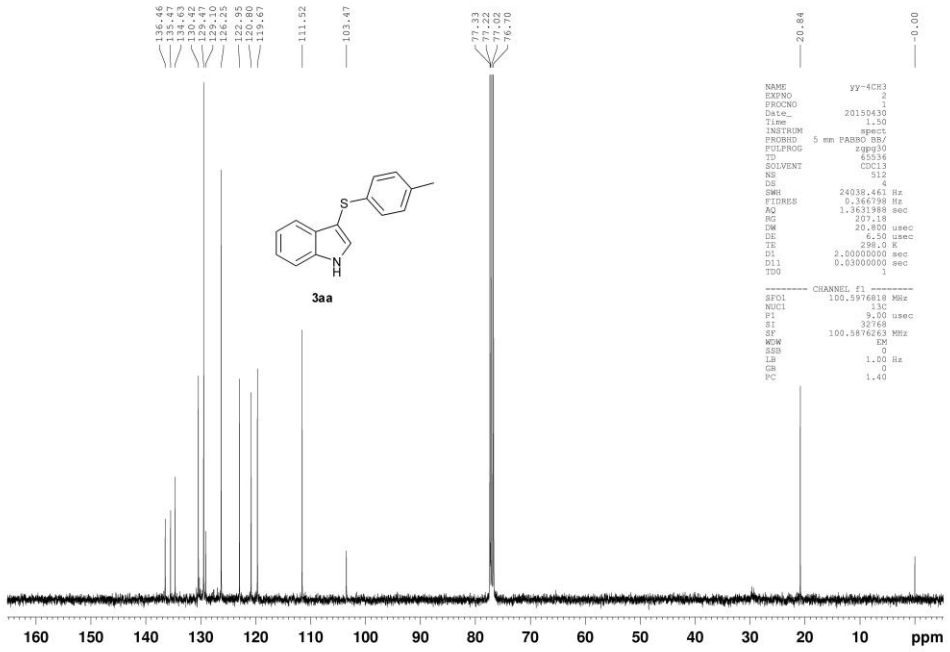
4-methylbenzenesulfonothioic acid S-(4-methylphenyl) ester (5). [1] The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 57% yield; mp = 91-93 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.36 (s, 3H), 2.41 (s, 3H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.21 (t, *J* = 8.0 Hz, 4H), 7.44 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.7, 142.1, 140.4, 136.5, 130.3, 129.4, 127.6, 124.5, 21.7, 21.5.

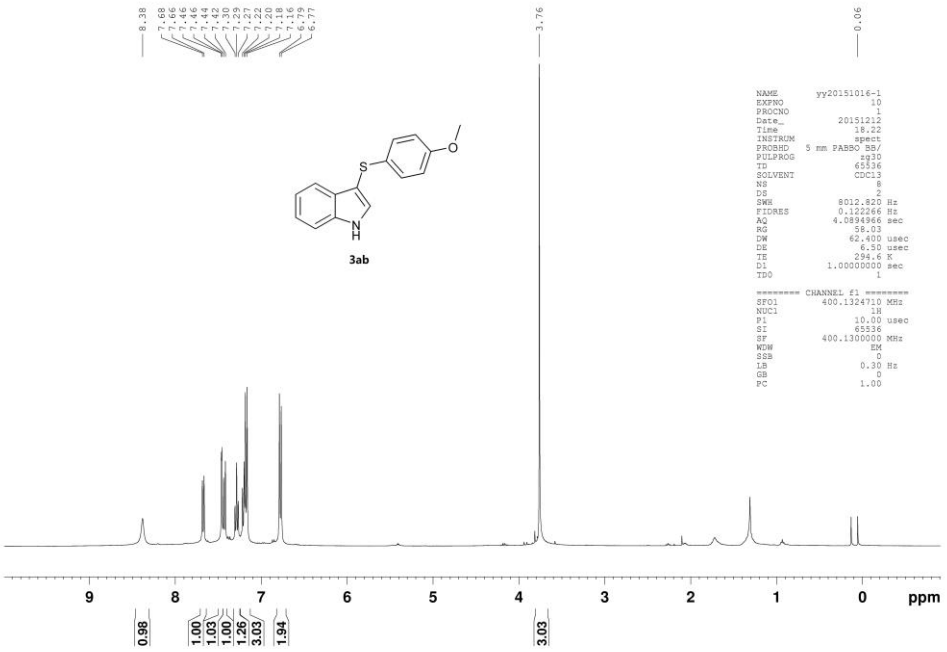
Reference

[1] F.-L. Yang, X.-T. Ma and S.-K. Tian, *Chem. Eur. J.*, 2012, **18**, 1582.
[2] F. Xiao, H. Xie, S. Liu, G.-J. Deng, *Adv. Synth. Catal.*, 2014, **356**, 364–368.
[3]G. L. Regina, V. Gatti, V. Famiglini, F. Piscitelli, and R. Silvestri, *ACS Comb. Sci.*, 2012, **14**, 258−262.
[4] M. Tudge, M. Tamiya, C. Savarin and G. R. Humphrey, *Org. Lett.*,2006, **8**, 565-568.

NMR Spectra of products



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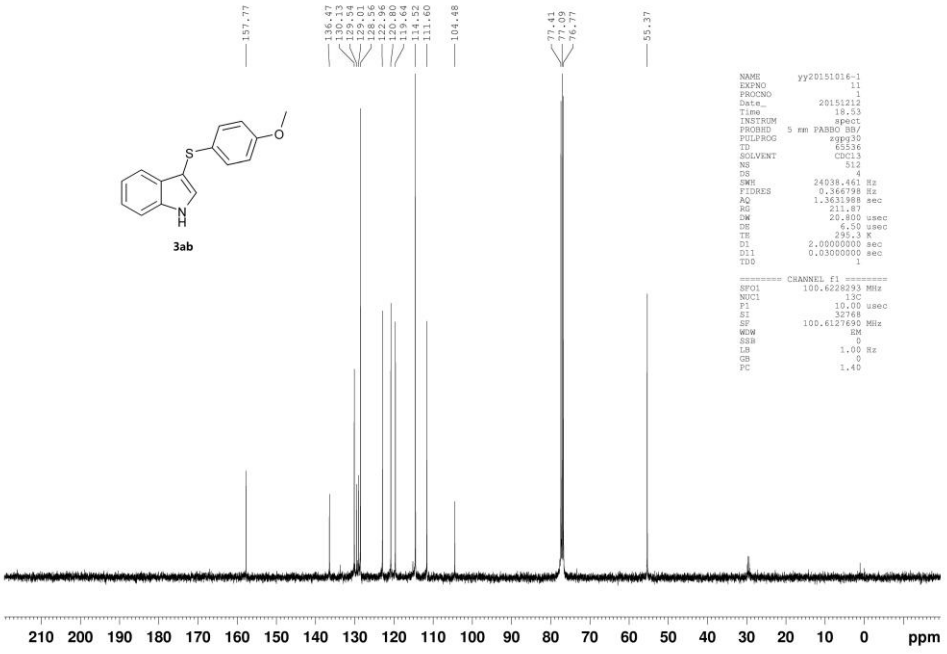
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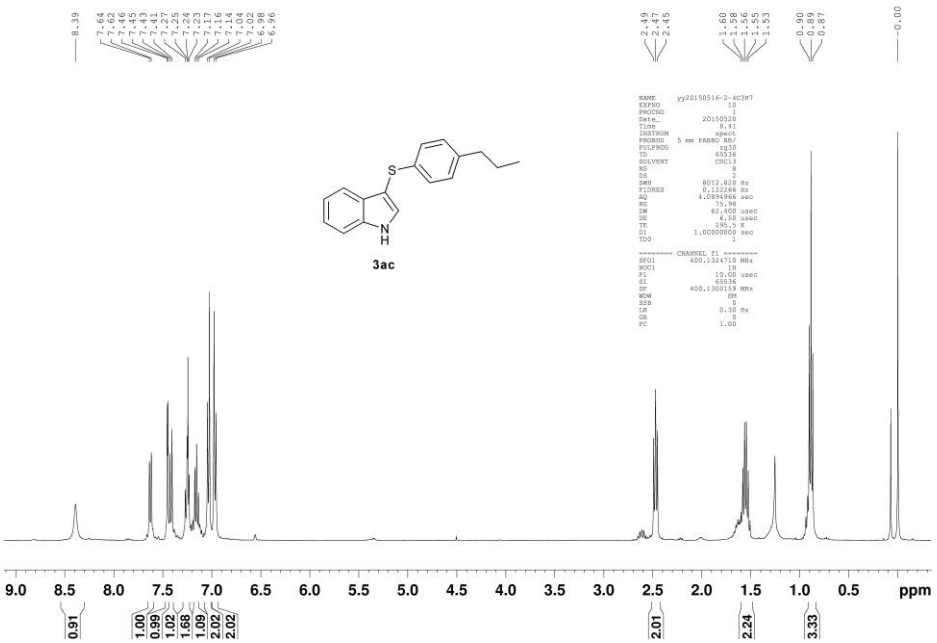
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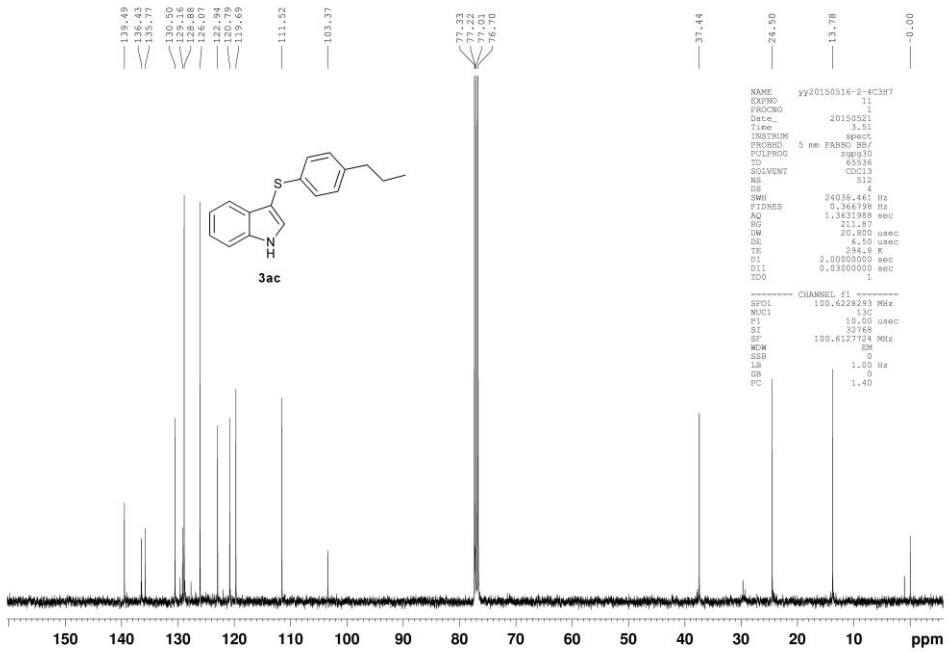
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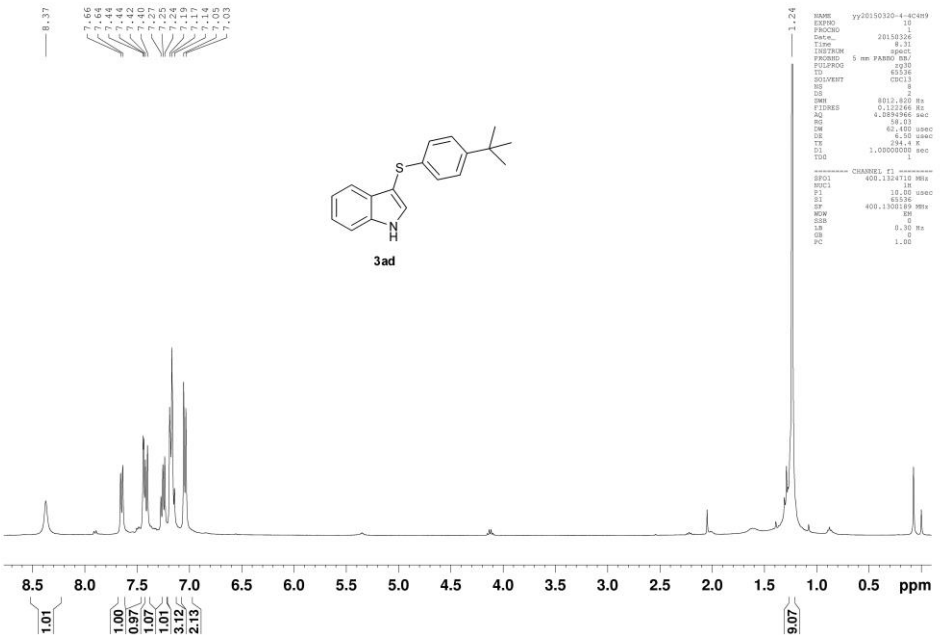
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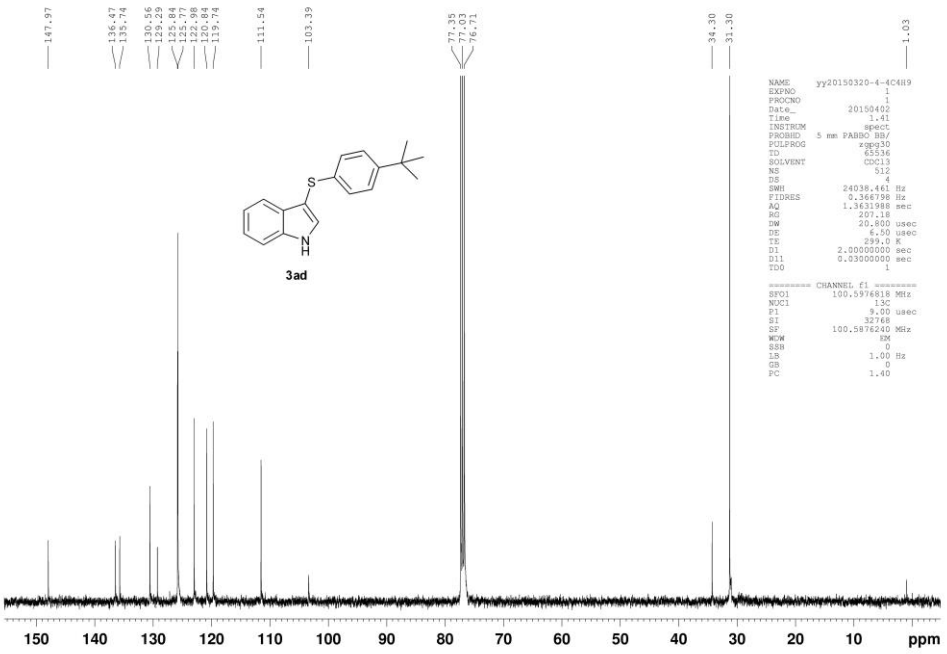
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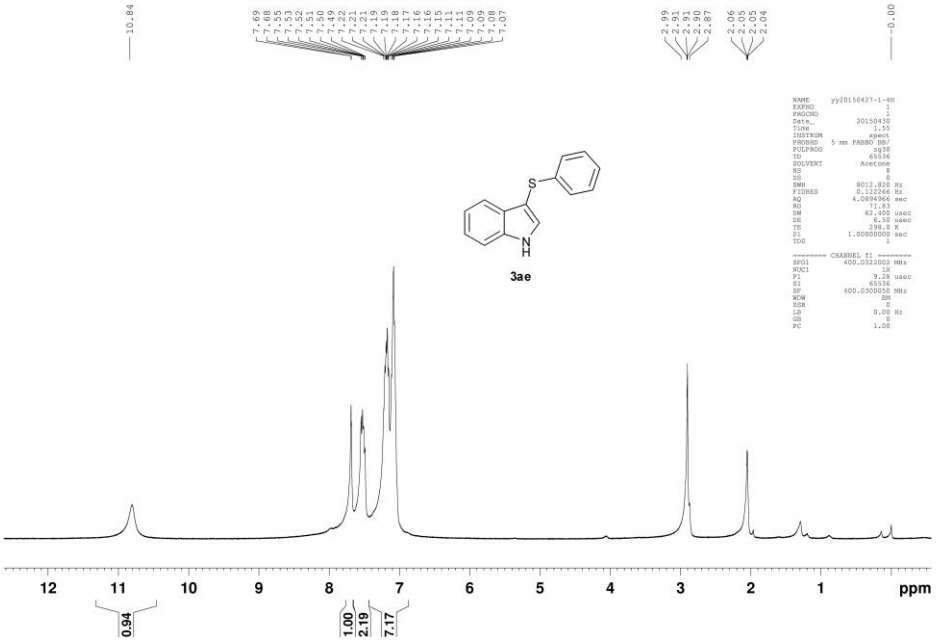
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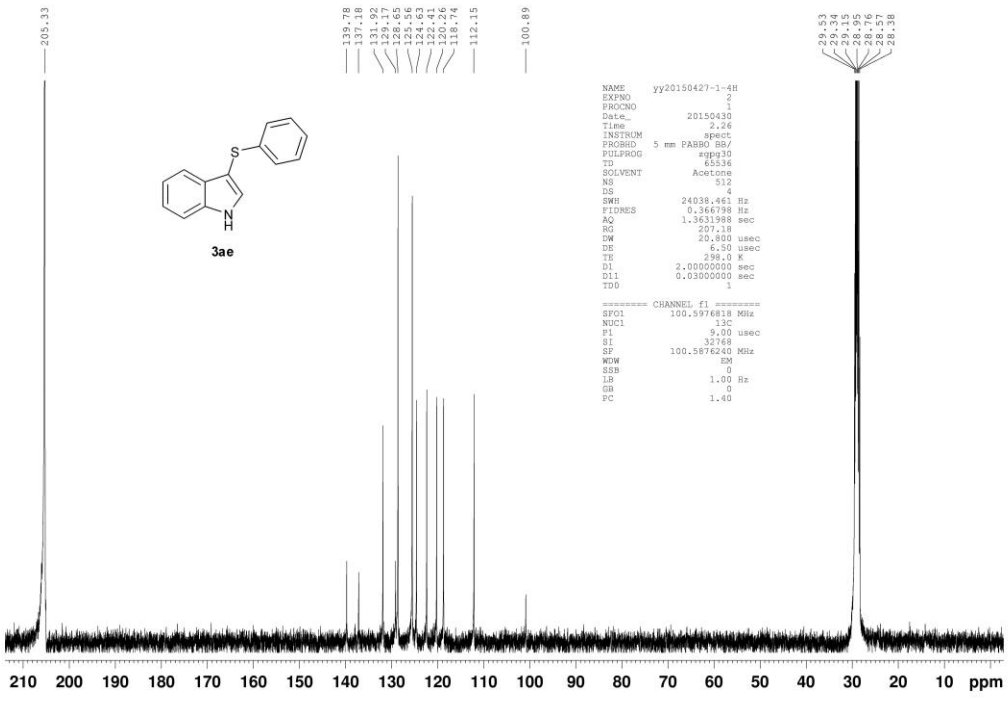
7.19
7.19
7.18
7.17
7.16
7.15
7.11
7.11
7.09
7.08
7.07
2.99
2.91
2.90
2.87
2.06
2.05
2.05
2.04
0.00
3ae
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.94
1.00
2.19
7.17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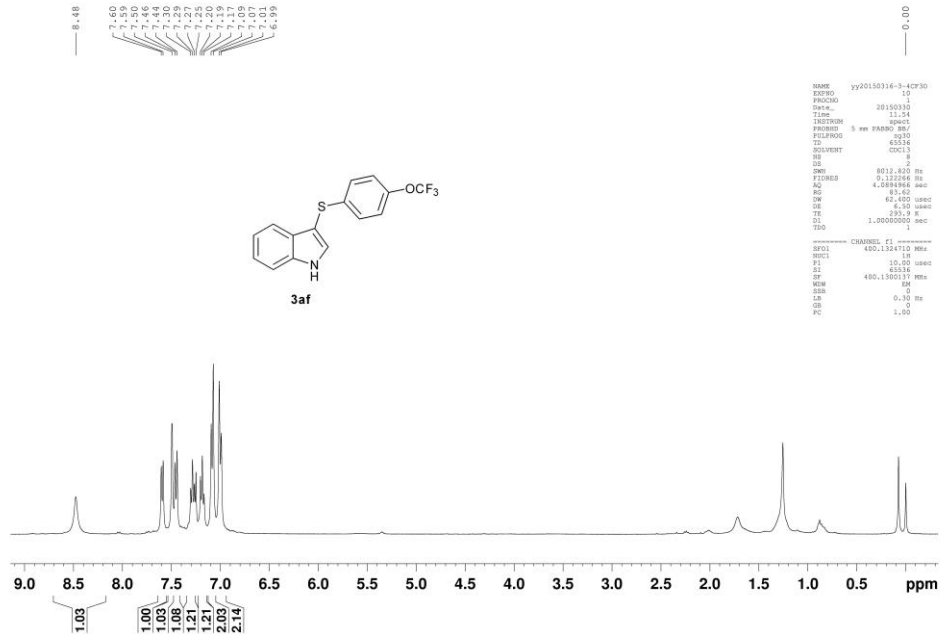
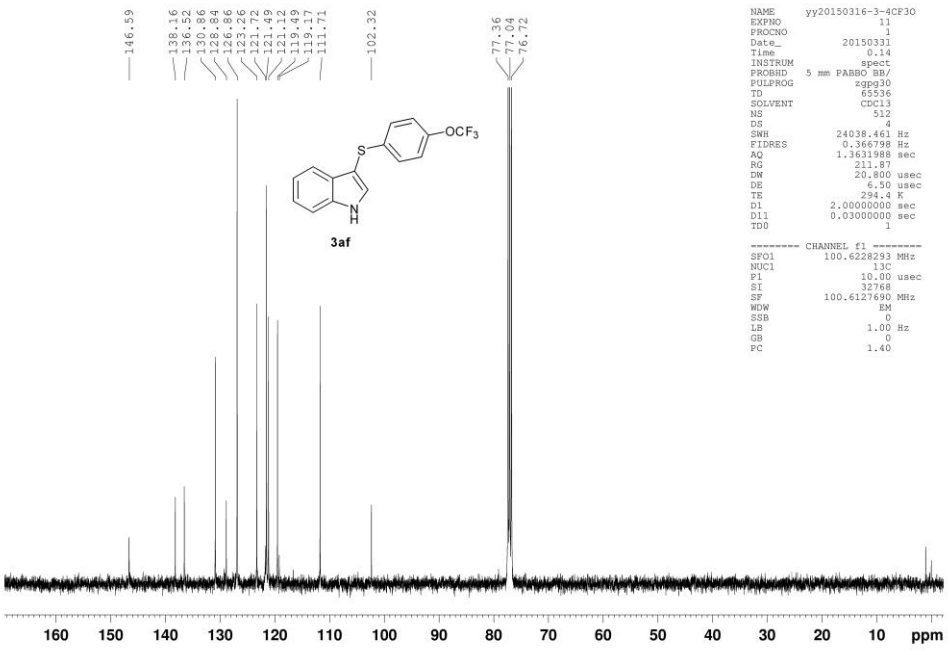
139.78
137.18
131.92
129.17
128.65
125.56
124.43
122.41
120.26
118.74
112.15
100.89
29.53
29.34
29.15
28.95
28.76
28.57
28.38
3ae
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

