Application of Ynamides in the Synthesis of 2-Aminoindoles

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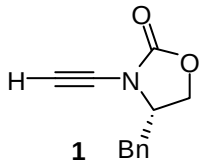
General methods

Solvents were dried according to standard procedures and flash chromatography was carried out on silica gel 60 (230–400 mesh). The ^{1}H NMR spectra were recorded at 400 MHz, ^{13}C NMR spectra were recorded at 100 MHz and ^{19}F NMR spectra were recorded at 377 MHz on a Varian Mercury 400 spectrometer. The chemical shifts are reported in ppm relative to solvent residual peak.[1] ^{1}H NMR spectra are reported as follows (s = singlet, d = doublet, t = triplet, q = quartet, quin. = quintet, hex. = hextet, hep. = heptet, oct. = octet, br = broad; coupling constant(s) in Hz; integration). MS spectra were recorded on a LC TOF (ES) apparatus. Melting points were measured with a Büchi Melting Point B-540 apparatus. All indole reactions were carried out in 8 mL sample vials with a teflon sealed screwcap in a glovebox under an argon atmosphere. All purchased chemicals were used as received without further purification. TIPS-protected ynamides were synthesized according to literature procedure.[2]

[1] Gottlieb, H. E.; Kotlyar, V.; Nudelman, A. *J. Org. Chem.* **1997**, *62*, 7512.
[2] Dooleweerdt, K.; Birkedal, H.; Ruhland, T.; Skrydstrup, T. *J. Org. Chem.* In press.

Synthesis of terminal alkynes 1, 3–4

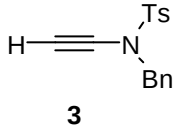
(*S*)-4-benzyl-3-ethynyloxazolidin-2-one (1)



1

(*S*)-4-benzyl-3-((triisopropylsilyl)ethynyl)oxazolidin-2-one (48.7 mmol, 17.4 g) in 500 mL dry THF under an atmosphere of argon was cooled to -18 °C and TBAF (1 M in THF, 97.3 mmol, 97 mL) was added. The reaction mixture was stirred for 10 min and then allowed to heat up to room temperature over 35 min. After quenching with 250 mL satd. aq. NH_4Cl the reaction mixture was extracted three times with Et_2O, dried over Na_2SO_4 and concentrated *in vacuo*. After flash chromatography on silica gel (EtOAc in heptane in a gradient from 20–40%) the title compound was obtained as a pale yellow solid (8.45 g, 42.0 mmol, 86%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.32 (m, 3H), 7.21 (d, 2H, J = 7.2 Hz), 4.29 (m, 2H), 4.13 (dd, 1H, J = 7.2 Hz, 4.4 Hz), 3.23 (dd, 1H, J = 13.6 Hz, 2.8 Hz), 2.99 (s, 1H), 2.95 (dd, 1H, J = 13.6 Hz, 8.0 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 155.8, 134.1, 129.5 (2C), 129.1 (2C), 127.6, 71.8, 67.5, 62.0, 58.0, 37.6. HRMS (ES) calcd for $C_{12}H_{11}NO_2Na$ (MNa+) 224.0682, found 224.0689. Spectroscopic data were in accordance with those reported in the literature.[3] mp = 65.3 – 66.1 °C (Et_2O) [lit: 61.5–62.3 °C][3].

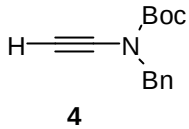
***N*-benzyl-*N*-ethynyl-4-methylbenzenesulfonamide (3)**



3

N-benzyl-4-methyl-*N*-((triisopropylsilyl)ethynyl)benzenesulfonamide (3.58 mmol, 1.58 g) in 40 mL dry THF under an atmosphere of argon was cooled to -18 °C and TBAF (1 M in THF, 7.15 mmol, 7.2 mL) was added. The reaction mixture was stirred for 10 min and then allowed to heat up to room temperature over 20 min. After quenching with 25 mL satd. aq. NH_4Cl the reaction mixture was extracted three times with Et_2O, dried over Na_2SO_4 and concentrated in vacuo. After flash chromatography on silica gel (10% Et_2O in pentane) and recrystallization from Et_2O the title compound was obtained as a colorless solid (459 mg, 1.61 mmol, 91%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.76 (d, 2H, J = 8.0 Hz), 7.30 (m, 7H), 4.50 (s, 2H), 2.67 (s, 1H), 2.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 144.9, 134.8, 134.4, 129.9 (2C), 128.8 (2C), 128.7 (2C), 128.5, 127.8 (2C), 76.4, 59.8, 55.4, 21.8. HRMS (ES) calcd for $C_{16}H_{15}NO_2SNa$ (MNa^+) 308.0721, found 308.0721. Spectroscopic data were in accordance with those reported in the literature.[4] mp = 109 – 112 °C (Et_2O) [lit: 102 – 103 °C][45].

***Tert*-butyl benzyl(ethynyl)carbamate (4)**



4

Tert-butyl benzyl((triisopropylsilyl)ethynyl)carbamate (5.31 mmol, 2.06 g) in 60 mL dry THF under an atmosphere of argon was cooled to -18 °C and TBAF (1 M in THF, 10.6 mmol, 10.6 mL) was added. The reaction mixture was stirred for 10 min and then allowed to heat up to room temperature over 30 min. After quenching with 25 mL satd. aq. NH_4Cl the reaction mixture was extracted three times with Et_2O, dried over Na_2SO_4 and concentrated *in vacuo*. After column chromatography on silica gel (Et_2O in pentane in a gradient from 4–5%) the title compound was obtained as an yellow oil (1.18 g, 5.12 mmol, 96%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.35 (m, 5H), 4.58 (s, 2H), 2.76 (s, 1H), 1.50 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 154.1, 136.4, 128.5 (2C), 128.1, 127.9 (2C), 82.8, 58.7, 53.0, 28.2 (3C). HRMS (ES) calcd for $C_{14}H_{17}NO_2Na$ (MNa^+) 254.1157, found 254.1151.

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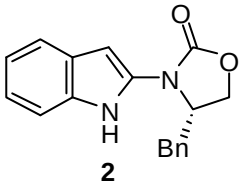
[3] Naud, S.; Cintrat, J. *Synthesis* **2003**, 1391.
[4] Tracey, MR.; Zhang, Y.; Frederick, MO.; Mulder, JA.; Hsung, RP. *Org. Lett.* **2004**, *6*, 2209.
[5] Brueckner, D. *Tetrahedron* **2006**, *62*, 3809.

Synthesis of indoles and benzofuranes (General method A)

To the 2-iodoaniline/2-iodophenol (1 equiv.) was added ynamide (1.2 equiv. unless otherwise stated) and Bu_4NOAc (3 equiv.) dissolved in 1.00 mL dry DMF followed by $Pd(OAc)_2$ (0.05 equiv.) and PPh_3 (0.20 equiv.) in 1.00 mL dry DMF. The sample vial was then fitted with a teflon sealed screwcap and removed from the glovebox. The reaction mixture was heated for the time stated for each product at 60 °C. After cooling to room temperature, water was added and the mixture was extracted three times with EtOAc, washed with brine, dried over $MgSO_4$ and concentrated *in vacuo*. The crude mixture was dissolved in CH_2Cl_2 and concentrated *in vacuo* on to a small amount of silica gel and purified by flash chromatography.

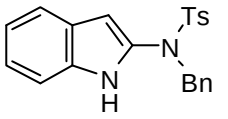
(*S*)-4-benzyl-3-(1*H*-indol-2-yl)oxazolidin-2-one (2) from General Method A



2

2-iodoaniline (0.50 mmol, 110 mg), **1** (0.60 mmol, 121 mg), Bu_4NOAc (1.50 mmol, 452 mg), $Pd(OAc)_2$ (0.025 mmol, 6.0 mg), PPh_3 (0.10 mmol, 26 mg) were reacted for 24 h. After flash chromatography (20% EtOAc in pentane) the title compound was obtained as a pale yellow solid (118 mg, 0.40 mmol, 81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.0 (s, 1H), 7.56 (dd, 1H, J = 6.8 Hz, 1.2 Hz), 7.30–7.38 (m, 5H), 7.12–7.20 (m, 4H), 6.00 (s, 1H), 4.59 (tt, 1H, J = 8.4 Hz, 2.8 Hz), 4.45 (t, 1H, J = 8.4 Hz), 4.35 (dd, 1H, J = 8.8 Hz, 3.2 Hz), 3.36 (dd, 1H, J = 14.4 Hz, 2.4 Hz), 3.05 (dd, 1H, J = 14.0 Hz, 9.2 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 155.1, 134.7, 133.4, 133.1, 129.5 (2C), 129.2 (2C), 127.6, 127.3, 121.3, 120.5, 119.4, 111.0, 85.9, 67.1, 56.9, 36.8. HRMS (ES) calcd for $C_{18}H_{16}N_2O_2Na$ (MNa^+) 315.1104, found 315.1102. mp = 142.8 – 144.6 °C.

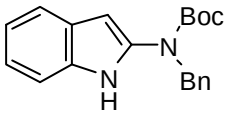
***N*-benzyl-*N*-(1*H*-indol-2-yl)-4-methylbenzenesulfonamide (5) from General Method A**



5

2-iodoaniline (0.30 mmol, 66 mg), **3** (0.36 mmol, 103 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 24 h. After flash chromatography (10% acetone in pentane) the title compound was obtained as a colorless solid (72 mg, 0.19 mmol, 64%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.69 (s, 1H), 7.55 (d, 2H, J = 8.4 Hz), 7.41 (d, 1H, J = 7.6 Hz), 7.32 (t, 2H, J = 7.2 Hz), 7.19–7.28 (m, 6H), 7.15 (t, 1H, J = 7.6 Hz), 7.05 (t, 1H, J = 7.2 Hz), 5.87 (s, 1H), 4.71 (s, 2H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 144.4, 135.7, 134.2, 134.1, 133.7, 129.9 (2C), 128.7 (2C), 128.2 (2C), 128.0, 127.7 (2C), 122.2, 120.3, 120.1, 111.0, 95.2, 54.3, 21.8. HRMS (ES) calcd for $C_{22}H_{20}N_2O_2SNa$ (MNa^+) 399.1143, found 399.1141. mp = 172.2 – 173.6 °C.

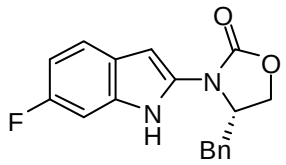
***Tert*-butyl benzyl(1*H*-indol-2-yl)carbamate (6) from General Method A**



6

2-iodoaniline (0.30 mmol, 66 mg), **4** (0.36 mmol, 69 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 25 h. After flash chromatography (5% acetone in pentane) the title compound was obtained as a colorless solid (39 mg, 0.12 mmol, 40%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.5 (s, 1H), 7.42 (d, 1H, J = 7.2 Hz), 7.24–7.34 (m, 6H), 7.07 (m, 2H), 5.84 (s, 1H), 4.98 (s, 2H), 1.50 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 128.7 (2C), 127.4 (2C), 126.6, 120.0, 119.2, 110.6, 87.0, 51.9, 28.3 (3C). HRMS (ES) calcd for $C_{20}H_{22}N_2O_2Na$ (MNa^+) 345.1579, found 345.1584.

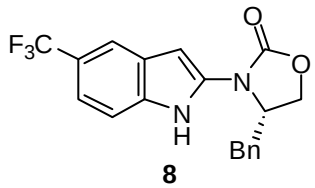
(*S*)-4-benzyl-3-(6-fluoro-1*H*-indol-2-yl)oxazolidin-2-one (7) from General Method A



7

5-fluoro-2-iodoaniline (0.30 mmol, 71 mg), **1** (0.36 mmol, 72 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 25 h. After flash chromatography (acetone in pentane in a gradient from 10–20%) the title compound was obtained as a colorless solid (64 mg, 0.21 mmol, 69%. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.06 (s, 1H), 7.44 (dd, 1H, J = 8.8 Hz, 5.2 Hz), 7.29–7.38 (m, 3H), 7.18 (d, 2H, J = 7.2 Hz), 7.07 (dd, 1H, J = 9.2 Hz, 2.4 Hz), 6.90 (ddd, 1H, J = 9.6 Hz, 8.8 Hz, 2.4 Hz), 5.96 (d, 1H, J = 2.0 Hz), 4.56 (tt, 1H, J = 8.4 Hz, 2.8 Hz), 4.44 (t, 1H, J = 8.0 Hz), 4.35 (dd, 1H, J = 8.4 Hz, 2.8 Hz), 3.35 (dd, 1H, J = 14.0 Hz, 2.8 Hz), 3.03 (dd, 1H, J = 14.0 Hz, 8.8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.6, 158.2, 155.2, 134.6, 133.6 (d, J = 3.2 Hz), 133.0 (d, J = 12.5 Hz), 129.5 (2C), 129.2 (2C), 127.7, 123.6, 120.0 (d, J = 9.7 Hz), 108.9 (d, J = 23.9 Hz), 95.7 (d, J = 26.5 Hz), 85.7, 67.1, 57.0, 36.8. ^{19}F NMR (377 MHz, $CDCl_3$) δ (ppm) -122.0 (td, J = 9.80 Hz, 5.28 Hz) . HRMS (ES) calcd for $C_{18}H_{15}ClN_2O_2Na$ (MNa^+) 333.1015, found 333.1024. mp = 148.8 – 150.1 °C.

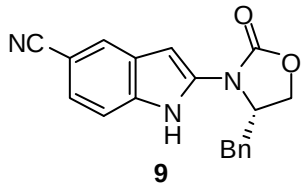
(*S*)-4-benzyl-3-(5-(trifluoromethyl)-1*H*-indol-2-yl)oxazolidin-2-one (8) from General Method A



8

2-iodo-4-(trifluoromethyl)aniline (0.30 mmol, 72 mg), 1 (0.36 mmol, 72 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 25 h. After flash chromatography (10% acetone in pentane) the title compound was obtained as a colorless solid (52 mg, 0.15 mmol, 48%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.3 (s, 1H), 7.84 (s, 1H), 7.29–7.45 (m, 5H), 7.18 (d, 2H, J = 6.8 Hz), 6.05 (s, 1H), 4.60 (tt, 1H, J = 8.4 Hz, 3.2 Hz), 4.47 (t, 1H, J = 8.4 Hz), 4.38 (dd, 1H, J = 9.2 Hz, 3.2 Hz), 3.34 (dd, 1H, J = 14.0 Hz, 3.2 Hz), 3.05 (dd, 1H, J = 14.0 Hz, 8.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 155.1, 135.4 (t, J = 5.8 Hz), 134.9, 134.53, 134.45, 130.4, 129.5 (2C), 129.3 (2C), 127.8, 126.80, 126.77, 124.1, 123.0 (q, J = 31.3 Hz), 121.4, 118.1 (q, J = 3.8 Hz), 117.0 (q, J = 3.8 Hz), 111.1, 86.3, 67.2, 56.9, 36.8. ^{19}F NMR (377 MHz, $CDCl_3$) δ (ppm) -60.8 (s). HRMS (ES) calcd for $C_{19}H_{15}F_3N_2O_2Na$ (MNa^+) 383.0983, found 383.0989. mp = 150.0 – 152.6 °C.

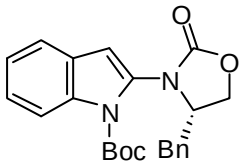
(*S*)-2-(4-benzyl-2-oxooxazolidin-3-yl)-1*H*-indole-5-carbonitrile (9) from General Method A



9

4-amino-3-iodobenzonitrile (0.30 mmol, 73 mg), **1** (0.36 mmol, 72 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 25 h. After flash chromatography (20% acetone in pentane) the title compound was obtained as a colorless solid (37 mg, 0.12 mmol, 38%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.4 (ppm) (s, 1H), 7.86 (s, 1H), 7.41 (s, 2H), 7.29–7.38 (m, 3H), 7.18 (d, 2H, J = 6.8 Hz), 6.03 (d, 1H, J = 2.0 Hz), 4.61 (tt, 1H, J = 8.0 Hz, 3.2 Hz), 4.49 (t, 1H, J = 8.0 Hz), 4.39 (dd, 1H, J = 8.8 Hz, 2.8 Hz), 3.35 (dd, 1H, J = 14.0 Hz, 2.8 Hz), 3.05 (dd, 1H, J = 13.6 Hz, 8.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 155.1, 135.4, 134.9, 134.3, 129.5 (2C), 129.3 (2C), 127.9, 127.3, 124.5 (2C), 120.8, 111.7, 103.8, 86.0, 67.3, 56.9, 36.8. HRMS (ES) calcd for $C_{19}H_{15}N_3O_2Na$ (MNa^+) 340.1062, found 340.1056. mp = 179.3 – 181.3 °C.

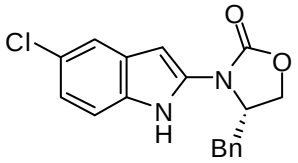
(*S*)-*tert*-butyl-2-(4-benzyl-2-oxooxazolidin-3-yl)-1*H*-indole-1-carboxylate (10) from General Method A



10

Tert-butyl 2-iodophenylcarbamate (0.30 mmol, 96 mg, 75 μL), **1** (0.50 mmol, 100 mg, 1.67 equiv.), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 25 h. After flash chromatography (10% acetone in pentane) the title compound was obtained as an yellow oil (90 mg, 0.23 mmol, 77%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.11 (d, 1H, J = 8.8 Hz), 7.56 (d, 1H, J = 8.4 Hz), 7.37 (ddd, 1H, J = 8.4 Hz, 7.2 Hz, 1.2 Hz), 7.22–7.31 (m, 4H), 7.12 (d, 2H, J = 6.8 Hz), 6.65 (s, 1H), 4.41–4.47 (m, 2H), 4.23 (m, 1H), 3.19 (dd, 1H, J = 14.0 Hz, 3.6 Hz), 2.84 (dd, 1H, J = 13.6 Hz, 9.6 Hz), 1.69 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 149.4, 135.34, 135.29, 130.0, 129.04 (2C), 129.01 (2C), 127.3, 127.1, 125.4, 123.2, 121.1, 116.0, 84.9, 68.6, 59.2, 39.3, 28.3 (3C). HRMS (ES) calcd for $C_{23}H_{24}N_2O_4Na$ (MNa^+) 415.1634, found 415.1644.

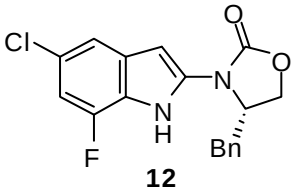
(*S*)-4-benzyl-3-(5-chloro-1*H*-indol-2-yl)oxazolidin-2-one (11) from General Method A



11

4-chloro-2-iodoaniline (0.30 mmol, 76 mg), **1** (0.36 mmol, 72 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 25 h. After flash chromatography (15% acetone in pentane) the title compound was obtained as a pale green solid (85 mg, 0.26 mmol, 87%. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.13 (s, 1H), 7.50 (d, 1H, J = 2.4 Hz), 7.28–7.38 (m, 4H), 7.17 (dd, 2H, J = 8.4 Hz, 1.6 Hz), 7.11 (dd, 1H, J = 8.4 Hz, 2.0 Hz), 5.91 (d, 1H, J = 2.0 Hz), 4.54 (tt, 1H, J = 8.4 Hz, 2.8 Hz), 4.43 (t, 1H, J = 8.4 Hz), 4.34 (dd, 1H, J = 9.2 Hz, 3.2 Hz), 3.32 (dd, 1H, J = 14.0 Hz, 2.8 Hz), 3.01 (dd, 1H, J = 13.6 Hz, 8.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 155.0, 134.54, 134.51, 131.4, 129.5 (2C), 129.2 (2C), 128.4, 127.7, 126.0, 121.4, 118.8, 112.0, 85.5, 67.1, 56.8, 36.8. HRMS (ES) calcd for $C_{18}H_{15}ClN_2O_2Na$ (MNa^+) 349.0720, found 349.0720. mp = 74.5 – 76.5 °C.

