Supporting Information

for

Synthesis of 1*H*-indole-3-sulfonates via Palladium-catalyzed Tandem Reaction of 2-Alkynyl Arylazides with Sulfonic Acids

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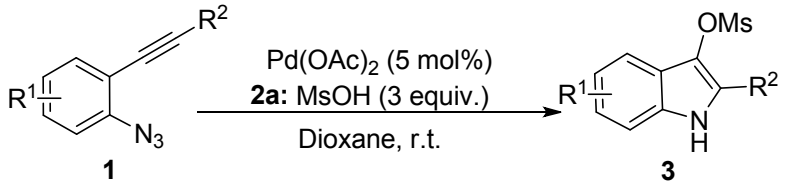
General Considerations

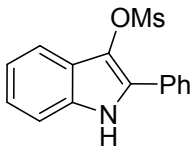
Unless specified, all reagents and starting materials were purchased from commercial sources and used as received. Solvents were purified following standard literature procedures. Analytical thin layer chromatography (TLC) was performed using pre-coated silica gel plate. Visualization was achieved by UV light (254 nm). Flash chromatography was performed using silica gel and a gradient solvent system (Ethyl acetate: Petrol ether as eluant). ^{1}H and ^{13}C NMR spectra were measured on 400 and 600 MHz spectrometers. Chemical shifts (ppm) were recorded with tetramethylsilane (TMS) as the internal reference standard. Multiplicities are given as: s (singlet), bs (broad singlet), d (doublet), t (triplet), dd (doublet of doublets) or m (multiplet). The number of protons (*n*) for a given resonance is indicated by *n*H and coupling constants are reported as a *J* value in Hz. Infrared spectra were recorded on a FTIR spectrometer. High resolution mass spectra (HRMS) were obtained on a LTQ Orbitrap LC/HRMS mass spectrometer. Starting materials **1** (2-alkynyl arylazides) were prepared by our reported methods.[1]

Representative Experimental Procedure for Preparation of 3



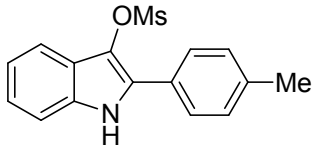
To a solution of 1-azido-2-(phenylethynyl)benzenes **1** (0.1 mmol) in dioxane (0.4 mL) was added $Pd(OAc)_2$ (5 mol%) and methanesulfonic acid **2a** or 4-methylbenzenesulfonic acid **2b** (0.3 mmol, 3 equiv.) in sequence at room temperature. The resulting solution was stirred and monitored by TLC analysis. On completion, the reaction mixture was directly subjected to purification by flash column chromatography on silica gel to give the desired product 2-phenyl-1*H*-indol-3-yl methanesulfonates **3**. (eluent: petrol ether: ethyl acetate = 8:1 to 4:1)

2-Phenyl-1*H*-indol-3-yl methanesulfonate (3a):



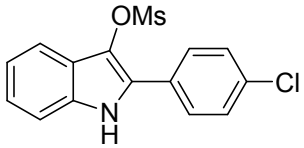
96% yield; brown solid; m.p. 92.7-95.3 ℃; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.87 (s, 3 H),7.18-7.24 (m, 2 H),7.32-7.40 (m, 2 H),7.46-7.49 (m, 2 H),7.72-7.76 (m, 3 H), 8.26 (br,1 H); ^{13}C NMR ($CDCl_3$,100 MHz) δ: 37.0, 111.4, 118.5, 121.2, 122.1, 123.6, 125.5, 126.8, 127.8, 128.6, 129.3, 129.8, 133.2; IR(KBr): 3379, 3035, 2950, 2853, 1344, 1183, 1158, 1056, 621, 558 cm^{-1}; HRMS (ESI) calcd. For $C_{15}H_{14}NO_3S$ $[M+H]^+$ 288.0694, found: 288.0695.

2-(*p*-Tolyl)-1*H*-indol-3-yl methanesulfonate (3b):



93% yield; brown solid; m.p. 143.3-145.5 ℃; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.42 (s, 3 H), 2.89 (s, 3 H), 7.20-7.28 (m, 2 H), 7.31 (d, J = 8 Hz,2 H), 7.35 (d, J = 7.6 Hz, 1 H), 7.65 (d, J =8 Hz, 2 H), 7.76 (d, J = 7.6 Hz,1 H), 8.26 (br,1 H); ^{13}C NMR ($CDCl_3$,100 MHz) δ: 21.4, 37.0, 111.3, 118.4, 121.2, 122.2, 123.4, 125.3, 126.7, 126.9, 128.0, 130.0, 133.1 ppm; IR(KBr): 3407, 3012, 2927, 2850, 1307, 1182, 1163, 1053, 646 cm^{-1}; HRMS (ESI) calcd. For $C_{16}H_{16}NO_3S$ $[M+H]^+$ 302.0851, found: 302.0853.

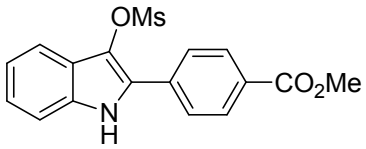
2-(4-Chlorophenyl)-1*H*-indol-3-yl methanesulfonate (3c):



82% yield; brown solid; m.p. 167.3-170.3 ℃; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.99 (s, 3 H),7.22-7.31 (m, 2 H),7.37 (d, J =7.6 Hz, 1 H), 7.48 (d, J = 8.8 Hz ,2 H), 7.71 (d, J = 8.8 Hz,2 H), 7.76 (d, J =8 Hz,1 H), 8.23 (br, 1 H);^{13}C NMR($CDCl_3$, 100 MHz) δ: 37.4, 111.5, 118.6, 121.5, 122.1, 123.9, 125.7, 126.8, 128.0, 128.3, 129.6, 133.4, 134.6 ppm; IR(KBr) : 3390, 2955, 2924, 2852,

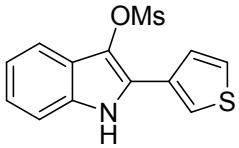
1349, 1183, 1166, 1090, 619, 540 cm^{-1}; HRMS (ESI) calcd. For $C_{15}H_{13}NO_3SCl$ $[M+H]^+$ 322.0305, found: 302.0306.

Methyl 4-(3-((methylsulfonyl)oxy)-1*H*-indol-2-yl)benzoate (3d):



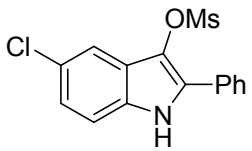
88% yield; yellow solid; m.p. 190.8-193.5 ℃; ^{1}H NMR($CDCl_3$, 400 MHz) δ: 2.99 (s,3 H), 3.98 (s, 3 H), 7.23-7.33 (m, 2 H), 7.41 (d, *J* = 8 Hz,1 H), 7.79 (d, *J* =8 Hz,1 H),7.87 (d, *J* = 8.4 Hz,2 H), 8.19 (d, *J* =8.4 Hz,2 H), 8.33 (br,1 H);^{13}C NMR($CDCl_3$, 100 MHz) δ: 37.4, 52.3, 111.5, 118.9, 121.6, 122.1, 124.4, 126.4, 126.6, 129.8, 130.5, 133.7, 134.1, 166.4 ppm; IR(KBr): 3344, 3020, 2924, 2854, 1343, 1185, 1144, 1052, 626, 520 cm^{-1}; HRMS (ESI) calcd. For $C_{17}H_{16}NO_5S$ $[M+H]^+$ 346.0749, found: 346.0751.

2-(Thiophen-3-yl)-1*H*-indol-3-yl methanesulfonate (3e):



83% yield; brown solid; m.p. 137.9-139.6 ℃; ^{1}H NMR ($CDCl_3$,400 MHz) δ: 3.04 (s, 3 H), 7.19-7.27 (m, 2 H), 7.34 (d, *J* = 7.6 Hz, 1 H), 7.47-7.49 (q, *J* = 2.8 Hz, 1 H), 7.53-7.55 (dd, J_1 = 5.2 Hz, J_2 = 1.6 Hz,1 H),7.68-7.69 (dd, J_1 = 2.8 Hz, J_2 = 1.2 Hz,1 H), 7.74 (d, *J* = 7.6 Hz, 1 H), 8.27 (br,1 H); ^{13}C NMR ($CDCl_3$,100 MHz) δ: 37.2, 111.4, 118.4, 121.3, 121.8, 122.4, 123.6, 124.5, 124.9, 125.7, 127.2, 130.2, 133.1; IR(KBr): 3396, 3007, 2927, 2854, 1331, 1171, 1154, 1054, 601, 523 cm^{-1}; HRMS (ESI) calcd. For $C_{13}H_{12}NO_3S_2$ $[M+H]^+$ 294.0259, found: 294.0260.

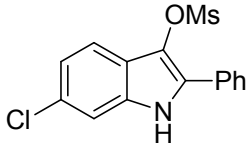
5-Chloro-2-phenyl-1*H*-indol-3-yl methanesulfonate (3f):



94% yield; brown solid; m.p. 129.0-130.7 ℃; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.92 (s, 3 H), 7.16-7.29 (m, 2 H), 7.41 (t, *J* = 7.2 Hz, 1 H), 7.49 (t, *J* = 7.2 Hz, 2 H), 7.70-7.73 (m, 3 H), 8.38 (br,1 H);

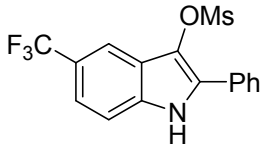
^{13}C NMR ($CDCl_3$, 100 MHz) δ: 37.2, 112.6, 117.9, 123.1, 124.0, 124.6, 126.8, 127.0, 128.9, 129.3, 129.4, 131.6; IR(KBr): 3405, 2959, 2931, 2853, 1359, 1178, 1153, 1079, 694, 523 cm^{-1}; HRMS (ESI) calcd. For $C_{15}H_{13}NO_3SCl$ $[M+H]^+$ 322.0305, found: 322.0307.

6-Chloro-2-phenyl-1*H*-indol-3-yl methanesulfonate (3g):



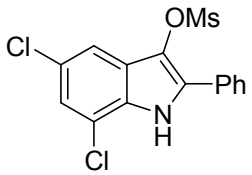
94% yield; brown solid; m.p. 144.0-145.5 ℃; ^1H NMR ($CDCl_3$, 400 MHz) δ: 2.90 (s, 3 H),7.16-7.19 (dd, J_1 = 8.4 Hz, J_2 = 1.6 Hz, 1 H), 7.32 (d, J = 1.6 Hz,1 H), 7.41 (t, J = 7.2 Hz, 1 H), 7.50 (t, J = 7.2 Hz, 2 H),7.67 (d, J = 8.8 Hz, 1 H), 7.72 (d, J = 7.6 Hz, 2 H), 8.33 (br,1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 37.1, 111.3, 119.6, 120.8, 122.1, 125.4, 126.8, 128.4, 128.9, 129.4, 129.5, 133.5; IR(KBr): 3410, 3035, 2923, 2852, 1364, 1183, 1159, 1063, 699, 528 cm^{-1}; HRMS (ESI) calcd. For $C_{15}H_{13}NO_3SCl$ $[M+H]^+$ 322.0305, found: 322.0306.

2-Phenyl-5-(trifluoromethyl)-1*H*-indol-3-yl methanesulfonate (3h):



93% yield; yellow solid; m.p. 167.6-168.4 ℃; ^1H NMR ($CDCl_3$, 400 MHz) δ: 2.95 (s, 3 H), 7.42-7.54 (m, 5 H),7.76 (d, J = 7.2 Hz, 2 H), 8.07 (s, 1 H), 8.52 (br, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 37.3, 111.8, 116.6 (q, J = 4.5 Hz), 120.3 (q, J = 3.5 Hz), 121.7, 123.5 (q, J = 270 Hz), 123.7 (q, J = 32 Hz), 125.7, 126.9, 129.1, 129.2, 129.4, 129.7, 134.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -60.70; IR(KBr): 3420, 3033, 2946, 2852, 1342, 1164, 1108, 1066, 697, 522 cm^{-1}; HRMS (ESI) calcd. For $C_{16}H_{13}NO_3SF_3$ $[M+H]^+$ 356.0568, found: 356.0569.

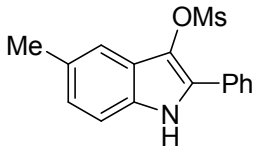
5,7-Dichloro-2-phenyl-1*H*-indol-3-yl methanesulfonate (3i):



90% yield; brown solid; m.p. 175.5- 178.3 ℃; ^1H NMR ($CDCl_3$, 400 MHz) δ: 2.92 (s, 3 H), 7.29

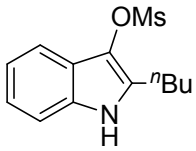
(d, J = 8.0 Hz, 1 H), 7.45-7.49 (m, 1 H), 7.54-7.58 (m, 2 H),7.68 (d, J = 1.2 Hz, 1 H), 7.78-7.80 (m, 2 H), 8.40 (br, 1 H);^{13}C NMR ($CDCl_3$, 100 MHz) δ: 37.3, 117.1, 117.4, 123.1, 124.1, 125.1, 127.1, 128.8, 128.9, 129.5, 130.2; IR(KBr): 3322, 3016, 2923, 2853, 1328, 1195, 1162, 1095, 627, 525 cm^{-1}; HRMS (ESI) calcd. For $C_{15}H_{12}NO_3SCl_2$ $[M+H]^+$ 355.9915, found: 355.9917.

5-Methyl-2-phenyl-1*H*-indol-3-yl methanesulfonate (3j):



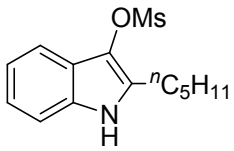
95% yield; brown solid; m.p. 119.3- 121.4 ℃;^{1}H NMR($CDCl_3$, 400 MHz) δ: 2.49 (s, 3 H), 2.88 (s, 3 H), 7.08-7.10 (dd, J_1 = 8.4 Hz, J_2 = 1.2 Hz, 1 H), 7.25 (d, J = 8.4 Hz, 1 H), 7.38-7.42 (m, 1 H), 7.50 (t, J = 7.6 Hz, 2 H), 7.55 (s, 1 H), 7.75 (d, J = 7.2 Hz, 2 H), 8.18 (br,1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 21.6, 36.9, 111.1, 117.9, 122.4, 125.5, 126.8, 127.8, 128.5, 129.3, 130.0, 130.7, 131.7 ; IR(KBr): 3384, 3018, 2923, 2852, 1320, 1197, 1174, 1051, 656, 523 cm^{-1}; HRMS (ESI) calcd. For $C_{16}H_{16}NO_3S$ $[M+H]^+$ 302.0851, found: 302.0853.

2-Butyl-1*H*-indol-3-yl methanesulfonate (3k):



96% yield; green solid; m.p. 45.4-47.2 ℃; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 0.97 (t, J =7.2 Hz, 3 H), 1.39-1.48 (m, 2 H), 1.67-1.75 (m, 2 H), 2.86 (t, J = 7.6 Hz, 2 H), 3.21 (s, 3 H), 7.15-7.22 (m, 2 H), 7.29-7.32 (m, 1 H), 7.56-7.58 (m, 1 H), 7.97 (br,1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 13.8, 22.5, 24.7, 30.8, 37.4, 111.2, 116.7, 120.7, 121.1, 122.2, 124.7, 131.4, 132.5; IR(KBr): 3392, 3028, 2925, 2856, 1348, 1177, 1079, 697, 522 cm^{-1}; HRMS (ESI) calcd. For $C_{13}H_{18}NO_3S$ $[M+H]^+$ 268.1007, found: 268.1008.

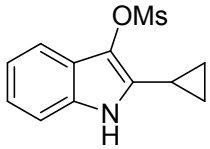
2-Pentyl-1*H*-indol-3-yl methanesulfonate (3l):



83% yield; green liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 0.93 (t, J = 6.8 Hz, 3 H), 1.37-1.41 (m, 4 H), 1.69-1.76 (m, 2 H), 2.85 (t, J = 7.6 Hz, 2 H), 3.21 (s, 3 H), 7.15-7.22 (m, 2 H), 7.29-7.32 (m, 1

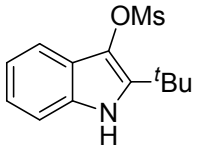
H), 7.56-7.58 (m, 1 H), 7.98 (br,1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 13.9, 22.4, 24.9, 28.3, 31.5, 37.4, 111.2, 116.7, 120.7, 121.1, 122.2, 124.7, 131.5, 132.5; IR(neat): 3398, 3033, 2927, 2857, 1343, 1178, 1153, 1087, 741, 520 cm^{-1}; HRMS (ESI) calcd. For $C_{14}H_{20}NO_3S$ $[M+H]^+$ 282.1164, found: 282.1165.

2-Cyclopropyl-1*H*-indol-3-yl methanesulfonate (3m):



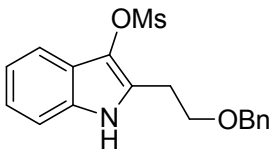
83% yield; black solid; m.p. 91.8-93.2 ℃; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 0.87-0.89 (q, J = 1.6 Hz, 2 H), 1.06-1.11 (m, 2 H), 2.15-2.22 (m, 1 H), 3.24 (s, 3 H), 7.14-7.21 (m, 2 H), 7.25-7.28 (m, 1 H), 7.56-7.58 (m, 1H), 7.75 (br,1 H); ^{13}C NMR($CDCl_3$, 100 MHz) δ: 6.5, 6.8, 37.3, 111.1, 116.7, 120.8, 121.5, 122.4, 126.1, 131.4, 132; IR(KBr): 3389, 3025, 2928, 2853, 1319, 1176, 1127, 1059, 577, 522 cm^{-1}; HRMS (ESI) calcd. For $C_{12}H_{14}NO_3S$ $[M+H]^+$ 252.0694, found: 252.0695.

2-(Tert-butyl)-1*H*-indol-3-yl methanesulfonate (3n):



96% yield; yellow solid; m.p. 132.1-135.6 ℃; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.52 (s, 9 H), 3.33 (s, 3 H), 7.14-7.21 (m, 2 H), 7.30 (d, J = 7.2 Hz, 1 H), 7.67 (d, J = 7.2 Hz, 1 H), 7.96 (br,1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 29.9, 32.4, 38.7, 111.0, 117.8, 120.5, 121.7, 122.3, 123.2, 131.7, 136.9; IR(KBr): 3384, 3024, 2957, 2866, 1335, 1172, 1122, 1088, 609, 515 cm^{-1}; HRMS (ESI) calcd. For $C_{13}H_{18}NO_3S$ $[M+H]^+$ 268.1007, found: 268.1009.

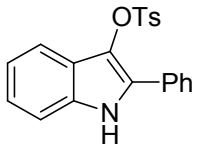
2-(2-(Benzyloxy)ethyl)-1*H*-indol-3-yl methanesulfonate (3o):



92% yield; brown liquid; ^{1}HNMR ($CDCl_3$, 400 MHz) δ: 3.16 (t, J = 5.6 Hz, 2 H), 3.19 (s, 3 H), 3.86 (s, 2 H), 4.63 (s, 2 H), 7.16-7.23 (m, 2 H), 7.37-7.45 (m, 6 H), 7.56 (d, J = 7.2 Hz, 1 H), 8.79

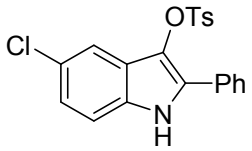
(br,1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 25.2, 37.3, 69.2, 73.5, 111.4, 116.6, 120.4, 120.5, 122.3, 124.4, 127.9, 128.1, 128.7, 130.0, 132.6, 137.8; IR(KBr): 3403, 3029, 2927, 2853, 2360, 1363, 1181, 1130, 1027, 523 cm^{-1}; HRMS (ESI) calcd. For $C_{18}H_{20}NO_4S$ $[M+H]^+$ 346.1113, found: 346.1115.

2-Phenyl-1*H*-indol-3-yl 4-methylbenzenesulfonate (3q):



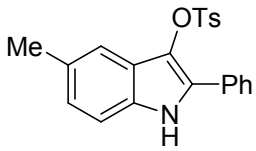
97% yield; brown solid; m.p.146.8-148.2 ℃; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.28 (s, 3 H), 6.91 (d, J = 8 Hz, 2 H), 7.15-7.19 (m, 1 H), 7.21-7.27 (m, 4 H), 7.32 (d, J = 8 Hz, 1 H), 7.34-7.37 (m, 2 H), 7.42 (d, J =8.4 Hz, 2 H), 7.66 (d, J = 8 Hz, 1 H), 8.06 (br,1 H); ^{13}C NMR($CDCl_3$, 100 MHz) δ: 21.5, 111.2, 118.9, 120.9, 122.4, 123.3, 125.5, 126.9, 127.8, 128.3, 128.4, 128.6, 129.0, 129.7, 131.2, 133.2, 144.9; IR(KBr): 3373,3053,2922,2852,1358,1191,1168,1055,652,547 cm^{-1}; HRMS (ESI) calcd. For $C_{21}H_{18}NO_3S$ $[M+H]^+$ 364.1007, found: 364.1008.