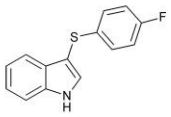



```
NAME          yy20150316-3-4CF3O
EXPNO                         10
PROCNO                         1
Date_                   20150330
Time                       11.54
INSTRUM                    spect
PROBHD   5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                            8
DS                            2
SWH                    8012.820 Hz
FIDRES                 0.122266 Hz
AQ                     4.0894964 sec
RG                         83.62
DW                        62.400 usec
DE                         6.50 usec
TE                        293.9 K
D1                     1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 400.1324710 MHz
NUC1                          1H
P1                         10.00 usec
SI                        65536
SF                   400.1300137 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

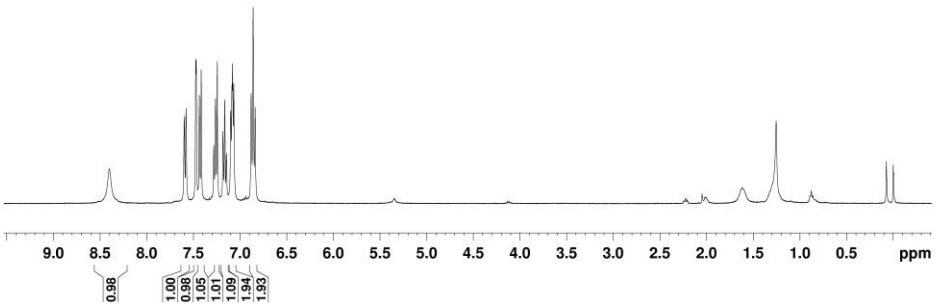


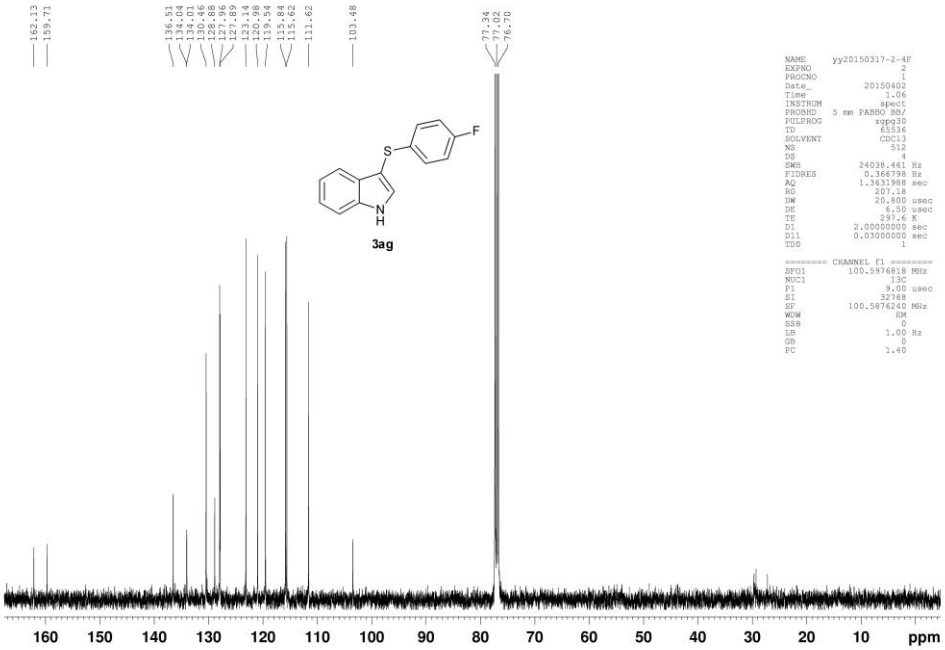

```
NAME          yy20150316-3-4CF3O
EXPNO                         11
PROCNO                         1
Date_                   20150331
Time                       0.14
INSTRUM                    spect
PROBHD   5 mm PABBO BB/
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          512
DS                            4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631988 sec
RG                       211.87
DW                       20.800 usec
DE                         6.50 usec
TE                        294.4 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 100.6228293 MHz
NUC1                          13C
P1                         10.00 usec
SI                        32768
SF                   100.6127690 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```


8.40
7.60
7.58
7.48
7.47
7.44
7.42
7.26
7.24
7.18
7.17
7.15
7.10
7.09
7.08
7.07
7.07
6.88
6.86
6.84

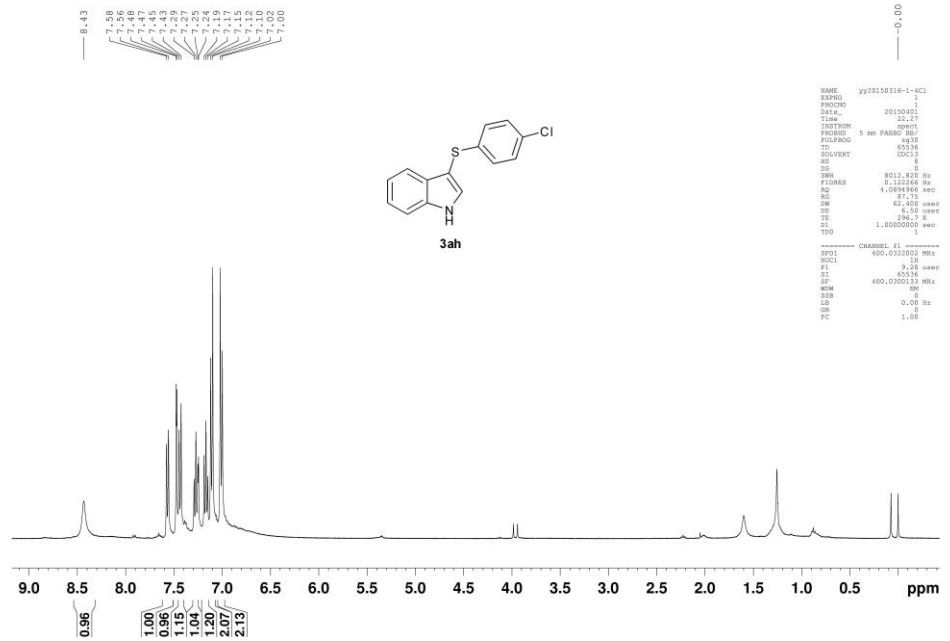

S
F
N
H

3ag


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
0.98
1.00
0.98
1.05
1.01
1.09
1.94
1.93

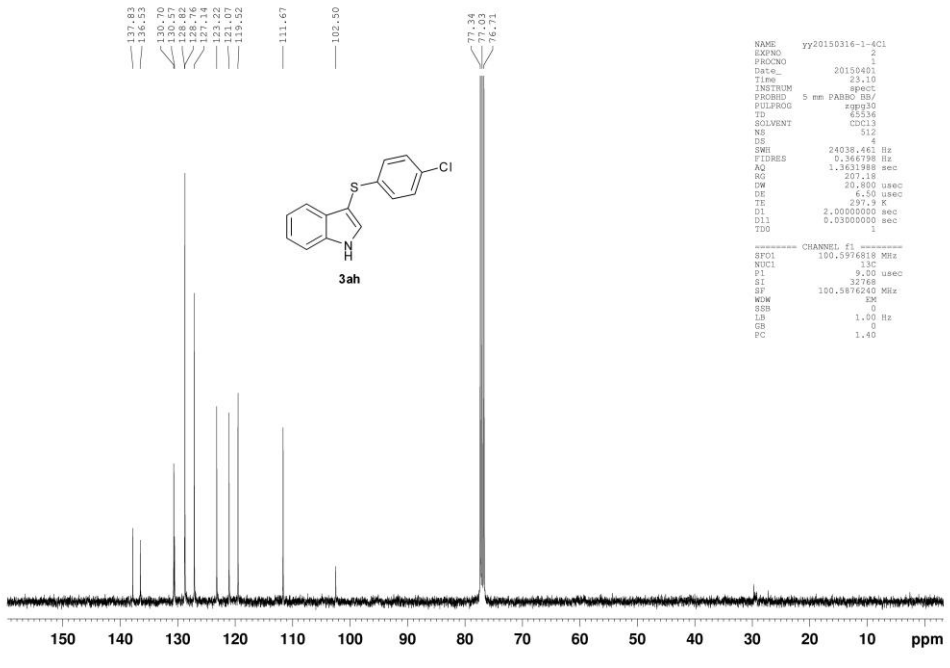

162.13
159.71
136.51
134.04
134.01
128.46
128.88
127.96
127.89
123.14
120.98
119.54
115.84
115.62
111.62
103.48
77.34
77.02
76.70
S
F
N
H
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