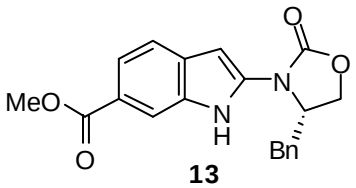
(*S*)-4-benzyl-3-(5-chloro-7-fluoro-1*H*-indol-2-yl)oxazolidin-2-one (12) from General Method A



12

4-chloro-2-fluoro-6-iodoaniline (0.30 mmol, 81 mg), **1** (0.36 mmol, 72 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 25 h. After flash chromatography (10% acetone in pentane) the title compound was obtained as a colorless solid (90 mg, 0.26 mmol, 87%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.2 (s, 1H), 7.29–7.39 (m, 3H), 7.28 (d, 1H, J = 1.2 Hz), 7.17 (dt, 2H, J = 6.8 Hz, 1.6 Hz), 6.90 (dd, 1H, J = 12.0 Hz, 1.6 Hz), 5.94 (t, 1H, J = 2.4 Hz), 4.57 (tt, 1H, J = 8.4 Hz, 3.2 Hz), 4.46 (t, 1H, J = 8.0 Hz), 4.37 (dd, 1H, J = 8.8 Hz, 2.8 Hz), 3.33 (dd, 1H, J = 13.6 Hz, 3.2 Hz), 3.04 (dd, 1H, J = 13.6 Hz, 8.8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 155.0, 149.8, 147.3, 135.1, 134.4, 131.03, 130.97, 129.5 (2C), 129.3 (2C), 127.8, 125.64, 125.56, 119.7, 119.6, 114.9, 114.8, 107.9, 107.7, 86.32, 67.2, 56.9, 36.9. ^{19}F NMR (377 MHz, CDCl3) δ (ppm) -132.8 (dd, J = 11.2 Hz, 4.2 Hz). HRMS (ES) calcd for $C_{18}H_{14}ClFN_2O_2Na$ (MNa^+) 367.0626, found 367.0619. mp = 133.5 – 135.0 °C.

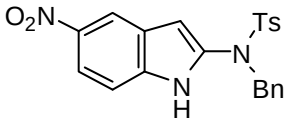
(*S*)-methyl 2-(4-benzyl-2-oxooxazolidin-3-yl)-1*H*-indole-6-carboxylate (13) from General Method A



13

Methyl 3-amino-4-iodobenzoate (0.30 mmol, 83 mg), **1** (0.36 mmol, 72 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 25 h. After flash chromatography (20% acetone in pentane) the title compound was obtained as an off-white solid (45 mg, 0.13 mmol, 42% . ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.25 (s, 1H), 8.10 (kv, 1H, J = 0.8 Hz), 7.85 (dd, 1H, J = 8.4 Hz, 1.6 Hz), 7.55 (dt, 1H, J = 8.4 Hz, 0.8 Hz), 7.29–7.38 (m, 3H), 7.18 (dt, 2H J = 6.8 Hz, 1.6 Hz), 6.02 (dd, 1H, J = 2.0 Hz, 0.8 Hz), 4.60 (tt, 1H, J = 8.8 Hz, 3.2 Hz), 4.46 (t, 1H, J = 8.4 Hz), 4.37 (dd, 1H, J = 9.2 Hz, 2.8 Hz), 3.94 (s, 3H), 3.36 (dd, 1H, J = 14.0 Hz, 3.2 Hz), 3.05 (dd, 1H, J = 14.0 Hz, 8.8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 168.1, 155.1, 136.3, 134.5, 132.3, 131.3, 129.5 (2C), 129.3 (2C), 127.8, 122.9, 122.0, 118.8, 113.1, 86.3, 67.2, 56.9, 52.0, 36.9. HRMS (ES) calcd for $C_{20}H_{18}N_2O_4Na$ (MNa^+) 373.1164, found 373.1166. mp = 179.0 – 181.5 °C.

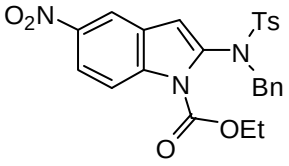
***N*-benzyl-4-methyl-*N*-(5-nitro-1*H*-indol-2-yl)benzenesulfonamide (14) from General Method A**



14

2-iodo-4-nitroaniline (0.30 mmol, 79 mg), **3** (0.36 mmol, 103 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 24 h. After flash chromatography (10% acetone in pentane) the title compound was obtained as an yellow solid (33 mg, 0.079 mmol, 26%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 9.18 (s, 1H), 8.35 (d, 1H, J = 2.4 Hz), 8.05 (dd, 1H, J = 8.8 Hz, 2.0 Hz), 7.53 (dt, 2H, J = 8.4 Hz, 2.0 Hz), 7.27–7.33 (m, 6H), 7.22–7.25 (m, 2H), 5.98 (dd, 1H, J = 2.4 Hz, 0.8 Hz), 4.72 (s, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 145.1, 142.2, 137.2, 136.6, 135.0, 133.6, 130.1 (2C), 128.8 (2C), 128.3, 128.1 (2C), 127.5 (2C), 126.3, 118.0, 117.3, 111.0, 96.2, 53.9, 21.8. HRMS (ES) calcd for $C_{22}H_{19}N_3O_4SNa$ (MNa^+) 444.0994, found 444.0996. mp = 203.0 – 205.4 °C.

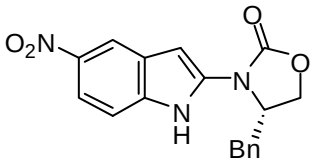
Ethyl 2-(*N*-benzyl-4-methylphenylsulfonamido)-5-nitro-1*H*-indole-1-carboxylate (15) from General Method A



15

Ethyl 2-iodo-4-nitrophenylcarbamate (0.30 mmol, 101 mg), **3** (0.36 mmol, 103 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 24 h. After flash chromatography (10% acetone in pentane) the title compound was obtained as an yellow solid (82 mg, 0.17 mmol, 55%) in addition there was obtained 28 mg (0.066 mmol, 22% of **14**. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.32 (dd, 1H, J = 2.0 Hz, 0.8 Hz), 8.22 (s, 1H), 8.21 (d, 1H, J = 2.0 Hz), 7.57 (dt, 2H, J = 8.4 Hz, 2.0 Hz), 7.29 (d, 2H, J = 8.0 Hz), 7.21–7.24 (m, 3H), 7.14–7.17 (m, 2H), 6.12 (d, 1H, J = 0.4 Hz), 4.77 (s, 2H), 4.51 (q, 2H, J = 7.2 Hz), 2.46 (s, 3H), 1.55 (s, 3H, J = 7.6 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 150.2, 144.3, 143.8, 138.3, 136.2, 136.0, 135.1, 129.8 (2C), 129.3 (2C), 128.6 (2C), 128.4, 127.8 (2C), 126.3, 120.5, 117.3, 116.1, 109.1, 64.6, 55.9, 21.8, 14.3. HRMS (ES) calcd for $C_{25}N_{23}N_3O_4SNa$ (MNa^+) 516.1205, found 516.1218. mp = 57.0 – 59.8 °C.

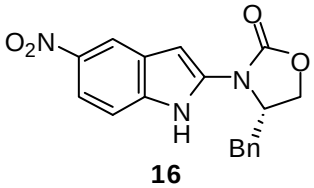
(*S*)-4-benzyl-3-(5-nitro-1*H*-indol-2-yl)oxazolidin-2-one (16) from General Method A *via* carbamate protected aniline



16

Ethyl 2-iodo-4-nitrophenylcarbamate (0.30 mmol, 101 mg), **1** (0.36 mmol, 72 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 23 h. After flash chromatography (20% acetone in pentane) the title compound was obtained as an yellow solid (80 mg, 0.24 mmol, 79%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.5 (s, 1H), 8.48 (d, 1H, *J* = 2.0 Hz), 8.08 (dd, 1H, *J* = 8.8 Hz, 2.4 Hz), 7.30–7.41 (m, 4H), 7.19 (d, 2H, *J* = 8.0 Hz), 6.11 (d, 1H, *J* = 1.6 Hz), 4.63 (tt, 1H, *J* = 8.4 Hz, 2.8 Hz), 4.51 (t, 1H, *J* = 8.4 Hz), 4.41 (dd, 1H, *J* = 9.2 Hz, 2.8 Hz), 3.37 (dd, 1H, *J* = 14.4 Hz, 3.2 Hz), 3.07 (dd, 1H, *J* = 14.4 Hz, 8.8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 155.1, 142.6, 136.3, 136.1, 134.3, 129.5 (2C), 129.4 (2C), 127.9, 126.9, 117.2, 116.3, 110.8, 87.2, 67.4, 56.9, 36.9. HRMS (ES) calcd for $C_{18}H_{15}N_3O_4Na$ (MNa^+) 360.0960, found 360.0966. mp = 187 – 189.3 °C.

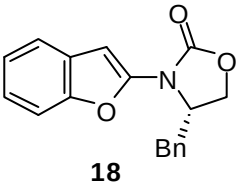
(*S*)-4-benzyl-3-(5-nitro-1*H*-indol-2-yl)oxazolidin-2-one (16) from General Method A



16

2-iodo-4-nitroaniline (0.30 mmol, 79 mg), **1** (0.50 mmol, 100 mg, 1.67 equiv.), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 22 h. After flash chromatography (EtOAc in pentane in a gradient 30–50%) the title compound was obtained as an yellow solid (32 mg, 0.09 mmol, 32%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 10.5 (s, 1H), 8.48 (d, 1H, *J* = 2.0 Hz), 8.08 (dd, 1H, *J* = 8.8 Hz, 2.4 Hz), 7.30–7.41 (m, 4H), 7.19 (d, 2H, *J* = 8.0 Hz), 6.11 (d, 1H, *J* = 1.6 Hz), 4.63 (tt, 1H, *J* = 8.4 Hz, 2.8 Hz), 4.51 (t, 1H, *J* = 8.4 Hz), 4.41 (dd, 1H, J = 9.2 Hz, 2.8 Hz), 3.37 (dd, 1H, *J* = 14.4 Hz, 3.2 Hz), 3.07 (dd, 1H, *J* = 14.4 Hz, 8.8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 155.1, 142.6, 136.3, 136.1, 134.3, 129.5 (2C), 129.4 (2C), 127.9, 126.9, 117.2, 116.3, 110.8, 87.2, 67.4, 56.9, 36.9. HRMS (ES) calcd for $C_{18}H_{15}N_3O_4Na$ (MNa+) 360.0960, found 360.0966. mp = 187 – 189.3 °C.

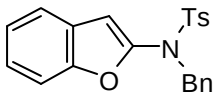
(*S*)-3-(benzofuran-2-yl)-4-benzyloxazolidin-2-one (18) from General Method A



18

2-iodophenol (0.30 mmol, 66 mg), **1** (0.51 mmol, 103 mg, 1.7 equiv.), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 23 h. After flash chromatography (10% acetone in pentane) 35 mg of a mixture of the title compound and (*S*)-3-acetyl-4-benzyloxazolidin-2-one was obtained in a 1.5 : 1 ratio. Corrected yield of (**18**) is 23.1 mg, 0.079 mmol, 26% (colorless solid). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) *inter alia* 7.45–7.47 (m, 1H), 7.34–7.36 (m, 1H), 7.11–7.28 (m, 7H), 6.68 (d, 1H, *J* = 0.8 Hz), 4.73 (tt, 1H, *J* = 9.2 Hz, 4.0 Hz), 4.33 (t, 1H, *J* = 8.4 Hz), 4.20 (dd, 1H, *J* = 9.2 Hz, 4.0 Hz), 2.22 (dd, 1H, *J* = 14.0, 4.0), 2.88 (dd, 1H, *J* = 13.6 Hz, 8.8 Hz). HRMS (ES) calcd for $C_{18}H_{15}NO_3Na$ (MNa^+) 316.0950, found 316.0948.

_N_-(benzofuran-2-yl)-_N_-benzyl-4-methylbenzenesulfonamide (19) from General Method A

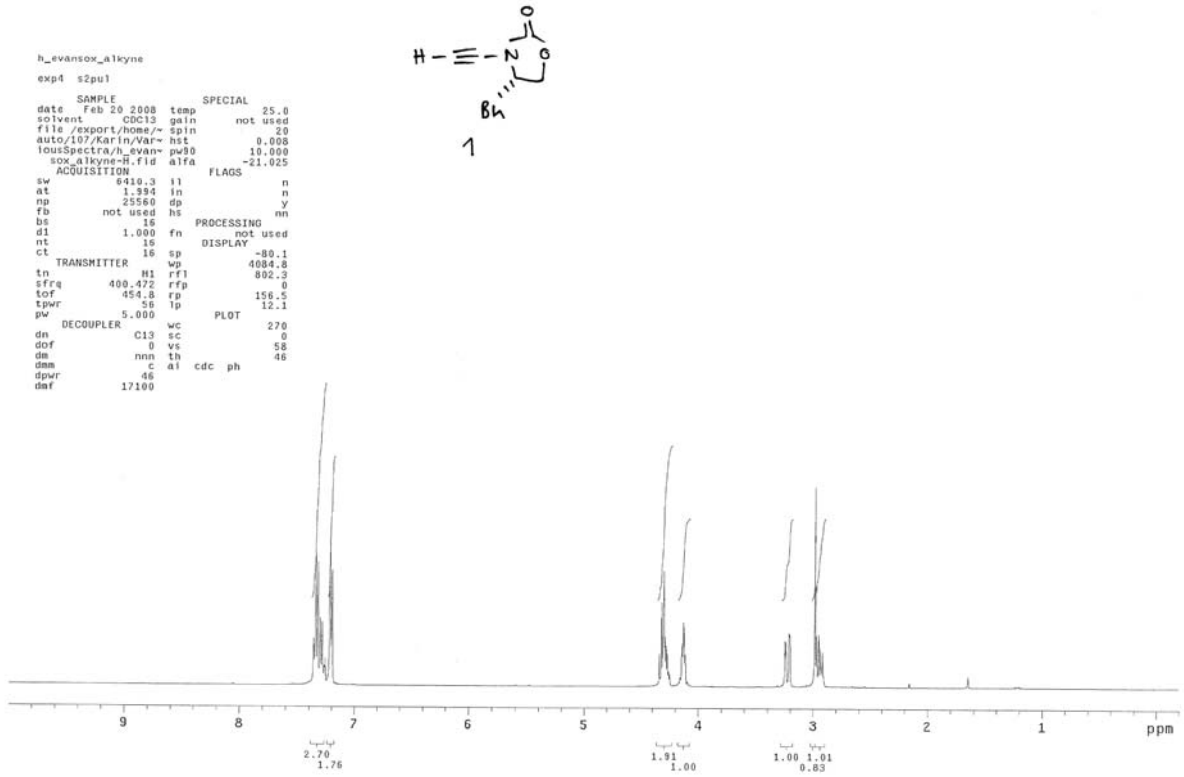


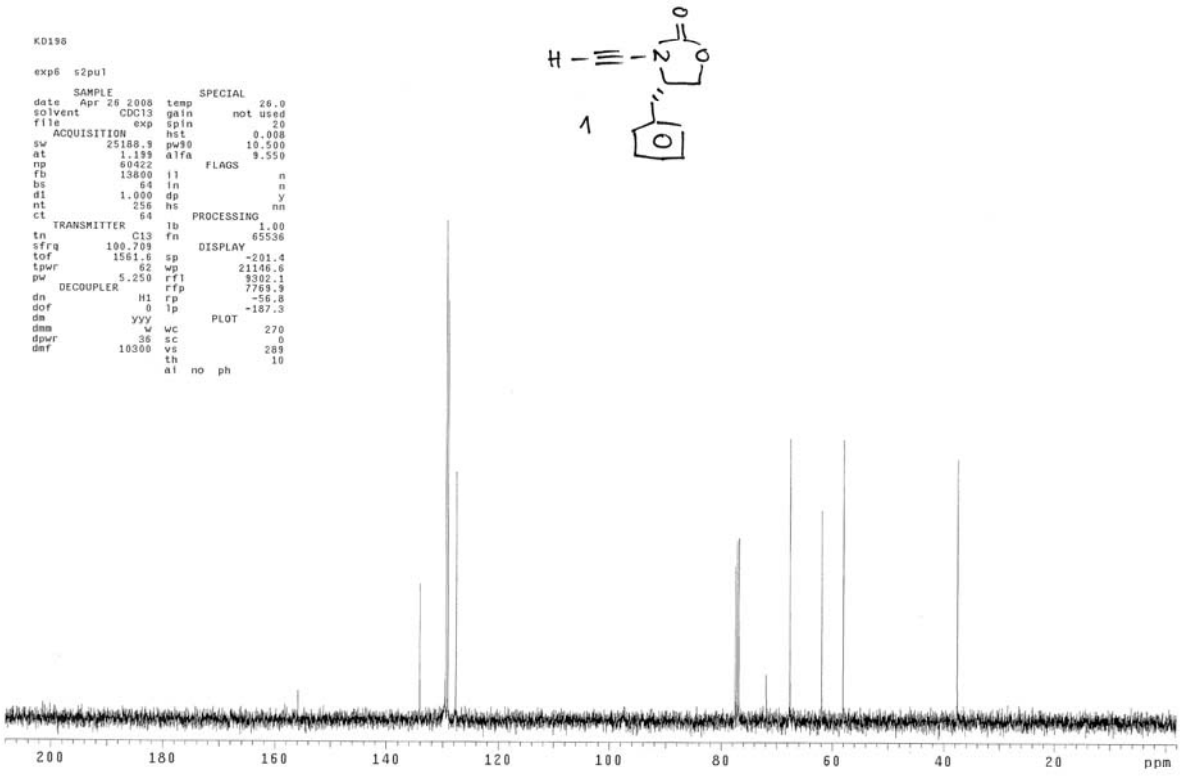
19

2-iodophenol (0.30 mmol, 66 mg), **3** (0.36 mmol, 103 mg), Bu_4NOAc (0.90 mmol, 271 mg), $Pd(OAc)_2$ (0.015 mmol, 3.4 mg), PPh_3 (0.060 mmol, 16 mg) were reacted for 24 h. After flash chromatography (5% acetone in pentane) the title compound was obtained as a colorless solid (35 mg, 0.09 mmol, 31%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.67 (d, 2H, J = 8.0 Hz), 7.45 (d, 1H, J = 7.2 Hz), 7.21–7.32 (m, 9H), 7.18 (td, 1H, J = 8.8 Hz, 1.2 Hz), 6.48 (d, 1H, J = 0.8 Hz), 4.81 (s, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, CDCl3) δ (ppm) 152.4, 147.8, 144.3, 136.0, 135.5, 129.9 (2C), 128.63 (2C), 128.59 (2C), 128.1, 127.8 (2C), 124.6, 123.1, 121.3, 111.1, 103.0, 53.3, 21.8. HRMS (ES) calcd for $C_{22}H_{19}NO_3SNa$ (MNa^+) 400.0983, found 400.0983. mp = 111.8 – 114.3 °C.

