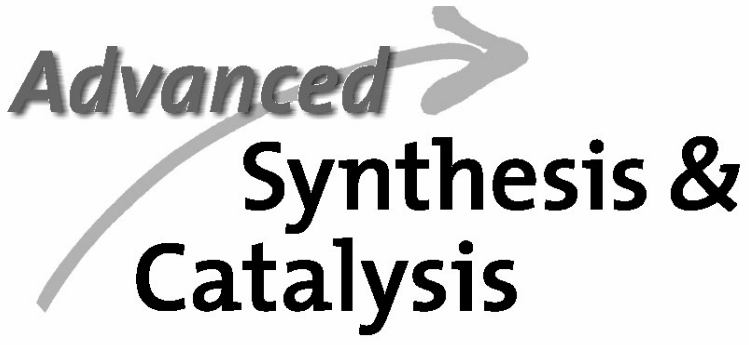

Supporting Information

Supporting Information

Proton-Promoted Hydroamination of 3-Dialkylthiomethylene-1,4-pentadiynes with *o*-Phenylenediamines: A Facile Route to Benzo[*b*][1,4]diazepines

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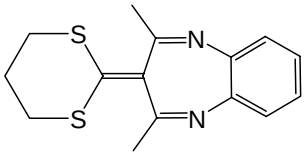
Experimental Section

General Information

All reagents were commercial and were used without further purification. Chromatography was carried on flash silica gel (300-400 mesh). All reactions were monitored by TLC, which was performed on precoated aluminum sheets of silica gel 60 (F_{254}). Melting points were uncorrected. The ^{1}H NMR and ^{13}C NMR spectra were determined on a VARIAN UNITY (500 or 400 MHz) spectrometer in $CDCl_3$ with TMS as internal standard. All shifts are given in ppm. IR (KBr) spectra were measured using a MAGNA-IR 560 spectrometer. Mass spectra were recorded on an Agilient 1100 LCMsD spectrometer. Elemental analyses were obtained on a PE-2400 analyser. The compounds **3a1**, **4** and **5** with dimensions 0.432 × 0.330 × 0.330 mm, 0.380 × 0.162 × 0.160 mm and 0.112 × 0.086 × 0.032 mm, respectively, were glued on a glass fiber. Data were collected on a Rigaku R-axis RAPID IP diffractometer at 293K using graphite-monochromated Mo Kα radiation (λ = 0.71073Å) and IP technique in the range $2.19^{\circ} < \theta < 27.48^{\circ}$. Empirical absorption correction was applied. The structures were solved by the direct method and refined by the full-matrix least-squares method on F^2 using the SHELXS 97 crystallographic software package. Anisotropic thermal parameters were used to refine all non-hydrogen atoms. Hydrogen atoms were located from difference Fourier maps.

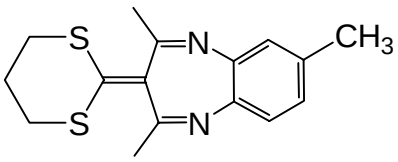
Typical Procedure for the Preparation of 3-5 (3a1 as Example): As a typical example, a solution of **2a** (1.0 mmol, 180 mg) and *o*-phenylenediamine (1.0 mmol, 108 mg) in C_2H_5OH (10 mL) was cooled to 0 ^oC in an ice bath, and $BF_3{\cdot}OEt_2$ (2.0 mmol, 0.25 mL) was added dropwise by syringe within 3 min. The reaction mixture was stirred for 0.5 h at room temperature. After **2a** was consumed (monitored by TLC), the reaction mixture was poured into water (30 mL). The solid crude product **3a1** was filtrated off, then purified by silica gel chromatography (diethyl/hexane = 1/1, v/v) to give 216 mg (75%) of **3a1**.

(1Z,4Z)-3-(1,3-dithian-2-ylidene)-2,4-dimethyl-3H-benzo[b][1,4]diazepine (Compound 3a1):



White crystal; m. p. 200-202 ^oC; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.03-2.24 (m, 2H), 2.76 (s, 6H), 2.78-2.82 (m, 2H), 2.94-2.99 (m, 2H), 7.20 (dt, J = 6.0, 3.5 Hz, 2H), 7.38 (dt, J = 6.0, 3.5 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 23.4, 25.4 (2C), 28.5 (2C), 124.8 (2C), 127.6 (2C), 129.9 (2C), 135.2 (2C), 140.1, 160.1; **IR** (KBr, cm^{-1}): 762, 790, 1213, 1272, 1365, 1426, 1460, 1559, 1620, 2916, 2956, 3442; **MS (EI)** m/z 289 $[(M+1)]^+$; **Anal**. Calcd (found) for: $C_{15}H_{16}N_2S_2$: C, 62.46 (62.59); H, 5.59 (5.64); N, 9.71 (9.76).

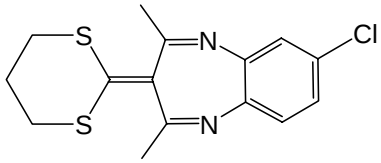
(1Z,4Z)-3-(1,3-dithian-2-ylidene)-2,4,7-trimethyl-3H-benzo[b][1,4]diazepine (Compound 3a2):



White crystal; m. p. 214-216 ^oC; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.02-2.14 (m, 2H), 2.36 (s, 3H), 2.45 (s, 6H), 2.77-2.80 (m, 2H), 2.93-2.96 (m, 2H), 7.00 (d, J = 8.0 Hz, 1H), 7.18 (s, 1H), 7.27 (d, J = 8.0 Hz, 1H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 20.8, 23.5, 25.4 (2C), 28.6 (2C), 126.1, 127.5, 127.8, 129.6, 134.7, 135.5, 137.9, 139.9, 159.1, 159.7; **IR** (KBr, cm^{-1}): 822, 1211, 1274, 1362,

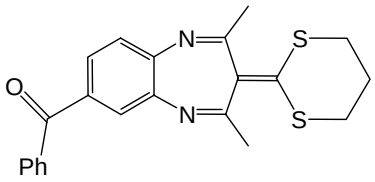
1423, 1473, 1562, 1621, 2855, 2919, 2956, 3053; **MS (EI)** m/z 303 $[(M+1)]^+$; **Anal**. Calcd (found) for: $C_{16}H_{18}N_2S_2$: C, 63.54 (63.59); H, 6.00 (6.09); N, 9.26 (9.31).

(1Z,4Z)-7-chloro-3-(1,3-dithian-2-ylidene)-2,4-dimethyl-3H-benzo[b][1,4]diazepine (Compound 3a3):



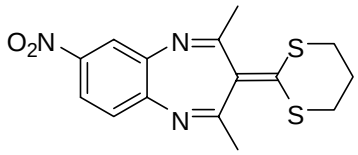
White crystal; m. p. 178-180 ^oC; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.04-2.16 (m. 2H), 2.46 (s, 6H), 2.79-2.84 (m, 2H), 2.94-2.80 (m, 2H), 7.13 (d, J = 9.0 Hz, 1H), 7.30 (d, J = 9.0 Hz, 1H), 7.37 (s, 1H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 23.6, 25.7 (2C), 28.7 (2C), 109.7, 125.3, 127.3, 129.1, 130.0, 134.8, 139.1, 141.3, 161.0, 161.7; **IR** (KBr, cm^{-1}): 818, 1246, 1364, 1423, 1458, 1561, 1622, 2854, 2923, 2957, 3446; **MS (EI)** m/z 323 $[(M+1)]^+$; **Anal**. Calcd (found) for: $C_{15}H_{15}ClN_2S_2$: C, 55.80 (55.88); H, 4.68 (4.79); N, 8.68 (8.63).

((1Z,4Z)-3-(1,3-dithian-2-ylidene)-2,4-dimethyl-3H-benzo[b][1,4]diazepin-7-yl)(phenyl)methanone (**Compound 3a4**)**:**



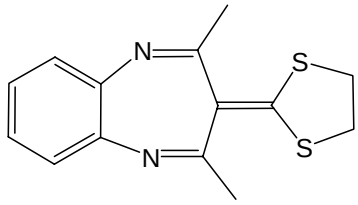
Yellow solid; m. p. 188-190 ^oC; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.03-2.15 (m, 2H), 2.47 (s, 3H), 2.50 (s, 3H), 2.77-2.82 (m, 2H), 2.93-3.00 (m, 2H), 7.46-7.49 (m, 3H), 7.57 (t, J = 7.5 Hz, 1H), 7.70 (q, J = 7.5 Hz, 1H), 7.81-7.83 (m, 3H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 23.8, 26.0, 26.1, 28.9 (2C), 126.5, 128.3, 128.5 (2C), 130.1 (2C), 131.0, 131.9, 132.4, 134.3, 135.1, 137.9, 140.1, 144.0, 162.2, 163.0, 196.1; **IR** (KBr, cm^{-1}): 698, 718, 1250, 1276, 1446, 1622, 1651, 2913, 3058, 3438; **MS (EI)** m/z 393 $[(M+1)]^+$.

(1Z,4Z)-3-(1,3-dithian-2-ylidene)-2,4-dimethyl-7-nitro-3H-benzo[b][1,4]diazepine (Compound 3a5):



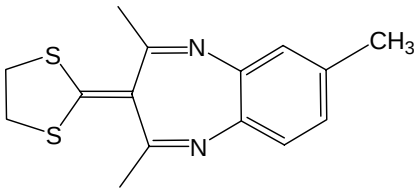
Yellow crystal; m. p. 144-146 ^{o}C; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.03-2.24 (m, 2H), 2.51 (s, 6H), 2.81-2.84 (m, 2H), 2.98-3.01 (m, 2H), 7.47 (d, *J* = 9.0 Hz, 1H), 8.00 (d, *J* = 8.5 Hz, 1H), 8.28 (s, 1H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 23.7, 26.1, 26.2, 28.9 (2C), 119.8, 124.1, 129.0, 133.4, 134.5, 140.8, 144.5, 145.7, 164.0, 164.7; **IR** (KBr, cm^{-1}): 876, 1080, 1201, 1248, 1339, 1417, 1512, 1539, 1628, 2921; **MS (EI)** m/z 334 $[(M+1)]^{+}$; **Anal**. Calcd (found) for: $C_{15}H_{15}N_3O_2S_2$: C, 54.03 (54.22); H, 4.53 (4.56); N, 12.60 (12.69).

(1Z,4Z)-3-(1,3-dithiolan-2-ylidene)-2,4-dimethyl-3H-benzo[b][1,4]diazepine (Compound 3b1):



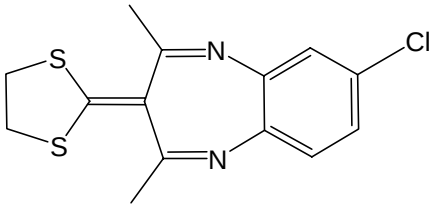
White crystal; m. p. 232-234 ^{o}C; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.47 (s, 6H), 3.32-3.36 (m, 2H), 3.38-3.43 (m, 2H), 7.19 (dt, *J* = 7.0, 3.0 Hz, 2H), 7.37 (dt, *J* = 7.0, 3.5 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 24.7 (2C); 38.2 (2C), 124.6 (2C), 125.2 (2C), 128.1 (2C), 138.7 (2C), 140.6, 161.3; **IR** (KBr, cm^{-1}): 761, 790, 845, 1211, 1277, 1366, 1458, 1622, 3441; **MS (EI)** m/z 275 $[(M+1)]^{+}$; **Anal**. Calcd (found) for: $C_{14}H_{14}N_2S_2$: C, 61.28 (61.43); H, 5.14 (5.19); N, 10.21(10.10)**.**

(1Z,4Z)-3-(1,3-dithiolan-2-ylidene)-2,4,7-trimethyl-3H-benzo[b][1,4]diazepine (Compound 3b2):



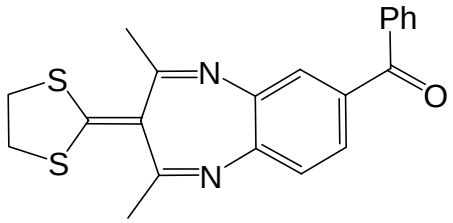
White crystal; m. p. 214-216 ^{o}C; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.34 (s, 3H), 2.43 (s, 6H), 3.28-3.29 (m, 2H), 3.35-3.36 (m, 2H), 6.99 (d, *J* = 8.0 Hz, 1H), 7.16 (s, 1H), 7.25 (d, *J* = 8.0 Hz, 1H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 20.8, 24.4 (2C), 38.0 (2C), 124.5, 126.2, 127.8, 128.0, 134.7, 138.2 (2C), 140.2, 160.1, 160.7; **IR** (KBr, cm^{-1}): 682, 821, 1210, 1279, 1366, 1427, 1475, 1567, 1619, 2919, 2985, 3446; **MS (EI)** m/z 289 $[(M+1)]^{+}$; **Anal**. Calcd (found) for: $C_{15}H_{16}N_2S_2$: C, 62.46 (62.62); H, 5.59 (5.69); N, 9.71 (9.75).

(1Z,4Z)-7-chloro-3-(1,3-dithiolan-2-ylidene)-2,4-dimethyl-3H-benzo[b][1,4]diazepine (Compound 3b3):



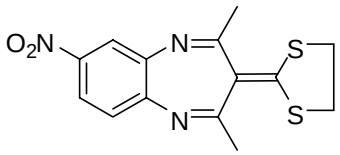
White crystal; m. p. 170-172 ^{o}C; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.45 (s, 6H), 3.30-3.38 (m, 2H), 3.40-3.45 (m, 2H), 7.13 (d, *J* = 8.5 Hz, 1H), 7.28 (d, *J* = 8.5 Hz, 1H), 7.36 (s, 1H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 24.5 (2C), 38.1 (2C), 124.2, 125.2, 127.4, 129.2, 129.8, 139.1, 139.2, 141.4, 161.7, 162.4; **IR** (KBr, cm^{-1}): 707, 819, 1217, 1280, 1366, 1460, 1561, 1622, 2854, 2925, 3444; **MS (EI)** m/z 309 $[(M+1)]^{+}$.