5-Chloro-2-phenyl-1*H*-indol-3-yl 4-methylbenzenesulfonate(3r):



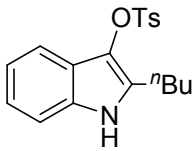
93% yield; brown solid; m.p. 140.8 -142.5 ℃; ^{1}H NMR($CDCl_3$, 400 MHz) δ: 2.32 (s, 3 H), 6.98 (d, J = 8 Hz, 2 H), 7.12-7.15 (dd, J_1 = 8.8 Hz, J_2 = 2 Hz, 1 H),7.21 (d, J = 8.4 Hz, 1 H), 7.26-7.29 (m, 3 H), 7.37-7.40 (m, 3 H), 7.45 (d, J = 8.4 Hz, 2 H), 8.15 (br, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 21.5, 112.3, 118.1, 123.2, 123.6, 124.8, 126.7, 126.8, 128.2, 128.5, 128.6, 129.2, 129.3, 129.9, 131.3, 131.4, 145.4; IR(KBr): 3418, 2952, 2921, 2852, 1319, 1191, 1178, 1090, 648, 546 cm^{-1}; HRMS (ESI) calcd. For $C_{21}H_{17}NO_3SCl$ $[M+H]^+$ 398.0618, found: 398.0618.

5-Methyl-2-phenyl-1*H*-indol-3-yl 4-methylbenzenesulfonate (3s):



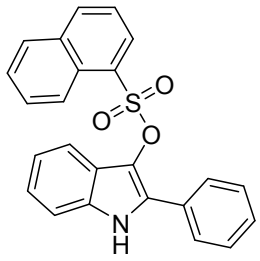
89% yield; yellow solid; m.p. 175.5- 176.6 ℃; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.30 (s, 3 H), 2.44 (s, 3 H), 6.94 (d, J = 8 Hz, 2 H), 7.04- 7.07 (dd, J_1 = 8.4 Hz, J_2 = 1.2 Hz, 1 H), 7.22 (d, J = 8.4 Hz, 1 H), 7.27 (d, J = 7.2 Hz, 3 H), 7.35-7.39 (m, 3 H),7.46 (d, J =8 Hz, 2 H), 7.86 (br,1 H); ^{13}C NMR ($CDCl_3$,100 MHz) δ : 21.4, 21.5, 110.8, 118.3, 122.6, 125.1, 125.3, 126.8, 127.7, 128.3, 128.4, 128.7, 128.9, 129.9, 130.3, 131.5, 131.6, 144.8; IR(KBr): 3407, 2952, 2921, 2852, 1305, 1186, 1131, 1054, 690, 558 cm^{-1}; HRMS (ESI) calcd. For $C_{22}H_{20}NO_3S$ $[M+H]^+$ 378.1164, found: 378.1166.

2-Butyl-1*H*-indol-3-yl 4-methylbenzenesulfonate(3t):



70% yield; yellow solid; m.p. 71.8-73.2 ℃; ^{1}H NMR ($CDCl_3$,400 MHz) δ: 0.91(t, J = 7.2 Hz, 3 H),1.31-1.37 (m, 2 H), 1.45-1.53 (m, 2 H),2.47 (s, 3 H), 2.49 (t, J = 7.6 Hz, 2 H), 6.96-7.00 (m, 1 H), 7.11 (t, J = 7.2 Hz, 2 H), 7.23 (d, J = 8 Hz, 1 H), 7.30 (d, J = 8 Hz, 2 H), 7.78 (d, J = 8 Hz, 2 H), 7.79 (br,1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 13.7, 21.7, 22.5, 24.3, 30.6, 110.7, 117.3, 120.2, 121.4, 121.9, 125.3, 128.8, 129.7, 131.0, 132.4, 132.8, 145.2; IR(KBr): 3403, 2954, 2926, 2857, 1356, 1188, 1134, 1090, 682, 556 cm^{-1}; HRMS (ESI) calcd. For $C_{19}H_{22}NO_3S$ $[M+H]^+$ 344.1320, found: 344.1321.

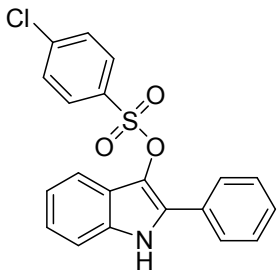
2-Phenyl-1*H*-indol-3-yl naphthalene-1-sulfonate (3u):



87%; brown solid; m.p. 133.5-136.3 ℃; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 6.95-6.98 (m, 2 H), 7.02-7.05 (m, 1 H), 7.08-7.12 (m,1 H), 7.19-7.24 (m, 3 H), 7.28 (d, J = 3.2 Hz, 1 H), 7.30 (d, J = 3.6 Hz, 1 H), 7.40 (d, J = 7.6 Hz, 1 H), 7.58-7.66 (m, 2 H), 7.84 (d, J = 7.2 Hz, 1 H), 7.93 (d, J = 8.4 Hz, 1 H), 7.98 (dd, J_1 = 0.8 Hz, J_2 = 7.6 Hz, 1 H), 8.03 (br, 1 H), 8.80 (d, J = 8.4 Hz, 1 H); ^{13}C NMR

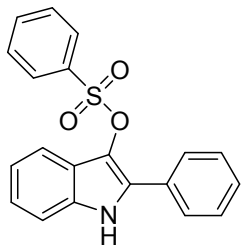
($CDCl_3$,100 MHz) δ: 111.2, 118.4, 120.9, 122.6, 123.2, 123.7, 125.3, 125.6, 126.6, 126.9, 127.7, 128.2, 128.5, 128.6, 128.7, 128.9, 129.1, 131.0, 131.1, 133.0, 133.9, 135.6; IR(KBr): 3397, 2955, 2922, 2853, 1457, 1365, 1182, 1137, 1028, 754, 504 cm^{-1}; HRMS (ESI) calcd. For $C_{24}H_{18}NO_3S$ $[M+H]^+$ 400.1007, found: 400.1009.

2-Phenyl-1*H*-indol-3-yl 4-chlorobenzenesulfonate (3v):



89%; brown solid; m.p. 160.7-162.7 ℃; ^1H NMR ($CDCl_3$, 400 MHz) *δ*: 6.99 (d, *J* = 8.8 Hz, 2 H), 7.16-7.31 (m, 8 H), 7.36 (d, *J* = 8.8 Hz, 2 H), 7.69 (d, *J* = 7.6 Hz, 1 H), 8.08 (br, 1 H); ^{13}C NMR ($CDCl_3$,100 MHz) *δ*: 111.3, 118.8, 121.2, 122.2, 123.5, 125.3, 127.0, 128.2, 128.4, 128.5, 128.6, 129.5, 129.9, 132.3, 133.2, 140.8；IR(KBr): 3390, 2952, 2923, 2852, 1371, 1188, 1160, 1089, 739, 621 cm^{-1}; HRMS (ESI) calcd. For $C_{20}H_{15}ClNO_3S$ $[M+H]^+$ 384.0461, found: 384.0462.

2-Phenyl-1*H*-indol-3-yl benzenesulfonate (3w):

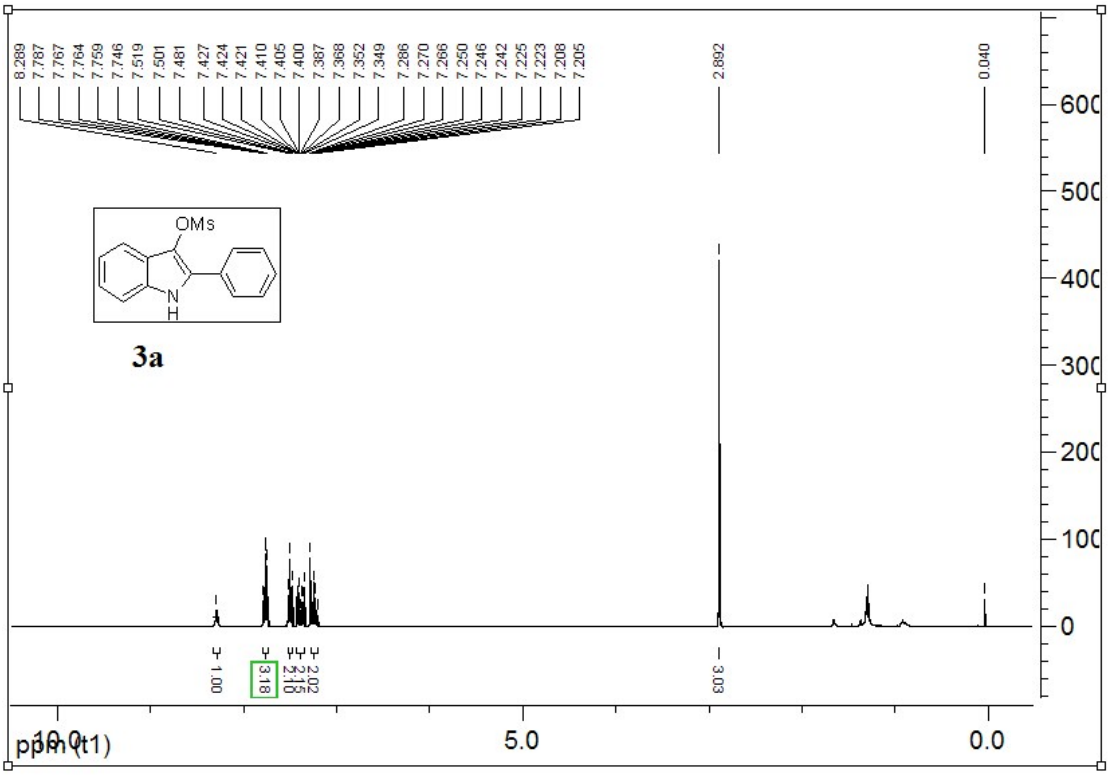


95%; brown solid; m.p. 121.5-122.7 ℃; ^1H NMR ($CDCl_3$, 400 MHz) *δ*: 7.13-7.18 (m, 3 H), 7.21-7.28 (m, 4 H), 7.31 (d, *J* = 8 Hz, 1H), 7.39-7.42 (m, 3 H), 7.57-7.60 (m, 3 H), 8.12 (br, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) *δ*: 111.3, 118.7, 121.0, 122.2, 123.3, 125.4, 126.8, 128.0, 128.4, 128.6, 128.7, 129.6, 133.1, 134.0, 134.5; IR(KBr): 3396, 2958, 2925, 2853, 1362, 1186, 1118, 1056, 694, 575 cm^{-1}; HRMS (ESI) calcd. For $C_{20}H_{16}NO_3S$ $[M+H]^+$ 350.0851, found: 350.0852.

Reference

1. (a) X. Zhang, X. Sun, H. Fan, C. Lyu, P. Li, H. Zhang and W. Rao, *RSC Adv.*, 2016, **6**,

56319; (b) X. Zhang, X. Sun, H. Fan, P. Li, C. Lyu and W. Rao, *Eur. J. Org. Chem.,* 2016, **25**, 4265.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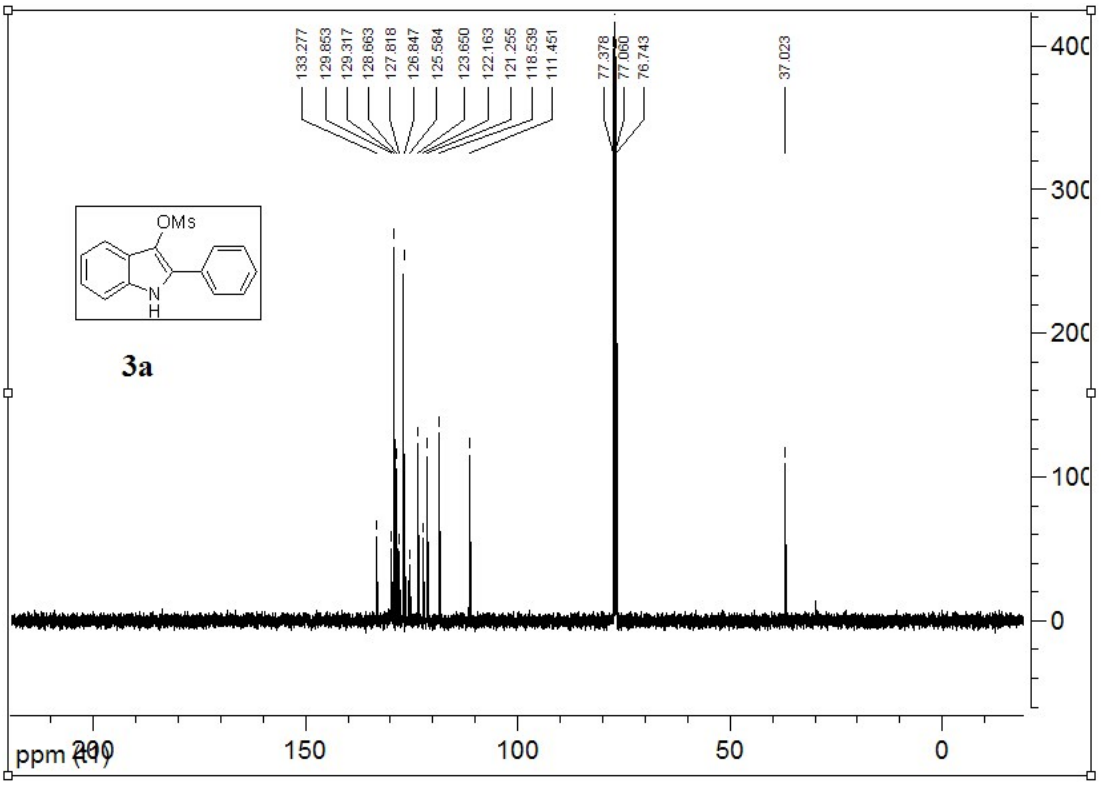
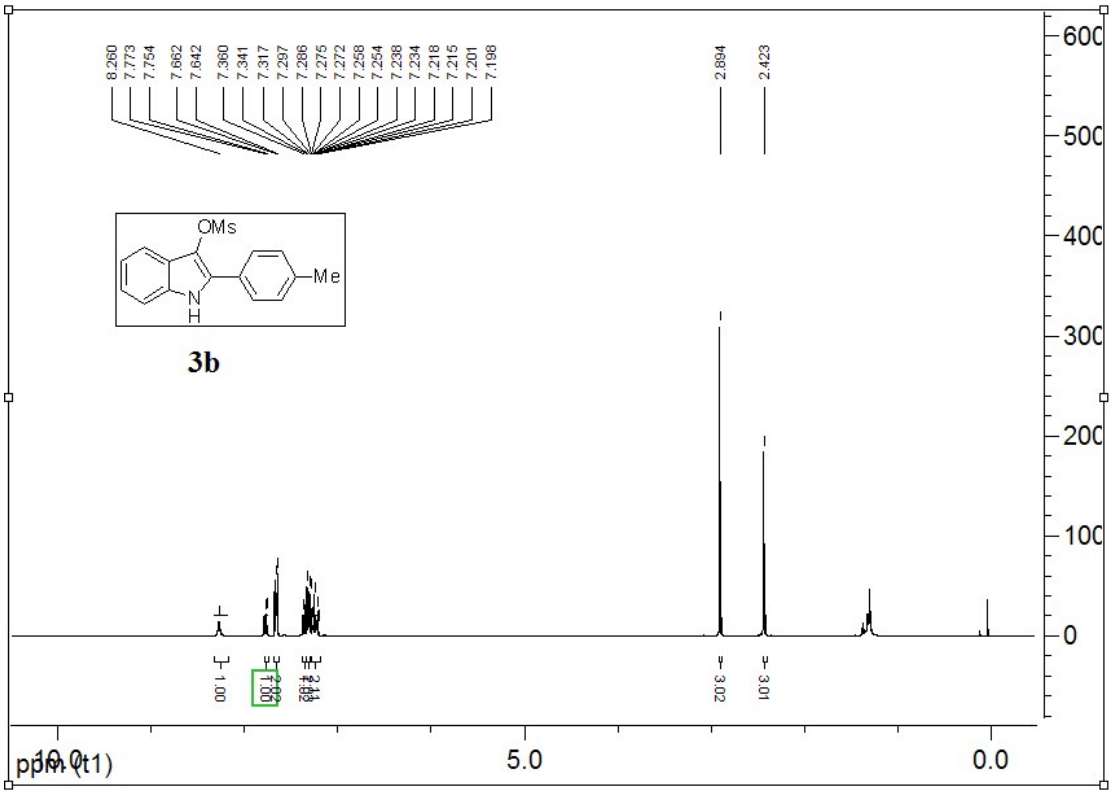
7.767
7.764
7.759
7.746
7.519
7.501
7.481
7.427
7.424
7.421
7.410
7.405
7.400
7.387
7.368
7.352
7.349
7.286
7.270
7.266
7.250
7.246
7.242
7.225
7.223
7.208
7.205
2.892
0.040
OMs
N
H
3a
1.00
3.18
2.15
2.10
2.22
3.03
600
500
400
300
200
100
0
10.0
5.0
0.0
ppm (t1)