NMR Spectra

Compound 1

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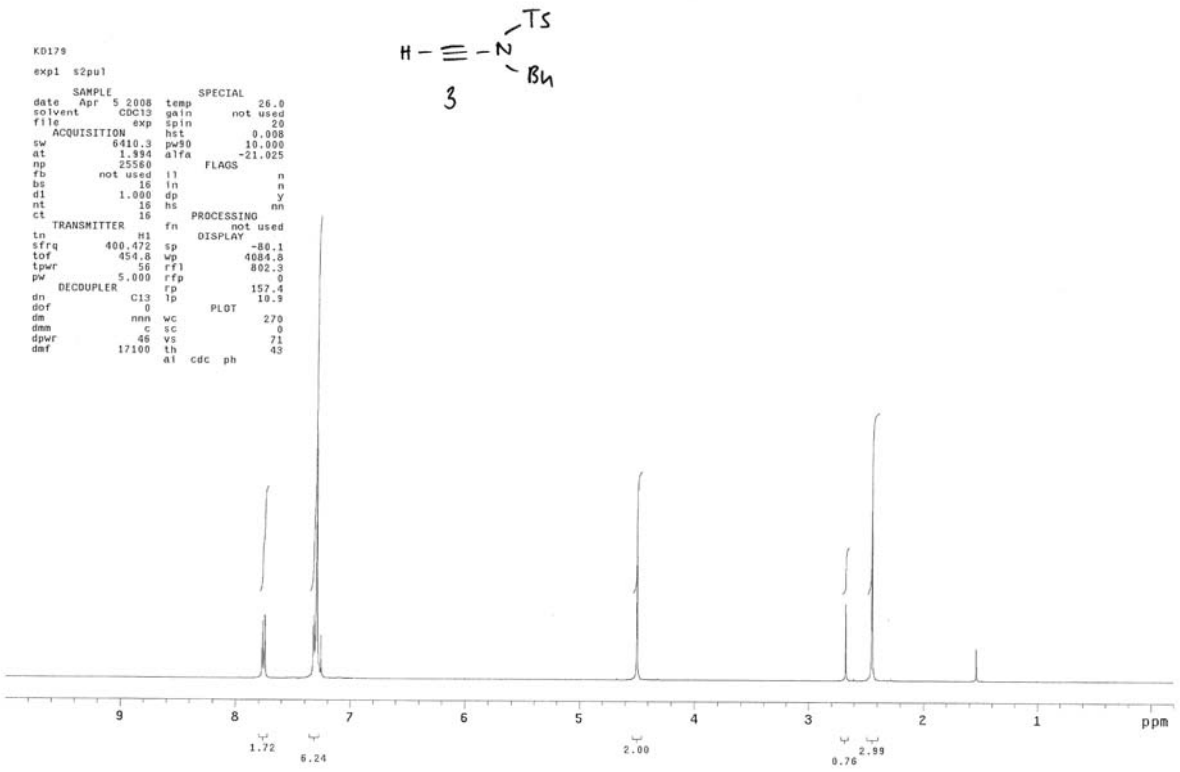
ppm

Compound 3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ct 16
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pw 5.000
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al cdc ph
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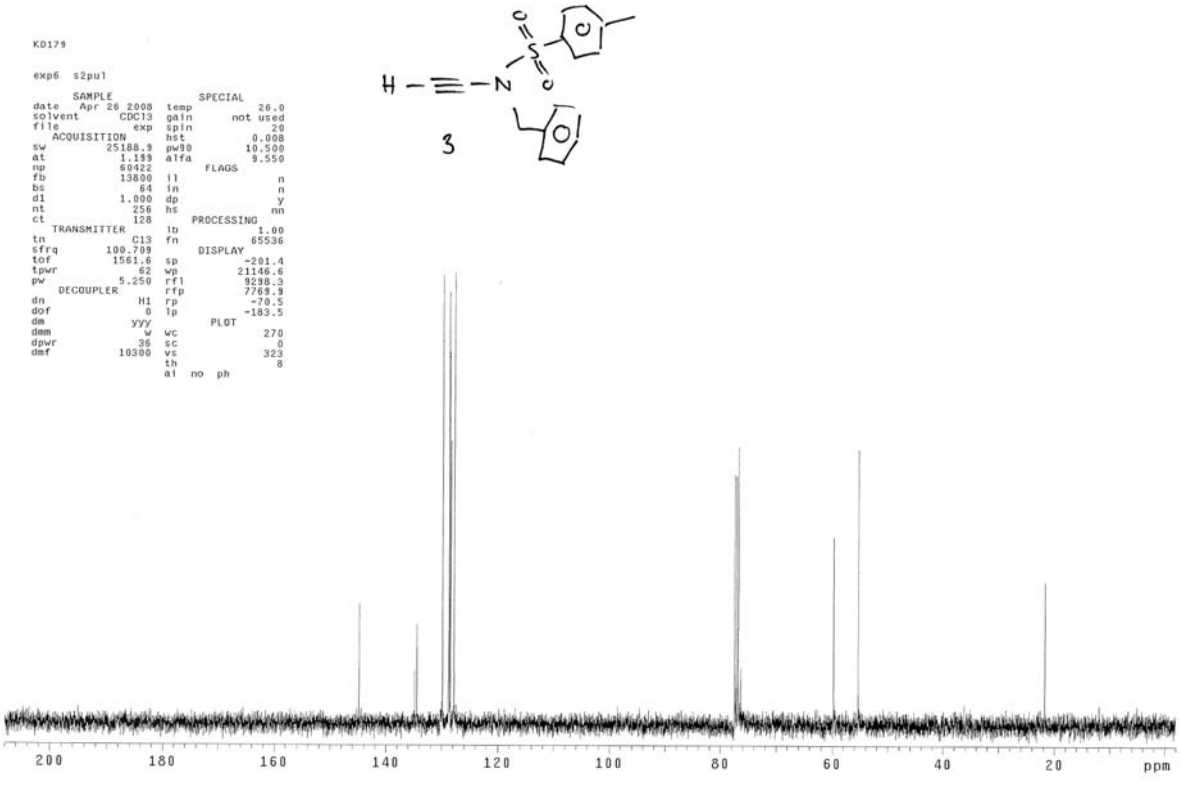
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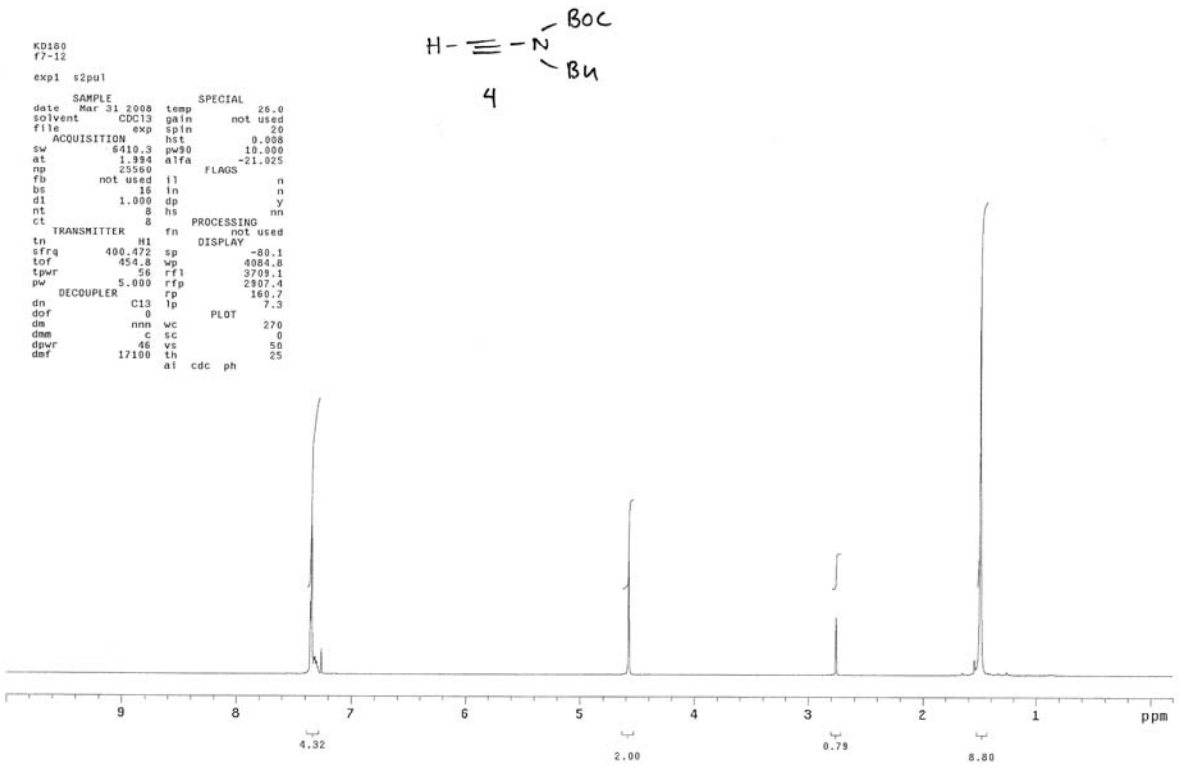
ppm

Compound 4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H-
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Bu
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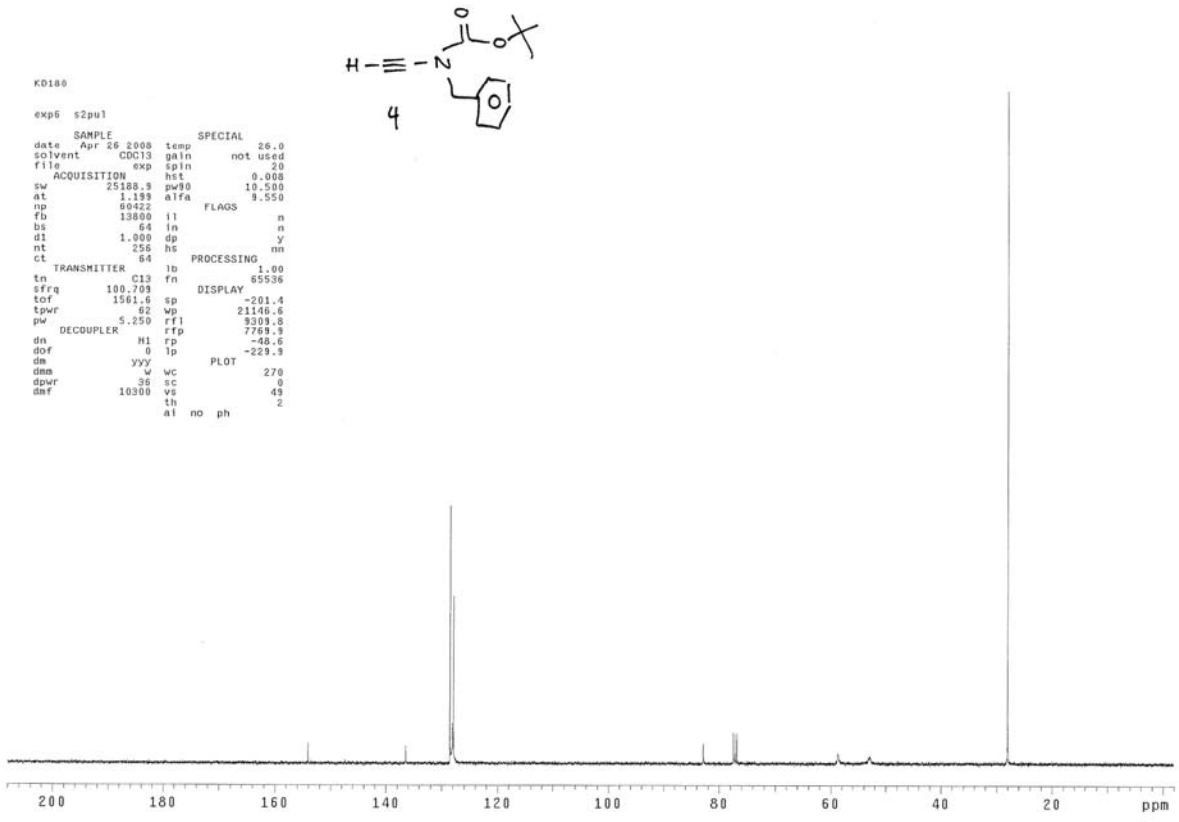
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date Apr 26 2008
solvent CDCl3
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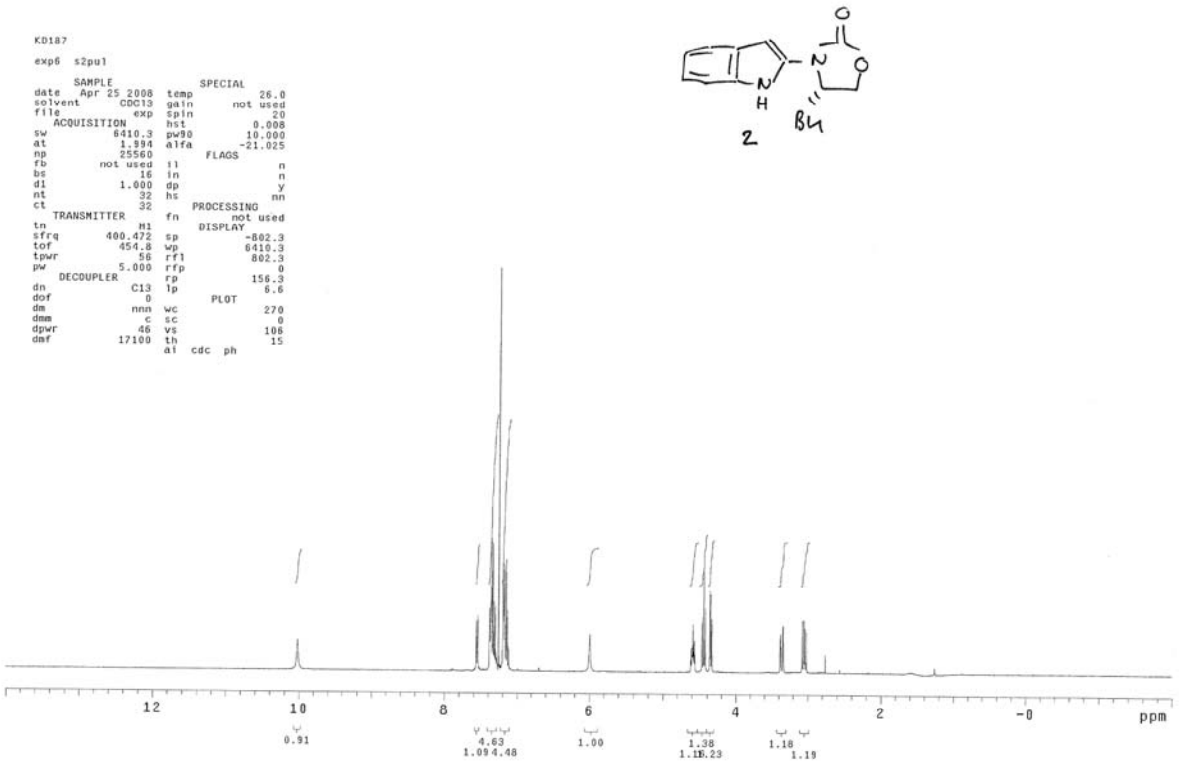
ppm

Compound 2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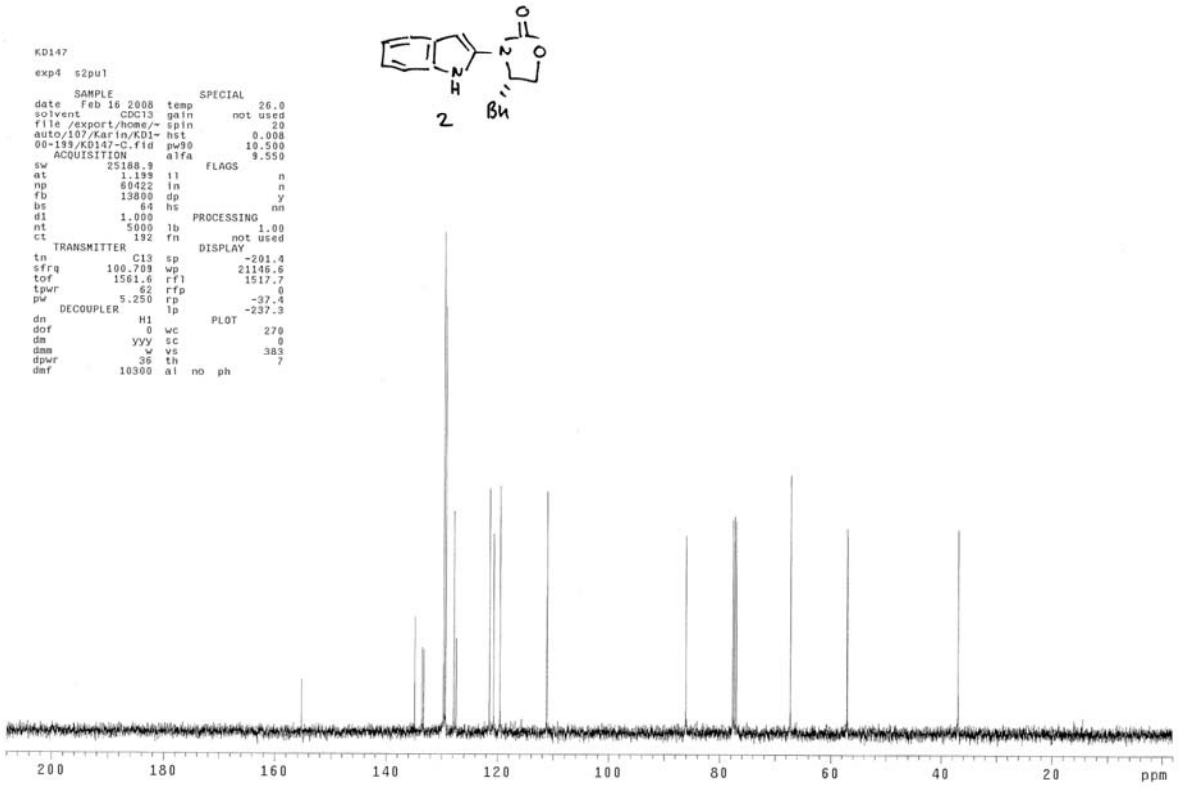
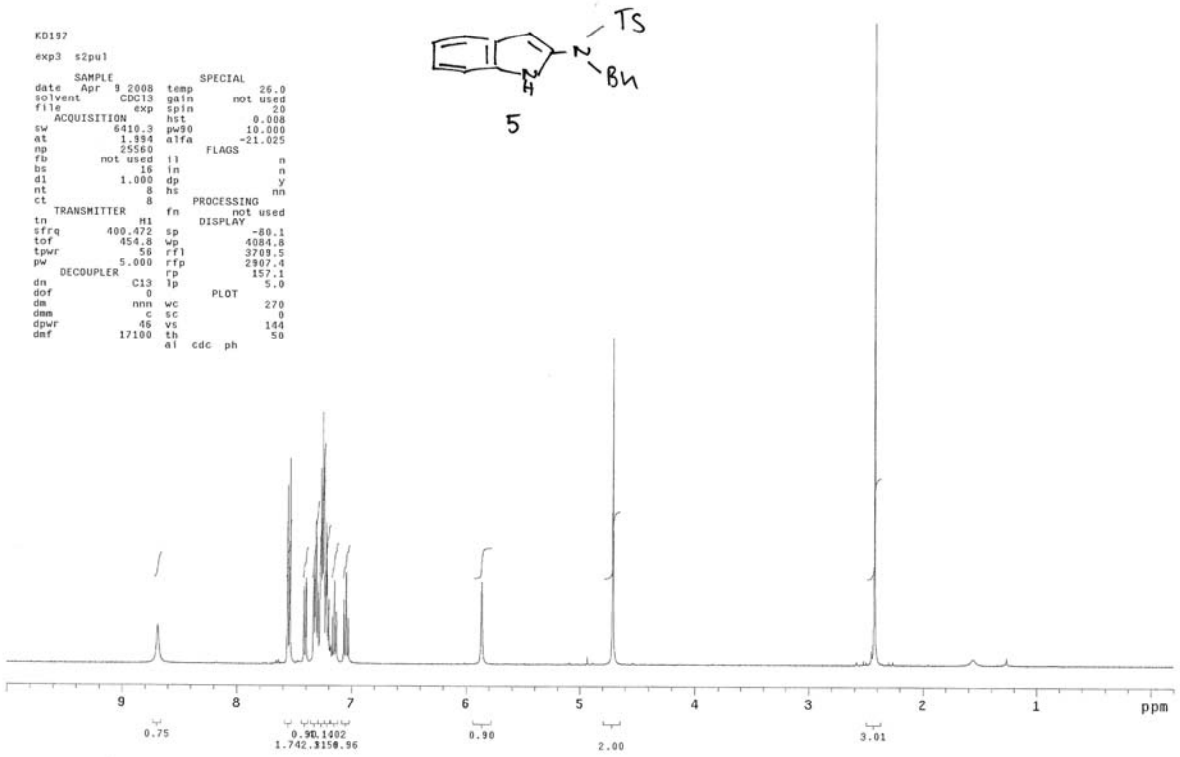
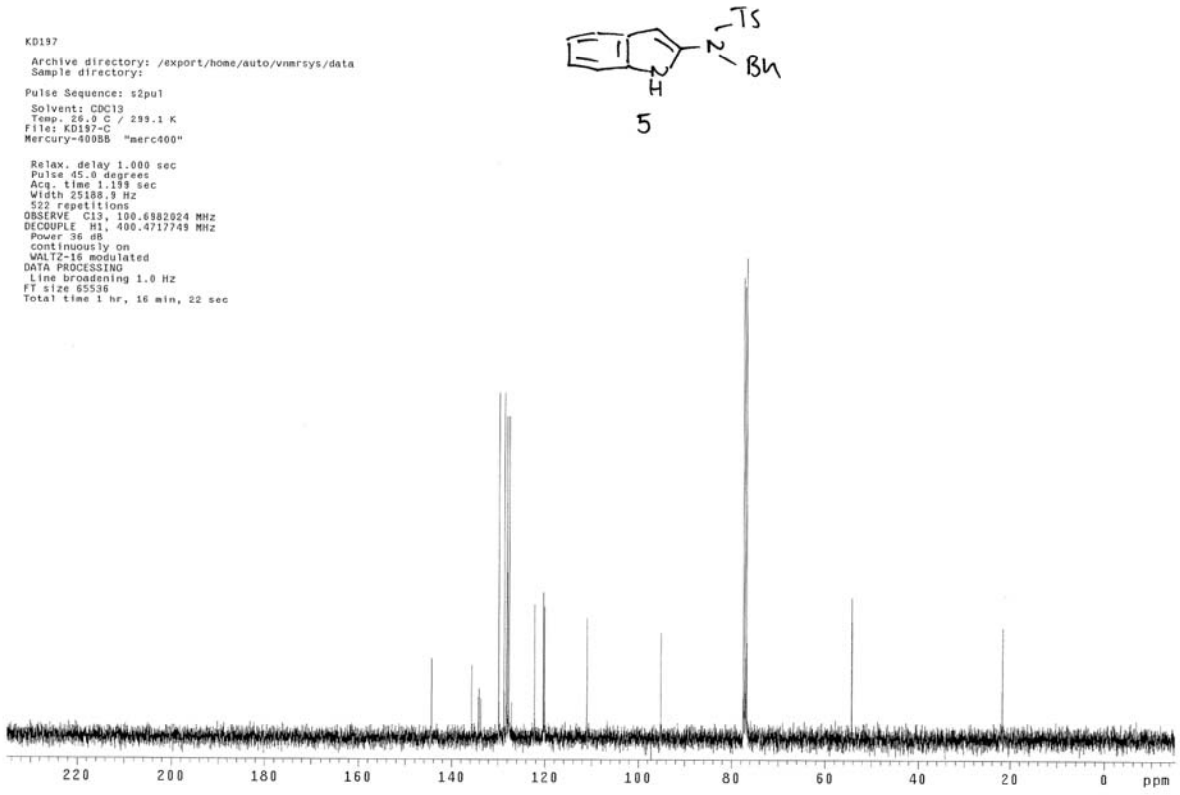
1.18
1.23
1.18
1.19