((1Z,4Z)-3-(1,3-dithiolan-2-ylidene)-2,4-dimethyl-3H-benzo[b][1,4]diazepin-7-yl)(phenyl)methanone (Compound 3b4):



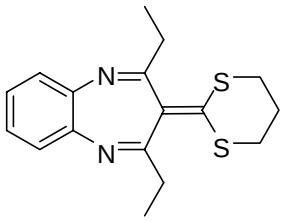
Yellow solid; m. p. 118-120 ^{o}C; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.47 (s, 3H), 2.50 (s, 3H), 3.33-3.38 (m, 2H), 3.40-3.45 (m, 2H), 7.45-7.49 (m, 3H), 7.57 (t, *J* = 8.5 Hz, 1H), 7.70 (q, *J* = 8.5 Hz, 1H), 7.79-7.83 (m, 3H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 24.9, 25.0, 38.5 (2C), 124.8, 126.6, 128.5 (2C), 130.1 (2C), 131.2, 132.4 (2C), 134.4, 137.9, 140.1, 140.4, 144.3, 163.1, 163.9, 196.1; **IR** (KBr, cm^{-1}): 719, 881, 1117, 1281, 1319, 1447, 1597, 1642, 2926, 3349; **MS (EI)** m/z 379 $[(M+1)]^{+}$.

(1Z,4Z)-3-(1,3-dithiolan-2-ylidene)-2,4-dimethyl-7-nitro-3H-benzo[b][1,4]diazepine (Compound 3b5):



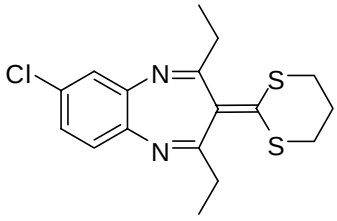
Yellow crystal; m. p. 164-166 ^{o}C; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.49 (s, 6H), 3.34-3.39 (m, 2H), 3.41-3.46 (m, 2H), 7.43 (d, *J* = 8.5 Hz, 1H), 7.98 (dd, *J* = 8.5, 2.5 Hz, 1H), 8.24 (d, *J* = 2.5 Hz, 1H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 24.8, 24.9; 38.3, 38.4, 119.6, 124.0, 124.4, 128.9, 140.8, 140.9, 144.4, 145.8, 164.4, 165.2; **IR** (KBr, cm^{-1}): 836, 1091, 1223, 1267, 1359, 1455, 1523, 1568, 1652, 2928; **MS (EI)** m/z 320 $[(M+1)]^{+}$; **Anal**. Calcd (found) for: $C_{14}H_{13}N_3O_2S_2$: C, 52.65 (52.85); H, 4.10 (4.16); N, 13.16 (13.28)**.**

(1Z,4Z)-3-(1,3-dithian-2-ylidene)-2,4-diethyl-3H-benzo[b][1,4]diazepine (Compound 3c1):



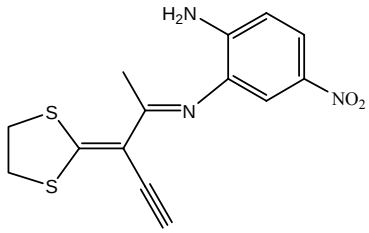
Yellow solid; m. p. 88-90 ^{o}C; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 1.23-1.26 (m, 6H), 2.02-2.11 (m, 2H), 2.62-2.77 (m, 4H), 2.90-2.98 (m, 4H), 7.17 (q, *J* = 7.5 Hz, 2H), 7.39 (q, *J* = 7.0 Hz, 2H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ: 11.0 (2C), 23.8, 28.9 (2C), 31.6 (2C), 124.9 (2C), 128.1 (2C), 129.6, 136.0, 140.5 (2C), 165.4 (2C); **IR** (KBr, cm^{-1}): 2932, 1622, 1558, 1541, 1458, 1416, 1346, 1242, 1167, 1060, 846, 760; **MS (EI)** m/z 317 $[(M+1)]^{+}$; **Anal**. Calcd (found) for: $C_{17}H_{20}N_2S_2$: C, 64.52 (64.68); H, 6.37 (6.35); N, 8.85 (8.94).

(1Z,4Z)-7-chloro-3-(1,3-dithian-2-ylidene)-2,4-diethyl-3H-benzo[b][1,4]diazepine (Compound 3c2):



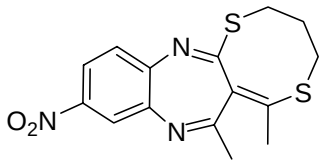
Yellow liquid; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 1.23-1.33 (m, 6H), 2.04-2.15 (m, 2H), 2.58-2.93 (m, 4H), 2.94-3.02 (m, 4H), 7.13 (d, *J* = 8.5 Hz, 1H), 7.32 (d, *J* = 8.5 Hz, 1H), 7.41 (s, 1H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ: 10.8, 10.9, 23.7, 28.7 (2C), 31.6, 31.7, 125.1, 127.6, 129.4, 129.8, 130.6, 135.4, 139.1, 141.4, 166.0, 166.6; **IR** (KBr, cm^{-1}): 2930, 1622, 1557, 1506, 1458, 1444, 1348, 1241, 1154, 1084, 880; **MS (EI)** m/z 351 $[(M+1)]^{+}$; **Anal**. Calcd (found) for: $C_{17}H_{19}ClN_2S_2$: C, 58.18 (58.41); H, 5.46 (5.55); N, 7.98 (7.86).

(E)-N1-(3-(1,3-dithiolan-2-ylidene)pent-4-yn-2-ylidene)-4-nitrobenzene-1,2-diamine (Compound 4):



Red crystal; m. p. 183-185 ^{o}C; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.26 (s, 3H), 3.40-3.44 (m, 4H), 3.72 (s, 1H), 4.65 (s, 2H), 6.68 (d, *J* = 9.0 Hz, 1H), 7.52 (s, 1H), 7.89 (q, *J* = 9.0 Hz, 1H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 19.3, 35.7, 40.7, 82.3, 86.5, 107.9, 112.9, 115.9, 122.1, 134.9, 138.9, 145.9, 165.1, 166.0; **IR** (KBr, cm^{-1}): 740, 830, 1212, 1264, 1309, 1460, 1484, 1500, 1592,1613, 3261, 3351, 3462; **MS (EI)** m/z 320 $[(M+1)]^{+}$; **Anal**. Calcd (found) for: $C_{14}H_{13}N_3O_2S_2$: C, 52.65 (52.70); H, 4.10 (4.05); N, 13.16 (13.24).

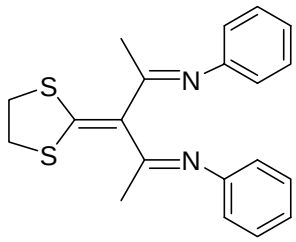
(1E,2Z,4Z,5aZ)-9-nitro-5,6-dimethyl-3H-[1,5]dithiocino[2,3-e] benzo[b][1,4]diazepine (Compound 5):



White crystal; m. p. 220-222 ^{o}C; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 1.88 (s, 3H), 2.36 (s, 3H), 2.38-2.41 (m, 2H), 2.58 (d, *J* = 16.0 Hz, 1H), 3.06 (d, *J* = 16.0 Hz, 1H), 3.75-3.81 (m, 1H), 3.93-3.99 (m, 1H), 7.56 (d, *J* = 9.0 Hz, 1H), 8.03 (d, *J* = 9.0 Hz, 1H), 8.28 (s, 1H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 20.9, 26.0, 27.4, 30.2, 36.2, 119.6, 123.3, 125.8, 128.5, 134.7, 139.9, 143.9, 145.0, 161.9, 163.2; **IR** (KBr, cm^{-1}): 884, 1085, 1168, 1211, 1248, 1343, 1450, 1511, 1623, 2962, 3433; **MS (EI)** m/z 334 $[(M+1)]^{+}$; **Anal**. Calcd (found) for: $C_{15}H_{15}N_3O_2S_2$: C, 54.03 (54.13); H, 4.53 (4.55); N, 12.60 (12.66).

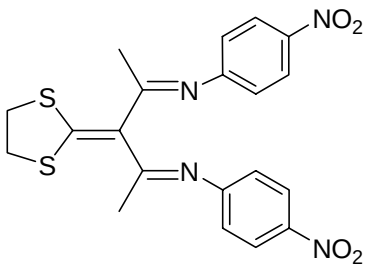
General Experimental Procedure For Compounds 6: A solution of **2b** (1.0 mmol, 166 mg) and 4-nitrobenzenamine (2.0 mmol, 276 mg) in C_2H_5OH (10 mL) was cooled to 0 ^oC in an ice bath, and $BF_3{\cdot}OEt_2$ (2.0 mmol, 0.25 mL) was added dropwise by syringe within 3 min. The reaction mixture was stirred for 2 h at room temperature. After **2b** was consumed (monitored by TLC), the reaction mixture was poured into water (50 mL) and extracted with CH_2Cl_2 (3×10 mL). The combined organic extracts were dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure to yield the corresponding crude product, which was purified by silica gel chromatography (diethyl/hexane = 1/1, v/v) to give 274 mg (62%) of **6b2** as a yellow crystal.

(E)-N-((E)-3-(1,3-dithiolan-2-ylidene)-4-(phenylimino)pentan-2-ylidene)benzenamine (Compound 6b1):



Yellow crystal; m. p. 112-114 ^oC; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.04 (s, 6H), 3.31 (s, 4H), 6.86 (d, *J* = 7.5 Hz, 4H), 7.08 (q, *J* = 7.5 Hz, 2H), 7.36 (t, *J* = 7.5 Hz, 4H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 20.1 (2C), 37.5 (2C), 119.8, 119.9 (2C), 120.3, 123.8 (2C), 124.5, 128.2, 128.8, 129.0, 129.3 (2C), 150.2 (2C), 153.8, 166.5; **IR** (KBr, cm^{-1}): 3378, 3060, 1618, 1592, 1483, 1364, 1212, 1159, 802, 701; **MS (EI)** m/z 353 $[(M+1)]^+$.

(E)-N-((E)-3-(1,3-dithiolan-2-ylidene)-4-(4-nitrophenylimino)pentan-2-ylidene)-4-nitrobenzenamine (Compound 6b2):

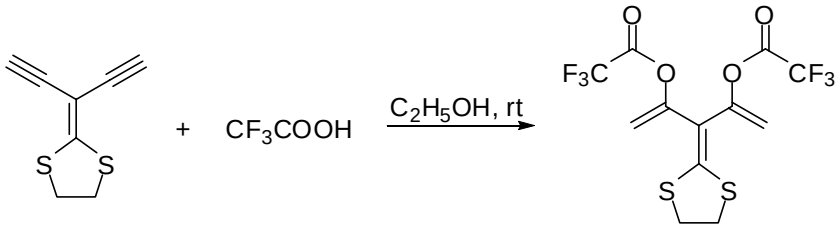


Yellow crystal; m. p. 185-186 ^{o}C; **^{1}H NMR** ($CDCl_3$, 500 MHz) δ: 2.08 (s, 6H), 3.38 (s, 4H), 6.94 (d, J = 8.5 Hz, 4H), 8.25 (d, J = 8.5 Hz, 4H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 20.7 (2C), 37.8 (2C), 119.8 (4C), 124.8 (2C), 125.4 (4C), 126.8 (2C), 144.2, 156.1 (2C), 166.5; **IR** (KBr, cm^{-1}): 1614, 1586, 1509, 1339, 1218, 1109, 864, 708; **MS (EI)** m/z 443 $[(M+1)]^+$.

General Experimental Procedure For Compounds 7:

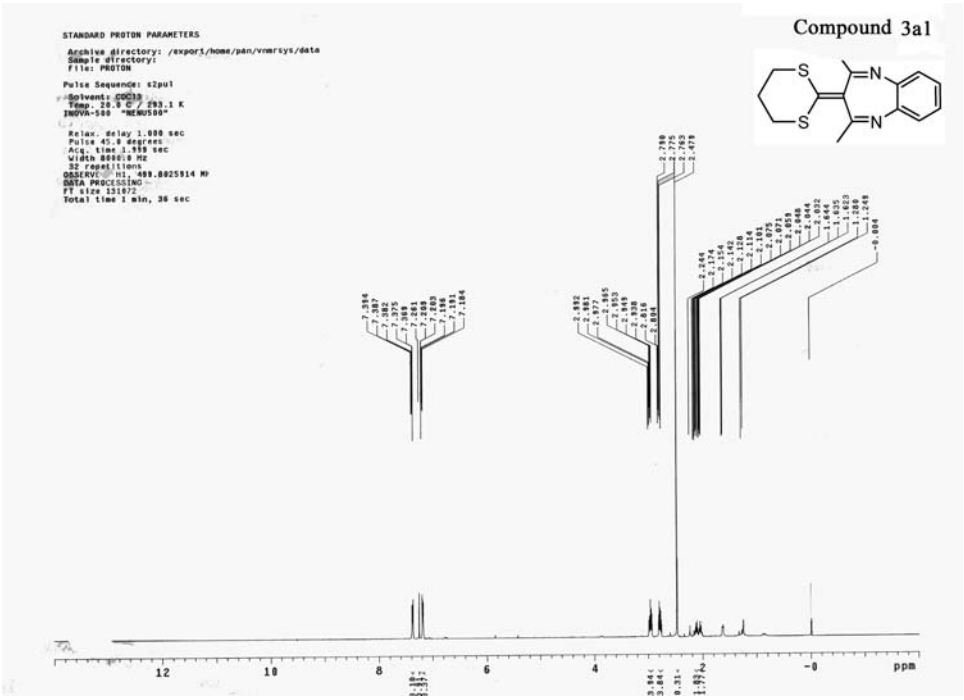
To a stirred solution of **2b** (1.0 mmol, 166 mg) in C_2H_5OH (10 mL) was added CF_3COOH (2.2 equiv) in one portion. The reaction mixture was stirred for 2-3 h at room temperature. After **2b** was consumed (monitored by TLC), the reaction mixture was poured into water (50 mL) and extracted with CH_2Cl_2 (3×10 mL). The combined organic extracts were dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure to yield the corresponding crude product, which was purified by silica gel chromatography (diethyl/hexane = 1/2, v/v) to give 197 mg (50%) of **7** as a white crystal.