133.277
129.853
129.317
128.663
127.818
126.847
125.584
123.650
122.163
121.255
118.539
111.451
77.378
77.060
76.743
37.023
OMs
N
H
3a
400
300
200
100
0
200
150
100
50
0
ppm (t1)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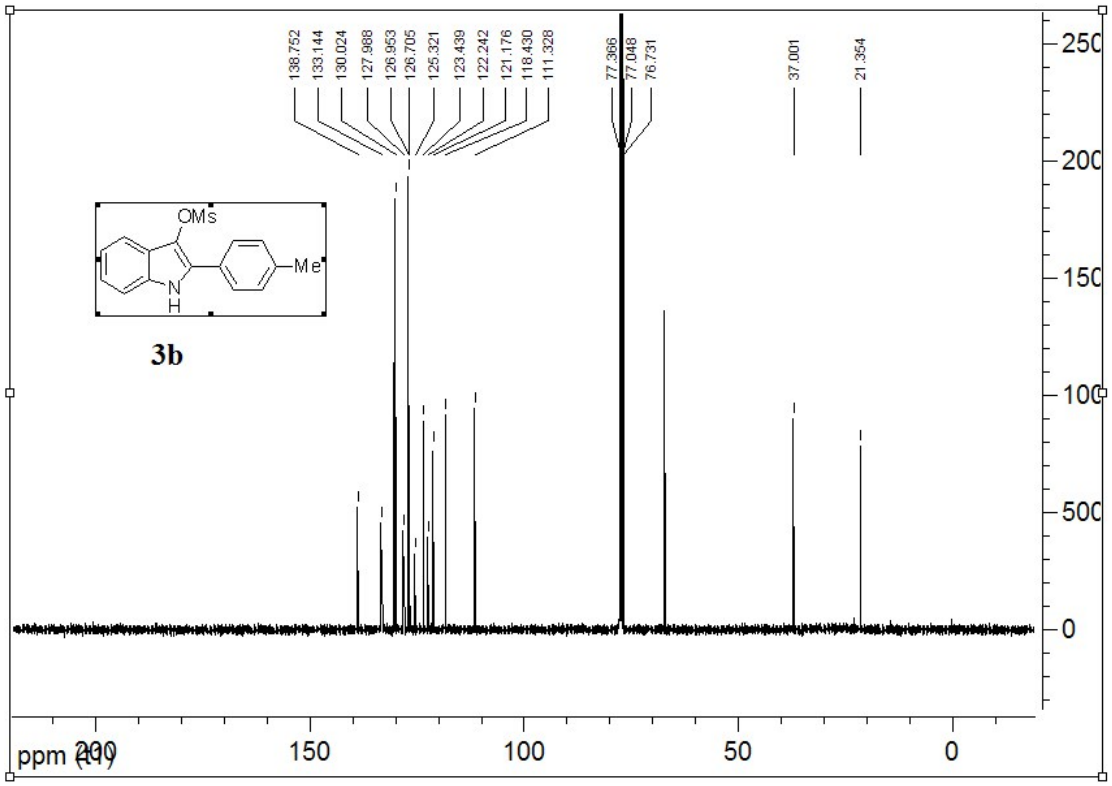
7.773
7.754
7.662
7.642
7.360
7.341
7.317
7.297
7.286
7.275
7.272
7.258
7.254
7.238
7.234
7.218
7.215
7.201
7.198
2.894
2.423
OMs
Me
N
H
3b
1.00
3.02
3.01
10.0
5.0
0.0
ppm (t1)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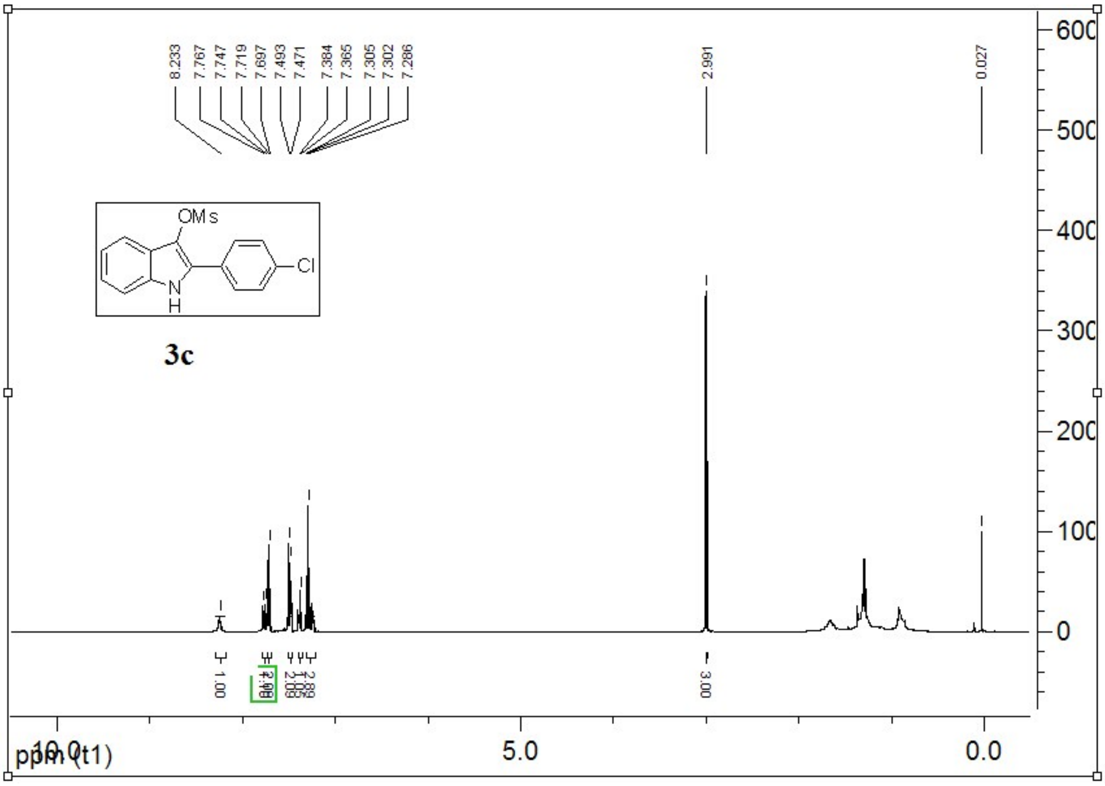
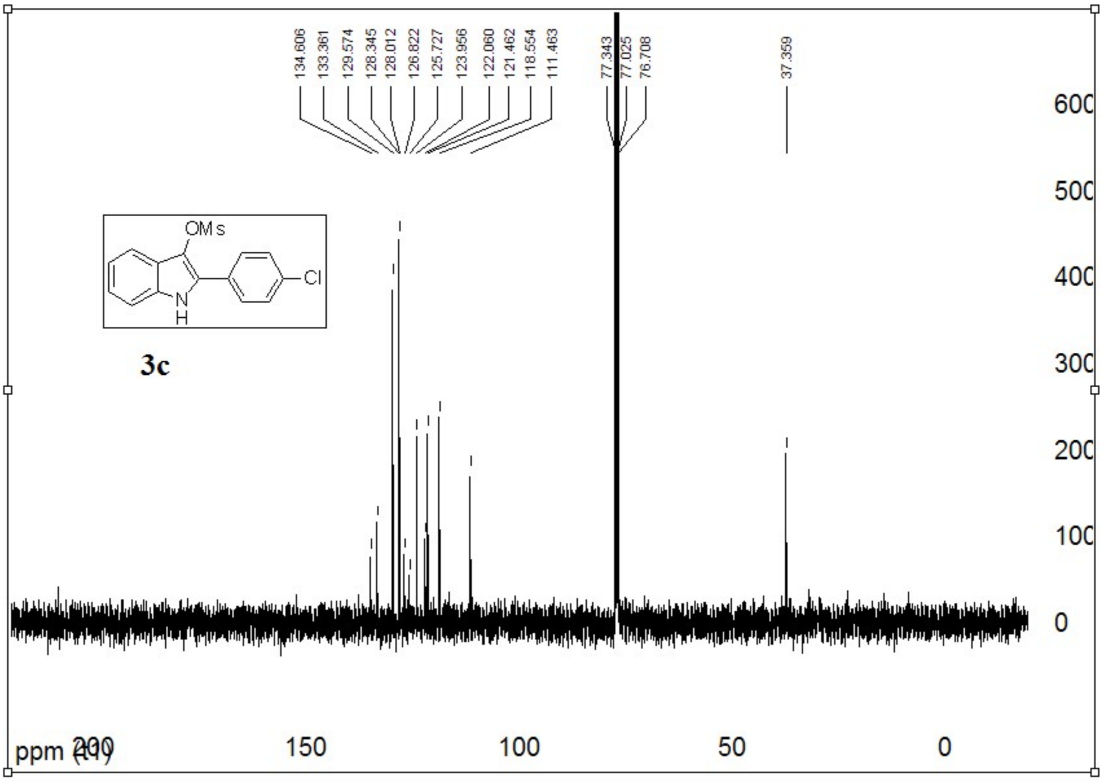
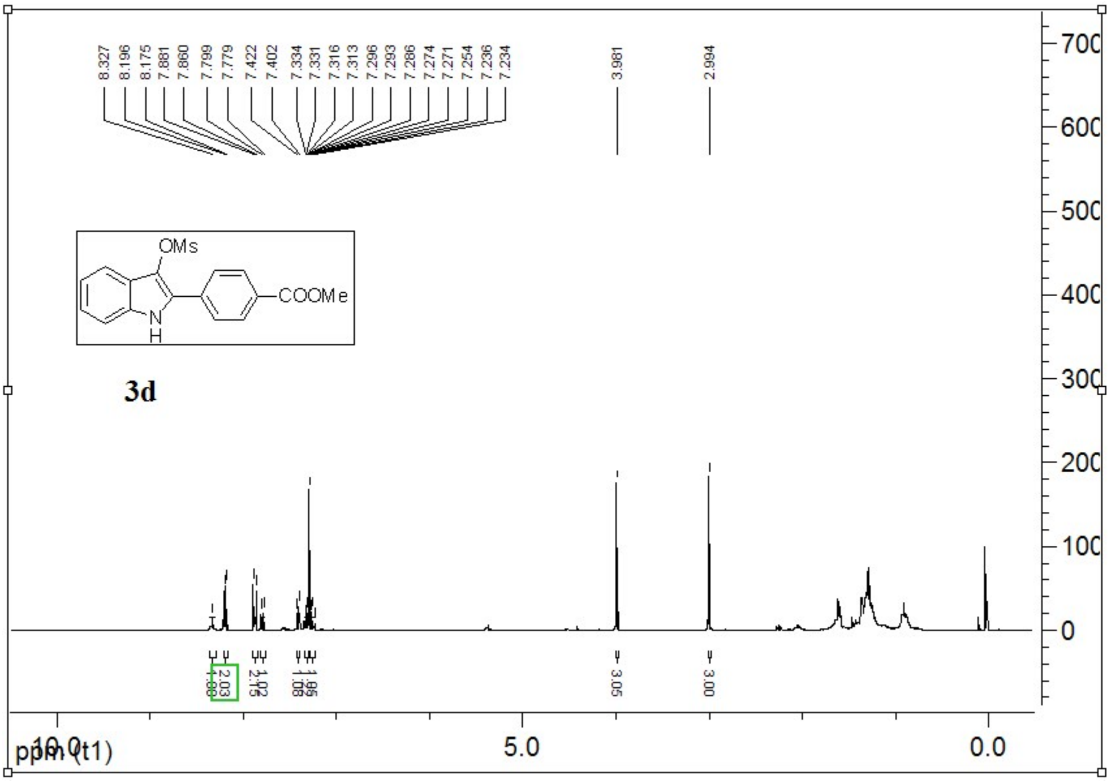
130.024
127.968
126.953
126.705
125.321
123.439
122.242
121.176
118.430
111.328
77.366
77.048
76.731
37.001
21.354
OMs
Me
N
H
3b
200
150
100
50
0
ppm (t1)


8.233
7.767
7.747
7.719
7.697
7.493
7.471
7.384
7.365
7.305
7.302
7.286
2.991
0.027
OMs
Cl
N
H
1.00
2.09
1.05
2.89
3.00
600
500
400
300
200
100
0
10.0
5.0
0.0
ppm (t1)

3c


134.606
133.361
129.574
128.345
128.012
126.822
125.727
123.956
122.060
121.462
118.554
111.463
77.343
77.025
76.708
37.359
OMs
Cl
N
H
600
500
400
300
200
100
0
200
150
100
50
0
ppm (t1)

3c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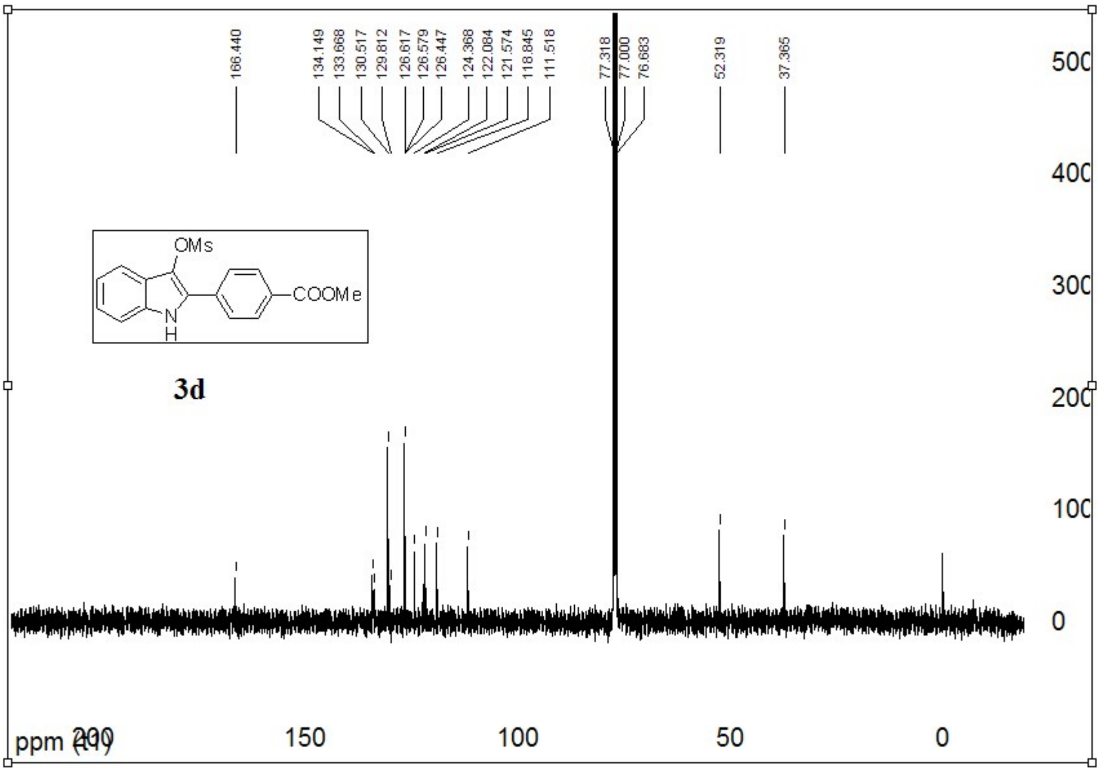
8.196
8.175
7.881
7.860
7.799
7.779
7.422
7.402
7.334
7.331
7.316
7.313
7.296
7.293
7.286
7.274
7.271
7.254
7.236
7.234
3.981
2.994
OMs
COOMe
N
H
3d
2.03
2.15
1.02
1.06
1.95
3.05
3.00
ppm (t1)
10.0
5.0
0.0
700
600
500
400
300
200
100
0


166.440
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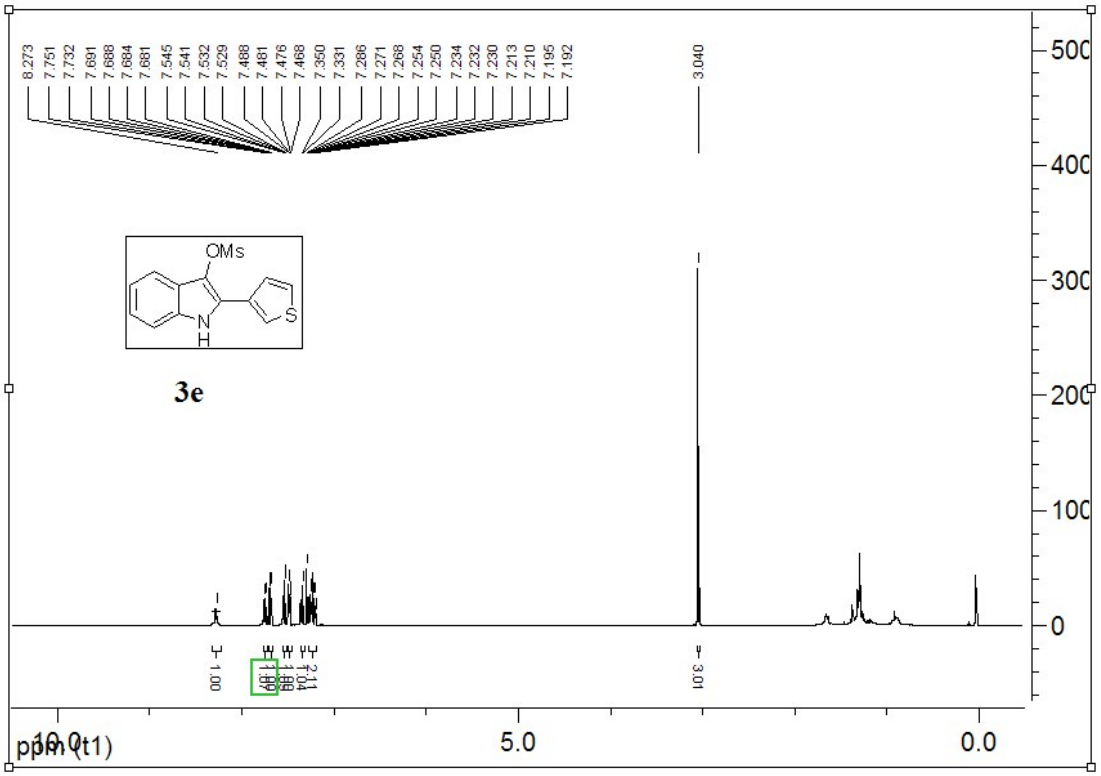
133.668
130.517
129.812
126.617
126.579
126.447
124.368
122.084
121.574
118.845
111.518
77.318
77.000
76.683
52.319
37.365
OMs
COOMe
N
H
3d
ppm (t1)
200
150
100
50
0
500
400
300
200
100
0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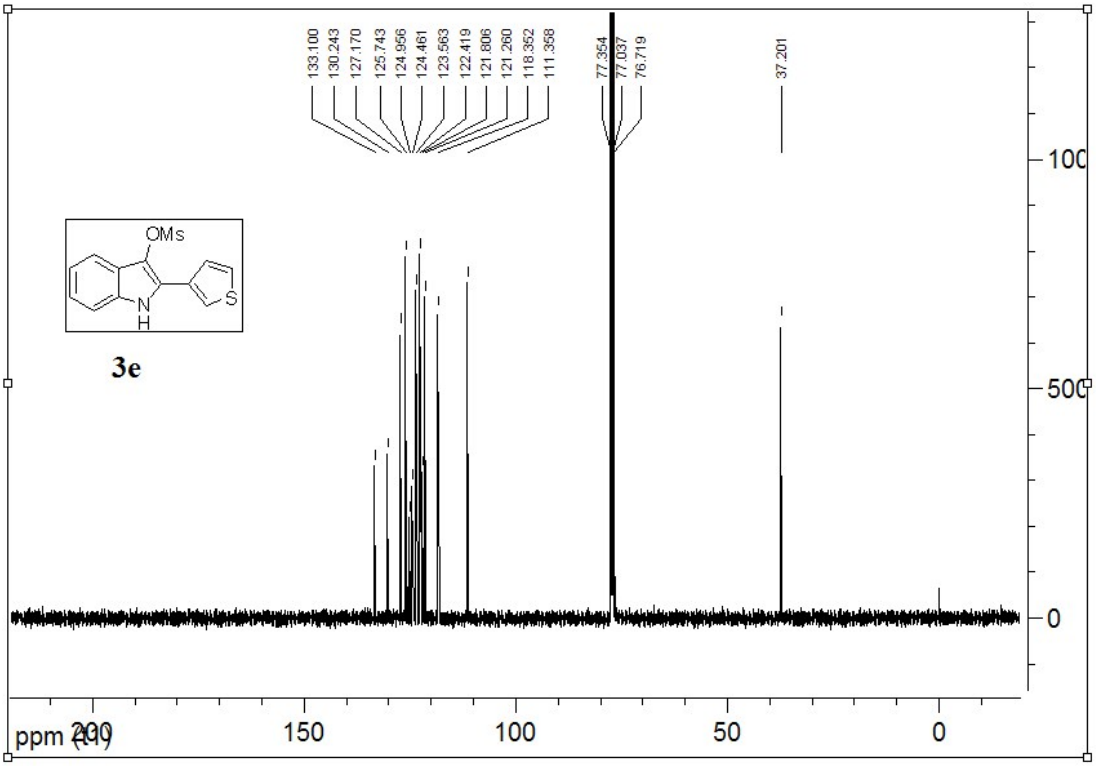
7.751
7.732
7.691
7.688
7.684
7.681
7.545
7.541
7.532
7.529
7.488
7.481
7.476
7.468
7.350
7.331
7.286
7.271
7.268
7.254
7.250
7.234
7.232
7.230
7.213
7.210
7.195
7.192
3.040
OMs
N
H
S
3e
1.00
2.11
3.01
ppm (t1)
10.0
5.0
0.0


133.100
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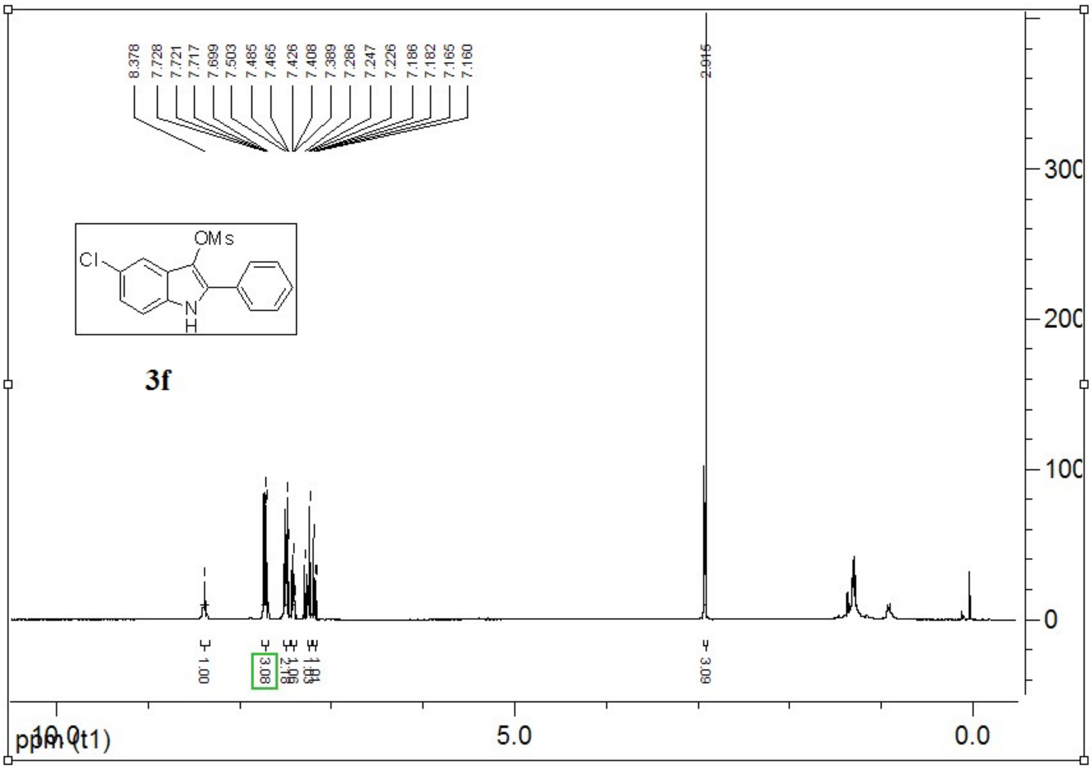
127.170
125.743
124.956
124.461
123.563
122.419
121.806
121.260
118.352
111.358
77.354
77.037
76.719
37.201
OMs
N
H
S
3e
ppm (t1)
200
150
100
50
0