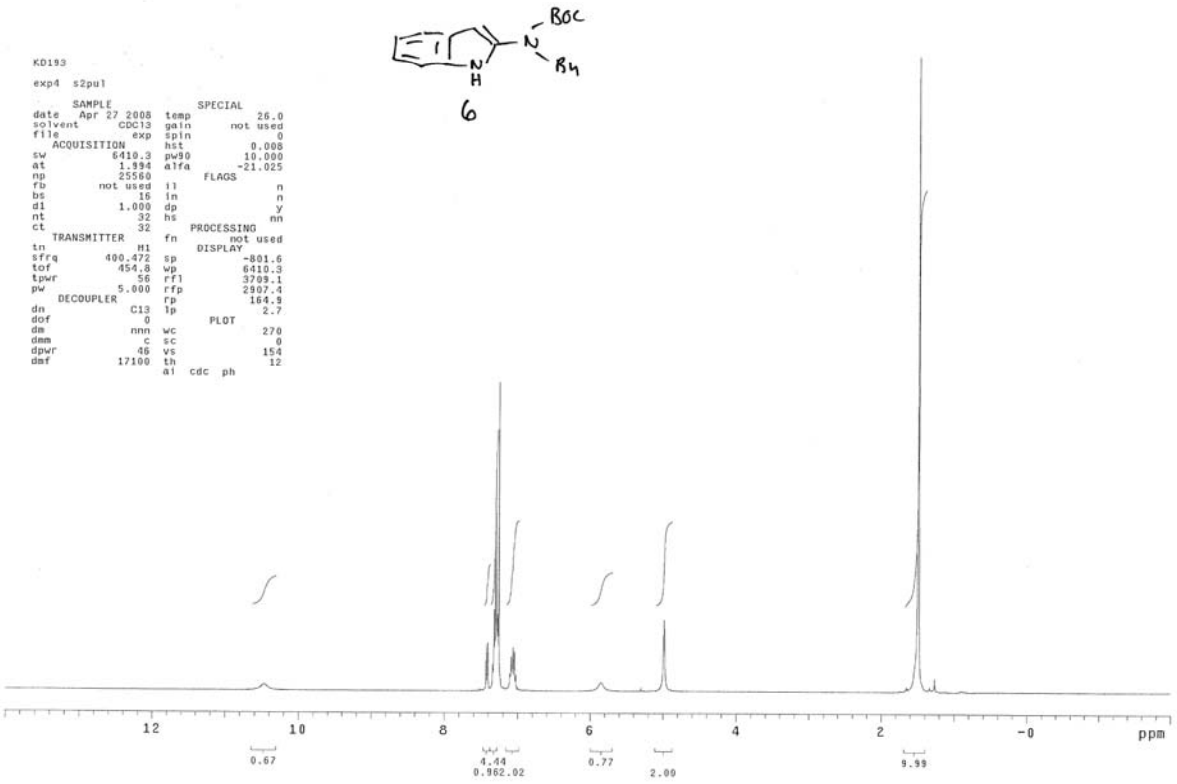

KD147
exp4 s2pul
SAMPLE
date Feb 16 2008
solvent CDCl3
file /export/home/~
auto/107/Karin/KD1~
00-199/KD147-C.fid
ACQUISITION
sw 25188.9
at 1.199
np 60422
fb 13800
bs 64
d1 1.000
nt 5000
ct 192
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -201.4
wp 21146.6
rfl 1517.7
rfp 0
rp -37.4
lp -237.3
PLOT
wc 270
sc 0
vs 383
th 7
al no ph
2
Bn
200
180
160
140
120
100
80
60
40
20
ppm

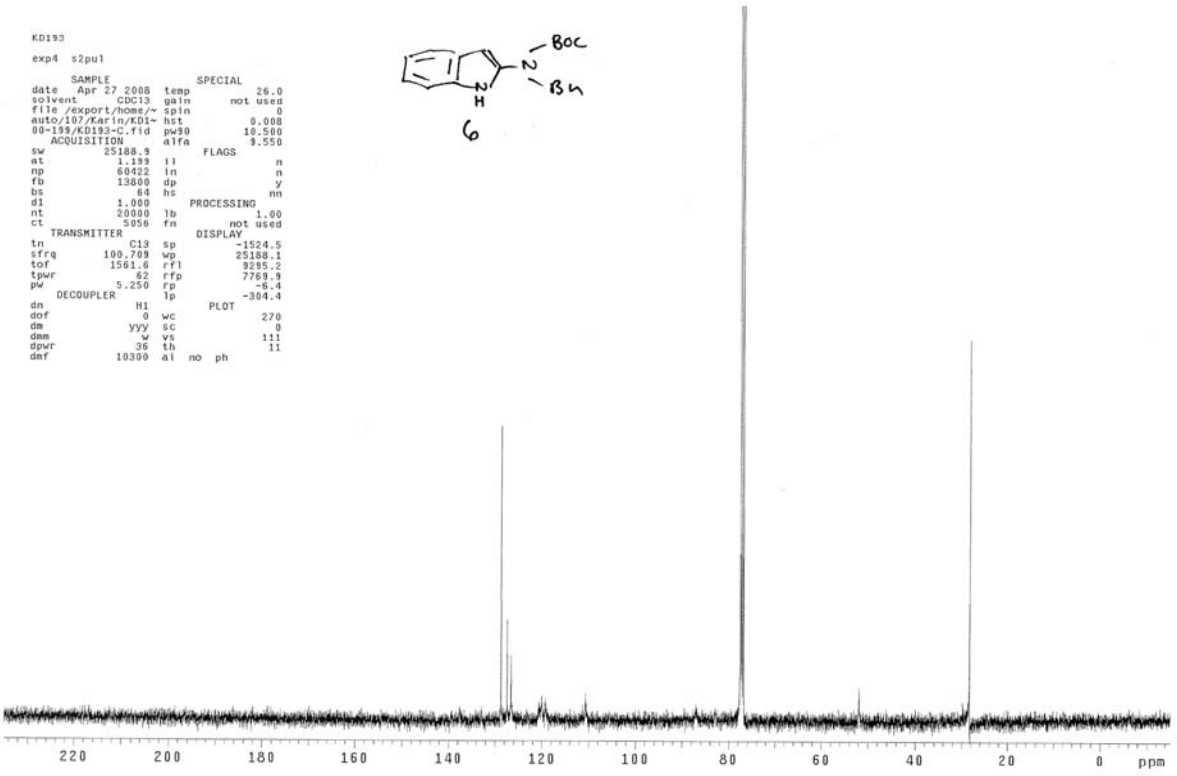
Compound 5


KD197
exp3 s2pul
SAMPLE
date Apr 9 2008
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -80.1
wp 4084.6
rfl 3709.5
rfp 2907.4
rp 157.1
lp 5.0
PLOT
wc 270
sc 0
vs 144
th 58
al cdc ph
Ts
Bn
5
9
8
7
6
5
4
3
2
1
ppm
0.75
0.90
2.00
3.01

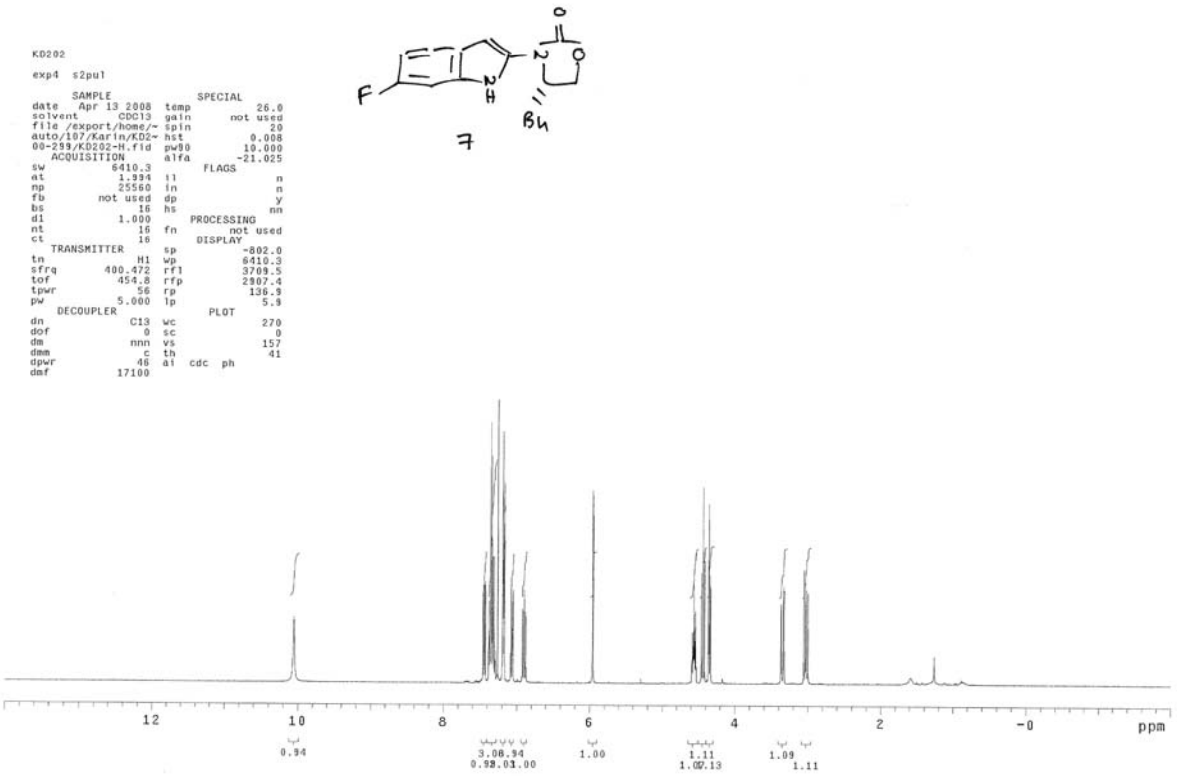

KD197
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: KD197-C
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25188.9 Hz
522 repetitions
OBSERVE C13, 100.6982024 MHz
DECOUPLE H1, 400.4717749 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 16 min, 22 sec
Ts
Bn
5
220
200
180
160
140
120
100
80
60
40
20
0
ppm

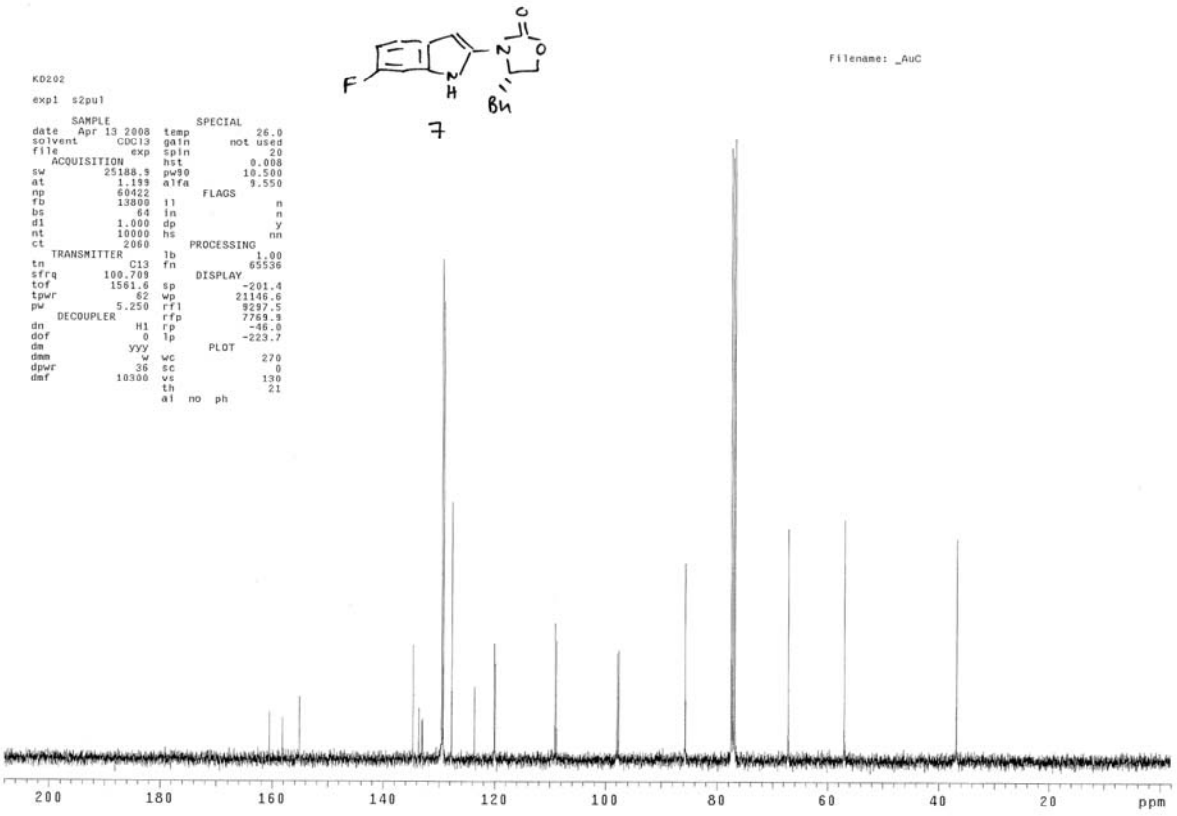
Compound 6

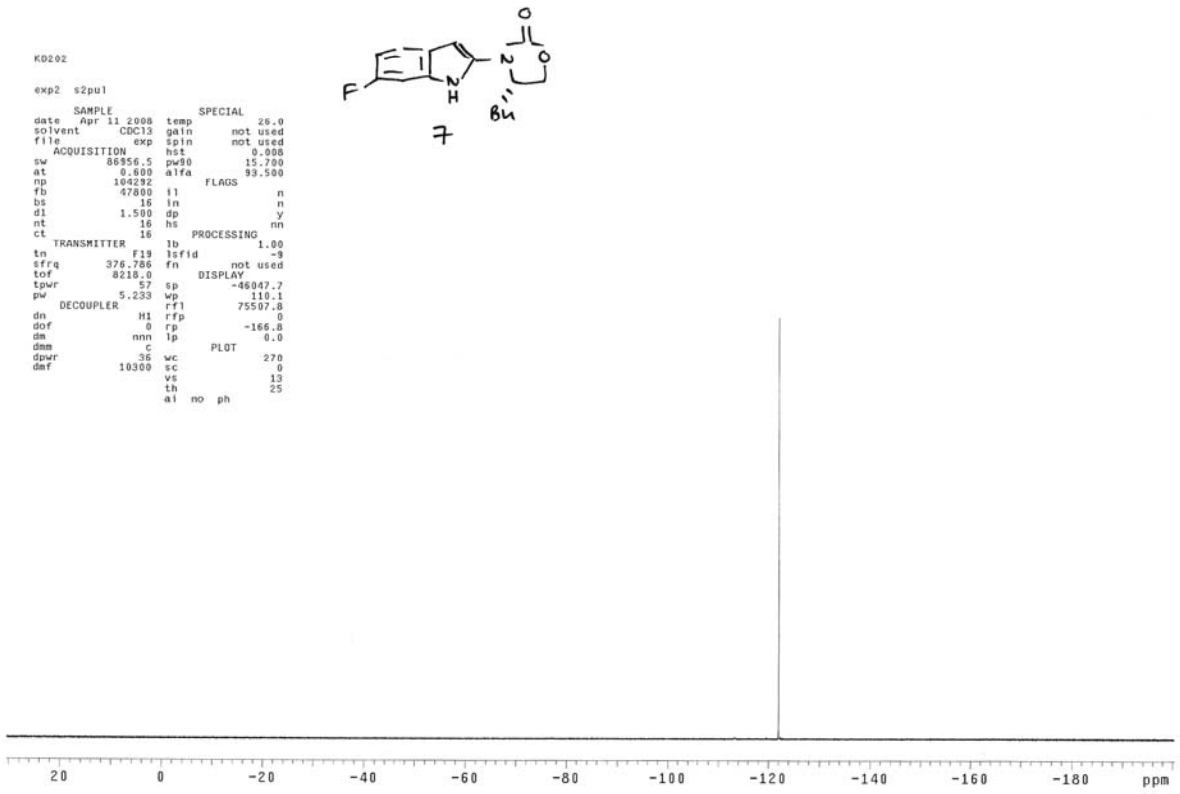

KD193
exp4 s2pul
SAMPLE
date Apr 27 2008
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 0
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -801.6
wp 6410.3
rfl 3709.1
rfp 2907.4
rp 164.9
lp 2.7
PLOT
wc 270
sc 0
vs 154
th 12
ai cdc ph
Boc
N
Bn
N
H
6
12
10
8
6
4
2
-0
ppm
0.67
4.48
0.962.02
0.77
2.00
9.99