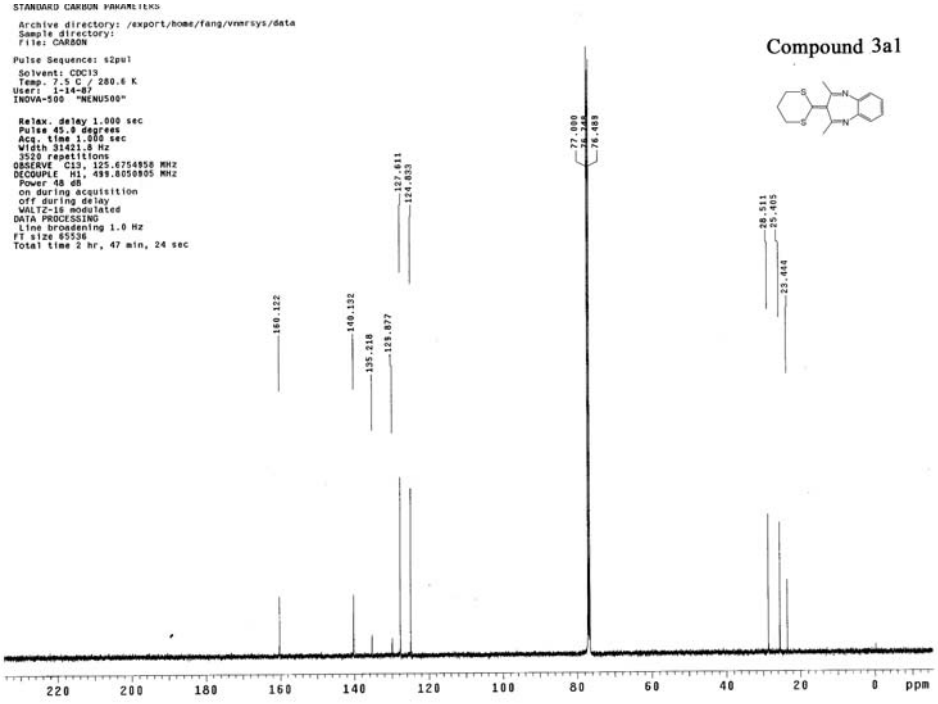
3-(1,3-dithiolan-2-ylidene)penta-1,4-diene-2,4-diyl bis(2,2,2-trifluoroacetate) (Compound 7):

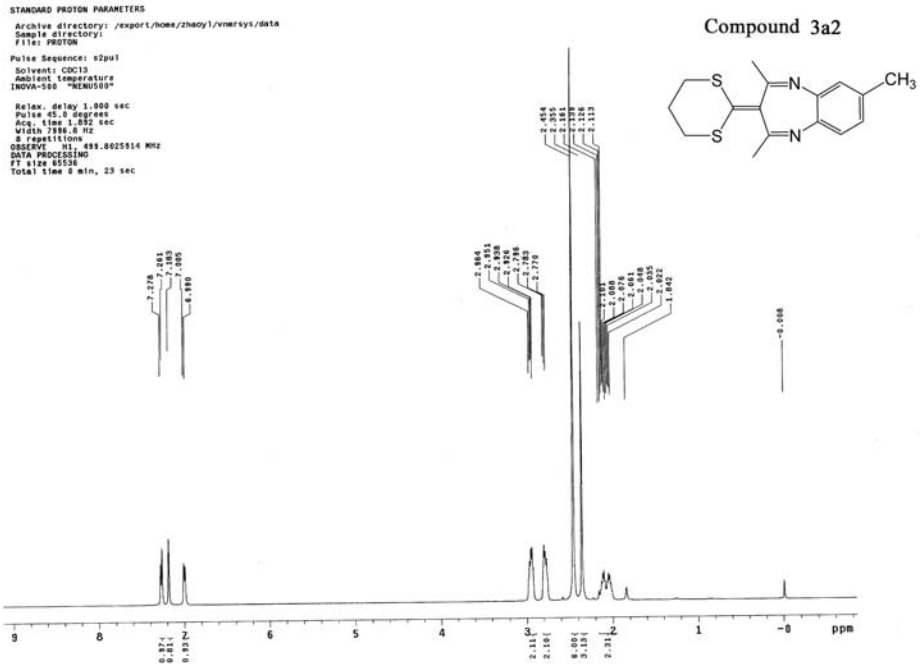


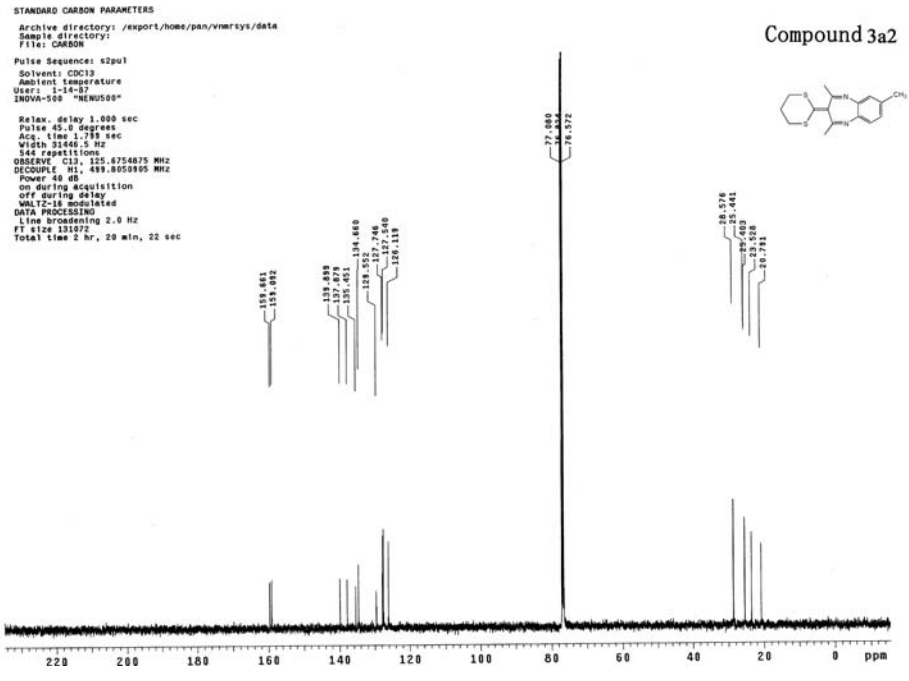
2b **7**

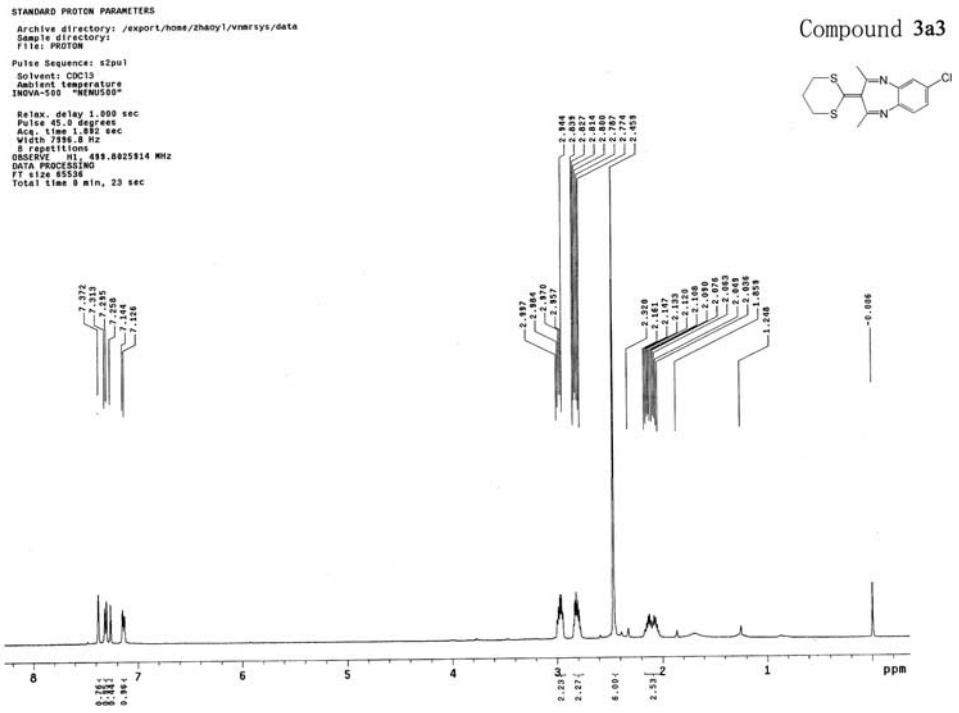
^{1}H NMR ($CDCl_3$, 500 MHz) δ: 3.48 (s, 4H), 5.29 (s, 2H), 5.38 (2H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ: 38.0 (2C), 108.9, 110.9, 111.8, 113.2, 115.4, 117.7, 149.1, 154.5, 154.9 (q, J = 170 Hz, 2C); **IR** (KBr, cm^{-1}): 3123, 2928, 1687, 1646, 1517, 1490, 1435, 1260; **MS (EI)** m/z 395 $[(M+1)]^+$. **Anal**. Calcd (found) for: $C_{12}H_8F_6O_4S_2$: C, 36.55 (36.78); H, 2.04 (2.18).

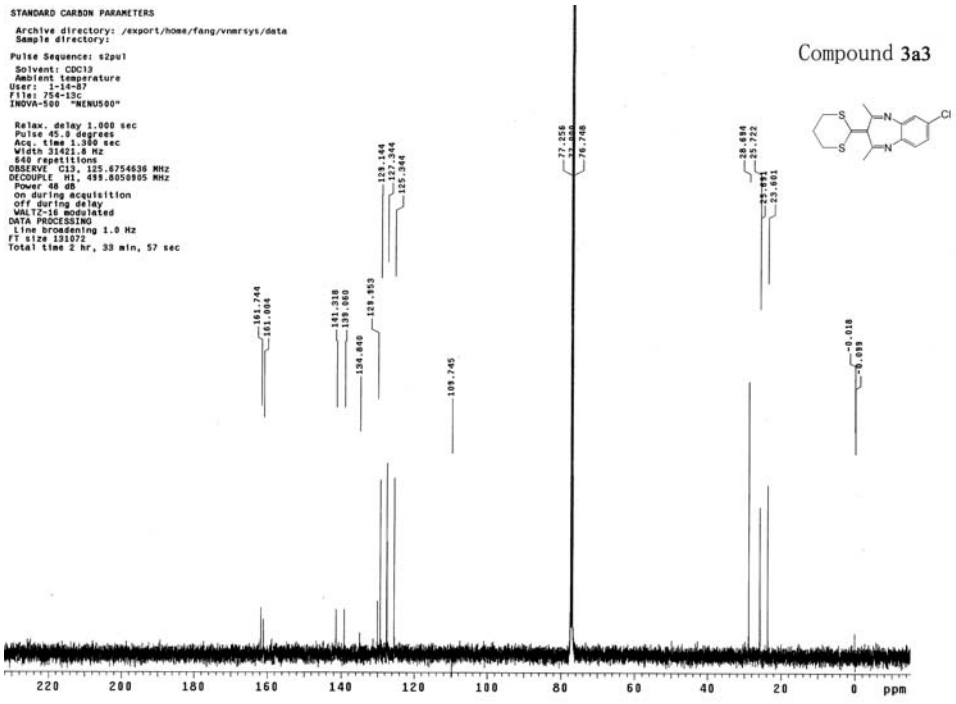

Compound 3a1
STANDARD PROTON PARAMETERS
Archive directory: /export/home/pan/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.999 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.8825914 MHz
DATA PROCESSING
FT size 131072
Total time 1 min, 36 sec
ppm

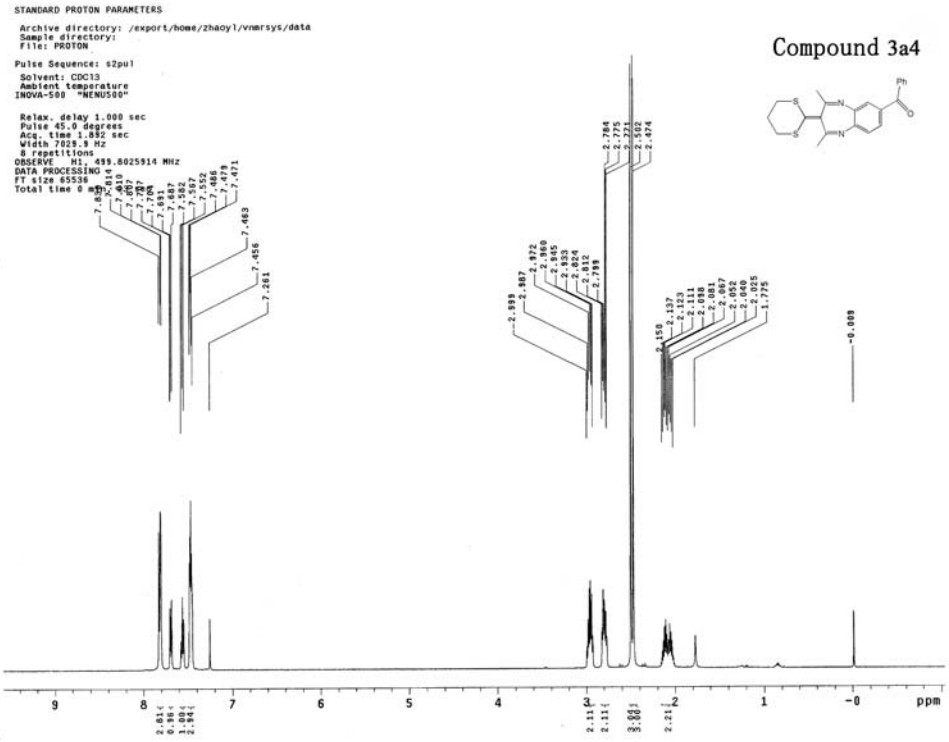

Compound 3a1
STANDARD CARBON PARAMETERS
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 31421.8 Hz
3520 repetitions
OBSERVE C13, 125.6754858 MHz
DECOUPLE H1, 499.8050805 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 2 hr, 47 min, 24 sec
160.122
140.132
135.218
129.877
127.611
124.680
77.000
76.368
76.469
28.511
25.405
23.444
ppm