8.378
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7.721
7.717
7.699
7.503
7.485
7.465
7.426
7.408
7.389
7.286
7.247
7.226
7.186
7.182
7.165
7.160
2.915
OMs
Cl
N
H
3f
1.00
3.08
3.09
ppm (t1)
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5.0
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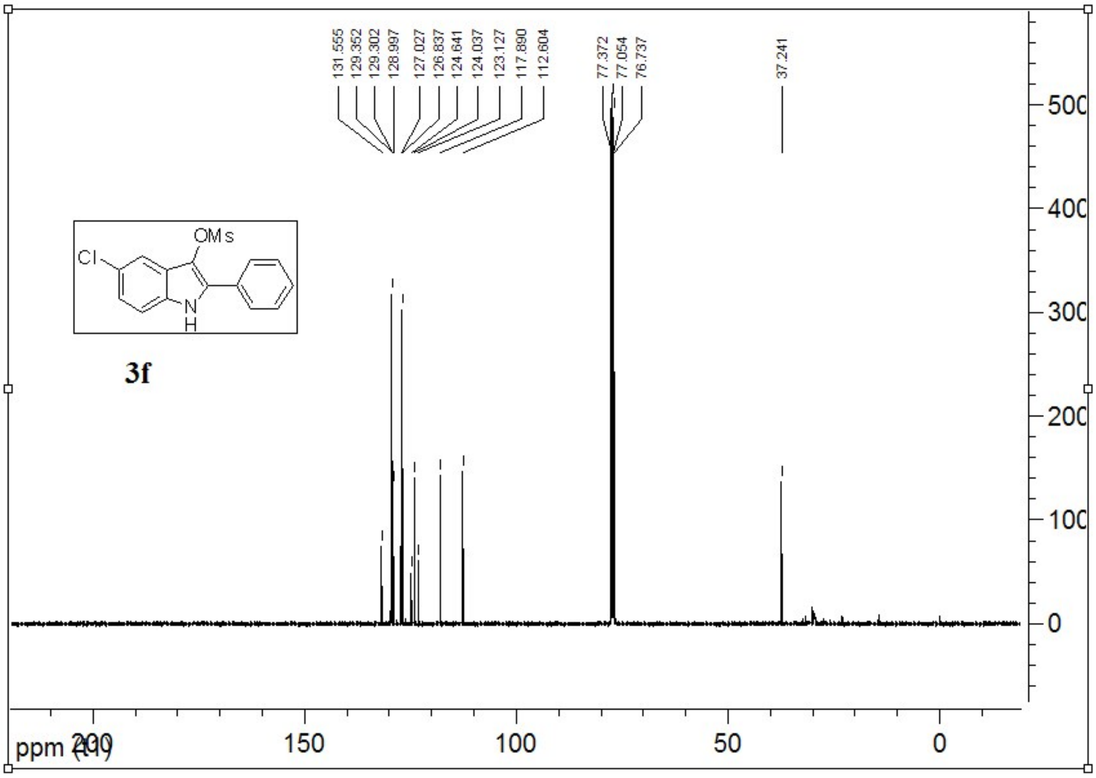
300
200
100
0



131.555
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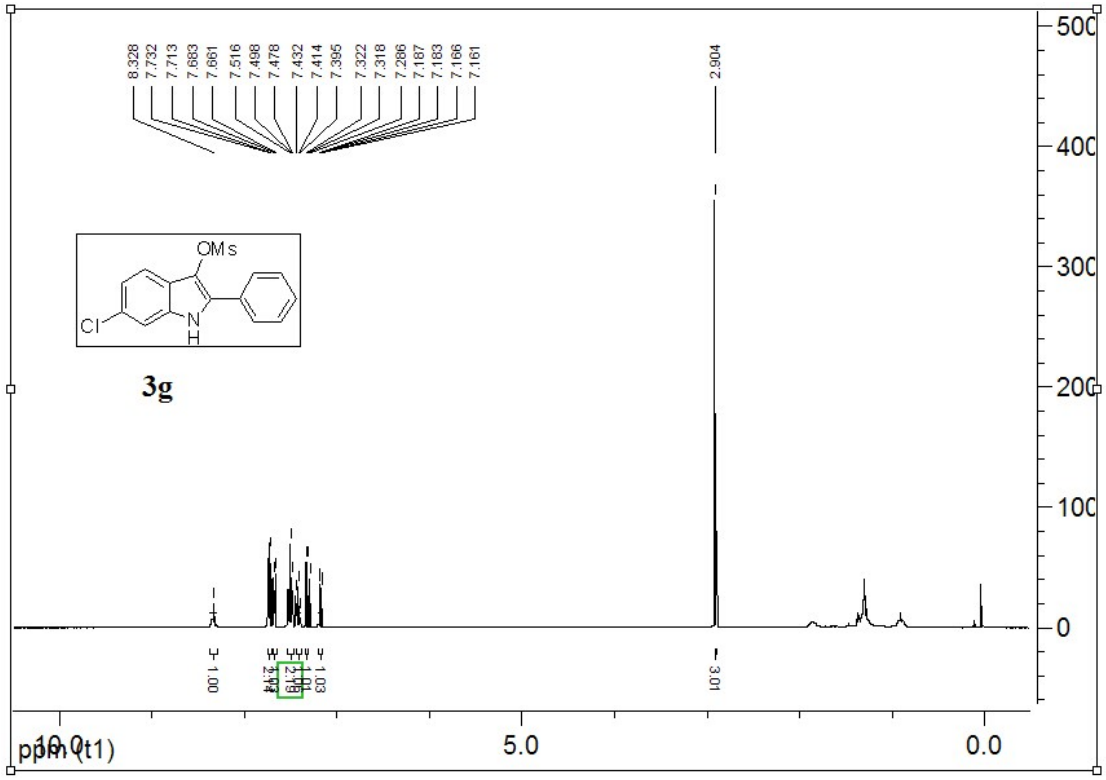
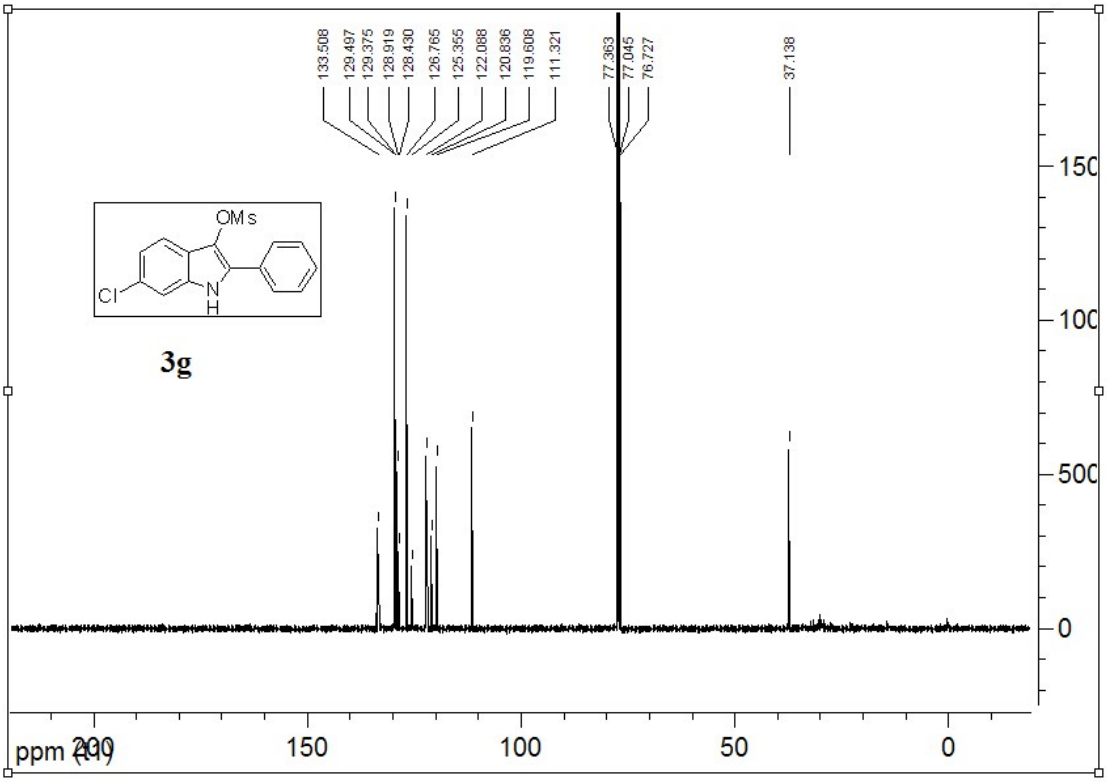
126.837
124.641
124.037
123.127
117.890
112.604
77.372
77.054
76.737
37.241
OMs
Cl
N
H
3f
ppm (t1)
200
150
100
50
0
500
400
300
200
100
0


8.328
7.732
7.713
7.683
7.661
7.516
7.498
7.478
7.432
7.414
7.395
7.322
7.318
7.286
7.187
7.183
7.166
7.161
2.904
OMs
Cl
N
H
3g
1.00
2.19
1.03
3.01
10.0
5.0
0.0
ppm (t1)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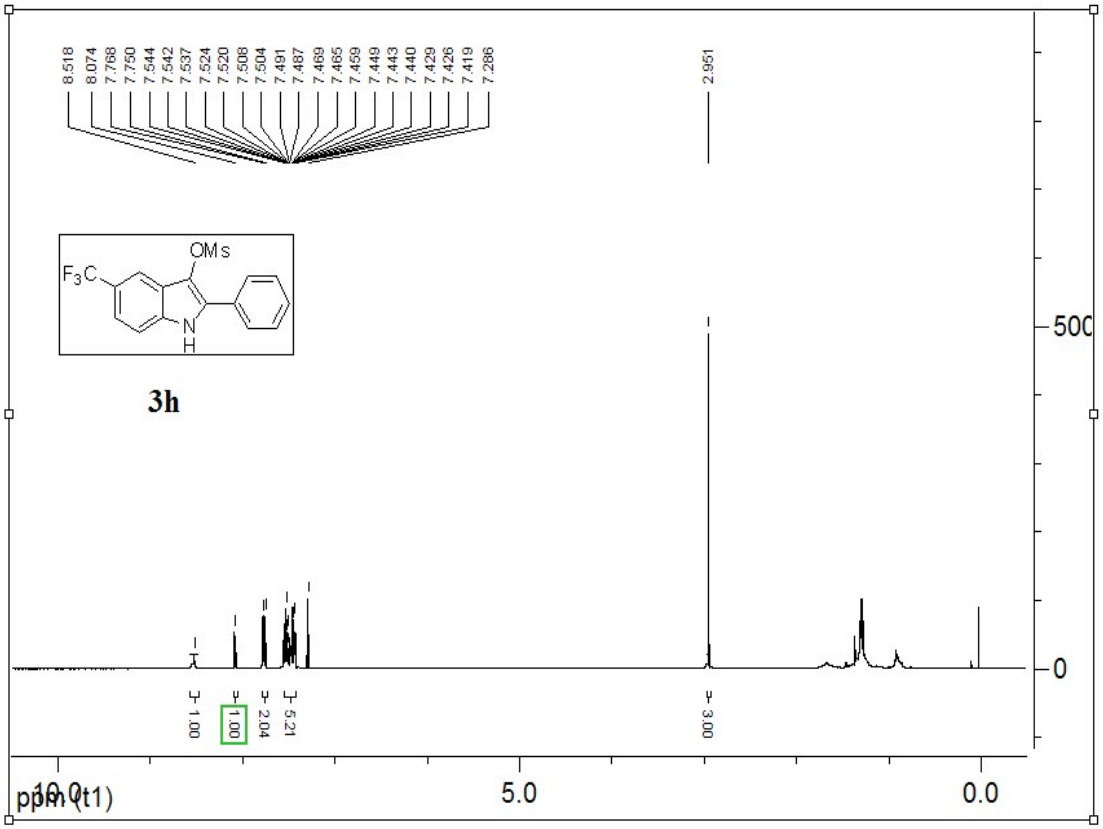
129.375
128.919
128.430
126.765
125.355
122.088
120.836
119.608
111.321
77.363
77.045
76.727
37.138
OMs
Cl
N
H
3g
200
150
100
50
0
ppm (t1)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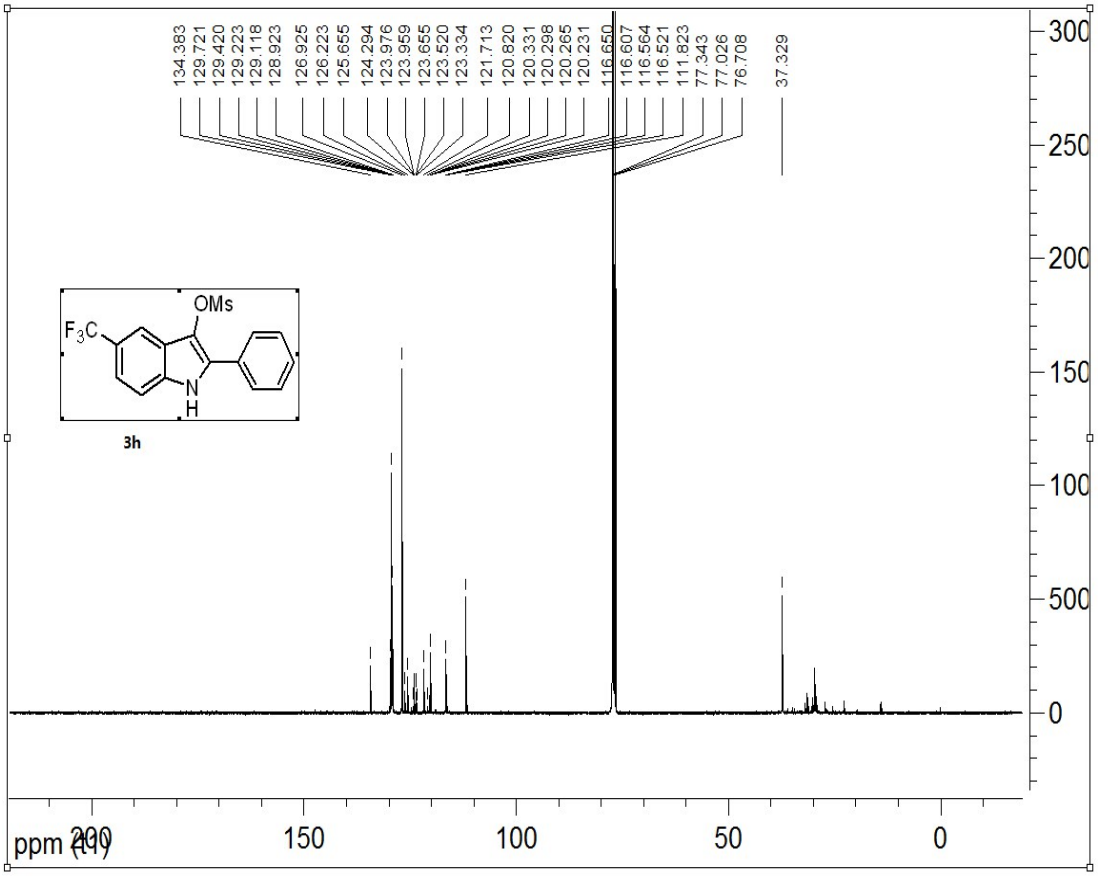
7.750
7.544
7.542
7.537
7.524
7.520
7.508
7.504
7.491
7.487
7.469
7.465
7.459
7.449
7.443
7.440
7.429
7.426
7.419
7.286
2.951
OMs
F3C
N
H
3h
3.00
1.00
2.04
5.21
3.00
500
0
ppm (t1)
5.0
0.0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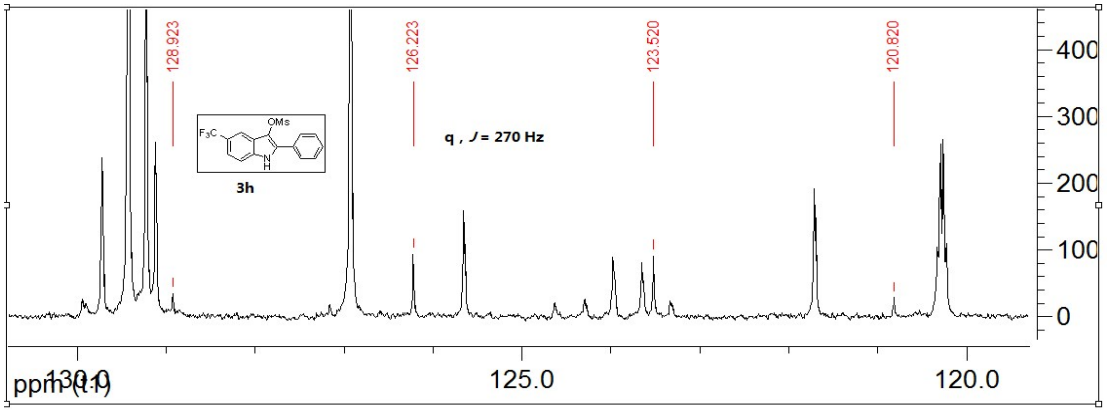
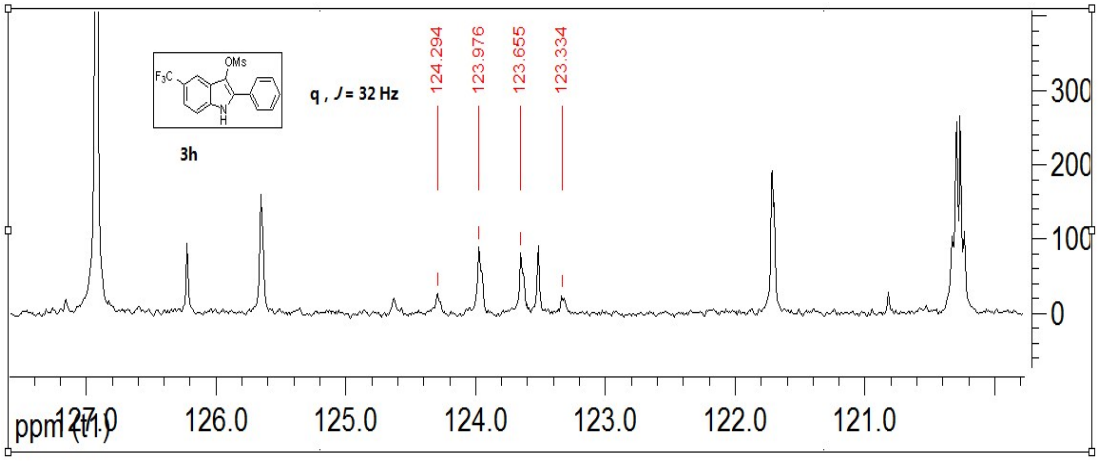
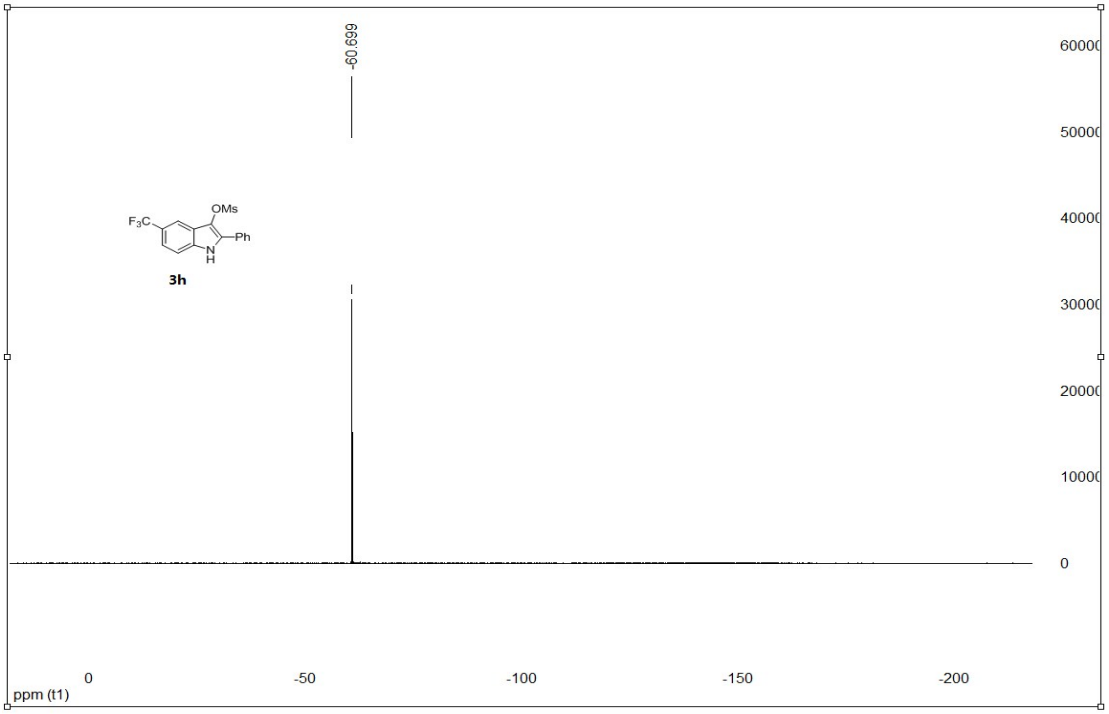
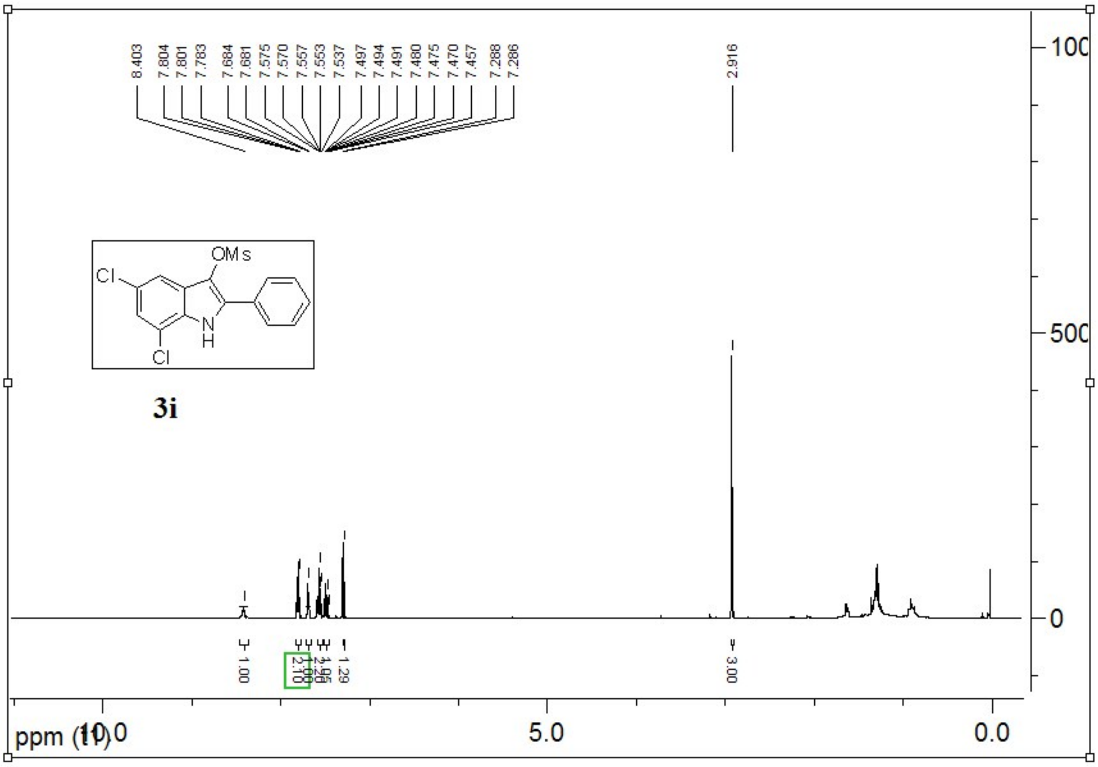
123.655
123.520
123.334
121.713
120.820
120.331
120.298
120.265
120.231
116.650
116.607
116.564
116.521
111.823
77.343
77.026
76.708
37.329
OMs
F3C
N
H
3h
300
250
200
150
100
500
0
ppm (t1)
200
150
100
50
0