KD193
exp4 s2pul
SAMPLE
date Apr 27 2008
solvent CDCl3
file /export/home/~
auto/107/Karin/KD1~
00-199/KD193-C.fid
ACQUISITION
sw 25188.9
at 1.139
np 60422
fb 13800
bs 64
d1 1.000
nt 20000
ct 5056
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 0
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1524.5
wp 25188.1
rfl 9295.2
rfp 7769.8
rp -6.4
lp -304.4
PLOT
wc 270
sc 0
vs 111
th 11
ai no ph
Boc
N
Bn
N
H
6
220
200
180
160
140
120
100
80
60
40
20
0
ppm

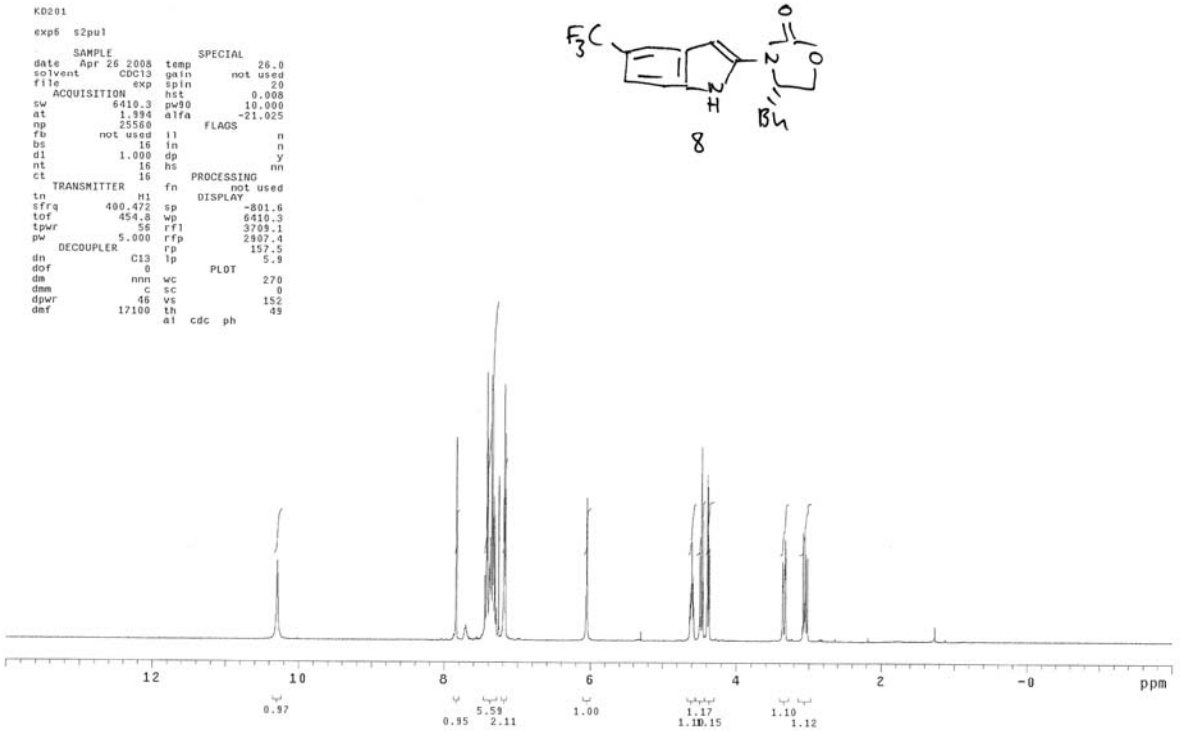
Compound 7

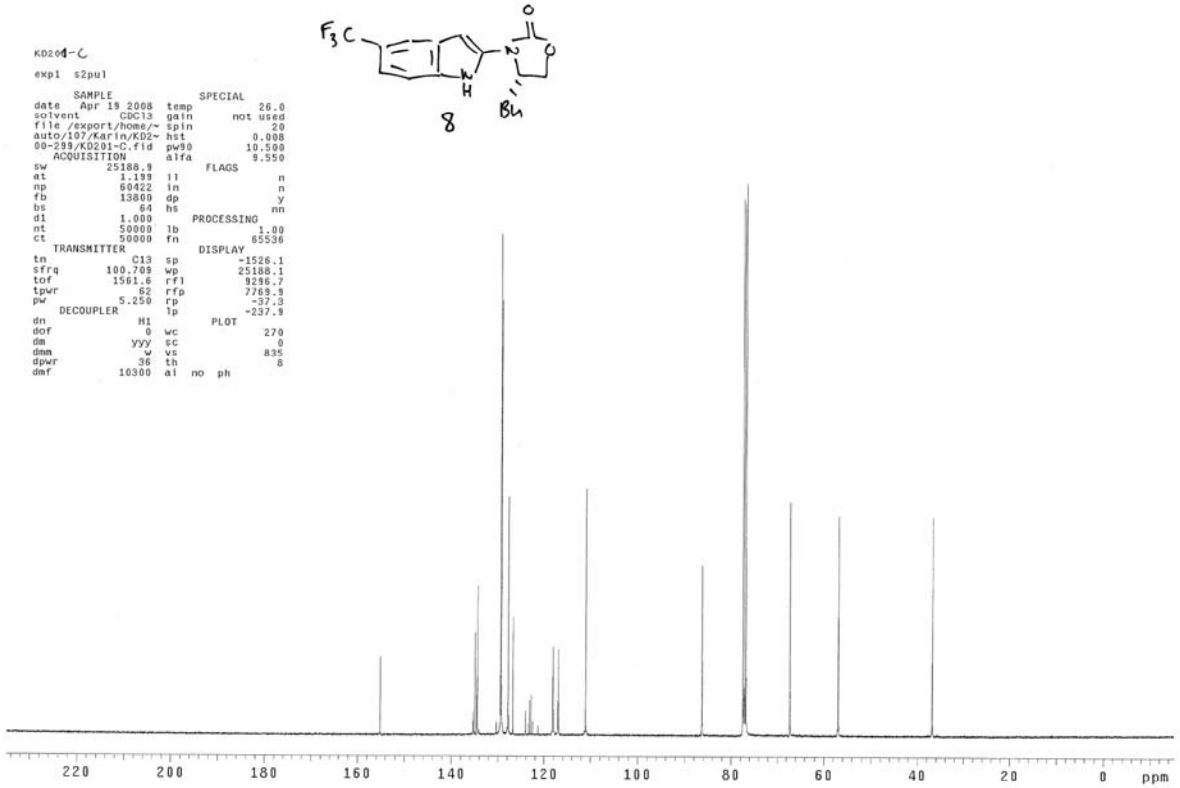

KD202
exp4 s2pul
SAMPLE
date Apr 13 2008
solvent CDCl3
file /export/home/~
auto/107/Karin/KD2~
00-299/KD202-H.fid
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -802.0
wp 6410.3
rf1 3709.5
rfp 2907.4
rp 136.9
lp 5.9
PLOT
wc 270
sc 0
vs 157
th 41
al cdc ph
F
N
H
O
O
N
Bu
7
12
10
8
6
4
2
-0
ppm
0.94
3.08.94
1.00
1.11
1.09
1.11

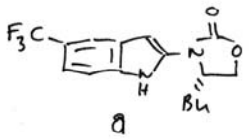

KD202
exp1 s2pul
SAMPLE
date Apr 13 2008
solvent CDCl3
file exp
ACQUISITION
sw 25188.9
at 1.199
np 60422
fb 13800
bs 64
d1 1.000
nt 10000
ct 2060
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn 65536
DISPLAY
sp -201.4
wp 21146.6
rf1 9297.5
rfp 7769.9
rp -46.8
lp -223.7
PLOT
wc 270
sc 0
vs 130
th 21
al no ph
Filename: _AuC
F
N
H
O
O
N
Bu
7
200
180
160
140
120
100
80
60
40
20
ppm


KD202
exp2 s2pul
SAMPLE
date Apr 11 2008
solvent CDCl3
file exp
ACQUISITION
sw 86956.5
at 0.600
np 104292
fb 47800
bs 16
d1 1.500
nt 16
ct 16
TRANSMITTER
tn F19
sfrq 376.786
tof 8218.0
tpwr 57
pw 5.233
DECOUPLER
dn H1
dof 0
dm nnn
dmm c
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin not used
hst 0.000
pw90 15.700
alfa 93.500
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
lsfid -9
fn not used
DISPLAY
sp -46047.7
wp 110.1
rfl 75507.8
rfp 0
rp -166.8
lp 0.0
PLOT
wc 270
sc 0
vs 13
th 25
ai no ph
F
N
H
N
O
O
Bn
7
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm

Compound 8


KD201
exp6 s2pul
SAMPLE
date Apr 26 2008
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -801.6
wp 6410.3
rf1 3709.1
rfp 2907.4
rp 157.5
lp 5.9
PLOT
wc 270
sc 0
vs 152
th 49
al cdc ph
F3C
N
H
O
O
BH
8
12
10
8
6
4
2
-0
ppm
0.97
0.95
5.59
2.11
1.00
1.17
1.15
1.10
1.12


KD201-C
exp1 s2pul
SAMPLE
date Apr 19 2008
solvent CDCl3
file /export/home/~
auto/107/Karin/KD2~
00-299/KD201-C.fid
ACQUISITION
sw 25188.9
at 1.199
np 60422
fb 13800
bs 64
d1 1.000
nt 50000
ct 50000
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn 65536
DISPLAY
sp -1526.1
wp 25188.1
rf1 9286.7
rfp 7769.9
rp -37.3
lp -237.9
PLOT
wc 270
sc 0
vs 835
th 8
al no ph
F3C
N
H
O
O
BH
8
220
200
180
160
140
120
100
80
60
40
20
0
ppm

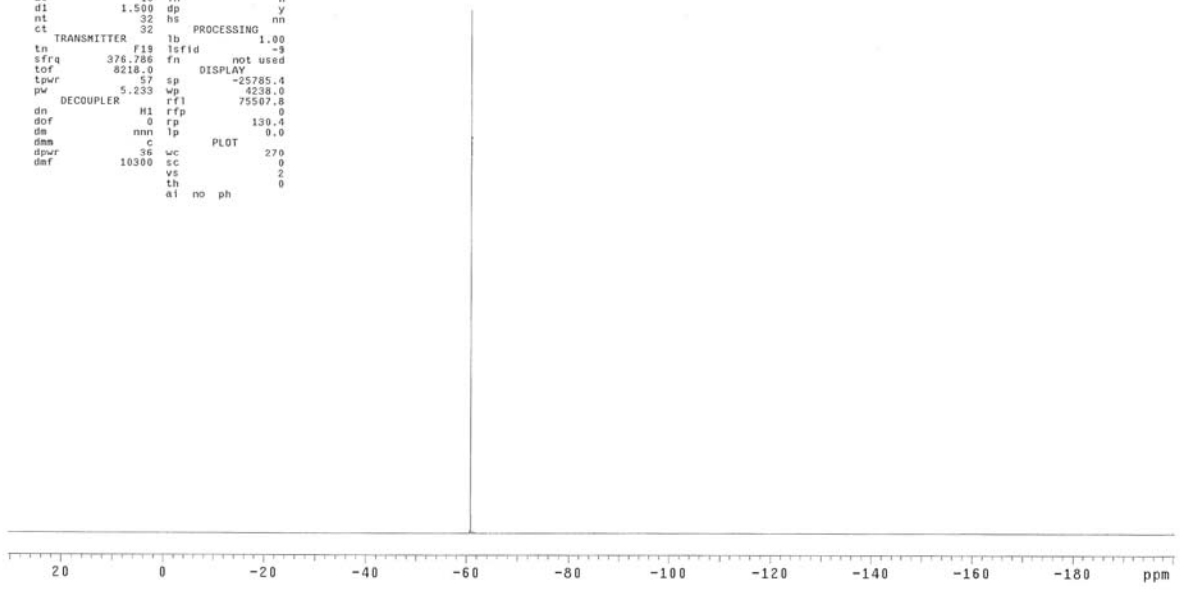


8a

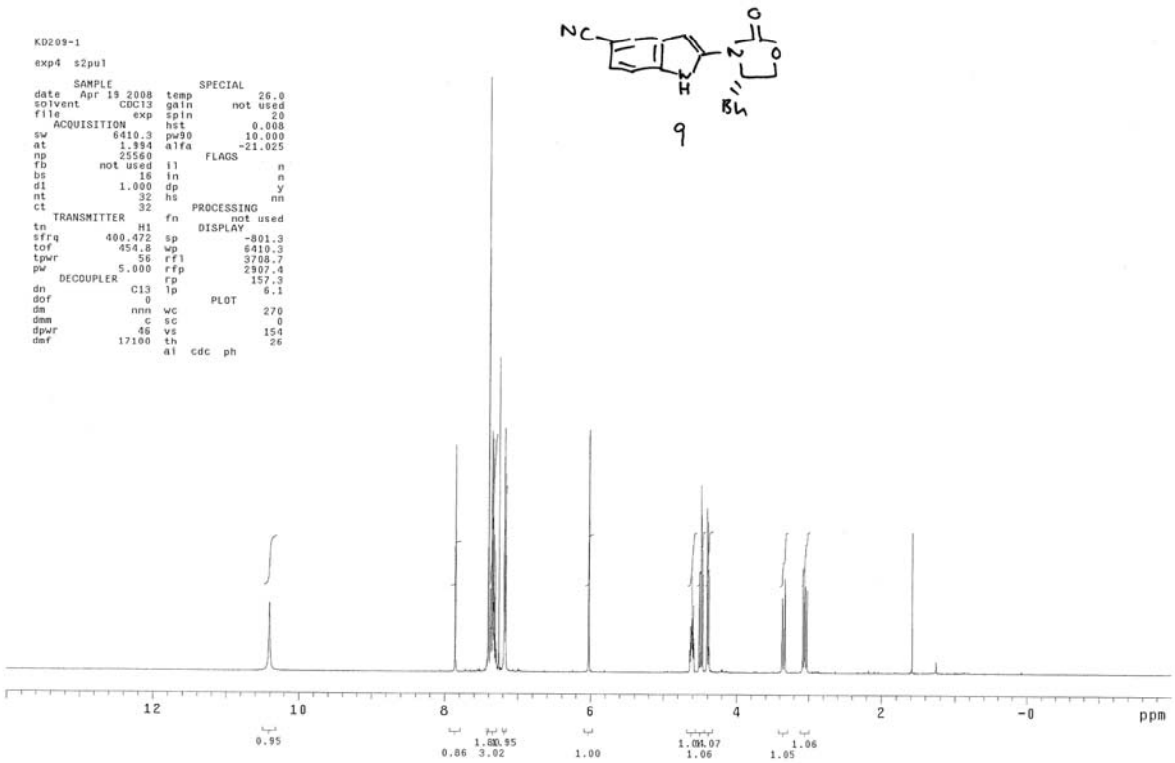
KD201
after recystallization

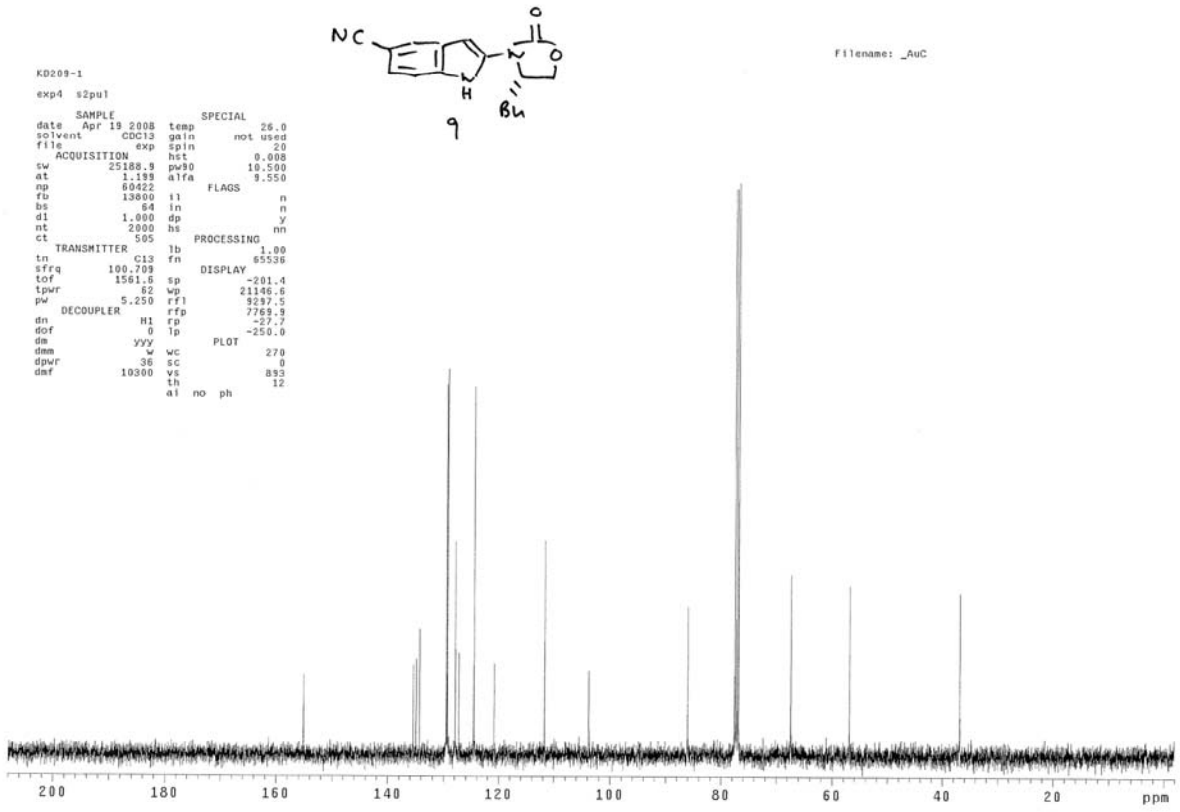
expl s2pul

SAMPLE		SPECIAL	
date	Apr 19 2008	temp	26.0
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	86956.5	pw90	15.700
at	0.600	alfa	93.500
np	104292	FLAGS	
fb	47800	il	n
bs	16	in	n
d1	1.500	dp	y
nt	32	hs	nn
ct	32	PROCESSING	
TRANSMITTER		lb	1.00
tn	F19	lsfid	-9
sfrq	376.786	fn	not used
tof	8218.0	DISPLAY	
tpwr	57	sp	-25785.4
pw	5.233	wp	4238.0
DECOUPLER		rfl	75507.8
dn	H1	rfp	0
dof	0	rp	130.4
dm	nnn	lp	0.0
dmm	c	PLOT	
dpwr	36	wc	270
dmf	10300	sc	0
		vs	2
		th	0
		ai no ph	