3ag



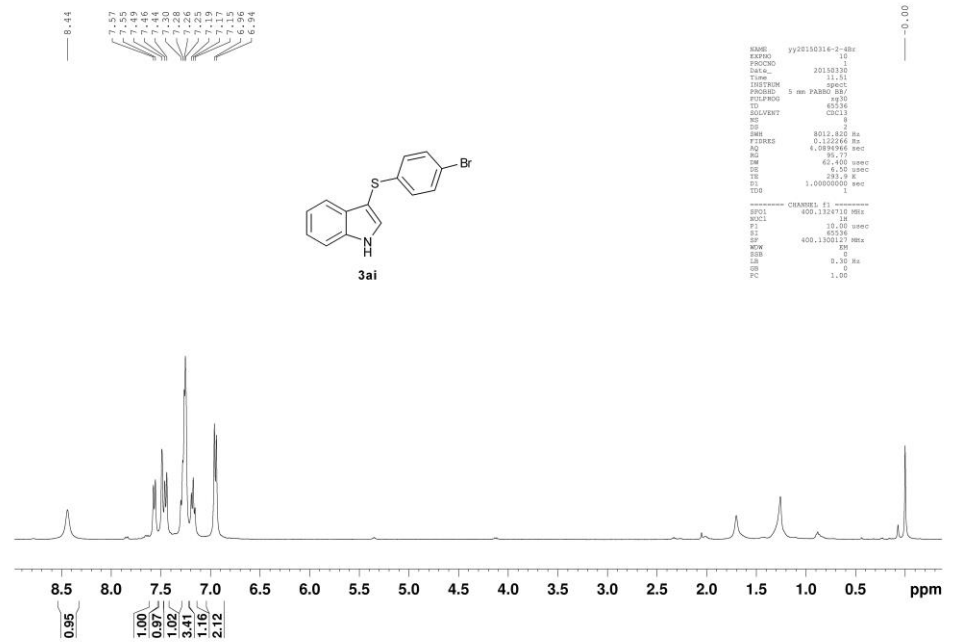
NAME yy20150316-1-4Cl
EXPNO 1
PROCNO 1
Date_ 20150401
Time 22.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 57.75
DW 62.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.0322002 MHz
NUC1 1H
P1 9.28 usec
SI 65536
SF 400.0300133 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



NAME yy20150316-1-4Cl
EXPNO 2
PROCNO 1
Date_ 20150401
Time 23.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 207.18
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.5976818 MHz
NUC1 13C
P1 9.00 usec
SI 32768
SF 100.5876240 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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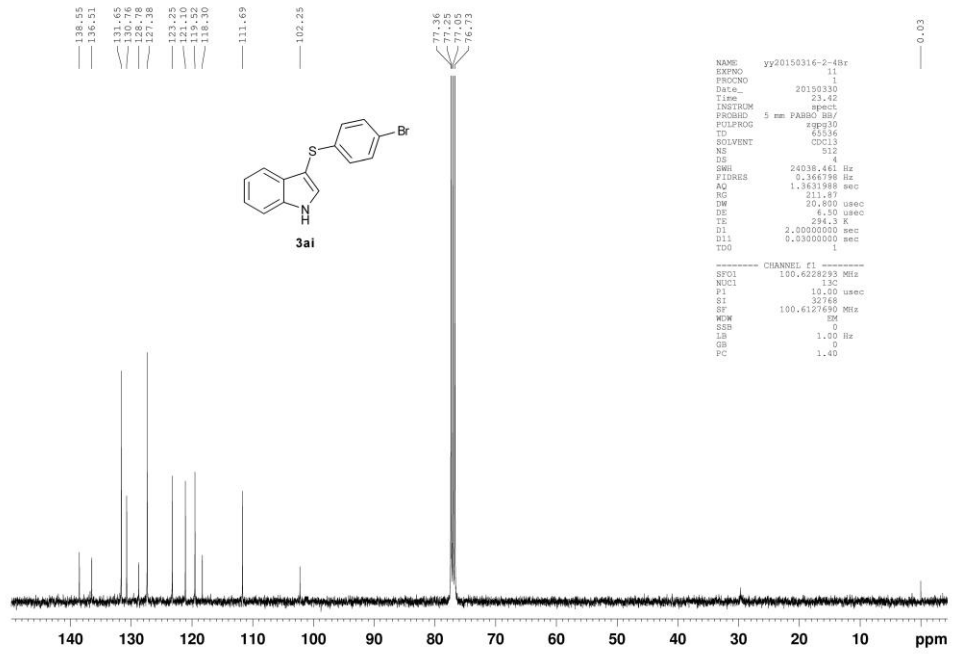
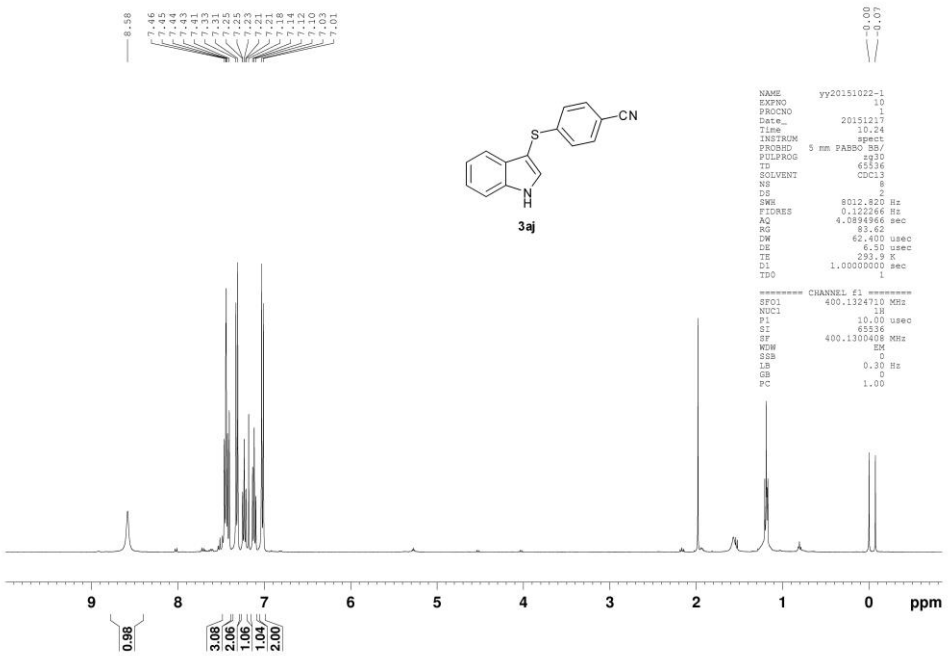
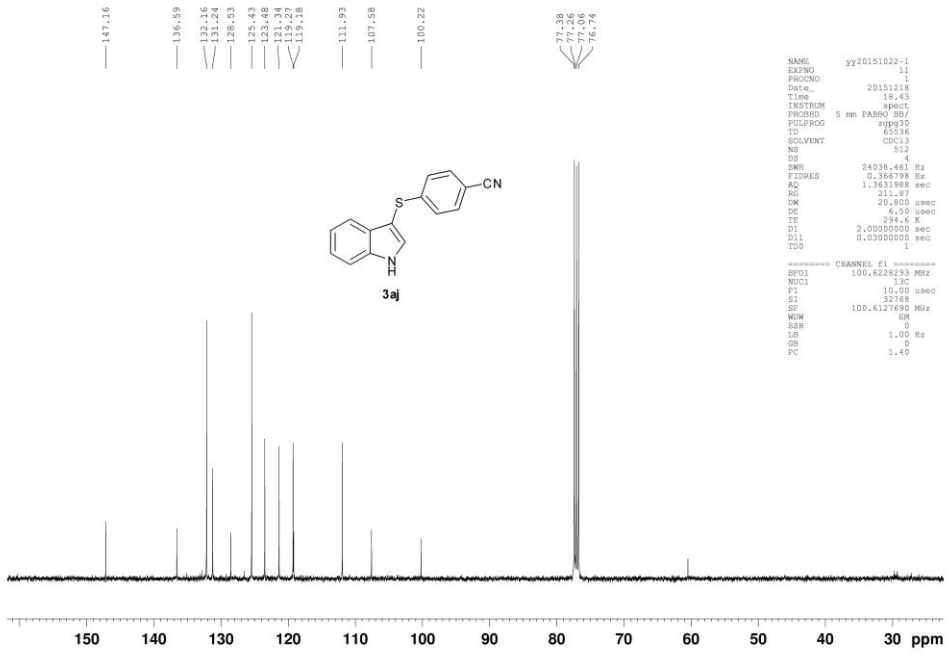
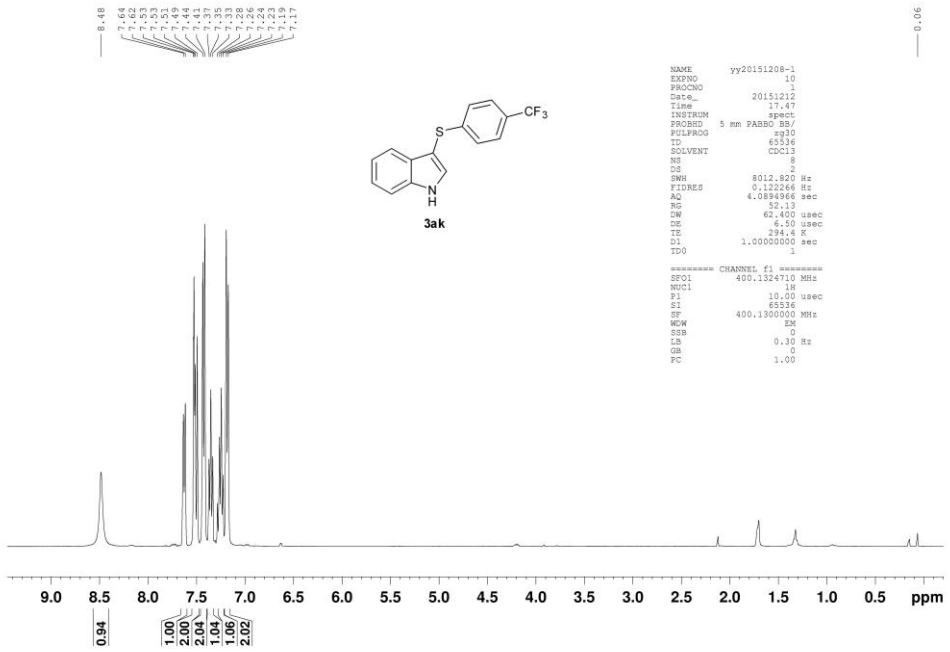
7.57
7.55
7.49
7.46
7.44
7.30
7.28
7.26
7.25
7.19
7.17
7.15
6.96
6.94
-0.00
Br
S
N
H
3ai
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.95
1.00
0.97
1.02
3.41
1.16
2.12


138.55
136.51
131.65
130.76
129.78
127.38
123.25
121.10
119.32
118.30
111.69
102.25
77.36
77.25
77.05
76.73
0.03
Br
S
N
H
3ai
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.58
7.46
7.45
7.44
7.43
7.41
7.33
7.31
7.25
7.25
7.23
7.21
7.21
7.18
7.14
7.12
7.10
7.03
7.01
0.00
0.07
CN
S
N
H
3aj
NAME yy20151022-1
EXPNO 10
PROCNO 1
Date_ 20151217
Time 10.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 83.62
DW 62.400 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 400.1300408 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
0.98
3.08
2.06
1.06
1.04
2.00


