

Iodine-mediated Regioselective C2-Amination of Indoles and Concise Total Synthesis of (±)-Folicanthine

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General Remarks

Column chromatography was carried out on silica gel. ^1H NMR spectra were recorded on 400 MHz in CDCl_3 and ^{13}C NMR spectra were recorded on 400 MHz in CDCl_3 . IR spectra were recorded on a FT-IR spectrometer and only major peaks are reported in cm^{-1} . Melting points were determined on a microscopic apparatus and were uncorrected. All compounds were further characterized by HRMS; copies of their ^1H NMR and ^{13}C NMR spectra are provided in the Supporting Information. Room temperature is 20–25°C. Commercially available reagents and solvents were used without further purification.

Experimental Section

General Procedure A: Starting materials 1a–1r were prepared by methylation, benzylation, or allylation of the corresponding indoles according to the literature procedure.¹ Starting materials 2a–2r were prepared by tosylation of the corresponding anilines according to the literature procedure²

A mixture of indole derivative (0.5 mmol) **1**, Cs₂CO₃ (325 mg, 1 mmol), *N*-tosylbenzenamine derivative (1.0 mmol) **2**, I₂ (254 mg, 1 mmol) and acetonitrile (CH₃CN) (2.5 mL) was stirred at room temperature under air for 3 h. The reaction was quenched with a saturation solution of Na₂S₂O₃ (5 mL) and then extracted with ethyl acetate (3 × 30 mL). The combined organic phases were washed with brine (50 mL), dried over anhydrous sodium sulfate, filtered, and concentrated. The remains was purified the mixture of petroleum ether and ethyl acetate as eluent to afford of the corresponding product **3** 42-90% yields.

Characterization Data of 3a-3r

1-methyl-N-p-tolyl-N-tosyl-1H-indol-2-amine 3a: Compound **3a** was isolated in 84% yield as a white solid following the general procedure A. Reaction time: 3h. mp: 141-145°C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.55 (d, *J* = 8.0 Hz, 2H), 7.50 (d, *J* = 8 Hz, 1H), 7.22-7.28 (m, 6H), 7.07-7.11 (m, 3H), 6.12 (s, 1H), 3.76 (s, 3H), 2.44 (s, 3H), 2.30 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 144.0, 137.7, 137.6, 135.4, 134.9, 134.7, 129.7, 129.3, 128.4, 127.7, 125.8, 122.5, 120.9, 119.8, 109.8, 99.4, 29.0, 21.6, 21.0; HRMS (ESI): calc for C₂₃H₂₂N₂O₂S [M+1]⁺ : 391.1475 found: 391.1471; IR (neat, cm⁻¹): 3413, 2924, 1383, 1357, 1165, 667.

1-methyl-N-phenyl-N-tosyl-1H-indol-2-amine 3b: Compound **3b** was isolated in 55% yield as a white solid following the general procedure A. Reaction time: 3h. mp: 177-180°C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.55 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.736-7.40 (m, 2H), 7.21-7.32 (m, 7H), 7.06-7.10 (m, 1H), 6.15 (s, 1H), 3.75 (s, 3H), 2.44 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 144.1, 140.2, 135.5, 135.0, 134.4, 129.4, 129.1, 128.4, 125.8, 122.6, 120.9, 119.9, 109.8, 99.7, 29.0, 21.6; HRMS (ESI): calc for C₂₂H₂₀N₂O₂S [M+1]⁺ : 377.1318 found: 377.1310 IR (neat,

cm⁻¹): 3394 , 2924, 1358, 1166, 752, 665, 565.

1-methyl-N-(3,4-dimethylphenyl)-N-tosyl-1H-indol-2-amine **3c**: Compound **3c** was isolated in 45% yield as a white solid following the general procedure **A**. Reaction time: 3h. mp: 174-178 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.56 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 7.6 Hz, 1H), 7.16-7.27 (m, 5H), 7.05-7.08 (m, 3H), 6.12 (s, 1H), 3.75 (s, 3H), 2.43 (s, 3H,), 2.19 (s, 6H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 143.9, 137.9, 137.5, 136.4, 135.7, 134.9, 134.8, 130.1, 129.3, 128.9, 128.4, 125.8, 125.0, 122.5, 120.8, 119.8, 109.7, 99.5, 29.0, 21.6, 19.8, 19.3; HRMS (ESI): calc for C₂₄H₂₄N₂O₂S [M+1]⁺ : 405.1631 found: 405.1635; IR (neat, cm⁻¹): 3404 , 2923, 1384, 1357, 1166, 753, 667.

1-methyl-N-tosyl-N-(4-(trifluoromethoxy)phenyl)-1H-indol-2-amine **3d**: Compound **3d** was isolated in 63% yield as a white solid following the general procedure **A**. Reaction time: 3h. mp: 158-162 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.55 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.41-7.45 (m, 2H), 7.12-7.41 (m, 7H), 6.62 (s, 1H), 3.89 (s, 3H), 3.76 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 148.0, 148.0, 144.5, 138.7, 135.1, 135.0, 133.9, 129.5, 128.8, 128.3, 125.7, 122.9, 121.4, 121.0, 120.1, 109.9; HRMS (ESI): calc for C₂₃H₁₉N₂O₃F₃S [M+1]⁺ :461.1141 found: 461.1146 IR (neat, cm⁻¹): 2927, 1503, 1361, 1263, 1167, 667.

N-(4-chlorophenyl)-1-methyl-N-tosyl-1H-indol-2-amine **3e**: Compound **3e** was isolated in 67% yield as a white solid following the general procedure **A**. Reaction time: 3h. mp: 155-158 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.50-7.55, (m, 3H), 7.31-7.34 (m, 2H), 7.23-7.28 (m, 6H), 7.08-7.10 (m, 1H), 6.12 (s, 1H), 3.72 (s, 3H), 3.2.43 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 144.4, 138.7, 135.1, 134.9, 133.9, 133.3, 129.5, 129.2, 128.7, 128.3, 125.7, 122.8, 120.9, 120.0, 29.0, 21.6; HRMS (ESI): calc for C₂₂H₁₉N₂O₂SCl [M+1]⁺ : 411.0929 found: 411.0934; IR (neat, cm⁻¹): 3398 , 2924, 1487, 1359, 1166, 1091, 754, 666.

1, 5-dimethyl-N-p-tolyl-N-tosyl-1H-indol-2-amine **3f**: Compound **3f** was isolated in 45% yield as a white solid following the general procedure **A**. Reaction time: 3h. mp: 78-80 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.55 (d, *J* = 8.4 Hz, 2H), 7.22-7.29 (m, 5H), 7.15 (d, *J* = 8.4 Hz, 1H), 7.09 (d, *J* = 8 Hz, 2H), 7.05 (d, *J* = 8.4 Hz, 1H), 6.02 (s,

1H), 3.73 (s, 3H), 2.45 (s, 3H), 2.40 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 144.0, 137.7, 137.6, 135.5, 134.6, 133.3, 129.7, 129.3, 129.0, 128.4, 127.6, 125.9, 124.2, 120.4, 109.5, 98.9, 29.0, 21.6, 21.4, 21.0; HRMS (ESI): calc for $\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_2\text{S}$ $[\text{M}+1]^+$: 405.1631 found: 405.1638 IR (neat, cm^{-1}): 3403, 2922, 1380, 1356, 1163, 671, 548.

1, 7-dimethyl-N-p-tolyl-N-tosyl-1H-indol-2-amine **3g**: Compound **3g** was isolated in 42% yield as a white solid following the general procedure A. Reaction time: 3h. Mp: 117-120°C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.54 (d, $J = 8.4\text{Hz}$, 2H), 7.25-7.34 (m, 5H), 7.11 (d, $J = 8.0\text{ Hz}$, 2H), 6.92 (d, $J = 6.8\text{ Hz}$, 2H), 6.07 (s, 1H), 4.01 (s, 3H), 2.73 (s, 3H), 2.46 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 144.0, 137.7, 137.6, 135.5, 134.8, 133.9, 129.7, 129.3, 128.4, 127.6, 126.5, 125.4, 121.5, 119.8, 118.9, 100.0, 31.9, 21.6, 21.0, 20.1; HRMS (ESI): calc for $\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_2\text{S}$ $[\text{M}+1]^+$: 405.1631 found: 405.1637 IR (neat, cm^{-1}): 3403, 2919, 2360, 1382, 1164, 666.

5-bromo-1-methyl-N-p-tolyl-N-tosyl-1H-indol-2-amine **3h**: Compound **3h** was isolated in 45% yield as a white solid following the general procedure A. Reaction time: 3h. mp: 140-142°C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.65 (d, $J = 1.6\text{ Hz}$, 2H), 7.53 (d, $J = 8.0\text{ Hz}$, 1H), 7.11-7.32 (m, 8H), 6.05 (s, 1H), 3.75 (s, 3H), 2.47 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 138.0, 137.0, 135.1, 128.4, 127.7, 122.4, 121.0, 120.4, 120.2, 120.1, 119.6, 109.6, 109.4, 107.4, 101.4, 33.0, 31.0; HRMS (ESI): calc for $\text{C}_{23}\text{H}_{21}\text{N}_2\text{O}_2\text{SBr}$ $[\text{M}+1]^+$: 469.0580 found: 469.0574 IR (neat, cm^{-1}): 3395, 2923, 1384, 1358, 1165, 668.

1, 4-dimethyl-N-p-tolyl-N-tosyl-1H-indol-2-amine Compound **3i**:

Compound **3i** was isolated in 57% yield as a colorless liquid following the general procedure A. Reaction time: 3h. ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.57 (d, $J = 8.0\text{ Hz}$, 2H), 7.26 (d, $J = 8\text{ Hz}$, 4H), 7.09-7.13 (m, 4H), 6.88 (d, $J = 8.0$, 1H), 6.13 (s, 1H), 3.74 (s, 3H), 2.44 (s, 6H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 144.0, 137.7, 137.6, 135.5, 134.7, 134.1, 130.3, 129.7, 129.3, 128.5, 127.6, 125.8, 122.7, 120.0, 107.4, 98.0, 29.2, 21.6, 21.0, 18.6; HRMS (ESI): calc for $\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_2\text{S}$ $[\text{M}+1]^+$: 405.1631

found: 405.1636 IR (neat, cm^{-1}): 2922, 1504, 1357, 1166, 761, 665, 596, 550.

N-(4-chlorophenyl)-1,3-dimethyl-*N*-tosyl-1*H*-indol-2-amine **3j**: Compound **3j** was isolated in 88% yield as a white solid following the general procedure A. Reaction time: 3h. Mp: 153-157°C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.67 (d, $J = 8.0$ Hz, 2H), 7.50 (d, $J = 8.0$ Hz, 1H), 7.20-7.27 (m, 8H), 7.08-7.12 (m, 1H), 3.67, (s, 3H), 2.41 (s, 3H), 1.83 (s, 3H,); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 144.4, 139.8, 136.9, 135.0, 131.6, 130.4, 129.7, 129.2, 127.9, 126.5, 125.5, 123.1, 119.4, 119.2, 109.6, 109.0, 29.5, 21.5, 8.9 ; HRMS (ESI): calc for $\text{C}_{23}\text{H}_{21}\text{N}_2\text{O}_2\text{SCl}$ $[\text{M}+1]^+$: 425.1085 found: 425.1090 IR (neat, cm^{-1}): 3397, 2921, 1595, 1359, 1166, 1092, 910, 736, 669, 576, 545.

1,3-dimethyl-*N*-tosyl-*N*-(4-(trifluoromethoxy)phenyl)-1*H*-indol-2-amine **3k**: Compound **3k** was isolated in 87% yield as a white solid following the general procedure A. Reaction time: 3h. mp: 132-136°C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.69 (d, $J = 8.4$ Hz, 2H), 7.51 (d, $J = 8$ Hz, 1H), 7.28-7.35 (m, 6H), 7.12 (t, 3H), 3.69 (s, 3H), 2.44 (s, 3H), 1.83 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 146.8, 144.5, 139.8, 136.9, 135.1, 130.4, 129.8, 127.9, 126.5, 125.6, 123.2, 121.6, 119.5, 119.3, 109.7, 109.1, 29.7, 29.5, 21.6, 8.9; HRMS (ESI): calc for $\text{C}_{24}\text{H}_{21}\text{N}_2\text{O}_3\text{F}_3\text{S}$ $[\text{M}+1]^+$: 475.1298 found: 475.1284 IR (neat, cm^{-1}): 3410, 2923, 1502, 1360, 1261, 1165, 670, 575, 545.

N-(2-methoxyphenyl)-1, 3-dimethyl-*N*-tosyl-1*H*-indol-2-amine **3l**: Compound **3l** was isolated in 78% yield as a white solid following the general procedure A. Reaction time: 3h. mp: 127-129°C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.63 (d, $J = 8.0$ Hz, 2H), 7.47 (d, $J = 7.6$ Hz, 1H), 7.34 (d, $J = 7.6$ Hz, 1H), 7.20-7.27 (m, 5H), 7.06-7.10 (m, 1H), 6.84-6.88 (m, 2H), 3.81 (s, 3H), 3.73 (s, 3H), 2.42 (s, 3H), 1.93 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 155.8, 143.6, 137.9, 135.0, 131.2, 129.5, 129.2, 128.2, 126.6, 122.6, 120.4, 119.2, 118.8, 112.3, 109.5, 108.8, 55.1, 30.0, 29.7, 21.6, 9.1; HRMS (ESI): calc for $\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_3\text{S}$ $[\text{M}+1]^+$: 421.1580 found: 421.1587 IR (neat, cm^{-1}): 3400, 2923, 1595, 1496, 1354, 1164, 749, 667.

1, 3-dimethyl-*N*-(3, 4-dimethylphenyl)-*N*-tosyl-1*H*-indol-2-amine **3m**: Compound

3m was isolated in 73% yield as a white solid following the general procedure **A**. Reaction time: 3h. mp: 172-173 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.69 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.23-7.29 (m, 4H), 7.01-7.12 (m, 4H), 3.72 (s, 3H), 2.44 (s, 3H), 2.19 (s, 3H), 2.18 (s, 3H), 1.89 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 143.9, 138.9, 137.7, 137.6, 135.2, 135.0, 131.3, 130.2, 129.6, 128.0, 126.6, 126.5, 122.8, 122.7, 119.3, 119.0, 109.5, 108.729.7, 29.6, 21.6, 20.0, 19.2, 9.0; HRMS (ESI): calc for C₂₅H₂₆N₂O₂S [M+1]⁺ : 419.1788 found: 419.1778 IR (neat, cm⁻¹): 3398, 2921, 1560, 1354, 1165, 745, 671.

1, 3-dimethyl-N-p-tolyl-N-tosyl-1H-indol-2-amine 3n: Compound **3n** was isolated in 90% yield as a colorless liquid following the general procedure **A**. Reaction time: 3h. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.20-7.27 (m, 6H), 7.06-7.09 (m, 3H), 3.70 (s, 3H), 2.40 (s, 3H), 2.27 (s, 3H), 1.88 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 144.0, 138.7, 137.5, 136.4, 135.0, 131.2, 129.8, 129.6, 127.9, 126.6, 125.1, 122.8, 119.3, 119.1, 109.5, 108.7; HRMS (ESI): calc for C₂₄H₂₄N₂O₂S [M+1]⁺ : 405.1631 found: 405.1636 IR (neat, cm⁻¹): 3418, 2920, 1379, 1355, 1164, 769, 670, 547.

3o: Compound **3o** was isolated in 56% yield as a white solid following the general procedure **A**. Reaction time: 3h. mp: 148-152 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.61-7.63 (dd, *J* = 2.0 Hz, 2H), 7.50-7.52 (dd, *J* = 2.0 Hz, 3H), 7.21-7.29 (m, 4H), 7.07-7.08 (m, 1H), 6.81-6.83 (dd, *J* = 2.4 Hz, 2H), 6.10 (s, 1H), 3.78 (s, 3H), 3.75 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 159.2, 137.2, 134.9, 134.6, 132.5, 132.0, 129.9, 129.5, 128.3, 125.7, 122.8, 120.9, 120.0, 114.4, 109.8, 99.1, 55.4, 29.0; HRMS (ESI): calc for C₂₂H₁₉N₂O₃SBr [M+1]⁺ : 471.0373 found: 471.0386 IR (neat, cm⁻¹): 3414, 2925, 1506, 1385, 1363, 1167, 770, 746.

3p: Compound **3p** was isolated in 62% yield as a white solid following the general procedure **A**. Reaction time: 3h. mp: 147-150 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.63 (s, 4H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.23-7.31 (m, 4H), 7.08-7.12 (m, 1H), 6.81-6.84 (m, 2H), 3.76 (s, 3H), 3.75 (s, 3H), 1.90 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 158.6, 139.4, 135.0, 133.5, 132.3, 131.1, 129.4, 128.2, 128.0, 126.5,

123.1, 119.4, 119.2, 114.5, 109.6, 108.4, 55.4, 29.7, 29.6, 9.2; HRMS (ESI): calc for $C_{23}H_{21}N_2O_3SBr$ $[M+1]^+$: 485.0529 found: 485.0538 IR (neat, cm^{-1}): 3396 , 2925, 1506, 1385, 1163, 741, 553.

N-(4-(4-methoxyphenoxy)phenyl)-1,3-dimethyl-*N*-tosyl-1*H*-indol-2-amine **3q**:

Compound **3q** was isolated in 45% yield as a white solid following the general procedure A. Reaction time: 3h. mp: 161-163 °C; 1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.67 (d, $J = 8.0$ Hz, 2H), 7.48 (d, $J = 8.0$ Hz, 1H), 7.23-7.29 (m, 6H), 7.08-7.10 (m, 1H), 6.93-6.95 (m, 2H), 6.83-6.87 (m, 4H), 3.78 (s, 3H), 3.73 (s, 3H), 2.44 (s, 3H), 1.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 157.3, 156.2, 149.4, 144.1, 137.5, 135.4, 135.0, 131.3, 129.7, 127.9, 127.5, 126.6, 122.9, 121.1, 119.3, 119.1, 117.7, 114.9, 109.6, 108.6, 55.6, 29.6, 21.6, 9.0; HRMS (ESI): calc for $C_{30}H_{28}N_2O_4S$ $[M+1]^+$: 513.1843 found: 513.1849 IR (neat, cm^{-1}): 3398 , 2922, 1496, 1356, 1225, 1164, 772.