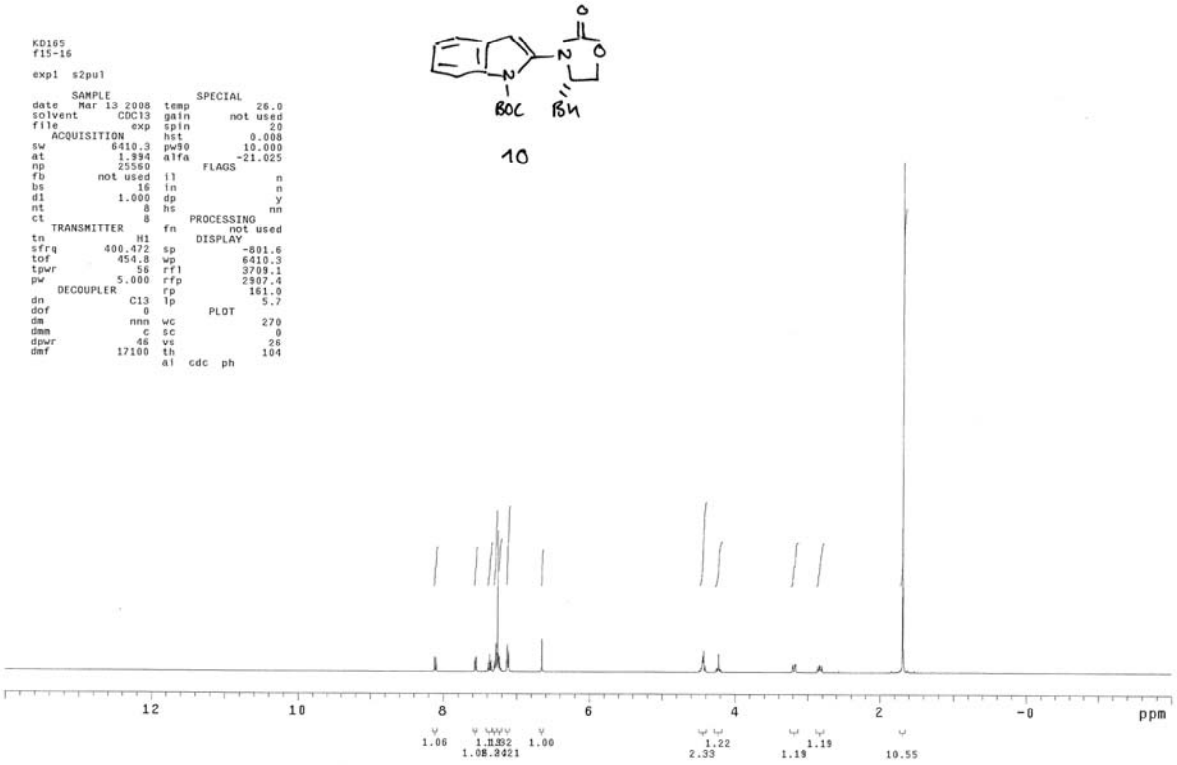


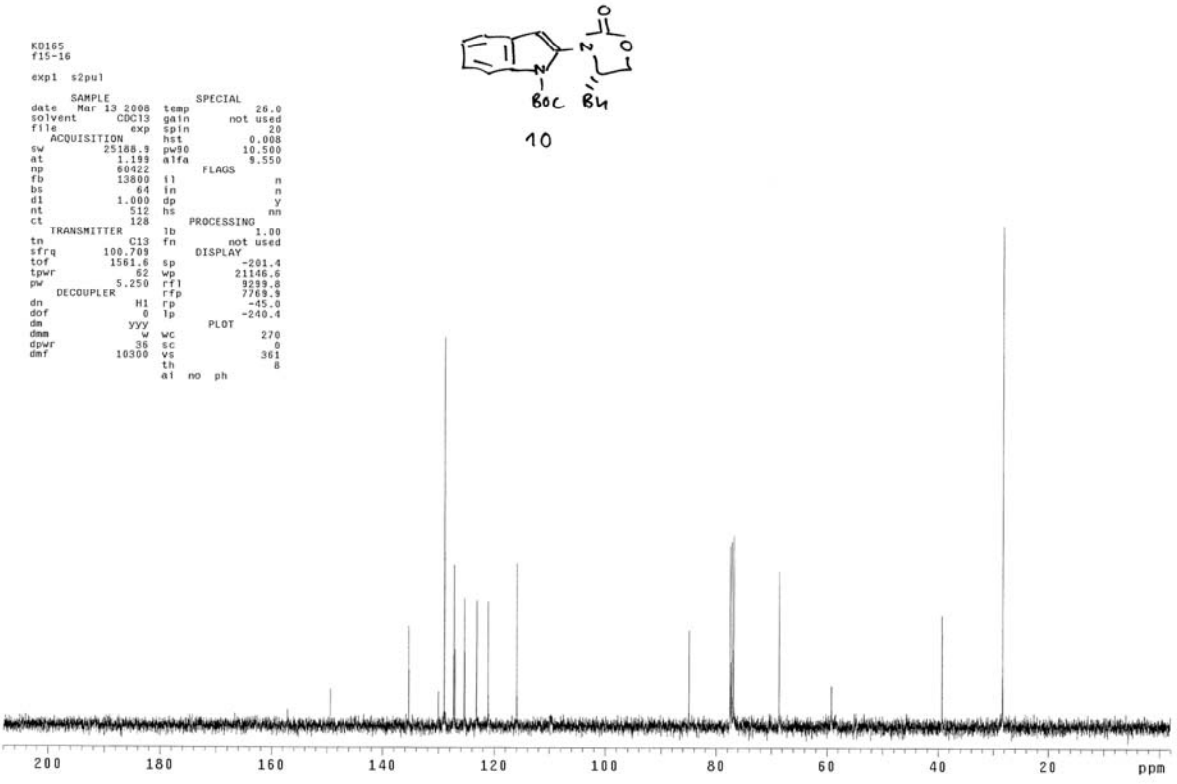
Compound 9


KD209-1
exp4 s2pul
SAMPLE
date Apr 19 2008
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -801.3
wp 6410.3
rf1 3708.7
rfp 2907.4
rp 157.3
lp 6.1
PLOT
wc 270
sc 0
vs 154
th 26
al cdc ph
NC
O
N
H
Bu
9
12
10
8
6
4
2
-0
ppm
0.95
0.86
3.02
1.00
1.06
1.05

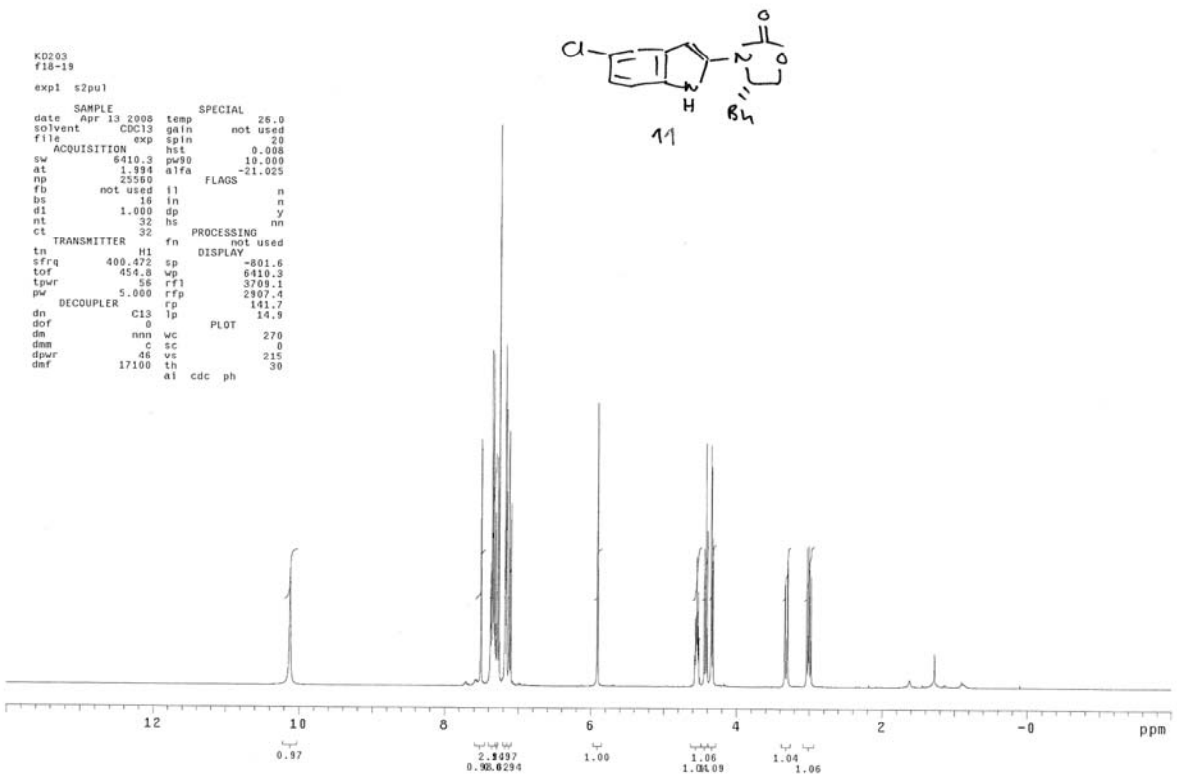

KD209-1
exp4 s2pul
SAMPLE
date Apr 19 2008
solvent CDCl3
file exp
ACQUISITION
sw 25188.9
at 1.199
np 60422
fb 13600
bs 64
d1 1.000
nt 2000
ct 505
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn 65536
DISPLAY
sp -201.4
wp 21146.6
rf1 9297.5
rfp 7769.9
rp -27.7
lp -250.0
PLOT
wc 270
sc 0
vs 893
th 12
al no ph
Filename: _AuC
NC
O
N
H
Bu
9
200
180
160
140
120
100
80
60
40
20
ppm

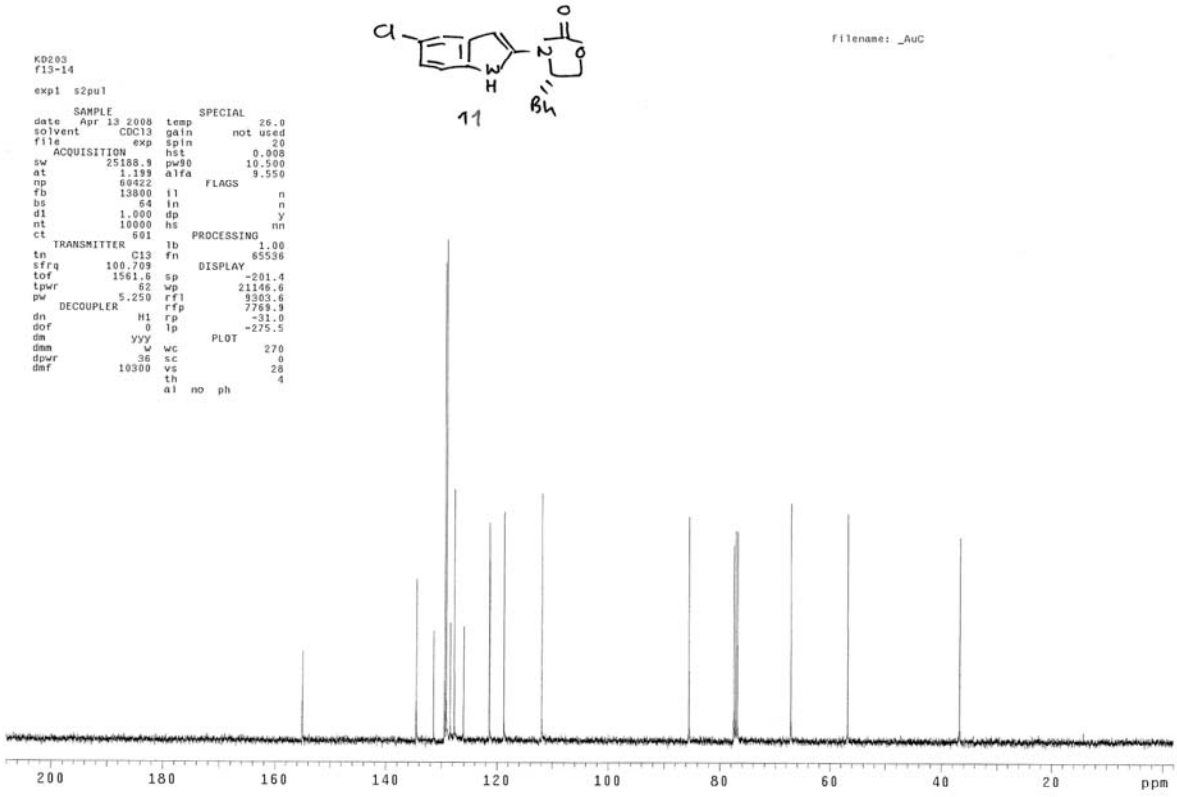
Compound 10


KD165
f15-16
exp1 s2pul
SAMPLE
date Mar 13 2008
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -801.6
wp 6410.3
rf1 3709.1
rfp 2907.4
rp 161.9
lp 5.7
PLOT
wc 270
sc 0
vs 26
th 104
al cdc ph
BOC
BH
10
12
10
8
6
4
2
-0
ppm
1.06
1.00
1.22
2.33
1.19
1.19
10.55

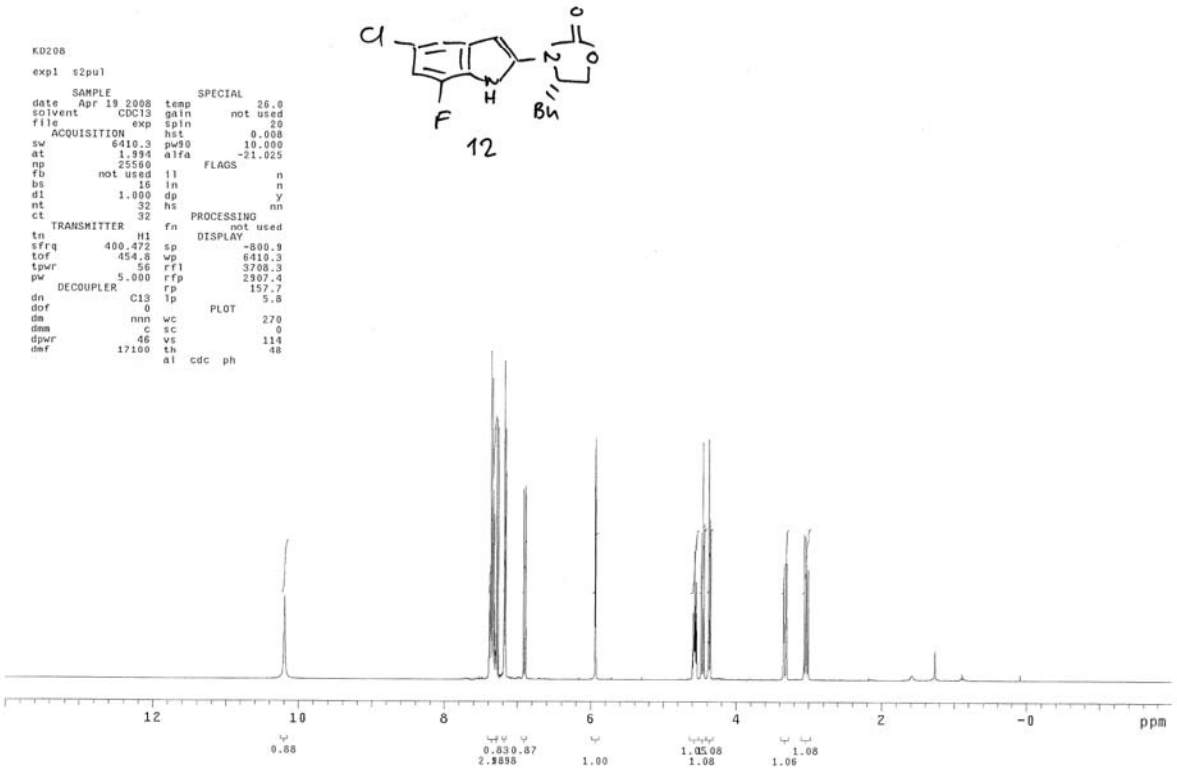

KD165
f15-16
exp1 s2pul
SAMPLE
date Mar 13 2008
solvent CDCl3
file exp
ACQUISITION
sw 25188.9
at 1.199
np 60422
fb 13800
bs 64
d1 1.000
nt 512
ct 128
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
lb 1.00
PROCESSING
fn not used
DISPLAY
sp -201.4
wp 21146.6
rf1 9239.8
rfp 7769.9
rp -45.0
lp -240.4
PLOT
wc 270
sc 0
vs 361
th 8
al no ph
BOC
BH
10
200
180
160
140
120
100
80
60
40
20
ppm

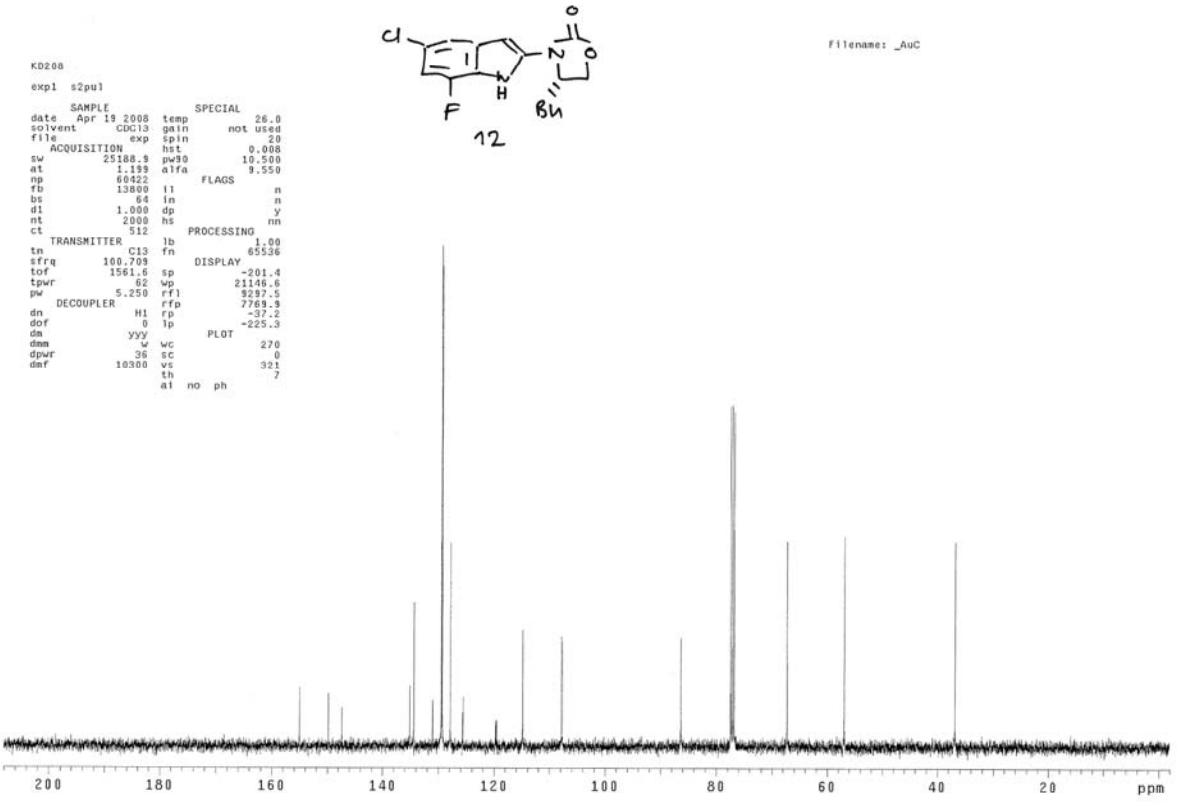
Compound 11

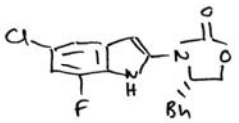

KD203
f18-19
exp1 s2pul
SAMPLE
date Apr 13 2008
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 1.994
np 25580
fb not used
bs 16
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -801.6
wp 6410.3
rf1 3709.1
rfp 2907.4
rp 141.7
lp 14.9
PLOT
wc 270
sc 0
vs 215
th 30
al cdc ph
11
12
10
8
6
4
2
-0
ppm
0.97
1.00
1.06
1.04
1.06