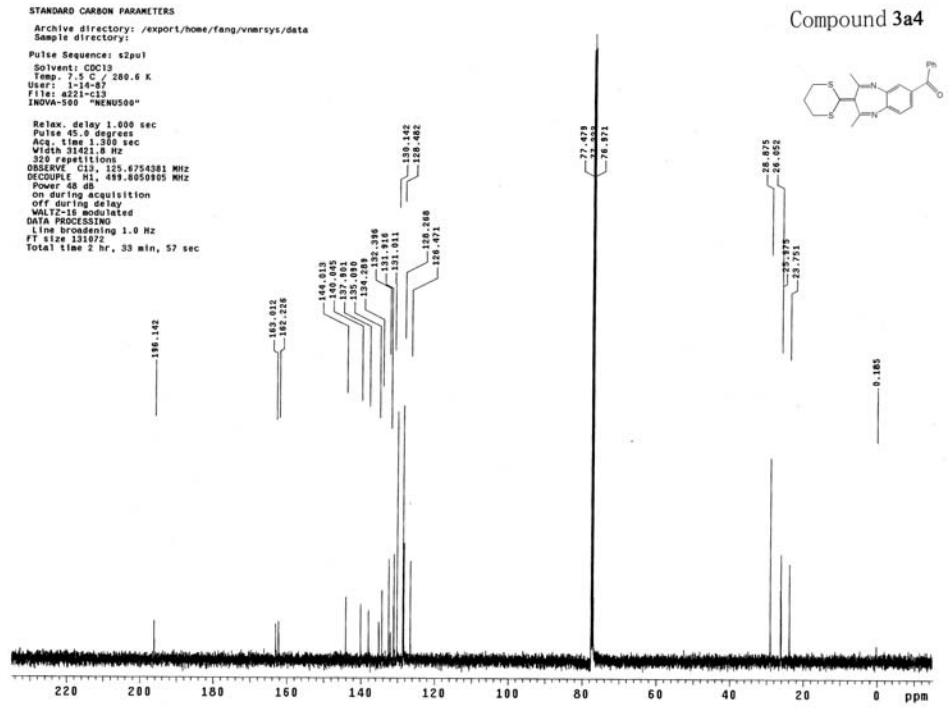

STANDARD PROTON PARAMETERS
Archive directory: /export/home/zhaoyl/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7886.8 Hz
8 repetitions
OBSERVE H1, 499.8025514 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Compound 3a2
S
S
N
N
CH3
ppm

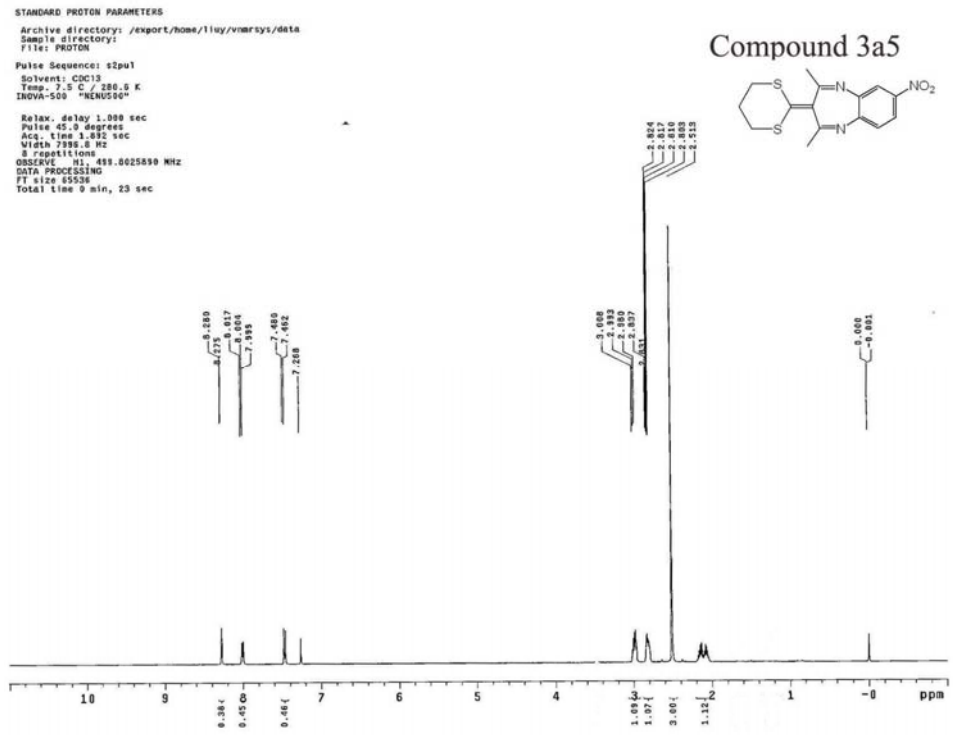

STANDARD CARBON PARAMETERS
Archive directory: /export/home/pan/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.755 sec
Width 31446.5 Hz
544 repetitions
OBSERVE C13, 125.6754875 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 2 hr, 20 min, 22 sec
Compound 3a2
ppm

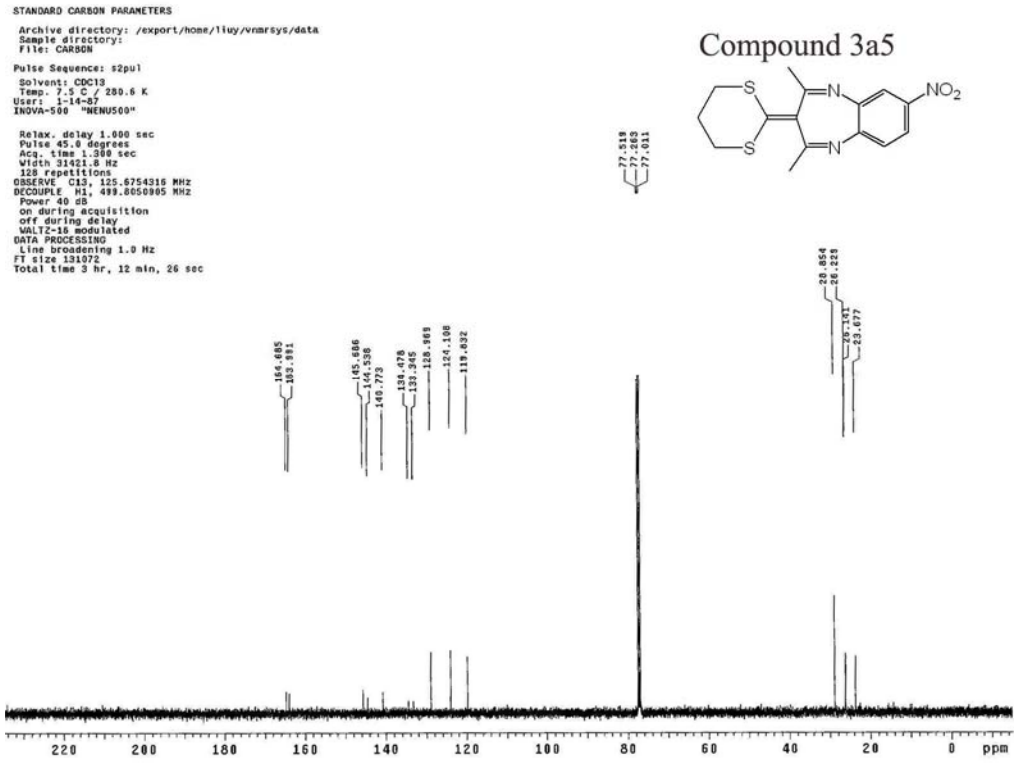

STANDARD PROTON PARAMETERS
Archive directory: /export/home/zhaoyl/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8825914 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Compound 3a3
Cl
ppm

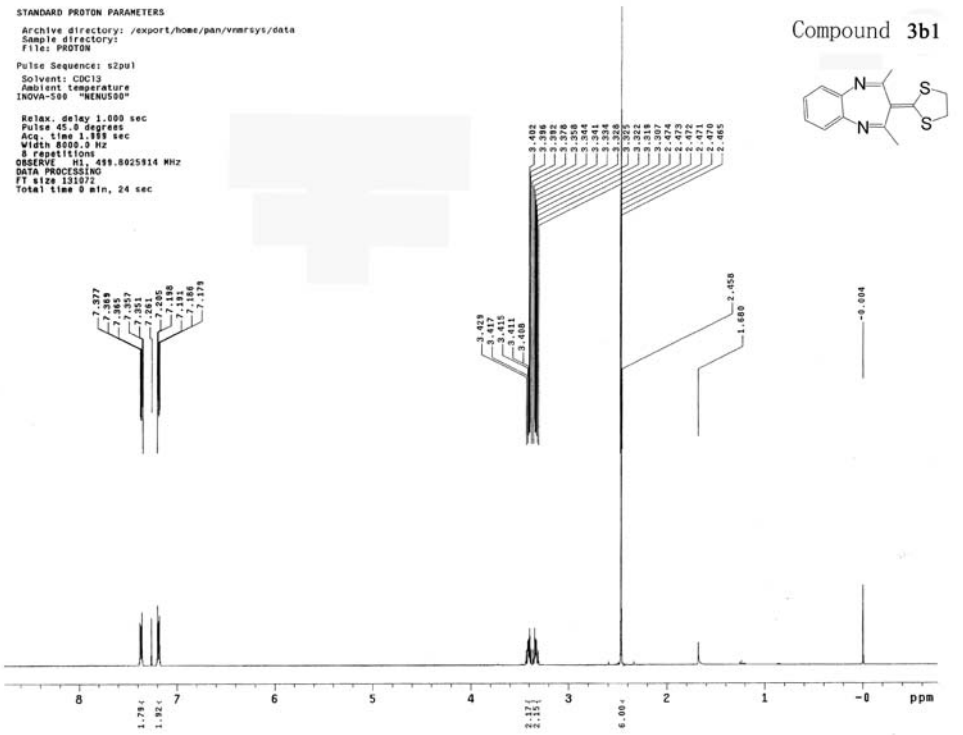

STANDARD CARBON PARAMETERS
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 754-13c
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
640 repetitions
OBSERVE C13, 125.8754636 MHz
DECOUPLE H1, 499.8059905 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 33 min, 57 sec
Compound 3a3
Cl
ppm

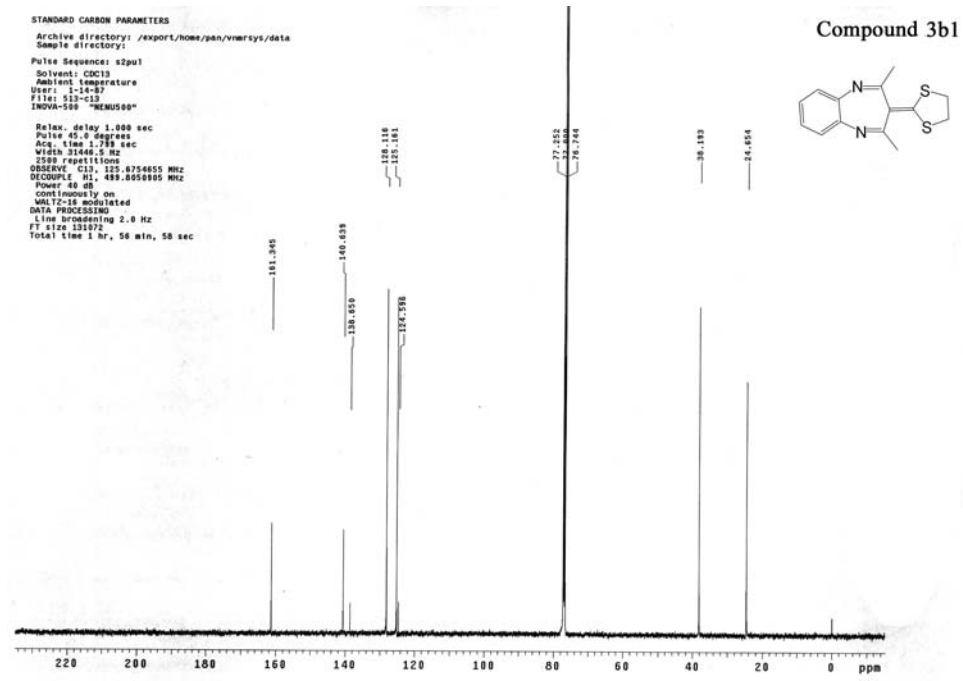

STANDARD PROTON PARAMETERS
Archive directory: /export/home/zhaoyl/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7029.8 Hz
8 repetitions
OBSERVE H1, 499.8025914 MHz
DATA PROCESSING
FT size 65536
Total time 0 min
Compound 3a4
ppm