128.923
126.223
123.520
120.820
q , J = 270 Hz
3h
400
300
200
100
0
130.0
125.0
120.0
ppm (t1)


124.294
123.976
123.655
123.334
q , J = 32 Hz
3h
300
200
100
0
127.0
126.0
125.0
124.0
123.0
122.0
121.0
ppm (t1)


-60.699
3h
60000
50000
40000
30000
20000
10000
0
0
-50
-100
-150
-200
ppm (t1)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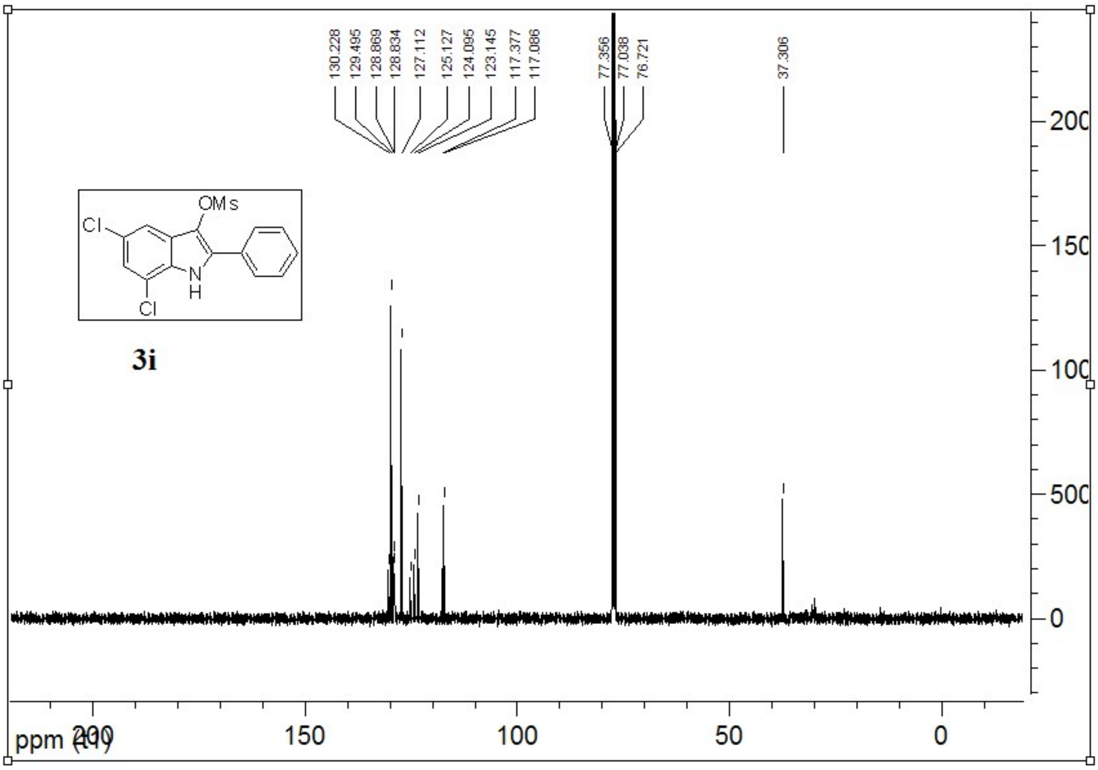
7.804
7.801
7.783
7.684
7.681
7.575
7.570
7.557
7.553
7.537
7.497
7.494
7.491
7.480
7.475
7.470
7.457
7.288
7.286
2.916
OMs
Cl
Cl
N
H
3i
1.00
2.10
1.29
3.00
1000
500
0
ppm (f1)
10.0
5.0
0.0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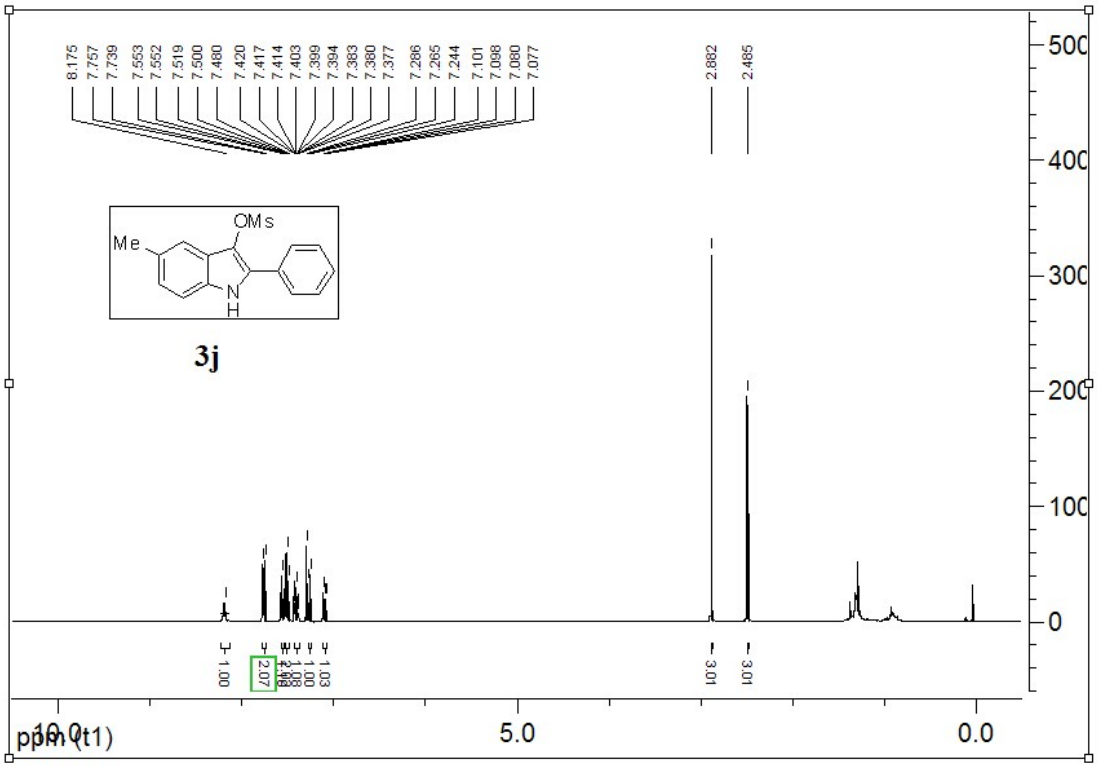
129.495
128.869
128.834
127.112
125.127
124.095
123.145
117.377
117.086
77.356
77.038
76.721
37.306
OMs
Cl
Cl
N
H
3i
2000
1500
1000
500
0
ppm (f1)
200
150
100
50
0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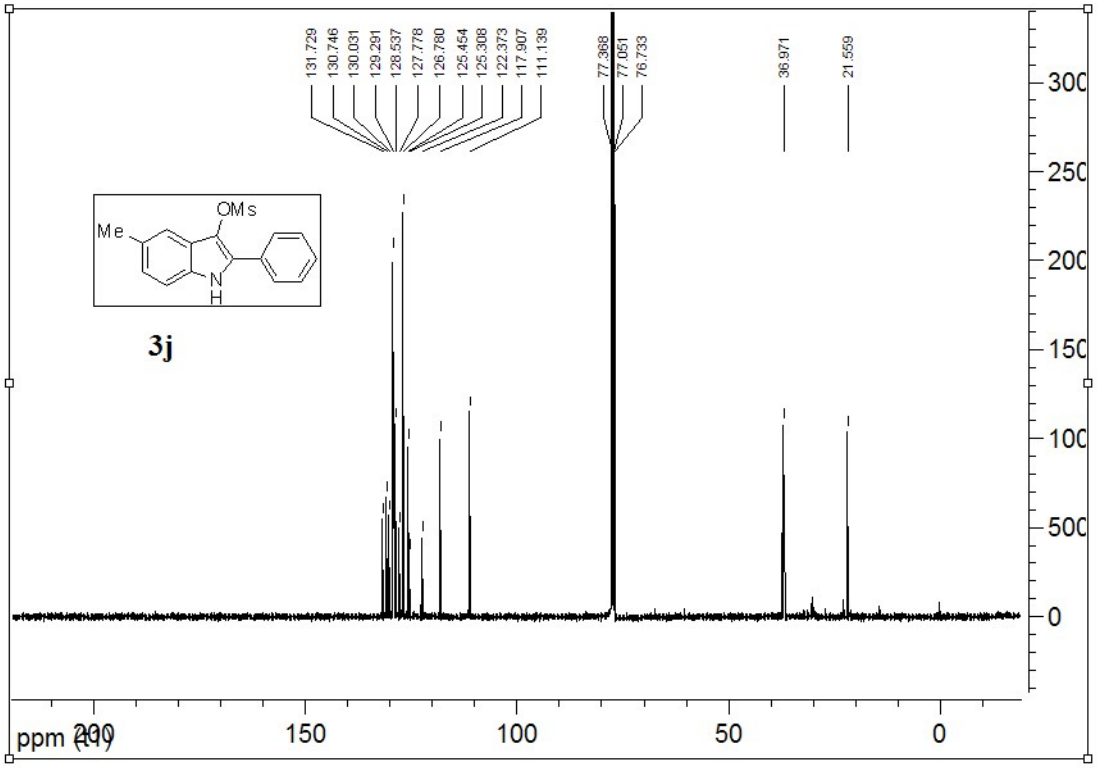
7.757
7.739
7.553
7.552
7.519
7.500
7.480
7.420
7.417
7.414
7.403
7.399
7.394
7.383
7.380
7.377
7.286
7.265
7.244
7.101
7.098
7.080
7.077
2.882
2.485
OMs
Me
N
H
3j
1.00
2.07
1.03
3.01
3.01
ppm (t1)
10.0
5.0
0.0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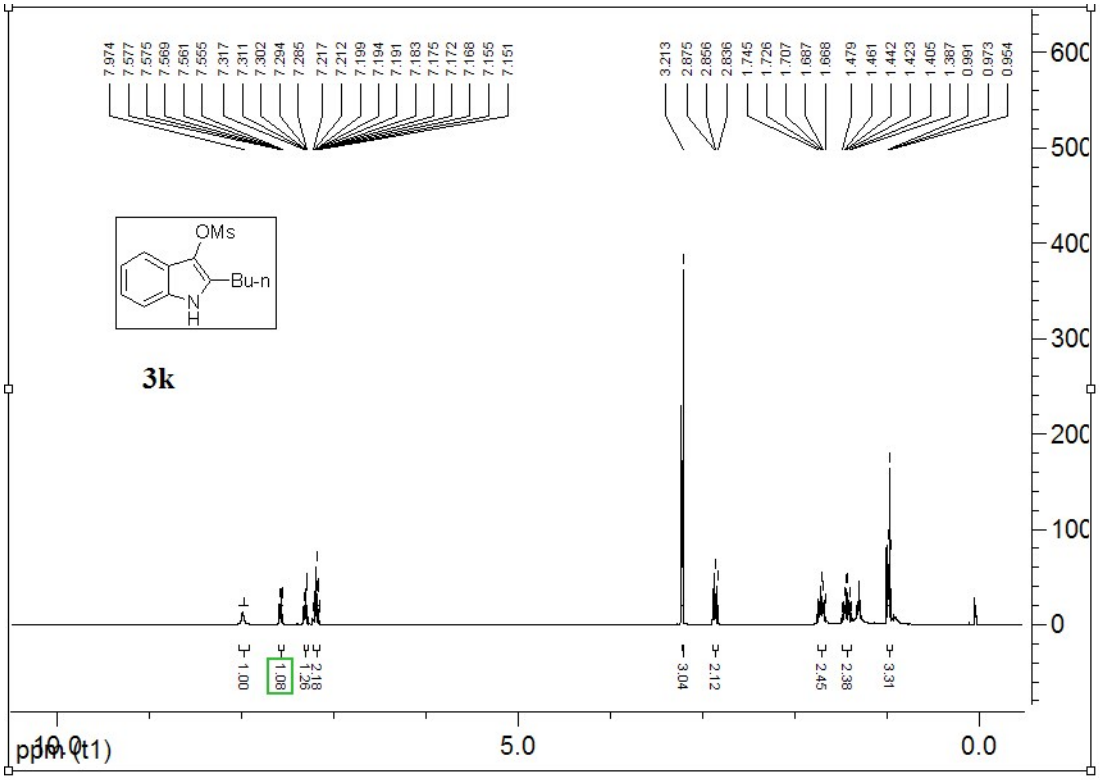
130.746
130.031
129.291
128.537
127.778
126.780
125.454
125.308
122.373
117.907
111.139
77.368
77.051
76.733
36.971
21.559
OMs
Me
N
H
3j
ppm (t1)
200
150
100
50
0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7.575
7.569
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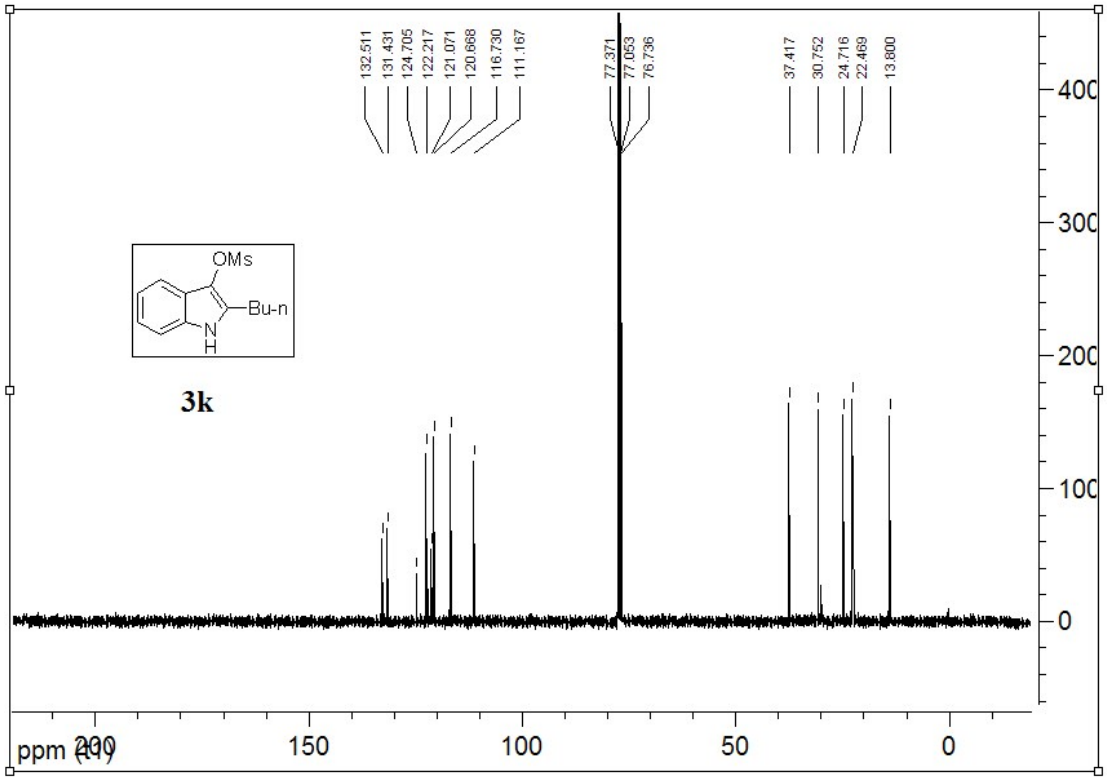
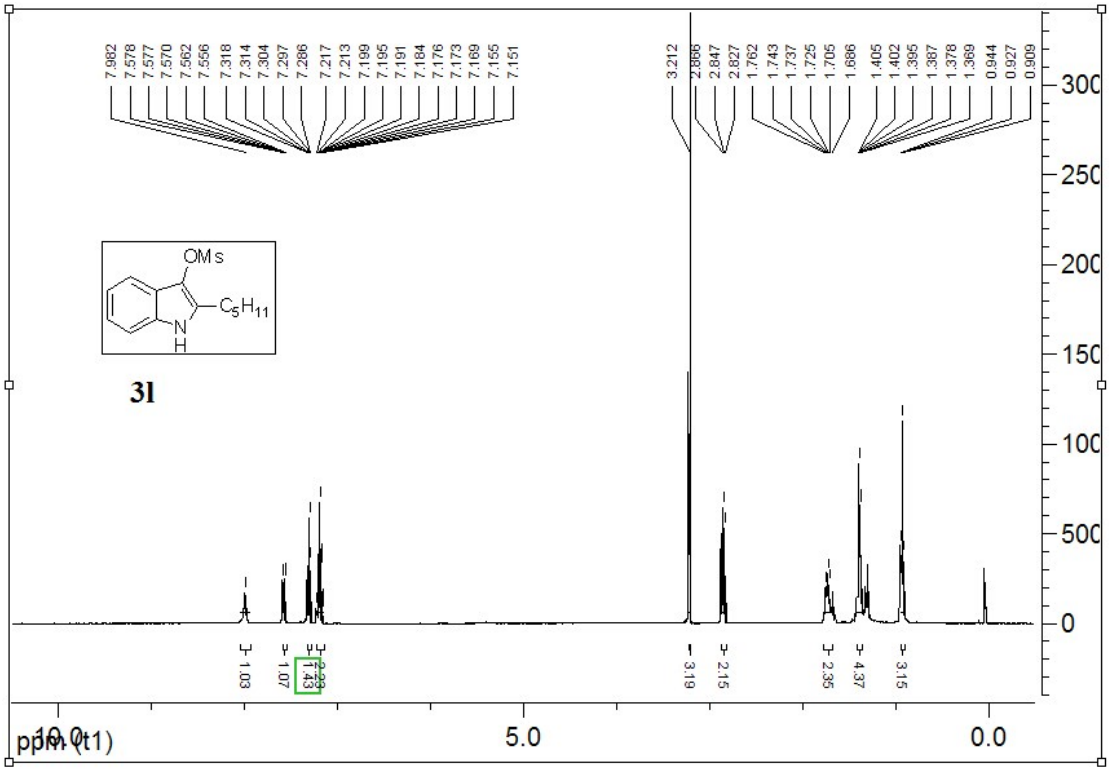
7.555
7.317
7.311
7.302
7.294
7.285
7.217
7.212
7.199
7.194
7.191
7.183
7.175
7.172
7.168
7.155
7.151
3.213
2.875
2.856
2.836
1.745
1.726
1.707
1.687
1.668
1.479
1.461
1.442
1.423
1.405
1.387
0.991
0.973
0.954
OMs
Bu-n
N
H
3k
1.00
1.08
2.18
1.26
3.04
2.12
2.45
2.38
3.31
ppm (t1)
10.0
5.0
0.0