147.16
136.59
132.16
131.24
128.53
125.43
123.48
121.34
119.27
119.18
111.93
107.58
100.22
77.38
77.26
77.06
76.74
CN
S
N
H
3aj
NAME yy20151022-1
EXPNO 11
PROCNO 1
Date_ 20151218
Time 18.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 211.87
DW 20.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm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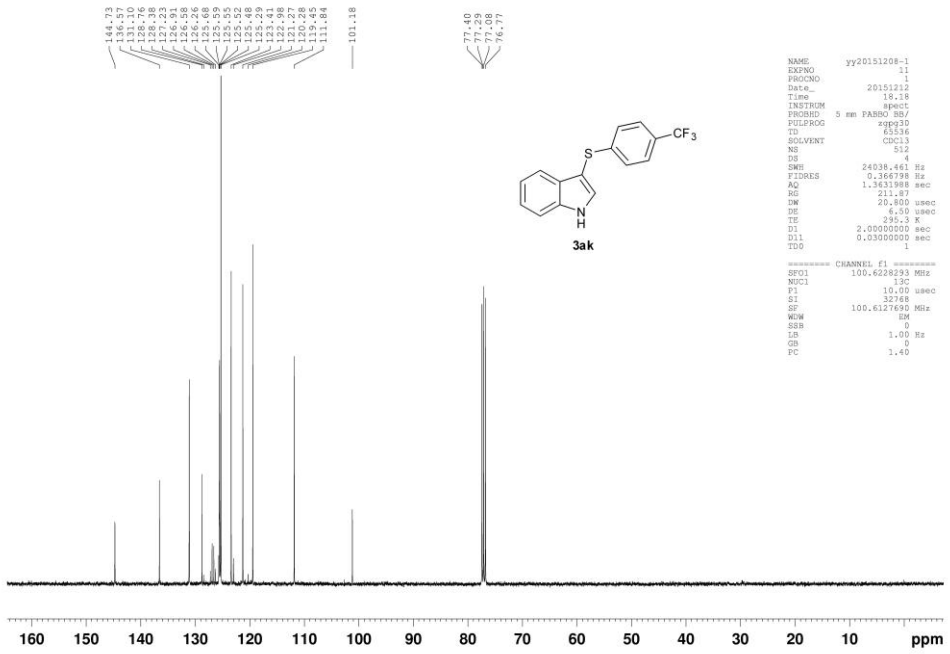
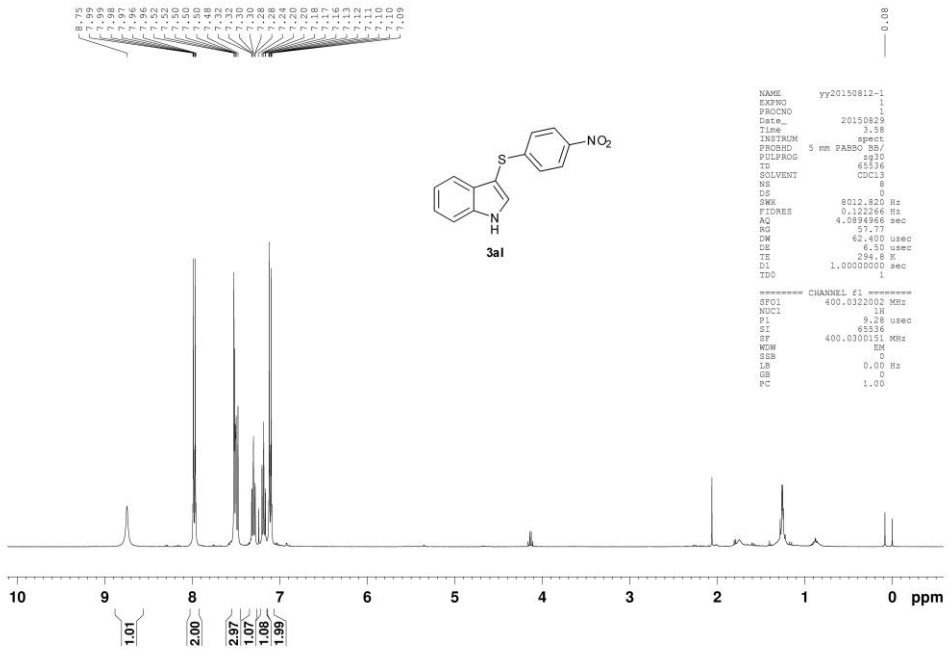
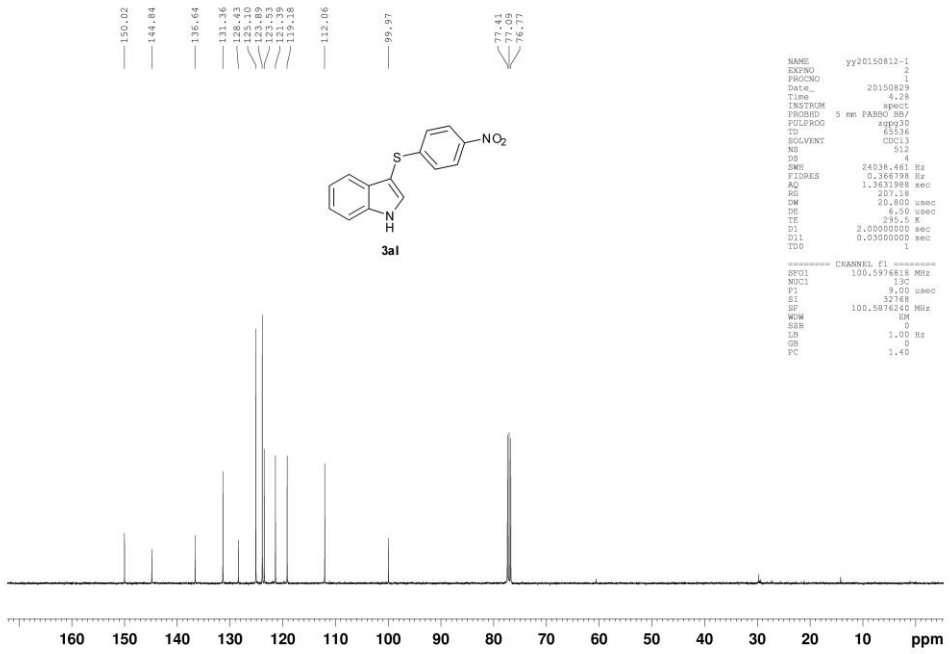
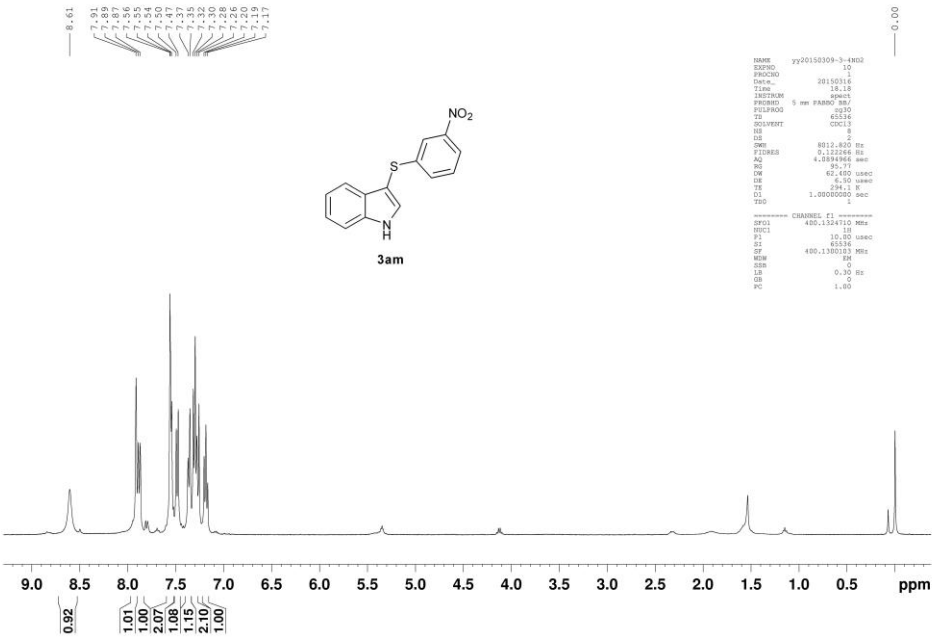
7.64
7.62
7.53
7.53
7.51
7.49
7.44
7.41
7.37
7.35
7.33
7.28
7.26
7.24
7.23
7.19
7.17
0.06
3ak
NAME yy20151208-1
EXPNO 10
PROCNO 1
Date_ 20151212
Time 17.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 52.13
DW 62.400 usec
DE 6.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
0.94
1.00
2.00
2.04
1.04
1.06
2.02


144.73
136.77
131.10
128.76
128.38
128.03
126.91
126.58
126.26
125.98
125.68
125.59
125.55
125.52
125.48
125.39
123.41
122.98
121.27
121.08
119.45
111.84
101.18
77.40
77.29
77.08
76.77
3ak
NAME yy20151208-1
EXPNO 11
PROCNO 1
Date_ 20151212
Time 18.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 211.87
DW 20.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


8.75
7.99
7.98
7.97
7.96
7.52
7.50
7.50
7.48
7.32
7.30
7.28
7.24
7.20
7.18
7.17
7.16
7.13
7.12
7.11
7.10
7.09
0.08
NO2
S
N
H
3al
NAME yy20150812-1
EXPNO 1
PROCNO 1
Date_ 20150829
Time 3.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 57.77
DW 62.400 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.0322002 MHz
NUC1 1H
P1 9.28 usec
SI 65536
SF 400.0300151 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
2.00
2.97
1.07
1.08
1.99


150.02
144.84
136.64
131.36
128.43
125.10
124.89
123.53
121.39
119.18
112.06
99.97
77.41
77.09
76.77
NO2
S
N
H
3al
NAME yy20150812-1
EXPNO 2
PROCNO 1
Date_ 20150829
Time 6.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 207.18
DW 20.800 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.5976818 MHz
NUC1 13C
P1 9.00 usec
SI 32768
SF 100.5876240 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.61
7.91
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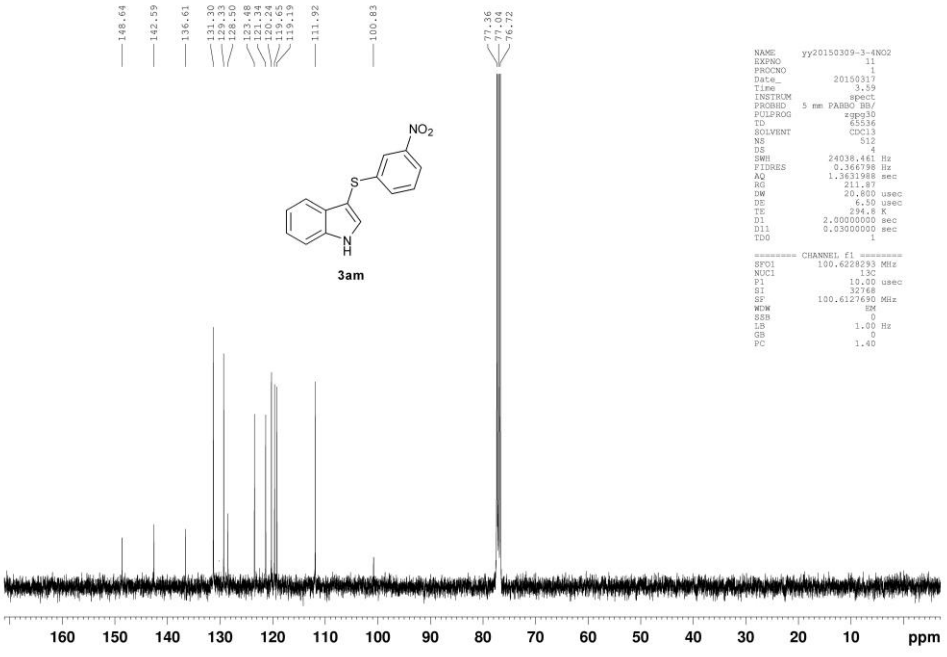
7.87
7.56
7.55
7.54
7.50
7.37
7.35
7.32
7.30
7.28
7.26
7.20
7.19
7.17
0.00
NO2
S
N
H
3am
NAME yy20150309-3-4NO2
EXPNO 10
PROCNO 1
Date_ 20150316
Time 18.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 95.77
DW 62.400 usec
DE 6.50 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
PL1 65536
SF 400.1300183 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
0.92
1.01
1.00
2.07
1.08
1.15
2.10
1.00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142.59
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131.30
129.33
128.50
123.48
121.34
120.34
119.65
119.19
111.92
100.83
77.36
77.04
76.72
NO2
S
N
H
3am
NAME yy20150309-3-4NO2
EXPNO 11
PROCNO 1
Date_ 20150317
Time 3.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 211.87
DW 20.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
PL1 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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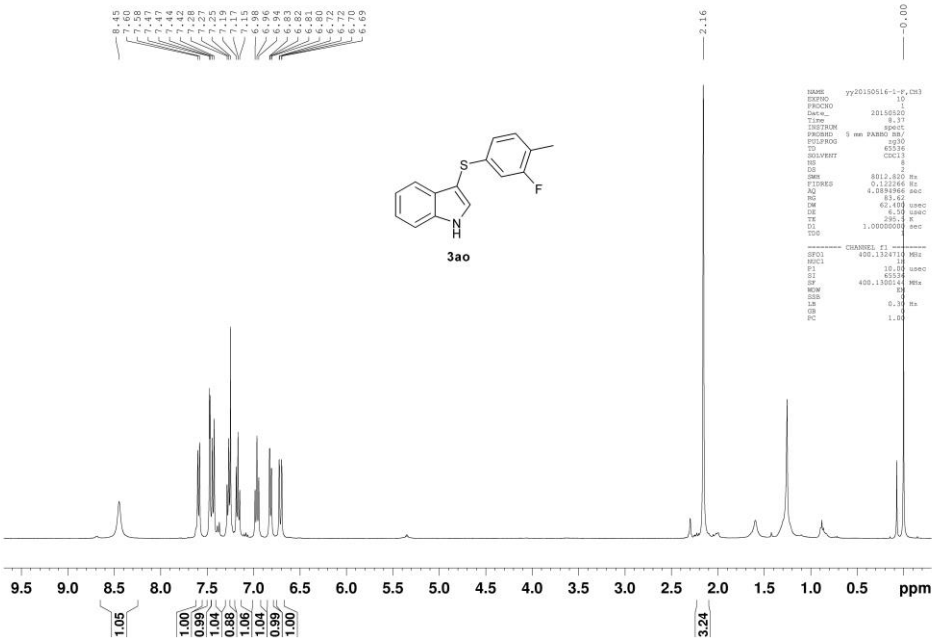
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.45
7.60
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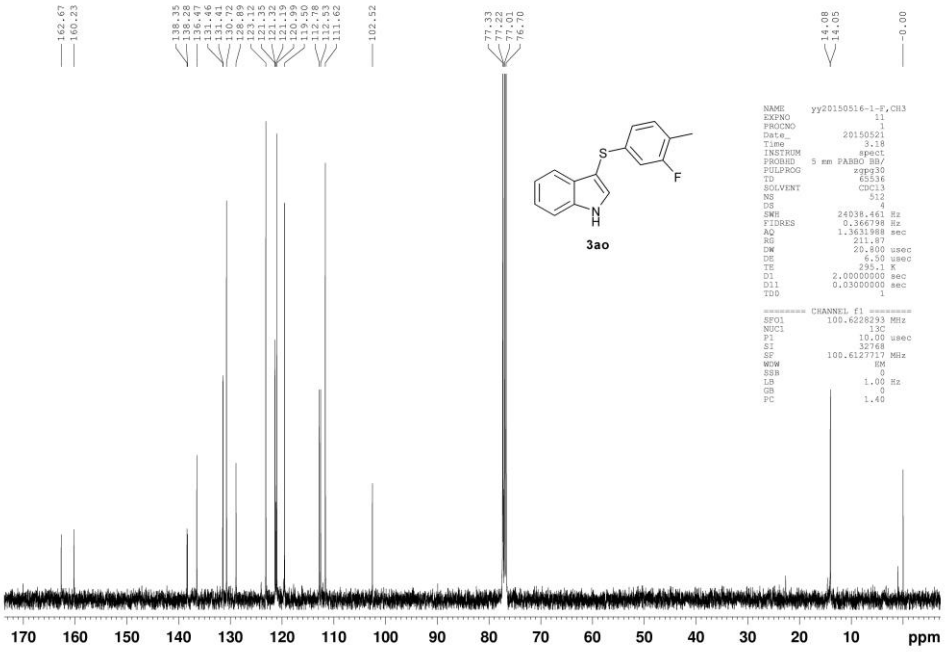
7.47
7.47
7.44
7.42
7.28
7.27
7.25
7.19
7.17
7.15
6.98
6.96
6.94
6.83
6.82
6.81
6.80
6.72
6.72
6.70
6.69
2.16
0.00
S
F
N
H
3ao
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.05
1.00
0.99
1.04
0.88
1.06
1.04
0.99
1.00
3.24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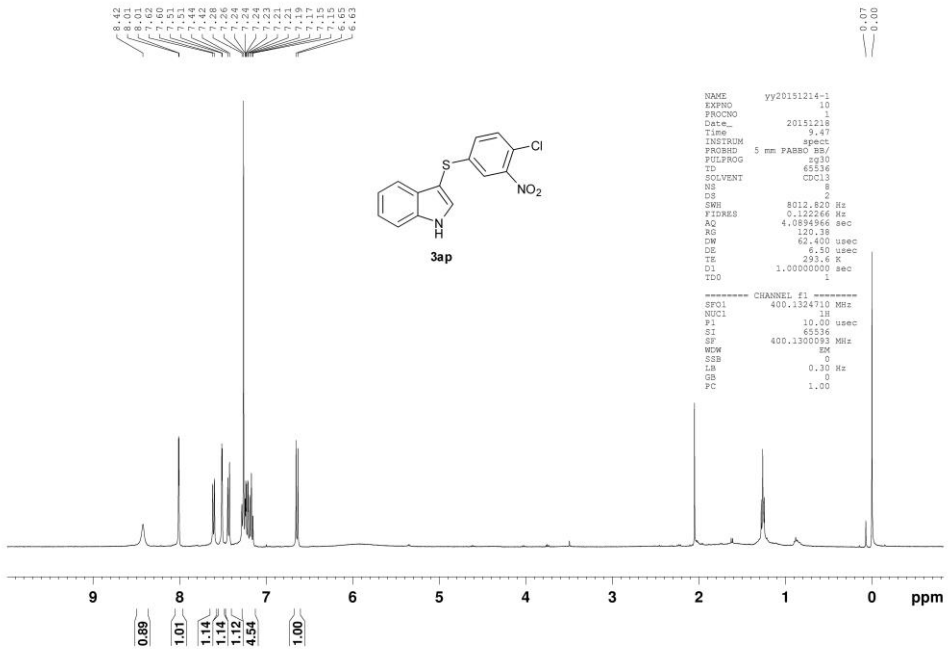
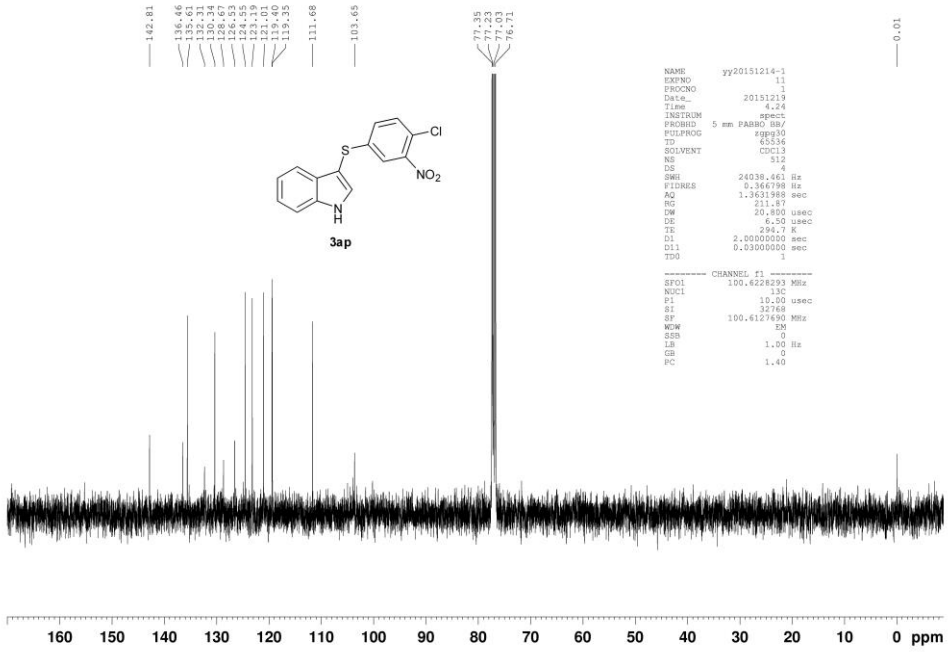
160.23
138.35
136.88
136.47
131.46
131.41
130.72
120.79
123.12
121.35
121.33
121.25
120.99
119.50
112.78
112.53
111.62
102.52
77.33
77.22
77.01
76.70
14.08
14.05
0.00
S
F
N
H
3ao
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