1-allyl-5-methoxy-*N*-*p*-tolyl-*N*-tosyl-1*H*-indol-2-amine **3r**: Compound **3r** was isolated in 42% yield as a white solid following the general procedure A. Reaction time: 3h. mp: 135-137 °C; 1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.54 (d, $J = 8.0$ Hz, 2H), 7.22-7.28 (m, 4H), 7.15 (d, $J = 8.8$ Hz, 1H), 7.08 (d, $J = 8.4$ Hz, 2H), 6.97 (d, $J = 2.4$ Hz, 1H), 6.85-6.88 (dd , $J = 2.4$ Hz, 1H), 6.07 (s, 1H), 5.73-5.80 (m, 1H), 5.01-5.04 (dd, $J = 1.2$ Hz, 1H), 4.92-4.97 (dd, $J = 1.2$ Hz, 1H), 4.83 (d, $J = 5.2$ Hz, 2H), 3.79 (s, 3H), 2.44 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, TMS) δ 154.2, 144.0, 137.6, 137.5, 135.3, 134.5, 133.3, 129.6, 129.3, 128.5, 127.8, 126.2, 116.8, 113.0, 111.4, 102.4, 99.4, 55.7, 45.2, 29.6, 21.6, 21.0; HRMS (ESI): calc for $C_{26}H_{26}N_2O_3S$ $[M+1]^+$: 447.1737 found: 447.1745 IR (neat, cm^{-1}): 3403, 2924, 1480, 1358, 1167, 669.

General Procedure B:A mixture of indole derivative (10 mmol) **4**, Cs_2CO_3 (20 mmol), I_2 (20 mmol) and acetonitrile (25 mL) was stirred at room temperature under air for 1 h. The reaction was quenched with a saturation solution of $Na_2S_2O_3$ (25 mL) and then extracted with ethyl acetate (3×50 mL). The combined organic phases were washed with brine (150 mL), dried over anhydrous sodium sulfate, filtered, and concentrated. The residue was purified by flash column chromatography using

mixture of petroleum ether and ethyl acetate as eluent to afford of the corresponding product **5** as a white solid.

To a solution of naphthalene (641 mg, 5.0 mmol) in THF (10 mL) was added metal sodium (115 mg, 5.0 mmol) at room temperature. The mixture was stirred at room temperature for 2 h to yield a deep-blue sodium-naphthalenide solution (0.5 M). The fresh-prepared sodium-naphthalenide solution was added slowly to a solution of **5** (122 mg, 0.19 mmol) in THF (5 ml) at $-78\text{ }^{\circ}\text{C}$ until the color of reaction mixture maintained unchanged blue (about 5 mL, 10 equiv was used). After being stirred at $-78\text{ }^{\circ}\text{C}$ for 30 min, saturated aqueous NH_4Cl solution (2 mL) was added. The resulting mixture was warmed to room temperature and diluted with EtOAc (50 mL), washed with brine (20 mL \times 3), dried over sodium sulfate and concentrated. The crude residue was purified by column chromatography (10% EtOAc/petroleum to 25% MeOH/ CH_2Cl_2) to afford crude secondary amine as a colorless foam. The crude amine was used immediately.

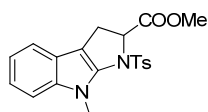
Formalin (37%, 5.5 μL , 0.0734 mmol, 5.2 equiv) followed by solid sodium triacetoxyborohydride (15.6 mg, 0.0734 mmol, 5.2 equiv) were added to a solution of **6** (5.0 mg, 0.0145 mmol, 1 equiv) in acetonitrile (700 μL) at $23\text{ }^{\circ}\text{C}$ and placed under an argon atmosphere. After 30 min, a solution of methanol (5:95) in dichloromethane saturated with ammonia was added slowly. After 5 min, the resulting slurry was concentrated under reduced pressure and the residue was purified by flash column chromatography (1% methanol in dichloromethane saturated with ammonia) to afford (\pm)-folicanthine **1** as a white solid (100 %). All spectral data were in agreement with the literature.

1,2,3,3a,8,8a-hexahydro-3a-(1,2,3,3a,8,8a-hexahydro-8-methyl-1-tosylpyrrolo[2,3-b]indol-3a-yl)-8-methyl-1-tosylpyrrolo[2,3-b]indole **5**: Compound **5** was isolated in 90% yield as a white solid following the general procedure **B**. Reaction time: 1h. mp: $149\text{-}152\text{ }^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.36 (d, $J = 8.0$ Hz, 4H), 7.21-7.27 (m, 6H), 6.86 (d, $J = 7.6$ Hz, 2H), 6.65 (t, $J = 4.8$ Hz, 2H), 6.50 (d, $J = 7.6$ Hz, 2H), 5.05 (s, 2H), 3.21-3.25 (m, 2H), 2.99 (s, 6H), 2.74-2.76 (m, 2H), 2.44 (s, 6H)

1.79-1.87 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 151.6, 142.9, 136.1, 129.9, 129.5, 127.7, 127.2, 124.6, 117.7, 106.9, 87.23, 60.4, 47.4, 32.6, 31.5, 21.5; HRMS (ESI): calc for $\text{C}_{36}\text{H}_{38}\text{N}_4\text{O}_4\text{S}_2$ $[\text{M}+1]^+$: 655.2407 found: 655.2401 IR (neat, cm^{-1}): 3398, 2951, 1602, 1491, 1342, 1157, 1012, 752, 660.

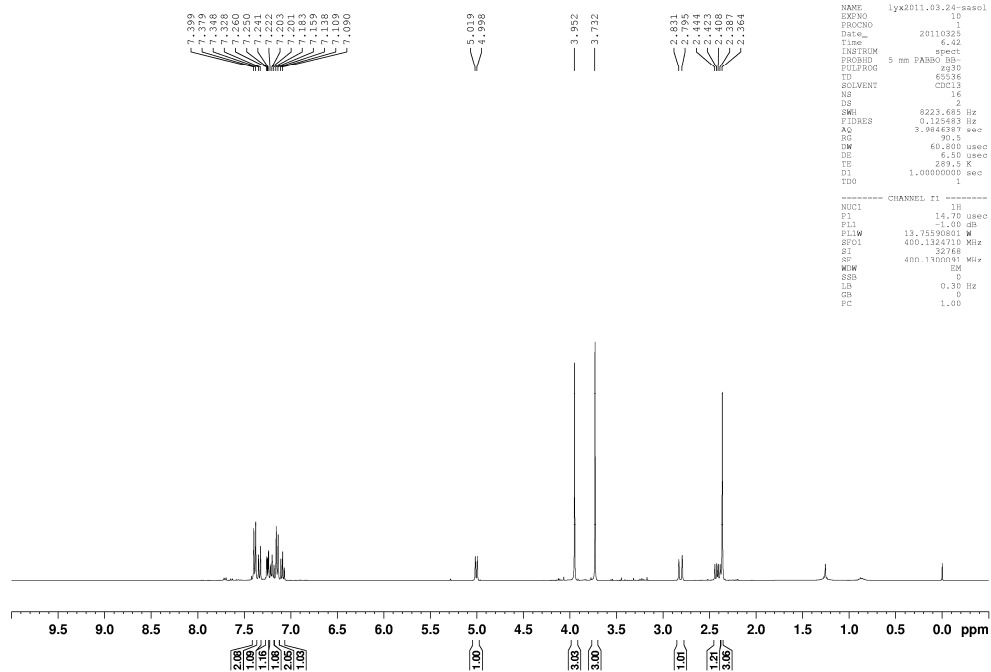
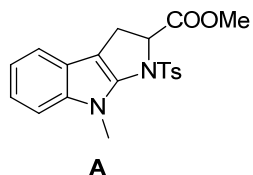
1,2,3,3a,8,8a-hexahydro-3a-(1,2,3,3a,8,8a-hexahydro-8-methylpyrrolo[2,3-b]indol-3a-yl)-8-methylpyrrolo[2,3-b]indole **6** Compound **6** was isolated in 56% yield as a white solid, Mp: 186-188°C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.17 (d, $J = 7.2$ Hz, 2H), 7.08 (t, $J = 7.6$ Hz, 2H), 6.57 (t, $J = 7.2$ Hz, 2H), 6.29 (d, $J = 8.0$, 2H), 4.38 (s, 2H), 2.98-3.00 (m, 2H), 2.80 (s, 6H), 2.42-2.49 (m, 4H), 2.12-2.16 (m, 2H), 1.81 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 152.6, 131.4, 128.5, 124.2, 116.1, 104.8, 87.5, 62.1, 45.8, 38.7, 31.1; HRMS (ESI): calc for $\text{C}_{22}\text{H}_{26}\text{N}_4$ $[\text{M}+1]^+$: 347.2230 found: 347.2236 IR (neat, cm^{-1}): 3395, 2925, 1603, 1494, 1383, 748.

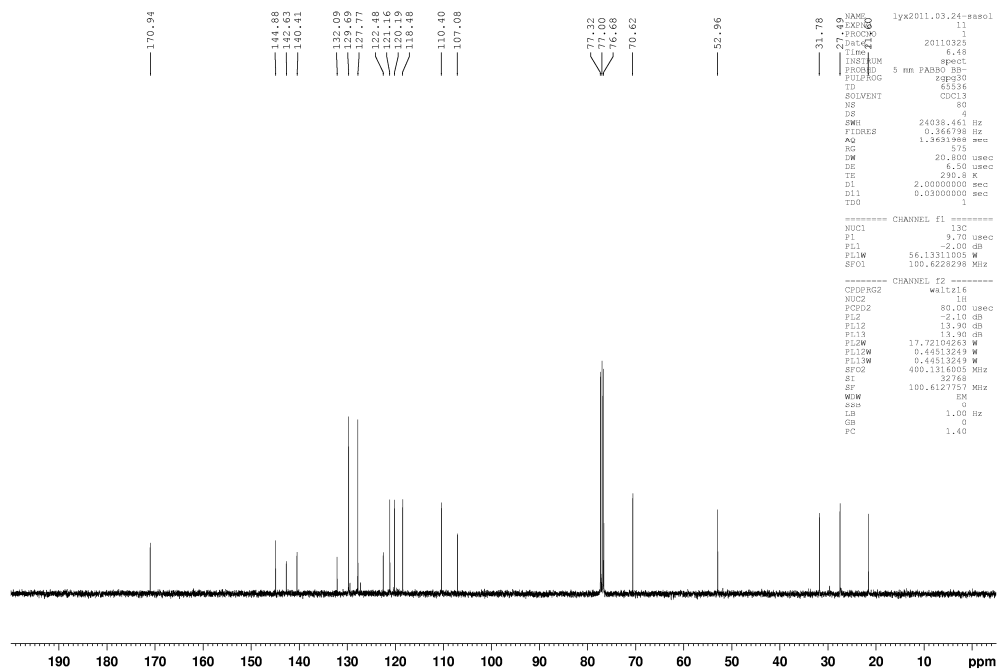
1,2,3,3a,8,8a-hexahydro-3a-(1,2,3,3a,8,8a-hexahydro-1,8-dimethylpyrrolo[2,3-b]indol-3a-yl)-1,8-dimethylpyrrolo[2,3-b]indole **1** Compound **1** was isolated in 99% yield as a white solid, Mp: 164-167°C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 6.91-6.99 (m, 4H), 6.48-6.51 (t, 2H), 6.26 (d, $J = 7.6$ Hz, 2H), 4.39 (s, 2H), 2.99 (s, 6H), 2.64 (s, 2H), 2.40-2.47 (m, 10H), 1.94-1.98 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 152.8, 132.7, 128.0, 123.6, 116.6, 105.8, 91.9, 62.6, 52.6, 37.8, 35.4, 35.2; HRMS (ESI): calc for $\text{C}_{24}\text{H}_{30}\text{N}_4$ $[\text{M}+1]^+$: 375.2543 found: 375.2547 IR (neat, cm^{-1}): 3397, 2912, 2787, 1602, 1492, 1381, 1155, 1022, 741.



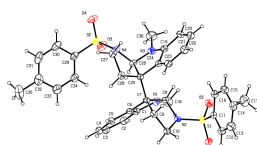
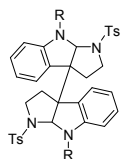
A

Compound **A** was isolated in 61% yield as a white solid ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.40-7.38 (d, $J = 8$ Hz, 2H), 7.35-7.33 (d, $J = 8$ Hz, 1H), 7.26-7.24 (t, $J = 4$ Hz, 1H), 7.22-7.20 (m, 1H), 7.18-7.09 (m, 3H), 5.02-5.00 (d, $J = 8.4$ Hz, 1H), 3.95 (s, 1H), 3.73 (s, 1H), 2.83-2.80 (d, $J = 14.4$ Hz, 1H), 2.44-2.39 (q, 1H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 170.9, 144.9, 142.6, 140.4, 132.1, 129.7, 127.8, 122.5, 121.2, 120.2, 118.5, 110.4, 107.1, 70.6, 53.0, 31.8, 27.5, 21.6; HRMS (ESI): calc for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_4\text{S}$ $[\text{M}+1]^+$: 385.1217 found: 385.1223.





Crystallographic data of 6



checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: c2c

Bond precision:	C-C = 0.0067 Å	Wavelength=0.71073	
Cell:	a=31.563 (7)	b=12.194 (3)	c=19.897 (4)
	alpha=90	beta=102.099 (3)	gamma=90
Temperature:	296 K		
	Calculated	Reported	
Volume	7488 (3)	7488 (3)	
Space group	C 2/c	C2/c	
Hall group	-C 2yc	?	
Moisty formula	C36 H38 N4 O4 S2	?	
Sum formula	C36 H38 N4 O4 S2	C36 H38 N4 O4 S2	
Mr	654.84	654.82	
Dx, g cm ⁻³	1.162	1.162	
Z	8	8	
Mu (mm ⁻¹)	0.183	0.183	
F000	2768.0	2768.0	
F000'	2771.04		
h, k, lmax	38, 14, 24	37, 14, 24	
Nref	6990	6776	
Tmin, Tmax	0.955, 0.961	0.956, 0.961	
Tmin'	0.955		

Correction method= MULTI-SCAN

Data completeness= 0.969 Theta(max)= 25.500

R(reflections)= 0.0653 (3339) wR2(reflections)= 0.2020 (6776)

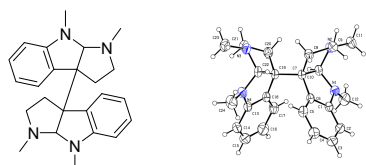
S = 0.976 Npar= 419

The following ALERTS were generated. Each ALERT has the format
test-name ALERT alert-type alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT026_ALERT_3_C	Ratio Observed / Unique Reflections too Low	49 Perc.
PLAT029_ALERT_3_C	_diffn_measured_fraction_theta_full Low	0.97
PLAT094_ALERT_2_C	Ratio of Maximum / Minimum Residual Density	2.68
PLAT220_ALERT_2_C	Large Non-Solvent C Ueq(max)/Ueq(min) ...	3.44 Ratio

Crystallographic data of 7



checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

No syntax errors found CIF dictionary Interpreting this report

Datablock: c2c

Bond precision: C-C = 0.0034 Å Wavelength=0.71073

Cell: a=28.442(8) b=16.060(5) c=13.506(4)
 alpha=90 beta=90.289(3) gamma=90
Temperature: 296 K

	Calculated	Reported
Volume	6169(3)	6169(3)
Space group	C 2/c	C2/c
Hall group	-C 2yc	?
Moiety formula	C24 H30 N4	?
Sum formula	C24 H30 N4	C36 H45 N6
Mr	374.52	561.78
Dx, g cm-3	1.210	1.210
Z	12	8
Mu (mm-1)	0.073	0.073
F000	2424.0	2424.0
F000'	2424.74	
h,k,lmax	34, 19, 16	34, 19, 16
Nref	5748	5714
Tmin, Tmax	0.982, 0.984	0.982, 0.984
Tmin'	0.982	

Correction method= MULTI-SCAN

Data completeness= 0.994 Theta(max)= 25.490

R(reflections)= 0.0526(3055) wR2(reflections)= 0.1406(5714)

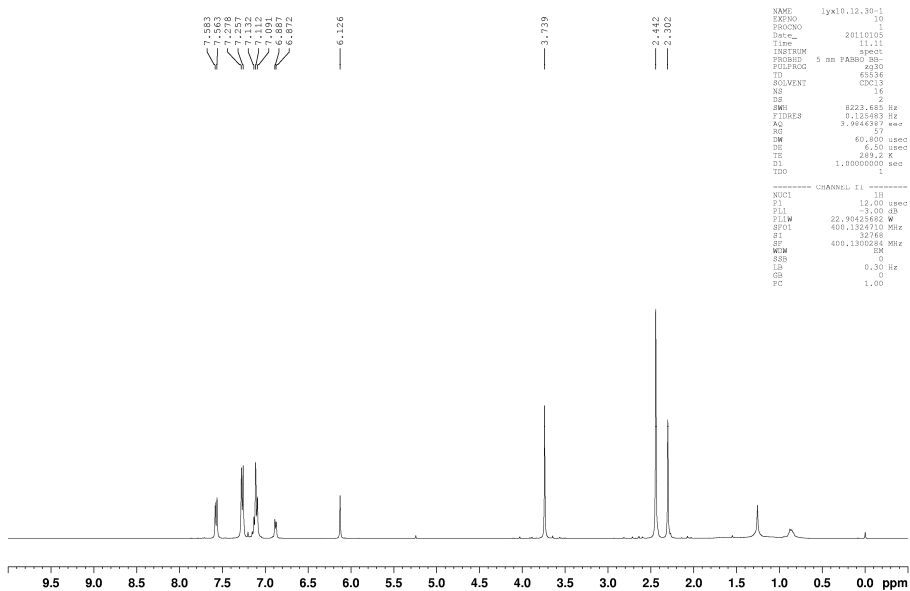
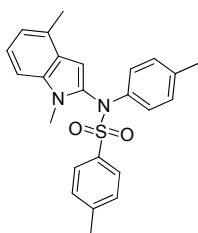
S = 1.006 Npar= 385

The following ALERTS were generated. Each ALERT has the format
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Click on the hyperlinks for more details of the test.