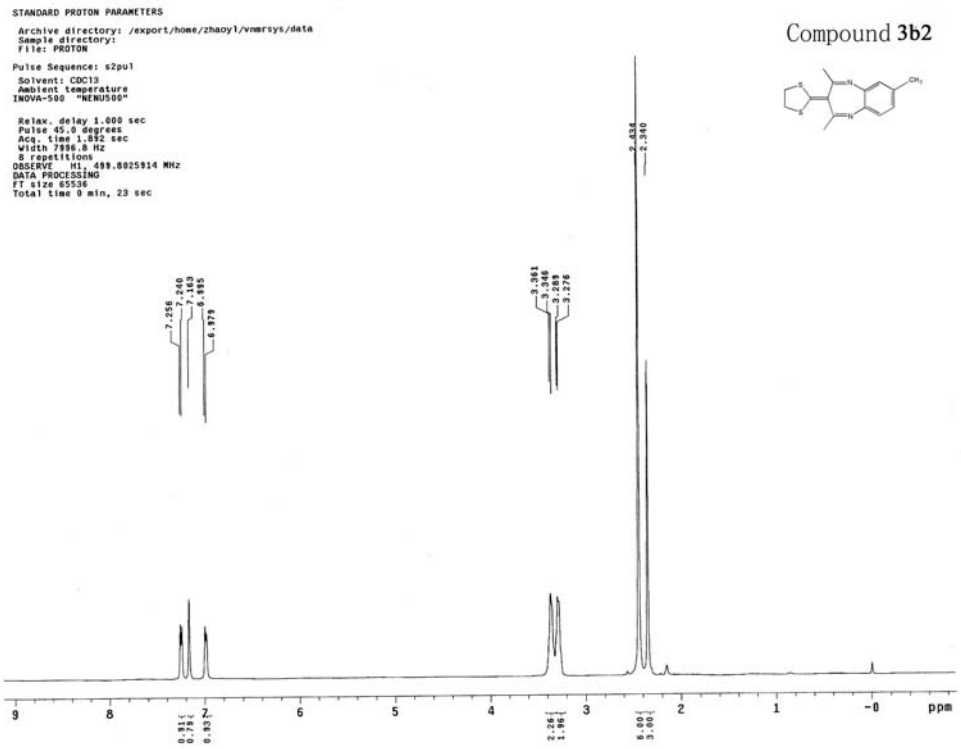

STANDARD CARBON PARAMETERS
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-07
File: a221-c13
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754381 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 33 min, 57 sec
Compound 3a4
ppm

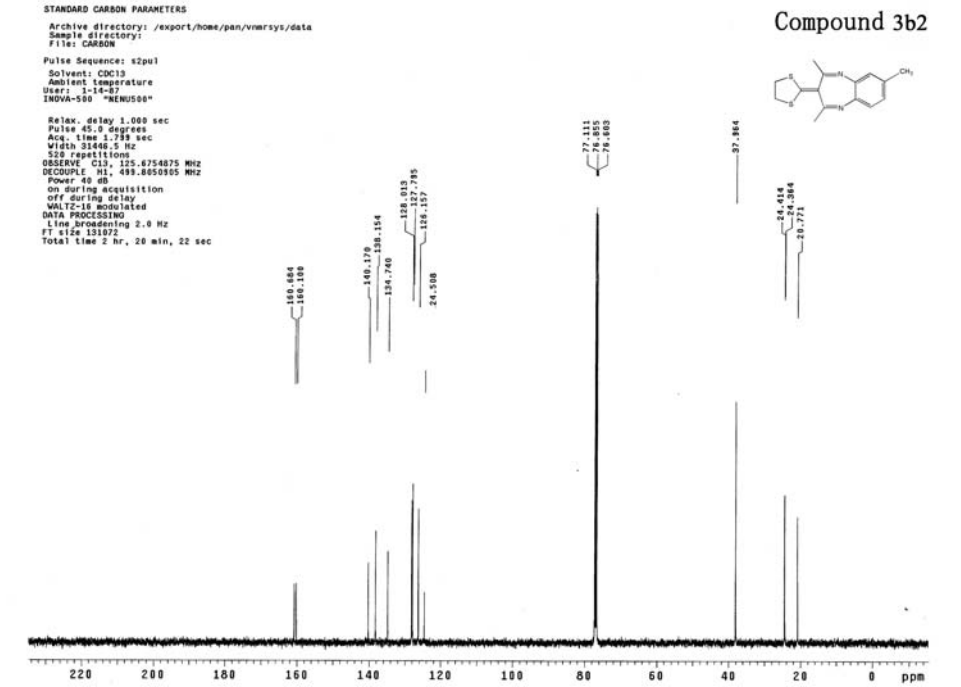

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7998.8 Hz
8 repetitions
OBSERVE H1, 499.8025859 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Compound 3a5
NO2
ppm

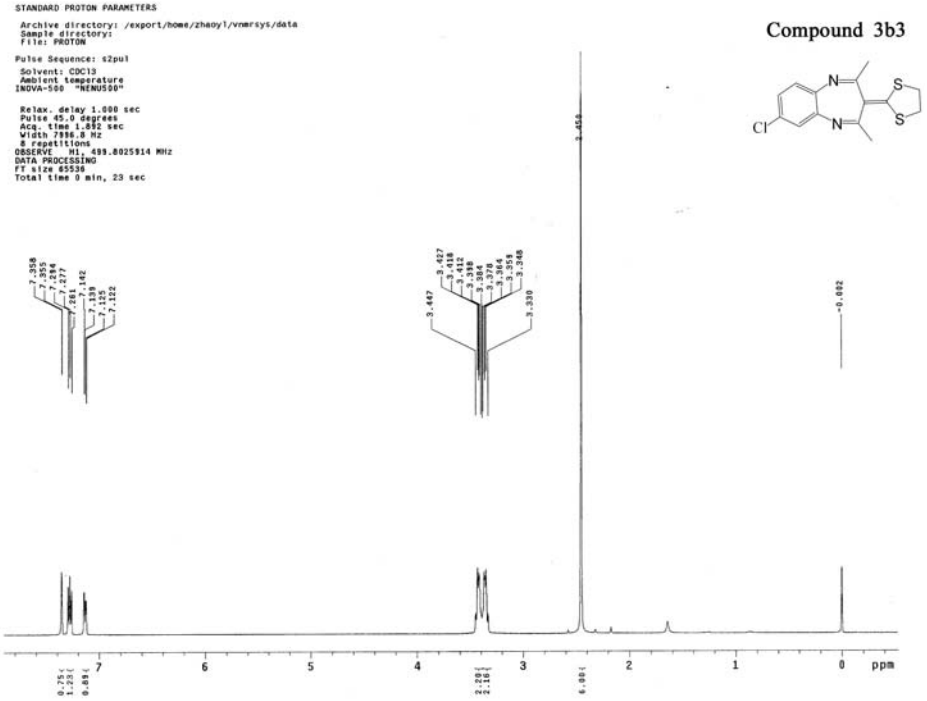

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
Compound 3a5
NO2
ppm

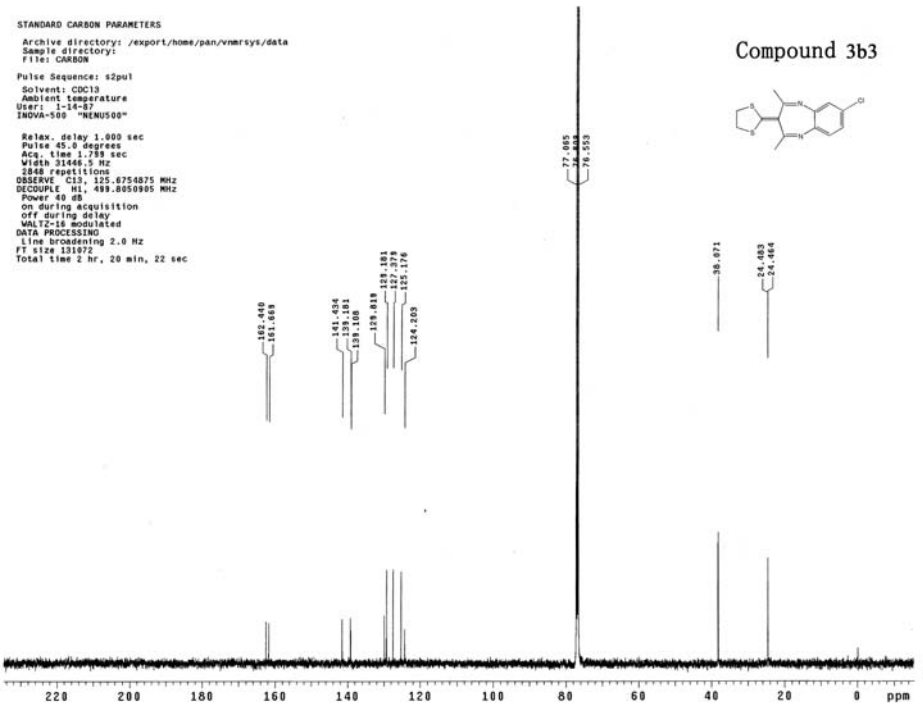

STANDARD PROTON PARAMETERS
Archive directory: /export/home/pan/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.999 sec
Width 8000.9 Hz
8 repetitions
OBSERVE H1, 499.8025914 MHz
DATA PROCESSING
FT size 131072
Total time 0 min, 24 sec
Compound 3b1
ppm

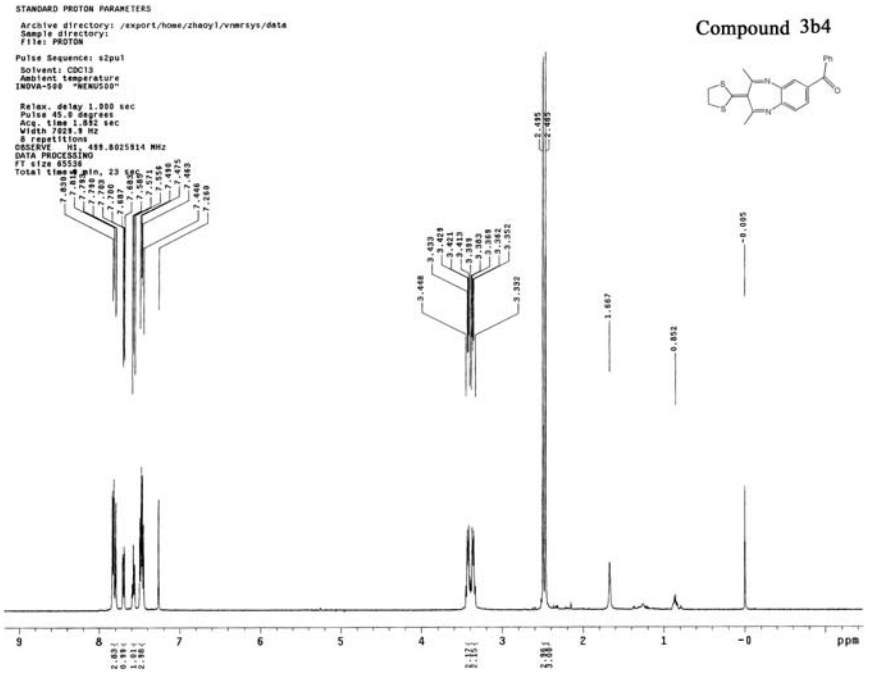

STANDARD CARBON PARAMETERS
Archive directory: /export/home/pan/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: I-14-87
File: 513-c13
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.799 sec
Width 31446.5 Hz
2500 repetitions
OBSERVE C13, 125.6754655 MHz
DECOUPLE H1, 499.8050805 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 1 hr, 56 min, 58 sec
Compound 3b1
ppm

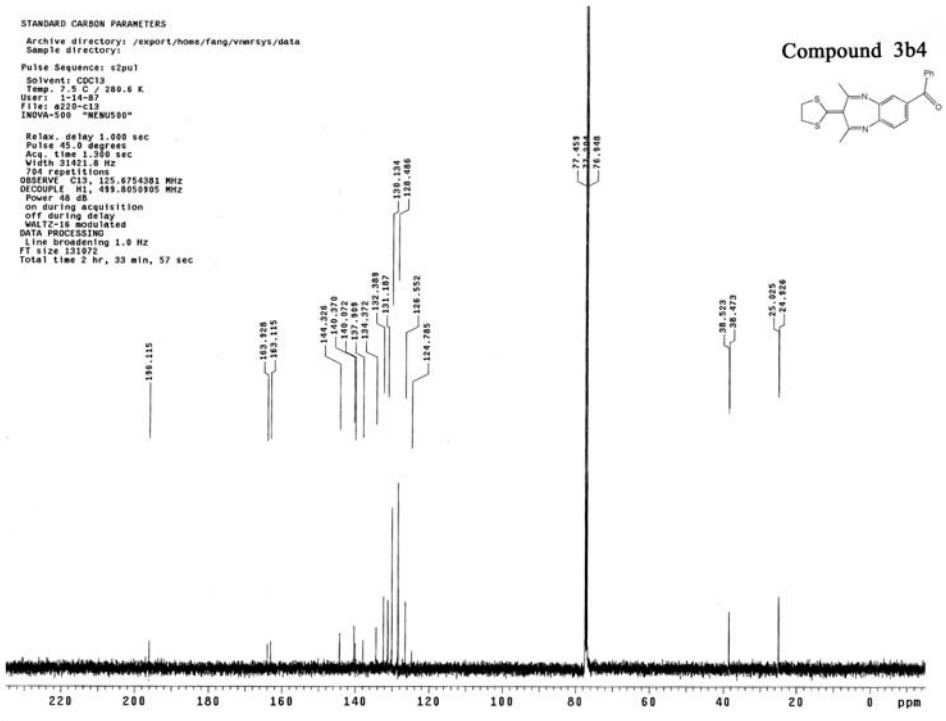

STANDARD PROTON PARAMETERS
Archive directory: /export/home/zhaoyl/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.832 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025914 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Compound 3b2
7.256
7.240
7.163
6.895
6.879
3.061
3.346
3.269
3.276
2.434
2.340
0.81
0.78
0.93
2.06
1.96
6.00
3.00
9
8
7
6
5
4
3
2
1
-0
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/pan/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.799 sec
Width 31446.5 Hz
520 repetitions
OBSERVE C13, 125.6754875 MHz
DECOUPLE H1, 499.8050505 MHz
Power 40 dB
On during acquisition
Off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 2 hr, 20 min, 22 sec
Compound 3b2
160.484
160.100
140.178
138.154
134.740
128.013
127.795
126.157
24.508
77.111
76.855
76.603
57.864
24.414
24.364
20.771
220
200
180
160
140
120
100
80
60
40
20
0
ppm