8.42
8.01
8.01
7.62
7.60
7.51
7.51
7.44
7.42
7.28
7.26
7.24
7.24
7.24
7.23
7.21
7.19
7.17
7.15
6.65
6.63
0.07
0.00
Cl
S
NO2
N
H
3ap
NAME yy20151214-1
EXPNO 10
PROCNO 1
Date_ 20151218
Time 9.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 120.38
DW 62.400 usec
DE 6.50 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
0.89
1.01
1.14
1.14
1.12
4.54
1.00


142.81
136.46
135.51
132.31
130.34
128.67
124.53
124.55
123.19
121.01
119.40
119.35
111.68
103.65
77.35
77.23
77.03
76.71
0.01
Cl
S
NO2
N
H
3ap
NAME yy20151214-1
EXPNO 11
PROCNO 1
Date_ 20151219
Time 4.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 211.87
DW 20.800 usec
DE 6.50 usec
TE 294.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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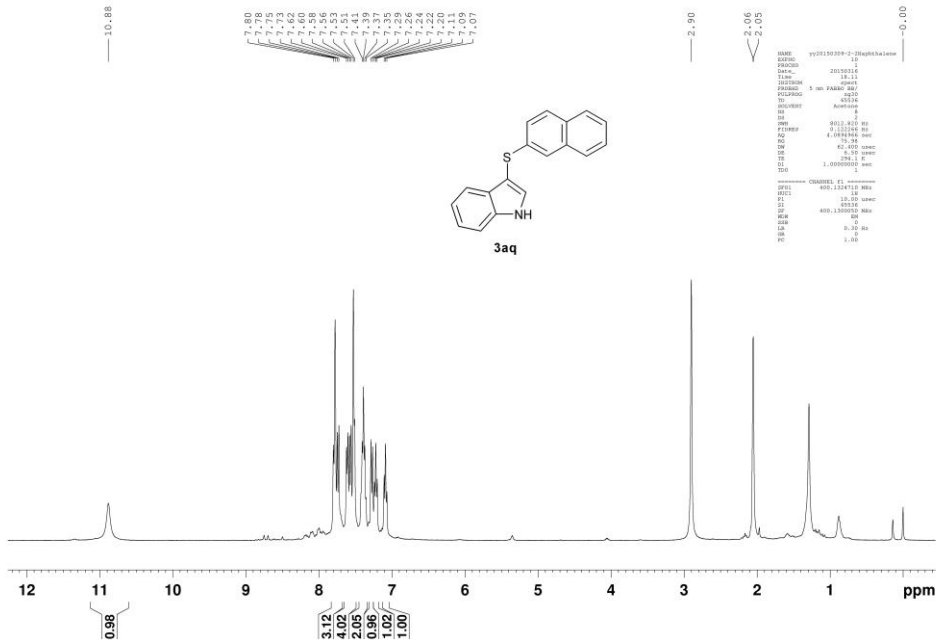
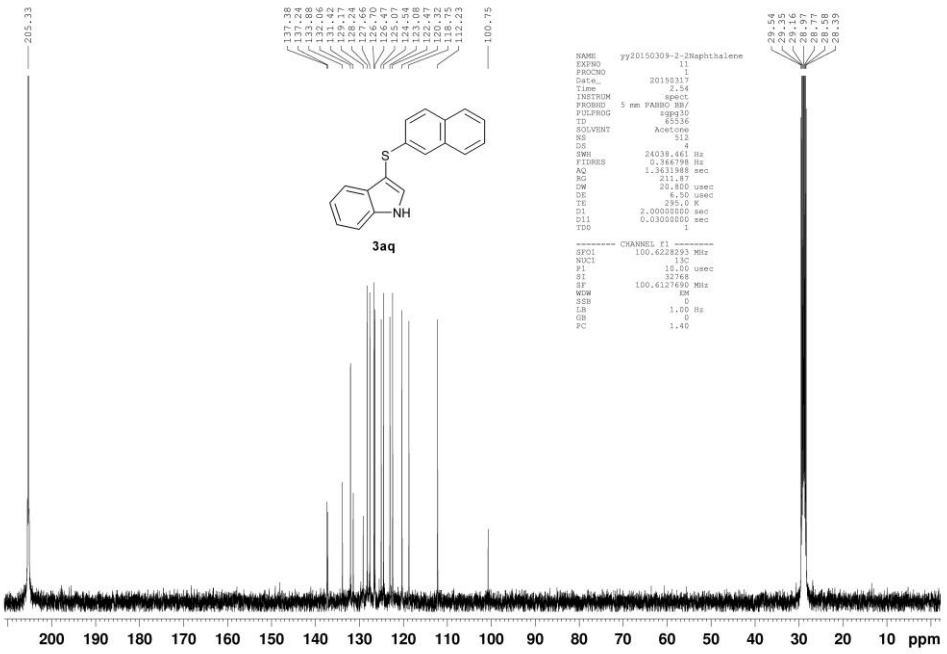
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


10.88
7.80
7.78
7.75
7.73
7.62
7.60
7.58
7.56
7.53
7.51
7.44
7.39
7.37
7.35
7.29
7.26
7.24
7.22
7.20
7.11
7.09
7.07
2.90
2.06
2.05
0.00
S
NH
3aq
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.98
3.12
4.02
2.05
0.96
1.02
1.00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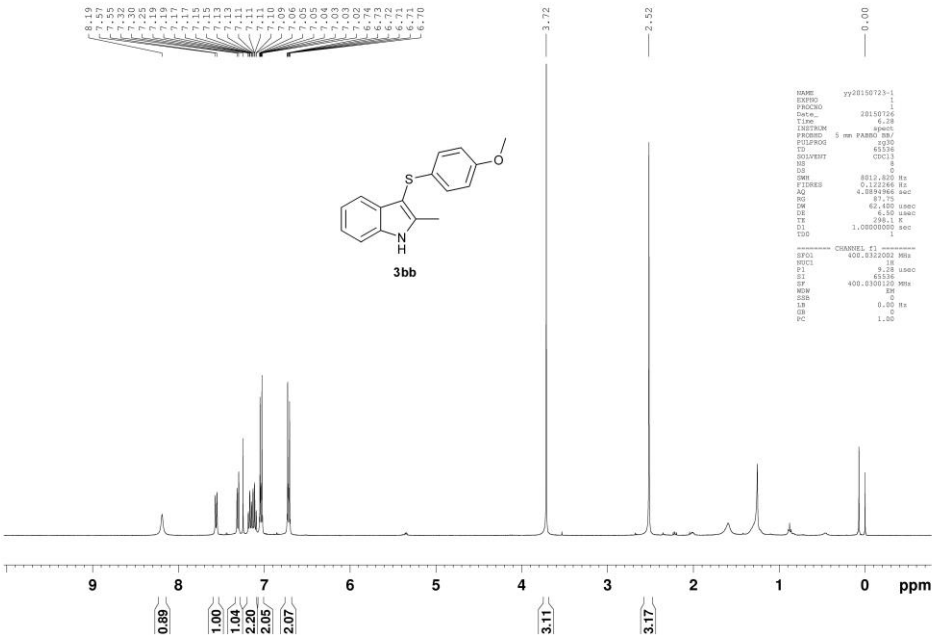
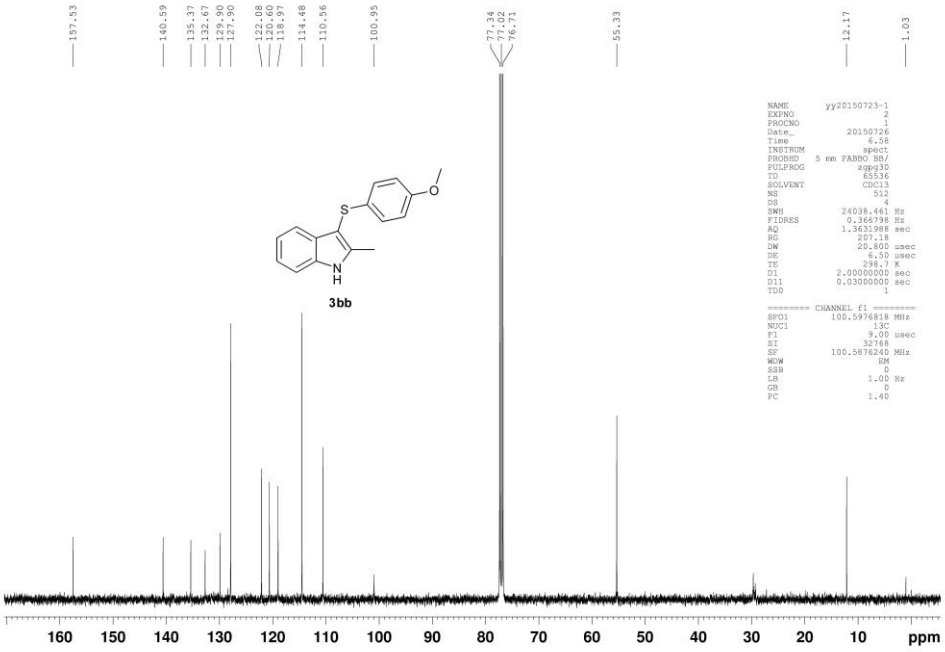
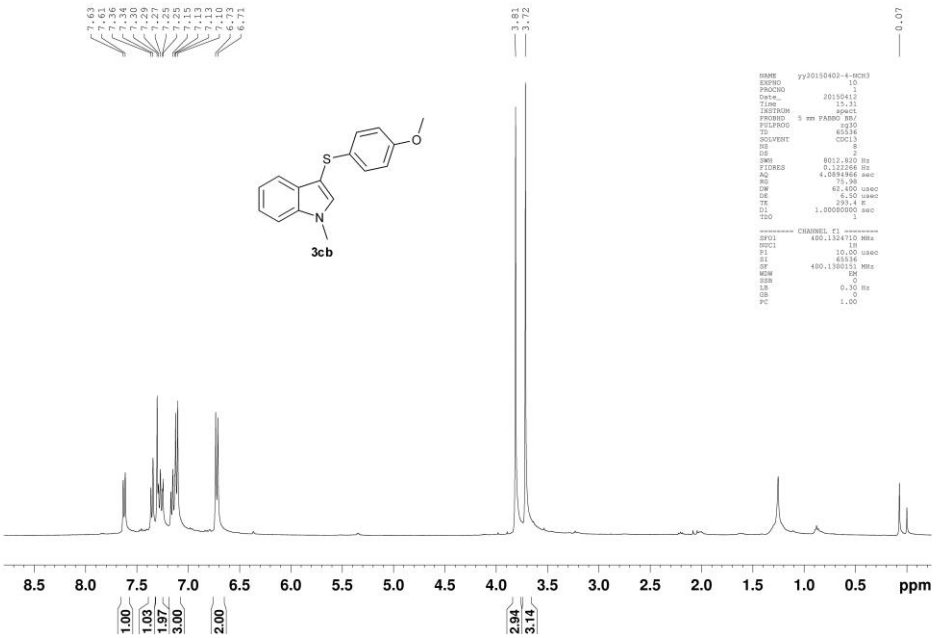
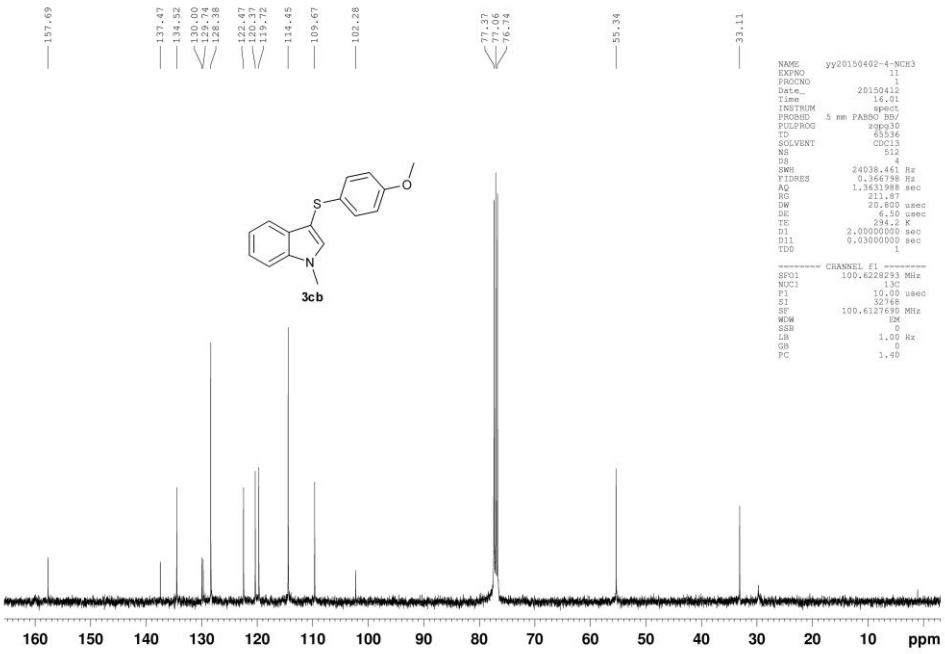
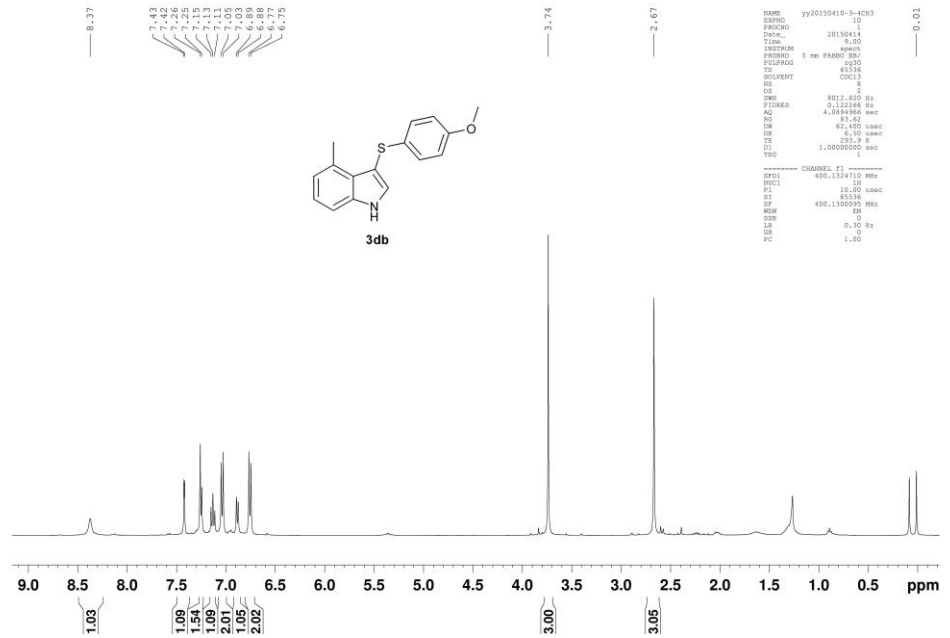
135.34
133.88
132.06
131.42
128.87
128.24
127.66
126.70
126.07
125.77
124.54
123.08
122.97
122.22
118.75
112.23
100.75
29.54
29.35
29.16
28.97
28.77
28.58
28.39
S
NH
3aq
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