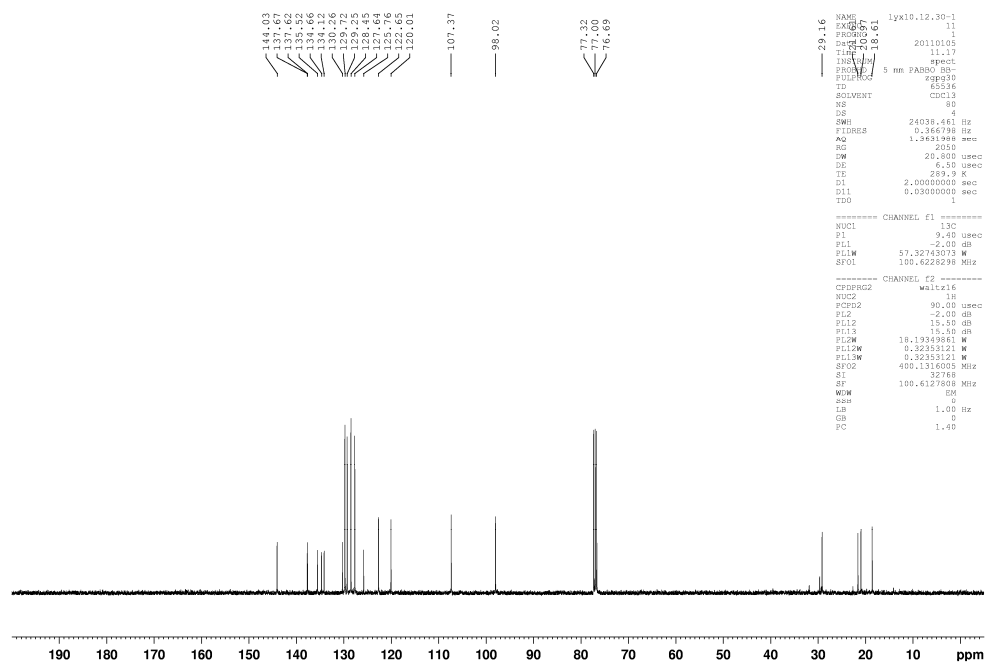
Alert level C

PLAT045_ALERT_1_C Calculated and Reported Z Differ by 1.50 Ratio
PLAT230_ALERT_2_C Hirshfeld Test Diff for N5 -- C33 .. 7.00 Å



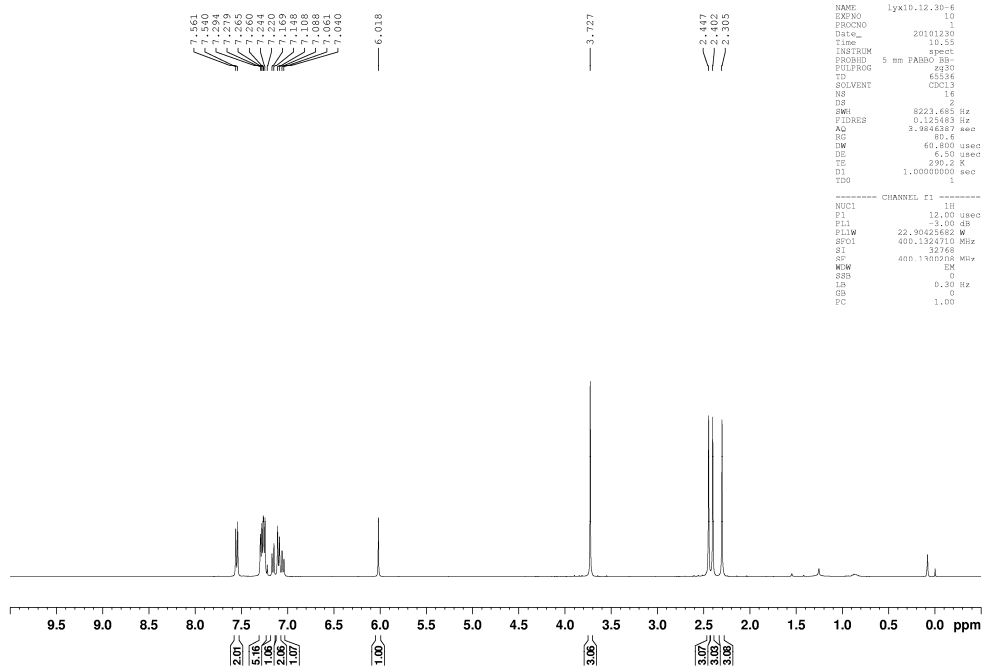
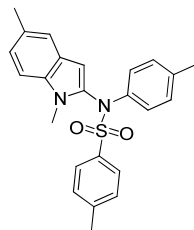
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PROCNO 1
Date_ 20110105
Time 11.11
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 8223.685 Hz
FIDRES 0.124863 Hz
AQ 3.9844397 sec
RG 327.500
DE 6.50 usec
TE 289.2 K
D1 1.8000000 sec
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D99 1
D100 1
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PC 1.00
    
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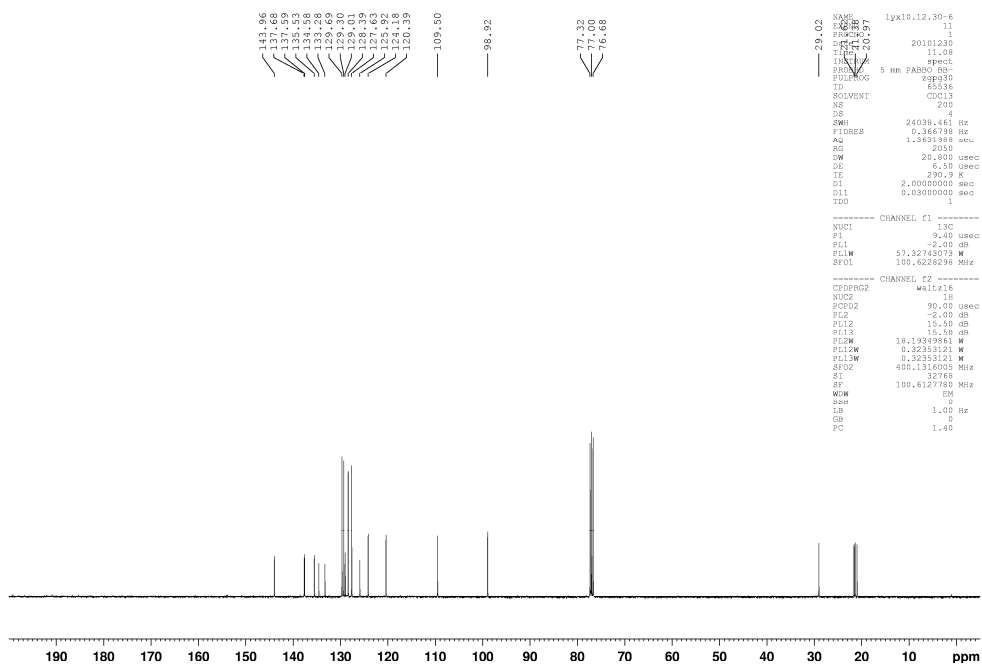
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SOLVENT CDCl3
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DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
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RG 327.500
DE 6.50 usec
TE 289.2 K
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D11 0.0300000 sec
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PROCNO 1
Date_ 20110105
Time 11.17
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3951988 sec
RG 327.500
DE 6.50 usec
TE 289.2 K
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D11 0.0300000 sec
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EXPNO 10
PROCNO 1
Date_ 20110105
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
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DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
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RG 327.500
DE 6.50 usec
TE 289.2 K
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NAME lyx10.12.30-6
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 PROCNO 1
 DSQU 20101230
 T2Ac 10.55
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65336
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
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 DW 60.800 usec
 DE 6.50 usec
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 DI 1.00000000 sec
 TDO 1

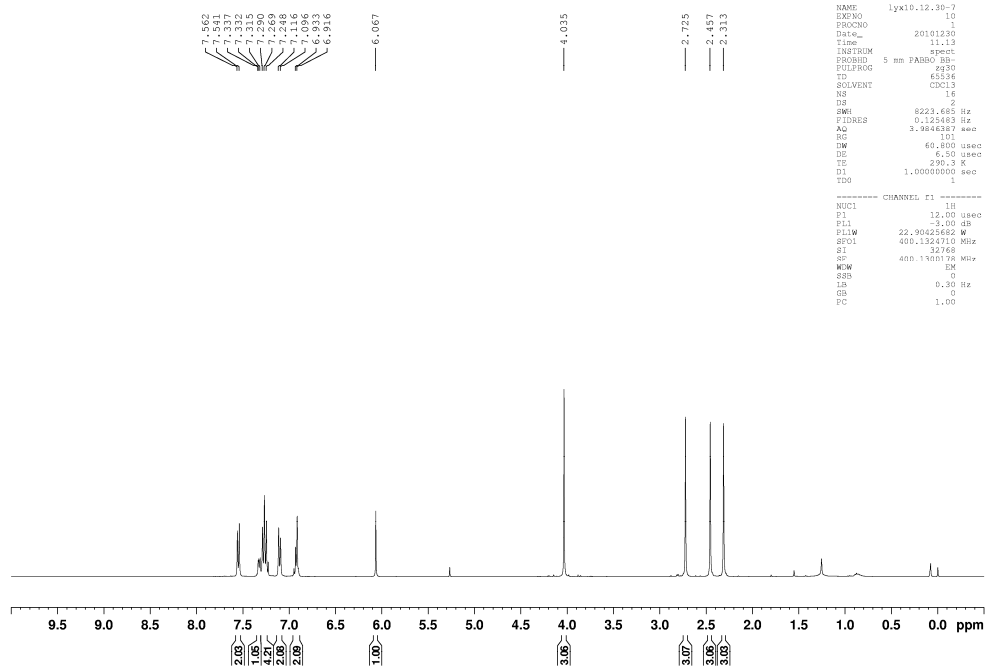
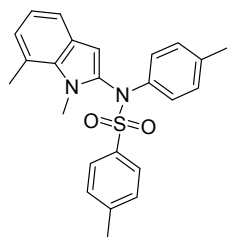
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 SF 400.13160500 MHz
 MCHW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



NAME lyx10.12.30-6
 INPRO 1
 PROCNO 1
 DSQU 20101230
 T2Ac 11.00
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65336
 SOLVENT CDCl3
 NS 209
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.166798 Hz
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 RG 2403
 DW 28.800 usec
 DE 6.50 usec
 TE 299.9 K
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 TDO 1

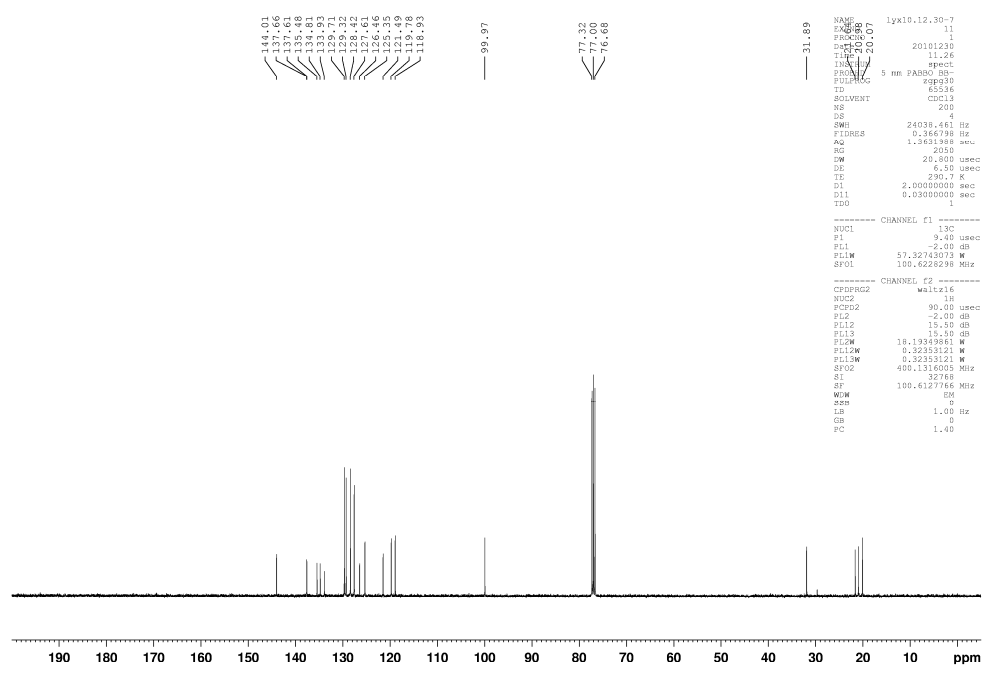
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 POC2 90.00 usec
 PL2 -2.00 dB
 PL2 15.00 dB
 PL23 15.50 dB
 PL2M 18.13949461 M
 PL2W 0.32353121 W
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 SSB 0
 LB 1.00 Hz
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 PC 1.40



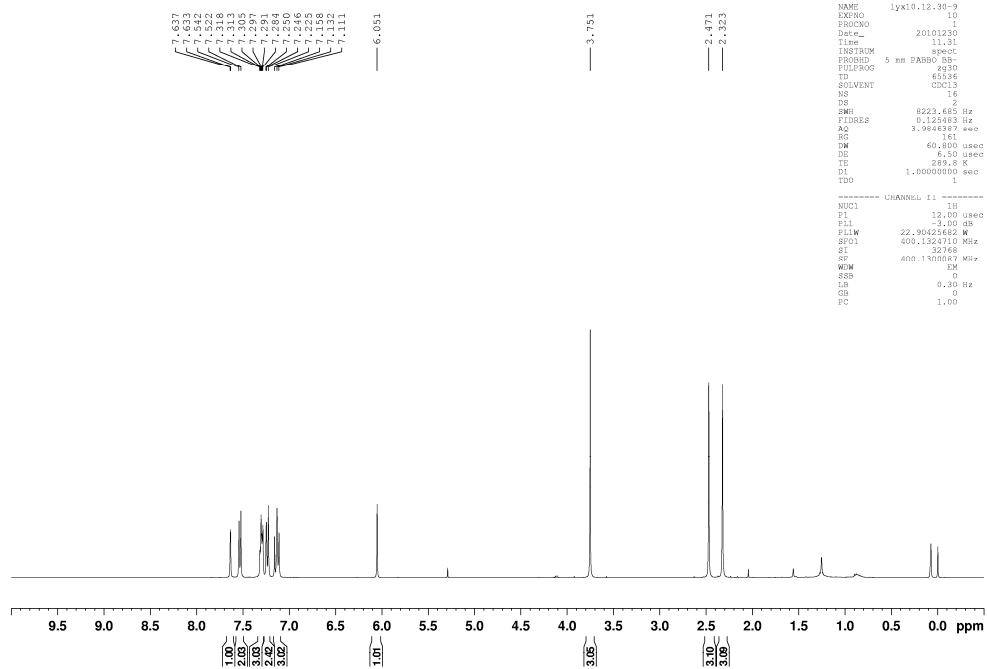
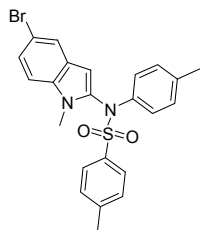
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Date_ 20101230
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TD 65336
SOLVENT CDCl3
NS 16
DS 4
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 1.864687 sec
RG 101
AQ 1.864687 sec
DE 6.50 usec
TE 290.2 K
D1 1.00000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
PL1W 22.90425682 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1324710 MHz
MCHW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
    
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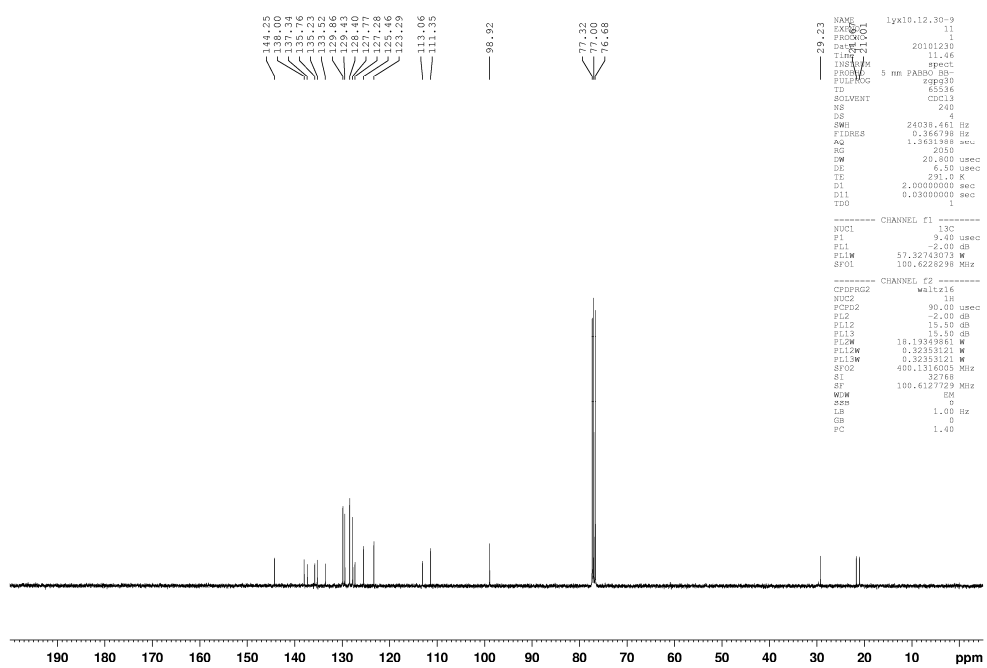
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NAME lyx10.12.30-7
EXPNO 1
PROCNO 1
Date_ 20101230
Time 11.24
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PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 4
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2400
AQ 1.3631988 sec
DE 6.50 usec
TE 290.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 13C
P1 9.40 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6262308 MHz
----- CHANNEL f2 -----
NAME waltz16
EXPNO 1
PROCNO 1
Date_ 20101230
Time 11.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 4
DS 4
SWH 100.6127786 MHz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2400
AQ 1.3631988 sec
DE 6.50 usec
TE 290.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
----- CHANNEL f2 -----
NUC1 13C
P1 9.40 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6262308 MHz
----- CHANNEL f2 -----
NAME waltz16
EXPNO 1
PROCNO 1
Date_ 20101230
Time 11.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 4
DS 4
SWH 100.6127786 MHz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2400
AQ 1.3631988 sec
DE 6.50 usec
TE 290.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
----- CHANNEL f2 -----
NUC1 13C
P1 9.40 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6262308 MHz
    