132.511
131.431
124.705
122.217
121.071
120.668
116.730
111.167
77.371
77.053
76.736
37.417
30.752
24.716
22.469
13.800
OMs
Bu-n
N
H
3k
ppm (t1)
200
150
100
50
0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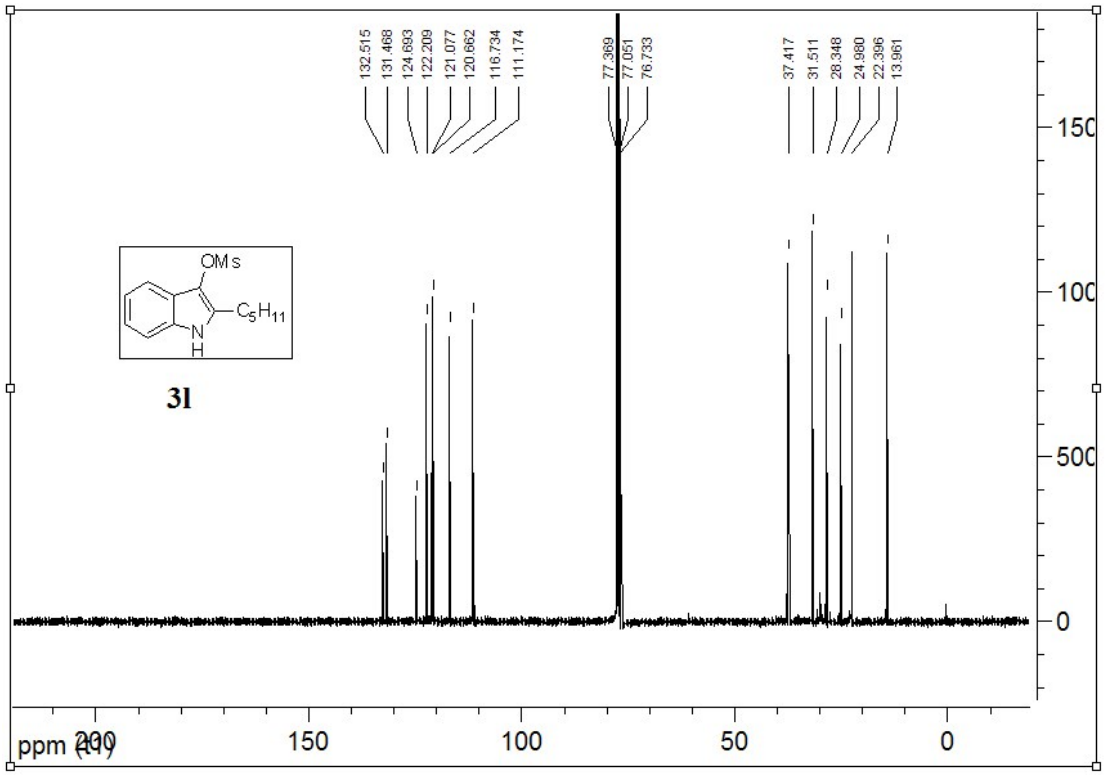
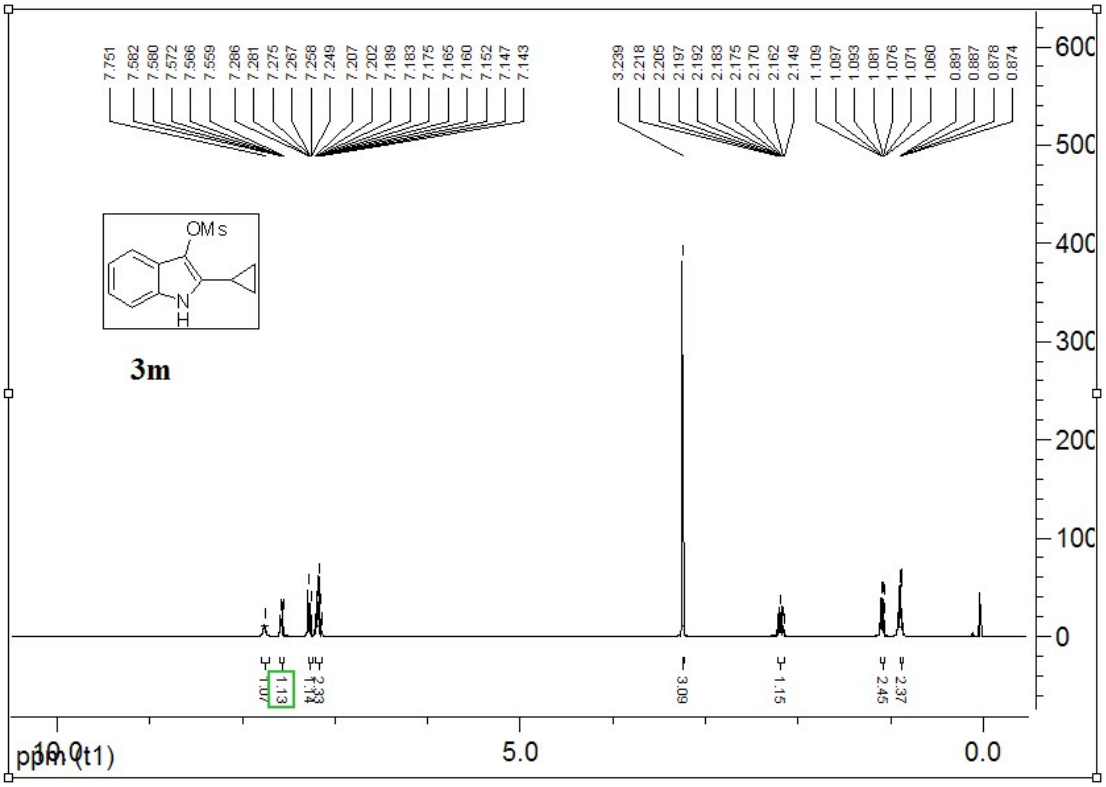
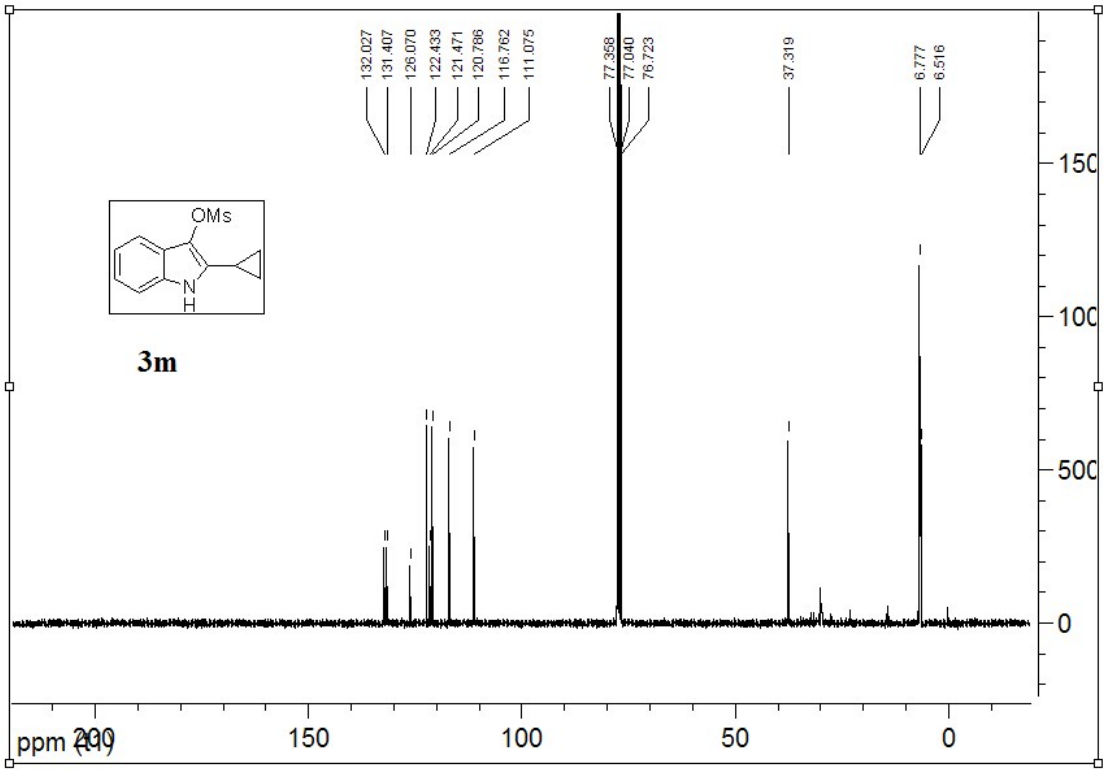
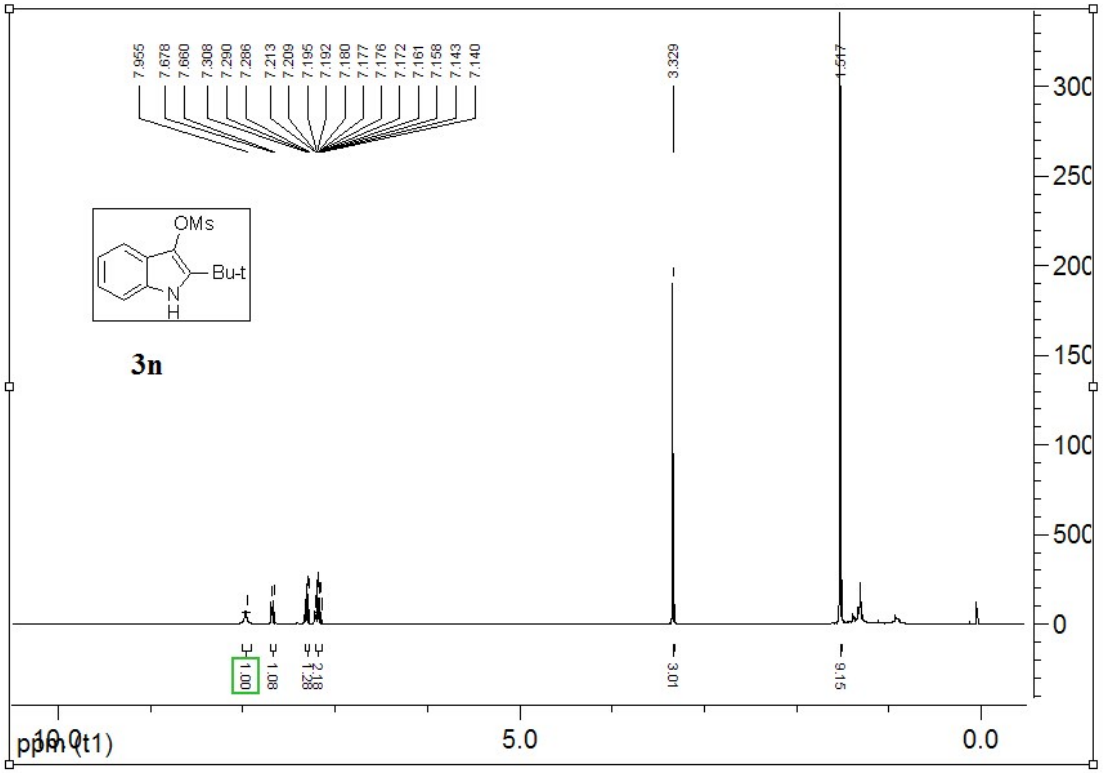
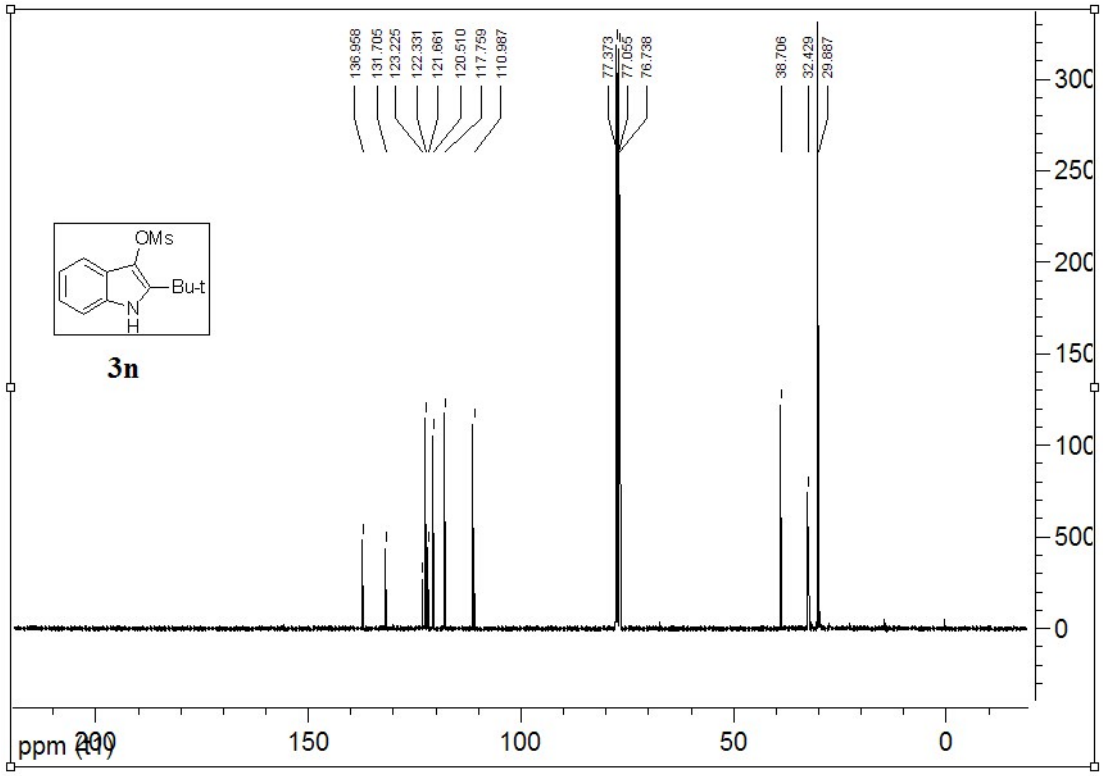
7.577
7.570
7.562
7.556
7.318
7.314
7.304
7.297
7.286
7.217
7.213
7.199
7.195
7.191
7.184
7.176
7.173
7.169
7.155
7.151
3.212
2.866
2.847
2.827
1.762
1.743
1.737
1.725
1.705
1.686
1.405
1.402
1.395
1.387
1.378
1.369
0.944
0.927
0.909
OMs
C_5H_{11}
N
H
3l
1.03
1.07
1.43
3.19
2.15
2.35
4.37
3.15
ppm (t1)
5.0
0.0

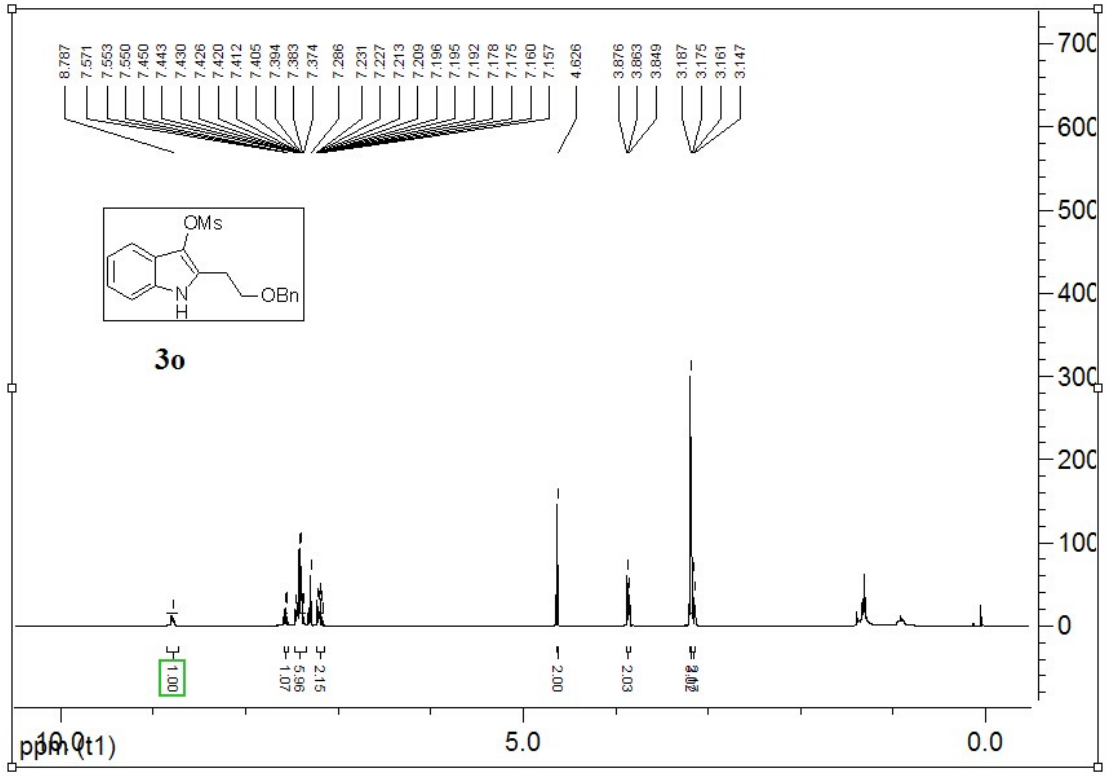

132.515
131.468
124.693
122.209
121.077
120.662
116.734
111.174
77.369
77.051
76.733
37.417
31.511
28.348
24.980
22.396
13.961
OMs
C_5H_{11}
N
H
3l
ppm (t1)
150
100
50
0


7.751
7.582
7.580
7.572
7.566
7.559
7.286
7.281
7.275
7.267
7.258
7.249
7.207
7.202
7.189
7.183
7.175
7.165
7.160
7.152
7.147
7.143
3.239
2.218
2.205
2.197
2.192
2.183
2.175
2.170
2.162
2.149
1.109
1.097
1.093
1.081
1.076
1.071
1.060
0.891
0.887
0.878
0.874
OMs
N
H
3m
1.07
1.13
2.33
2.37
3.09
1.15
2.45
2.37
ppm (t1)
5.0
0.0


132.027
131.407
126.070
122.433
121.471
120.786
116.762
111.075
77.358
77.040
76.723
37.319
6.777
6.516
OMs
N
H
3m
ppm (t1)
150
100
50
0


7.955
7.678
7.660
7.308
7.290
7.286
7.213
7.209
7.195
7.192
7.180
7.177
7.176
7.172
7.161
7.158
7.143
7.140
3.329
1.517
OMs
Bu-t
N
H
3n
1.00
1.08
2.88
3.01
9.15
ppm (t1)
5.0
0.0


136.958
131.705
123.225
122.331
121.661
120.510
117.759
110.987
77.373
77.055
76.738
38.706
32.429
29.887
OMs
Bu-t
N
H
3n
ppm (t1)
150
100
50
0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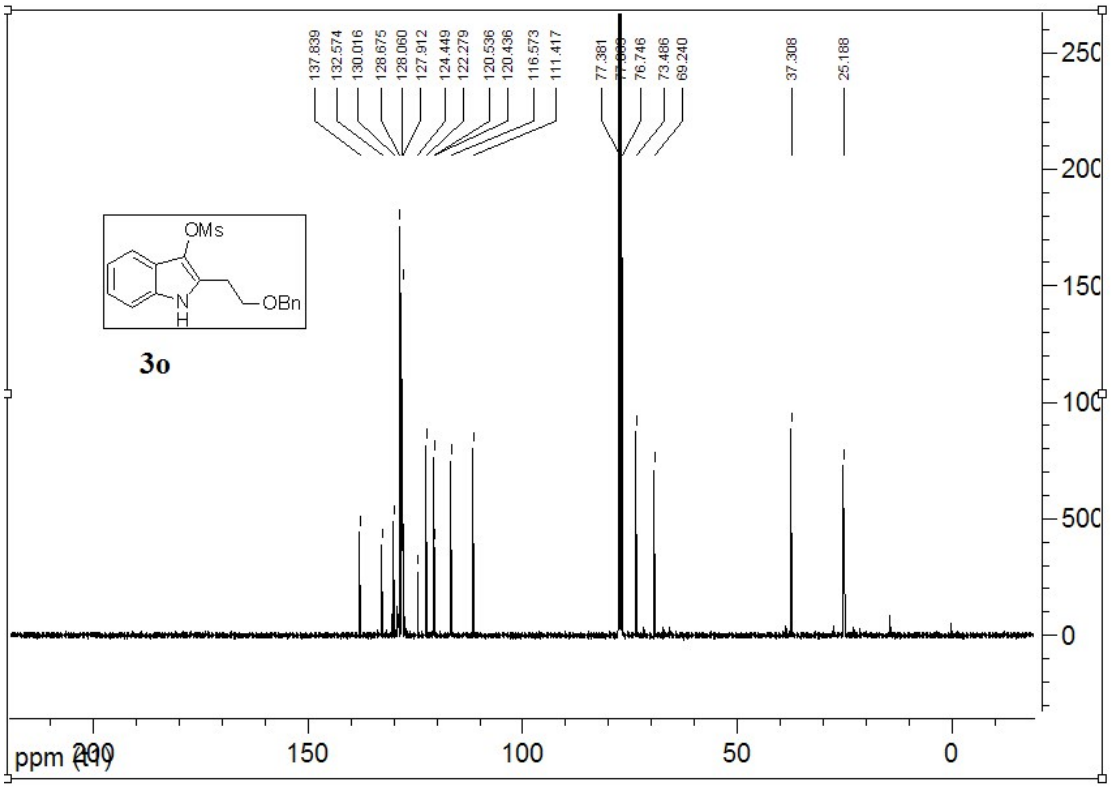
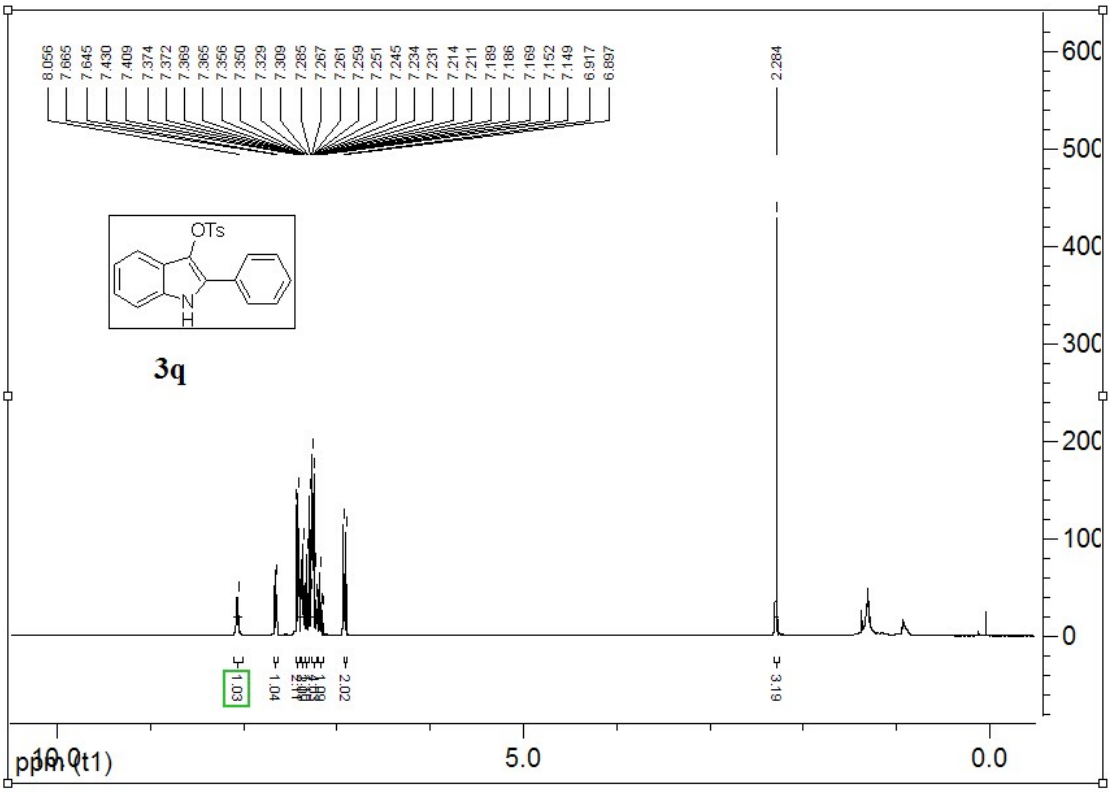
7.553
7.550
7.450
7.443
7.430
7.426
7.420
7.412
7.405
7.394
7.383
7.374
7.286
7.231
7.227
7.213
7.209
7.196
7.195
7.192
7.178
7.175
7.160
7.157
4.626
3.876
3.863
3.849
3.187
3.175
3.161
3.147
OMs
OBn
N
H
3o
1.00
1.07
0.96
2.15
2.00
2.03
ppm (t1)
5.0
0.0