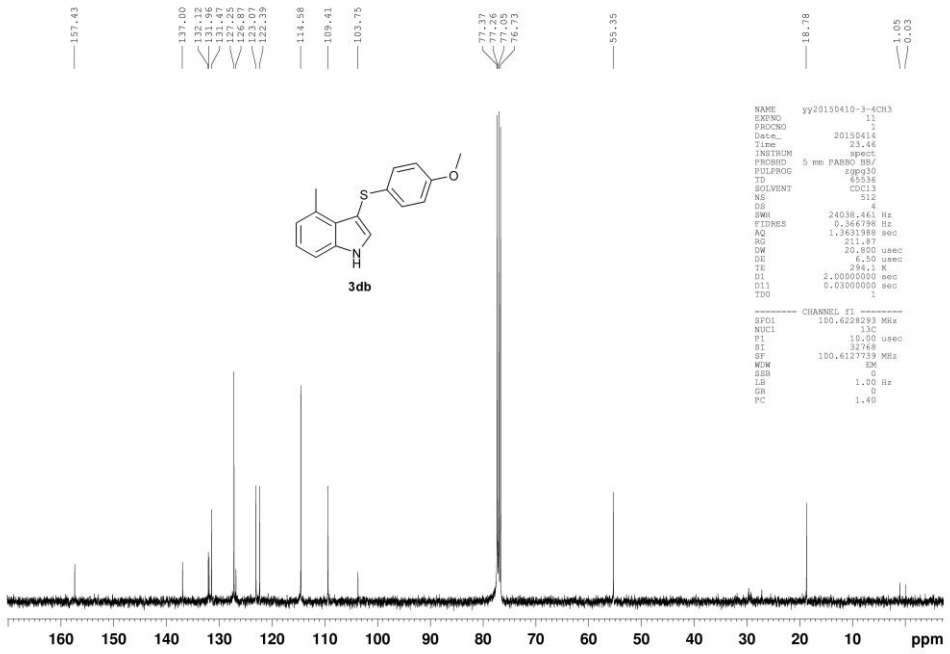

8.19
7.57
7.35
7.32
7.30
7.25
7.24
7.19
7.17
7.17
7.15
7.15
7.13
7.13
7.11
7.11
7.11
7.10
7.09
7.06
7.05
7.05
7.04
7.03
7.03
7.02
6.74
6.73
6.72
6.71
6.71
6.70
3.72
2.52
0.00
3bb
9
8
7
6
5
4
3
2
1
0
ppm
0.89
1.00
1.04
2.20
2.05
2.07
3.11
3.17

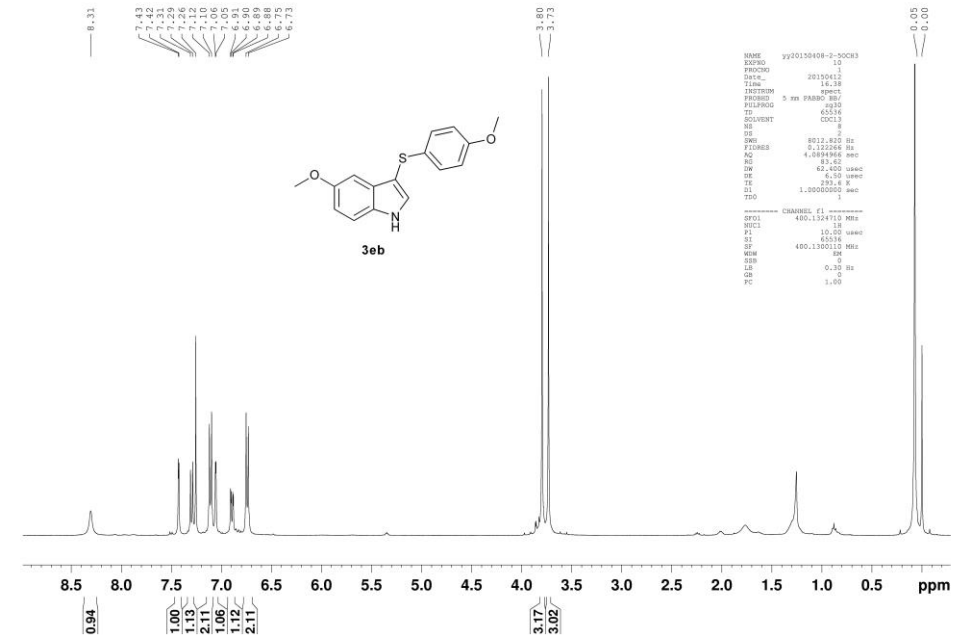

157.53
140.59
135.37
132.67
129.90
127.90
122.08
120.60
118.97
114.48
110.56
100.95
77.34
77.02
76.71
55.33
12.17
1.03
3bb
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

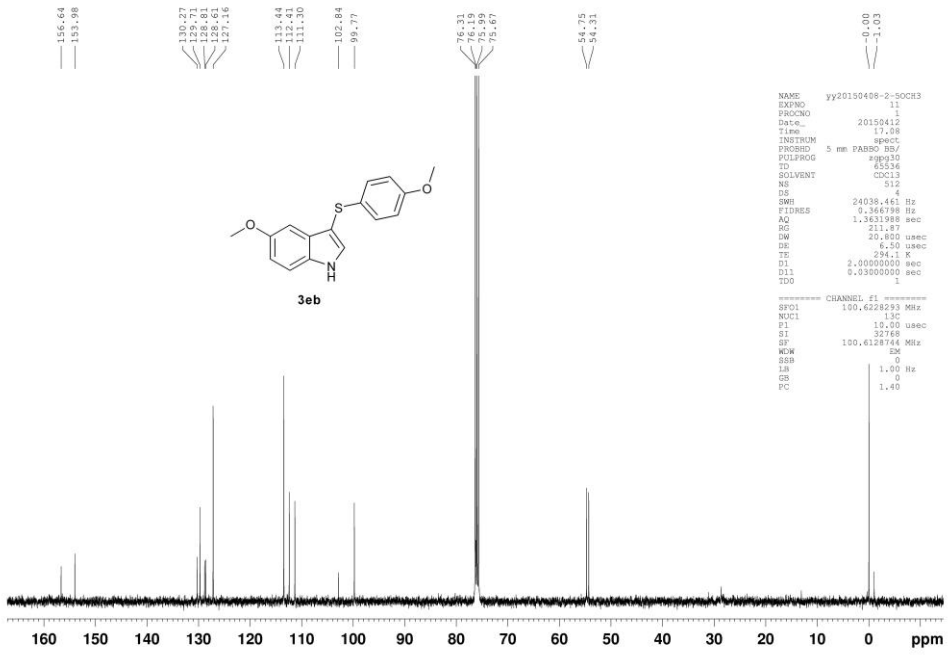

7.63
7.61
7.46
7.34
7.30
7.29
7.27
7.25
7.15
7.13
7.13
7.10
6.73
6.71
3.81
3.72
0.07
3cb
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.03
1.97
3.00
2.00
2.94
3.14

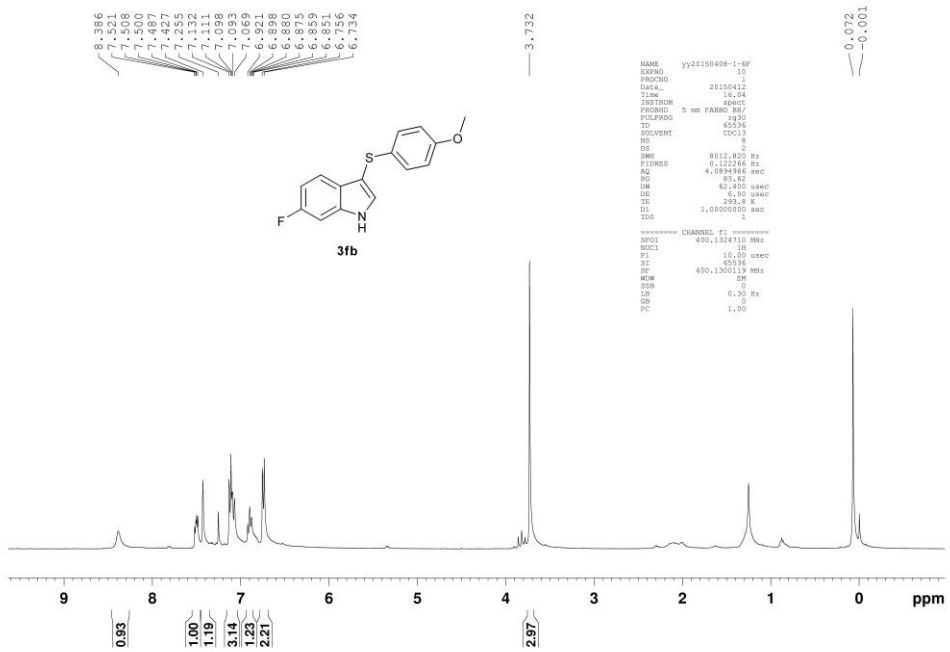

157.69
137.47
134.52
130.00
129.74
128.38
122.47
120.37
119.72
114.45
109.67
102.28
77.37
77.06
76.74
55.34
33.11
3cb
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

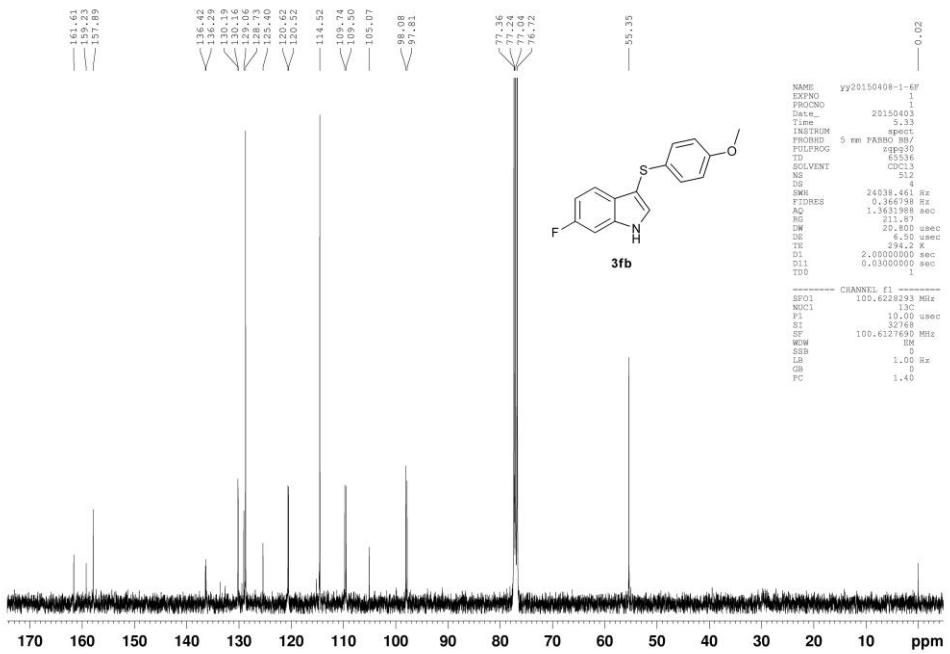

8.37
7.43
7.42
7.26
7.25
7.15
7.13
7.11
7.05
7.03
6.89
6.88
6.77
6.75
3.74
2.67
0.01
3db
NAME yy20150410-3-4CH3
EXPNO 10
PROCNO 1
Date_ 20150414
Time 9.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 83.62
DW 62.400 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.03
1.09
1.54
1.09
2.01
1.05
2.02
3.00
3.05

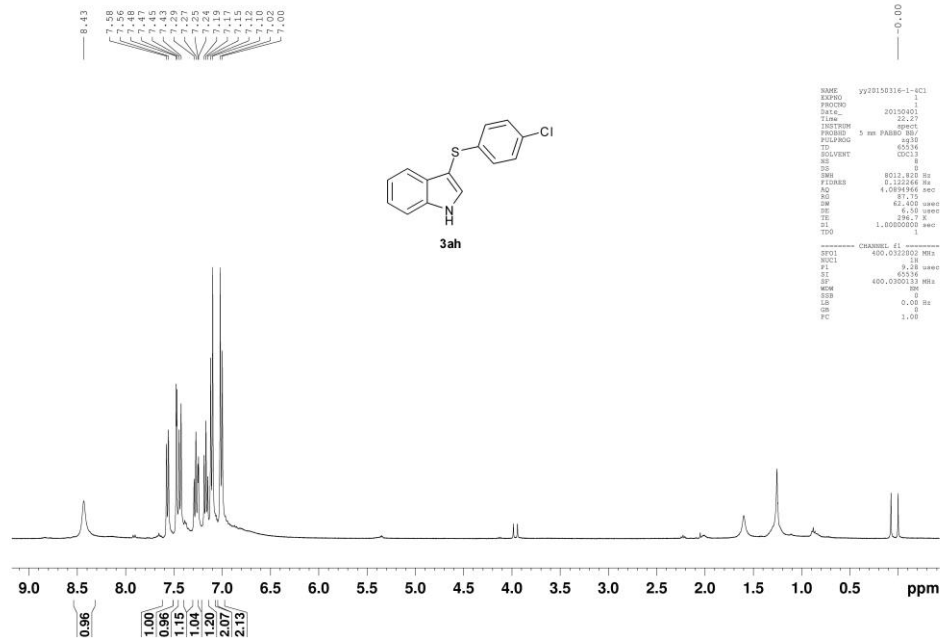

157.43
137.00
132.12
131.96
131.37
123.25
126.87
123.07
122.39
114.58
109.41
103.75
77.37
77.26
77.05
76.73
55.35
18.78
1.05
0.03
3db
NAME yy20150410-3-4CH3
EXPNO 11
PROCNO 1
Date_ 20150414
Time 23.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 211.87
DW 20.800 usec
DE 6.50 usec
TE 294.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127739 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


8.31
7.43
7.42
7.31
7.29
7.26
7.12
7.10
7.06
7.05
6.91
6.90
6.89
6.88
6.75
6.73
3.80
3.73
0.05
0.00
3eb
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
0.94
1.00
1.13
2.11
1.06
1.12
2.11
3.17
3.02