```



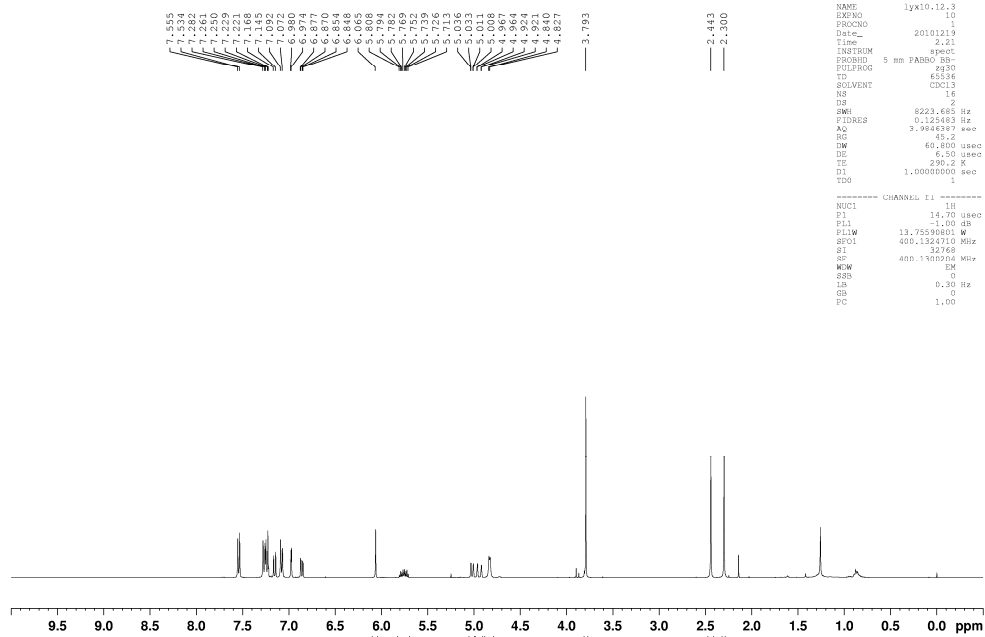
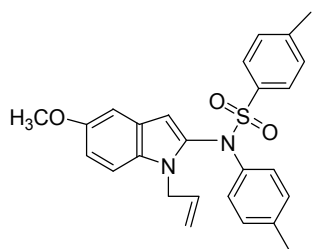
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NAME lyx10.12.30-9
EXPNO 1
PROCNO 1
Date_ 20101230
Time 11.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 15
DS 4
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.384387 sec
RG 161
TM 60.000 usec
DE 6.50 usec
TE 289.0 K
D1 1.0000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
PL12 22.90425662 M
SFO1 400.132010 MHz
SI 32768
SF 400.132010 MHz
WDM EX
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
    
```



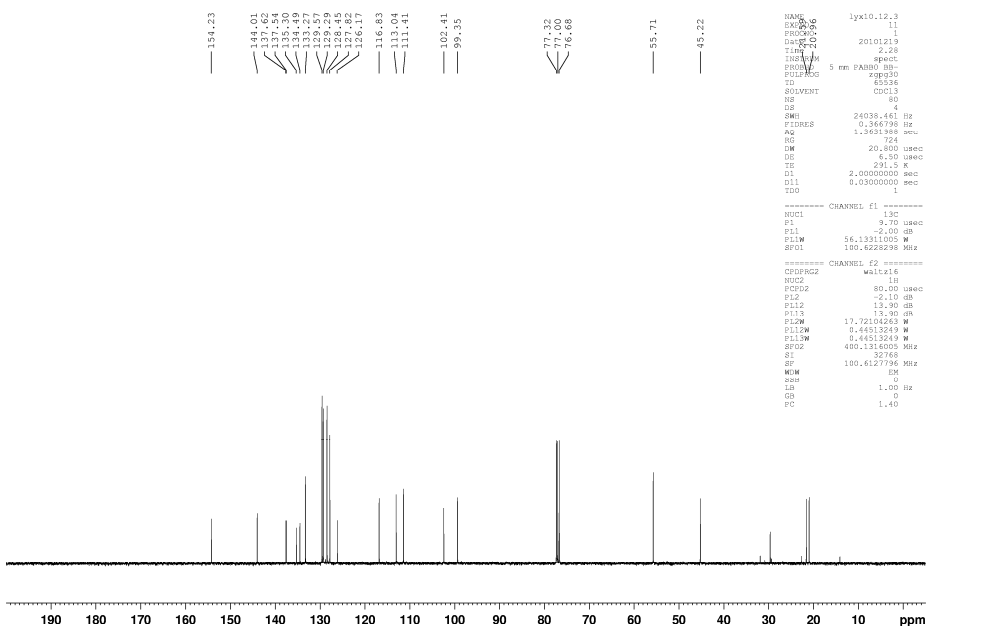
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NAME lyx10.12.30-9
EXPNO 1
PROCNO 1
Date_ 20101230
Time 11.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.356198 sec
RG 2400
DM 28.800 usec
DE 6.50 usec
TE 291.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 13C
P1 9.40 usec
PL1 -2.00 dB
PL12 57.32743073 M
SFO1 100.6228298 MHz
----- CHANNEL f2 -----
NAME waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL14 15.00 dB
PL15 15.00 dB
PL16 15.00 dB
PL17 15.00 dB
PL18 15.00 dB
PL19 15.00 dB
PL20 15.00 dB
PL21 15.00 dB
PL22 15.00 dB
PL23 15.00 dB
PL24 15.00 dB
PL25 15.00 dB
PL26 15.00 dB
PL27 15.00 dB
PL28 15.00 dB
PL29 15.00 dB
PL30 15.00 dB
PL31 15.00 dB
PL32 15.00 dB
PL33 15.00 dB
PL34 15.00 dB
PL35 15.00 dB
PL36 15.00 dB
PL37 15.00 dB
PL38 15.00 dB
PL39 15.00 dB
PL40 15.00 dB
PL41 15.00 dB
PL42 15.00 dB
PL43 15.00 dB
PL44 15.00 dB
PL45 15.00 dB
PL46 15.00 dB
PL47 15.00 dB
PL48 15.00 dB
PL49 15.00 dB
PL50 15.00 dB
PL51 15.00 dB
PL52 15.00 dB
PL53 15.00 dB
PL54 15.00 dB
PL55 15.00 dB
PL56 15.00 dB
PL57 15.00 dB
PL58 15.00 dB
PL59 15.00 dB
PL60 15.00 dB
PL61 15.00 dB
PL62 15.00 dB
PL63 15.00 dB
PL64 15.00 dB
PL65 15.00 dB
PL66 15.00 dB
PL67 15.00 dB
PL68 15.00 dB
PL69 15.00 dB
PL70 15.00 dB
PL71 15.00 dB
PL72 15.00 dB
PL73 15.00 dB
PL74 15.00 dB
PL75 15.00 dB
PL76 15.00 dB
PL77 15.00 dB
PL78 15.00 dB
PL79 15.00 dB
PL80 15.00 dB
PL81 15.00 dB
PL82 15.00 dB
PL83 15.00 dB
PL84 15.00 dB
PL85 15.00 dB
PL86 15.00 dB
PL87 15.00 dB
PL88 15.00 dB
PL89 15.00 dB
PL90 15.00 dB
PL91 15.00 dB
PL92 15.00 dB
PL93 15.00 dB
PL94 15.00 dB
PL95 15.00 dB
PL96 15.00 dB
PL97 15.00 dB
PL98 15.00 dB
PL99 15.00 dB
PL100 15.00 dB
    
```



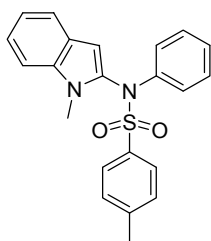
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NAME lyx10.12.3
EXPNO 1
PROCNO 1
Date_ 20101219
Time 2.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.984587 sec
RG 45.2
AQ 40.800 usec
DE 6.50 usec
TE 290.2 K
D1 1.00000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 12.75590801 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300904 MHz
MCHW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
    
```

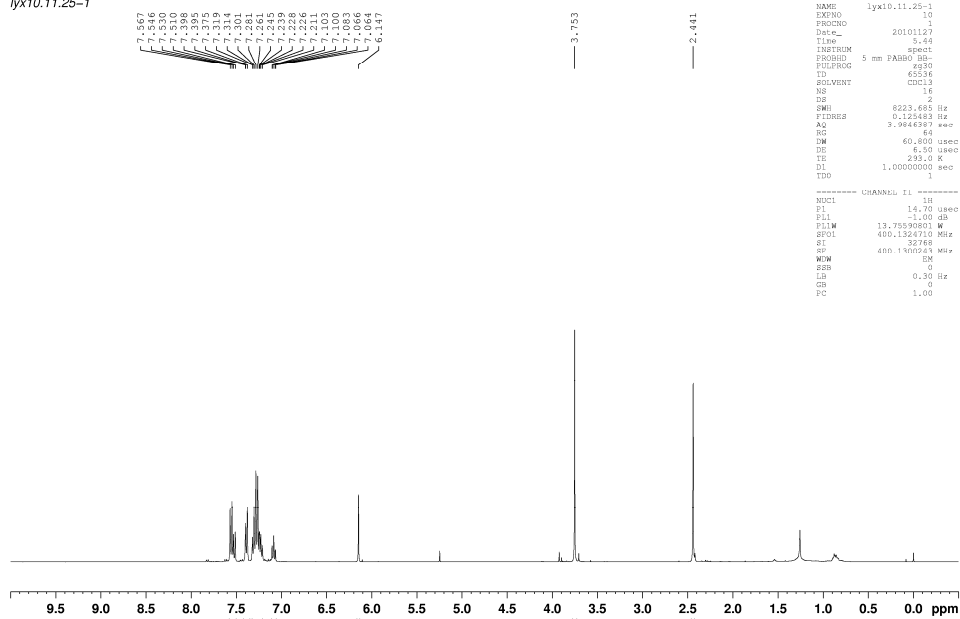


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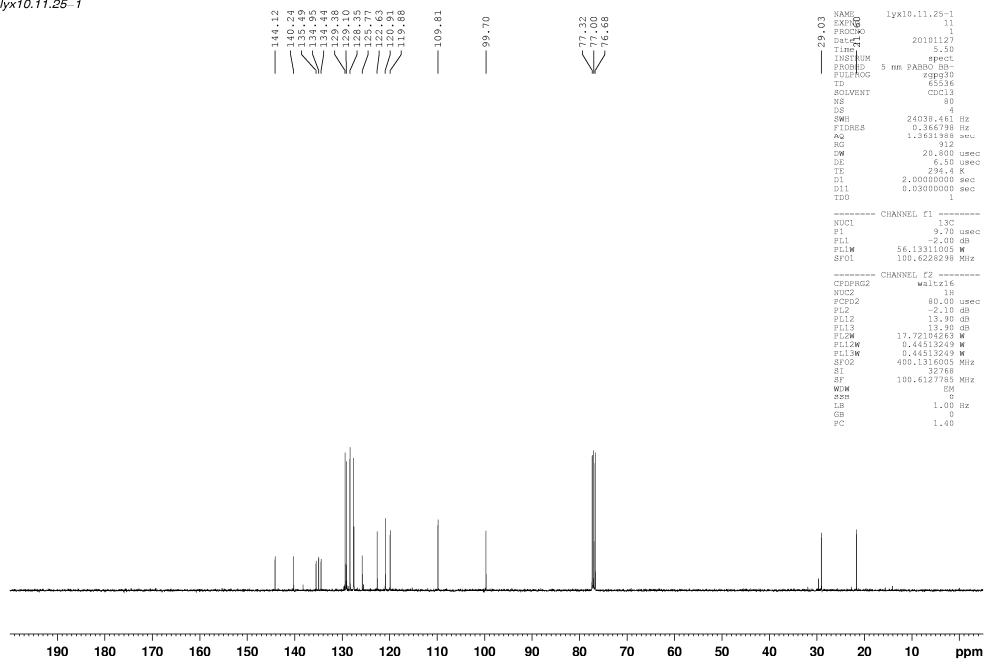
NAME lyx10.12.3
EXPNO 1
PROCNO 1
Date_ 20101219
Time 2.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 24038.461 Hz
FIDRES 0.364798 Hz
AQ 1.385138 sec
RG 724
AQ 20.800 usec
DE 6.50 usec
TE 291.5 K
D1 2.00000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 58.13311008 W
SFO1 100.6262628 MHz
----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL2 13.90 dB
PL3 13.90 dB
PL2W 17.72104263 W
PL3W 0.44513249 W
SFO2 400.1311005 MHz
SI 32768
SF 100.6127796 MHz
MCHW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```

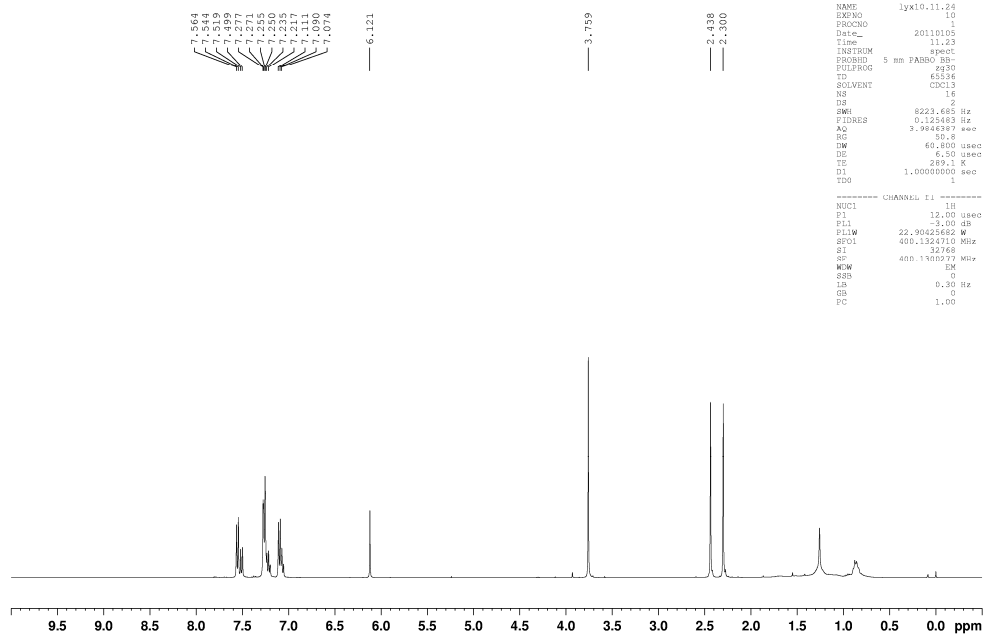
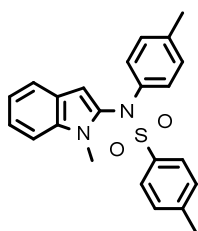


lyx10.11.25-1



lyx10.11.25-1

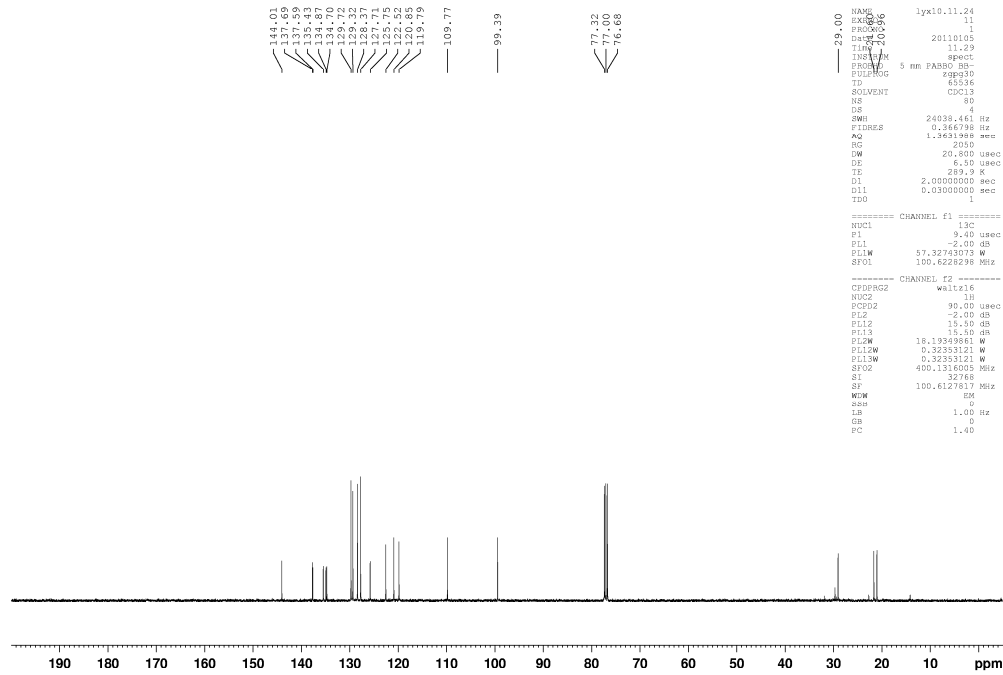




```

NAME lyx10.11.24
EXPNO 11
PROCNO 1
Date_ 20110105
Time 11.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 16
DS 4
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.984887 sec
RG 50.8
AQ 60.800 usec
DE 6.50 usec
TE 289.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
PL1W 22.90425682 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1324710 MHz
MCK EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
    
```