137.839
132.574
130.016
128.675
128.060
127.912
124.449
122.279
120.536
120.436
116.573
111.417
77.381
76.746
73.486
69.240
37.308
25.188
OMs
OBn
N
H
3o
ppm (t1)
150
100
50
0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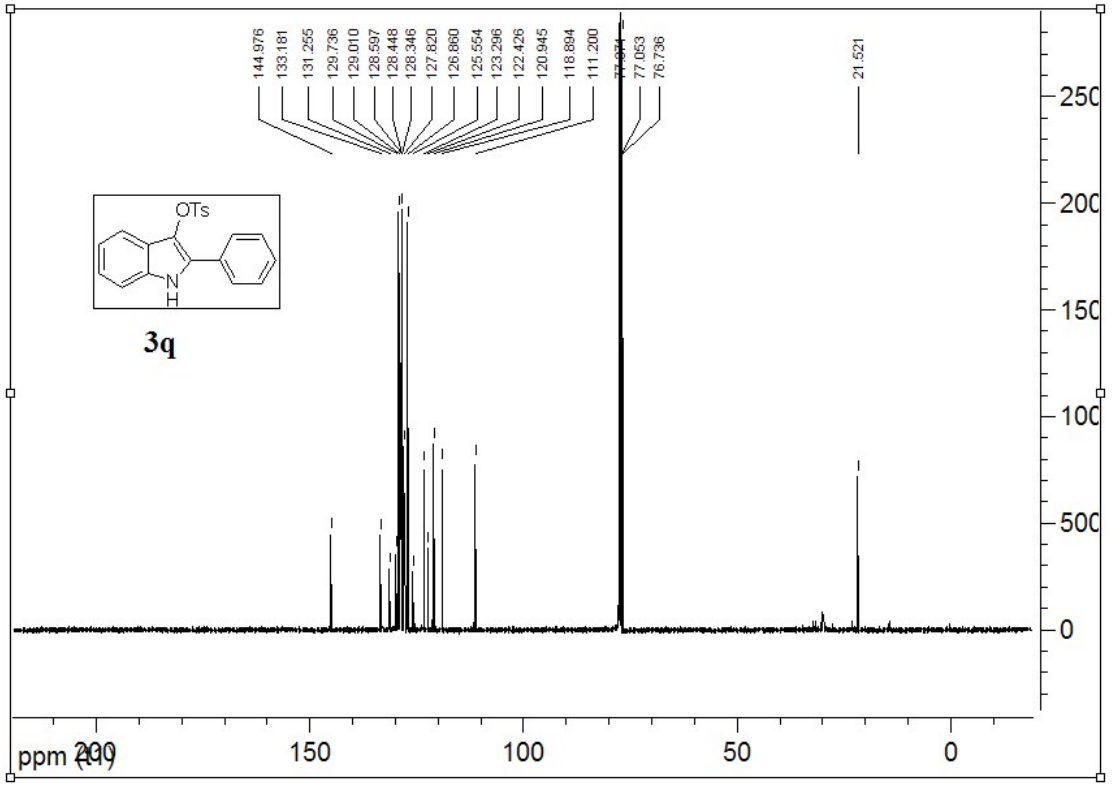
7.665
7.645
7.430
7.409
7.374
7.372
7.369
7.365
7.356
7.350
7.329
7.309
7.285
7.267
7.261
7.259
7.251
7.245
7.234
7.231
7.214
7.211
7.189
7.186
7.169
7.152
7.149
6.917
6.897
2.284
OTs
N
H
3q
1.03
1.04
2.02
3.19
600
500
400
300
200
100
0
ppm (t1)
5.0
0.0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131.255
129.736
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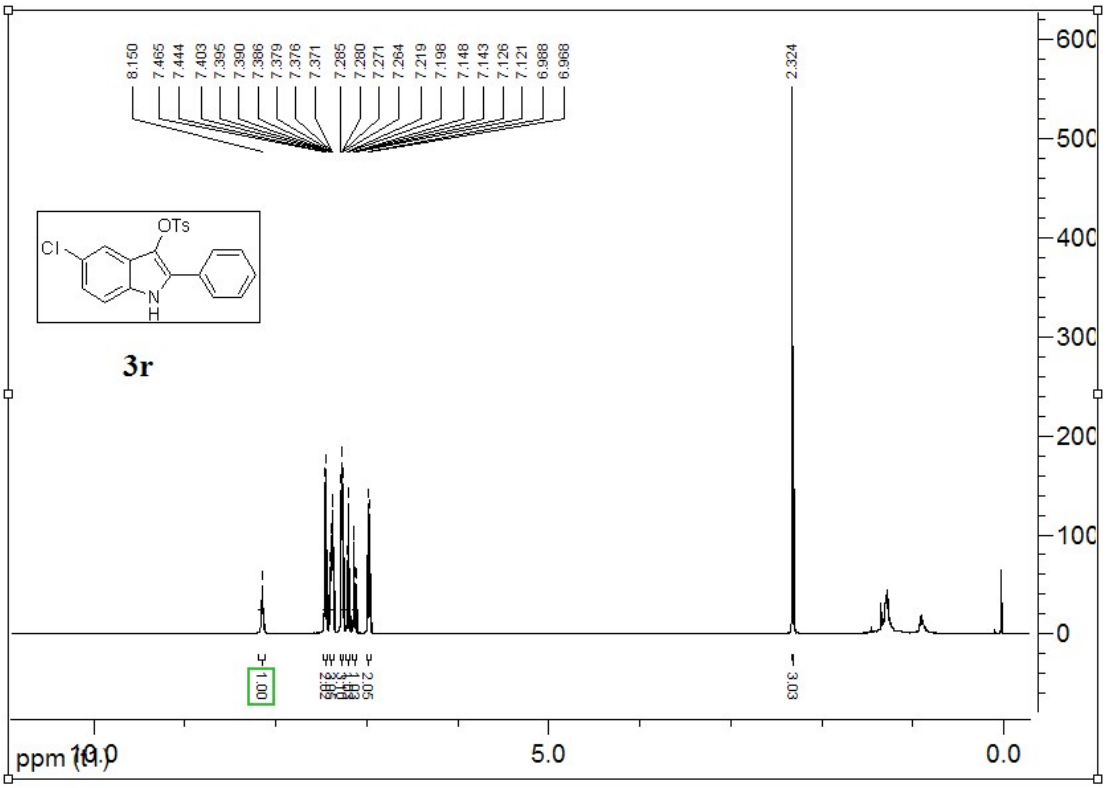
128.448
128.346
127.820
126.860
125.554
123.296
122.426
120.945
118.894
111.200
77.053
76.736
21.521
OTs
N
H
3q
250
200
150
100
50
0
ppm (t1)
150
100
50
0


8.150
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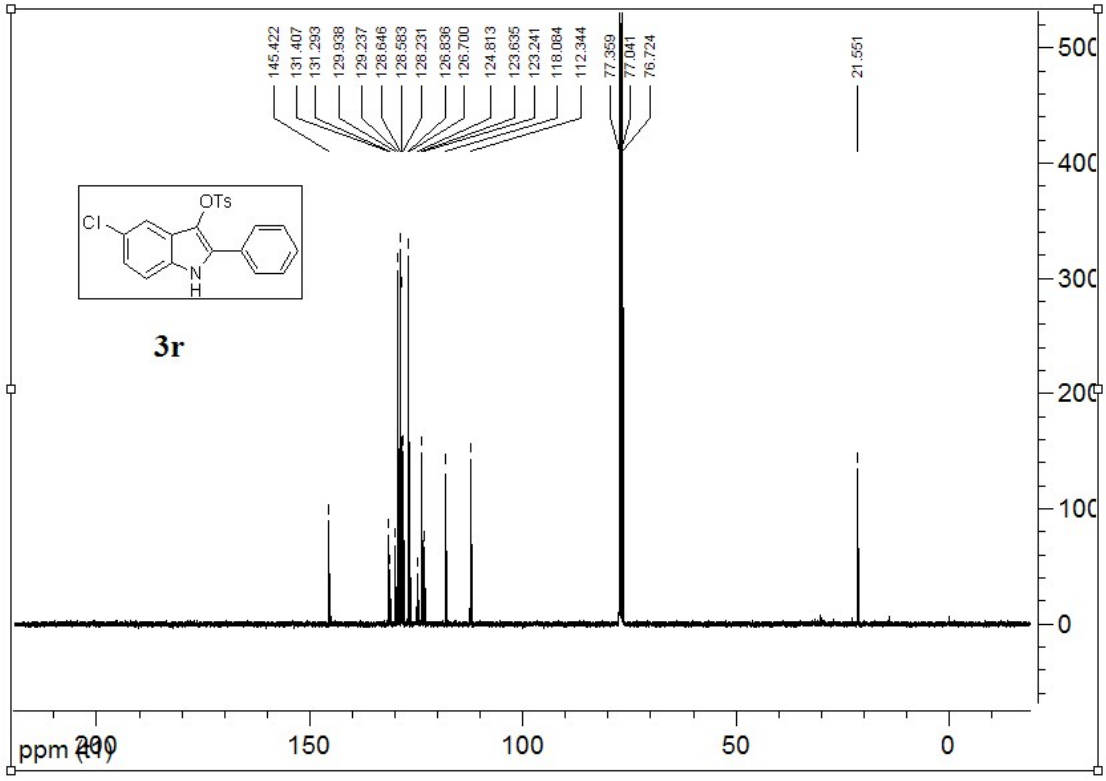
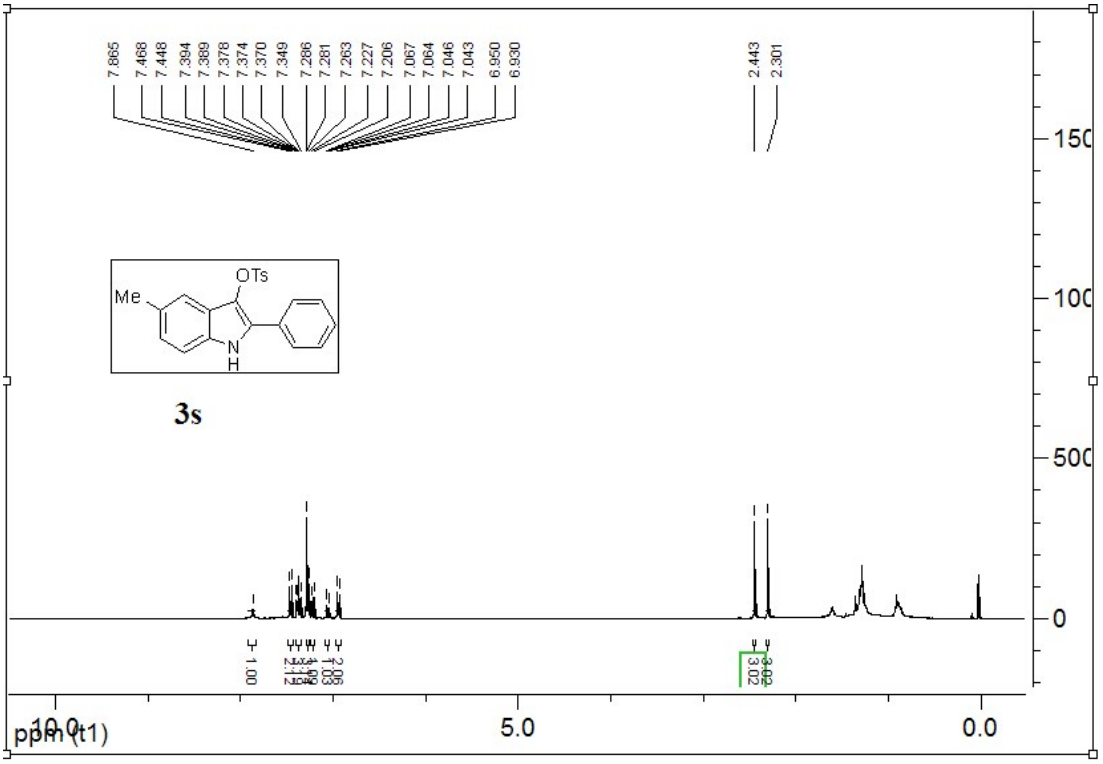
7.444
7.403
7.395
7.390
7.386
7.379
7.376
7.371
7.285
7.280
7.271
7.264
7.219
7.198
7.148
7.143
7.126
7.121
6.988
6.968
2.324
OTs
Cl
N
H
3r
1.00
2.05
3.03
ppm (t1)
10.0
5.0
0.0


145.422
131.407
131.293
129.938
129.237
128.646
128.583
128.231
126.836
126.700
124.813
123.635
123.241
118.084
112.344
77.359
77.041
76.724
21.551
OTs
Cl
N
H
3r
ppm (t1)
200
150
100
50
0


7.865
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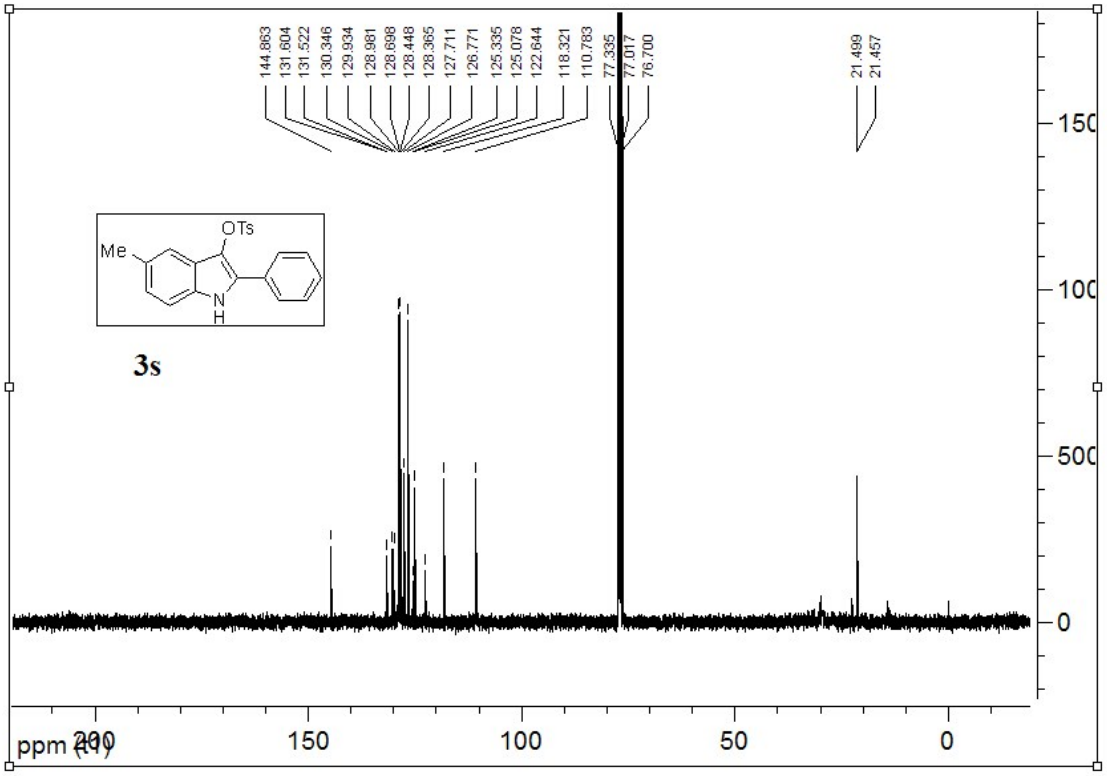
7.448
7.394
7.389
7.378
7.374
7.370
7.349
7.286
7.261
7.263
7.227
7.206
7.067
7.064
7.046
7.043
6.950
6.930
2.443
2.301
OTs
Me
N
H
3s
150
100
500
0
1.00
2.08
3.02
10.0
5.0
0.0
ppm (t1)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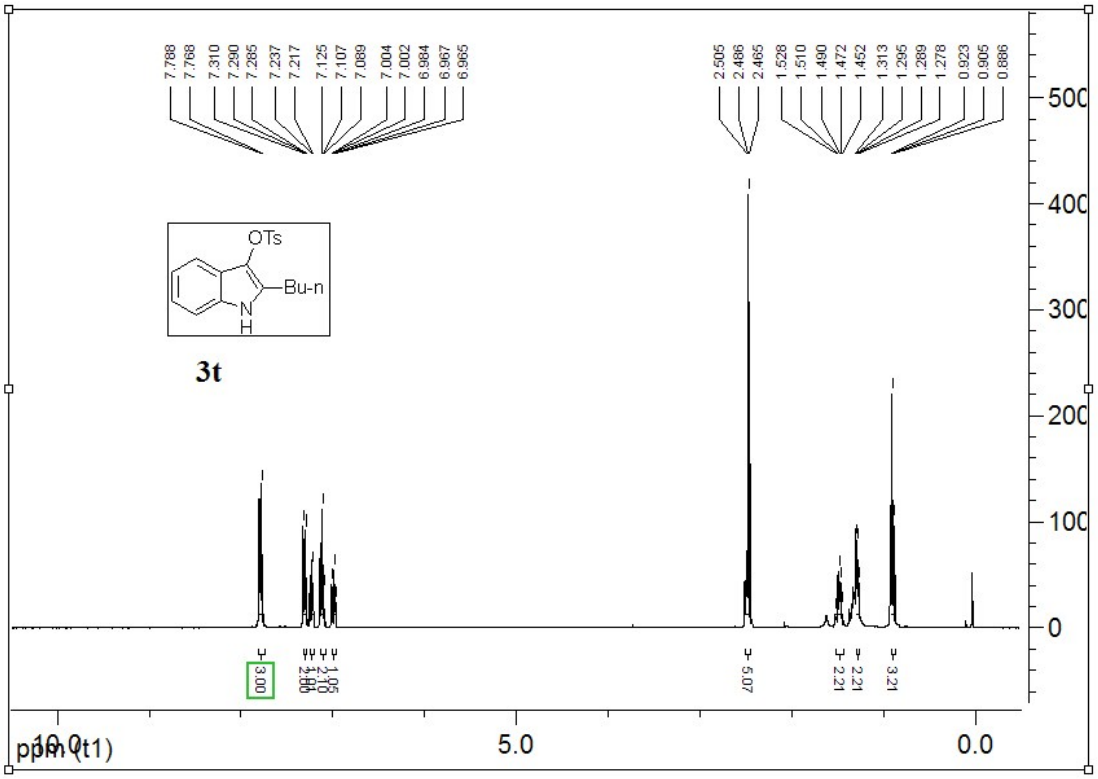
131.522
130.346
129.934
128.981
128.698
128.448
128.365
127.711
126.771
125.335
125.078
122.644
118.321
110.783
77.335
77.017
76.700
21.499
21.457
OTs
Me
N
H
3s
150
100
500
0
200
150
100
50
0
ppm (t1)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7.290
7.285
7.237
7.217
7.125
7.107
7.089
7.004
7.002
6.984
6.967
6.965
2.505
2.486
2.465
1.528
1.510
1.490
1.472
1.452
1.313
1.295
1.289
1.278
0.923
0.905
0.886
OTs
Bu-n
N
H
3t
3.00
2.05
2.00
1.00
5.07
2.21
2.21
3.21
ppm (t1)
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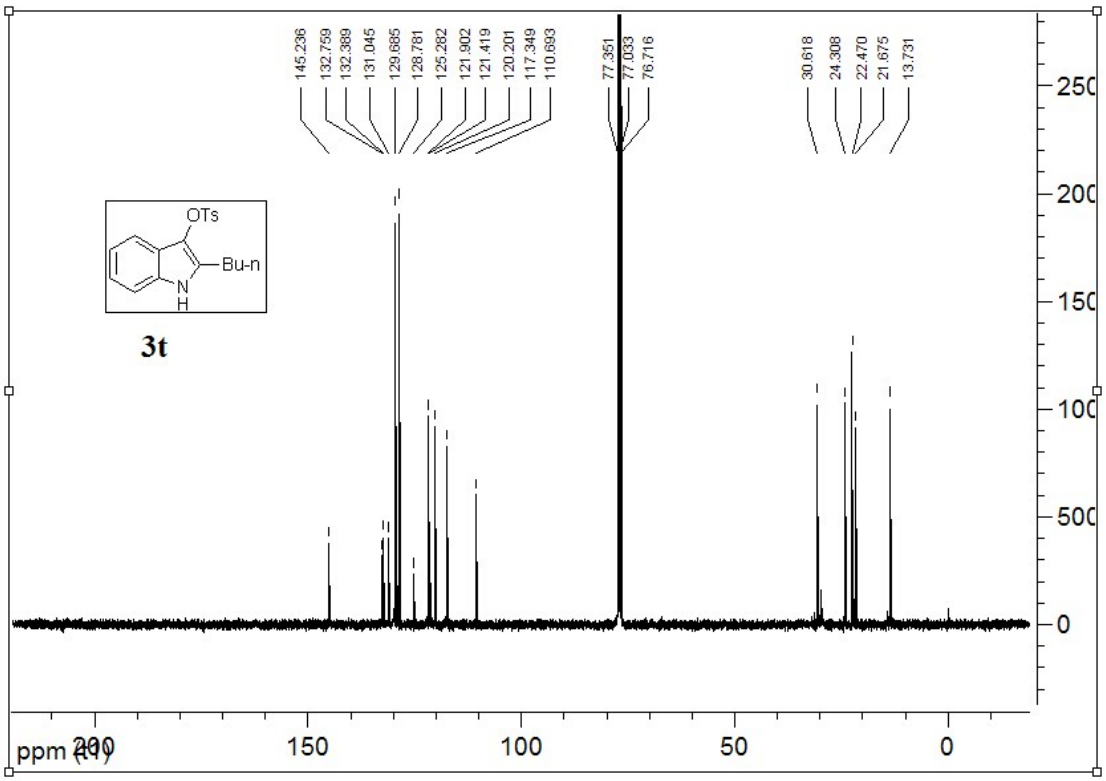
5.0
0.0