KD203
f13-14
exp1 s2pul
SAMPLE
date Apr 13 2008
solvent CDCl3
file exp
ACQUISITION
sw 25188.9
at 1.189
np 60422
fb 13800
bs 64
d1 1.000
nt 10000
ct 601
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn 65536
DISPLAY
sp -201.4
wp 21146.6
rf1 9303.6
rfp 7769.9
rp -31.0
lp -275.5
PLOT
wc 270
sc 0
vs 28
th 4
al no ph
Filename: _AuC
11
200
180
160
140
120
100
80
60
40
20
ppm

Compound 12


KD208
expl s2pul
SAMPLE
date Apr 19 2008
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -800.9
wp 6410.3
rfl 3208.3
rfp 2907.4
rp 157.7
lp 5.8
PLOT
wc 270
sc 0
vs 114
th 48
al cdc ph
Cl
O
N
O
H
F
Bn
12
12
10
8
6
4
2
-0
ppm
0.88
0.83 0.87
2.9898
1.00
1.05 1.08
1.08
1.08
1.06


KD208
expl s2pul
SAMPLE
date Apr 19 2008
solvent CDCl3
file exp
ACQUISITION
sw 25188.9
at 1.199
np 60422
fb 13800
bs 64
d1 1.000
nt 2000
ct 512
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn 65536
DISPLAY
sp -201.4
wp 21146.6
rf1 9297.5
rfp 7769.9
rp -37.2
lp -225.3
PLOT
wc 270
sc 0
vs 321
th 7
al no ph
Filename: _AuC
Cl
O
N
O
H
F
Bn
12
200
180
160
140
120
100
80
60
40
20
ppm

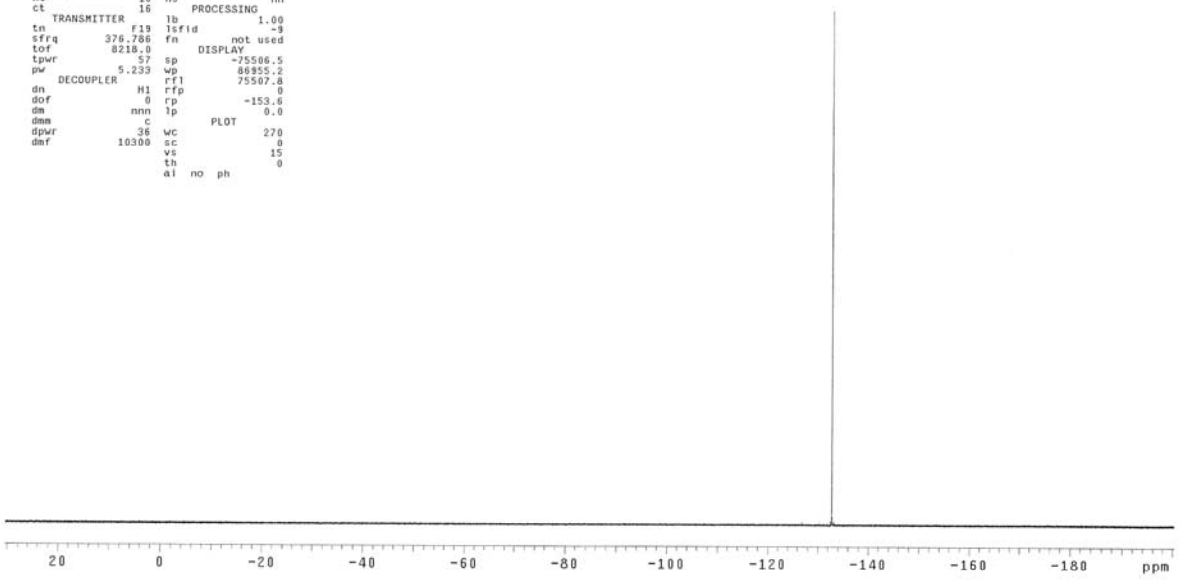


12

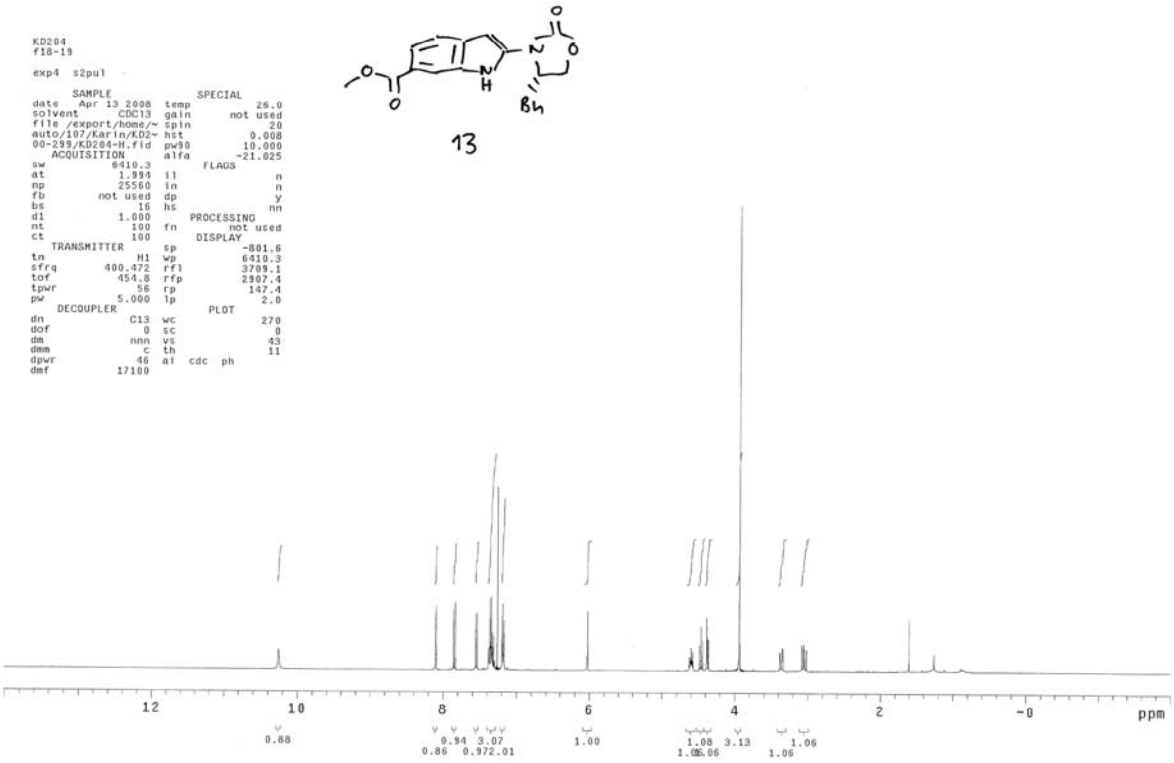
KD208

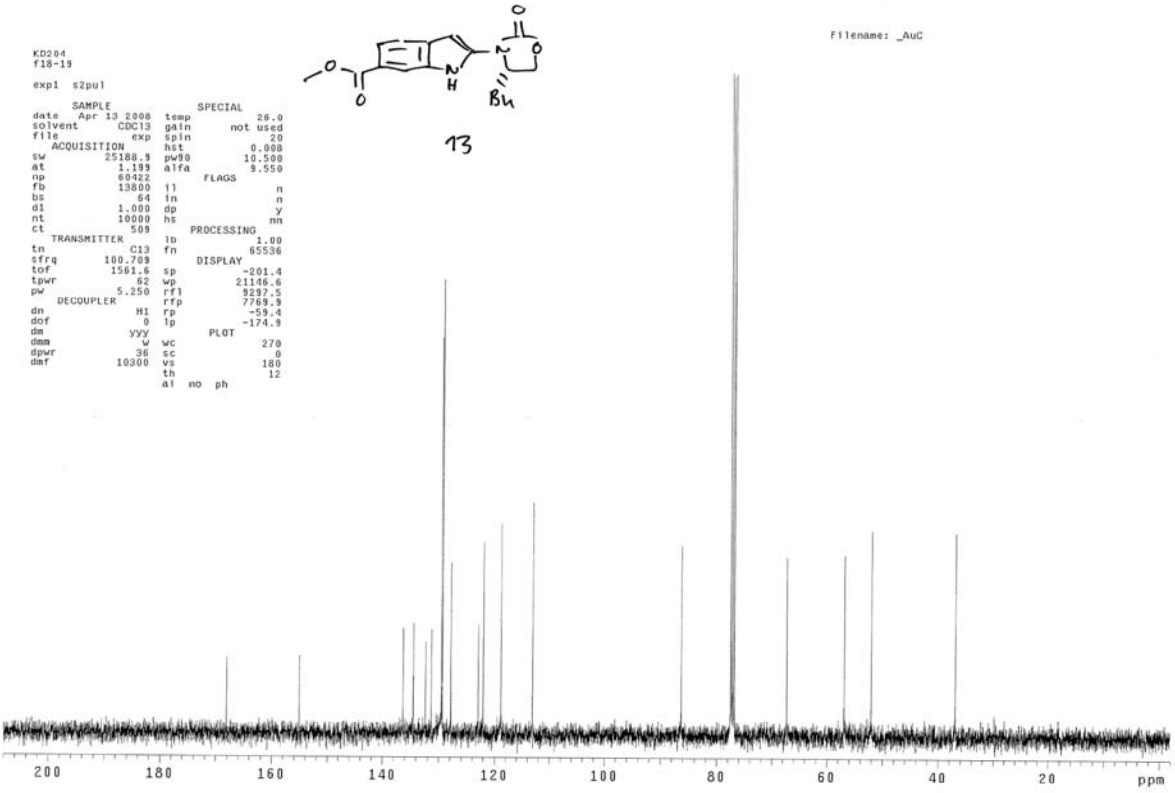
expl s2pul

SAMPLE		SPECIAL	
date	Apr 19 2008	temp	26.0
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	86956.5	pw90	15.700
at	0.600	alfa	93.500
np	104292	FLAGS	
fb	47800	il	n
bs	16	in	n
d1	1.500	dp	y
nt	16	hs	nn
ct	16	PROCESSING	
TRANSMITTER		lb	1.00
tn	F19	lsfid	-9
sfrq	376.786	fn	not used
tof	8218.0	DISPLAY	
tpwr	57	sp	-75506.5
pw	5.233	wp	86955.2
DECOUPLER		rfl	75507.8
dn	H1	rfp	0
dof	0	rp	-153.6
dm	nnn	lp	0.0
dmm	c	PLOT	
dpwr	36	wc	270
dmf	10300	sc	0
		vs	15
		th	0
		al no ph	

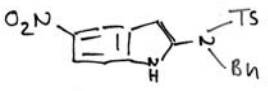


Compound 13


KD204
f18-19
exp4 s2pul
SAMPLE
date Apr 13 2008
solvent CDCl3
file /export/home/~
auto/107/Karin/KD2~
00-299/KD204-H.fid
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 100
ct 100
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -801.6
wp 6410.3
rf1 3709.1
rfp 2907.4
rp 147.4
lp 2.0
PLOT
wc 270
sc 0
vs 43
th 11
al cdc ph
13
12
10
8
6
4
2
-0
ppm
0.88
0.94
3.07
0.86
0.97
2.01
1.00
1.08
3.13
1.06
1.06
1.06


KD204
f18-19
exp1 s2pul
SAMPLE
date Apr 13 2008
solvent CDCl3
file exp
ACQUISITION
sw 25188.8
at 1.189
np 60422
fb 13800
bs 64
d1 1.000
nt 10000
ct 509
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn 65536
DISPLAY
sp -201.4
wp 21146.6
rf1 9297.5
rfp 7769.9
rp -59.4
lp -174.9
PLOT
wc 270
sc 0
vs 180
th 12
al no ph
13
Filename: _AuC
200
180
160
140
120
100
80
60
40
20
ppm

Compound 14

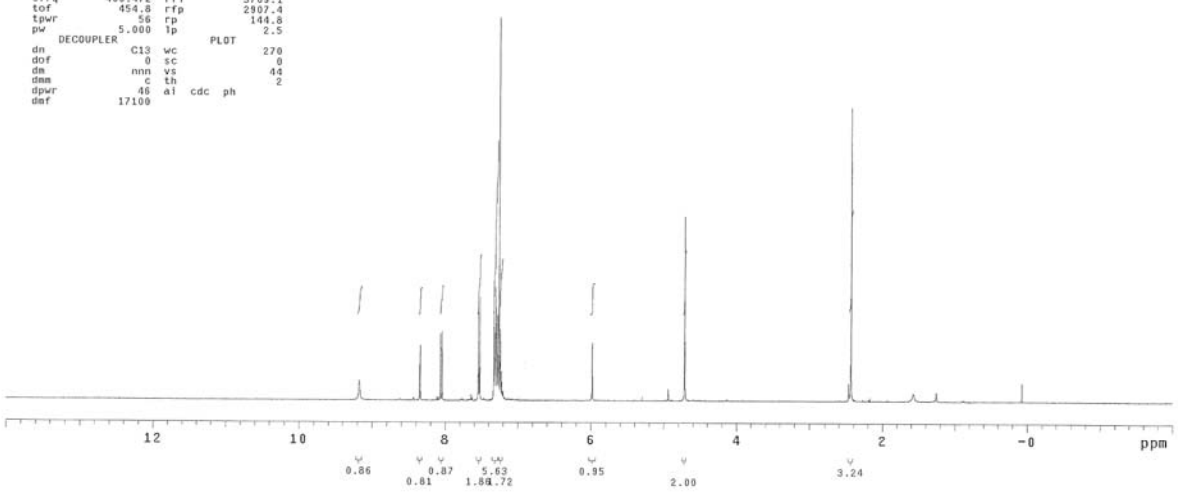


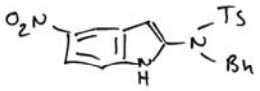
14

KD199
f34-35

exp4 s2pul

SAMPLE		SPECIAL	
date	Apr 13 2008	temp	26.0
solvent	CDCl3	gain	not used
file	/export/home/~	spin	20
auto/107/Karin/KD1~		hst	0.008
00-199/KD199-H.fid		pw90	10.000
ACQUISITION		alfa	-21.025
sw	8410.3	FLAGS	
at	1.994	il	n
np	25560	in	n
fb	not used	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	16	fn	not used
ct	16	DISPLAY	
TRANSMITTER		sp	-801.6
tn	H1	wp	6410.3
sfrq	400.472	rf1	3709.1
tof	454.8	rfp	2907.4
tpwr	56	rp	144.8
pw	5.000	lp	2.5
DECOUPLER		PLOT	
dn	C13	wc	270
dof	0	sc	0
dm	nnn	vs	44
dmm	c	th	2
dpwr	46	al cdc ph	
dmf	17100		