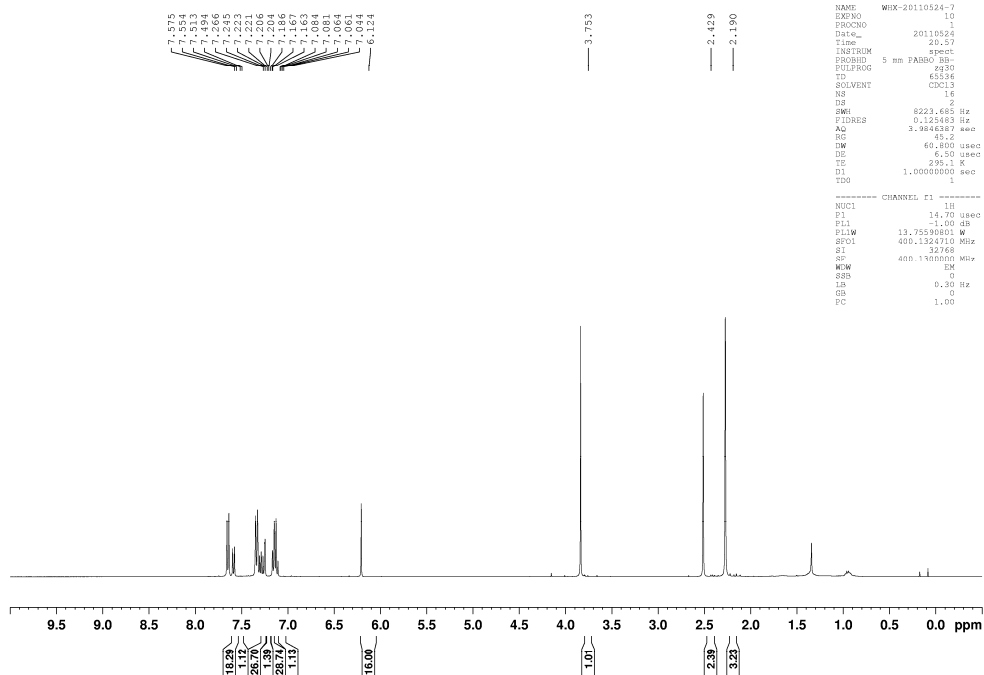
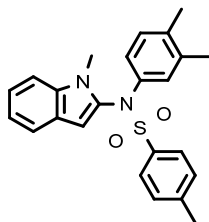


```

NAME lyx10.11.24
EXPNO 11
PROCNO 1
Date_ 20110105
Time 11.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 16
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3661988 sec
RG 2030
AQ 20.800 usec
DE 6.50 usec
TE 289.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

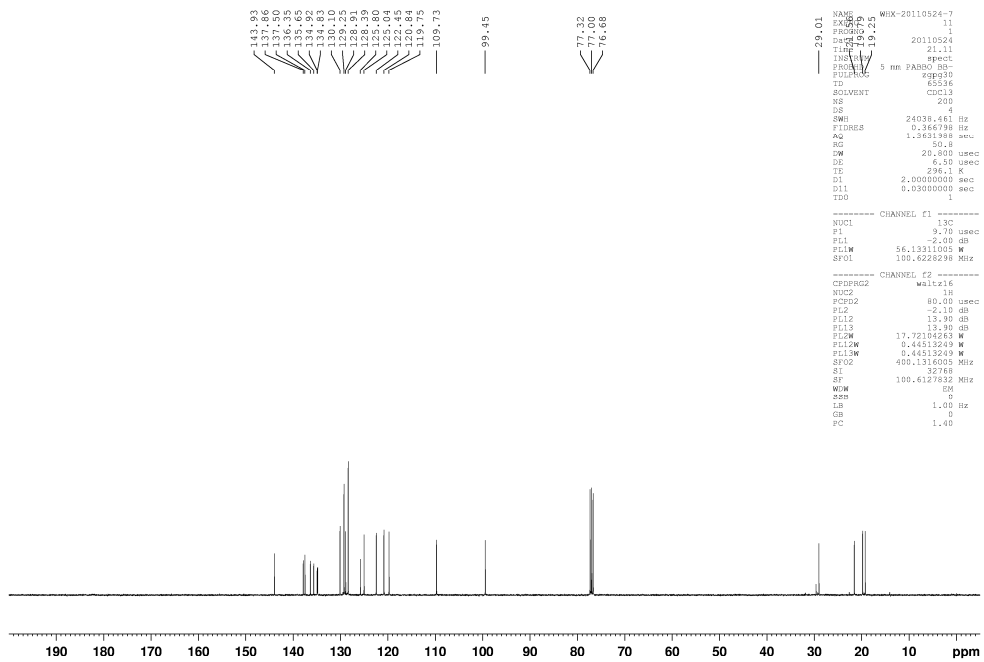
===== CHANNEL f1 =====
NUC1 13C
P1 9.40 usec
PL1 -2.00 dB
PL1W 57.327493073 W
SFO1 100.6228239 MHz

===== CHANNEL f2 =====
CEPRPG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.50 dB
PL13 15.50 dB
PL1W 18.19309863 W
PL1W 0.32353121 W
PL1W 0.32353121 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127813 MHz
MCK EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```



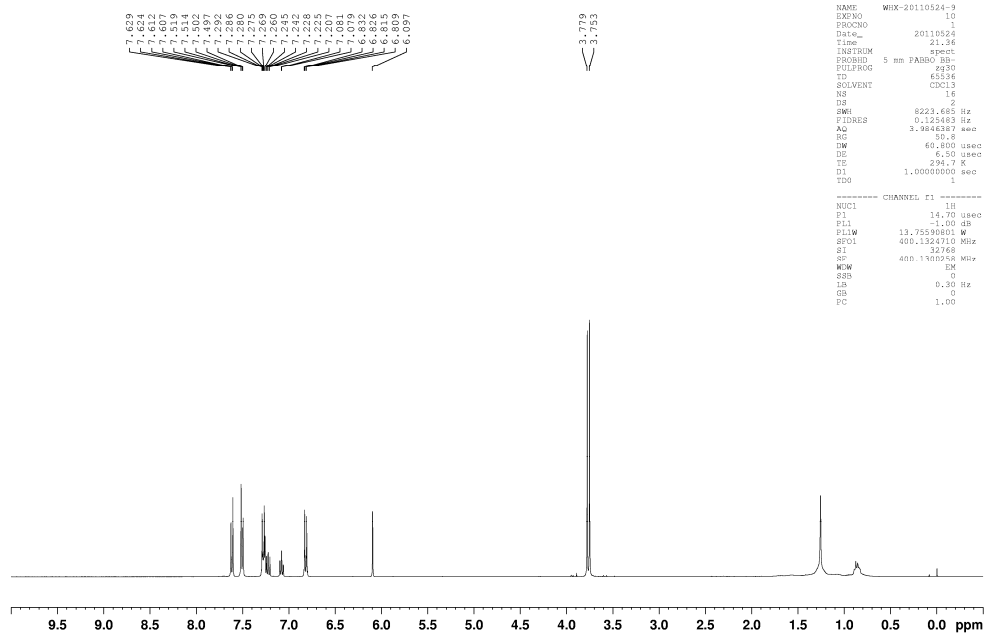
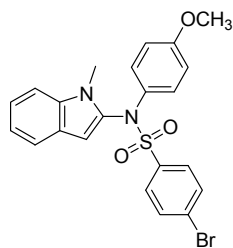
```

NAME      WIX-20110524-7
EXPNO    1
PROCNO   1
Date_    20110524
Time     21.11
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65336
SOLVENT  CDCl3
NS       16
DS       4
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       1.3848287 sec
RG       48.2
RW       46.800 usec
DE       6.50 usec
TE       296.1 K
D1       1.00000000 sec
TD0      1
    
```



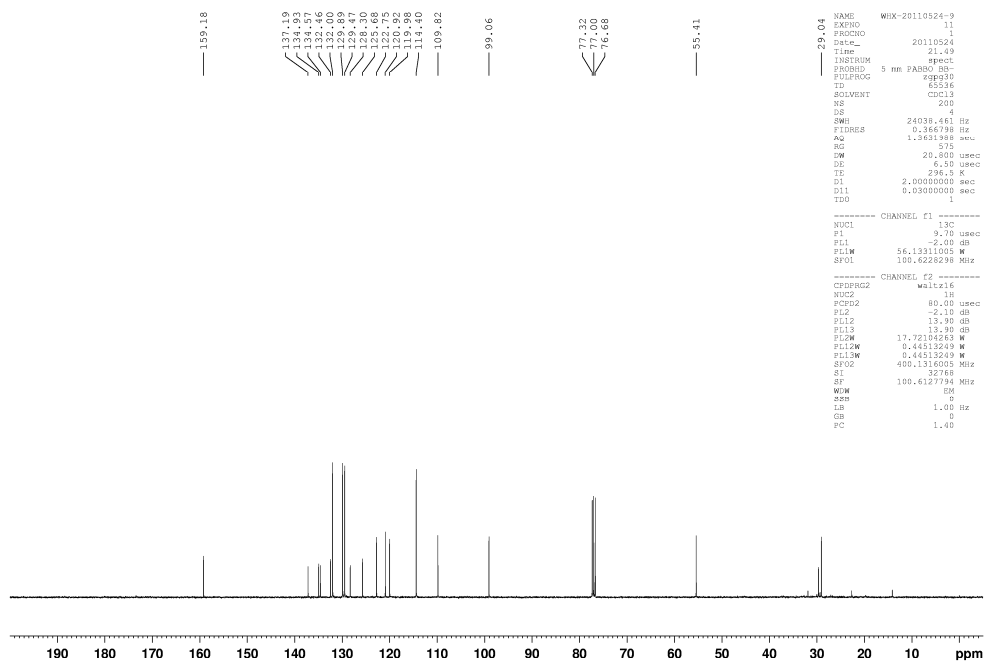
```

NAME      WIX-20110524-7
EXPNO    1
PROCNO   1
Date_    20110524
Time     21.11
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65336
SOLVENT  CDCl3
NS       200
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3961988 sec
RG       50.4
RW       28.800 usec
DE       6.50 usec
TE       296.1 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1
    
```



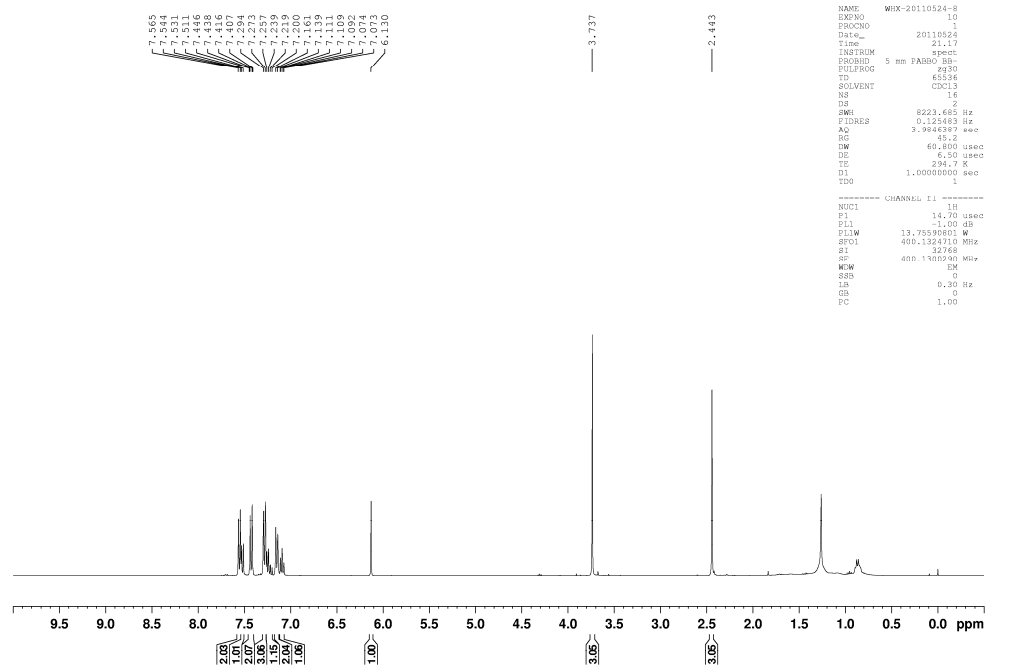
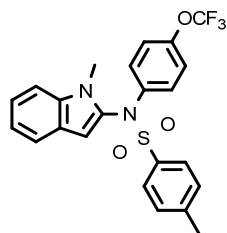
```

NAME      WIX-20110524-9
EXPNO    1
PROCNO   1
Date_    20110524
Time     21.49
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65336
SOLVENT  CDCl3
NS       4
DS       4
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       1.3648587 sec
RG       512
DW       60.800 usec
DE       6.50 usec
TE       294.7 K
D1       1.00000000 sec
TD0      1
----- CHANNEL f1 -----
NUC1     1H
P1       14.70 usec
PL1      -1.00 dB
PL1W     11.75590801 W
SFO1     400.1324710 MHz
SI       32768
SF       400.1300934 MHz
MCHW     EM
SFO2     0
LB       0.30 Hz
GB       0
PC       1.00
    
```



```

NAME      WIX-20110524-9
EXPNO    11
PROCNO   1
Date_    20110524
Time     21.49
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65336
SOLVENT  CDCl3
NS       4
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631998 sec
RG       512
DW       20.800 usec
DE       6.50 usec
TE       296.5 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1
----- CHANNEL f1 -----
NUC1     13C
P1       9.70 usec
PL1      -2.00 dB
PL1W     56.13311095 W
SFO1     100.6228298 MHz
----- CHANNEL f2 -----
NAME      waltz16
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65336
SOLVENT  CDCl3
NS       4
DS       4
SWH      100.6127794 MHz
FIDRES   0
AQ       1.00 Hz
RG       0
DW       1.40
TE       296.5 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1
    
```

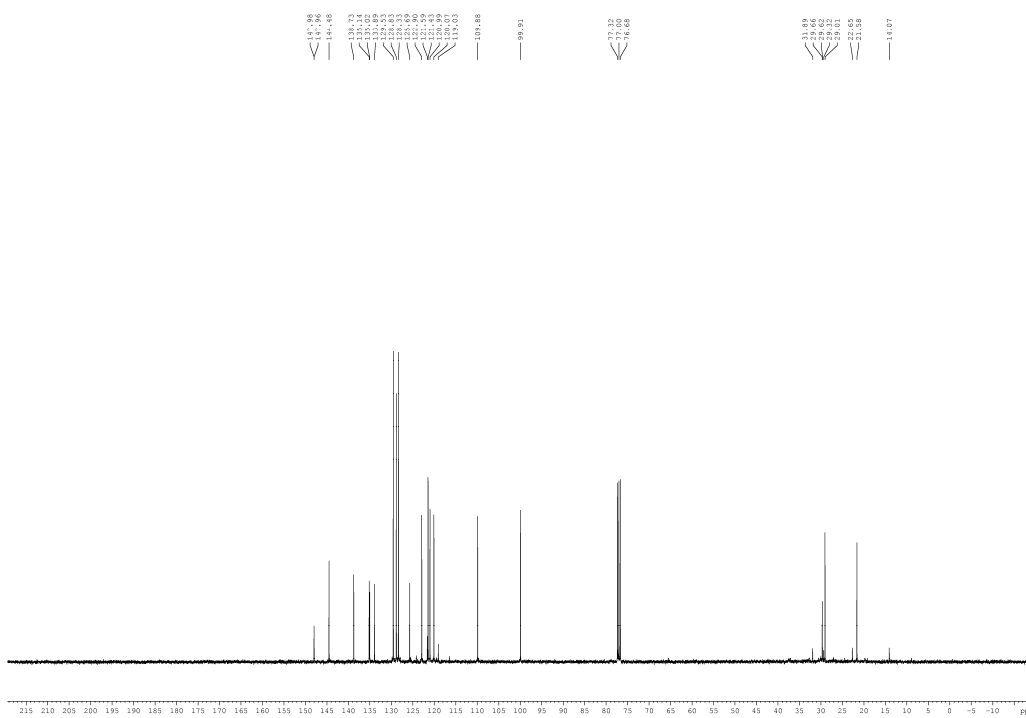


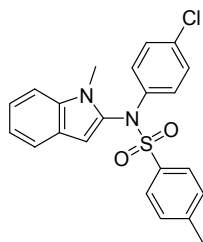
```

NAME      WIX-20110524-8
EXPNO    10
PROCNO   1
Date_    20110524
Time     21.17
INSTRUM  spect
PROBHD   5 mm F400
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        18
DS        2
SMBW     8223.683 Hz
FIDRES   0.125483 Hz
AQ        9.964597 sec
RG        48.2
DM        60.000 usec
DE        6.50 usec
TE        294.2 K
D1        1.00000000 sec
YD0       1