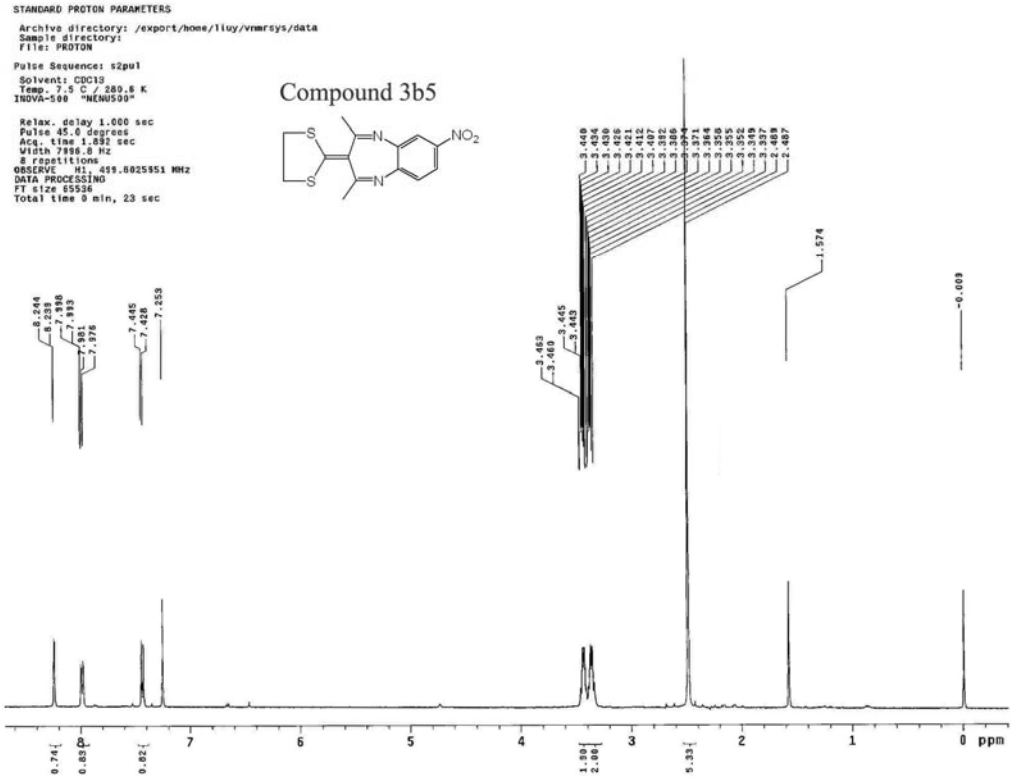

STANDARD PROTON PARAMETERS
Archive directory: /export/home/zhaoyl/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7886.8 Hz
8 repetitions
OBSERVE H1, 499.8025914 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Compound 3b3
7.358
7.355
7.304
7.277
7.281
7.142
7.139
7.125
7.122
3.447
3.427
3.418
3.415
3.398
3.384
3.374
3.364
3.359
3.348
3.330
3.458
-0.002
ppm

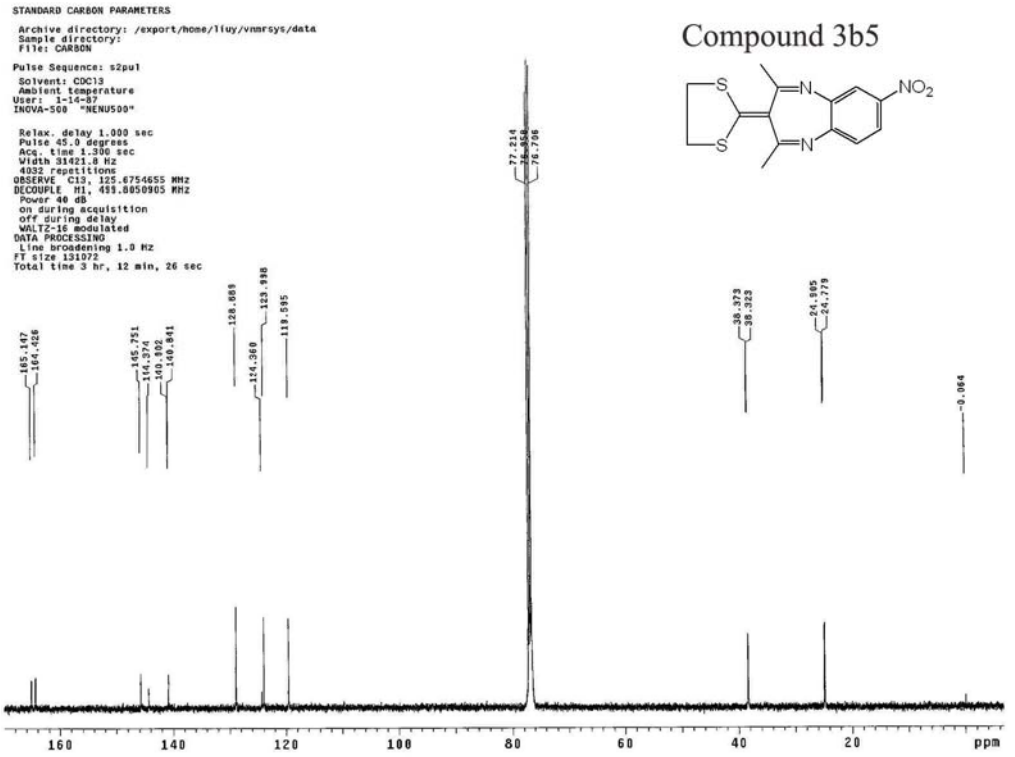

STANDARD CARBON PARAMETERS
Archive directory: /export/home/pan/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.789 sec
Width 31446.5 Hz
2848 repetitions
OBSERVE C13, 125.6754875 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 2 hr, 20 min, 22 sec
Compound 3b3
162.440
161.669
141.494
139.881
139.108
129.819
128.181
127.379
125.176
124.203
77.065
76.444
76.553
38.071
24.483
24.464
ppm

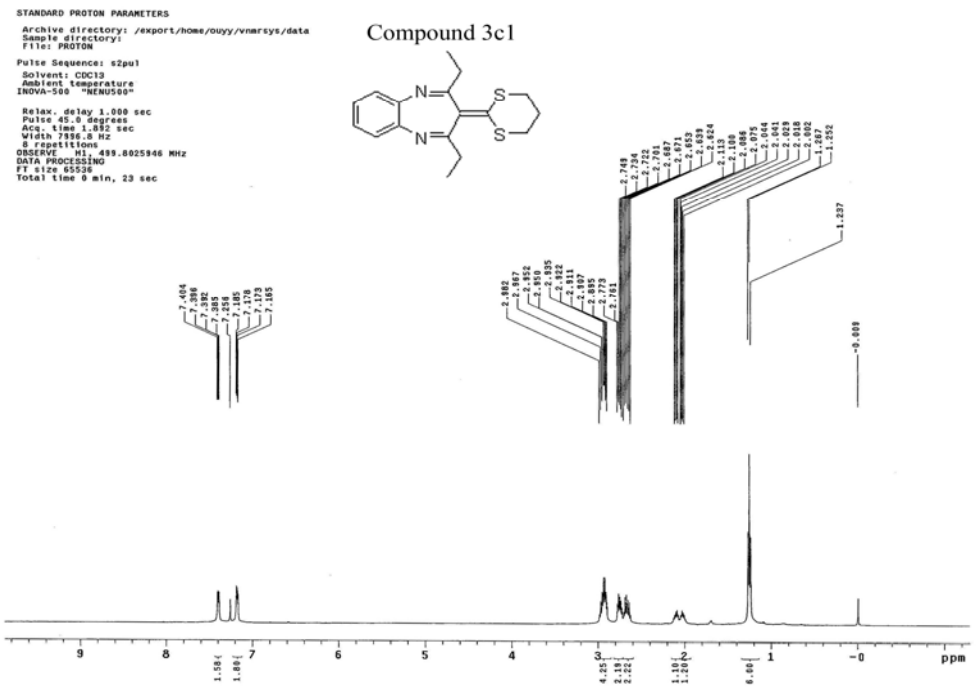

STANDARD PROTON PARAMETERS
Archive directory: /export/home/zhaoyl/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.852 sec
Width 7028.9 Hz
8 repetitions
OBSERVE H1, 499.8025914 MHz
DATA PROCESSING
FT size 65536
Compound 3b4
ppm

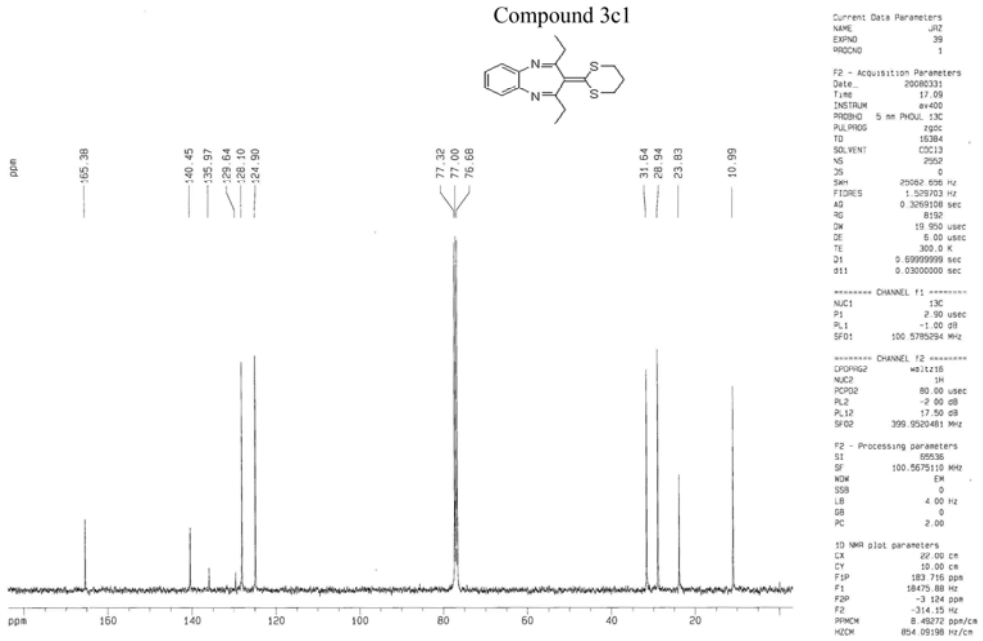

STANDARD CARBON PARAMETERS
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
File: a220-c13
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
704 repetitions
OBSERVE C13, 125.6754381 MHz
DECOUPLE H1, 499.8050905 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 33 min, 57 sec
Compound 3b4
ppm

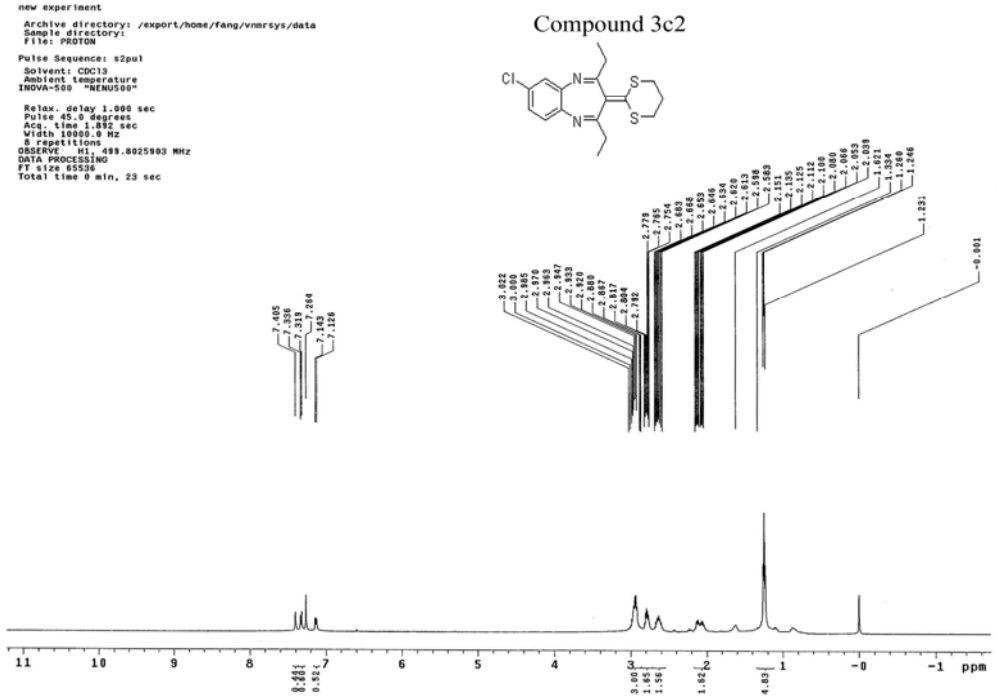
]