145.236
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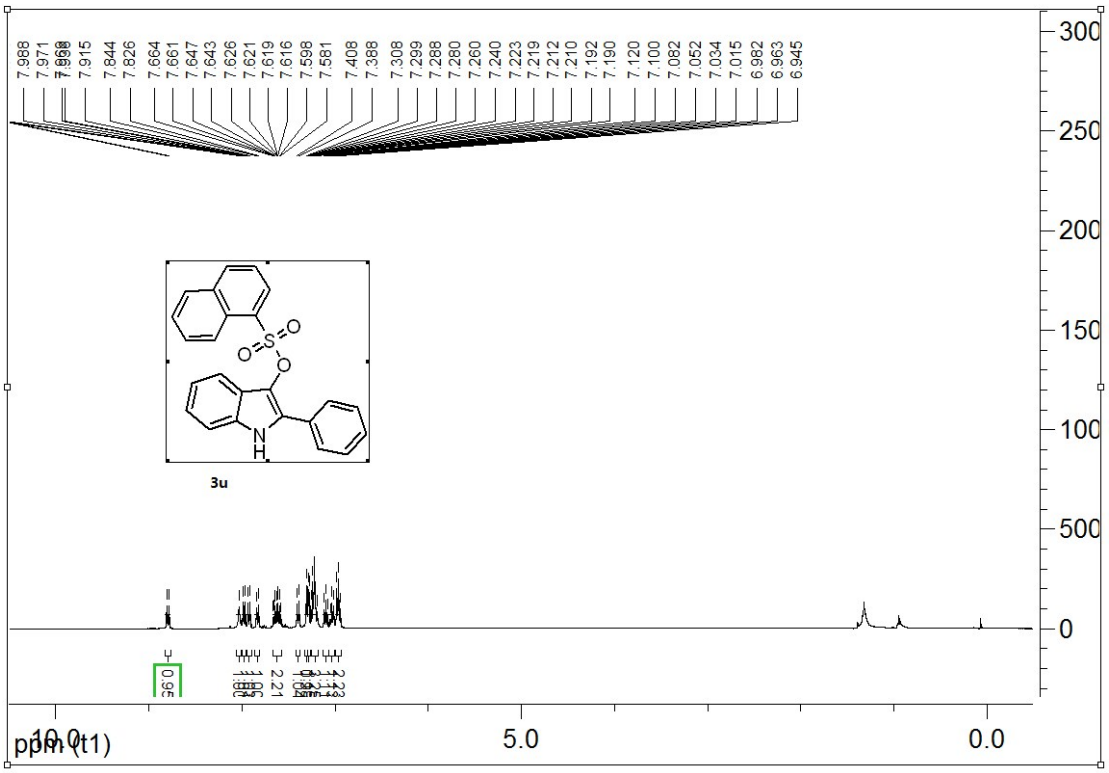
132.389
131.045
129.685
128.781
125.282
121.902
121.419
120.201
117.349
110.693
77.351
77.033
76.716
30.618
24.308
22.470
21.675
13.731
OTs
Bu-n
N
H
3t
ppm (t1)
200
150
100
50
0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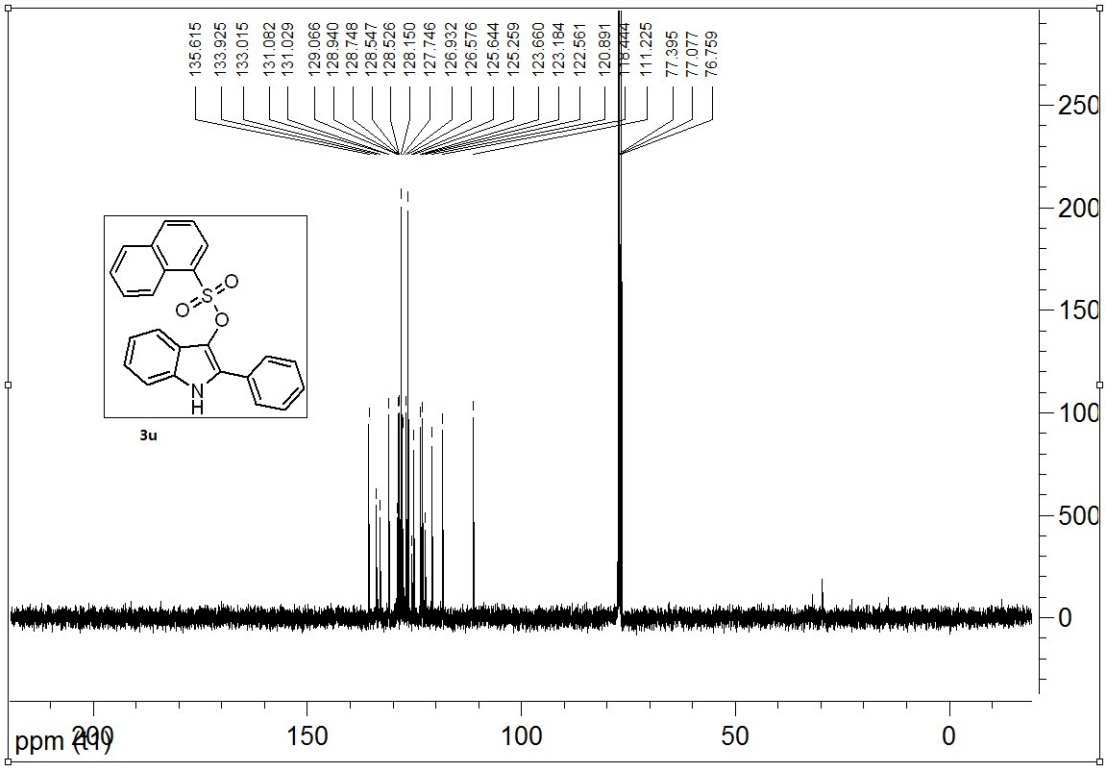
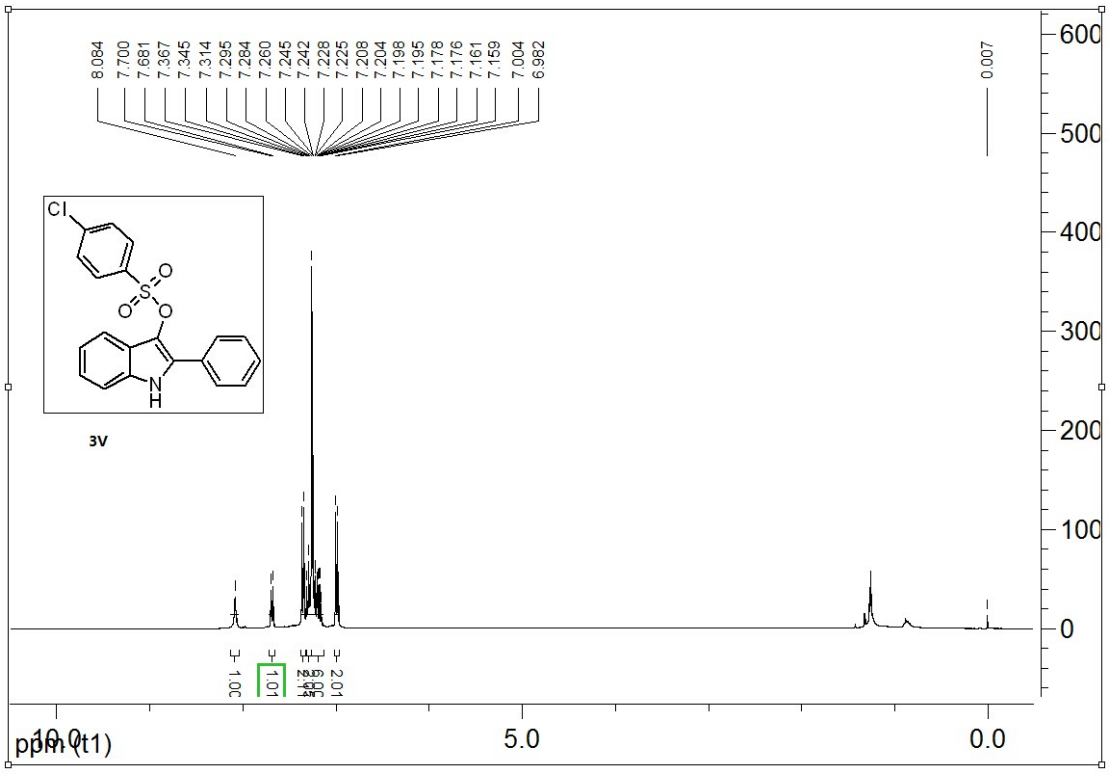
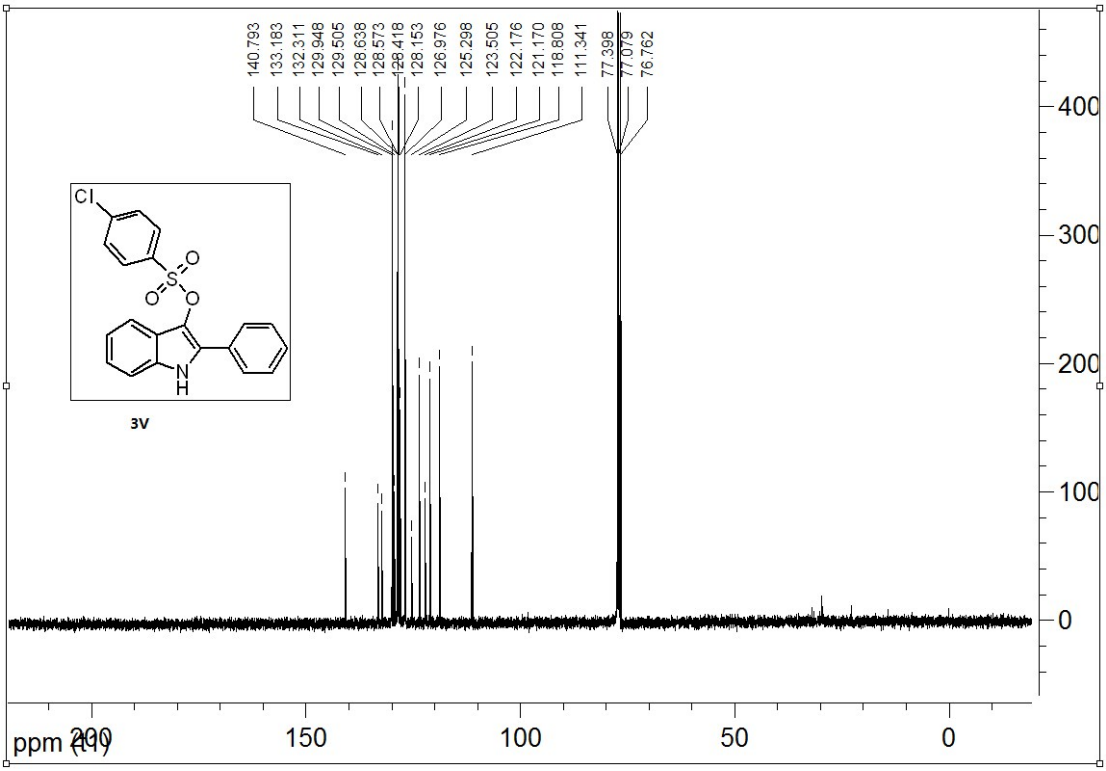
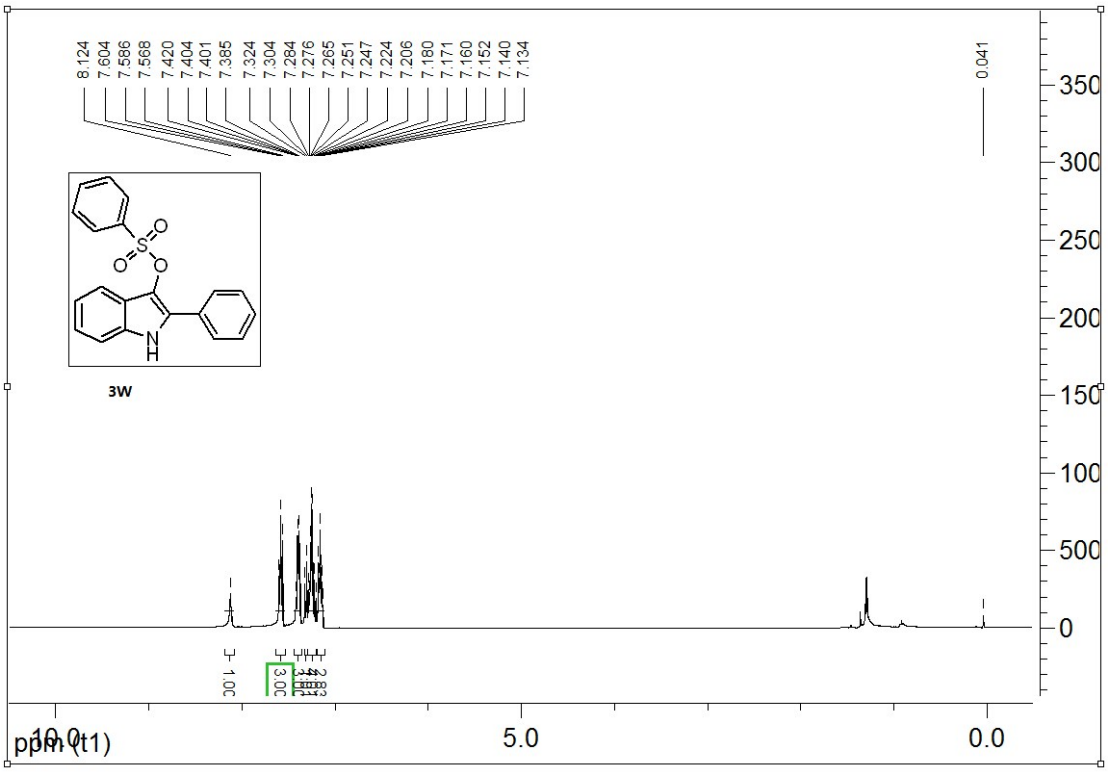
7.844
7.826
7.664
7.661
7.647
7.643
7.626
7.621
7.619
7.616
7.598
7.581
7.408
7.388
7.308
7.299
7.288
7.280
7.260
7.240
7.223
7.219
7.212
7.210
7.192
7.190
7.120
7.100
7.082
7.052
7.034
7.015
6.982
6.963
6.945
3u
ppm (t1)
5.0
0.0


135.615
133.925
133.015
131.082
131.029
129.066
128.940
128.748
128.547
128.526
128.150
127.746
126.932
126.576
125.644
125.259
123.660
123.184
122.561
120.891
111.225
77.395
77.077
76.759
3u
ppm (t1)
150
100
50
0


8.084
7.700
7.681
7.367
7.345
7.314
7.295
7.284
7.260
7.245
7.242
7.228
7.225
7.208
7.204
7.198
7.195
7.178
7.176
7.161
7.159
7.004
6.982
0.007
Cl
3V
ppm (t1)


140.793
133.183
132.311
129.948
129.505
128.638
128.573
128.418
128.153
126.976
125.298
123.505
122.176
121.170
118.808
111.341
77.398
77.079
76.762
Cl
3V
ppm (t1)


8.124
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7.586
7.568
7.420
7.404
7.401
7.385
7.324
7.304
7.284
7.276
7.265
7.251
7.247
7.224
7.206
7.180
7.171
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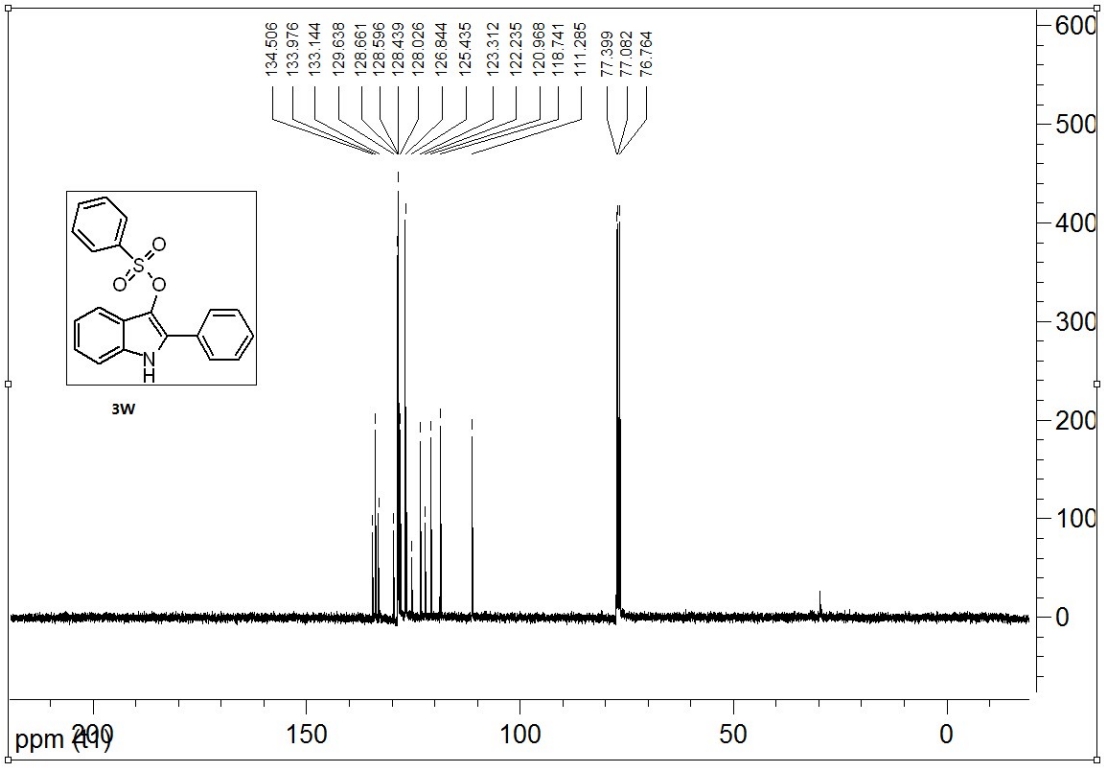
7.152
7.140
7.134
0.041
3W
ppm (t1)
10.0
5.0
0.0


134.506
133.976
133.144
129.638
128.661
128.596
128.439
128.026
126.844
125.435
123.312
122.235
120.968
118.741
111.285
77.399
77.082
76.764
3W
ppm (t1)
200
150
100
50
0