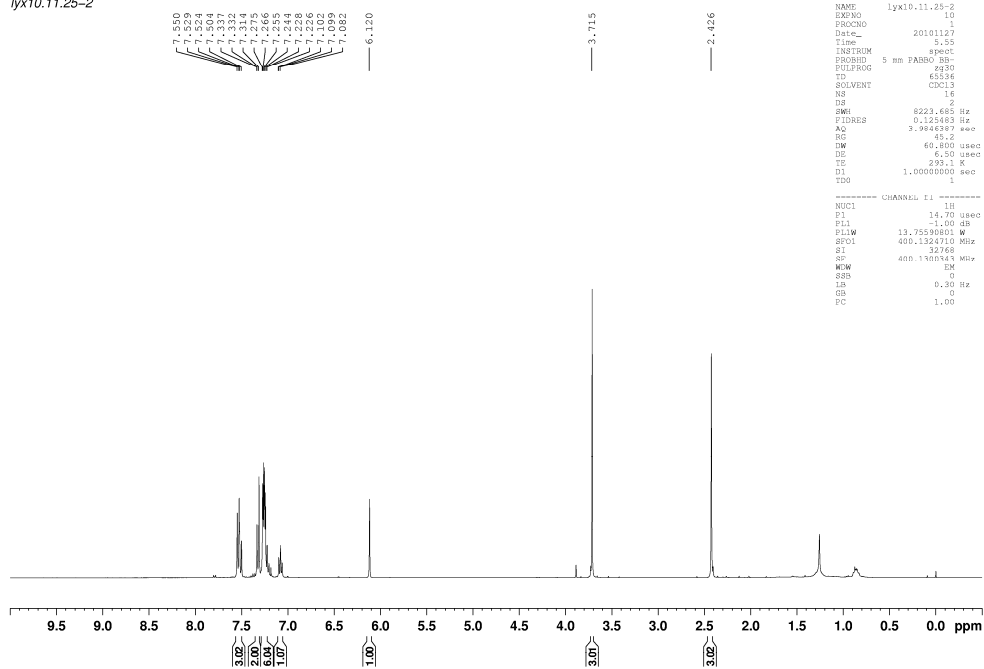
----- CHANNEL f1 -----
NUC1     1H
P1       14.70 usec
PL1      -1.00 dB
PL12     13.75590801 W
SFO1     400.1324710 MHz
SFO2     327.68 MHz
AQ       999.1505940 Mcyc
MCHW     EM
SGB      0
LB       0.30 Hz
GB       0
PC       1.00

```





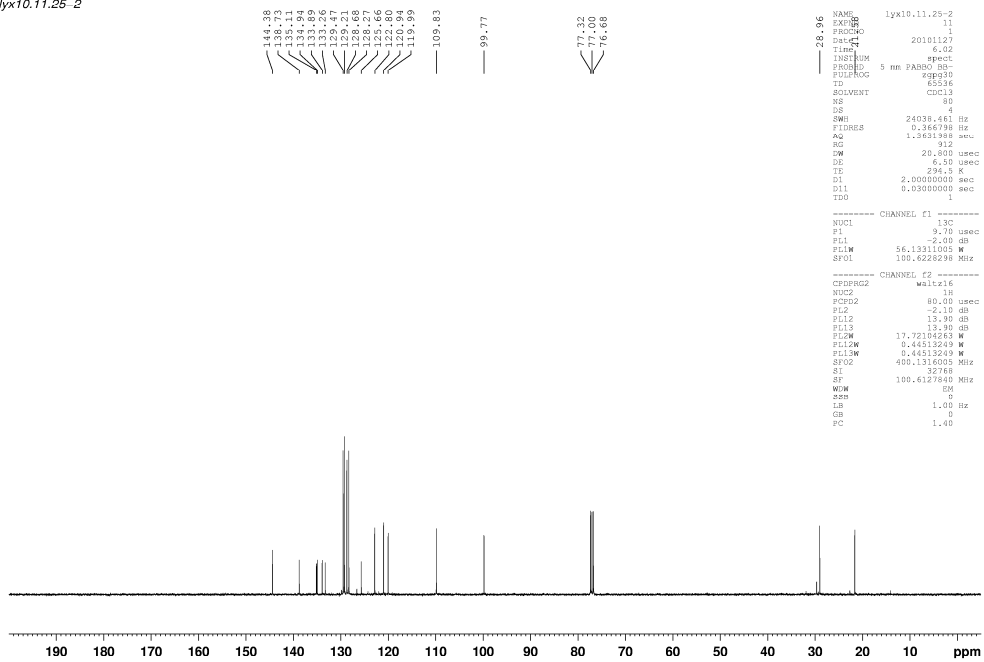
lyx10.11.25-2



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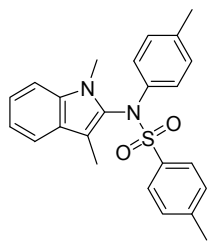
NAME lyx10.11.25-2
EXPNO 1
PROCNO 1
Date_ 20101127
Time 6.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 8
DS 4
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.984887 sec
RG 48.2
AQ 40.800 usec
DE 6.50 usec
TE 293.1 K
D1 1.00000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 12.75390801 W
SFO1 400.1324700 MHz
SI 32768
SF 400.1305644 MHz
MCHW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
    
```

lyx10.11.25-2

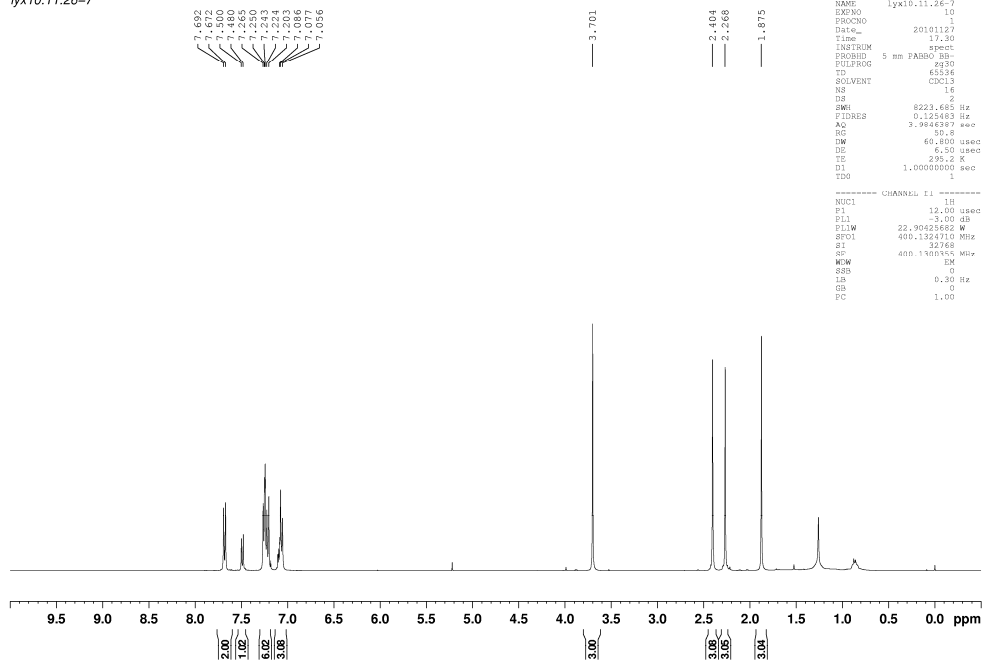


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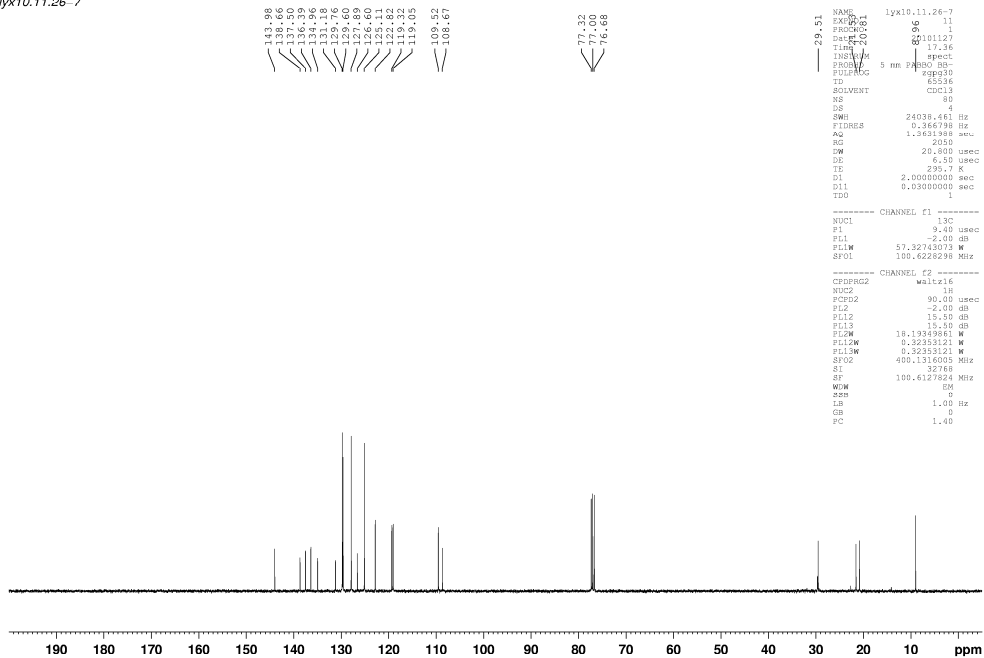
NAME lyx10.11.25-2
EXPNO 1
PROCNO 1
Date_ 20101127
Time 6.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 8
DS 4
SWH 24038.461 Hz
FIDRES 0.266798 Hz
AQ 1.3631988 sec
RG 132
AQ 20.800 usec
DE 6.50 usec
TE 294.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311095 W
SFO1 100.6228298 MHz
----- CHANNEL f2 -----
NAME waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL2 13.90 dB
PL13 13.90 dB
PL1W 17.92104583 W
SFO2 0.44513249 M
SFO2 0.44513249 M
SF02 400.1316035 MHz
SI 32768
SF 100.6127840 MHz
MCHW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```

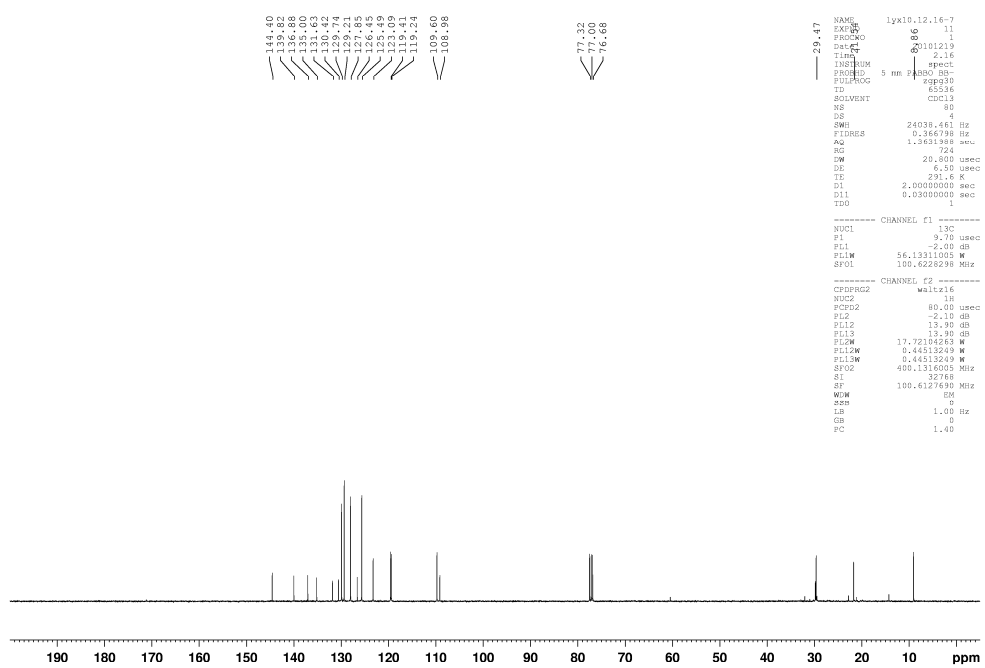
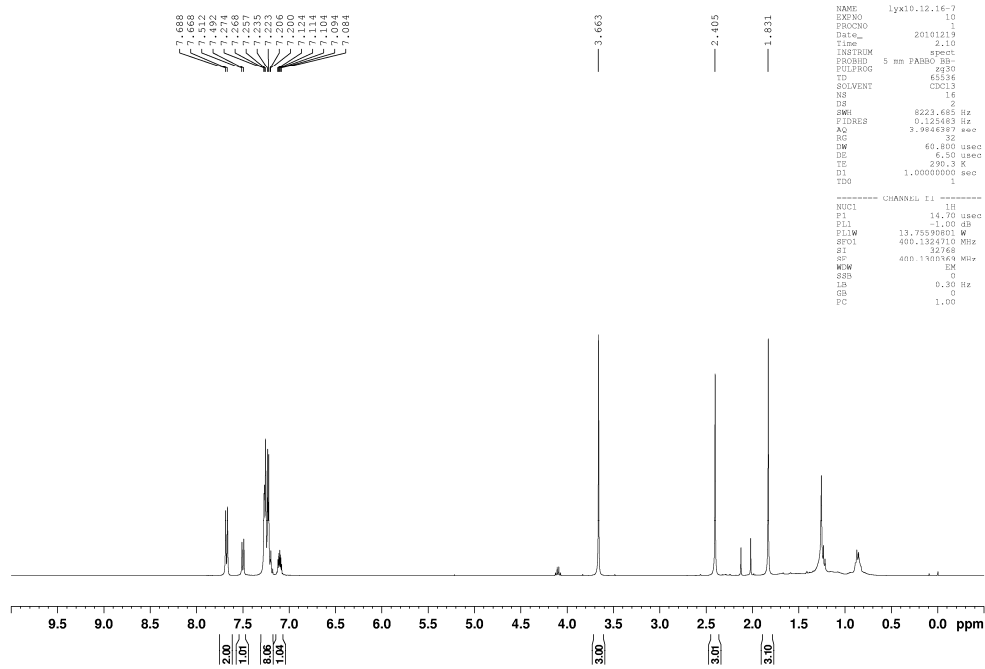
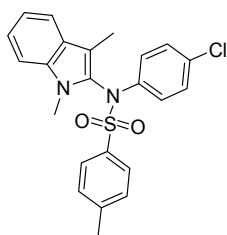


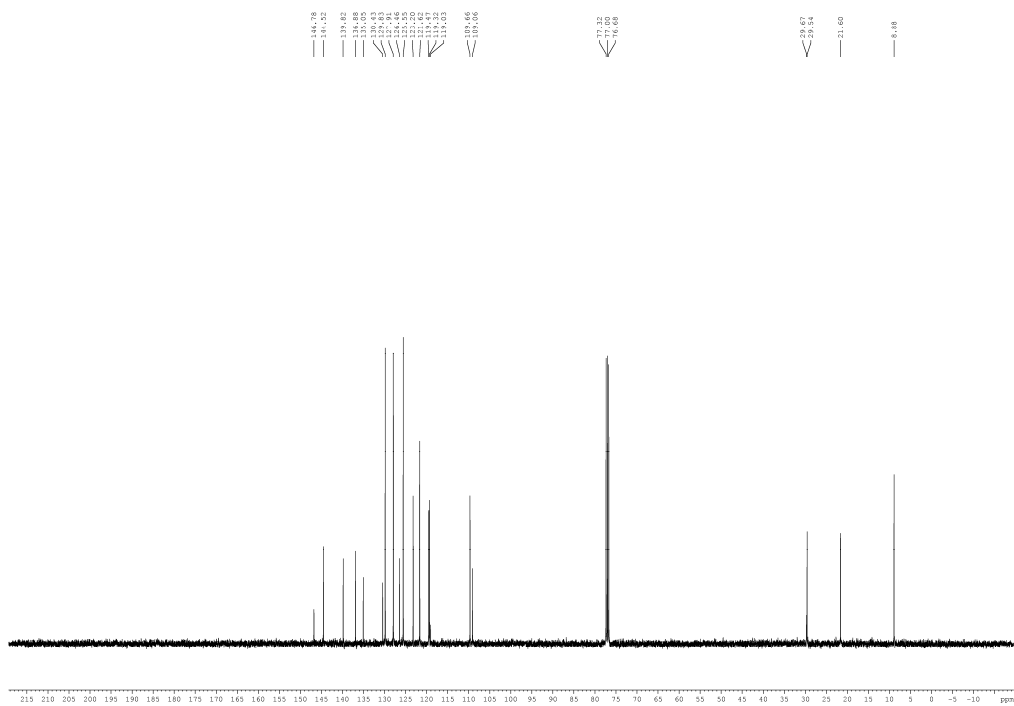
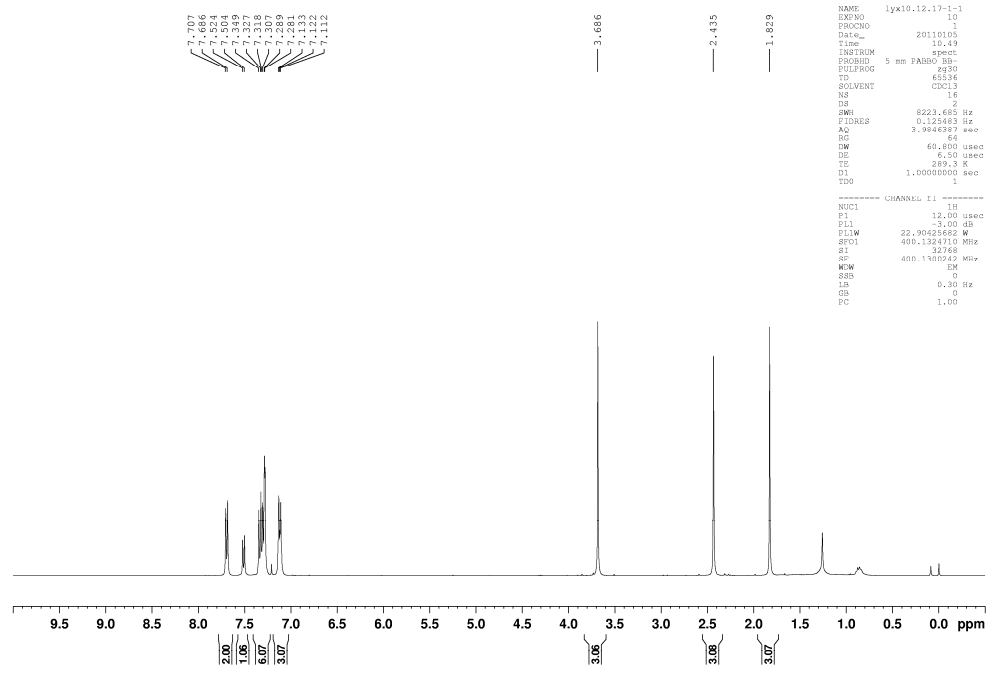
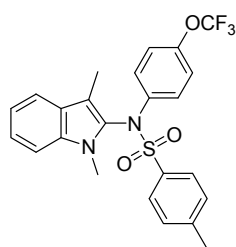
lyx10.11.26-7