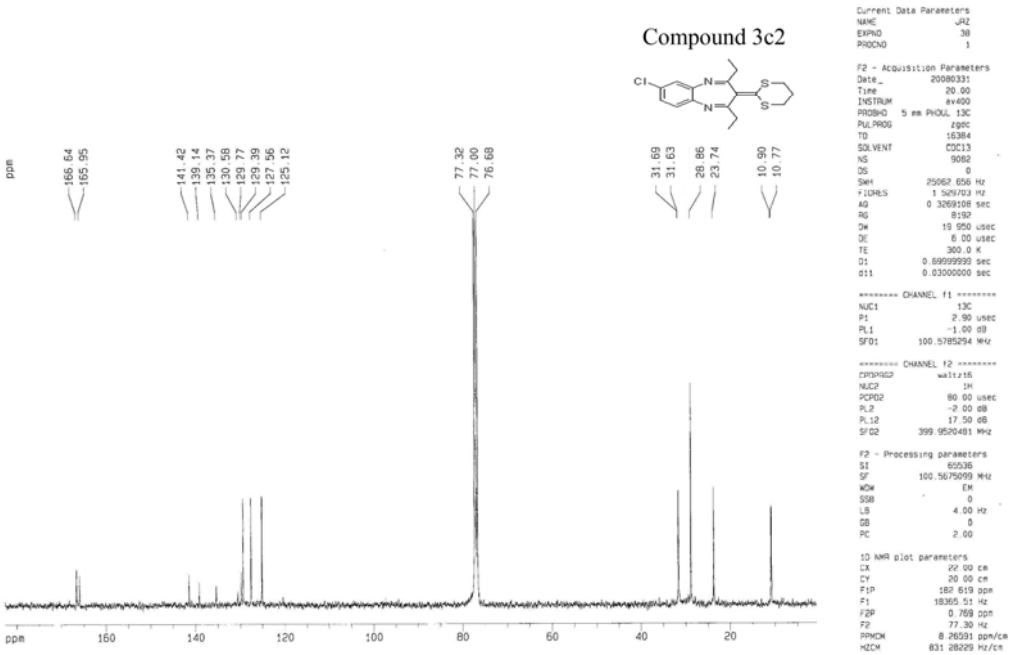

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7998.8 Hz
8 repetitions
OBSERVE H1, 499.8025951 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Compound 3b5
NO2
ppm

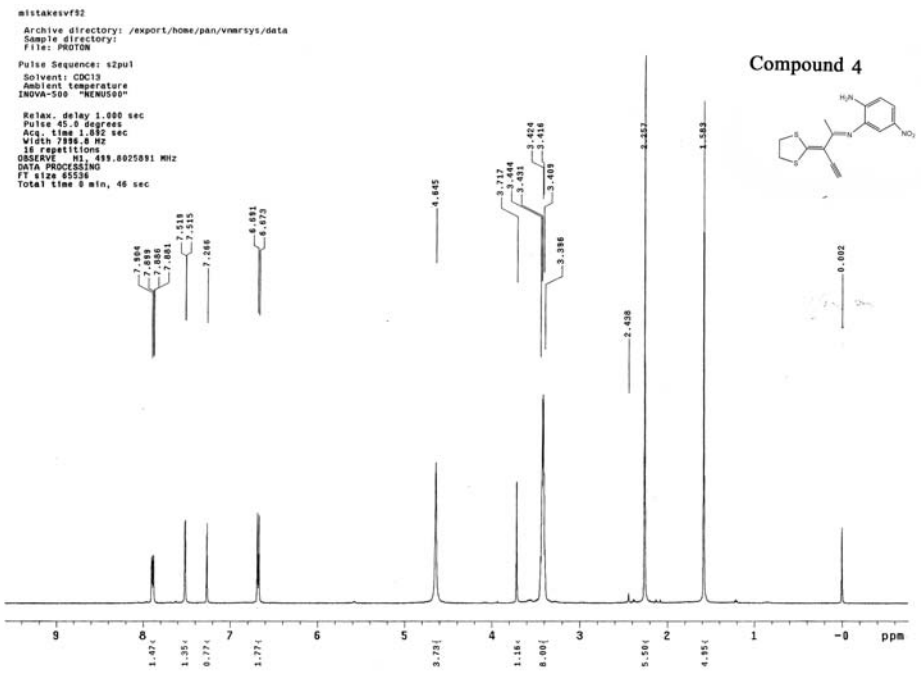

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-16-07
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4032 repetitions
OBSERVE C13, 125.6754655 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
Compound 3b5
NO2
ppm

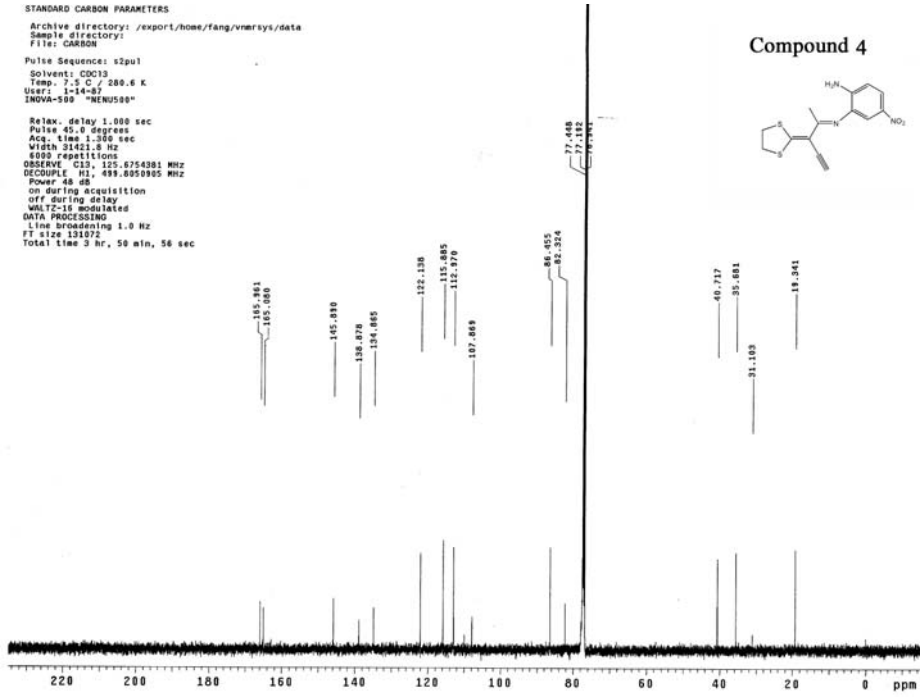

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025946 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Compound 3c1
ppm

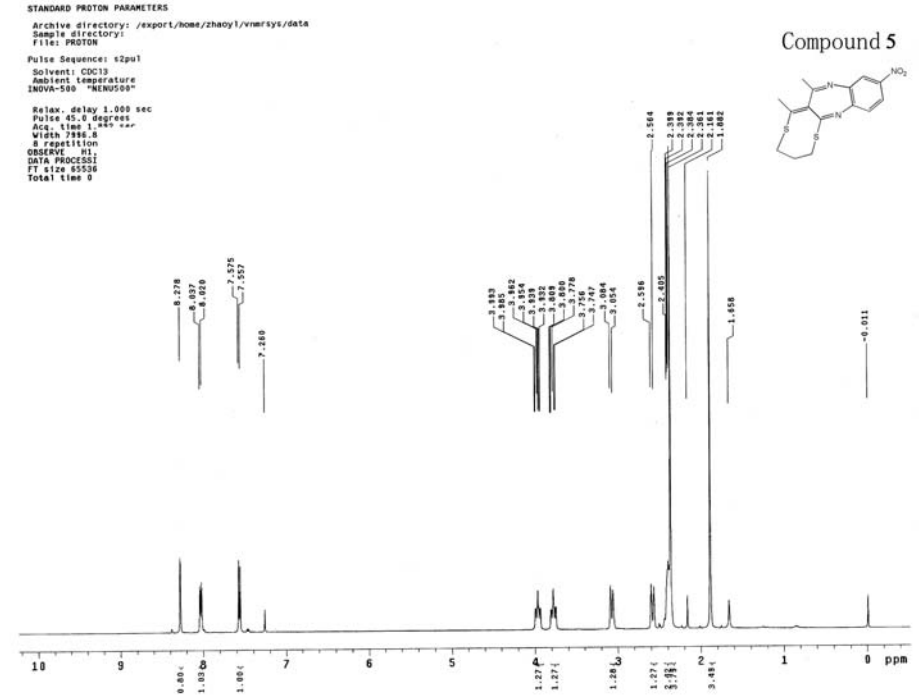

Compound 3c1
ppm
Current Data Parameters
NAME JAZ
EXPNO 39
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080331
Time 17.09
INSTRUM av400
PROBHD 5 mm PHOUL 13C
PULPROG zgpg
TD 16384
SOLVENT CDCl3
NS 2952
DS 0
SWH 25062.656 Hz
FIDRES 1.529703 Hz
AQ 0.3269108 sec
RG 8192
DW 19.950 usec
DE 6.00 usec
TE 300.0 K
D1 0.69999999 sec
d11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 2.90 usec
PL1 -1.00 dB
SF01 100.5785294 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.50 dB
SF02 399.9520481 MHz
F2 - Processing parameters
SI 65536
SF 100.5675110 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 27.00 cm
CY 10.00 cm
F1P 183.716 ppm
F1 18475.88 Hz
F2P -3.124 ppm
F2 -314.15 Hz
PPMCM 8.49272 ppm/cm
HZCM 854.09198 Hz/cm

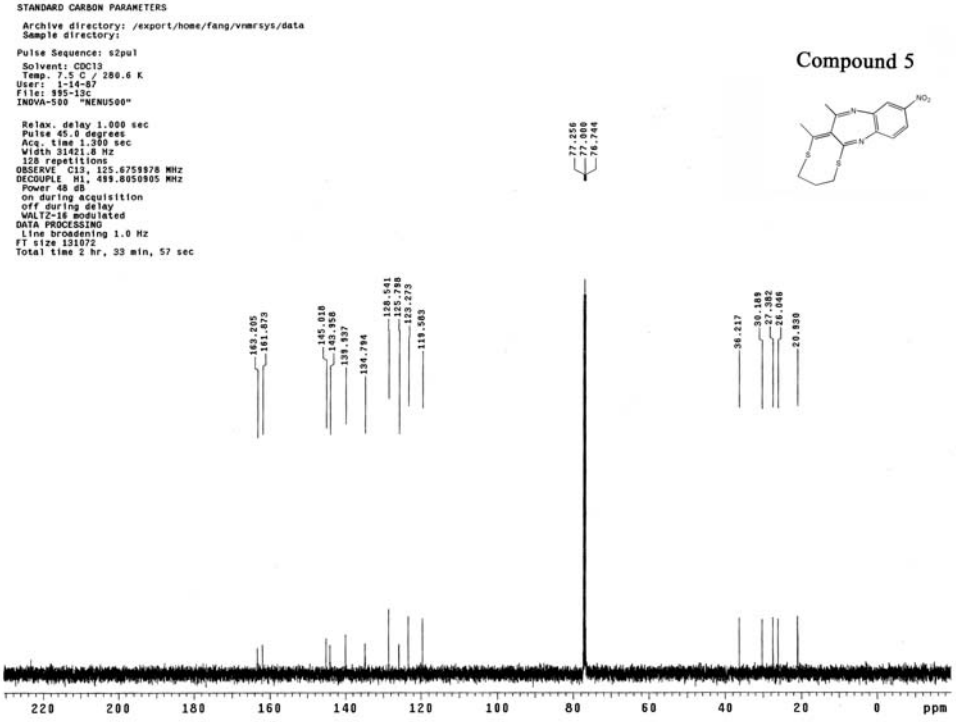

new experiment
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 10000.0 Hz
8 repetitions
OBSERVE H1, 499.8025903 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Compound 3c2
Cl
N
S
7.405
7.386
7.319
7.264
7.143
7.126
3.022
3.000
1.985
2.970
2.963
2.933
2.920
2.880
2.817
2.804
2.782
2.778
2.755
2.754
2.683
2.666
2.653
2.634
2.620
2.613
2.588
2.569
2.151
2.135
2.125
2.112
2.100
2.080
2.066
2.053
2.039
1.621
1.334
1.260
1.246
1.231
-0.001
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm

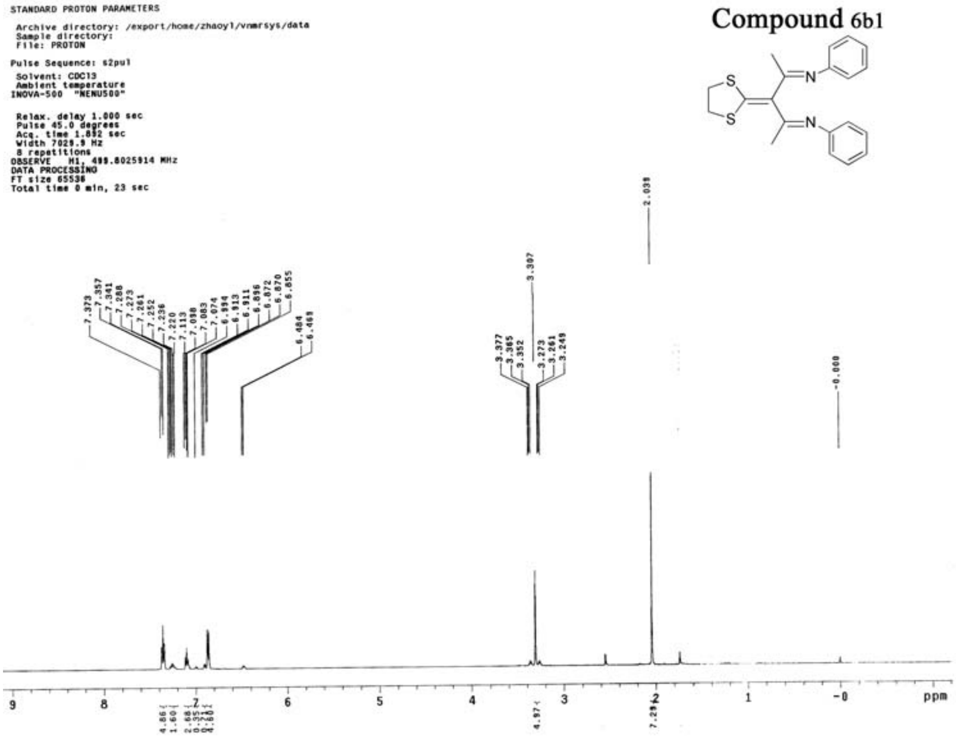

Compound 3c2
Cl
N
S
ppm
166.64
165.95
141.42
139.14
135.37
130.58
129.77
129.39
127.56
125.12
77.32
77.00
76.68
31.69
31.63
28.86
23.74
10.90
10.77
160
140
120
100
80
60
40
20
Current Data Parameters
NAME JAZ
EXPNO 38
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080331
Time 20.00
INSTRUM av400
PROBHD 5 mm PHOUL 13C
PULPROG zgpg
TD 16384
SOLVENT CDCl3
NS 9082
DS 0
SWH 25062.656 Hz
FIDRES 1.529703 Hz
AQ 0.3269108 sec
RG 8192
DW 19.950 usec
DE 6.00 usec
TE 300.0 K
D1 0.69999999 sec
d11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 2.90 usec
PL1 -1.00 dB
SFO1 100.5785294 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.50 dB
SFO2 399.9520481 MHz
F2 - Processing parameters
SI 65536
SF 100.5675099 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.00 cm
CY 20.00 cm
F1P 182.619 ppm
F1 18365.51 Hz
F2P 0.769 ppm
F2 77.30 Hz
PPMCM 8.26591 ppm/cm
HZCM 831.28229 Hz/cm