156.64
153.98
130.27
129.71
128.81
128.61
127.16
113.44
112.31
111.00
102.84
99.77
76.31
77.19
76.69
75.67
54.75
54.31
0.00
1.03
3eb
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

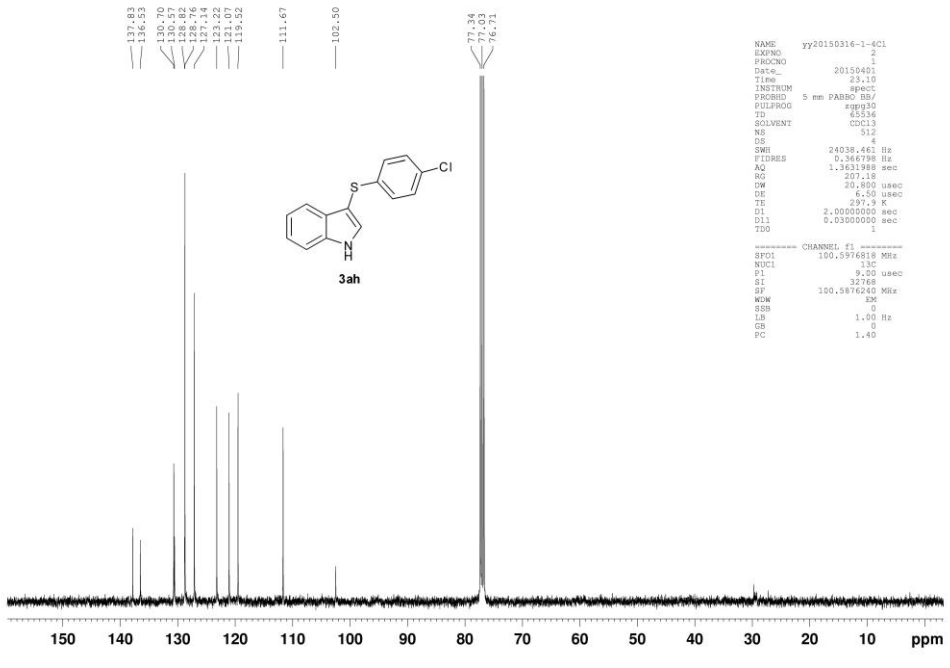

8.386
7.521
7.508
7.500
7.487
7.427
7.255
7.132
7.111
7.098
7.093
7.059
6.921
6.888
6.880
6.867
6.859
6.851
6.756
6.734
3.732
0.072
0.001
3fb
9
8
7
6
5
4
3
2
1
0
ppm
0.93
1.00
1.19
3.14
1.23
2.21
2.97


161.61
159.23
157.89
136.42
136.29
130.19
130.16
129.66
128.73
125.40
120.62
120.52
114.52
109.74
109.50
105.07
98.08
97.81
77.36
77.04
76.72
55.35
0.02
3fb
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



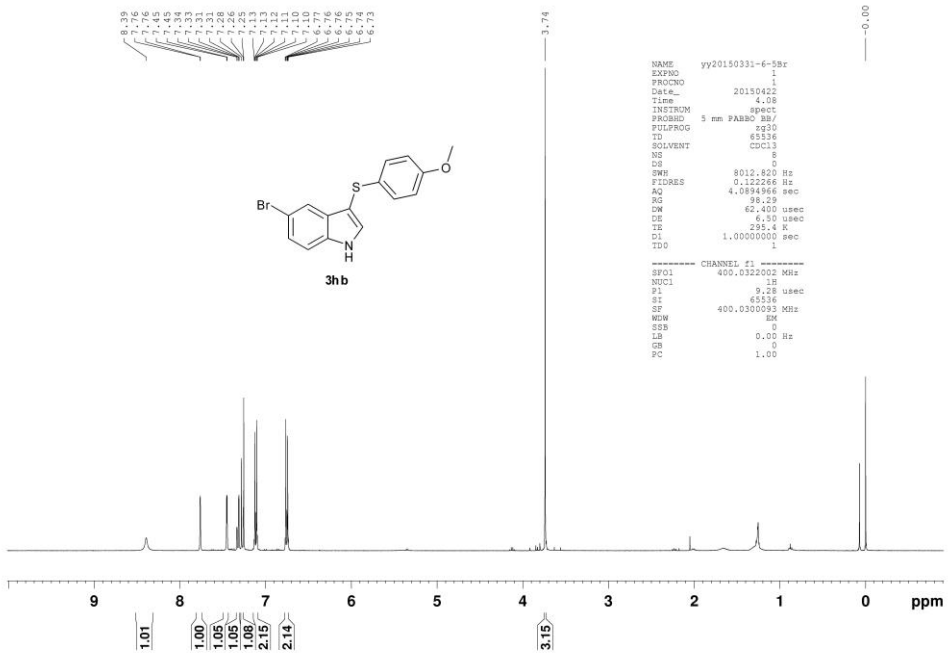
NAME yy20150316-1-4Cl
EXPNO 1
PROCNO 1
Date_ 20150401
Time 22.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 57.75
DW 62.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1

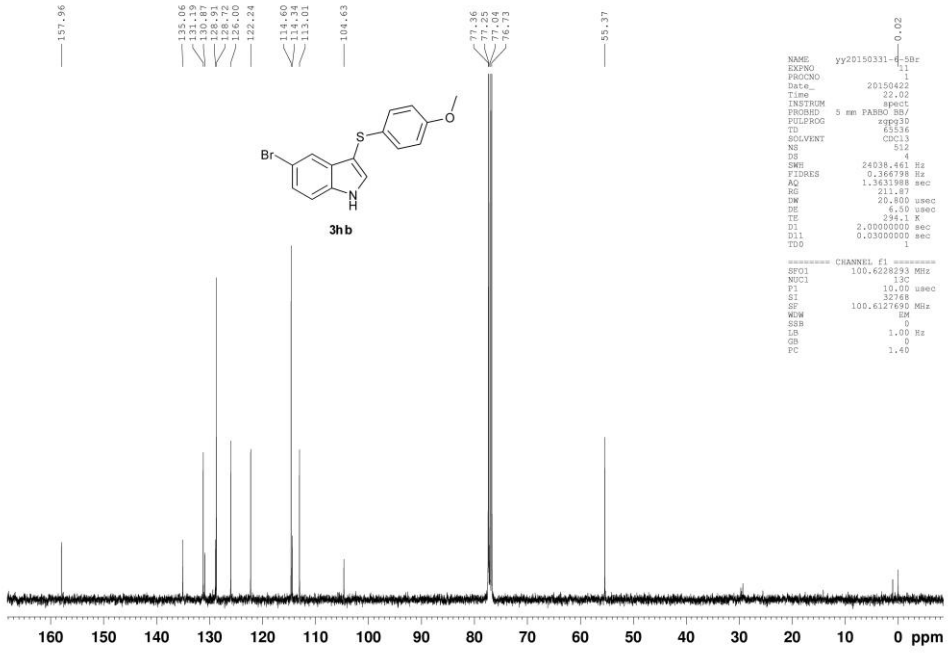
======== CHANNEL f1 ========
SFO1 400.0322002 MHz
NUC1 1H
P1 9.28 usec
SI 65536
SF 400.0300133 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

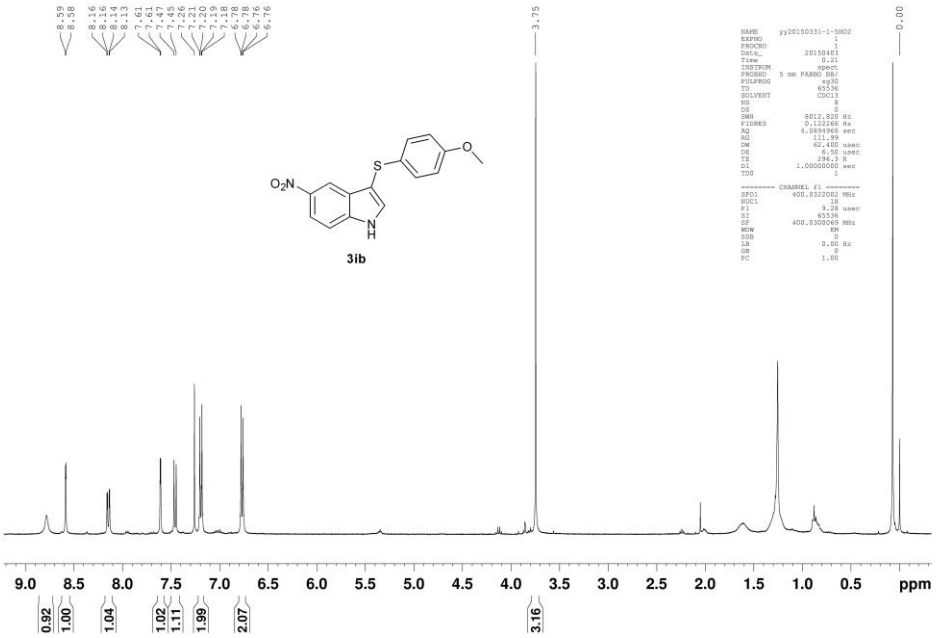


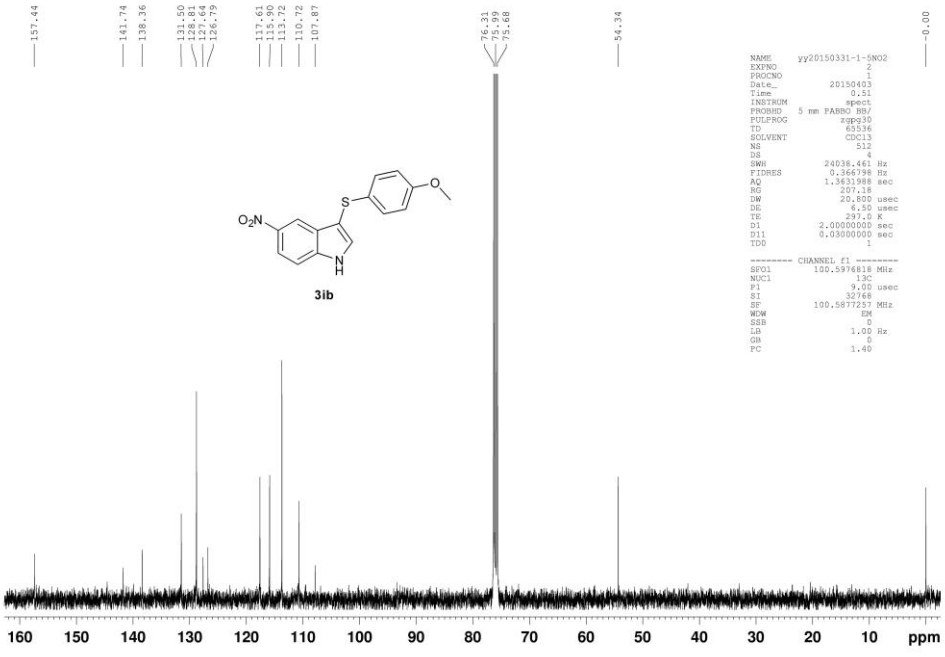
NAME yy20150316-1-4Cl
EXPNO 2
PROCNO 1
Date_ 20150401
Time 23.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 207.18
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

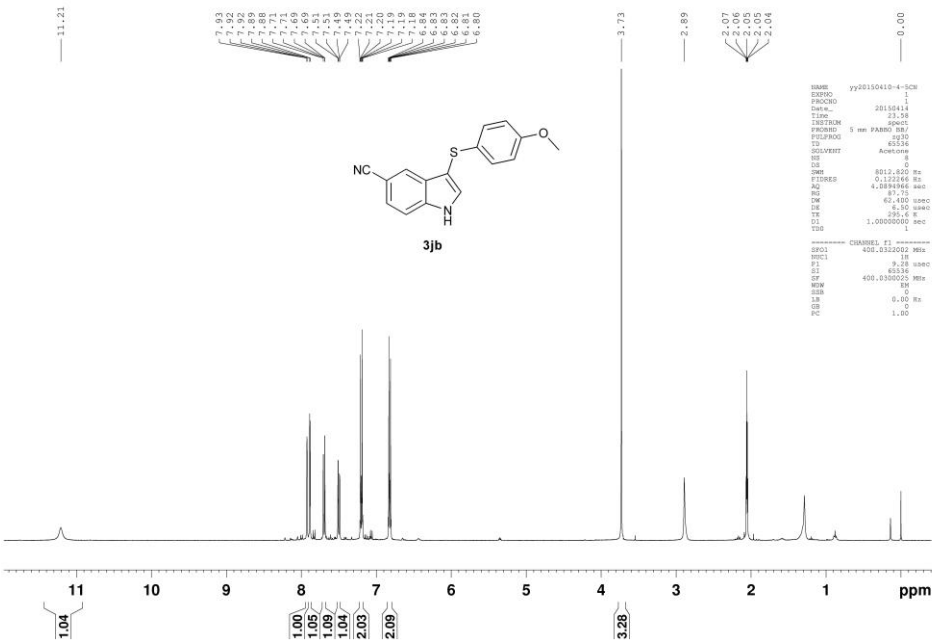
======== CHANNEL f1 ========
SFO1 100.5976818 MHz
NUC1 13C
P1 9.00 usec
SI 32768
SF 100.5876240 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

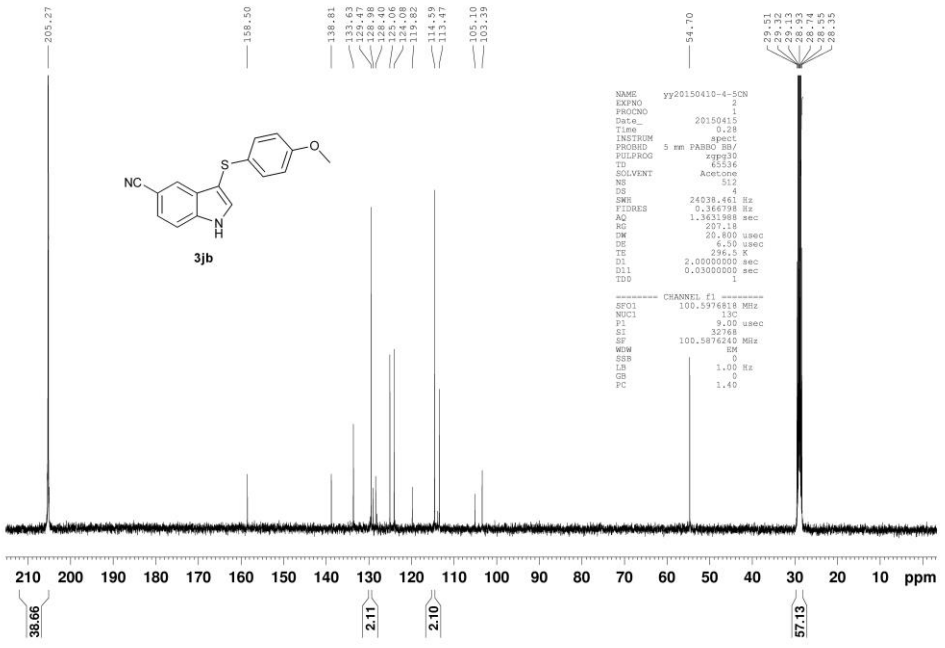

8.39
7.76
7.45
7.34
7.31
7.28
7.26
7.13
7.12
7.10
6.77
6.76
6.75
6.74
6.73
3.74
-0.00
Br
S
O
N
H
3hb
NAME yy20150331-6-5Br
EXPNO 1
PROCNO 1
Date_ 20150422
Time 4.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 98.29
DW 62.400 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.0322002 MHz
NUC1 1H
P1 9.28 usec
SI 65536
SF 400.0300093 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.01
1.00
1.05
1.05
1.08
2.15
2.14
3.15