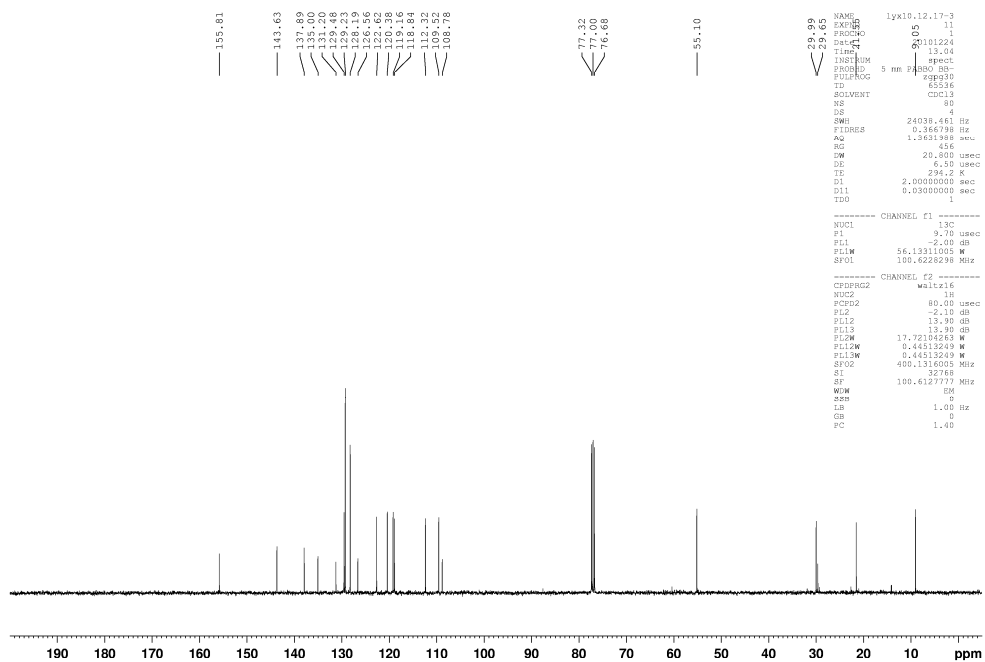
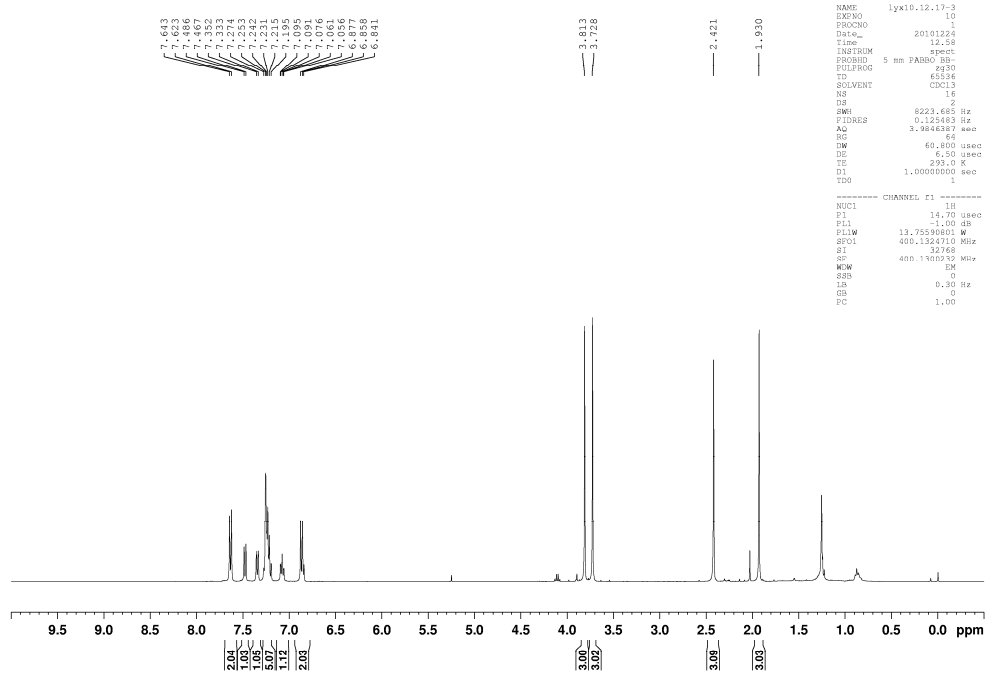
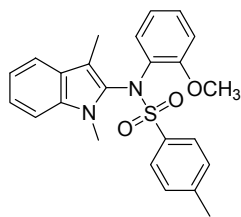


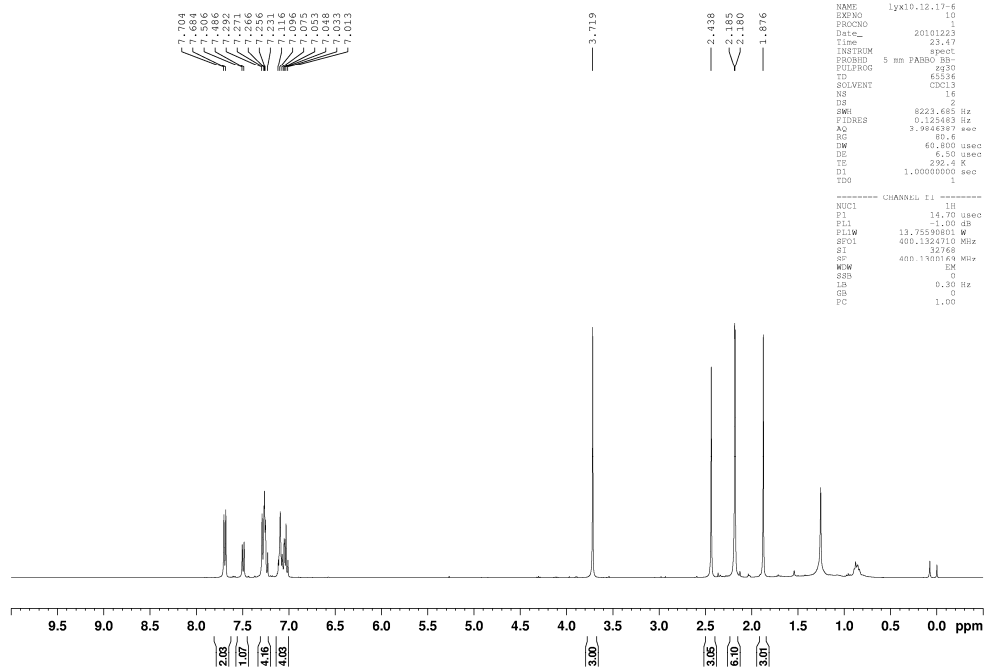
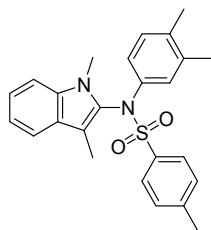
lyx10.11.26-7





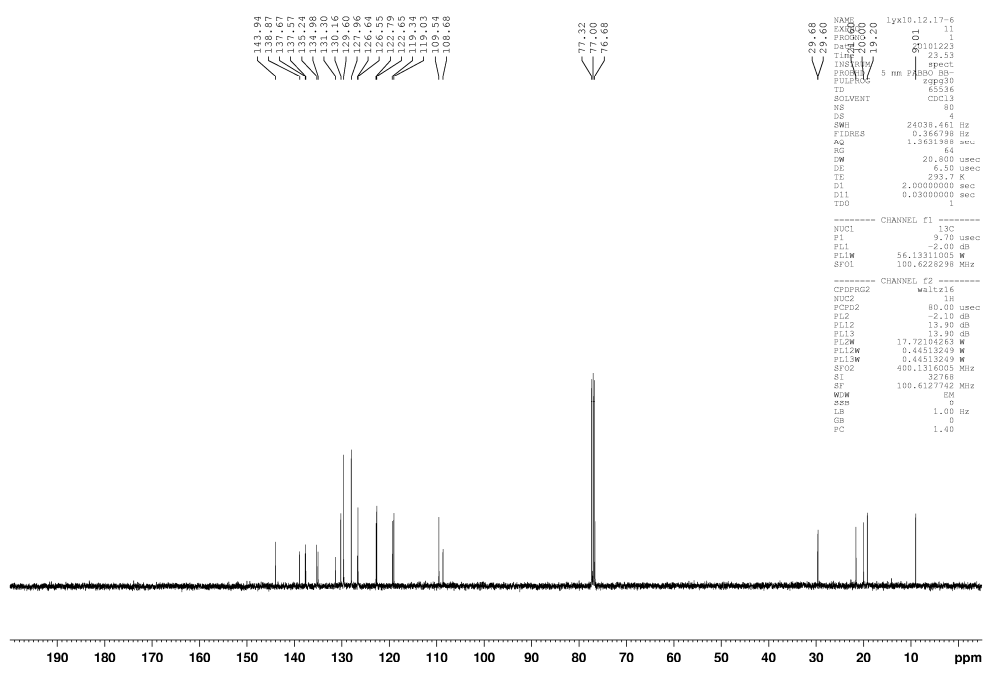






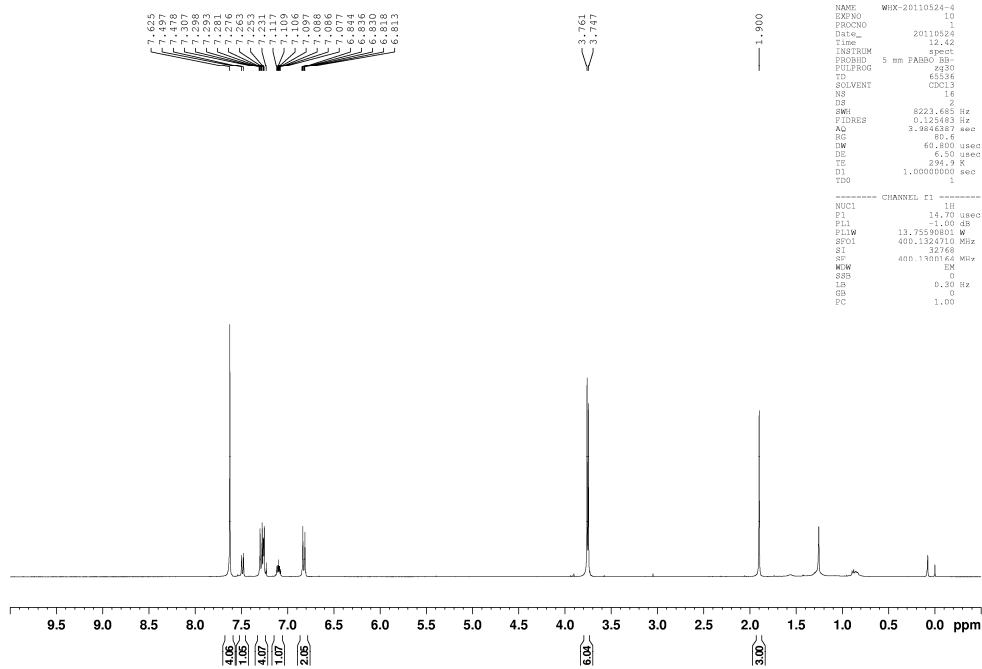
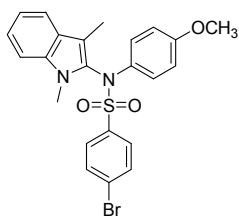
```

NAME Lyk10.12.17-6
EXPNO 1
PROCNO 1
Date_ 20101123
Time 23.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 4
DS 4
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.984587 sec
RG 64
DM 60.800 usec
DE 6.50 usec
TE 293.2 K
D1 1.00000000 sec
TD0 1
    
```



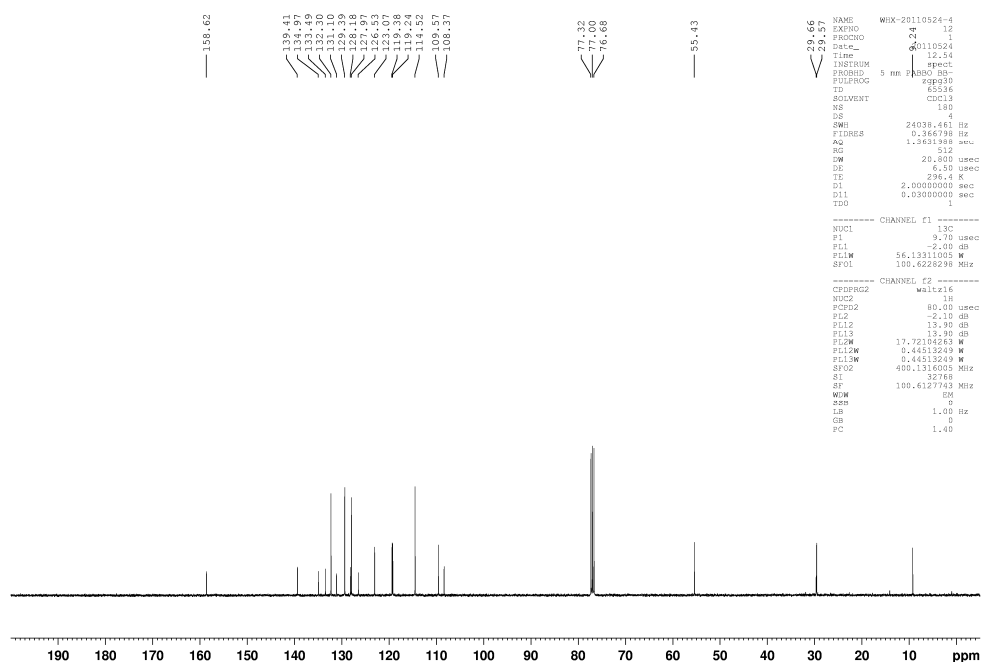
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NAME Lyk10.12.17-6
EXPNO 1
PROCNO 1
Date_ 20101123
Time 23.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 4
DS 4
SWH 24038.461 Hz
FIDRES 0.266798 Hz
AQ 1.363198 sec
RG 64
DM 20.800 usec
DE 6.50 usec
TE 293.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
    
```



```
NAME WHX-20110524-4
EXPNO 12
PROCNO 1
Date_ 20110524
Time 12.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 16
DS 4
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 1.3846387 sec
RG 60.6
DW 60.800 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
```

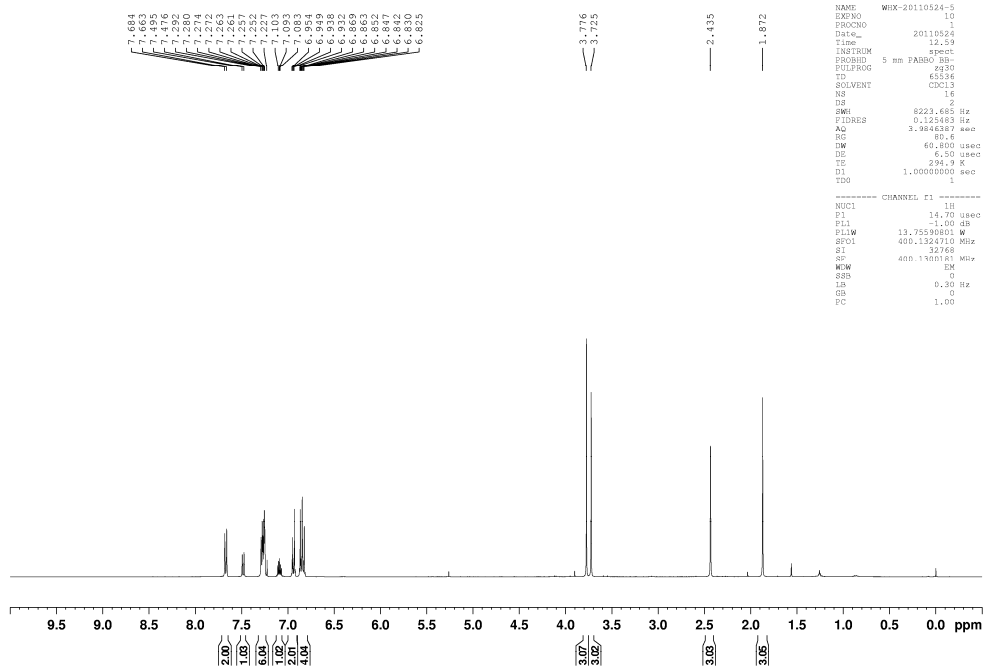
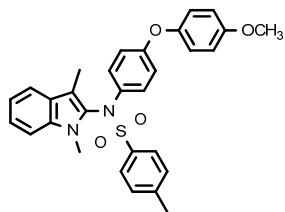
----- CHANNEL f1 -----
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 17.75590801 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1316095 MHz
MCHW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