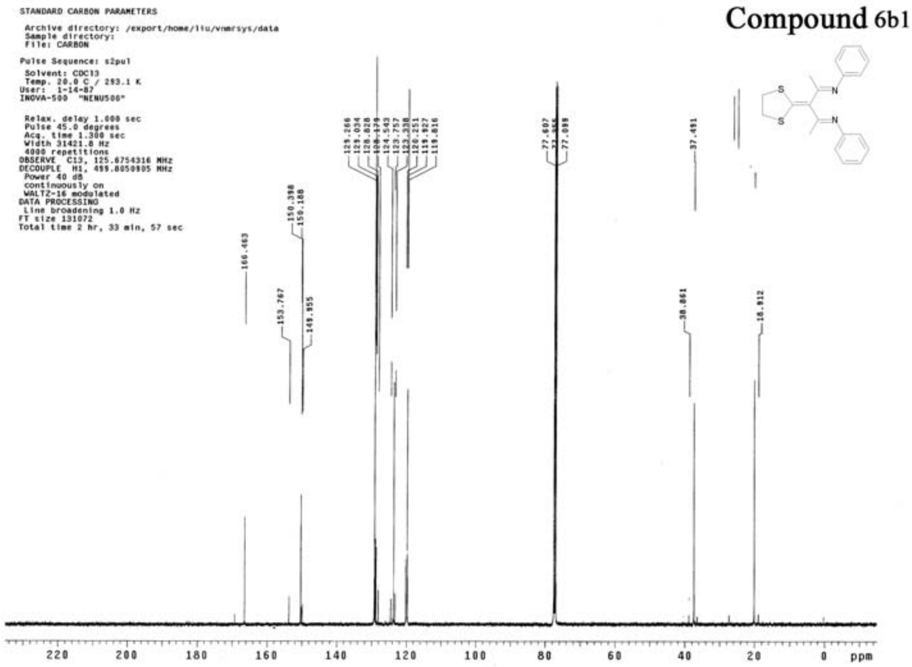

mistakesvf92
Archive directory: /export/home/pan/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
16 repetitions
OBSERVE H1, 499.8025891 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 46 sec
Compound 4
ppm

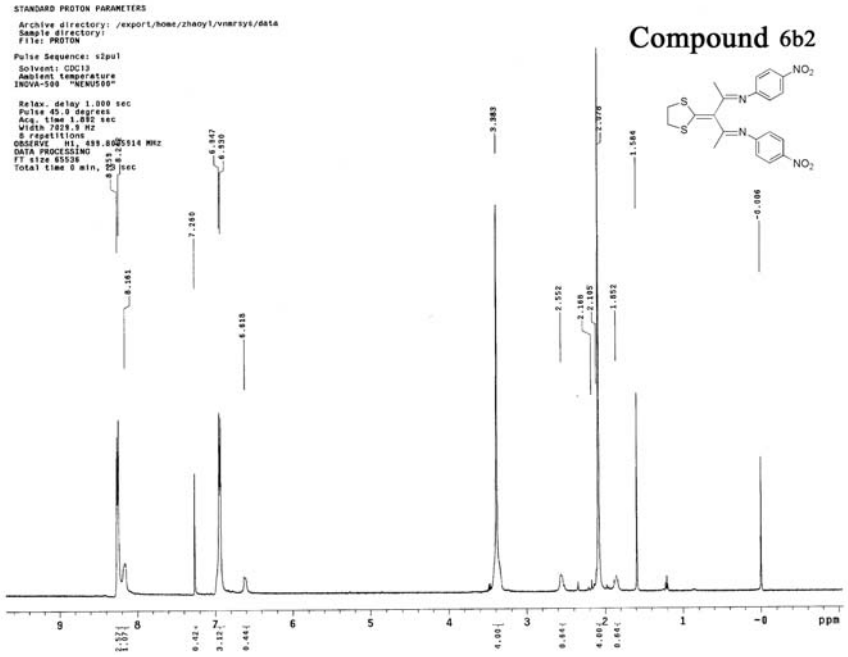

STANDARD CARBON PARAMETERS
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1=14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
6000 repetitions
OBSERVE C13, 125.6754381 MHz
DECOUPLE H1, 499.8050905 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 50 min, 56 sec
Compound 4
ppm

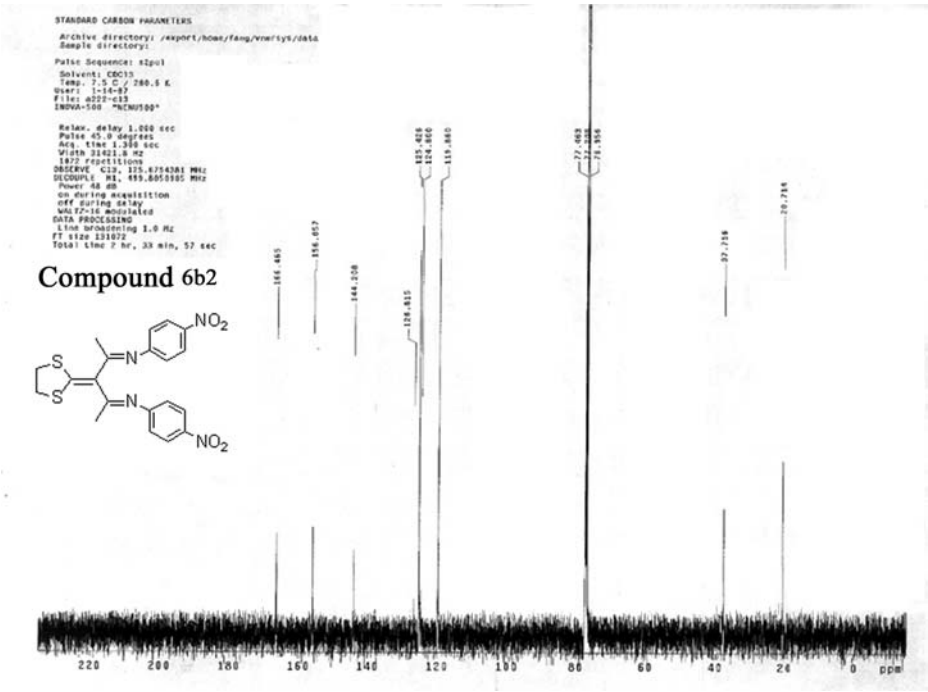

STANDARD PROTON PARAMETERS
Archive directory: /export/home/zhaoyl/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Width 7986.8
8 repetition
OBSERVE H1,
DATA PROCESSI
FT size 65536
Total time 0
Compound 5
8.278
8.037
8.020
7.575
7.557
7.260
3.893
3.885
3.882
3.865
3.932
3.939
3.800
3.778
3.756
3.747
3.054
2.596
2.564
2.405
2.399
2.392
2.386
2.361
2.361
1.682
1.458
-0.011
10
9
8
7
6
5
4
3
2
1
0 ppm

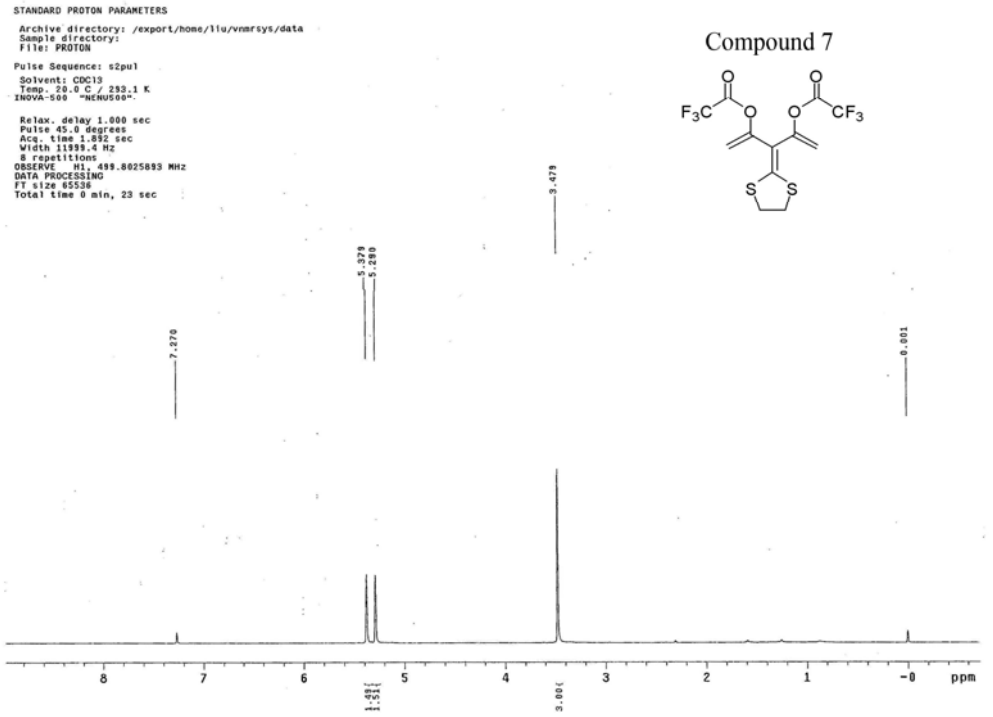

STANDARD CARBON PARAMETERS
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
File: 895-13c
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6759878 MHz
DECOUPLE H1, 499.8050905 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 33 min, 57 sec
Compound 5
77.256
77.000
76.744
163.205
161.873
145.018
143.958
139.337
134.794
128.541
125.708
123.273
119.583
56.217
30.189
27.382
26.046
20.830
220
200
180
160
140
120
100
80
60
40
20
0 ppm

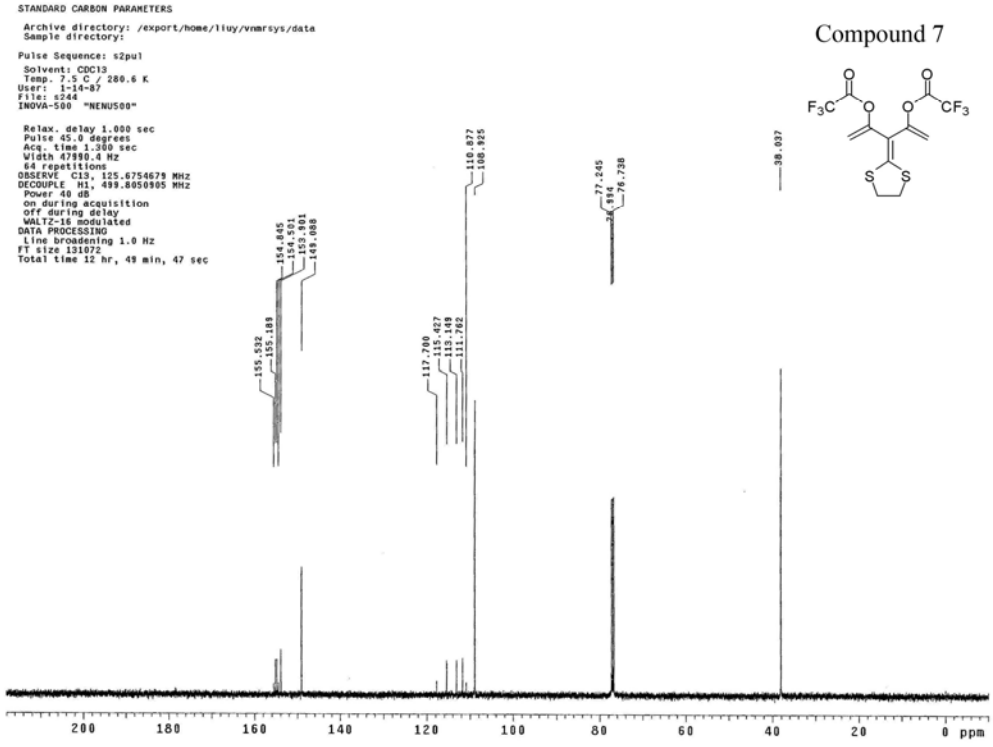

STANDARD PROTON PARAMETERS
Archive directory: /export/home/zhaoyl/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
8 repetitions
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Compound 6b1
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liu/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4000 repetitions
OBSERVE C13, 125.6754316 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 33 min, 57 sec
Compound 6b1
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/zhaoyl/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7029.5 Hz
8 repetitions
OBSERVE H1, 499.8855514 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 25 sec
Compound 6b2
NO2
NO2
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.5 K
User: 1-14-07
File: a022-c13
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1072 repetitions
OBSERVE C13, 175.6754381 MHz
DECOUPLE H1, 499.8859805 MHz
Power 48 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 33 min, 57 sec
Compound 6b2
NO2
NO2
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 11999.4 Hz
8 repetitions
OBSERVE H1, 499.8025893 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Compound 7
7.270
5.279
5.269
3.479
0.001
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
File: s244
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 47890.4 Hz
64 repetitions
OBSERVE C13, 125.6754679 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
FT size 131072
Total time 12 hr, 49 min, 47 sec
Compound 7
155.532
155.188
154.845
154.501
153.901
149.088
117.700
115.427
113.149
111.792
110.877
108.925
77.245
77.034
76.738
38.037
ppm