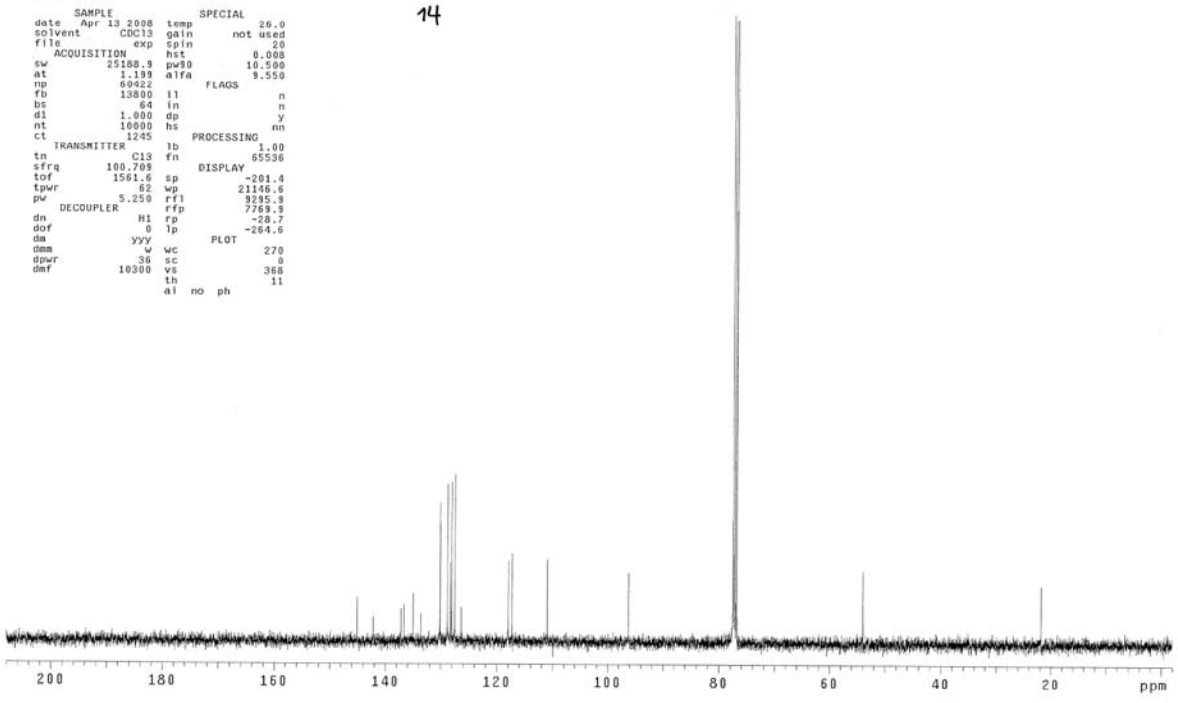
14

Filename: _AuC

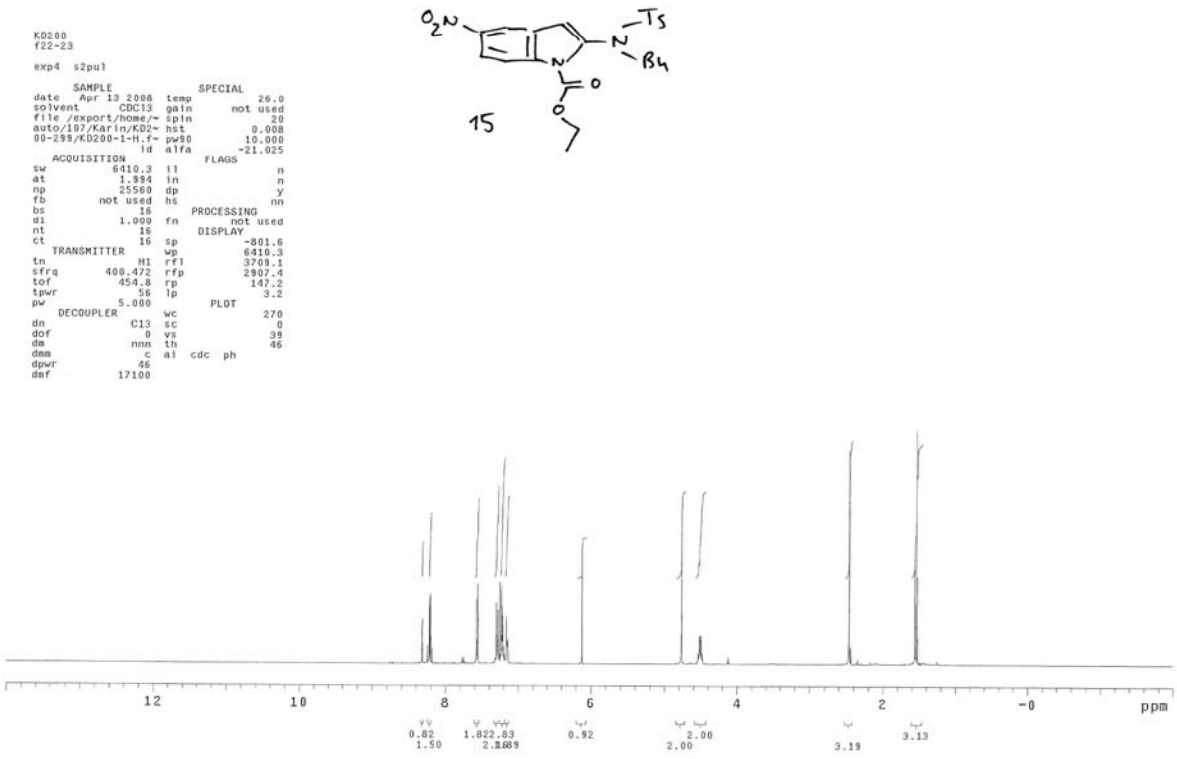
KD199
f34-35

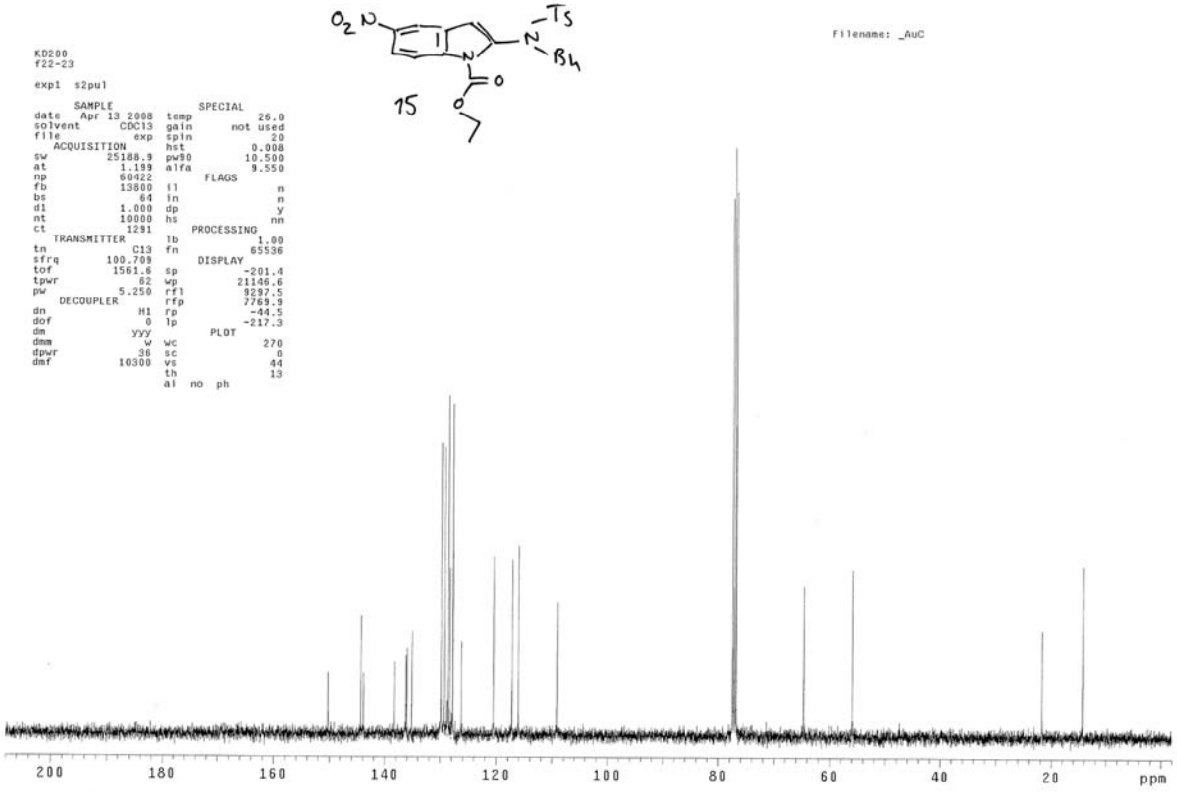
exp1 s2pul

SAMPLE		SPECIAL	
date	Apr 13 2008	temp	26.0
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	25188.9	pw90	10.500
at	1.199	alfa	9.550
np	60422	FLAGS	
fb	13800	il	n
bs	64	in	n
d1	1.000	dp	y
nt	10000	hs	nn
ct	1245	PROCESSING	
TRANSMITTER		lb	1.00
tn	C13	fn	65536
sfrq	100.708	DISPLAY	
tof	1561.6	sp	-201.4
tpwr	62	wp	21146.6
pw	5.250	rf1	9295.9
DECOUPLER		rfp	7769.9
dn	H1	rp	-28.7
dof	0	lp	-264.6
dm	yyy	PLOT	
dmm	w	wc	270
dpwr	36	sc	0
dmf	10300	vs	368
		th	11
		al no ph	

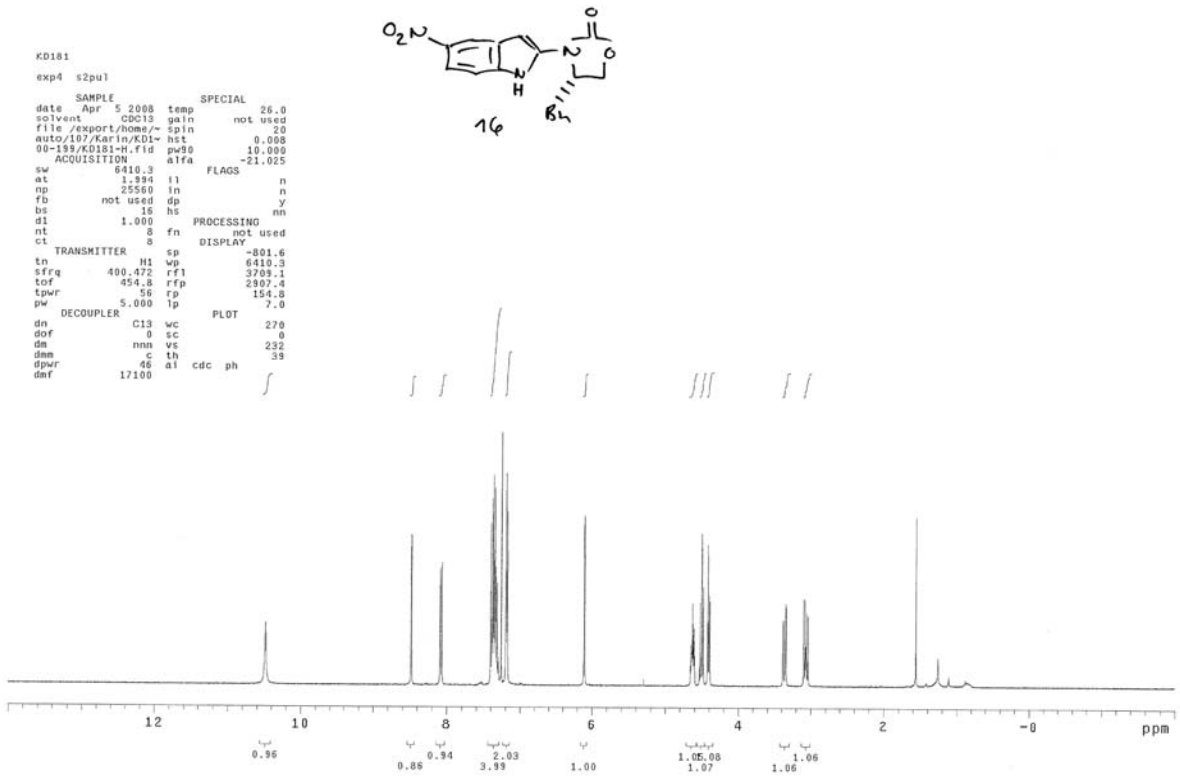


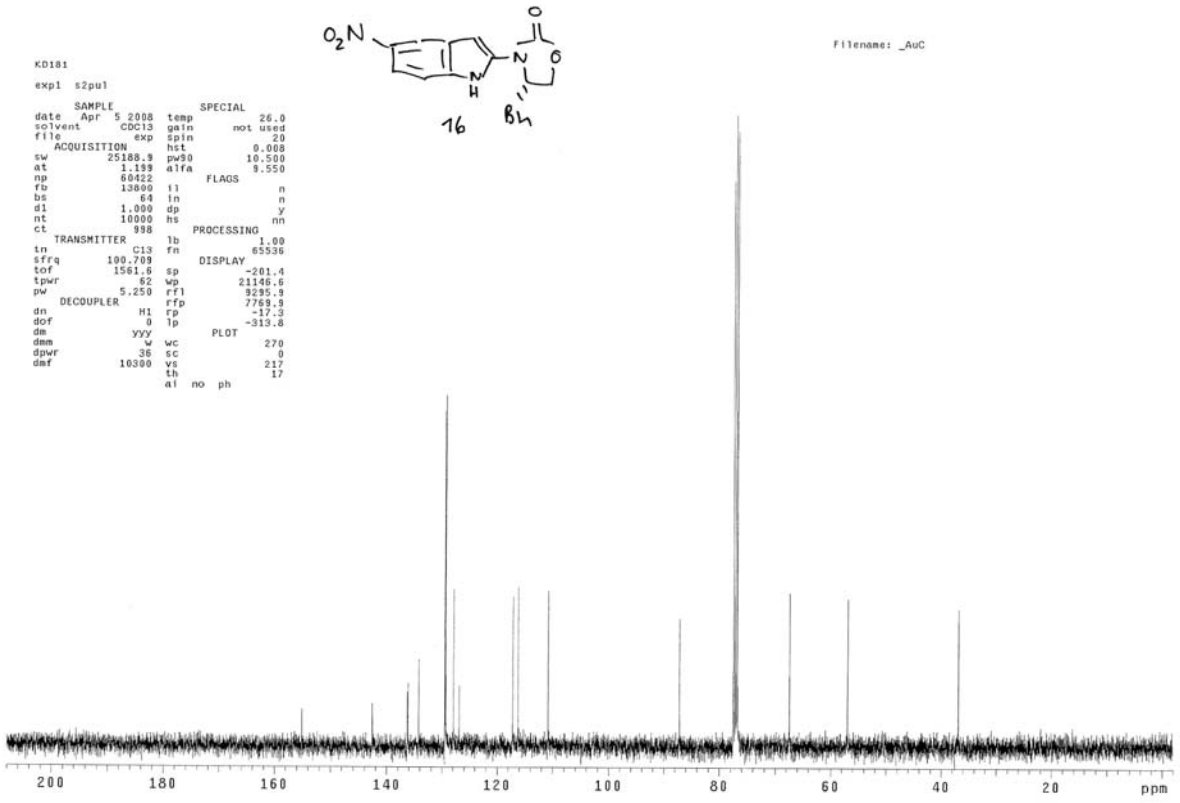
Compound 15


KD200
f22-23
exp4 s2pul
SAMPLE
date Apr 13 2008
solvent CDCl3
file /export/home/~
auto/107/Karin/KD2~
00-299/KD200-1-H.f~
id
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -801.6
wp 6410.3
rf1 3709.1
rfp 2907.4
rp 147.2
lp 3.2
PLOT
wc 270
sc 0
vs 39
th 46
al cdc ph
15
12
10
8
6
4
2
-0
ppm
0.82
1.50
1.82
2.83
2.89
0.92
2.00
2.00
3.19
3.13

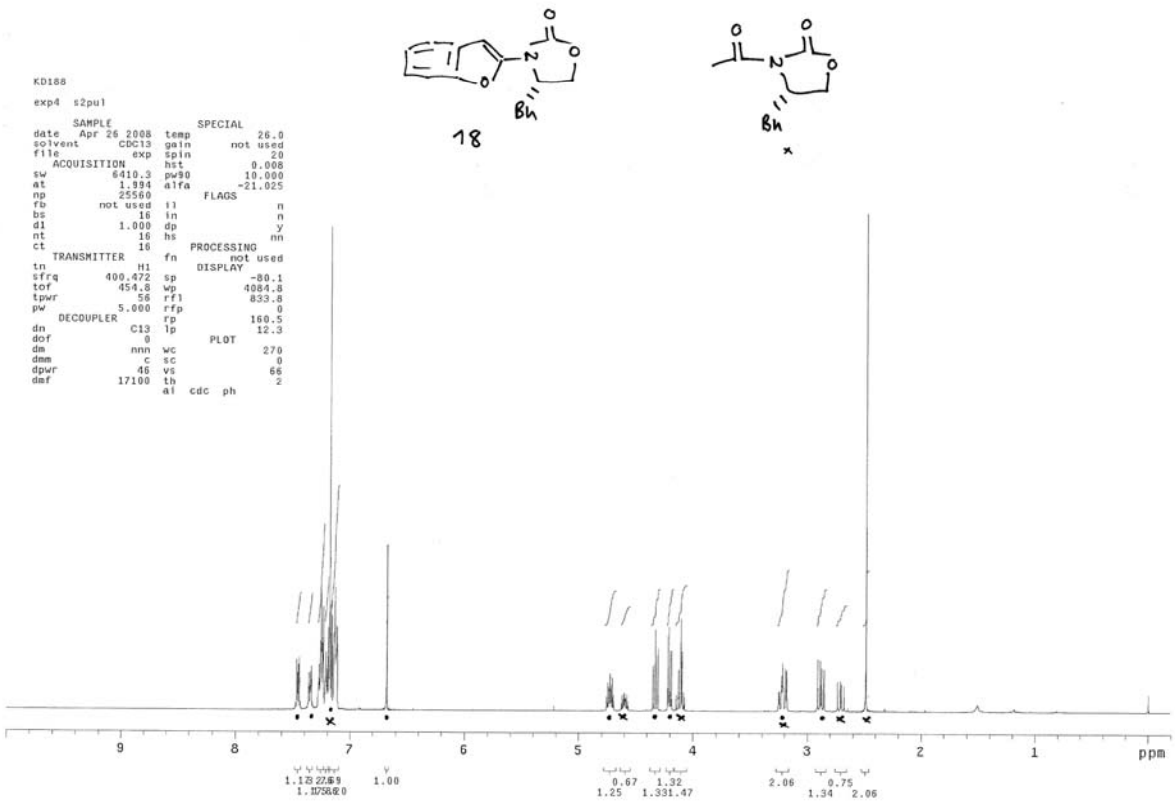

KD200
f22-23
exp1 s2pul
SAMPLE
date Apr 13 2008
solvent CDCl3
file exp
ACQUISITION
sw 25188.9
at 1.189
np 60422
fb 13800
bs 64
d1 1.000
nt 10000
ct 1291
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn 65536
DISPLAY
sp -201.4
wp 21146.6
rf1 9297.5
rfp 7769.9
rp -44.5
lp -217.3
PLOT
wc 270
sc 0
vs 44
th 13
al no ph
Filename: _AuC
15
200
180
160
140
120
100
80
60
40
20
ppm

Compound 16

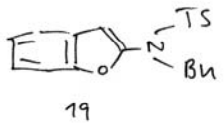

KD181
exp4 s2pul
SAMPLE
date Apr 5 2008
solvent CDCl3
file /export/home/~
auto/107/Karin/KD1~
00-199/KD181-H.fid
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -801.6
wp 6410.3
rfl 3709.1
rfp 2907.4
rp 154.8
lp 7.0
PLOT
wc 270
sc 0
vs 232
th 39
al cdc ph
16
12
10
8
6
4
2
-0
ppm
0.96
0.86
0.94
2.03
3.99
1.00
1.05.08
1.07
1.06
1.06


KD181
exp1 s2pul
SAMPLE
date Apr 5 2008
solvent CDCl3
file exp
ACQUISITION
sw 25188.9
at 1.199
np 60422
fb 13800
bs 64
d1 1.000
nt 10000
ct 998
TRANSMITTER
tn C13
sfrq 100.709
tof 1561.6
tpwr 62
pw 5.250
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 36
dmf 10300
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.500
alfa 9.550
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn 65536
DISPLAY
sp -201.4
wp 21146.6
rfl 9295.9
rfp 7769.9
rp -17.3
lp -313.8
PLOT
wc 270
sc 0
vs 217
th 17
al no ph
16
Filename: _AuC
200
180
160
140
120
100
80
60
40
20
ppm

Compound 18


KD188
exp4 s2pul
SAMPLE
date Apr 26 2008
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -80.1
wp 4084.8
rfl 833.8
rfp 0
rp 160.5
lp 12.3
PLOT
wc 270
sc 0
vs 66
th 2
al cdc ph
18
Bn
Bn
9
8
7
6
5
4
3
2
1
ppm
1.13 2.59 1.00
0.67 1.32
1.25 1.33 1.47
2.06
0.75
1.34 2.06

Compound 19

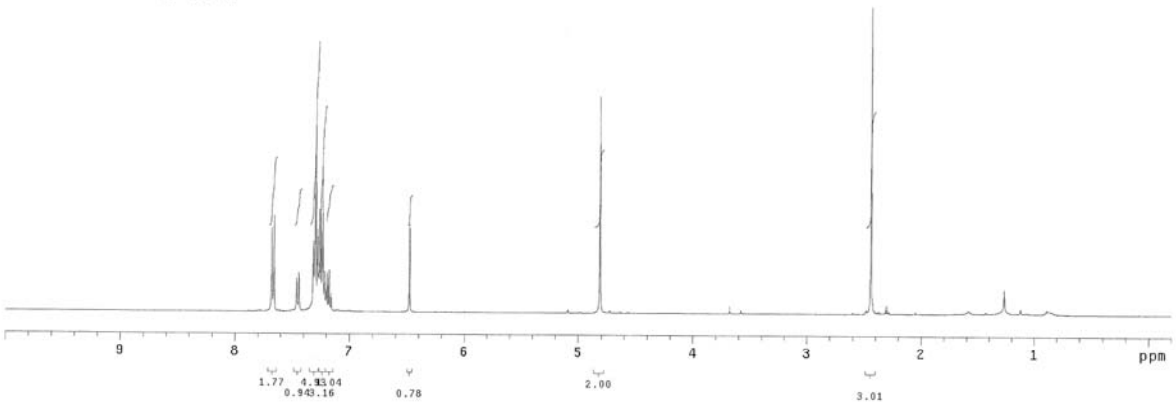


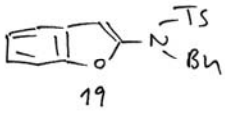
19

```
KD191

expl  s2pul

      SAMPLE                  SPECIAL
date   Apr  6 2008   temp          26.0
solvent       CDCl3  gain       not used
file           exp   spin            20
   ACQUISITION       hst          0.008
sw            6410.3  pw90        10.000
at             1.994  alfa       -21.025
np             25560      FLAGS
fb         not used   il               n
bs               16   in               n
d1            1.000   dp               y
nt                8   hs              nn
ct                8      PROCESSING
   TRANSMITTER        fn         not used
tn                H1       DISPLAY
sfrq         400.472   sp            -80.1
tof            454.8   wp           4004.8
tpwr              56   rfl          3709.1
pw             5.000   rfp          2907.4
      DECOUPLER        rp            152.4
dn                C13   lp             13.1
dof                 0        PLOT
dm                nnn   wc             270
dmm                 c   sc               0
dpwr               46   vs              69
dmf             17100   th              50
                        al  cdc  ph
```





19

Filename: _AuC

```
KD191

expl  s2pul

      SAMPLE                  SPECIAL
date   Apr  6 2008   temp          26.0
solvent       CDCl3  gain       not used
file           exp   spin            20
   ACQUISITION       hst          0.008
sw           25188.9  pw90        10.500
at             1.199  alfa         9.550
np             60422      FLAGS
fb             13800   il               n
bs                64   in               n
d1             1.000   dp               y
nt             10000   hs              nn
ct              2326      PROCESSING
   TRANSMITTER        lb              1.00
tn                C13   fn         not used
sfrq         100.703       DISPLAY
tof            1561.6   sp           -201.4
tpwr               62   wp          21146.6
pw             5.250   rfl          9295.9
      DECOUPLER        rfp          7769.9
dn                 H1   rp            -30.7
dof                 0   lp            -261.9
dm                yyy        PLOT
dmm                 w   wc             270
dpwr               36   sc               0
dmf             10300   vs            1251
                        th               7
                        al  no  ph
```