```
NAME WHX-20110524-4
EXPNO 12
PROCNO 24
Date_ 20110524
Time 12.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65336
SOLVENT CDCl3
NS 160
DS 4
SWH 24038.461 Hz
FIDRES 0.266798 Hz
AQ 1.3931988 sec
RG 612
DW 28.800 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
```

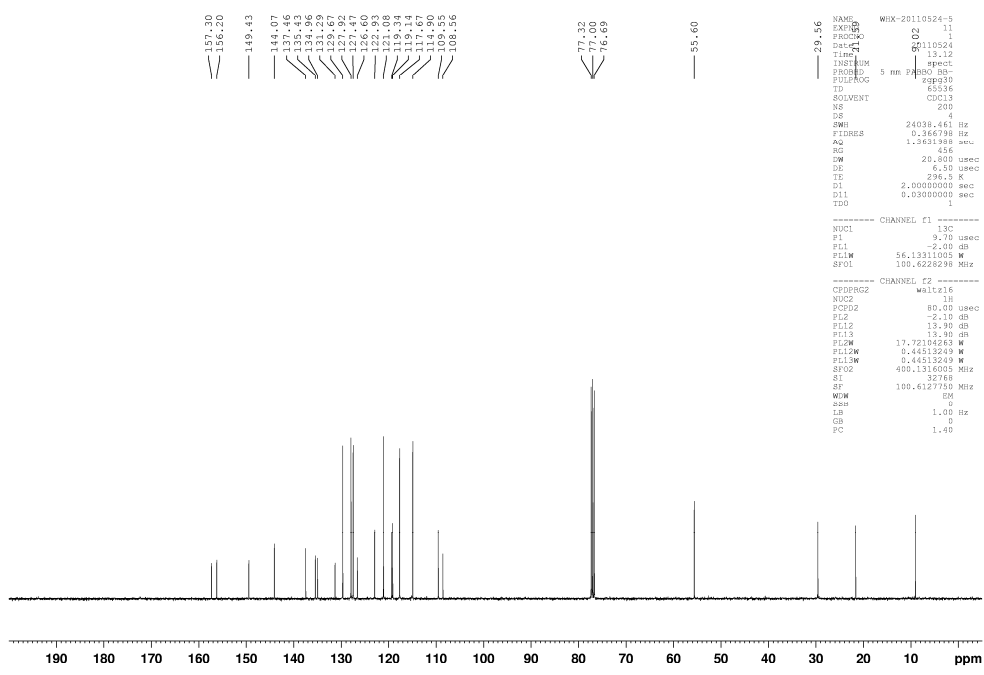
----- CHANNEL f1 -----
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311095 W
SFO1 100.6226298 MHz

----- CHANNEL f2 -----
NAME waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL2 13.90 dB
PL23 13.90 dB
PL2W 17.72104583 W
SFO2 400.1316095 MHz
SI 32768
SF 100.6127743 MHz
MCHW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



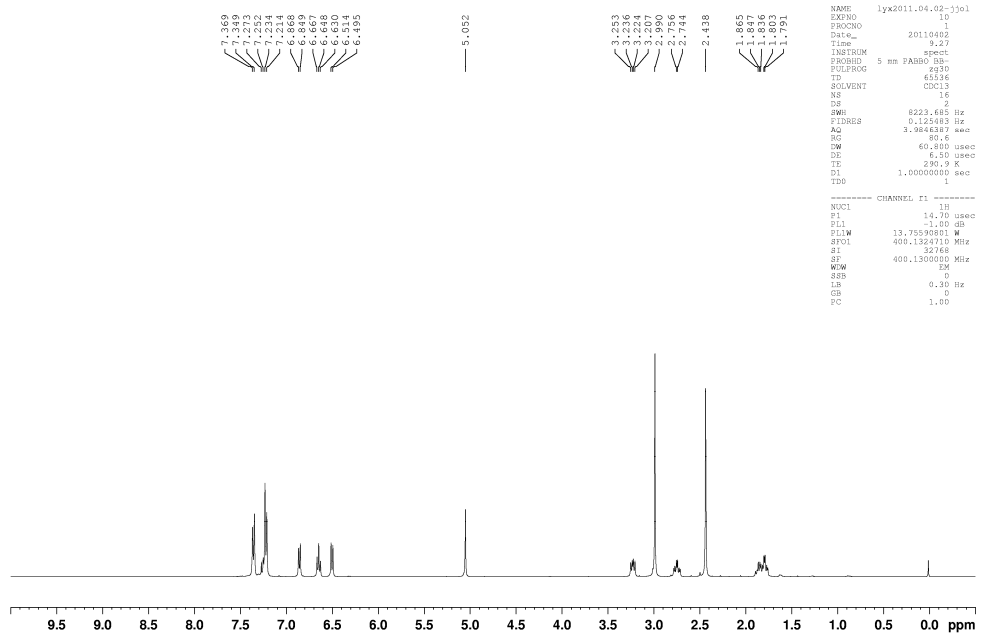
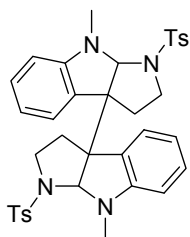
```

NAME      WIX-20110524-5
EXPNO    1
PROCNO   1
Date_    20110524
Time     12.59
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        4
SWH       8223.685 Hz
FIDRES   0.125483 Hz
AQ        1.848487 sec
RG         60.6
AQ        60.600 usec
DE         6.50 usec
TE        294.2 K
D1        1.00000000 sec
TD0       1
    
```



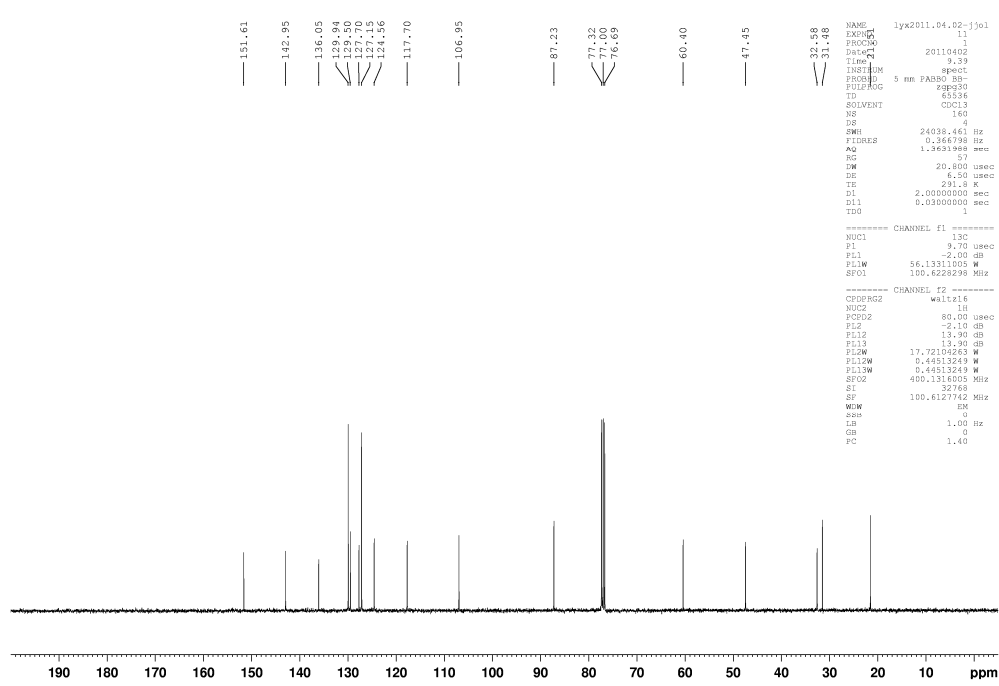
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NAME      WIX-20110524-5
EXPNO    1
PROCNO   1
Date_    20110524
Time     13.12
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        200
DS        4
SWH       24038.461 Hz
FIDRES   0.346798 Hz
AQ        1.3631988 sec
RG         656
AQ        20.800 usec
DE         6.50 usec
TE        296.5 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
    
```

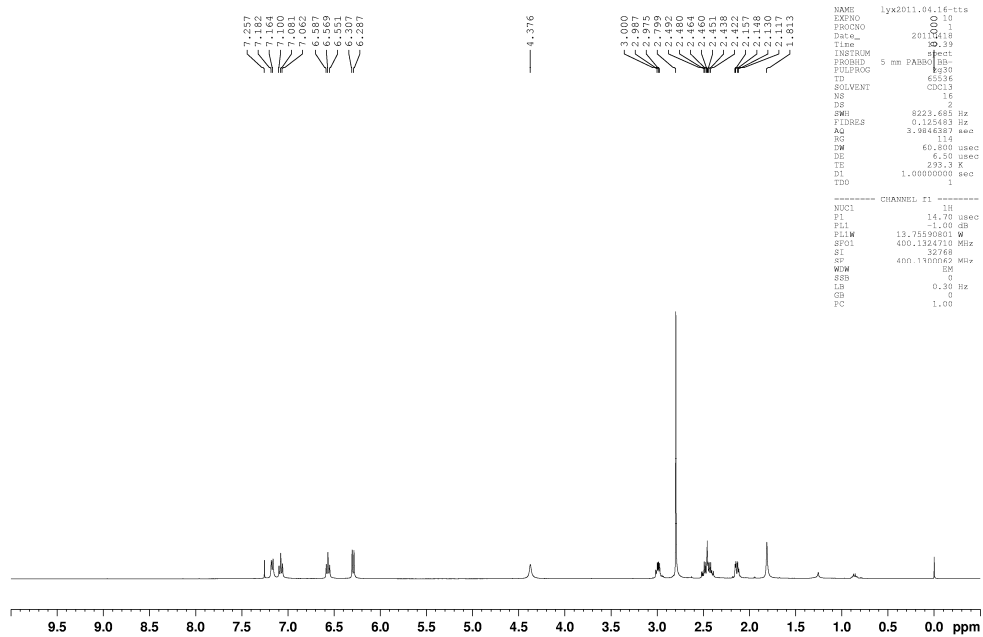
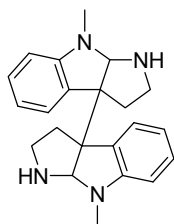
```

NAME lyx2011.04.02-1j01
EXPNO 10
PROCNO 1
DATE_ 20110402
Time 9.27
INSTRUM spect
PROBHD 5 mm PABBO QNP-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.683 Hz
FIDRES 0.123483 Hz
AQ 3.9246217 sec
RG 80.6
DW 60.800 usec
DE 6.30 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.75598901 M
SFO1 400.1324710 MHz
SI 32768
SF 400.13300000 MHz
WVW EM
SOLV 0
LB 0.30 Hz
GB 0
PC 1.00
    
```



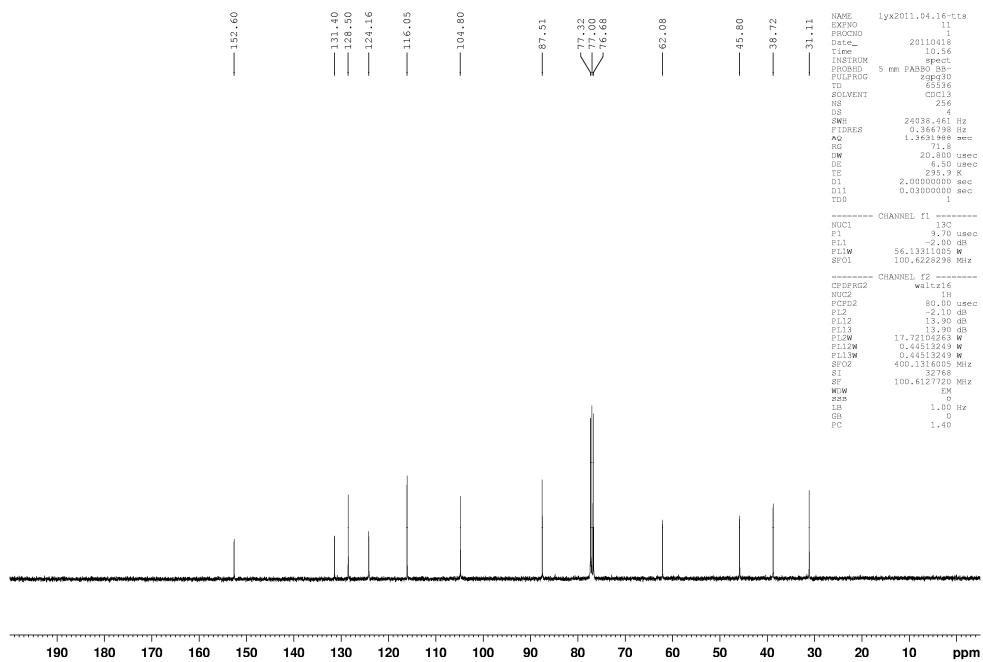
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NAME lyx2011.04.02-1j01
EXPNO 11
PROCNO 1
DATE_ 20110402
Time 9.39
INSTRUM spect
PROBHD 5 mm PABBO QNP-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3651989 sec
RG 57
DW 20.800 usec
DE 6.50 usec
TE 291.9 K
D1 2.00000000 sec
D12 0.03000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311005 M
SFO1 100.6228298 MHz
----- CHANNEL f2 -----
NAME lyx2011.04.02-1j01
EXPNO 12
PROCNO 1
DATE_ 20110402
Time 9.39
INSTRUM spect
PROBHD 5 mm PABBO QNP-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 100.6127742 MHz
FIDRES 0.366798 Hz
AQ 1.3651989 sec
RG 57
DW 20.800 usec
DE 6.50 usec
TE 291.9 K
D1 2.00000000 sec
D12 0.03000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.75598901 M
SFO1 400.1324710 MHz
SI 32768
SF 400.13300000 MHz
WVW EM
SOLV 0
LB 0.30 Hz
GB 0
PC 1.00
    
```



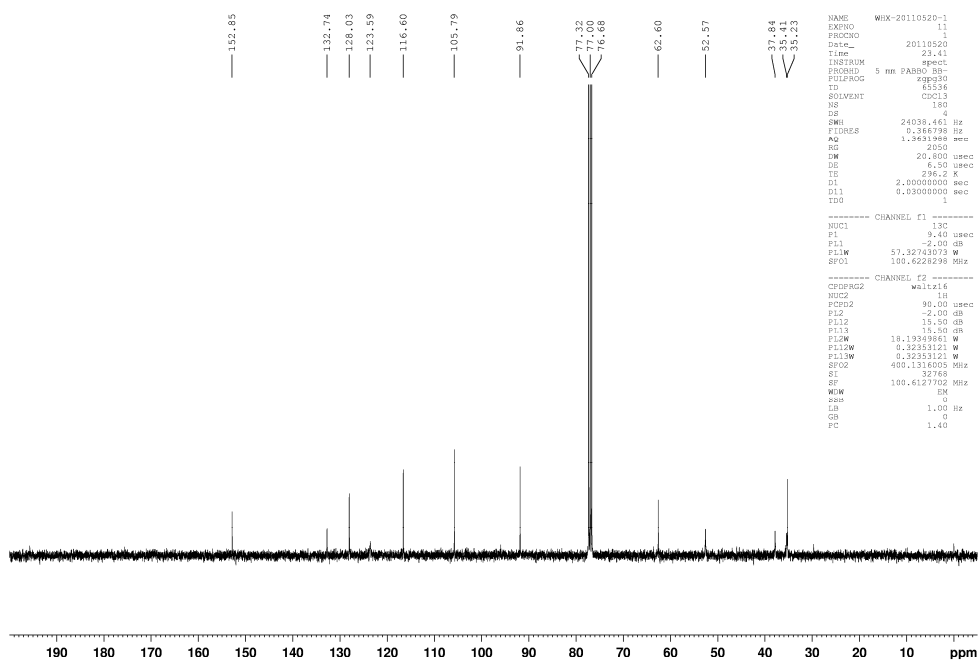
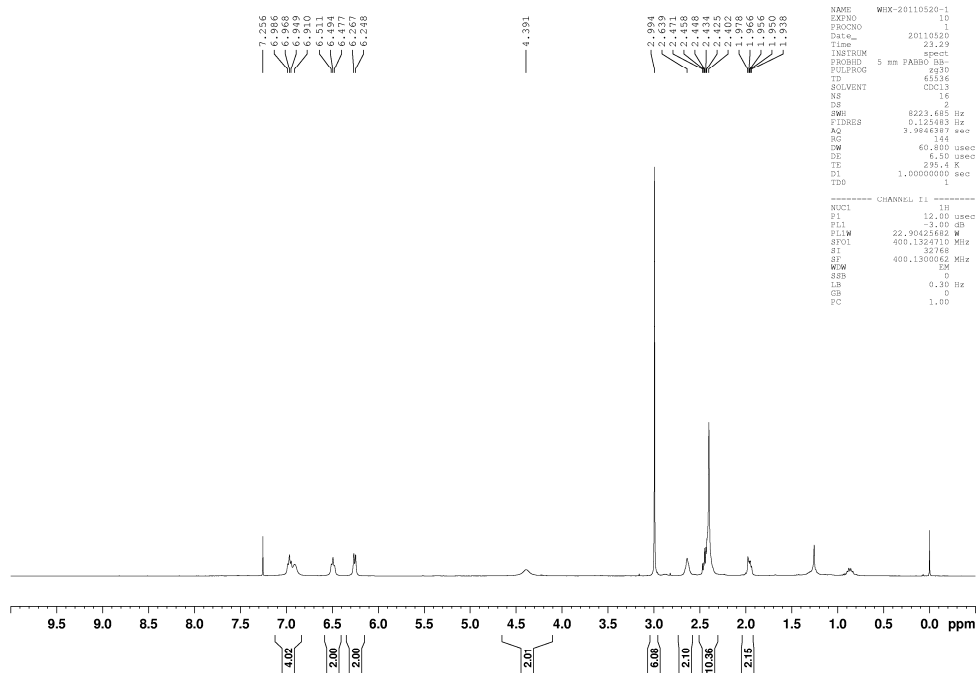
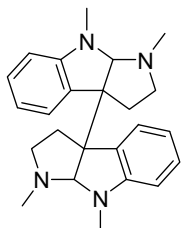
```

NAME lyx2011.04.16-tts
EXPNO 1
PROCNO 1
Date_ 20110416
Time 09.29
INSTRUM spect
PROBHD 5 mm PABBO
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.364287 sec
RG 134
TM 60.500 usec
DE 6.50 usec
TE 295.3 K
D1 1.0000000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.7559001 W
SFO1 400.1324510 MHz
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
    
```



```

NAME lyx2011.04.16-tts
EXPNO 1
PROCNO 1
Date_ 20110416
Time 10.56
INSTRUM spect
PROBHD 5 mm PABBO
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 24038.461 Hz
FIDRES 0.266198 Hz
AQ 1.3631968 sec
RG 71.8
DM 20.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
----- CHANNEL f1 -----
NUC1 13C
P1 9.70 usec
PL1 -2.00 dB
PL1W 56.13311005 W
SFO1 100.6228298 MHz
----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.10 dB
PL2 13.300 dB
PL3 13.30 dB
PL3W 17.72102683 W
PL2W 0.44513249 W
PL3W 0.44513249 W
SFO2 400.1316005 MHz
SF 100.6127220 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```



(1) C. A. Merlic; Y. You; D. M. McInnes; A. L. Zechman; M. M. Miller; Q. Deng, *Tetrahedron* 2001, 57, 5199–5212.

(2) K. K. Park, J. J. Lee, J. Ryu, *Tetrahedron*, 2003, 59, 7651-7659.