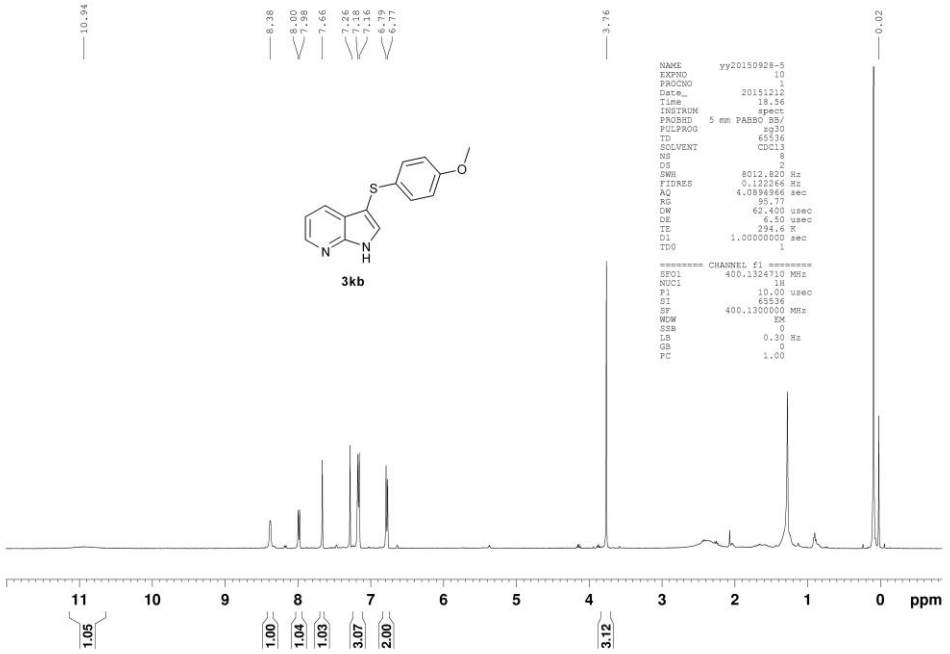

157.96
135.06
131.19
130.37
128.91
128.72
126.00
122.24
114.60
114.34
113.01
104.63
77.36
77.05
77.04
76.73
55.37
0.02
Br
S
O
N
H
3hb
NAME yy20150331-6-5Br
EXPNO 11
PROCNO 1
Date_ 20150422
Time 22.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 211.87
DW 20.800 usec
DE 6.50 usec
TE 294.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

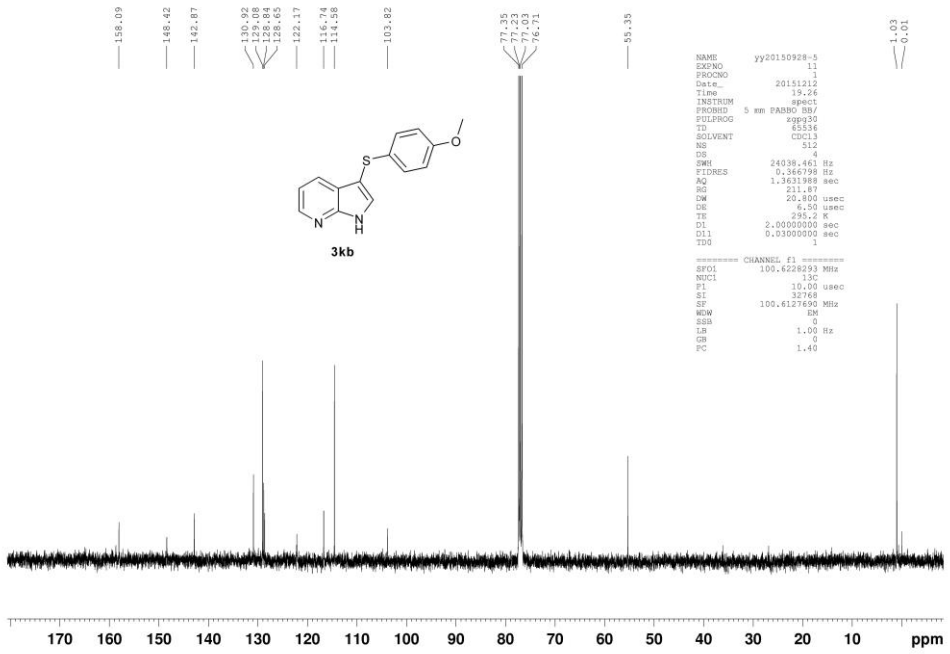

8.59
8.58
8.16
8.16
8.14
8.13
7.61
7.61
7.47
7.45
7.26
7.21
7.20
7.19
7.18
6.78
6.78
6.76
6.76
3.75
0.00
O2N
S
O
N
H
3ib
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.92
1.00
1.04
1.02
1.11
1.99
2.07
3.16

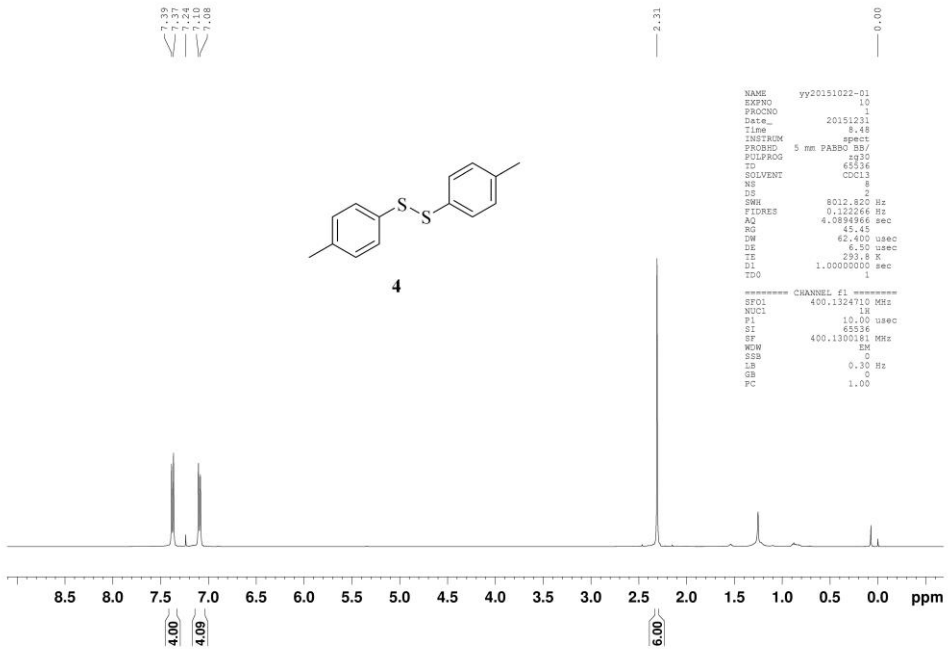

157.44
141.74
138.36
131.50
128.81
127.64
126.79
117.61
115.90
113.72
110.72
107.87
76.31
77.00
75.68
54.34
0.00
O2N
S
O
N
H
3ib
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

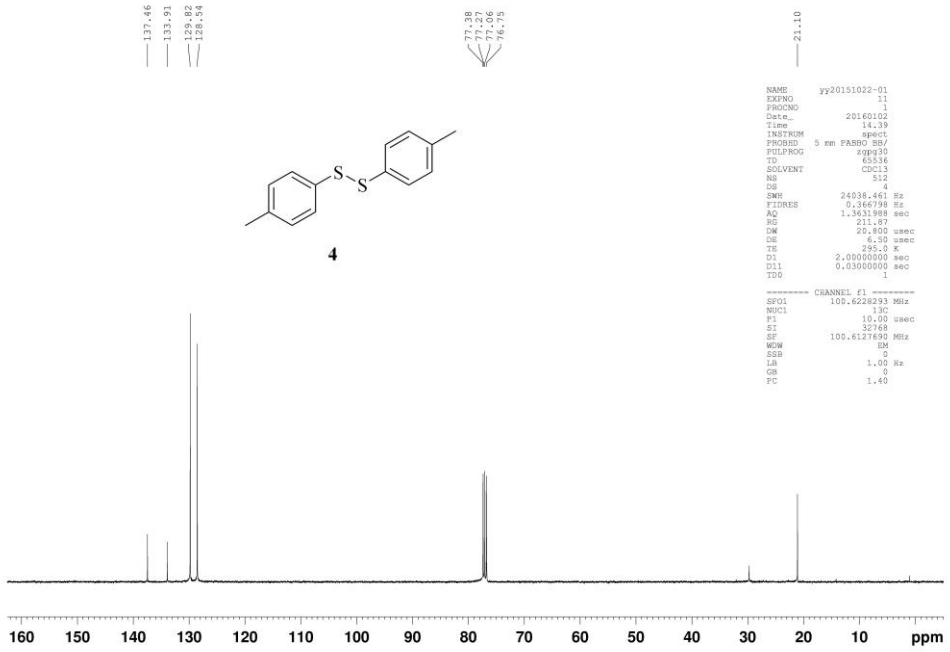

11.21
7.93
7.92
7.92
7.89
7.88
7.71
7.71
7.69
7.69
7.51
7.51
7.49
7.49
7.22
7.21
7.20
7.19
7.19
7.18
6.84
6.83
6.83
6.82
6.81
6.80
3.73
2.89
2.07
2.06
2.05
2.05
2.04
0.00
NC
S
O
N
H
3jb
11
10
9
8
7
6
5
4
3
2
1
ppm
1.04
1.00
1.05
1.09
1.04
2.03
2.09
3.28

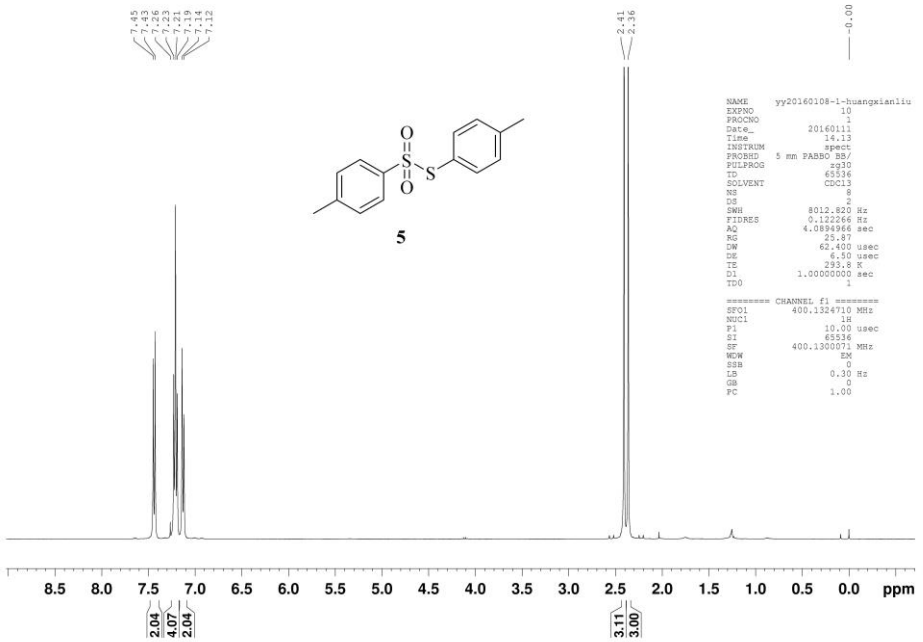

205.27
158.50
138.81
133.63
129.47
128.88
128.40
125.06
124.08
119.02
114.59
113.47
105.10
103.39
54.70
29.51
29.22
29.13
28.93
28.74
28.55
28.35
NC
S
O
N
H
3jb
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
38.66
2.11
2.10
57.13

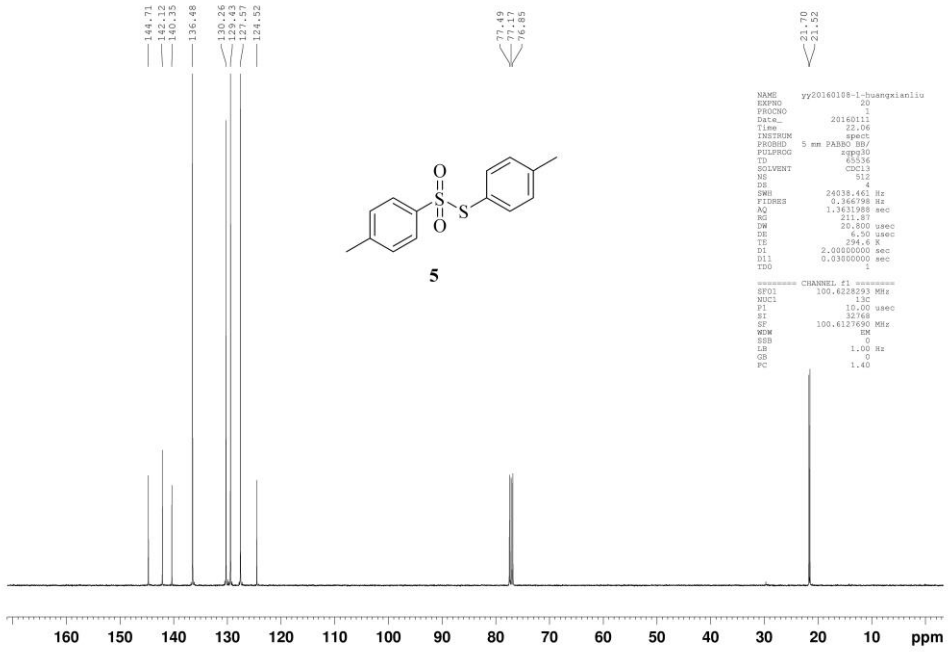

10.94
8.38
8.00
7.98
7.66
7.26
7.18
7.16
6.79
6.77
3.76
0.02
3kb
NAME yy20150928-5
EXPNO 10
PROCNO 1
Date_ 20151212
Time 18.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.08949466 sec
RG 90.77
DW 62.400 usec
DE 6.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.05
1.00
1.04
1.03
3.07
2.00
3.12


158.09
148.42
142.87
130.92
129.08
128.84
128.65
122.17
116.74
114.58
103.82
77.35
77.23
77.03
76.71
55.35
1.03
0.01
3kb
NAME yy20150928-5
EXPNO 11
PROCNO 1
Date_ 20151212
Time 19.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 211.87
DW 20.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.39
7.37
7.24
7.10
7.08
2.31
0.00
S
S
4
NAME yy20151022-01
EXPNO 10
PROCNO 1
Date_ 20151231
Time 8.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 45.45
DW 62.400 usec
DE 6.50 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 400.1300181 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 0.0 ppm
4.00
4.09
6.00


137.46
133.91
129.82
128.54
77.38
77.27
77.06
76.75
21.10
S
S
4
NAME yy20151022-01
EXPNO 11
PROCNO 1
Date_ 20160102
Time 14.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 211.87
DW 20.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.45
7.43
7.26
7.23
7.21
7.19
7.14
7.12
2.41
2.36
0.00
5
NAME yy20160108-1-huangxianliu
EXPNO 10
PROCNO 1
Date_ 20160111
Time 14.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 25.87
DW 62.400 usec
DE 6.50 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 400.1300071 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 0.0 ppm
2.04
4.07
2.04
3.11
3.00


144.71
143.72
140.35
136.48
130.26
129.83
127.57
124.52
77.49
77.17
76.85
21.70
21.52
5
NAME yy20160108-1-huangxianliu
EXPNO 20
PROCNO 1
Date_ 20160111
Time 22.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 211.87
DW 20.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm