Supplementary Material (ESI) for Chemical Communications

Supporting Information

Tandem Nazarov cyclization-halovinylation of divinyl ketones under Vilsmeier conditions: synthesis of highly substituted cyclopentadienes

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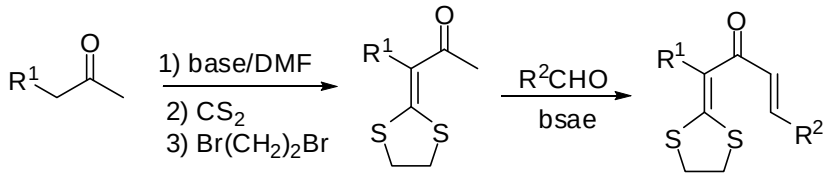
I. General information

All reagents were commercial and used without further purification, unless otherwise indicated. 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 (F254). Melting points were uncorrected. The ^{1}H NMR and ^{13}C NMR spectra were determined at 25ºC on a 500 MHz and 125 MHz, respectively, and TMS as internal standard. All shifts are given in ppm. IR (KBr) spectra were recorded on in the range of 400−4000 cm^{-1}. Mass spectra were obtained using the ESI method. Elemental analyses were measured on a GmbH VarioEL analyzer (Elementar Analysensysteme). The compound **2f** with dimension 0.40×0.30×0.25 mm, was glued on a glass fiber. Data were collected at 273K using graphite-monochromated Mo Kα radiation (λ = 0.71073Å) and Bruker APEX CCD area-detector in the range 1.70° < θ < 26.07°. Absorption corrections were applied using multi-scan technique. The structure was solved by Direct Method of SHELXS-97[1] and refined by full-matrix least-squares techniques using the SHELXL-97 program[2] within WINGX.[3] Non-hydrogen atoms were refined with anisotropic temperature parameters except C7, C8, C7′ and C8′. The disordered atoms C7 and C8 have been refined using C atoms split over two sites, with a total occupancy of 1. The hydrogen atoms of the organic ligands were refined as rigid groups. Substrates **1** were prepared following the procedure described in our previous work.[4] **D**, **1m** and **1n**[4a] are known compounds.

II. Synthetic procedures/analytical data of compounds 1.



A: R^1 = 4-$MeOC_6H_4$
B: R^1 = 2-$MeOC_6H_4$
C: R^1 = 4-FC_6H_4
D: R^1 = Me

1

General procedure for the synthesis of 1a–g, 1k and 1*l* (taking 1a as an example): To a well-stirred suspension of 1-(4-methoxyphenyl)propan-2-one (3.28 g, 20 mmol), anhydrous K_2CO_3 (6.90 g, 50 mmol) and anhydrous DMF (50 mL) was added CS_2 (1.46 mL, 24 mmol) at 0 °C. After the reaction mixture was stirred at 0 °C for 0.5 h, 1,2-dibromoethane (1.74 mL, 20 mmol) was added dropwise within 15 min. The resulting mixture was stirred for 10 h at room temperature. Then, the mixture was poured into 200 mL of saturated aqueous NH_4Cl and extracted with CH_2Cl_2 (50 mL × 3). The combined organic phase was washed with water (50 mL × 3), dried over anhydrous $MgSO_4$, filtered and concentrated in vacuo. The crude product was purified by flash chromatography (silica gel, petroleum ether/diethyl ether: 10/1) to give 1-(1,3-dithiolan-2-ylidene)-1-(4-methoxyphenyl) propan-2-one **A** (4.69 g, 88%) as a yellow crystal. Then, to a stirred solution of **A** (266 mg, 1.0 mmol) and benzaldehyde (0.11 mL, 1.1 mmol) in EtOH (5.0 mL) was added NaOH (60 mg, 1.5 mmol) in one portion at room temperature. After **A** was consumed as indicated by TLC, the resulting mixture was quenched by ice-water (20 mL) under stirring and neutralized with dilute hydrochloric acid. The precipitate was collected by filtration, washed with water (15 mL × 3) and dried at ambience to give **1a** (326 mg, 92%) as a yellow crystal.

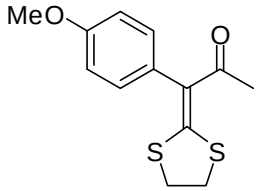
General procedure for the synthesis of 1h–j (taking 1h as an example): To a well-stirred suspension of anhydrous *t*-BuOK (4.94 g, 44 mmol) and 1-(2-methoxyphenyl)propan-2-one (3.12 mL, 20 mmol) in 50 mL of anhydrous DMF were added CS_2 (1.32 mL, 20 mmol) at 0 ºC. After the

reaction mixture was stirred at 0 °C for 0.5 h, 1,2-dibromoethane (1.74 mL, 20 mmol) was added dropwise within 30 min. The mixture was allowed to warm to room temperature and stirred for 10 h, and then poured into saturated aqueous NH_4Cl (200 mL) under stirring. The resulting mixture was extracted with CH_2Cl_2 (50 mL × 3). The combined organic phase was washed with water (50 mL × 3), dried over anhydrous $MgSO_4$, filtered and concentrated in vacuo. The crude product was purified by flash chromatography (silica gel, petroleum ether/diethyl ether: 2/1) to give 1-(1,3-dithiolan-2-ylidene)-1-(2-methoxyphenyl) propan-2-one **B** (3.46 g, 65%) as a yellow crystal. Then, to a solution of **B** (266 mg, 1.0 mmol) in *t*-BuOH (5 mL) was added *t*-BuOK (168 mg, 1.5 mmol) and benzaldehyde (0.11 mL, 1.1 mmol) under stirring. The reaction mixture was stirred at 30 °C for 5.0 h. After the starting material **B** was consumed as indicated by TLC, the resulting mixture was poured into water and neutralized with dilute hydrochloric acid. The resulting precipitate was collected by filtration, washed with water (10 mL × 3) and dried at ambience to give **1h** (304 mg, 86%) as a yellow crystal.

Synthesis of 1m: To a well-stirred suspension of anhydrous *t*-BuOK (4.94 g, 44 mmol) and 2-butanone (1.8 mL, 20 mmol) in 50 mL of anhydrous DMF were added CS_2 (1.32 mL, 20 mmol) at 0 ºC. After the reaction mixture was stirred at 0 °C for 1.5 h, 1,2-dibromoethane (1.74 mL, 20 mmol) was added dropwise within 30 min. The mixture was allowed to warm to room temperature and stirred for 10 h, and then poured into saturated aqueous NH_4Cl (200 mL) under stirring. The resulting mixture was extracted with CH_2Cl_2 (50 mL × 3). The combined organic phase was washed with water (50 mL × 3), dried over anhydrous $MgSO_4$, filtered and concentrated in vacuo. The crude product was purified by flash chromatography (silica gel, petroleum ether/diethyl ether: 2/1) to give 3-(1,3-dithiolan-2-ylidene)butan-2-one **D** (695 mg, 20%) as a white solid. Then, to a stirred solution of **D** (174 mg, 1.0 mmol) and benzaldehyde (0.11 mL, 1.1 mmol) in EtOH (5.0 mL) was added NaOH (60 mg, 1.5 mmol) in one portion at room temperature. After **C** was consumed as indicated by TLC, the resulting mixture was quenched by ice-water (20 mL) under stirring and neutralized with dilute hydrochloric acid. The precipitate was collected by filtration, washed with water (15 mL × 3) and dried at ambience to give **1m** (212 mg, 81%) as a yellow crystal.

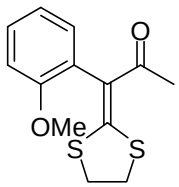
General procedure for the synthesis of 1′a–c (taking 1′a as an example): A solution of 1-(4-methoxyphenyl)propan-2-one (1.64 g, 10 mmol), benzaldehyde (4.0 ml, 40 mmol), piperidine (4.0 ml, 4.0 mmol), and acetic acid (0.6 ml, 6.0 mmol) in benzene (25 mL) was heated under reflux for 24 h with azotropic removal of water using a Dean–Stark trap. The reaction mixture was diluted with EtOAc (200 mL), washed with H_2O (100 mL × 3), saturated aqueous $NaHCO_3$ (100 mL × 3), dried ($MgSO_4$), and concentrated in vacuo. The residue was chromatographed over silica gel (elute, petroleum ether/diethyl ether: 6/1) to give **1′a** (1.74 g, 51%).

1-(1,3-Dithiolan-2-ylidene)-1-(4-methoxyphenyl)propan-2-one (A)



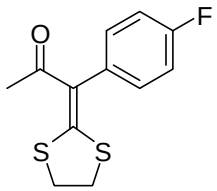
Yellow crystal; mp 80−82 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 1.98 (s, 3H), 3.21 (t, *J* = 6.5 Hz, 2H), 3.46 (t, *J* = 6.0 Hz, 2H), 3.85 (s, 3H), 6.96 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 194.1, 164.7, 159.4, 133.2, 131.1 (2C), 126.6, 114.5 (2C), 55.5, 40.1, 35.9, 28.7. IR (KBr): 3061, 2926, 2837, 1634, 1513, 1468, 1281 cm^{-1}. MS (ESI): *m/z* = 267.0 $[(M+1)]^+$. Anal. Calcd for $C_{13}H_{14}O_2S_2$: C, 58.62; H, 5.30; Found: C, 58.83; H, 5.21.

1-(1,3-Dithiolan-2-ylidene)-1-(2-methoxyphenyl)propan-2-one (B)



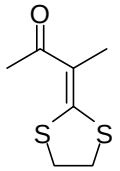
Yellow crystal; mp 118−120 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 1.94 (s, 3H), 3.20 (t, *J* = 6.5 Hz, 2H), 3.43−3.47 (m, 2H), 3.79 (s, 3H), 6.96−7.01 (m, 2H), 7.20−7.21 (m, 1H), 7.35−7.38 (m, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 193.8, 163.0, 156.67, 131.2, 129.7, 129.0, 122.8, 120.7, 111.0, 55.3, 39.5, 35.3, 27.4. IR (KBr): 3059, 2996, 1643, 1474, 1466, 1303, 1264 cm^{-1}. MS (ESI): *m/z* = 267.0 $[(M+1)]^+$. Anal. Calcd for $C_{13}H_{14}O_2S_2$: C, 58.62; H, 5.30; Found: C, 58.90; H, 5.13.

1-(1,3-Dithiolan-2-ylidene)-1-(4-fluorophenyl)propan-2-one (C)



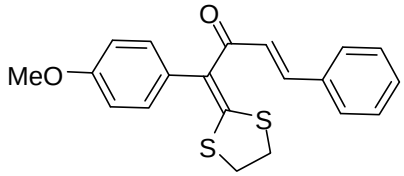
Yellow crystal; mp 126−127°C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 1.97 (s, 3H), 3.22 (t, *J* = 6.5 Hz, 2H), 3.45 (t, *J* = 6.5 Hz, 2H), 7.12−7.14 (m, 2H), 7.21−7.23 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 193.2. 164.9, 162.3 (d, *J* = 246.0 Hz, 1C), 136.5, 131.5, 131.4, 125.7, 116.0 (d, *J* = 21.5 Hz, 2C), 39.7, 35.7, 28.4. IR (KBr): 3056, 1626, 1509, 1548, 1253, 1236 cm^{-1}. MS (ESI): *m/z* = 255.0 $[(M + 1)]^+$. Anal. Calcd for $C_{12}H_{11}FOS_2$: C, 56.67; H, 4.36; Found: C, 56.41; H, 4.50.

3-(1,3-Dithiolan-2-ylidene)butan-2-one (D)



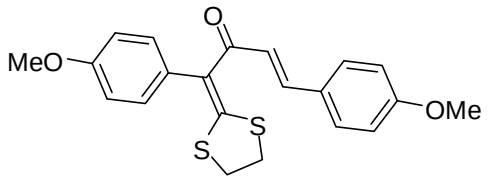
White crystal; mp 56−58 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 2.19 (s, 3H), 2.27 (s, 3H), 3.31−3.41 (m, 4H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 194.5, 159.3, 119.5, 39.5, 35.7, 27.7, 19.9. IR (KBr): 3024, 2967, 1510, 1239, 829 cm^{-1}. MS (ESI): *m/z* = 175.0 $[(M+1)]^+$. Anal. Calcd for $C_7H_{10}OS_2$: C, 48.24; H, 5.78; Found: C, 48.01; H, 5.64.

(*E*)-1-(1,3-Dithiolan-2-ylidene)-1-(4-methoxyphenyl)-4-phenylbut-3-en-2-one (1a)



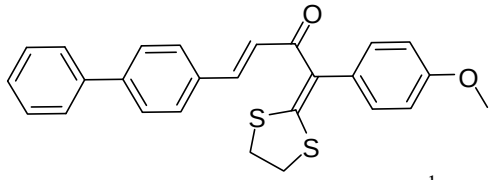
Light yellow crystal; mp 136−138 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.25 (t, *J* = 6.0 Hz, 2H), 3.49 (t, *J* = 6.0 Hz, 2H), 3.88 (s, 3H), 6.65 (d, *J* = 16.0 Hz, 1H), 6.99 (d, *J* = 9.0 Hz, 2H), 7.24 (t, *J* = 8.5 Hz, 2H), 7.26–7.29 (m, 3H), 7.35 (t, *J* = 7.5 Hz, 2H), 7.67 (d, *J* = 16.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 184.6, 167.0, 159.5, 142.6, 135.6, 132.2, 131.6 (2C), 130.1, 128.9 (2C), 128.5 (2C), 127.3, 123.9, 114.5 (2C), 55.5, 40.1, 35.9. IR (KBr): 3047, 2915, 1636, 1571, 1452, 1241 cm^{-1}. MS (ESI): *m/z* = 355.0 $[(M+1)]^+$. Anal. Calcd for $C_{20}H_{18}O_2S_2$: C, 67.76; H, 5.12; Found: C, 67.99; H, 5.04.

(*E*)-1-(1,3-Dithiolan-2-ylidene)-1,4-bis(4-methoxyphenyl)but-3-en-2-one (1b)



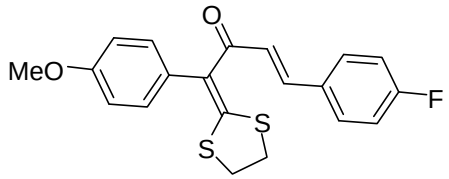
Light yellow crystal; mp 208−210 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.24 (t, *J* = 5.5 Hz, 2H), 3.47−3.49 (m, 2H), 3.79 (s, 3H), 3.88 (s, 3H), 6.51 (d, *J* = 15.5 Hz, 1H), 6.81 (t, *J* = 7.0 Hz, 2H), 6.98 (t, *J* = 7.0 Hz, 2H), 7.22(t, *J* = 7.0 Hz, 2H), 7.30 (t, *J* = 7.0 Hz, 2H), 7.63 (d, *J* = 15.5 Hz, 1H), ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 184.8, 161.3, 159.5, 142.4, 132.4, 131.6 (2C), 130.2 (2C), 128.4, 127.4, 121.7 (2C), 114.4 (4C), 55.6, 55.5, 40.1, 35.9. IR (KBr): 3069, 2919, 1604, 1565, 1511, 1458, 1243 cm^{-1}. MS (ESI): *m/z* = 385.0 [(M+1)]$^+$. Anal. Calcd for $C_{21}H_{20}O_3S_2$: C, 65.60; H, 5.24; Found: C, 67.81; H, 5.16.

(*E*)-4-(biphenyl-4-yl)-1-(1,3-dithiolan-2-ylidene)-1-(4-methoxyphenyl)but-3-en-2-one (1c)



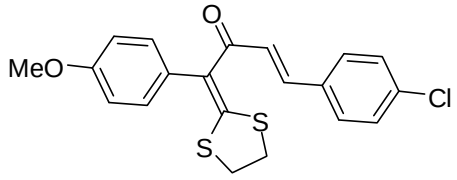
Yellow crystal; mp 208−210 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.23 (t, *J* = 6.0 Hz, 2H), 3.47 (t, *J* = 6.0 Hz, 2H), 3.88 (s, 3H), 6.66 (d, *J* = 15.5 Hz, 1H), 7.00 (d, *J* = 8.5 Hz, 2H), 7.23 (d, *J* = 8.5 Hz, 2H), 7.41−7.43 (m, 4H), 7.51−7.56 (m, 5H), 7.69 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 184.2, 166.6, 159.2, 142.5, 141.8, 140.1, 134.3, 131.9, 131.3, 130.9, 128.8 (2C), 128.7 (2C), 127.6, 127.3, 127.0, 126.9 (2C), 126.1, 123.5, 114.5, 114.2, 55.2, 39.8, 35.6. IR (KBr): 3064, 2920, 1635, 1407, 1239, 664 cm^{-1}. MS (ESI): *m/z* = 431.1 [(M+1)]$^+$. Anal. Calcd for $C_{26}H_{22}O_2S_2$: C, 72.52; H, 5.15; Found: C, 72.21; H, 5.20.

(*E*)-1-(1,3-Dithiolan-2-ylidene)-4-(4-fluorophenyl)-1-(4-methoxyphenyl)but-3-en-2-one (1d)



Light yellow crystal; mp 142−144 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.26 (t, *J* = 6.0 Hz, 2H), 3.51 (t, *J* = 6.0 Hz, 2H), 3.89 (s, 3H), 6.56 (d, *J* = 15.5 Hz, 1H), 6.96–7.00 (m, 4H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.33−7.34 (m, 2H), 7.62 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 184.4, 167.1, 161.5, 159.6, 141.3, 132.2, 131.6 (2C), 130.3 (2C), 130.2, 127.2, 123.5, 116.1 (d, *J* = 22.0 Hz, 2C), 114.5 (2C), 55.5, 40.1, 35.9. IR (KBr): 3063, 2918, 1601, 1571, 1509, 1453, 1241, 1157 cm^{-1}. MS (ESI): *m/z* = 373.0 [(M+1)]$^+$. Anal. Calcd for $C_{20}H_{17}FO_2S_2$: C, 64.49; H, 4.60; Found: C, 64.80; H, 4.41.

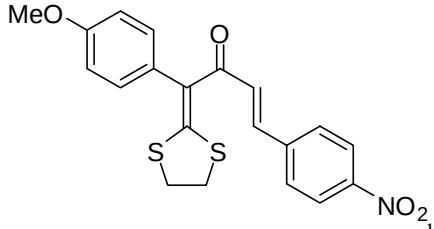
(*E*)-4-(4-Chlorophenyl)-1-(1,3-dithiolan-2-ylidene)-1-(4-methoxyphenyl)but-3-en-2-one (1e)



Light yellow crystal; mp 152−154 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.26 (t, *J* = 6.5 Hz, 2H), 3.50 (t, *J* = 6.5 Hz, 2H), 3.88 (s, 3H), 6.60 (d, *J* = 15.5 Hz, 1H), 6.99 (d, *J* = 9.0 Hz, 2H), 7.22 (d, *J* = 8.5 Hz, 2H), 7.25 (t, *J* = 8.5 Hz, 4H), 7.60 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 184.3, 167.5, 159.6, 141.17, 135.8, 134.1, 132.1, 131.6 (2C), 129.6 (2C), 129.2 (2C), 127.2, 124.3, 114.5 (2C), 55.5, 40.1, 36.0. IR (KBr): 3055, 2920, 1637, 1578, 1453, 1242 cm^{-1}. MS (ESI): *m/z* =

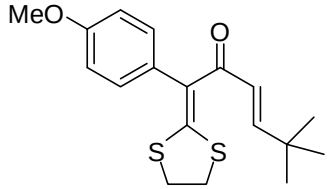
389.0 $[(M+1)]^+$. Anal. Calcd for $C_{20}H_{17}ClO_2S_2$: C, 61.76; H, 4.41; Found: C, 61.94; H, 4.30.

(*E*)-1-(1,3-Dithiolan-2-ylidene)-1-(4-methoxyphenyl)-4-(4-nitrophenyl)but-3-en-2-one (1f)



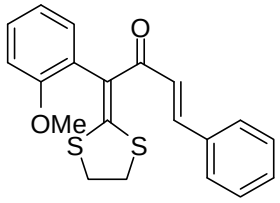
Yellow crystal; mp 168−170 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.27 (t, *J* = 6.0 Hz, 2H), 3.51 (t, *J* = 6.0 Hz, 2H), 3.87 (s, 3H), 6.72 (d, *J* = 16.0 Hz, 1H), 6.98 (d, *J* = 8.5 Hz, 2H), 7.21 (d, *J* = 8.5 Hz, 2H), 7.45 (d, *J* = 8.5 Hz, 2H), 7.63 (d, *J* = 15.5 Hz, 1H), 8.12 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 183.5, 169.2, 159.7, 148.2, 141.9, 139.3, 131.7, 131.6, 128.9 (2C), 127.7, 127.0, 126.7, 124.2, 123.8, 114.6 (2C), 55.5, 40.2, 36.1. IR (KBr): 3044, 2833, 1582, 1510, 1443, 1334, 1281, 1243 cm^{-1}. MS (ESI): *m/z* = 400.0 $[(M+1)]^+$. Anal. Calcd for $C_{20}H_{17}NO_4S_2$: C, 60.13; H, 4.29; N, 3.51; Found: C, 60.33; H, 4.20; N, 3.56.

(*E*)-1-(1,3-Dithiolan-2-ylidene)-1-(4-methoxyphenyl)-5,5-dimethylhex-3-en-2-one (1g)



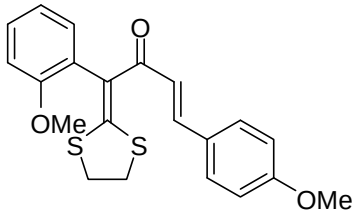
Light yellow crystal; mp 140−142 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 0.93 (s, 9H), 3.23 (t, *J* = 6.5 Hz, 2H), 3.47 (t, *J* = 6.5 Hz, 2H), 3.86 (s, 3H), 5.91 (d, *J* = 15.5 Hz, 1H), 6.91 (d, *J* = 15.5 Hz, 1H), 6.95 (d, *J* = 8.5 Hz, 2H), 7.17 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 185.3, 165.6, 159.1, 157.0, 132.1, 131.2 (2C), 126.8, 121.8, 114.1 (2C), 55.2, 39.7, 35.5, 33.7, 28.6 (3C). IR (KBr): 3038, 2965, 1642, 1585, 1513, 1445, 1324, 1247 cm^{-1}. MS (ESI): *m/z* = 335.0 [(M+1)]+. Anal. Calcd for $C_{18}H_{22}O_2S_2$: C, 64.63; H, 6.63; Found: C, 64.90; H, 6.52.

(*E*)-1-(1,3-Dithiolan-2-ylidene)-1-(2-methoxyphenyl)-4-phenylbut-3-en-2-one (1h)



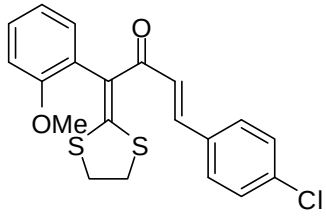
Light yellow crystal; mp 102−104 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.24 (t, *J* = 6.0 Hz, 2H), 3.48 (t, *J* = 6.0 Hz, 2H), 3.75 (s, 3H), 6.57 (d, *J* = 15.5 Hz, 1H), 7.00 (d, *J* = 8.5 Hz, 1H), 7.04 (t, *J* = 7.0 Hz, 1H), 7.27 (t, *J* = 7.0 Hz, 4H), 7.32 (t, *J* = 7.0 Hz, 2H), 7.40−7.44 (m, 1H), 7.67 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 184.4, 165.6, 157.2, 141.8, 135.5, 131.8 (2C), 130.0, 129.6, 128.6 (2C), 128.3, 128.0 (2C), 123.5, 120.9, 111.3, 55.6, 39.7, 35.5. IR (KBr): 3025, 2900, 1639, 1584, 1445, 1332, 1245 cm^{-1}. MS (ESI): *m/z* = 355.0 $[(M+1)]^+$. Anal. Calcd for $C_{20}H_{18}O_2S_2$: C, 67.76; H, 5.12; Found: C, 67.96; H, 5.03.

(*E*)-1-(1,3-Dithiolan-2-ylidene)-1-(2-methoxyphenyl)-4-(4-methoxyphenyl)but-3-en-2-one (1i)



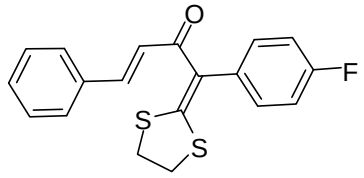
Light yellow crystal; mp 150−152 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.20–3.23 (m, 2H), 3.46–3.48 (m, 2H), 3.74 (s, 3H), 3.76 (s, 3H), 6.45 (d, *J* = 15.5 Hz, 1H), 6.79 (d, *J* = 6.5 Hz, 2H), 7.00–7.05 (m, 2H), 7.24–7.28 (m, 3H), 7.40–7.42 (m, 1H), 7.64 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 184.4, 164.8, 160.8, 141.5, 131.7 (2C), 129.9, 129.6 (2C), 128.4, 128.1, 123.5, 121.2, 120.8, 114.0 (2C), 111.2, 55.6, 55.2, 39.6, 35.4. IR (KBr): 3054, 2932, 2832, 1637, 1573, 1460, 1244, 1140 cm^{-1}. MS (ESI): *m/z* = 385.0 $[(M+1)]^+$. Anal. Calcd for $C_{21}H_{20}O_3S_2$: C, 65.60; H, 5.24; Found: C, 67.81; H, 5.15.

(*E*)-4-(4-Chlorophenyl)-1-(1,3-dithiolan-2-ylidene)-1-(2-methoxyphenyl)but-3-en-2-one (1j)



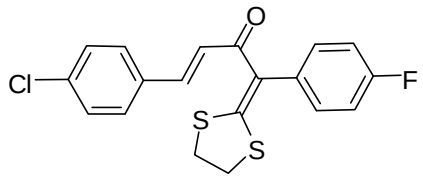
Light yellow crystal; mp 117−119 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.25 (t, *J* = 6.0 Hz, 2H), 3.49 (t, *J* = 6.0 Hz, 2H), 3.75 (s, 3H), 6.53 (d, *J* = 16.0 Hz, 1H), 7.00 (d, *J* = 8.0 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 7.23–7.26 (m, 5H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.60 (d, *J* = 16.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 184.0, 166.1, 157.1, 140.3, 135.3, 134.0, 131.8, 130.1, 129.1 (2C), 128.8 (2C), 128.1, 124.0, 123.4, 120.9, 111.3, 55.6, 39.7, 35.5. IR (KBr): 3063, 2938, 1639, 1584, 1445, 1245 cm^{-1}. MS (ESI): *m/z* = 389.0 $[(M+1)]^+$. Anal. Calcd for $C_{20}H_{17}ClO_2S_2$: C, 61.76; H, 4.41; Found: C, 62.02; H, 4.33.

(*E*)-1-(1,3-Dithiolan-2-ylidene)-1-(4-fluorophenyl)-4-phenylbut-3-en-2-one (1k)



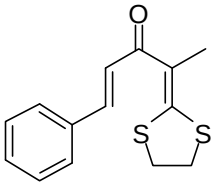
Yellow crystal; mp 174−175°C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.25 (t, *J* = 6.5 Hz, 2H), 3.48 (t, *J* = 6.5 Hz, 2H), 6.55 (d, *J* = 15.5 Hz, 1H), 7.14 (t, *J* = 8.5 Hz, 2H), 7.29−7.33 (m, 7H), 7.66 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 183.8, 167.2, 162.4 (d, *J* = 247.0 Hz, 1C), 142.6, 135.5, 135.1, 132.0, 131.9, 129.9, 128.7 (2C), 128.1 (2C), 126.2, 123.2, 116.0 (d, *J* = 20.2 Hz, 2C), 39.7, 35.7. IR (KBr): 3052, 2913, 1637, 1574, 1445, 1246, 1161 cm^{-1}. MS (ESI): *m/z* = 343.1 $[(M + 1)]^+$. Anal. Calcd for $C_{19}H_{15}FOS_2$: C, 66.64; H, 4.41; Found: C, 66.82; H, 4.30.

(*E*)-4-(4-Chlorophenyl)-1-(1,3-dithiolan-2-ylidene)-1-(4-fluorophenyl)but-3-en-2-one (1*l*)



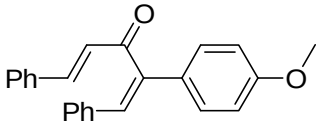
Yellow crystal; mp 180−181°C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.27 (t, *J* = 6.0 Hz, 2H), 3.49 (t, *J* = 6.0 Hz, 2H), 6.51 (d, *J* = 15.5 Hz, 1H), 7.14 (t, *J* = 8.5 Hz, 2H), 7.28−7.29 (m, 6H), 7.59 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 183.5, 167.7, 162.4 (d, *J* = 247.0 Hz, 1C), 141.1, 135.7, 135.4, 133.7, 132.0, 131.9, 129.3 (2C), 128.9 (2C), 126.1, 123.6, 116.3 (d, *J* = 20.5 Hz, 2C), 39.7, 35.8. IR (KBr): 3055, 1597, 1445, 1336, 1161, 734 cm^{-1}. MS (ESI): *m/z* = 377.0 $[(M + 1)]^+$. Anal. Calcd for $C_{19}H_{14}ClFOS_2$: C, 60.55; H, 3.74; Found: C, 60.33; H, 3.91.

(*E*)-4-(1,3-Dithiolan-2-ylidene)-1-phenylpent-1-en-3-one (1m)



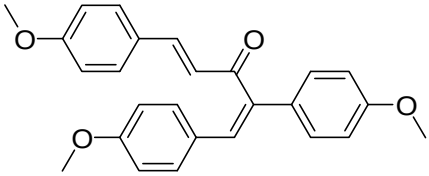
Yellow crystal; mp 98−100 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 2.34 (s, 3H), 3.35−3.43 (m, 4H), 7.23 (d, *J* = 15.5 Hz, 1H), 7.37−7.40 (m, 3H), 7.57−7.59 (m, 2H), 7.72 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 185.5, 161.5, 143.1, 135.4, 129.9, 128.8 (2C), 128.2 (2C), 122.2, 119.9, 39.4, 35.8, 19.4. IR (KBr): 3061, 2930, 1643, 1577, 1479, 1230, 675 cm^{-1}. MS (ESI): *m/z* = 263.0 $[(M+1)]^+$. Anal. Calcd for: $C_{14}H_{14}OS_2$: C, 64.08; H, 5.83; Found: C, 64.22; H, 5.76.

(1*Z*,4*E*)-2-(4-Methoxyphenyl)-1,5-diphenylpenta-1,4-dien-3-one (1′a)



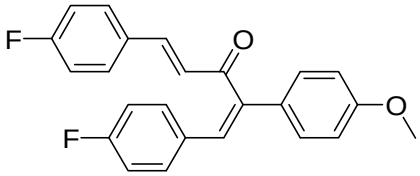
Yellow crystal; mp 136−138 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.87 (s, 3H), 6.94 (d, *J* = 7.0 Hz, 2H), 6.97 (s, 1H), 7.13−7.23 (m, 7H), 7.34−7.35 (m, 3H), 7.45−7.47 (m, 2H), 7.69 (s, 1H), 7.72 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 191.3, 159.3, 143.6, 140.8, 138.3, 135.1, 134.9, 131.0 (2C), 130.8 (2C), 130.3, 129.0 (2C), 128.8 (2C), 128.4 (2C), 128.2 (2C), 123.3, 114.5 (2C), 55.2. IR (KBr): 3064, 2921, 1658, 1610, 1408, 1246, 690 cm^{-1}. MS (ESI): *m/z* = 341.0 $[(M+1)]^+$. Anal. Calcd for: $C_{24}H_{20}O_2$: C, 84.68; H, 5.92; Found: C, 84.80; H, 5.78.

(1*Z*,4*E*)-1,2,5-Tris(4-methoxyphenyl)penta-1,4-dien-3-one (1′b)



Yellow crystal; mp 38−40 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.76 (s, 3H), 3.81 (s, 3H), 3.88 (s, 3H), 6.70 (d, *J* = 9.0 Hz, 2H), 6.79 (d, *J* = 15.5 Hz, 1H), 6.84 (d, *J* = 8.5 Hz, 2H), 6.97 (d, *J* = 8.5 Hz, 2H), 7.07 (d, *J* = 8.5 Hz, 2H), 7.16 (d, *J* = 8.5 Hz, 2H), 7.40 (d, *J* = 8.5 Hz, 2H), 7.68 (t, *J* = 11.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 190.9, 161.3, 160.1, 159.2, 143.1, 138.9, 138.0, 132.6 (2C), 131.1 (2C), 131.2, 130.1 (2C), 129.5, 127.9, 121.2, 114.6 (2C), 114.2 (2C), 113.7 (2C), 55.3, 55.2 (2C). IR (KBr): 3065, 2935, 2837, 1649, 1510, 1304, 1175, 831 cm^{-1}. MS (ESI): *m/z* = 401.0 $[(M+1)]^+$. Anal. Calcd for: $C_{26}H_{24}O_4$: C, 77.98; H, 6.04; Found: C, 78.09; H, 6.09.

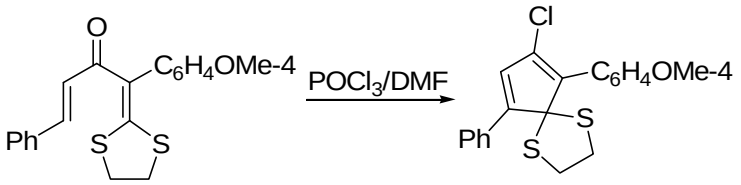
(1*Z*,4*E*)-1,5-Bis(4-fluorophenyl)-2-(4-methoxyphenyl)penta-1,4-dien-3-one (1′c)



Yellow crystal; mp 108−110 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.88 (s, 3H), 6.82 (d, *J* = 15.5 Hz, 1H), 6.88 (t, *J* = 9.0 Hz, 2H), 6.97 (d, *J* = 8.5 Hz, 2H), 7.03 (t, *J* = 9.0 Hz, 2H), 7.09−7.16 (m, 4H), 7.42−7.45 (m, 2H), 7.65 (s, 1H), 7.69 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 190.8, 163.0 (d, *J* = 150 Hz, 1C), 162.0 (d, *J* = 150 Hz, 1C), 159.5, 142.4, 140.5, 137.1, 132.8, 132.7, 131.2, 131.0 (2C), 130.3 (2C), 130.2, 128.5, 122.9, 115.0 (d, *J* = 21.5 Hz, 2C), 115.3 (d, *J* = 21.5 Hz, 2C), 114.8 (2C), 55.3. IR (KBr): 3016, 2965, 1611, 1506, 1175, 832 cm^{-1}. MS (ESI): *m/z* = 377.0 $[(M+1)]^+$. Anal. Calcd for: $C_{24}H_{18}F_2O_2$: C, 76.58; H, 4.82; Found: C, 76.42; H, 5.01.

III. Synthetic procedures/analytical data of compounds 2 and III.

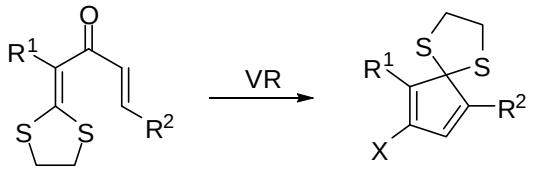
Optimization of the reaction conditions.



1a **2a**

Entry[a]	DMF (mL)	$POCl_3$ (equiv)	t (h)	*T* (ºC)	Yield (%)[b]
1	10	1.0	12	rt	NR[c]
2	10	2.0	12	rt	10[d]
3	10	2.0	5.0	60	47
4	5.0	2.0	4.0	60	61
5	5.0	2.0	3.0	90	83
6	***5.0***	***2.5***	***2.0***	***90***	***86***
7	5.0	3.0	2.0	90	78
8	5.0	2.5	2.0	100	74

[a] Reagent: **1a** (1.0 mmol). [b] Isolated yield. [c] No reaction. [d] **1a** was recovered in 81% yield.



1 **2**

General procedure for the preparation of cyclopentadienes 2 and III′d (taking 2a as an example): To a well-stirred solution of **1a** (354 mg, 1.0 mmol) in DMF (5.0 mL) was added $POCl_3$ (0.23 mL, 2.5 mmol) in one portion at room temperature. Then, the reaction mixture was heated to 90°C and stirred for 2.5 h. After **1a** was consumed (monitored by TLC), the reaction mixture was poured into water (30 mL), neutralized with saturated aqueous $NaHCO_3$ to pH 7, and extracted with CH_2Cl_2 (15 mL × 3). The combined organic extracts were washed with water (15 mL × 3), dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure to yield the crude product, which was purified by silica gel chromatography (eluent, petroleum ether/diethyl ether: 75/1, v/v) to give **2a** (320 mg, 86%) as a light yellow crystal.

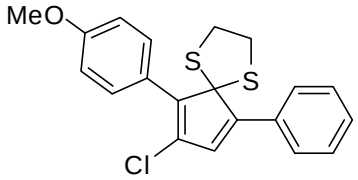
Procedure for the detection of IIIa: The reaction of **1a** (340 mg, 1.0 mmol) and $POCl_3$ (0.23 mL, 2.5 mmol) in DMF (5.0 mL) was allowed at room temperature for 1.0 h. Then, the reaction mixture was poured into water (30 mL), neutralized with saturated aqueous $NaHCO_3$ to pH 7, and extracted with CH_2Cl_2 (15 mL × 3). The combined organic extracts were washed with water (15 mL × 3), dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by silica gel chromatography (eluent, petroleum ether/diethyl ether: 75/1, v/v) to give a mixture of **2a** and **IIIa** in 21% yield with the ratio of 1 to 2 based on ^{1}H NMR (**2a** and **IIIa** has the same polarity on silica gel chromatography).

General procedure for the preparation of cyclopentadienes 2′ and 2″ (taking 2′a and 2″a as example): To a well-stirred solution of **1′a** (340 mg, 1.0 mmol) in DMF (5.0 mL) was added $POCl_3$ (0.23 mL, 2.5 mmol) in one portion at room temperature. Then, the reaction mixture was stirred at room temperature for 7.0 h. After **1′a** was consumed (monitored by TLC), the reaction mixture was poured into water (30 mL), neutralized with saturated aqueous $NaHCO_3$ to pH 7, and extracted with CH_2Cl_2 (15 mL × 3). The combined organic extracts were washed with water (15 mL × 3), dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure to yield the crude product, which was purified by silica gel chromatography (eluent, petroleum ether/diethyl ether: 100/1, v/v)

to give **2′a** (272 mg, 76%) and **2′′a** (18 mg, 5.0%), respectively.

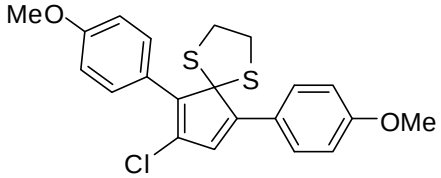
Synthesis of cyclopentenone III′a and its chlorovinylation under Vilsmeier conditions: A solution of **1′a** (340 mg, 1.0 mmol), concentrated HCl (0.25 mL, M = 12 mol L^{-1}, 3.0 mmol) in CH_2Cl_2 (5.0 mL) was stirred at room temperature for 4.0 h. The reaction mixture was diluted with water (30 mL), neutralized with saturated aqueous $NaHCO_3$ to pH 7, and extracted with CH_2Cl_2 (10 mL × 3). The combined organic extracts were washed with water (15 mL), dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure to yield the crude product, which was purified by silica gel chromatography (eluent, petroleum ether/diethyl ether: 10/1, v/v) to give **III′a** (265 mg, 78%) as a white crystal. Then, to a well-stirred solution of **III′a** (170 mg, 0.5 mmol) in DMF (2.5 mL) was added $POCl_3$ (0.12 mL, 1.3 mmol) in one portion at room temperature. After the reaction mixture was stirred at room temperature for 1.5 h monitored by TLC, water (15 mL) was added. The resulting aqueous mixture was neutralized with saturated aqueous $NaHCO_3$ to PH 7 and extracted with CH_2Cl_2 (10 mL × 3). The combined organic extracts were washed with water (15 mL × 3), dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure to yield the crude product, which was purified by silica gel chromatography (eluent, petroleum ether/diethyl ether: 100/1, v/v) to give **2′a** (70 mg, 39%) and **2′′a** (72 mg, 40%), respectively.

7-Chloro-6-(4-methoxyphenyl)-9-phenyl-1,4-dithiaspiro[4.4]nona-6,8-diene (2a)



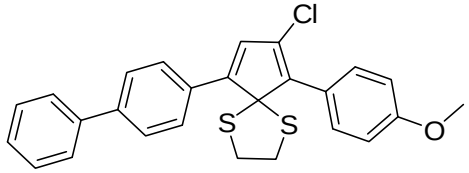
Light yellow crystal; mp 134–136 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.05–3.10 (m, 2H), 3.27–3.32 (m, 2H), 3.86 (s, 3H), 6.60 (s, 1H), 6.94 (d, *J* = 8.5 Hz, 2H), 7.31–7.39 (m, 3H), 7.56 (d, *J* = 8.5 Hz, 2H), 7.81 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 159.5, 148.1, 147.0, 133.4, 131.8 (2C), 128.9, 128.2, 128.1 (2C), 127.8 (2C), 127.7, 125.4, 113.5 (2C), 74.6, 55.2, 41.6 (2C). IR (KBr): 3059, 2960, 1597, 1501, 1246, 1173 cm^{-1}. MS (ESI): *m/z* = 373.0 $[(M+1)]^+$. Anal. Calcd for $C_{20}H_{17}ClOS_2$: C, 64.41; H, 4.59; Found: C, 64.68; H, 4.48.

7-Chloro-6,9-bis(4-methoxyphenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2b)



Light yellow crystal; mp 144–146 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.06−3.10 (m, *J* = 5.0 Hz, 2H), 3.30−3.36 (m, 2H), 3.85 (s, 3H), 3.86 (s, 3H), 6.54 (s, 1H), 6.93 (d, *J* = 8.5 Hz, 2H), 6.96 (d, *J* = 8.0 Hz, 2H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.79 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 159.7, 159.7, 147.8, 146.5, 132.1 (2C), 129.2 (2C), 128.1, 127.7, 126.3, 125.7, 113.7 (2C), 113.6 (2C), 74.8, 55.5, 55.4, 41.8 (2C). IR (KBr): 3004, 2956, 2833, 1601, 1504, 1245, 1175 cm^{-1}. MS (ESI): *m/z* = 403.0 $[(M+1)]^+$. Anal. Calcd for $C_{21}H_{19}ClO_2S_2$: C, 62.59; H, 4.75; Found: C, 62.79; H, 4.64.

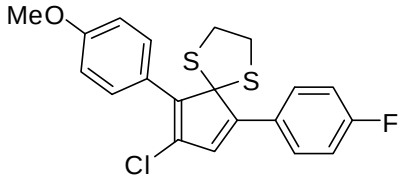
9-(Biphenyl-4-yl)-7-chloro-6-(4-methoxyphenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2c)



Yellow crystal; mp 173−174 °C; ^{1}H NMR (DMSO, 500 MHz) δ = 3.08 (t, *J* = 5.0 Hz, 2H), 3.52 (t, *J*

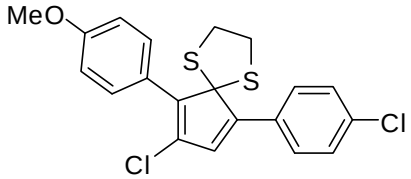
= 5.0 Hz, 2H), 3.80 (s, 3H), 7.01 (d, *J* = 8.5 Hz, 2H), 7.13 (s, 1H), 7.38 (m, 2H), 7.46−7.49 (m, 3H), 7.69−7.71 (m, 4H), 7.98 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (DMSO, 125 MHz) δ = 164.4, 153.6, 151.5, 144.7, 144.6, 136.8 (2C), 136.4, 134.2 (2C), 133.9, 132.9 (2C), 132.8, 131.8 (2C), 131.7, 131.4 (2C), 129.8, 118.7 (2C), 78.7, 60.3, 46.7 (2C). IR (KBr): 3062, 2919, 1647, 1407, 1172, 720 cm^{-1}. MS (ESI): *m/z* = 449.0 [(M+1)]$^+$. Anal. Calcd for $C_{26}H_{21}ClOS_2$: C, 69.55; H, 4.71; Found: C, 69.35; H, 4.80.

7-Chloro-9-(4-fluorophenyl)-6-(4-methoxyphenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2d)



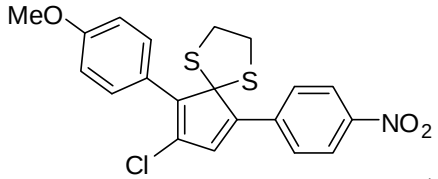
Light yellow crystal; mp 146–148 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.05−3.10 (m, 2H), 3.27−3.32 (m, 2H), 3.85 (s, 3H), 6.55 (s, 1H), 6.95 (d, *J* = 8.5 Hz, 2H), 7.06 (t, *J* = 8.5 Hz, 2H), 7.56 (d, *J* = 9.0 Hz, 2H), 7.78–7.81 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 162.6 (d, *J* = 247.0 Hz, 1C), 159.5, 147.0, 146.7, 131.7 (2C), 129.6 (2C), 129.5, 128.8, 127.6, 125.2, 115.0 (d, *J* = 20.9 Hz, 2C), 113.4 (2C), 74.7, 55.1, 41.4 (2C). IR (KBr): 3065, 2833, 1617, 1502, 1292, 1246 cm^{-1}. MS (ESI): *m/z* = 391.0 [(M+1)]$^+$. Anal. Calcd for $C_{20}H_{16}ClFOS_2$: C, 61.45; H, 4.13; Found: C, 61.66; H, 4.02.

7-Chloro-9-(4-chlorophenyl)-6-(4-methoxyphenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2e)



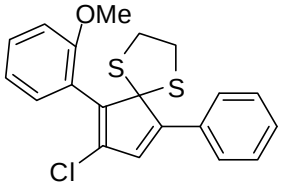
Light yellow crystal; mp 145–147 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.05−3.09 (m, 2H), 3.30−3.34 (m, 2H), 3.86 (s, 3H), 6.62 (s, 1H), 6.96 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.56 (t, *J* = 8.5 Hz, 2H), 7.77 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 159.8, 147.7, 146.8, 134.2, 132.0 (2C), 130.1, 129.6, 129.2 (2C), 128.9, 128.5 (2C), 125.4, 113.7 (2C), 74.8, 55.5, 41.8 (2C). IR (KBr): 3038, 2956, 1598, 1502, 1245, 1230 cm^{-1}. MS (ESI): *m/z* = 407.0 [(M+1)]$^+$. Anal. Calcd for $C_{20}H_{16}Cl_2OS_2$: C, 58.97; H, 3.96; Found: C, 59.19; H, 3.87.

7-Chloro-6-(4-methoxyphenyl)-9-(4-nitrophenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2f)



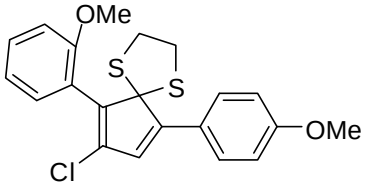
Yellow crystal; mp 193–195 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.07−3.12 (m, 2H), 3.35−3.41 (m, 2H), 3.86 (s, 3H), 6.88 (s, 1H), 6.97 (d, *J* = 8.5 Hz, 2H), 7.54 (d, *J* = 8.5 Hz, 2H), 8.00 (d, *J* = 8.5 Hz, 2H), 8.23 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 160.1, 150.4, 147.1, 145.0, 139.4, 132.8, 131.9 (2C), 128.2 (2C), 127.8, 124.9, 123.5 (2C), 113.8 (2C), 74.5, 55.5, 41.8 (2C). IR (KBr): 3062, 2834, 1592, 1505, 1343, 1241 cm^{-1}. MS (ESI): *m/z* = 418.0 [(M+1)]$^+$. Anal. Calcd for $C_{20}H_{16}ClNO_3S_2$: C, 57.48; H, 3.86; N, 3.35; Found: C, 57.68; H, 3.75; N, 3.40.

7-Chloro-6-(2-methoxyphenyl)-9-phenyl-1,4-dithiaspiro[4.4]nona-6,8-diene (2g)



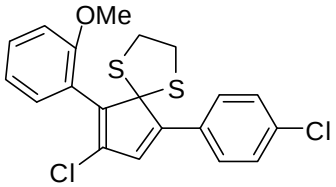
Light yellow crystal; mp 122−124 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.04−3.08 (m, 2H), 3.30 (s, broad, 2H), 3.84 (s, 3H), 6.59 (s, 1H), 7.00 (t, *J* = 7.0 Hz, 2H), 7.33 (t, *J* = 7.0 Hz, 1H), 7.38−7.41 (m, 3H), 7.71 (d, *J* = 7.0 Hz, 1H), 7.82 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 158.3, 149.2, 143.2, 133.8, 131.4, 129.8, 129.5, 128.8, 128.7, 128.0 (2C), 127.8, 127.7, 122.0, 120.0, 111.4, 74.9, 55.9, 41.6 (2C). IR (KBr): 3057, 3009, 2921, 2834, 1487, 1258, 1022 cm^{-1}. MS (ESI): *m/z* = 373.0 $[(M+1)]^+$. Anal. Calcd for $C_{20}H_{17}ClOS_2$: C, 64.41; H, 4.59; Found: C, 64.65; H, 4.50.

7-Chloro-6-(2-methoxyphenyl)-9-(4-methoxyphenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2h)



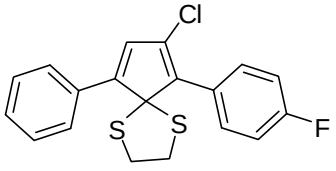
Light yellow crystal; mp 153−155 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.03−3.07 (m, 2H), 3.29 (s, broad, 2H), 3.83 (s, 6H), 6.50 (s, 1H), 6.90 (t, *J* = 9.0 Hz, 2H), 6.98 (t, *J* = 8.0 Hz, 2H), 7.35–7.38 (m, 1H), 7.69–7.71 (m, 1H), 7.77 (d, *J* = 9.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 159.4, 158.3, 148.6, 142.5, 131.5, 129.8, 129.5 (2C), 128.9, 127.3, 126.4, 122.1, 120.0, 113.4 (2C), 111.4, 74.9, 56.0, 55.2, 41.5 (2C). IR (KBr): 3063, 2957, 2930, 2832, 1597, 1504, 1252 cm^{-1}. MS (ESI): *m/z* = 403.0 $[(M+1)]^+$. Anal. Calcd for $C_{21}H_{19}ClO_2S_2$: C, 62.59; H, 4.75; Found: C, 62.88; H, 4.70.

7-Chloro-9-(4-chlorophenyl)-6-(2-methoxyphenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2i)



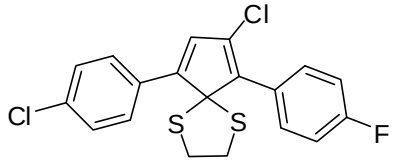
Light yellow crystal; mp 104−106 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.03−3.08 (m, 2H), 3.29 (s, broad, 2H), 3.83 (s, 3H), 6.59 (s, 1H), 6.99 (d, *J* = 7.5 Hz, 2H), 7.34 (d, *J* = 9.0 Hz, 2H), 7.37 (dd, *J* = 2.0, 8.0 Hz, 1H), 7.69 (dd, *J* = 1.5, 7.5 Hz, 1H), 7.76 (d, *J* = 9.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 158.2, 147.6, 143.8, 133.8, 132.1, 131.3, 129.9, 129.4, 129.2, 128.9 (2C), 128.2 (2C), 121.8, 120.0, 111.4, 74.8, 55.9, 41.6 (2C). IR (KBr): 3053, 3006, 2964, 2927, 2868, 2831, 1487, 1255 cm^{-1}. MS (ESI): *m/z* = 407.0 $[(M+1)]^+$. Anal. Calcd for $C_{20}H_{16}Cl_2OS_2$: C, 58.97; H, 3.96; Found: C, 59.26; H, 3.85.

7-Chloro-6-(4-fluorophenyl)-9-phenyl-1,4-dithiaspiro[4.4]nona-6,8-diene (2j)



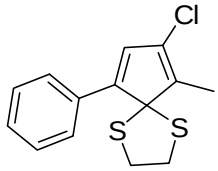
Yellow crystal; mp 121−122°C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.02−3.06 (m, 2H), 3.30−3.34 (m, 2H), 6.59 (s, 1H), 7.09 (t, *J* = 8.5 Hz, 2H), 7.34−7.40 (m, 3H), 7.59−7.62 (m, 2H), 7.81 (d, *J* = 7.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 162.7 (d, *J* = 247.0 Hz, 1C), 148.7, 145.9, 138.3, 133.3, 132.5, 131.8, 129.0, 128.9, 128.7, 128.2, 128.1, 127.7, 127.6, 115.0 (d, *J* = 20.0 Hz, 2C), 74.6, 41.6 (2C). IR (KBr): 3056, 2957, 1770, 1501, 1261, 1183, 835 cm^{-1}. MS (ESI): *m/z* = 361.0 $[(M + 1)]^+$. Anal. Calcd for $C_{19}H_{14}ClFS_2$: C, 63.23; H, 3.91; Found: C, 63.04; H, 3.79.

7-Chloro-9-(4-chlorophenyl)-6-(4-fluorophenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2k)



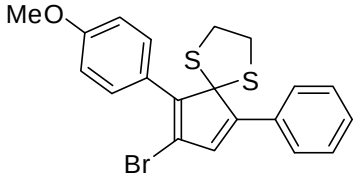
Yellow crystal; mp 210−211°C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.01−3.05 (m, 2H), 3.29−3.33 (m, 2H), 6.59 (s, 1H), 7.09 (t, *J* = 9.0 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.57−7.59 (m, 2H), 7.75 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 162.8 (d, *J* = 247.0 Hz, 1C), 147.2, 146.5, 134.1, 132.5, 132.4, 131.6, 129.2, 128.9, 128.8, 128.3 (2C), 128.2 (2C), 115.1 (d, *J* = 20.5 Hz, 2C), 74.5, 41.5 (2C). IR (KBr): 3065, 2962, 1697, 1500, 1292, 1091, 825 cm^{-1}. MS (ESI): *m/z* = 395.0 [(M + 1)]$^+$. Anal. Calcd for $C_{19}H_{13}Cl_2FS_2$: C, 57.72; H, 3.31; Found: C, 58.02; H, 3.45.

7-Chloro-6-methyl-9-phenyl-1,4-dithiaspiro[4.4]nona-6,8-diene (2*l*)



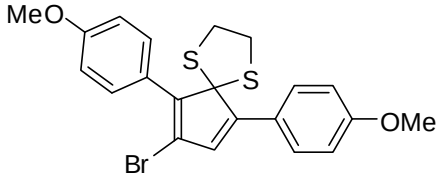
White crystal; mp 96−98 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 2.06 (s, 3H), 3.44−3.46 (m, 2H), 3.53−3.55 (m, 2H), 6.39 (s, 1H), 7.31−7.35 (m, 3H), 7.73−7.75 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 149.6, 144.0, 134.1, 128.5, 128.0, 127.9 (2C), 127.6 (2C), 125.5, 73.8, 42.0 (2C), 10.3. IR (KBr): 3058, 2959, 2916, 1693, 1516, 760 cm^{-1}. MS (ESI): *m/z* = 281.0 [(M+1)]$^+$. Anal. Calcd for $C_{14}H_{13}ClS_2$: C, 59.87; H, 4.67; Found: C, 59.71; H, 4.80.

7-Bromo-6-(4-methoxyphenyl)-9-phenyl-1,4-dithiaspiro[4.4]nona-6,8-diene (2m)



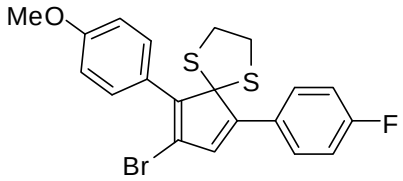
Light yellow crystal; mp 132−134 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.01−3.05 (m, 2H), 3.30−3.33 (m, 2H), 3.85 (s, 3H), 6.68 (s, 1H), 6.94 (d, *J* = 7.5 Hz, 2H), 7.32−7.39 (m, 3H), 7.53 (d, *J* = 7.0 Hz, 2H), 7.82 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 159.3, 150.8, 147.8, 133.1, 131.6 (2C), 130.5, 128.2 (2C), 127.9 (2C), 127.5, 126.2, 116.8, 113.2 (2C), 74.8, 55.0, 41.3 (2C). IR (KBr): 3056, 2925, 1601, 1501, 1288, 1247, 1173 cm^{-1}. MS (ESI): *m/z* = 417.0 [(M+1)]$^+$. Anal. Calcd for $C_{20}H_{17}BrOS_2$: C, 57.55; H, 4.11; Found: C, 57.76; H, 4.02.

7-Bromo-6,9-bis(4-methoxyphenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2n)



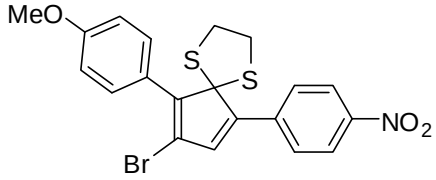
Light yellow crystal; mp 154−156 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.00−3.03 (m, 2H), 3.30−3.33 (m, 2H), 3.83 (s, 3H), 3.84 (s, 3H), 6.59 (s, 1H), 6.91 (d, *J* = 8.5 Hz, 2H), 6.94 (d, *J* = 8.5 Hz, 2H), 7.52 (d, *J* = 8.5 Hz, 2H), 7.77 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 159.3, 159.3, 150.1, 147.2, 131.7 (2C), 129.0, 128.7 (2C), 126.3, 125.7, 117.0, 113.3 (2C), 113.2 (2C), 74.7, 55.1, 55.0, 41.2 (2C). IR (KBr): 3060, 2925, 1604, 1503, 1243, 1176 cm^{-1}. MS (ESI): *m/z* = 447.1 [(M+1)]$^+$. Anal. Calcd for $C_{21}H_{19}BrO_2S_2$: C, 56.37; H, 4.28; Found: C, 56.59; H, 4.19.

7-Bromo-9-(4-fluorophenyl)-6-(4-methoxyphenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2o)



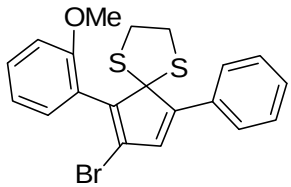
Light yellow crystal; mp 128−130 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.00−3.04 (m, 2H), 3.27−3.30 (m, 2H), 3.85 (s, 3H), 6.61 (s, 1H), 6.95 (d, *J* = 9.0 Hz, 2H), 7.05 (t, *J* = 8.5 Hz, 2H), 7.51 (d, *J* = 8.5 Hz, 2H), 7.78−7.81 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 162.5 (d, *J* = 247 Hz, 1C), 159.5, 150.8, 146.7, 131.7 (2C), 130.5, 129.4 (2C), 129.3, 126.1, 116.9, 115.0 (d, *J* = 21.5 Hz, 2C), 113.3 (2C), 74.8, 55.1, 41.4 (2C). IR (KBr): 3064, 2915, 1594, 1501, 1242, 1167 cm^{-1}. MS (ESI): *m/z* = 436.1 [(M+1)]$^+$. Anal. Calcd for $C_{20}H_{16}BrFOS_2$: C, 55.17; H, 3.70; Found: C, 55.38; H, 3.59.

7-Bromo-6-(4-methoxyphenyl)-9-(4-nitrophenyl)-1,4-dithiaspiro[4.4]nona-6,8-diene (2p)



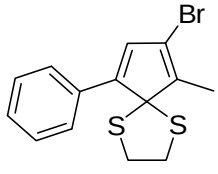
Yellow crystal; mp 198−200 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.03−3.06 (m, 2H), 3.35−3.39 (m, 2H), 3.86 (s, 3H), 6.93 (s, 1H), 6.96 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 8.00 (d, *J* = 8.0 Hz, 2H), 8.23 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 159.6, 153.8, 146.7, 144.6, 138.9, 134.3, 131.5 (2C), 127.7 (2C), 125.5, 123.1 (2C), 117.8, 113.3 (2C), 74.5, 55.0, 41.2 (2C). IR (KBr): 3048, 2925, 1590, 1505, 1341, 1282, 1242 cm^{-1}. MS (ESI): *m/z* = 462.0 [(M+1)]$^+$. Anal. Calcd for $C_{20}H_{16}BrNO_3S_2$: C, 51.95; H, 3.49; N, 3.03; Found: C, 52.18; H, 3.40; N, 3.07.

7-Bromo-6-(2-methoxyphenyl)-9-phenyl-1,4-dithiaspiro[4.4]nona-6,8-diene (2q)



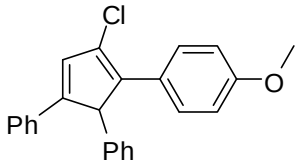
Light yellow crystal; mp 130−132 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 3.03−3.06 (m, 2H), 3.29 (s, broad, 2H), 3.84 (s, 3H), 6.65 (s, 1H), 6.99 (t, *J* = 7.5 Hz, 2H), 7.32 (t, *J* = 7.5 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 3H), 7.68 (d, *J* = 7.5 Hz, 1H), 7.82 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 158.0, 149.1, 147.2, 133.7, 131.4, 130.4, 129.8 (2C), 128.0 (2C), 127.7 (2C), 122.9, 120.0, 118.9, 111.4, 75.4, 55.9, 41.5 (2C). IR (KBr): 3051, 3006, 2921, 1485, 1430, 1258, 1118 cm^{-1}. MS (ESI): *m/z* = 417.1 [(M+1)]$^+$. Anal. Calcd for $C_{20}H_{17}BrOS_2$: C, 57.55; H, 4.11; Found: C, 57.86; H, 4.01.

7-Bromo-6-methyl-9-phenyl-1,4-dithiaspiro[4.4]nona-6,8-diene (2r)



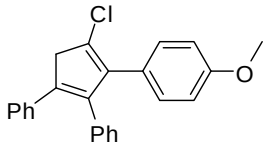
Yellow crystal; mp 58−60 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 2.07 (s, 3H), 3.45−3.47 (m, 2H), 3.53−3.56 (m, 2H), 6.44 (s, 1H), 7.33−7.36 (m, 3H), 7.73 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 149.8, 147.7, 134.1, 130.1, 128.0, 127.9 (2C), 127.7 (2C), 114.6, 74.1, 42.0 (2C), 12.0. IR (KBr): 3058, 2969, 1646, 1513, 1426, 676 cm^{-1}. MS (ESI): *m/z* = 325.0 [(M+1)]$^+$. Anal. Calcd for $C_{14}H_{13}BrS_2$: C, 51.69; H, 4.03; Found: C, 51.41; H, 4.20.

(4-Chloro-5-(4-methoxyphenyl)cyclopenta-2,4-diene-1,2-diyl)dibenzene (2′a)



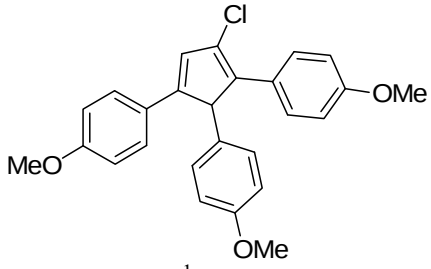
White crystal; mp 94−96 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.77 (s, 3H), 5.04 (s, 1H), 6.81−6.83 (m, 3H), 7.13−7.14 (m, 1H), 7.16−7.17 (m, 5H), 7.22−7.25 (m, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.50 (d, *J* = 9.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 158.6, 149.7, 143.3, 137.5, 133.9, 130.1, 129.3 (2C), 128.7 (2C), 128.4 (2C), 128.0 (2C), 127.4, 127.0, 126.8, 126.0, 125.8 (2C), 113.5 (2C), 59.0, 55.1. IR (KBr): 3064, 1647, 1407, 1174, 638 cm^{-1}. MS (ESI): *m/z* = 359.0 $[(M+1)]^+$. Anal. Calcd for: $C_{24}H_{19}ClO$: C, 80.33; H, 5.34; Found: C, 80.09; H, 5.22.

(4-Chloro-3-(4-methoxyphenyl)cyclopenta-1,3-diene-1,2-diyl)dibenzene (2″a)



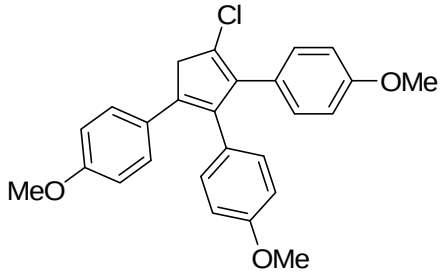
White crystal; mp 145−146 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.76 (s, 3H), 3.77 (s, 2H), 6.76 (t, *J* = 6.5 Hz, 2H), 6.96−7.00 (m, 4H), 7.09−7.19 (m, 8H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 158.6, 142.5, 142.4, 138.0, 135.9, 130.7 (2C), 129.8 (3C), 128.7, 128.2 (2C), 128.1 (2C), 127.8 (2C), 127.0, 126.6, 125.6, 113.2 (2C), 55.1, 47.9. IR (KBr): 3050, 2962, 1504, 1246, 1176, 780 cm^{-1}. MS (ESI): *m/z* = 359.0 $[(M+1)]^+$. Anal. Calcd for: $C_{24}H_{19}ClO$: C, 80.33; H, 5.34; Found: C, 80.20; H, 5.49.

4,4',4''-(4-Chlorocyclopenta-3,5-diene-1,2,3-triyl)tris(methoxybenzene) (2′b)



Colorless oil; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.67 (s, 3H), 3.75 (s, 3H), 3.76 (s, 3H), 4.92 (s, 1H), 6.65 (d, *J* = 7.5 Hz, 3H), 6.75 (d, *J* = 9.0 Hz, 2H), 6.79 (d, *J* = 9.0 Hz, 2H), 7.00 (d, *J* = 8.5 Hz, 2H), 7.31 (d, *J* = 9.0 Hz, 2H), 7.44 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 159.0, 158.5, 158.1, 142.6, 142.3, 130.9, 129.6, 129.3, 129.0, 128.8, 128.0, 127.2, 127.0 (2C), 126.9, 126.2, 114.1 (2C), 113.9 (2C), 113.5 (2C), 58.4, 55.2, 55.1, 55.0. IR (KBr): 3064, 2955, 1716, 1508, 1249, 1175, 1072 cm^{-1}. MS (ESI): *m/z* = 419.0 $[(M+1)]^+$. Anal. Calcd for: $C_{26}H_{23}ClO_3$: C, 74.55; H, 5.53; Found: C, 74.20; H, 5.69.

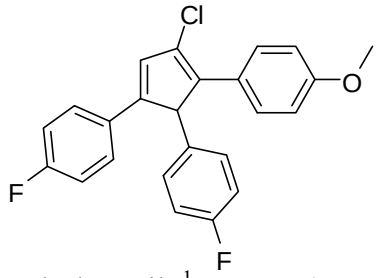
4,4',4''-(4-Chlorocyclopenta-1,3-diene-1,2,3-triyl)tris(methoxybenzene) (2″b)



Colorless oil; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.71 (s, 2H), 3.76 (s, 3H), 3.77 (s, 3H), 3.78 (s, 3H), 6.71 (d, *J* = 9.0 Hz, 2H), 6.72 (d, *J* = 8.5 Hz, 2H), 6.76 (d, *J* = 9.0 Hz, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 7.00 (d, *J* = 9.0 Hz, 2H), 7.06 (d, *J* = 9.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 158.5,

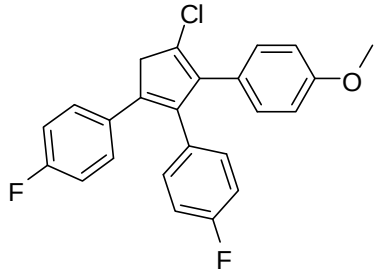
158.4, 158.2, 142.5, 140.6, 137.2, 131.0 (2C), 130.7 (2C), 130.5, 128.9 (2C), 128.4, 127.7, 125.9, 113.6 (4C), 113.1 (2C), 55.1 (3C), 47.8. IR (KBr): 3062, 2955, 2918, 1509, 1251, 1175, 831 cm^{-1}. MS (ESI): *m/z* = 419.0 $[(M+1)]^+$. Anal. Calcd for: $C_{26}H_{23}ClO_3$: C, 74.55; H, 5.53; Found: C, 74.32; H, 5.69.

4,4'-(4Chloro-5-(4-methoxyphenyl)cyclopenta-2,4-diene-1,2-diyl)bis(fluorobenzene) (2′c)



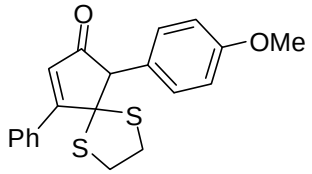
Colorless oil; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.75 (s, 3H), 4.95 (s, 1H), 6.72 (s, 1H), 6.79−6.82 (m, 4H), 6.92 (t, *J* = 8.5 Hz, 2H), 7.04 (dd, *J* = 5.5, 8.5 Hz, 2H), 7.31 (dd, *J* = 5.5, 8.5 Hz, 2H), 7.44 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 162.1 (d, *J* = 234.0 Hz, 1C), 161.4 (d, *J* = 244.1 Hz, 1C), 158.8, 148.6, 143.1, 133.0, 130.1, 129.9, 129.5, 129.4, 129.3 (2C), 127.5, 127.4, 127.0, 125.7, 115.6 (d, *J* = 22.0 Hz, 2C), 115.5 (d, *J* = 22.4 Hz, 2C), 113.7 (2C), 58.3, 55.1. IR (KBr): 3043, 2957, 2917, 1508, 1232, 1179, 834 cm^{-1}. MS (ESI): *m/z* = 395.0 $[(M+1)]^+$. Anal. Calcd for: $C_{24}H_{17}ClF_2O$: C, 73.01; H, 4.34; Found: C, 73.22; H, 4.20.

4,4'-(4-Chloro-3-(4-methoxyphenyl)cyclopenta-1,3-diene-1,2-diyl)bis(fluorobenzene) (2″c)



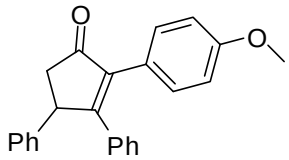
White crystal; mp 122−124 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.73 (s, 2H), 3.78 (s, 3H), 6.78 (d, *J* = 8.5 Hz, 2H), 6.85−6.91 (m, 6H), 6.98 (d, *J* = 6.5 Hz, 2H), 7.05 (dd, *J* = 5.5, 9.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 161.3 (d, *J* = 245.5 Hz, 1C), 159.4 (d, *J* = 276.1 Hz, 1C), 158.5, 142.0, 140.9, 137.0, 131.6, 131.2 (2C), 130.4 (2C), 129.2, 129.1 (2C), 128.5, 125.1, 115.1 (d, *J* = 21.0 Hz, 2C), 115.0 (d, *J* = 21.0 Hz, 2C), 113.0 (2C), 54.8, 47.7. IR (KBr): 3044, 2997, 2927, 1505, 1247, 1176, 1158, 831 cm^{-1}. MS (ESI): *m/z* = 395.0 $[(M+1)]^+$. Anal. Calcd for: $C_{24}H_{17}ClF_2O$: C, 73.01; H, 4.34; Found: C, 73.30; H, 4.51.

6-(4-Methoxyphenyl)-9-phenyl-1,4-dithiaspiro[4.4]non-8-en-7-one (IIIa)



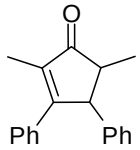
^{1}H NMR of a mixture of **IIIa** and **2a** ($CDCl_3$, 500 MHz) δ = 2.78–2.80 (m, 2H), 3.04–3.08 (m, 2H), 3.82 (s, 3H), 3.84 (s, 1H), 6.72 (s, 1H), 6.91 (d, *J* = 8.5 Hz, 2H), 7.43–7.46 (m, 5H), 7.65 (d, *J* = 8.5 Hz, 2H) for **IIIa** and δ = 3.02–3.09 (m, 2H), 3.30–3.32 (m, 2H), 3.86 (s, 3H), 6.59 (s, 1H), 6.93 (d, *J* = 8.5 Hz, 2H), 7.36–7.39 (m, 3H), 7.56 (d, *J* = 8.5 Hz, 2H), 7.81 (d, *J* = 7.0 Hz, 2H) for **2a**.

2-(4-Methoxyphenyl)-3,4-diphenylcyclopent-2-enone (III′a)



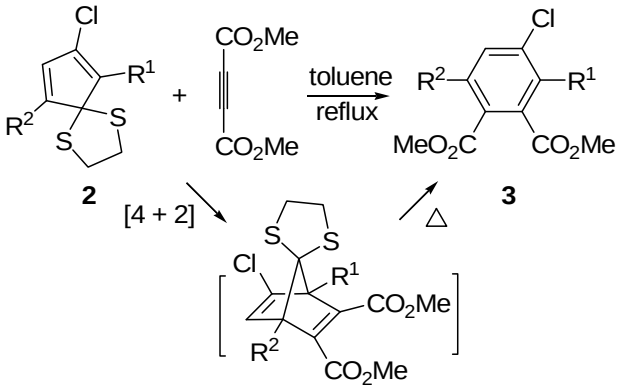
White crystal; mp 102−104 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 2.62 (dd, *J* = 2.0, 19.0 Hz, 1H), 3.22 (dd, *J* = 7.5, 19.0 Hz, 1H), 3.80 (s, 3H), 4.54 (dd, *J* = 2.0, 7.5 Hz, 1H), 6.86 (d, *J* = 8.5 Hz, 2H), 7.13−7.18 (m, 8H), 7.21−7.25 (m, 4H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 207.0, 169.2, 159.3, 142.3, 140.2, 135.1, 130.9 (2C), 129.1, 128.9 (2C), 128.7 (2C), 128.2 (2C), 127.4 (2C), 126.8, 123.9, 113.9 (2C), 55.2, 47.2, 45.9. IR (KBr): 3051, 2945, 2871, 1695, 1509, 1247, 1057, 732 cm^{-1}. MS (ESI): *m/z* = 341.0 $[(M+1)]^+$. Anal. Calcd for: $C_{24}H_{20}O_2$: C, 84.68; H, 5.92; Found : C, 83.84; H, 6.10.

2,5-Dimethyl-3,4-diphenylcyclopent-2-enone (III′d)



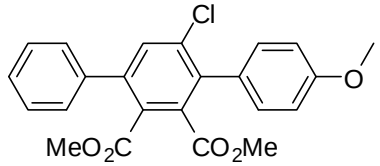
White crystal; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 1.35 (d, *J* = 7.5, 3H), 2.02 (s, 3H), 2.38–2.41 (m, 1H), 3.96–3.97 (m, 1H), 7.06 (d, *J* = 7.0, 2H), 7.12 (t, *J* = 7.0, 1H), 7.20 (t, *J* = 7.5, 2H), 7.24–7.32 (m, 5H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 211.0, 167.0, 142.0, 136.7, 135.1, 128.9, 128.7 (2C), 128.3 (2C), 128.2 (2C), 127.5 (2C), 126.6, 56.3, 51.2, 15.2, 10.1.

IV. Synthesis of polyaryls 3: [4 + 2] cycloaddition of 2 with dimethyl 2-butynedioate.



General procedure for the preparation of aryl halides 3 (taking 3a as an example): To a well-stirred solution of **2a** (372 mg, 1.0 mmol) in toluene (5.0 mL) was added dimethyl 2-butynedioate (0.61 mL, 5.0 mmol) in one portion at room temperature. Then, the reaction mixture was refluxed for 8.0 h. After **2a** was consumed (monitored by TLC), the reaction mixture was poured into water (20 mL) and extracted with CH_2Cl_2 (10 mL × 3). The combined organic extracts were dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure to yield the crude product, which was purified by silica gel chromatography (eluent, petroleum ether/diethyl ether: 10/1, v/v) to give **3a** (357 mg, 87%) as a white crystal.

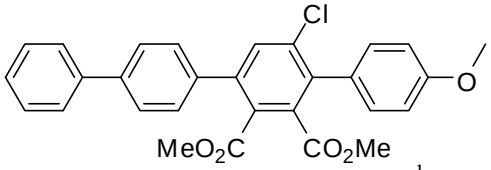
4-Methoxyl-2',3'-dimethoxycarbonyl-6'-chloro *p*-terphenyl (3a)



White crystal; mp 126−128 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.54 (s, 3H), 3.58 (s, 3H), 3.86 (s, 3H), 6.96 (d, *J* = 8.5 Hz, 2H), 7.22 (d, *J* = 8.5 Hz, 2H), 7.35−7.36 (m, 2H), 7.39−7.44 (m, 3H), 7.59

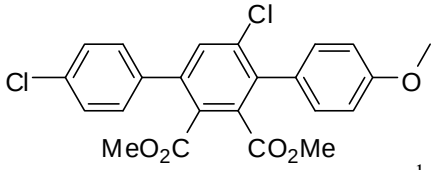
(s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 167.9, 167.6, 159.3, 141.3, 138.6, 137.4, 136.1, 135.5, 132.1, 130.4 (2C), 129.6, 128.4 (2C), 128.2 (2C), 128.0 (2C), 113.6, 113.4, 55.1, 52.4, 52.3. IR (KBr): 3065, 2946, 1743, 1718, 1251, 1020, 833 cm^{-1}. MS (ESI): *m/z* = 411.0 $[(M+1)]^+$. Anal. Calcd for: $C_{23}H_{19}ClO_5$: C, 67.24; H, 4.66; Found: C, 67.01, H, 4.52.

4-Methoxyl -2',3'-dimethoxycarbonyl-6'-chloro *p*-quaterphenyl (3b)



White crystal; mp 193−194 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.55 (s, 3H), 3.62 (s, 3H), 3.86 (s, 3H), 6.96 (d, *J* = 9.0 Hz, 2H), 7.23−7.26 (m, 2H), 7.42−7.44 (m, 1H), 7.45−7.48 (m, 4H), 7.64−7.68 (m, 5H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 168.0, 167.4, 159.4, 141.0, 140.9, 140.2, 137.6, 136.3, 135.7, 132.2, 130.5 (2C), 129.6, 128.9 (2C), 128.5 (2C), 128.3, 127.6 (2C), 127.2 (2C), 127.1 (2C), 113.5 (2C), 55.2, 52.2, 52.5. IR (KBr): 3062, 1742, 1647, 1242, cm^{-1}. MS (ESI): *m/z* = 487.0 $[(M+1)]^+$. Anal. Calcd for: $C_{29}H_{23}ClO_5$: C, 71.53; H, 4.76; Found: C, 72.41; H, 4.82.

4-Methoxyl-2',3'-dimethoxycarbonyl-6'-chloro-4''-chloro *p*-terphenyl (3c)



White crystal; mp 132−134 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 3.54 (s, 3H), 3.61 (s, 3H), 3.85 (s, 3H), 6.95 (d, *J* = 8.5 Hz, 2H), 7.21 (d, *J* = 8.5 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 2H), 7.39 (d, *J* = 8.5 Hz, 2H), 7.55 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 167.7, 167.5, 159.5, 140.1, 137.9, 137.1, 136.4, 135.8, 134.4, 132.0, 130.4, 129.5 (2C), 129.4 (2C), 128.7 (2C), 128.1, 113.5 (2C), 55.2, 52.6, 52.5. IR (KBr): 3059, 2955, 1751, 1518, 1306, 1028, 842 cm^{-1}. MS (ESI): *m/z* = 445.0 $[(M+1)]^+$. Anal. Calcd for: $C_{23}H_{18}Cl_2O_5$: C, 62.04; H, 4.07; Found: C, 62.21; H, 4.12.

V. Crystal data and ORTEP drawing of compound 2f.

$C_{20}H_{16}ClNO_3S_2$, yellow, *M* = 417.91, monoclinic, space group *P*21/c, *a* = 6.808(2), *b* = 16.045(5), *c* = 18.120(6) Å, *V* = 1967.4(11) Å^3, α = 90.00, β = 96.286(6), γ = 90.00, Z = 4, *T* = 273(2) K, *F*000 = 864, 10854 reflections collected, 3885 unique with *R*(int) = 0.0338, R_1 = 0.0603, wR_2 = 0.1416 ($I > 2\sigma(I)$).

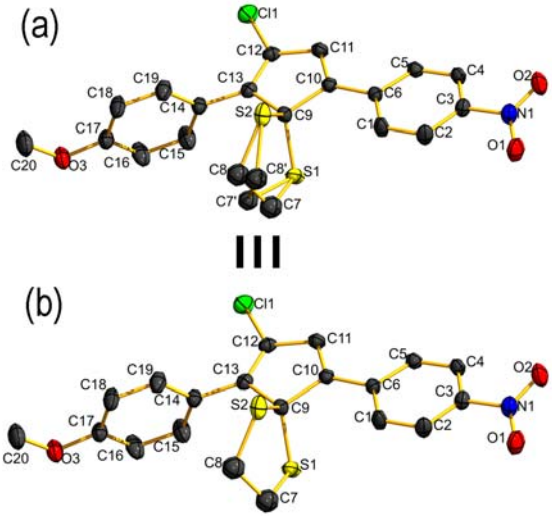


Fig. 1 (a) and (b) ORTEP diagram of **2f** (30% probability displacement ellipsoids and all hydrogen atoms are omitted, distorted C7′ and C8′ showing in (a)).

VI. References

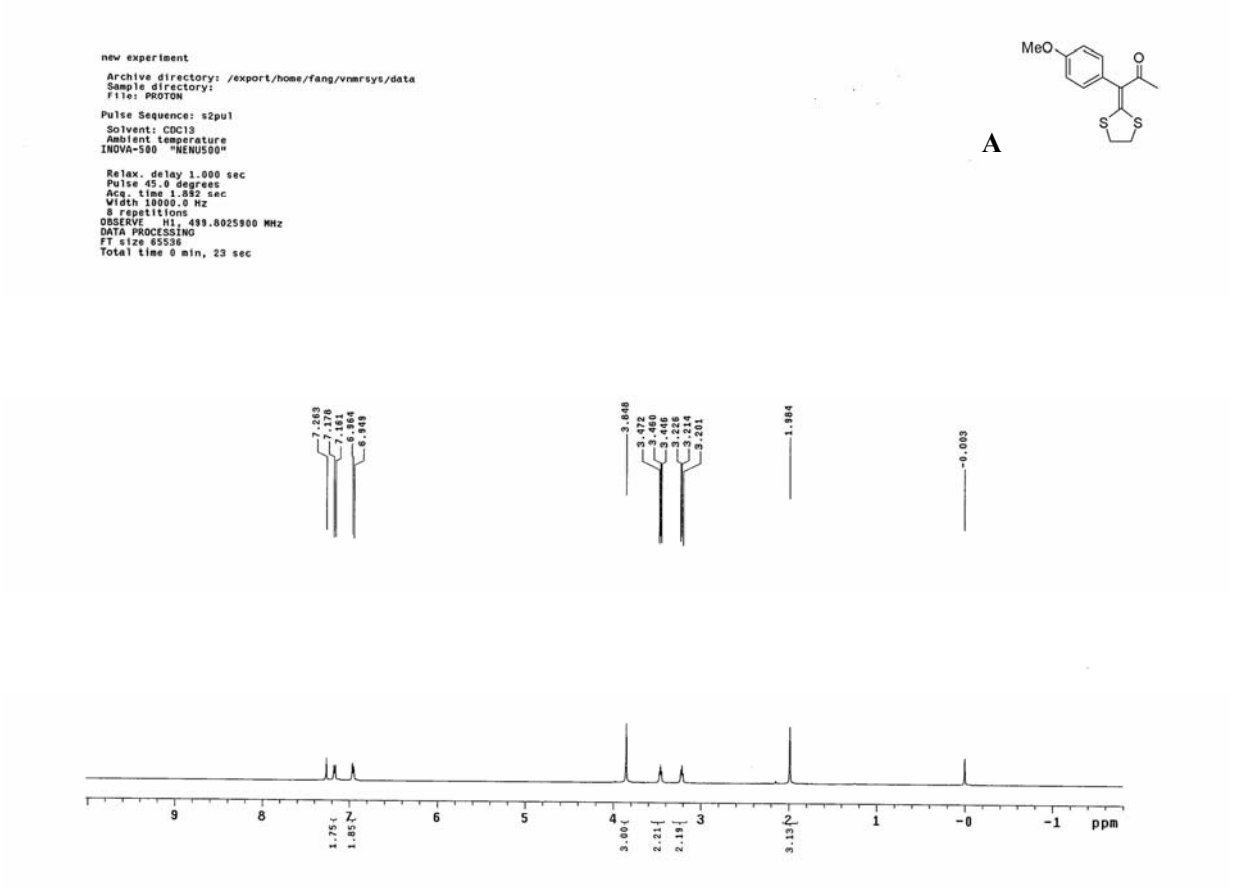
1 Sheldrick, G. M. *SHELXS-97*, Programs for X-ray Crystal Structure Solution, University of Göttingen: Göttingen, Germany, 1997.

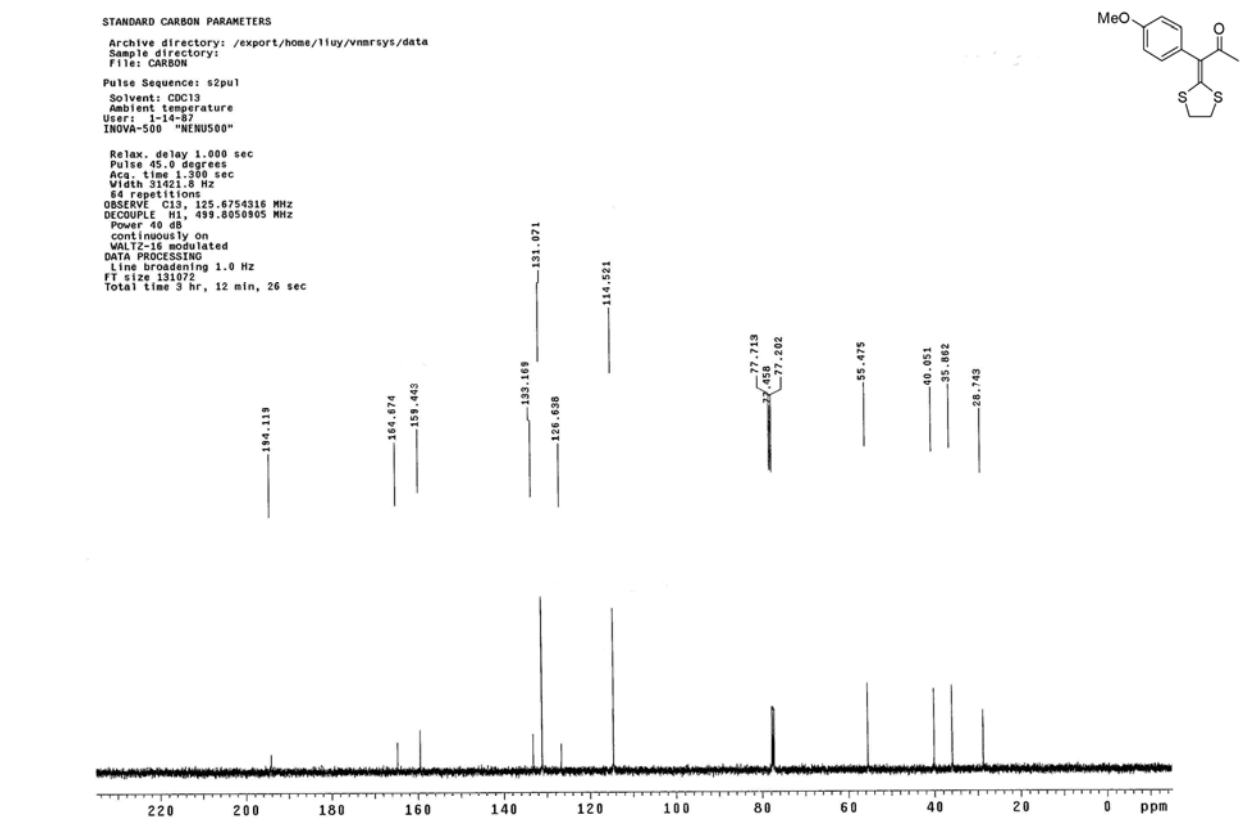
2 Sheldrick, G. M. *SHELXL-97*, Programs for X-ray Crystal Structure Refinement, University of Göttingen: Göttingen, Germany, 1997.

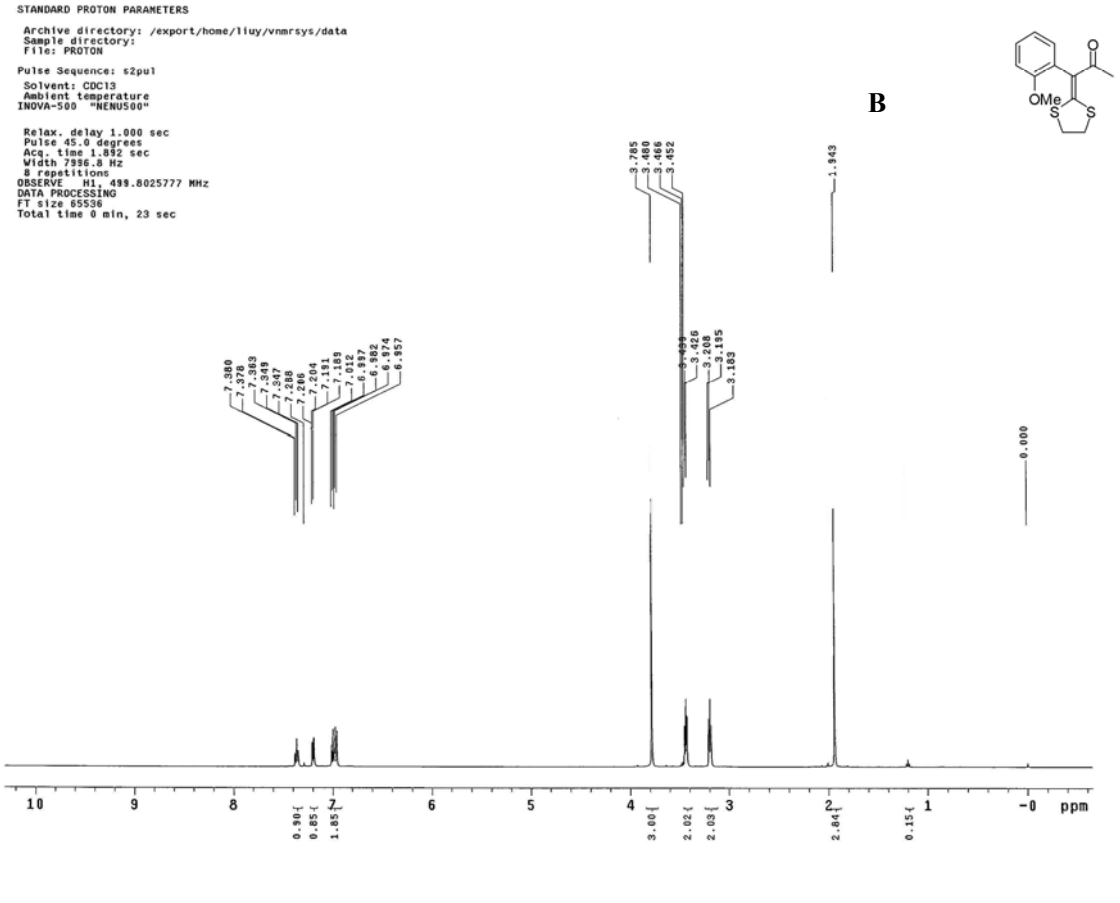
3 Farrugia, L. J. *WINGX, A Windows Program for Crystal Structure Analysis*; University of Glasgow; Glasgow, UK, 1988.

4 (*a*) Bi, X.; Dong, D.; Liu, Q.; Pan, W.; Zhao, L.; Li, B. *J. Am. Chem. Soc.,* 2005, **127**, 4578; (*b*) Liu, J.; Wang, M.; Li, B.; Liu, Q.; Zhao, Y. *J. Org. Chem.,* 2007, **72**, 4401.

VII. Copies of ^{1}H NMR and ^{13}C NMR spectra of new compounds



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Sample directory:
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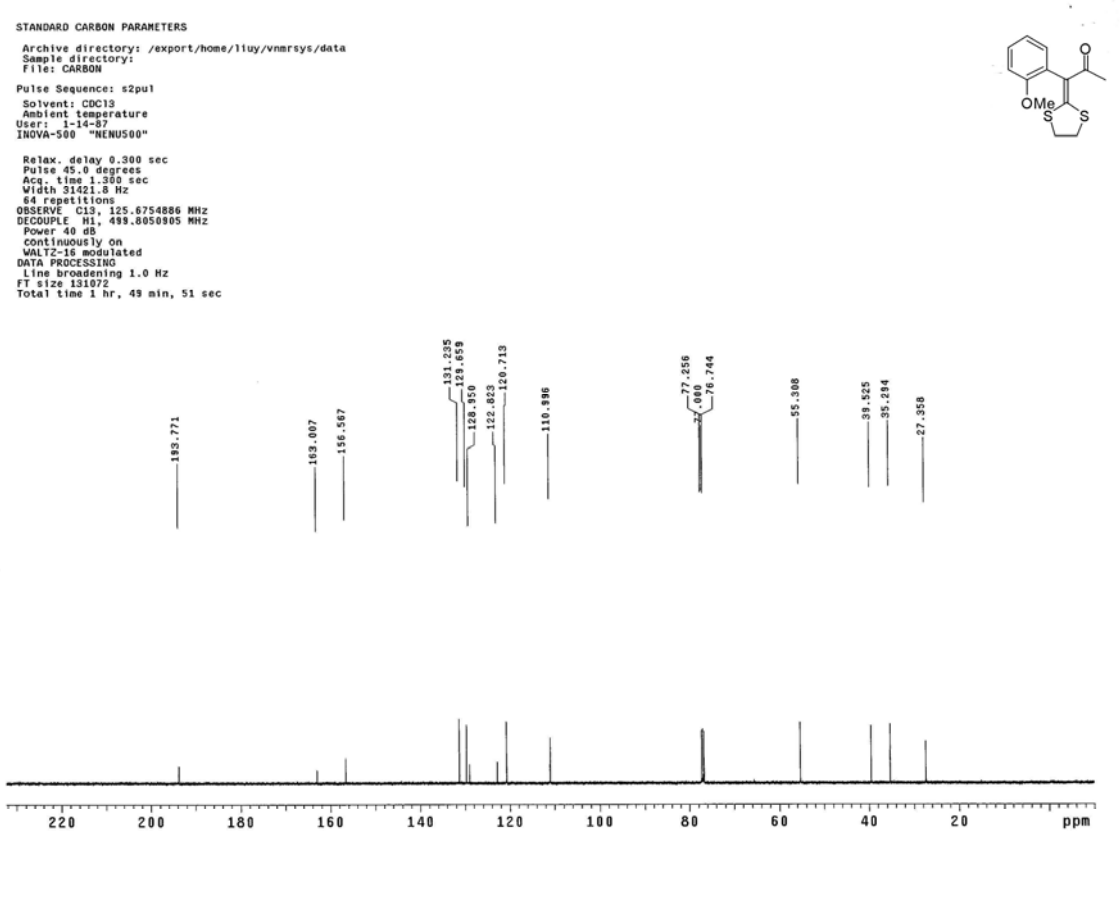
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7936.8 Hz
8 repetitions
OBSERVE H1, 499.8025777 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
B
OMe
S
S
O
7.380
7.378
7.363
7.349
7.347
7.288
7.266
7.204
7.191
7.189
7.017
7.015
6.982
6.974
6.957
3.785
3.480
3.466
3.452
3.439
3.426
3.208
3.195
3.183
1.943
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.90
0.85
1.85
3.60
2.02
2.03
2.84
0.15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Sample directory:
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Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754886 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
OMe
S
S
O
193.771
163.007
156.567
131.295
129.659
128.950
122.823
120.713
110.996
77.256
77.000
76.744
55.308
38.525
35.294
27.358
220
200
180
160
140
120
100
80
60
40
20
ppm

```
STANDARD PROTON PARAMETERS

 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: 1887
INOVA-500  "NENU500"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 7986.8 Hz
 8 repetitions
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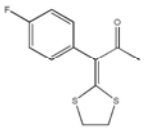
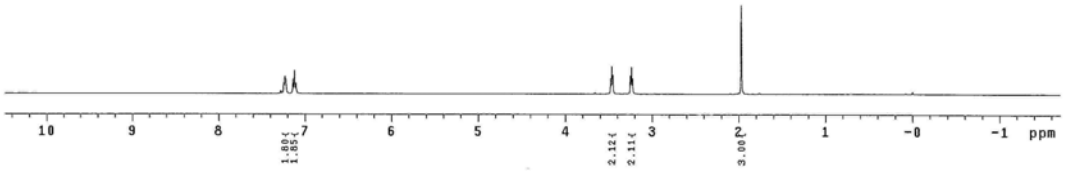
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
```



C

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STANDARD CARBON PARAMETERS

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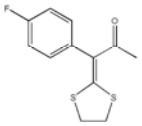
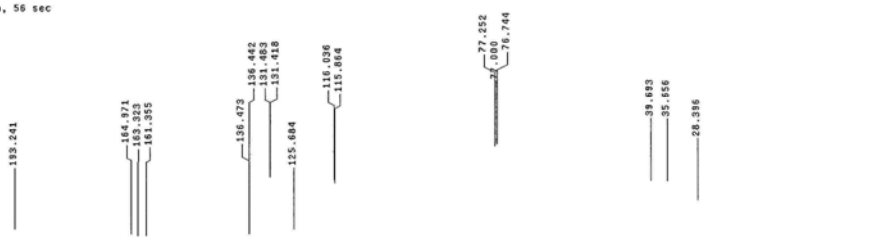
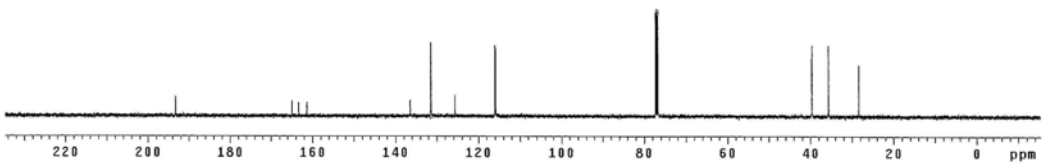
 Sample directory:

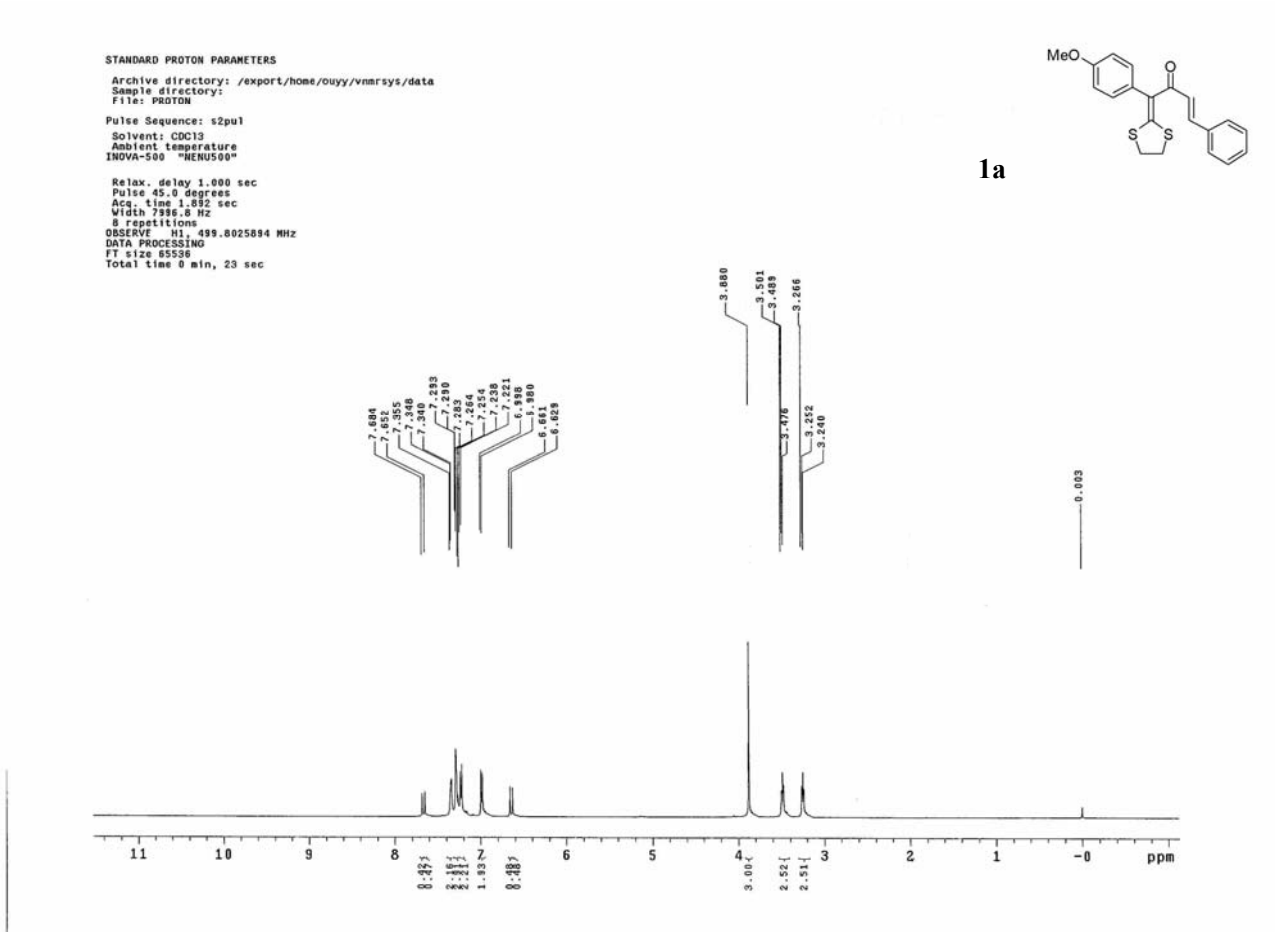
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
User:  1-14-87
File: 1888
INOVA-500  "NENU500"

 Relax. delay 0.300 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 128 repetitions
OBSERVE   C13, 125.6754714 MHz
DECOUPLE  H1, 499.8050905 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec
```





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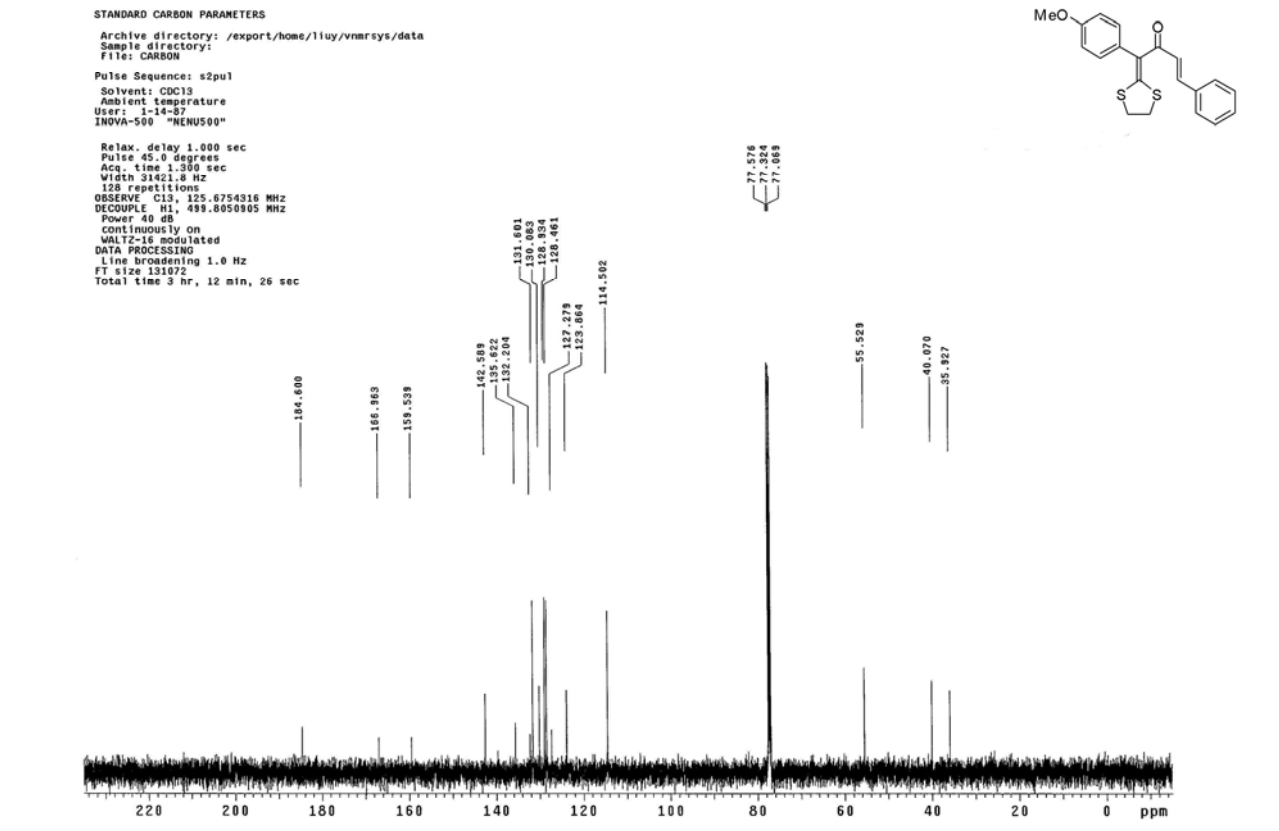
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7386.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 85536
Total time 0 min, 23 sec
MeO
1a
ppm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Sample directory:
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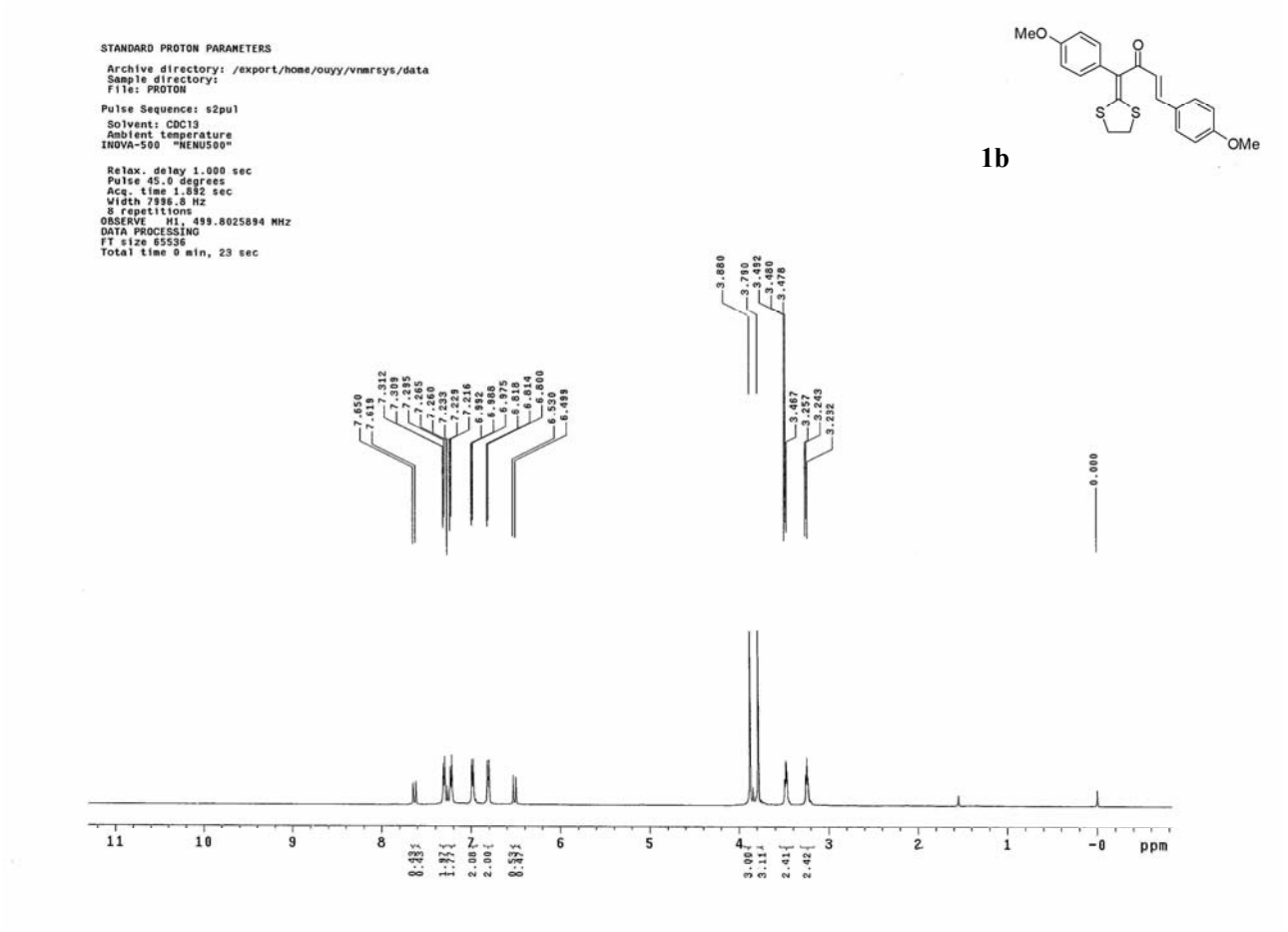
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
MeO
ppm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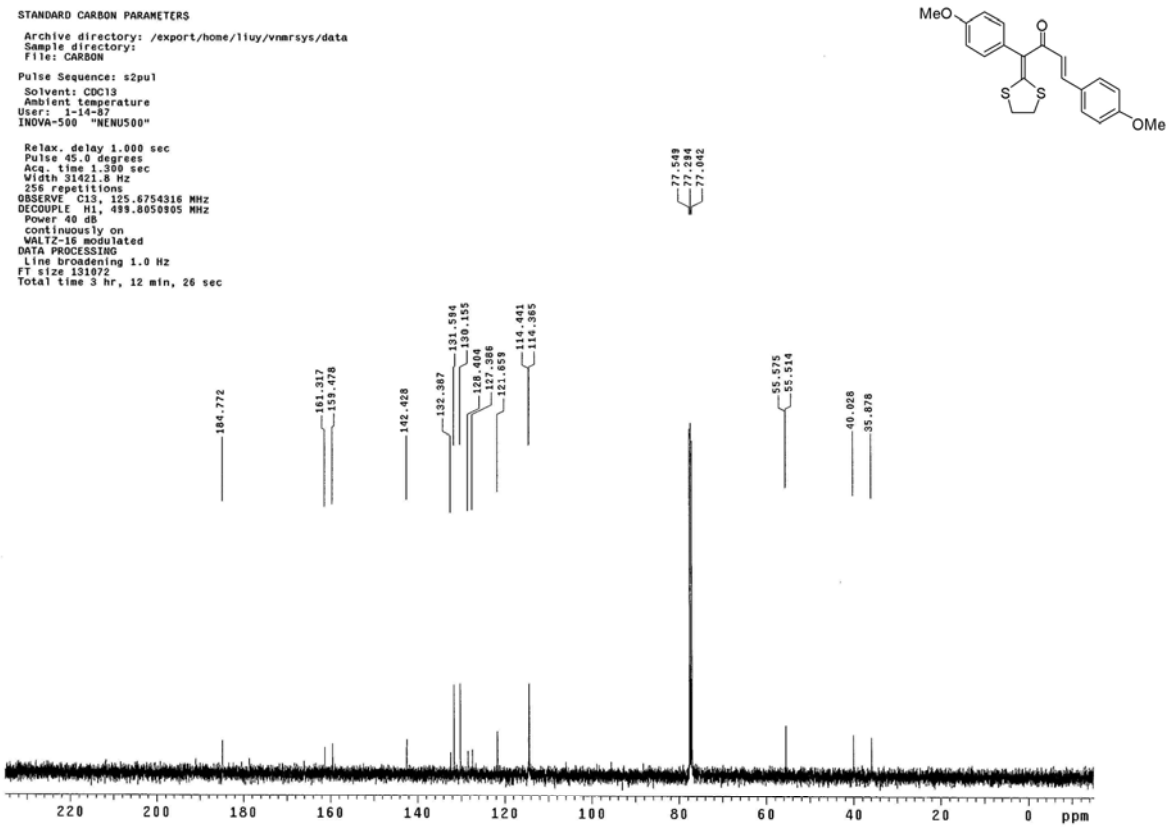
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
OMe
1b
7.650
7.619
7.312
7.301
7.295
7.265
7.260
7.233
7.223
7.216
7.202
6.988
6.975
6.814
6.810
6.530
6.489
3.880
3.790
3.492
3.480
3.478
3.467
3.257
3.243
3.232
0.000
11
10
9
8
7
6
5
4
3
2
1
-0
ppm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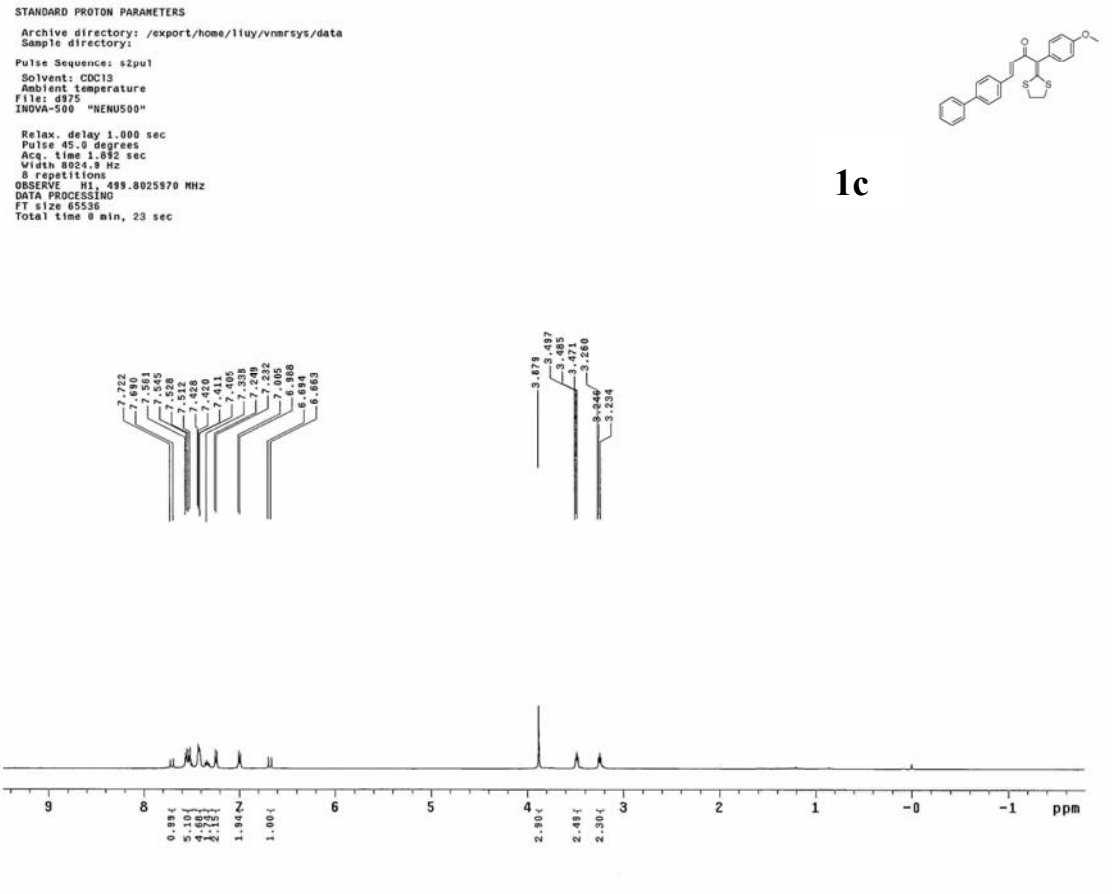
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050805 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
MeO
OMe
77.548
77.294
77.042
184.772
161.317
159.478
142.428
132.387
131.594
130.155
128.404
127.386
121.659
114.441
114.365
55.575
55.514
40.028
35.878
220
200
180
160
140
120
100
80
60
40
20
0
ppm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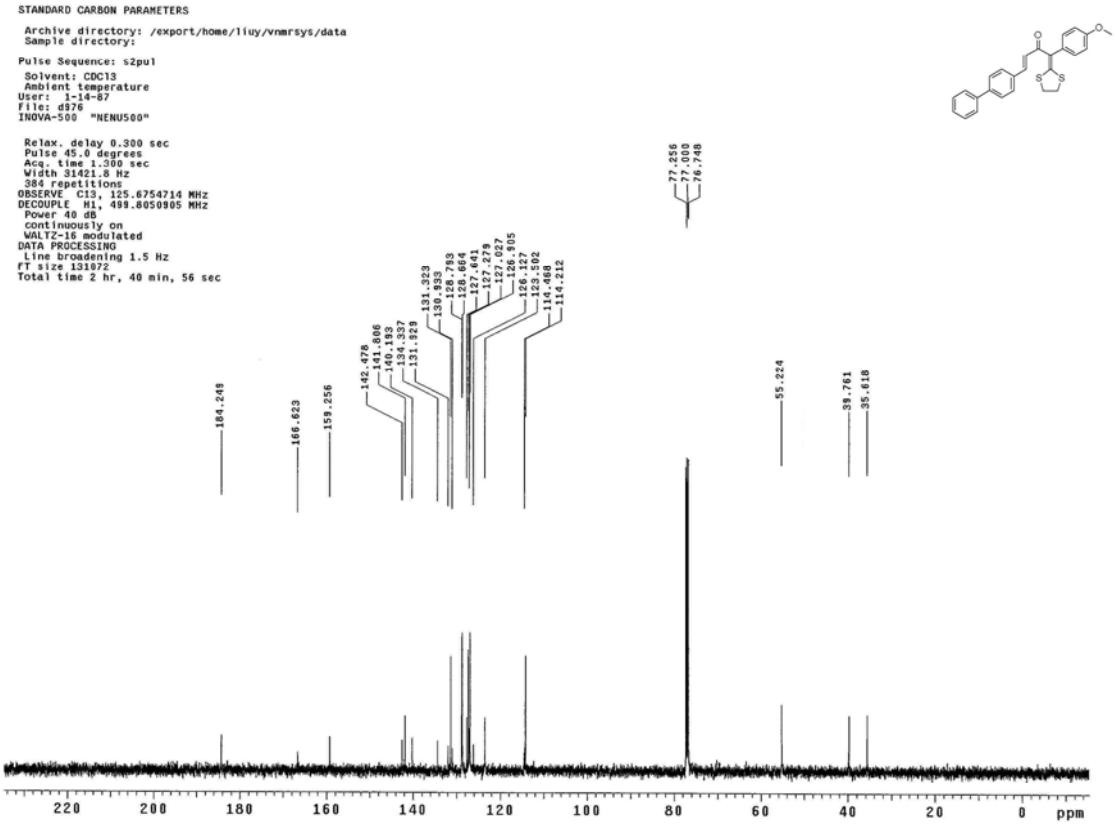
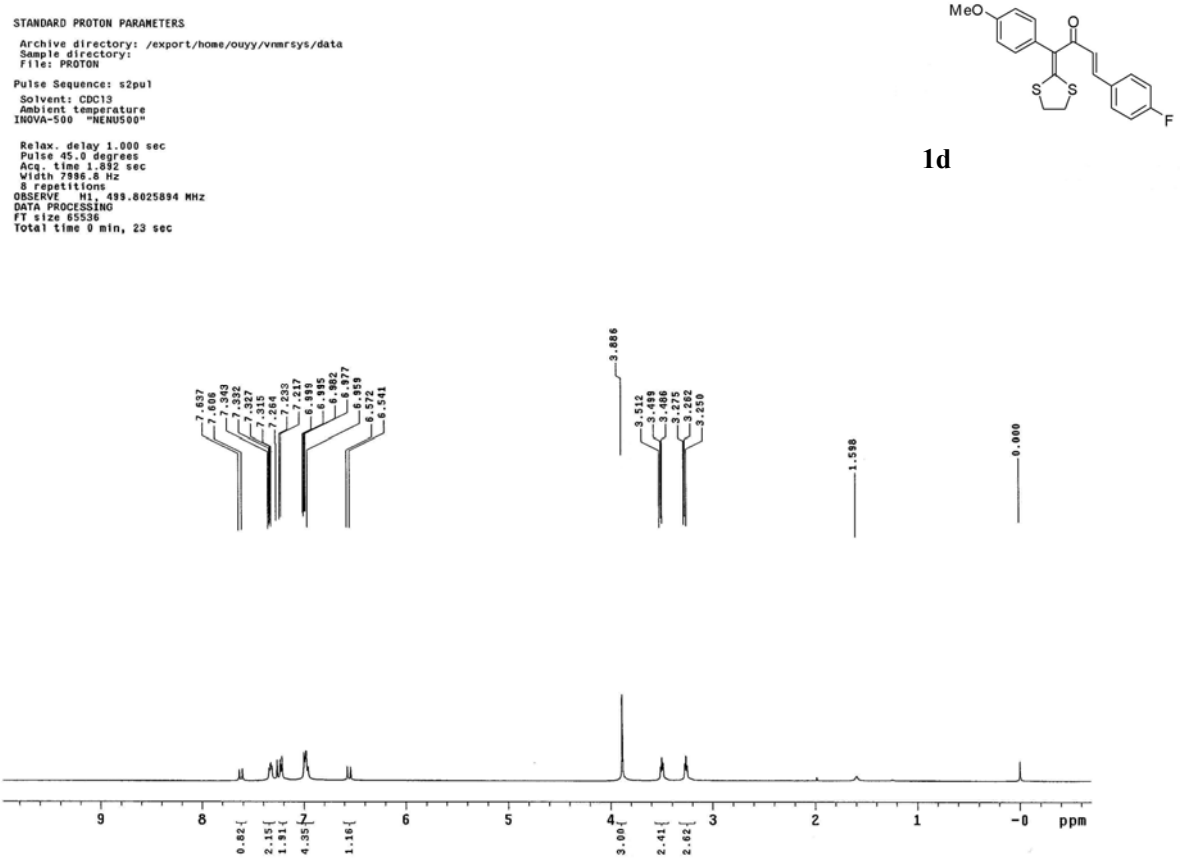
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d975
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.692 sec
Width 8024.9 Hz
8 repetitions
OBSERVE H1, 499.8025970 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
1c
7.722
7.690
7.561
7.545
7.528
7.512
7.428
7.420
7.411
7.406
7.398
7.249
7.232
7.005
6.988
6.884
6.863
3.479
3.487
3.485
3.471
3.260
3.246
3.234
9
8
7
6
5
4
3
2
1
-0
-1
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: d976
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
384 repetitions
OBSERVE C13, 125.6754714 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec
77.256
77.000
76.748
131.323
130.933
128.793
128.664
127.641
127.279
127.237
126.605
126.127
123.502
114.468
114.212
142.478
141.806
140.193
134.937
131.329
184.249
166.623
159.256
55.224
39.761
35.618
220
200
180
160
140
120
100
80
60
40
20
0
ppm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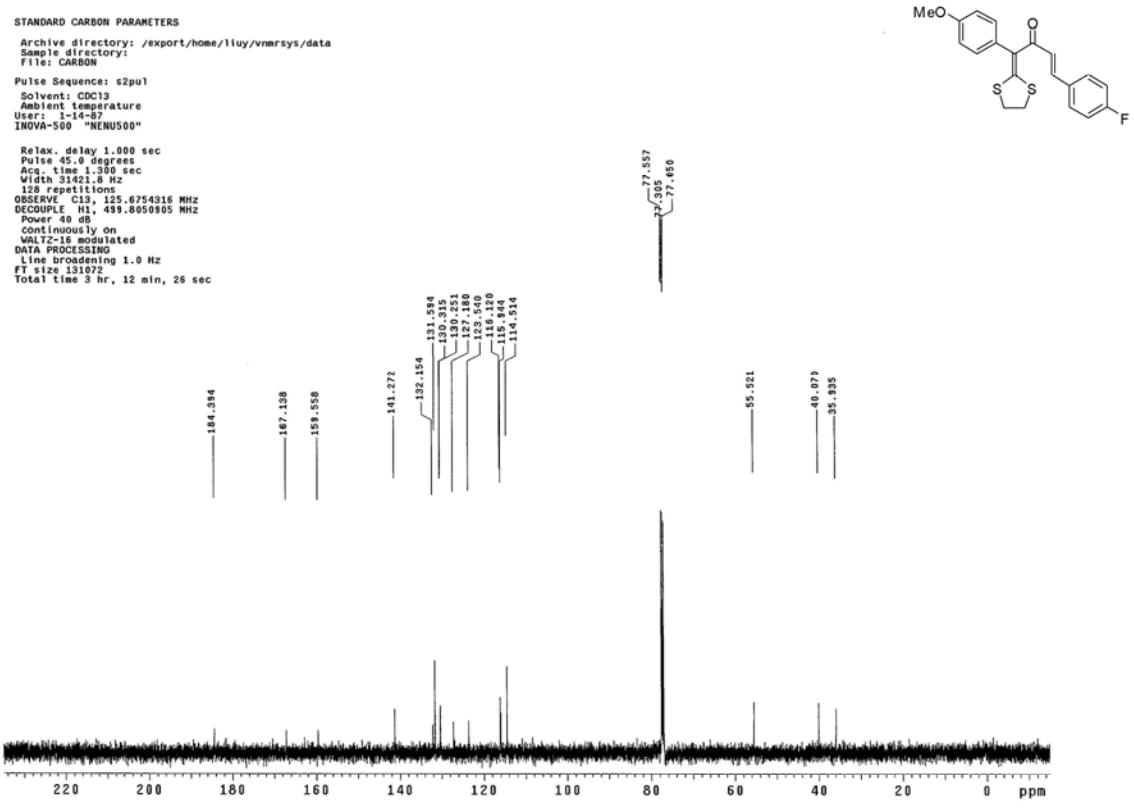
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
O
S
S
F
1d
7.637
7.606
7.343
7.332
7.327
7.315
7.264
7.233
7.217
6.999
6.995
6.982
6.977
6.959
6.572
6.541
3.886
3.512
3.499
3.486
3.275
3.262
3.250
1.598
0.000
ppm
0.82
2.15
1.91
4.35
1.16
3.00
2.41
2.62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Sample directory:
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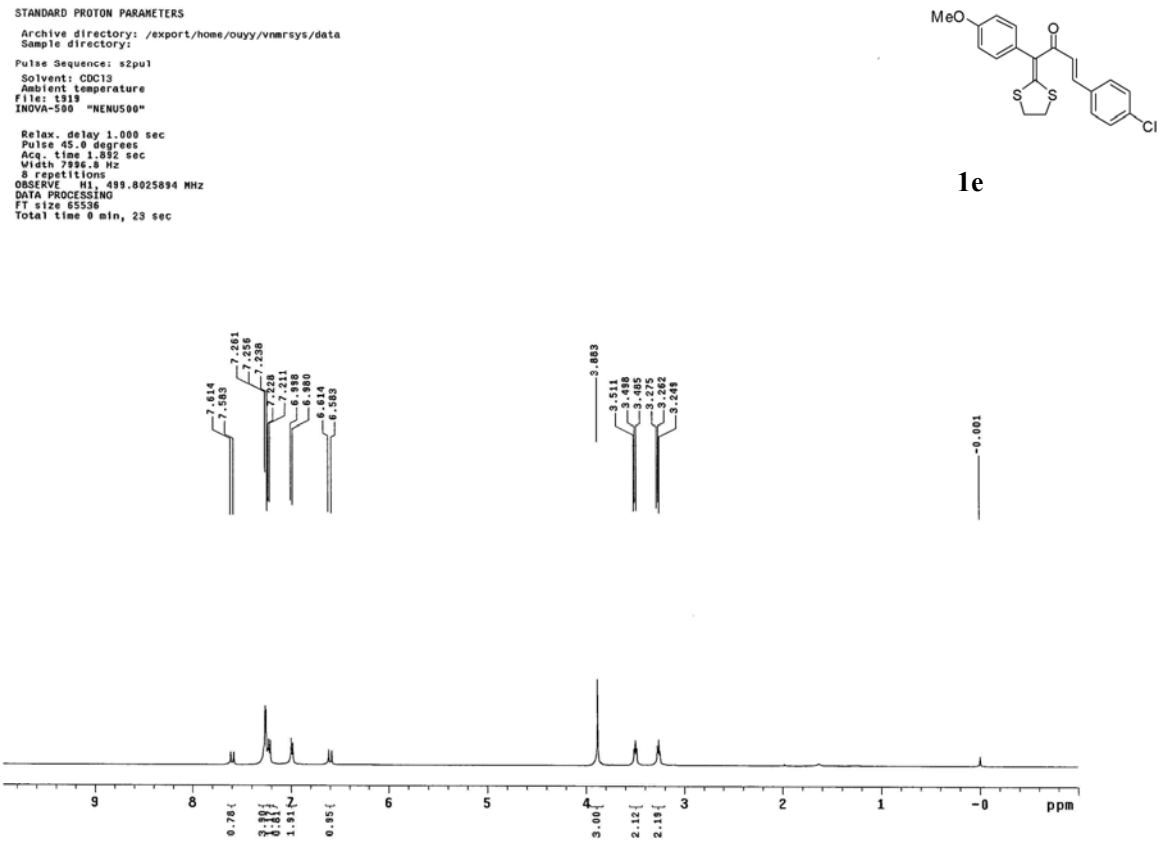
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
MeO
O
S
S
F
77.557
77.305
77.050
131.594
130.315
130.251
127.180
126.540
116.120
115.944
114.514
184.394
167.138
159.558
141.272
132.154
55.521
40.073
35.935
ppm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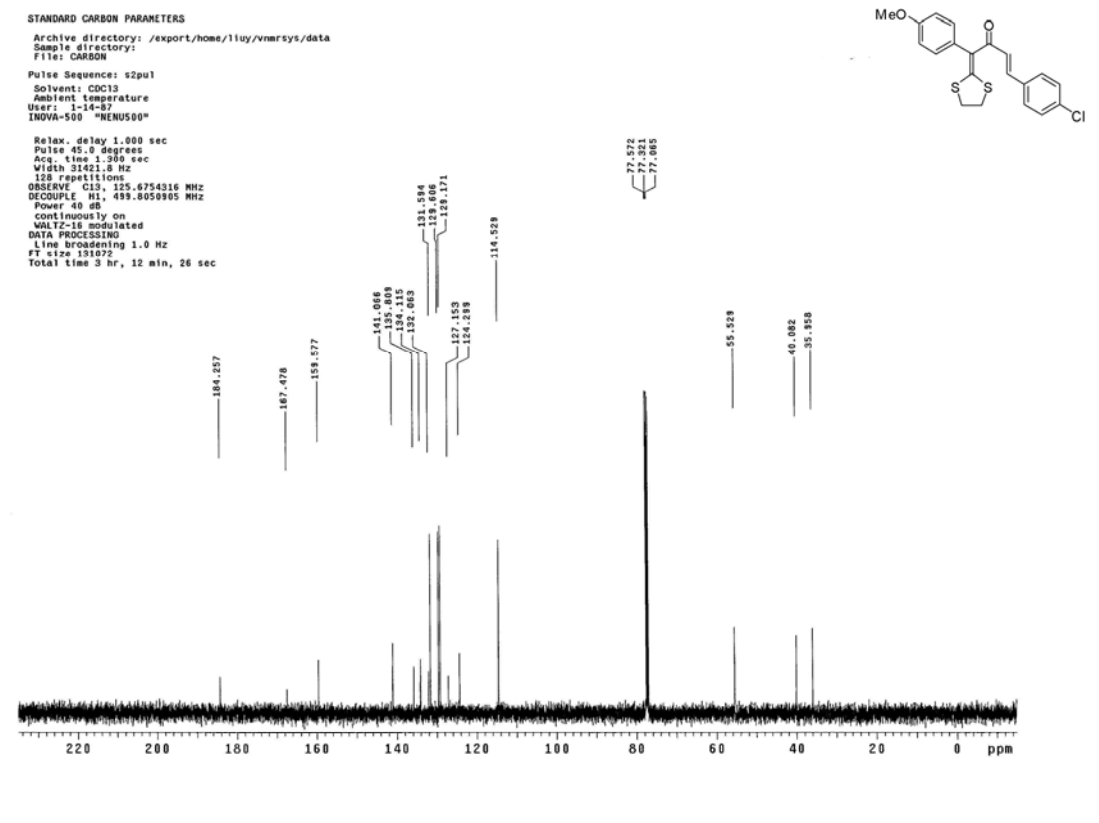
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: t919
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
O
S
S
Cl
1e
7.614
7.583
7.261
7.256
7.236
7.228
7.211
6.618
6.600
6.614
6.583
3.883
3.511
3.498
3.485
3.275
3.262
3.249
-0.001
0.78
3.00
0.85
1.91
2.12
2.18
3.00
9
8
7
6
5
4
3
2
1
-0
ppm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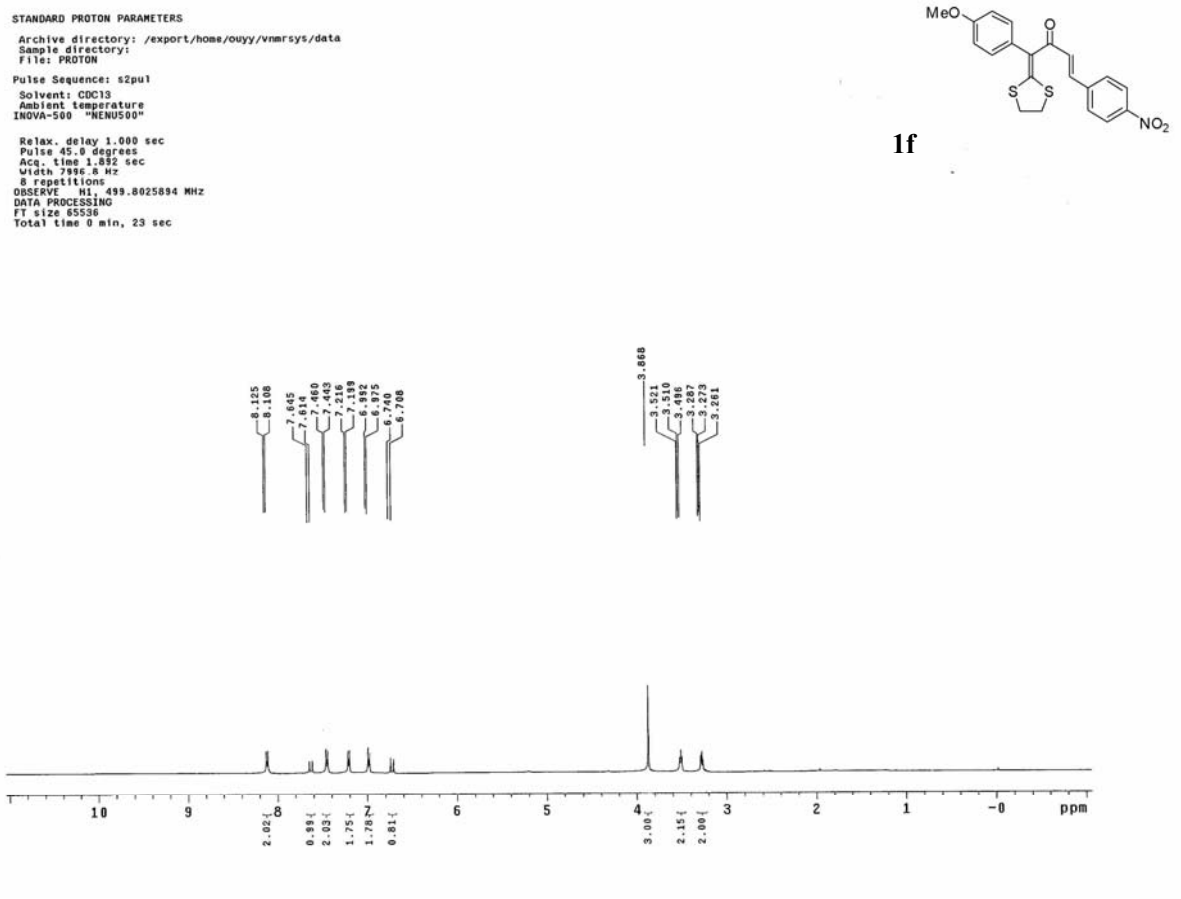
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
MeO
O
S
S
Cl
77.572
77.321
77.065
141.066
135.809
134.115
132.063
131.594
130.606
129.171
127.153
124.289
114.529
184.257
167.478
159.577
55.528
40.082
35.958
220
200
180
160
140
120
100
80
60
40
20
0
ppm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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 1.852 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
O
S
S
NO2
1f
8.125
8.108
7.645
7.614
7.460
7.443
7.216
7.199
6.752
6.735
6.740
6.708
3.868
3.521
3.510
3.496
3.287
3.273
3.261
10
9
8
6
5
4
3
2
1
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ppm
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2.03
1.75
1.78
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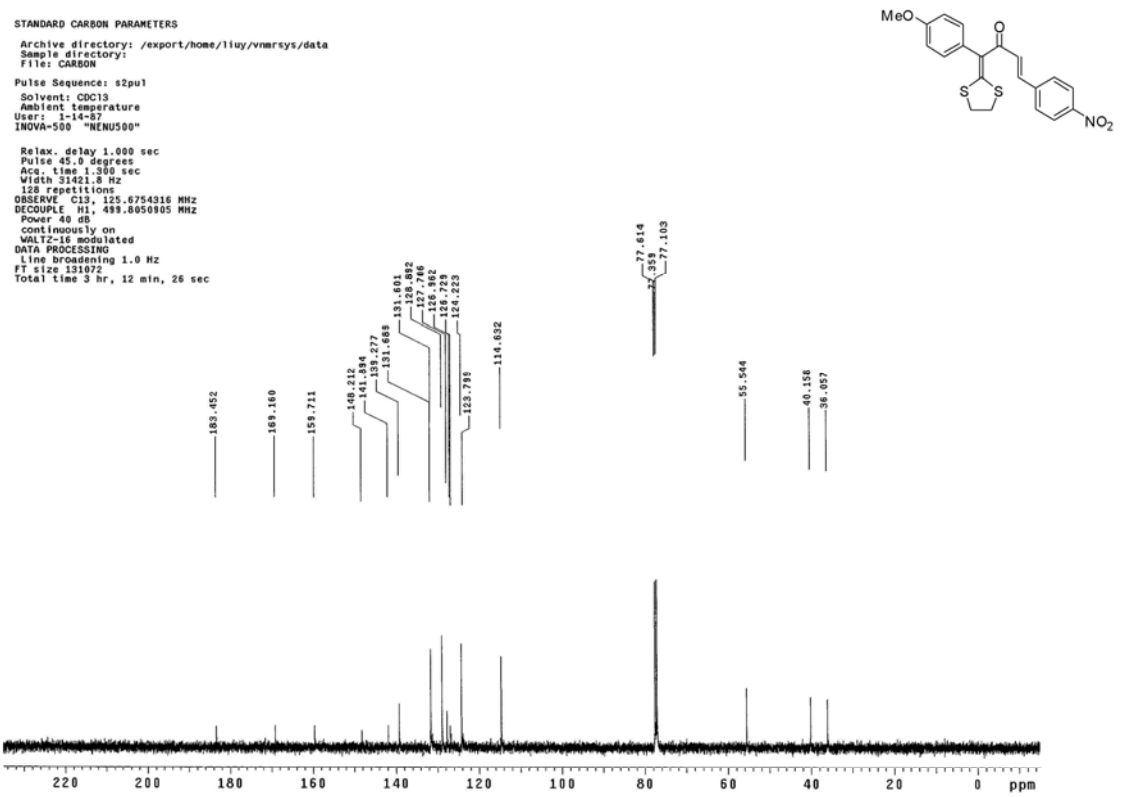
3.00
2.15
2.00


STANDARD CARBON PARAMETERS
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Sample directory:
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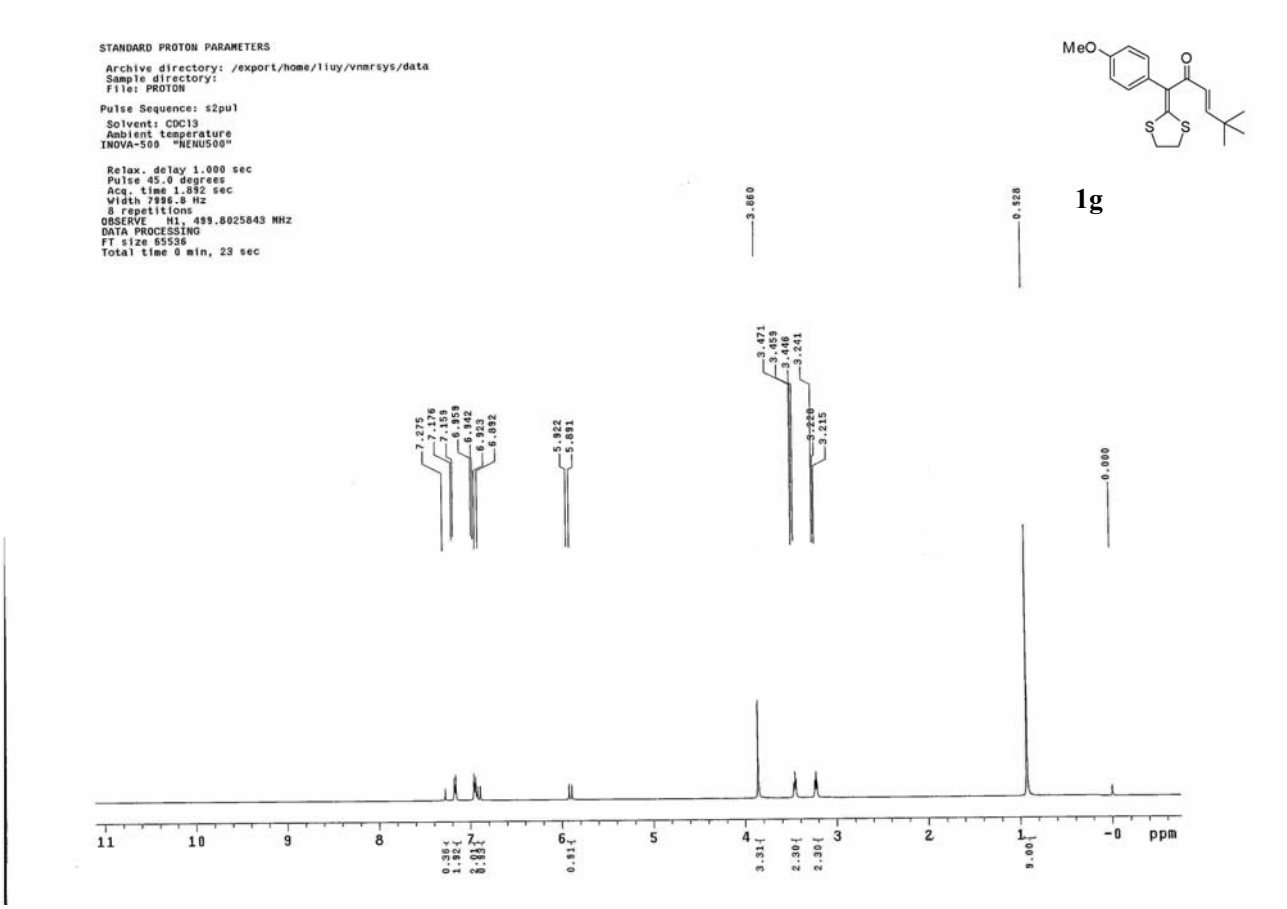
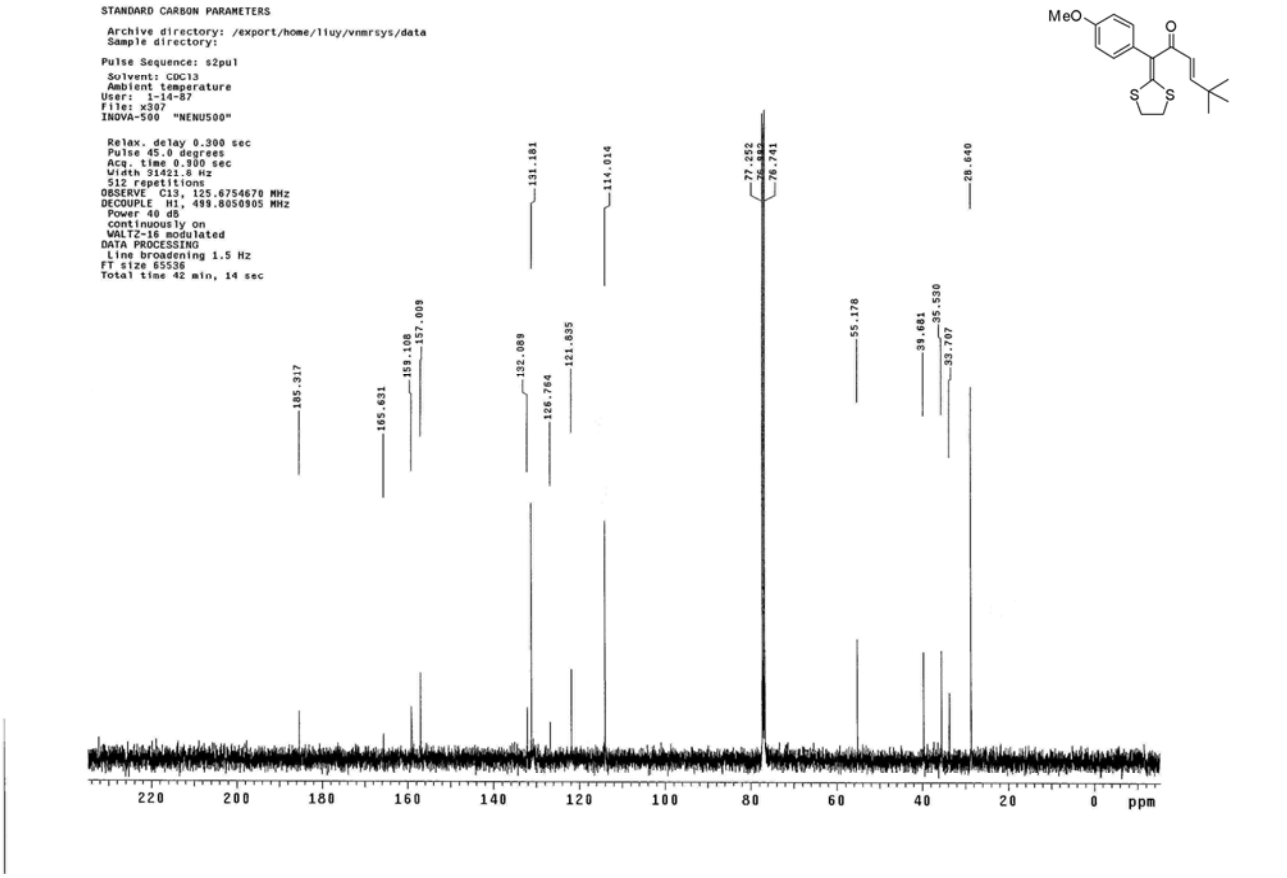
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
MeO
O
S
S
NO2
163.452
159.160
159.711
148.212
141.884
139.277
131.688
131.601
129.852
127.788
126.962
126.729
124.223
123.788
114.632
77.614
77.359
77.103
55.544
40.158
36.057
220
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/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
8 repetitions
OBSERVE H1, 499.8025843 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
1g
7.275
7.176
7.159
6.959
6.942
6.923
6.892
5.922
5.891
3.660
3.471
3.459
3.446
3.241
3.226
3.215
0.928
0.000
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

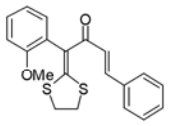

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: x307
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.900 sec
Width 31421.8 Hz
512 repetitions
OBSERVE C13, 125.6754670 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 42 min, 14 sec
MeO
185.317
165.631
159.108
157.009
132.089
131.181
126.764
121.635
114.014
77.252
76.741
55.178
39.681
35.530
33.707
28.640
220
200
180
160
140
120
100
80
60
40
20
0
ppm

STANDARD PROTON PARAMETERS

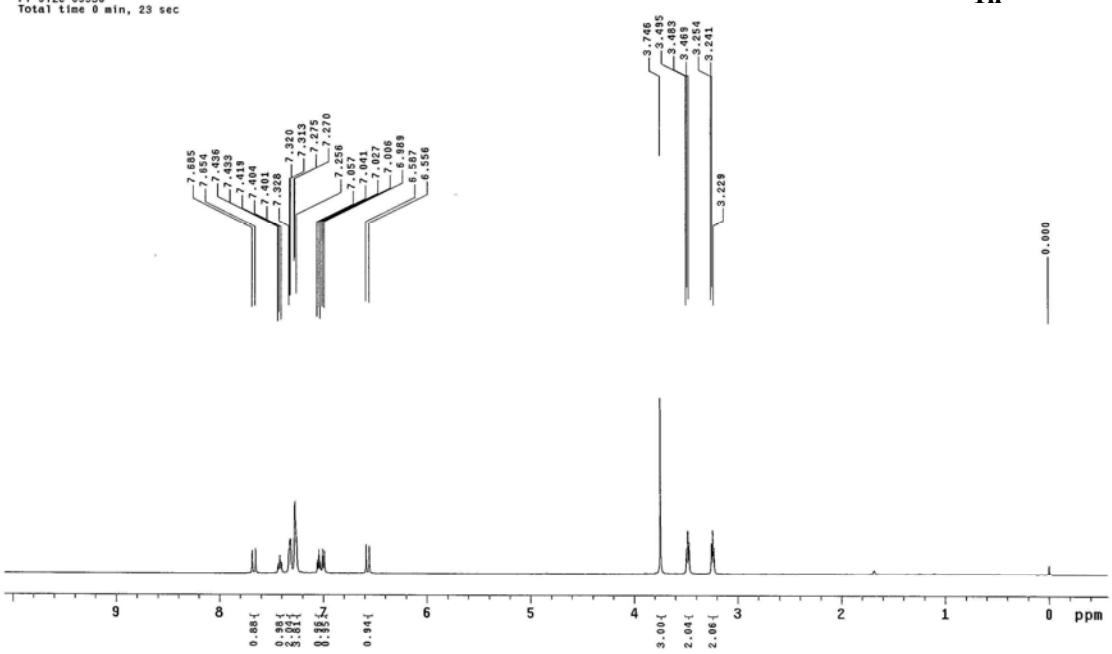
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: PROTON

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 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025931 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec



1h

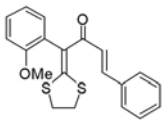


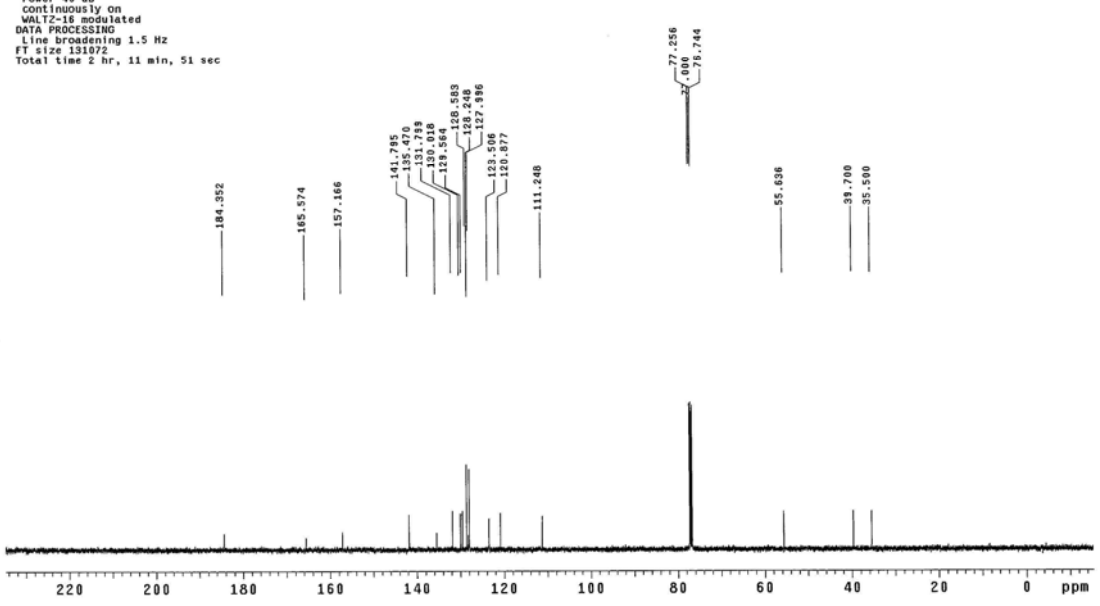
STANDARD CARBON PARAMETERS

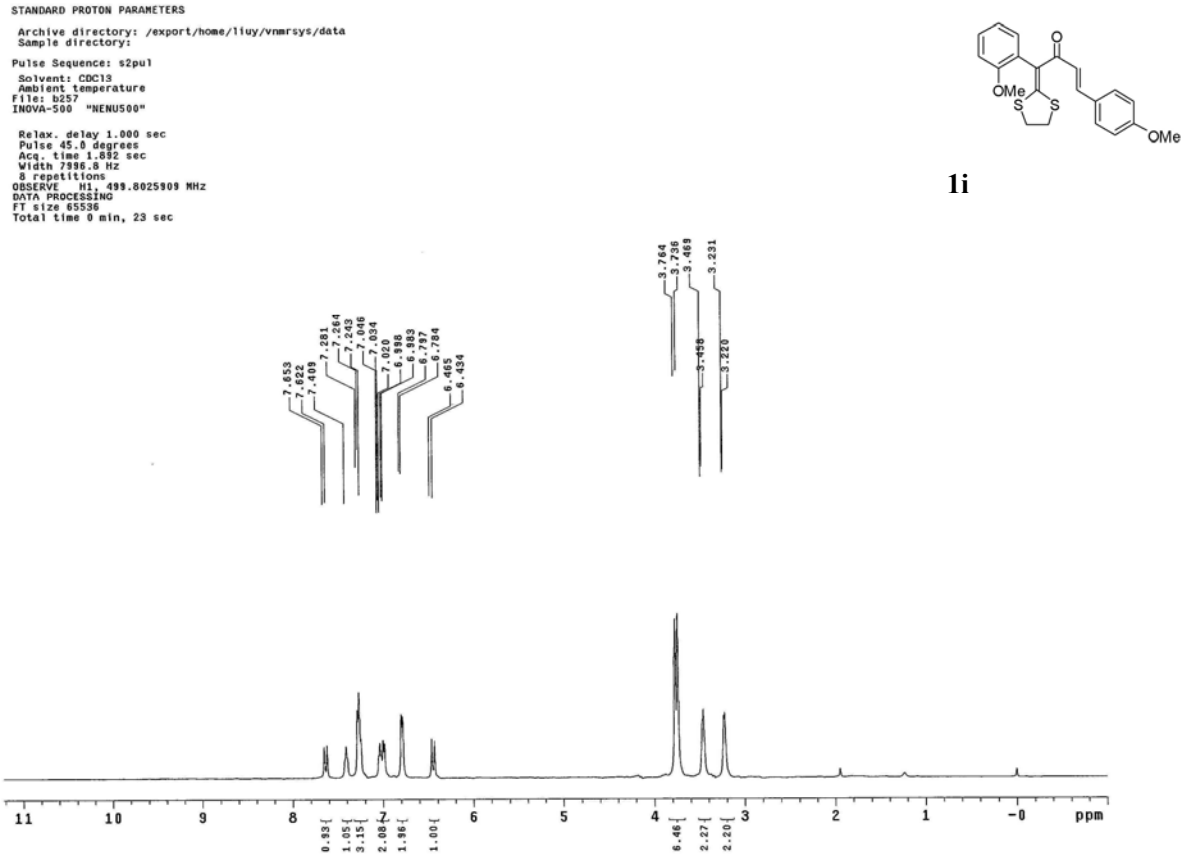
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON

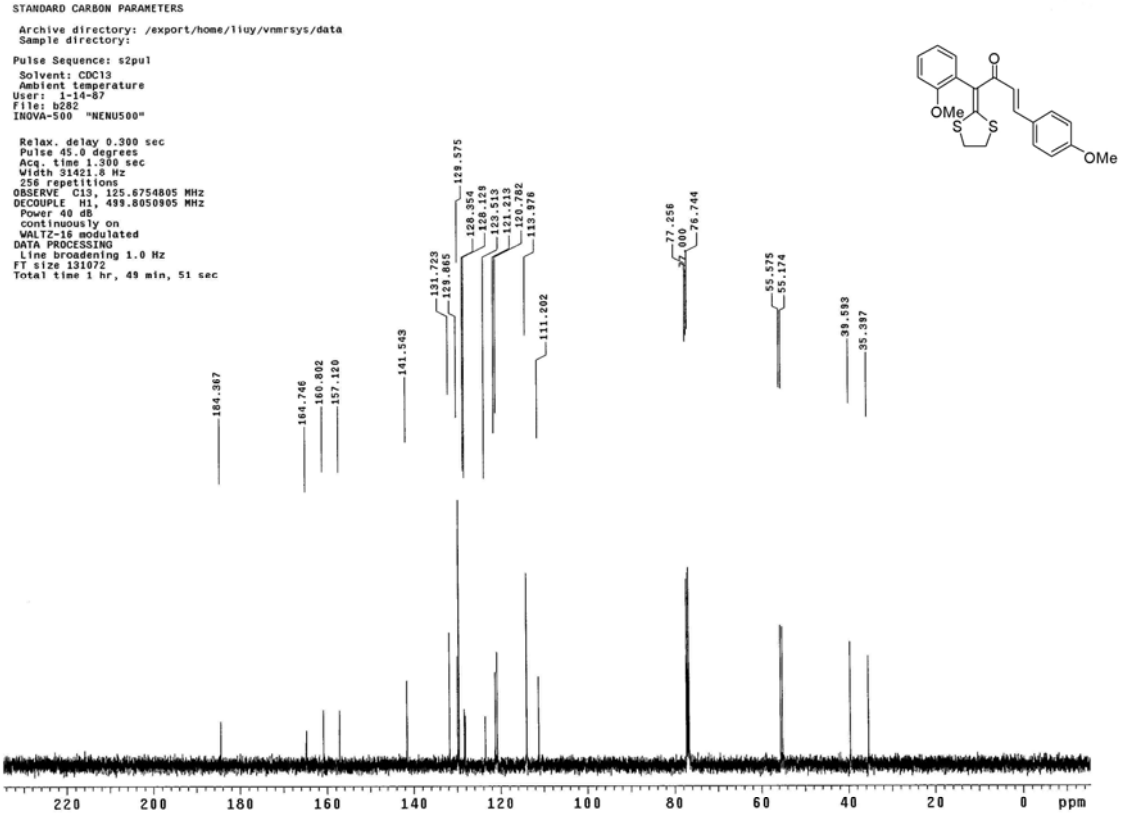
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
INOVA-500 "NENU500"

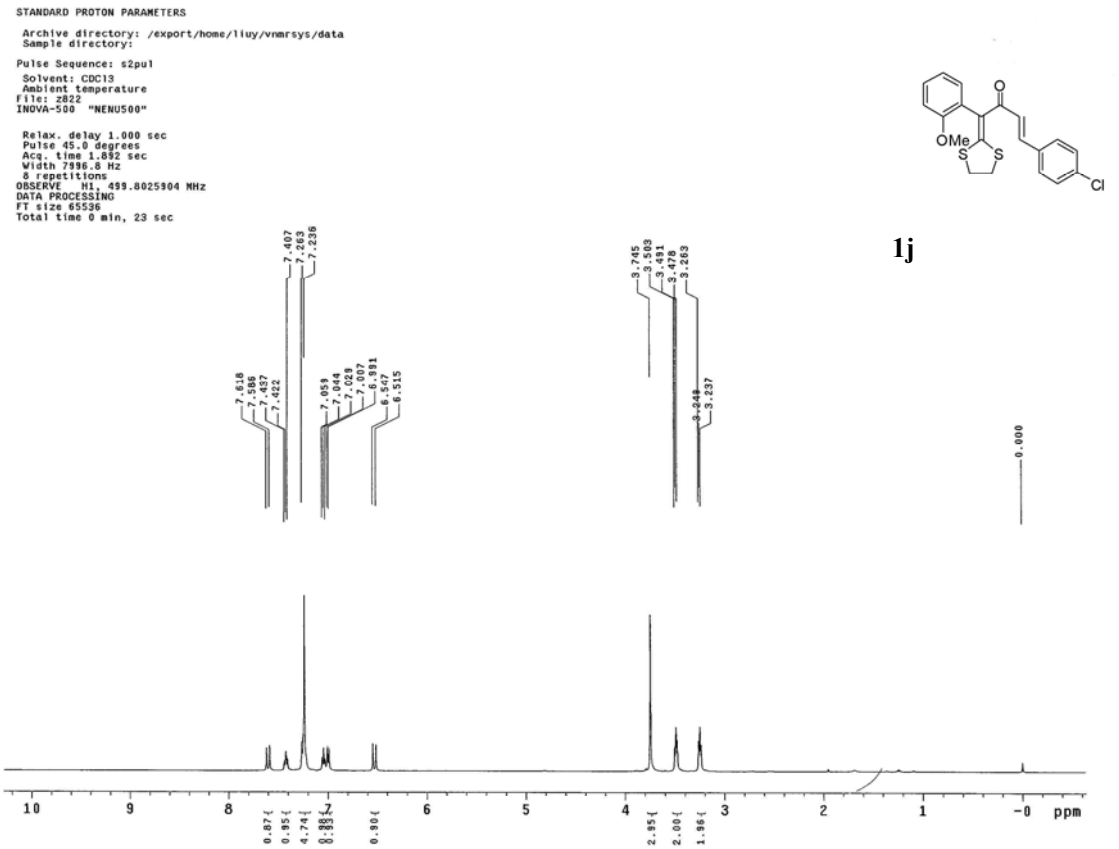
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754714 MHz
DECOUPLE H1, 499.8050305 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 11 min, 51 sec

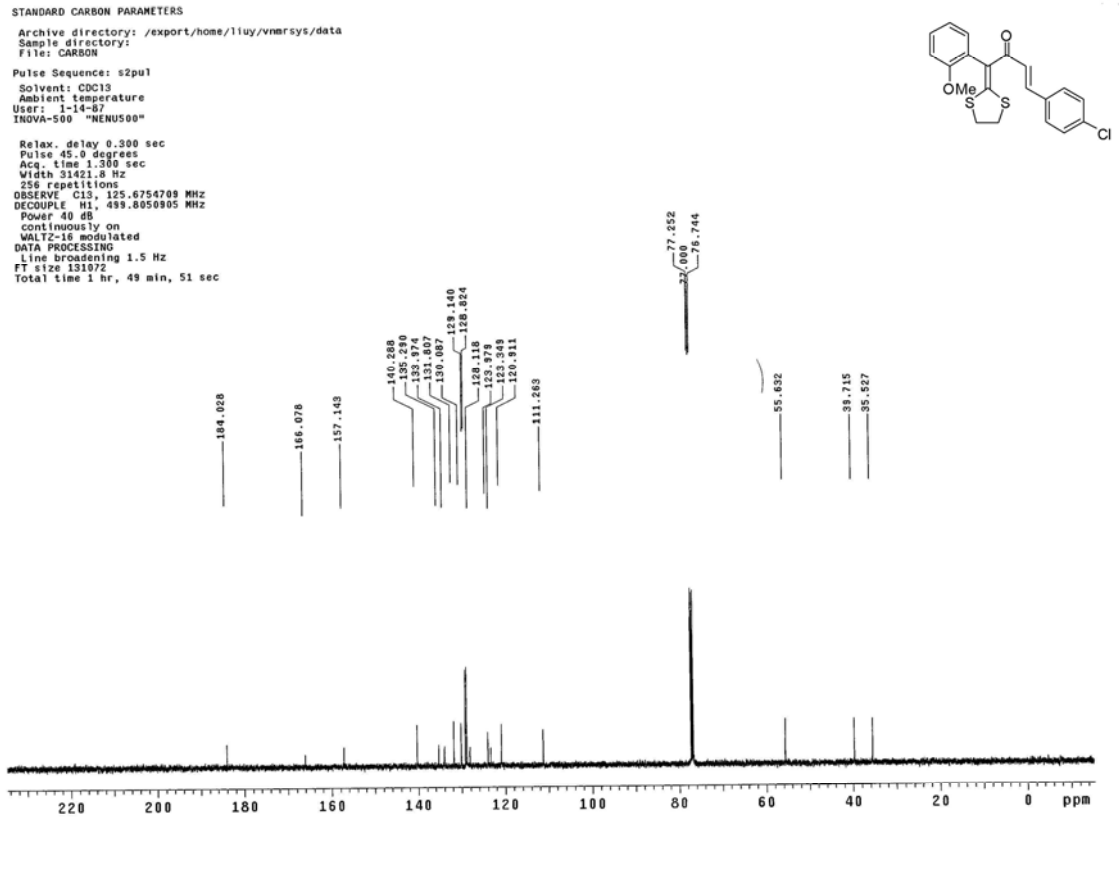


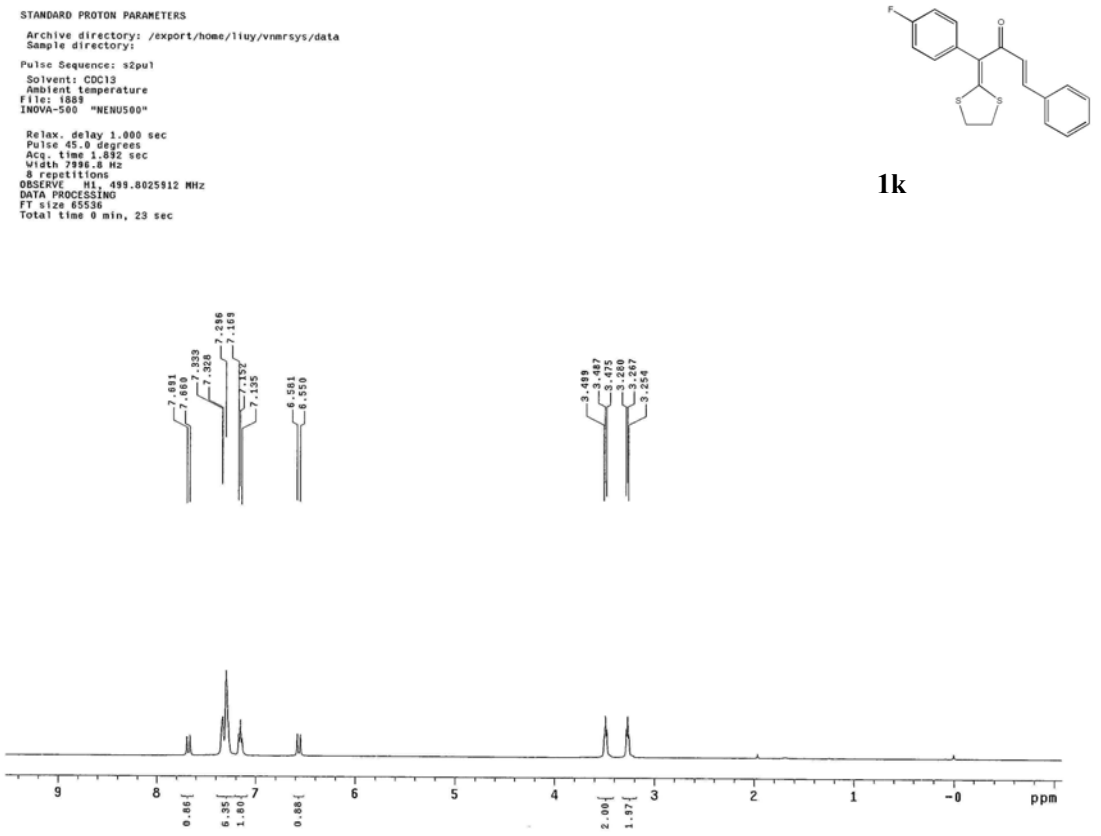



STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b257
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025909 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
OMe
S
S
O
OMe
1i
7.653
7.622
7.409
7.281
7.264
7.243
7.048
7.034
7.020
6.898
6.883
6.797
6.784
6.465
6.434
3.764
3.736
3.469
3.231
3.458
3.220
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.93
1.05
3.15
2.08
1.96
1.00
6.46
2.27
2.20

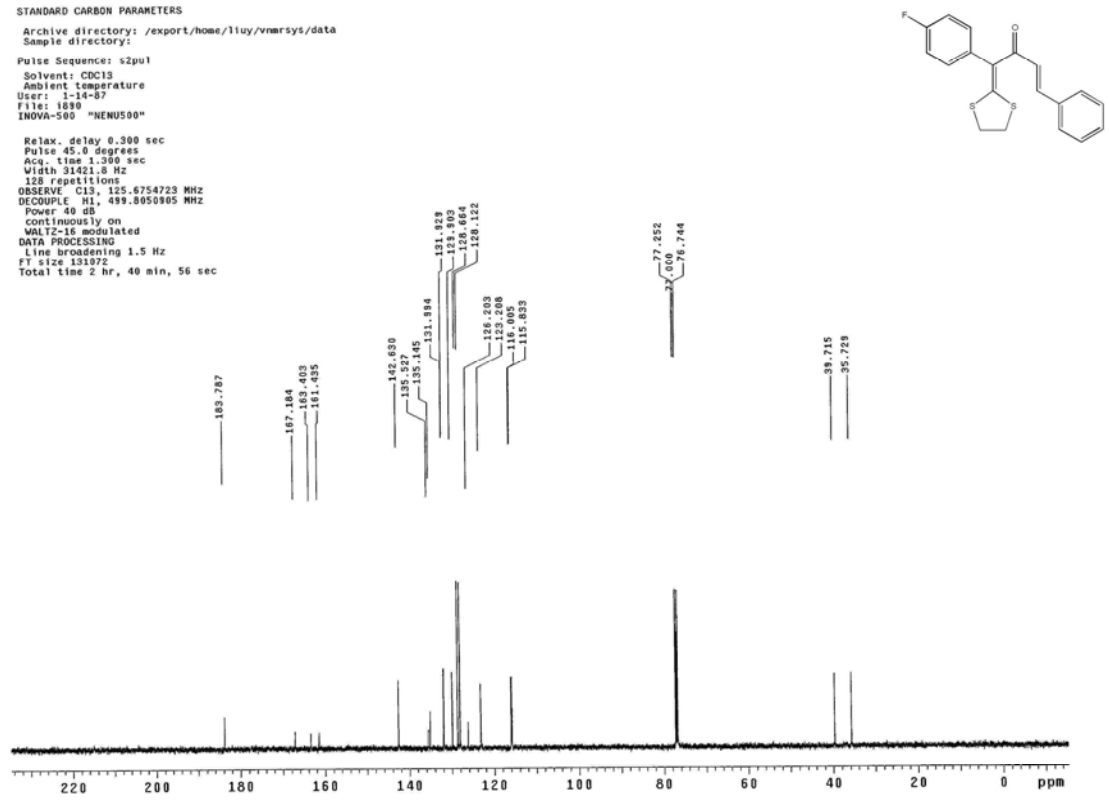

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: b282
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754805 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
OMe
S
S
O
OMe
184.367
164.746
160.802
157.120
141.543
131.723
129.465
129.575
128.354
128.129
123.513
121.213
120.782
113.976
111.202
77.256
77.000
76.744
55.575
55.174
39.593
35.387
220
200
180
160
140
120
100
80
60
40
20
0
ppm

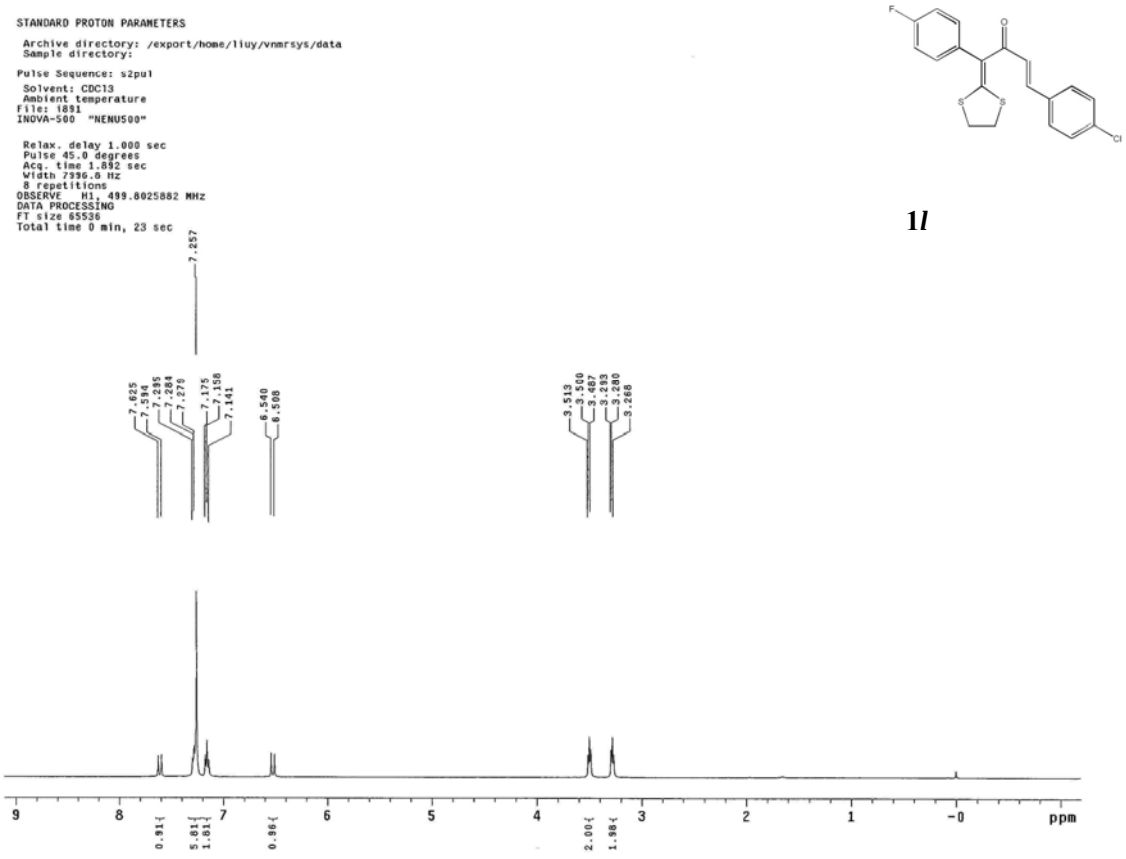

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: z822
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025904 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
1j
7.618
7.586
7.437
7.422
7.407
7.263
7.236
7.059
7.044
7.028
7.007
6.881
6.547
6.515
3.745
3.503
3.491
3.478
3.263
3.248
3.237
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.87
0.95
4.74
0.90
2.95
2.00
1.96


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754709 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
184.028
166.078
157.143
140.288
135.290
133.974
131.807
130.087
129.140
128.824
128.118
127.978
123.348
120.911
111.263
77.252
77.000
76.744
55.632
39.715
35.527
220
200
180
160
140
120
100
80
60
40
20
0
ppm

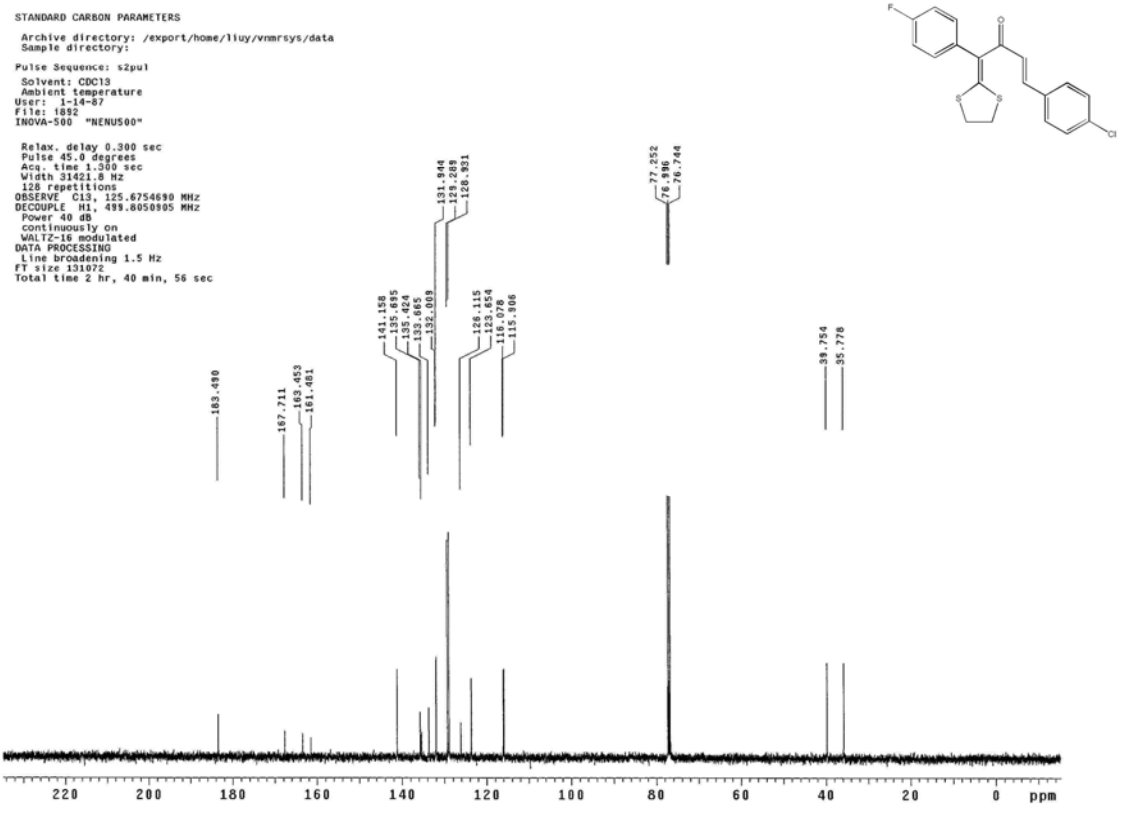

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: i889
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025812 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.691
7.680
7.333
7.328
7.096
7.169
7.152
7.135
6.561
6.550
3.498
3.487
3.475
3.260
3.267
3.254
0.86
6.35
1.80
0.88
2.00
1.97
ppm

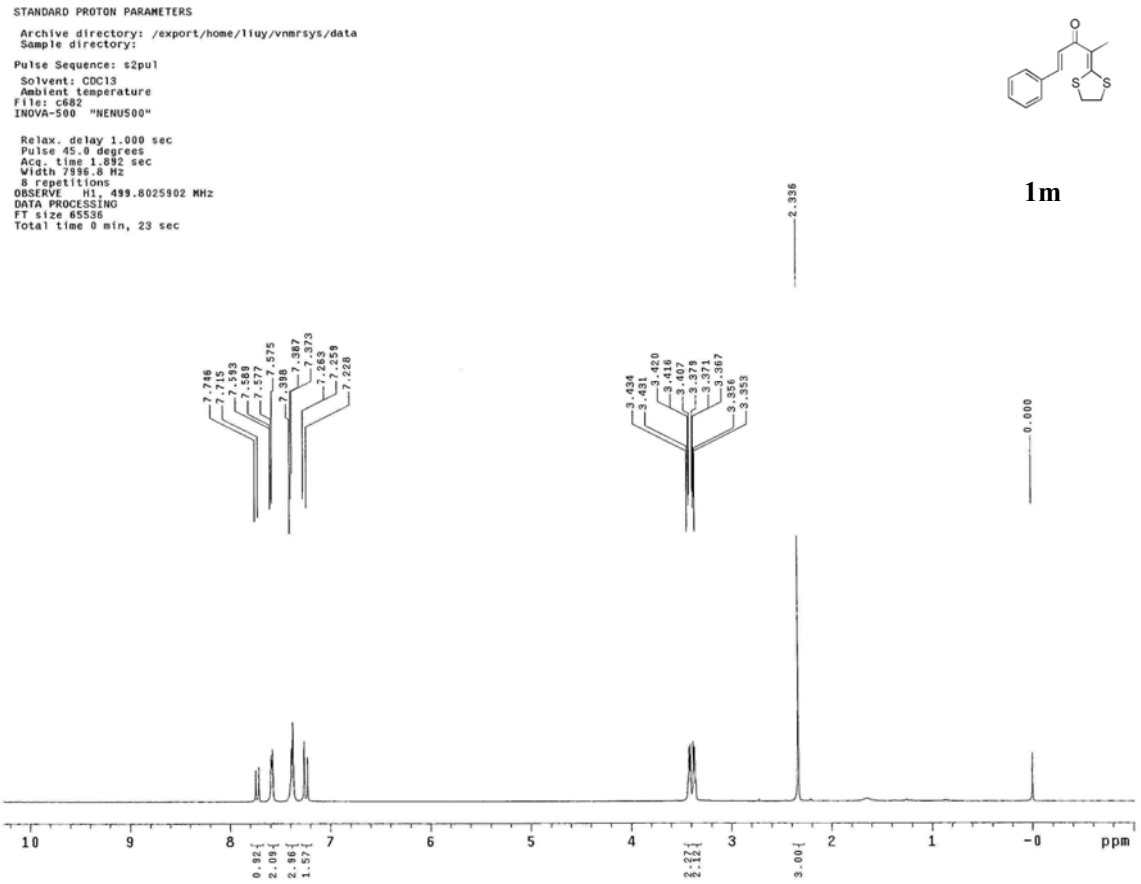
1k

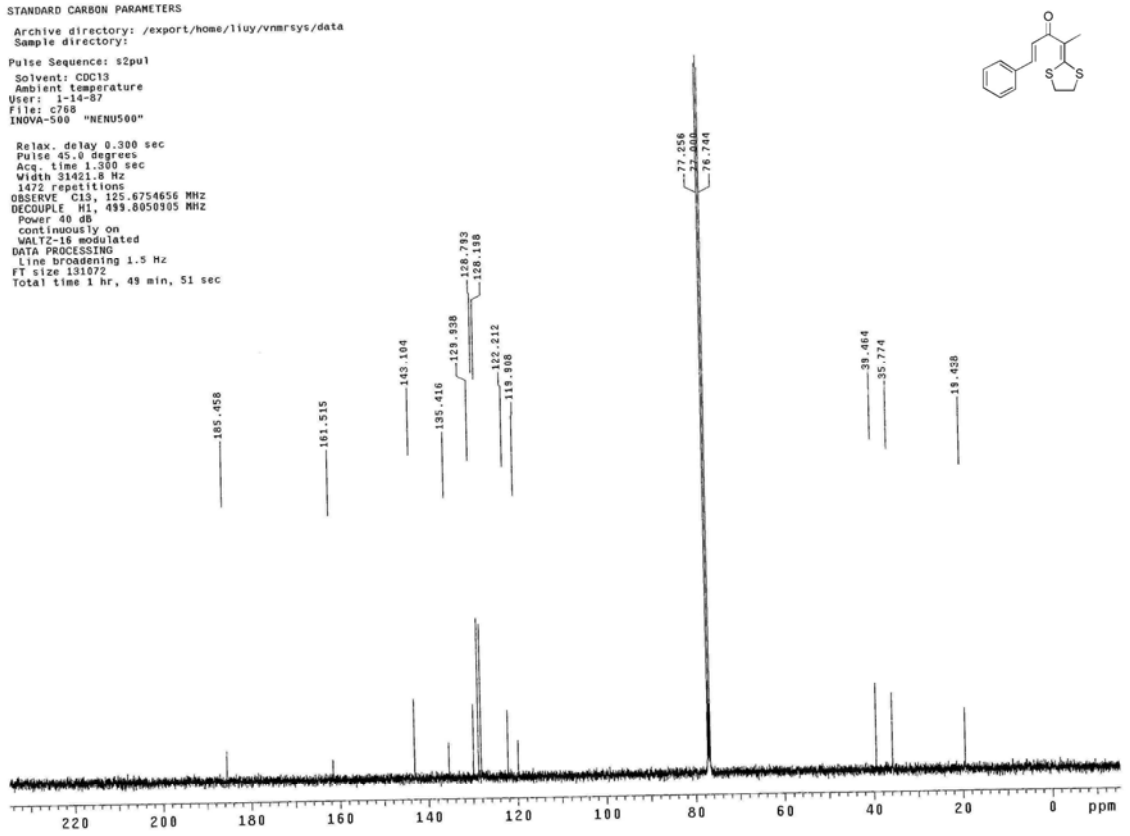

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
File: i890
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754723 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec
189.787
167.184
163.403
161.495
142.630
135.527
135.145
131.984
131.929
129.903
128.664
128.122
126.203
125.208
116.005
115.833
77.252
77.000
76.744
39.715
35.729
220
200
180
160
140
120
100
80
60
40
20
0
ppm

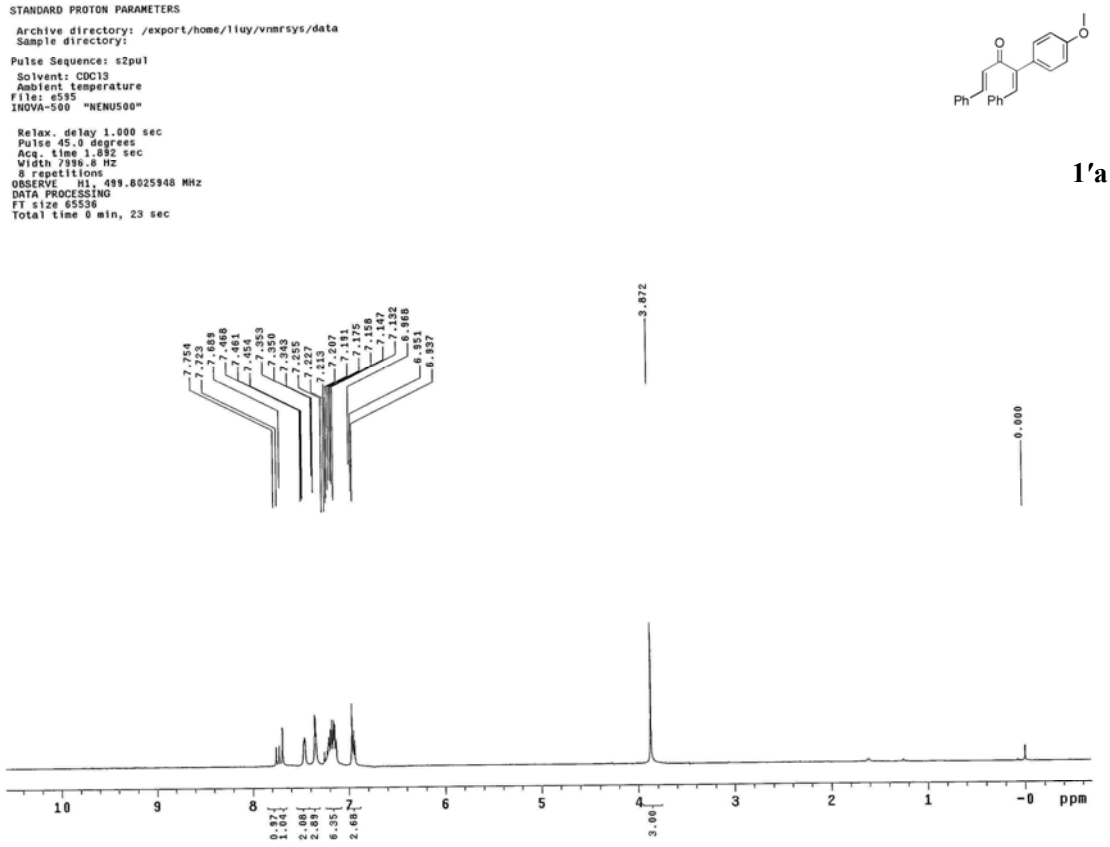

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1891
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025882 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.257
7.625
7.616
7.595
7.284
7.279
7.175
7.158
7.141
6.540
6.508
3.513
3.510
3.497
3.293
3.280
3.268
0.91
5.01
1.01
0.96
2.00
1.98
9
8
7
6
5
4
3
2
1
-0
ppm

1*l*

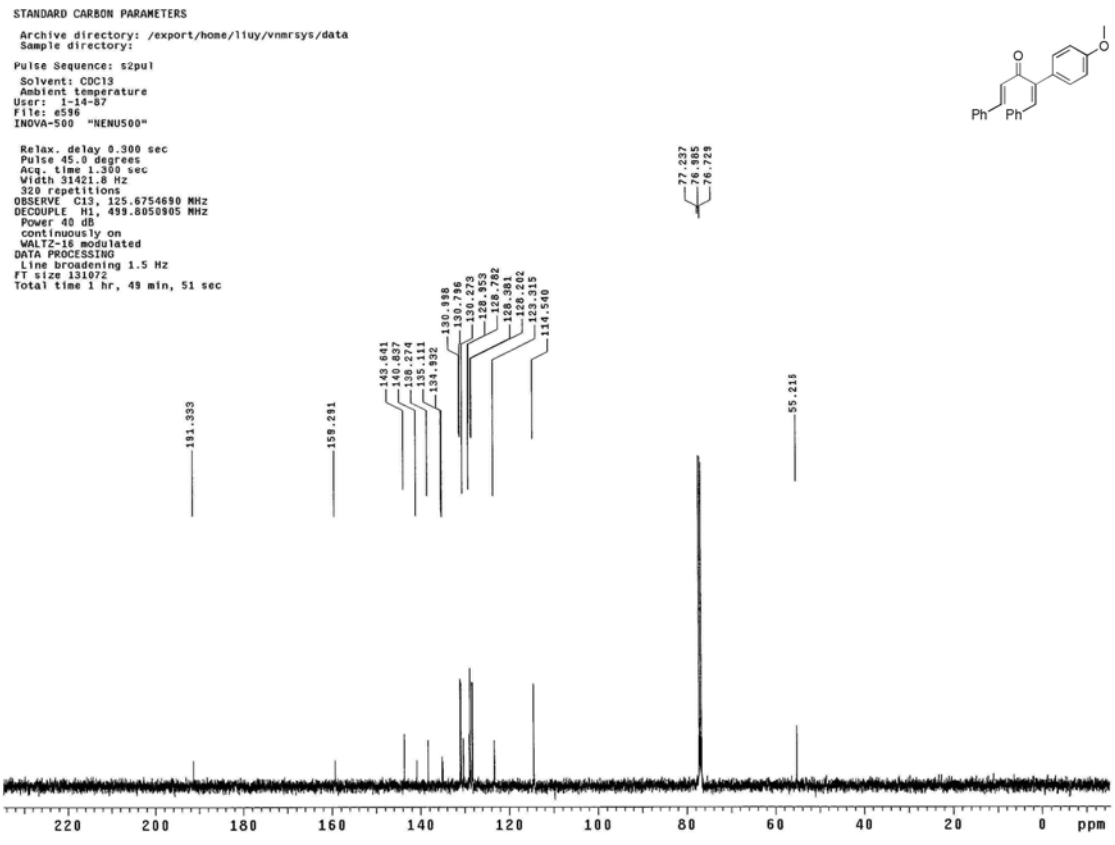

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1892
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754690 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec
183.490
167.711
163.453
161.481
141.158
135.695
135.424
133.665
132.069
131.946
131.248
128.931
126.115
123.654
116.078
115.906
77.252
76.996
76.744
39.754
35.778
220
200
180
160
140
120
100
80
60
40
20
0
ppm

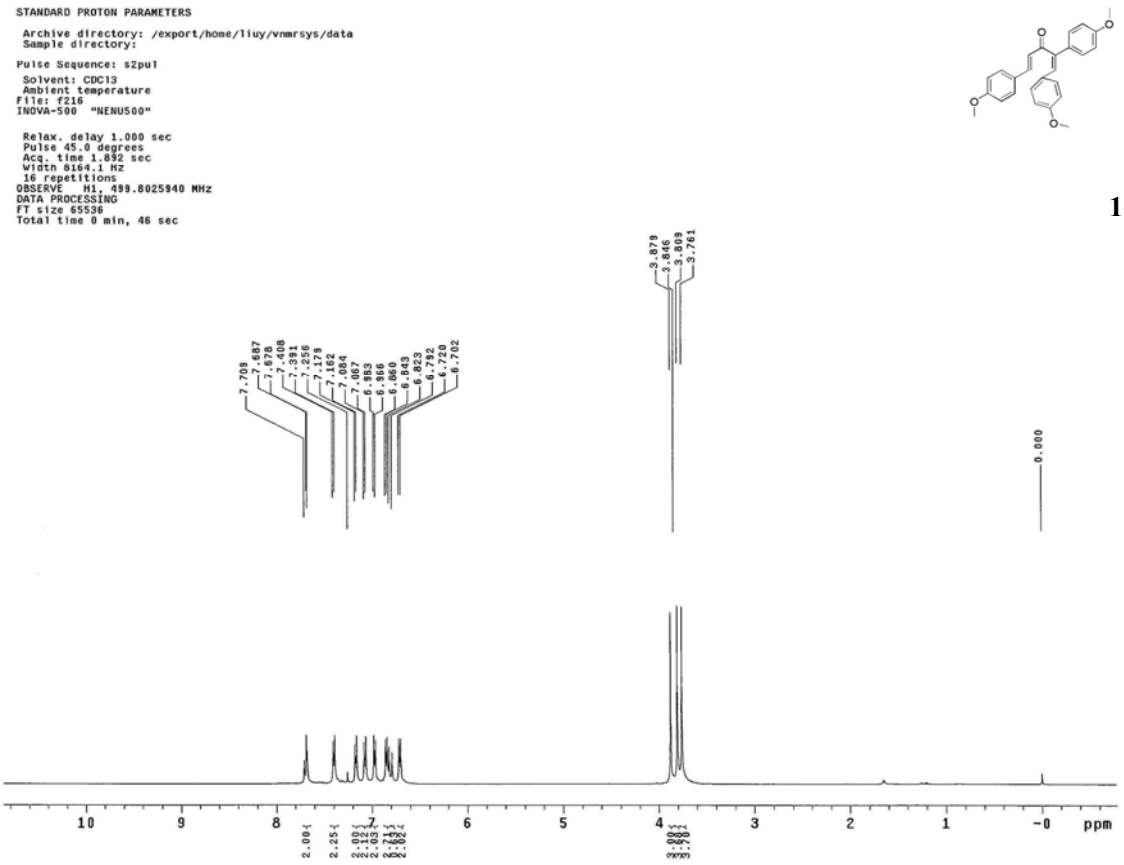

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: c682
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025902 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
1m
7.746
7.715
7.593
7.589
7.577
7.575
7.390
7.387
7.373
7.263
7.259
7.228
3.434
3.431
3.420
3.416
3.407
3.379
3.371
3.367
3.356
3.353
2.336
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.92
2.09
2.86
1.57
2.27
2.12
3.00


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: c768
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1472 repetitions
OBSERVE C13, 125.6754656 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
185.458
161.515
143.104
135.416
129.938
128.793
128.198
122.212
119.908
77.256
77.000
76.744
39.464
35.774
19.438
220
200
180
160
140
120
100
80
60
40
20
0
ppm

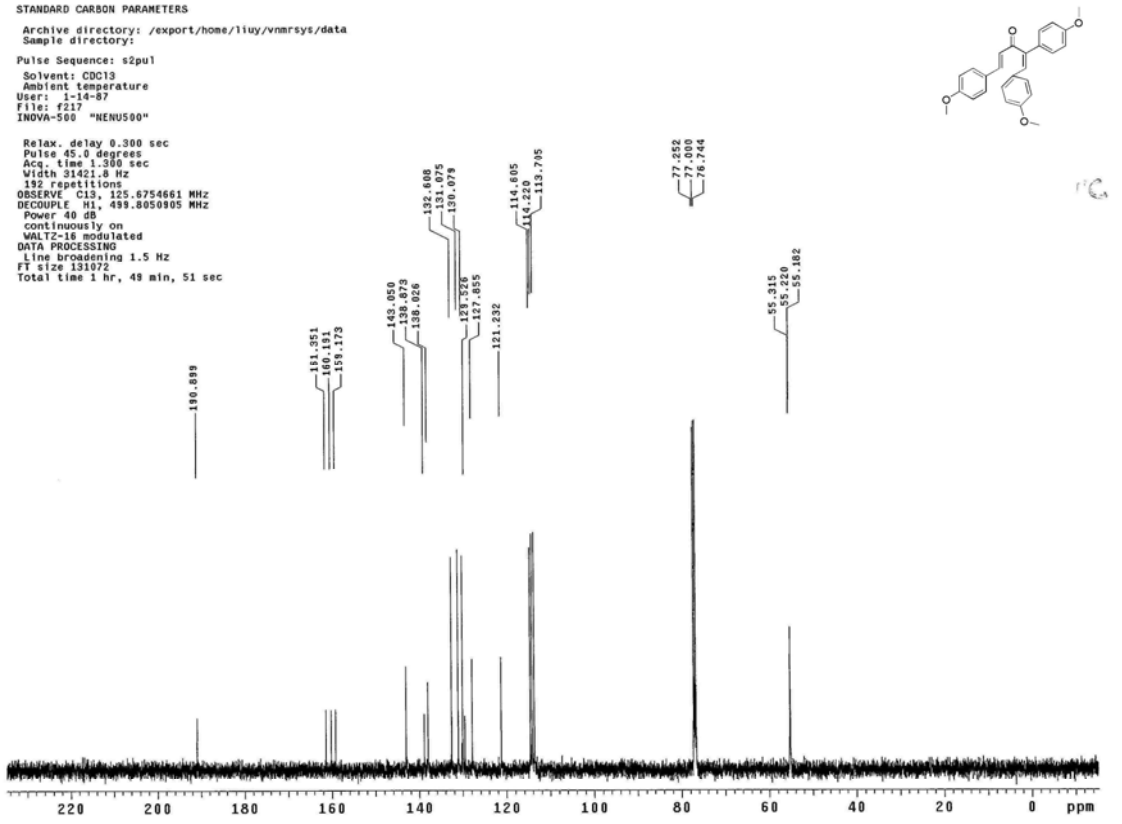

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: e595
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 HZ
8 repetitions
OBSERVE H1, 499.8025948 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.754
7.728
7.689
7.468
7.461
7.454
7.353
7.350
7.348
7.255
7.227
7.213
7.207
7.191
7.175
7.158
7.147
7.132
6.968
6.951
6.937
3.872
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.87
1.04
2.08
2.08
6.35
2.68
3.00

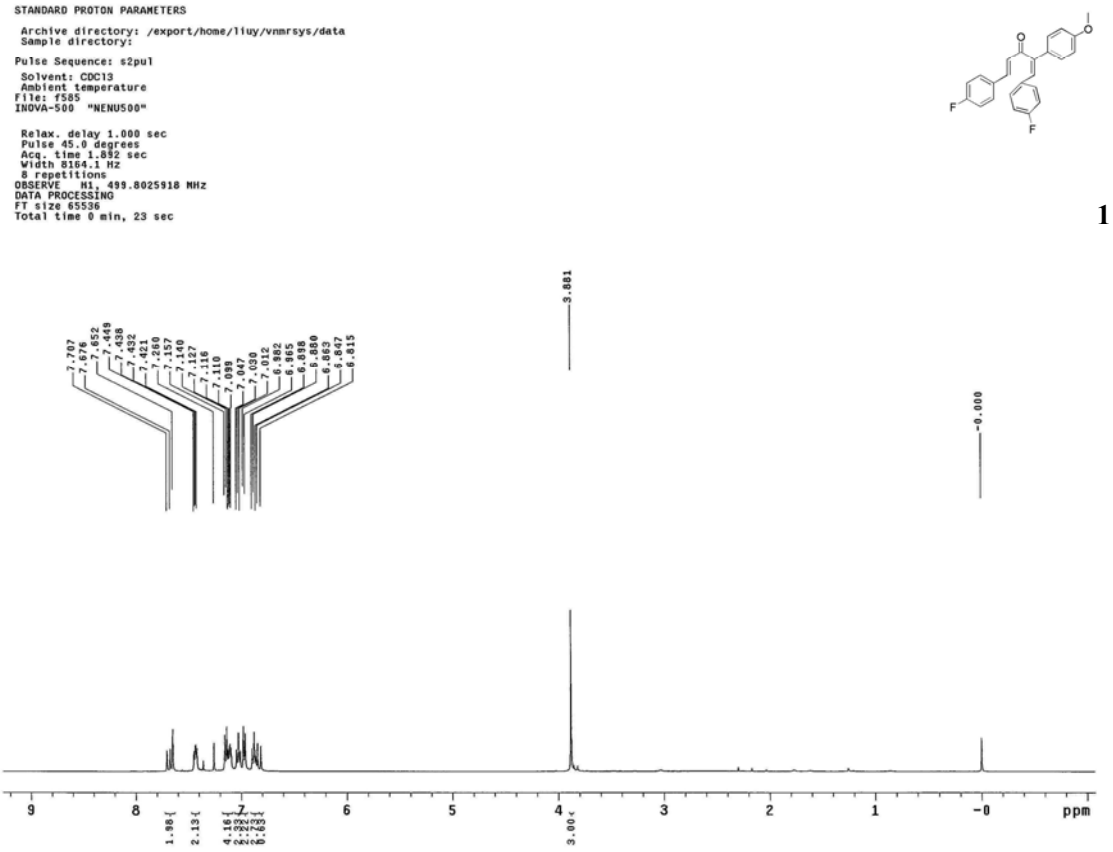
1′a


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: e596
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754690 MHz
DECOUPLE H1, 499.8050805 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
77.237
76.985
76.729
191.393
159.291
149.641
140.837
138.274
135.111
134.932
130.998
130.796
130.273
129.953
129.382
128.381
128.202
123.315
114.540
55.218
220
200
180
160
140
120
100
80
60
40
20
0
ppm

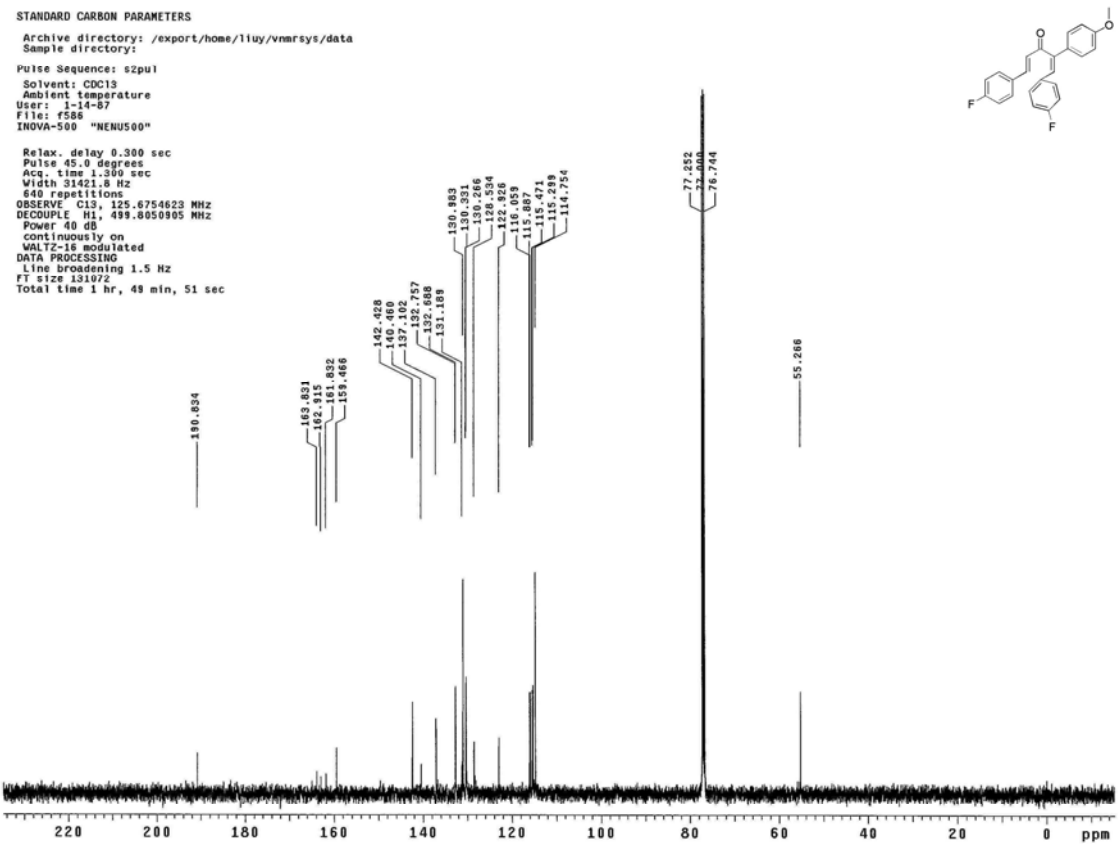

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f216
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8164.1 Hz
16 repetitions
OBSERVE H1, 499.8025940 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 46 sec
7.709
7.687
7.678
7.408
7.391
7.256
7.179
7.162
7.084
7.067
6.983
6.966
6.860
6.843
6.823
6.792
6.720
6.702
3.879
3.846
3.808
3.761
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm

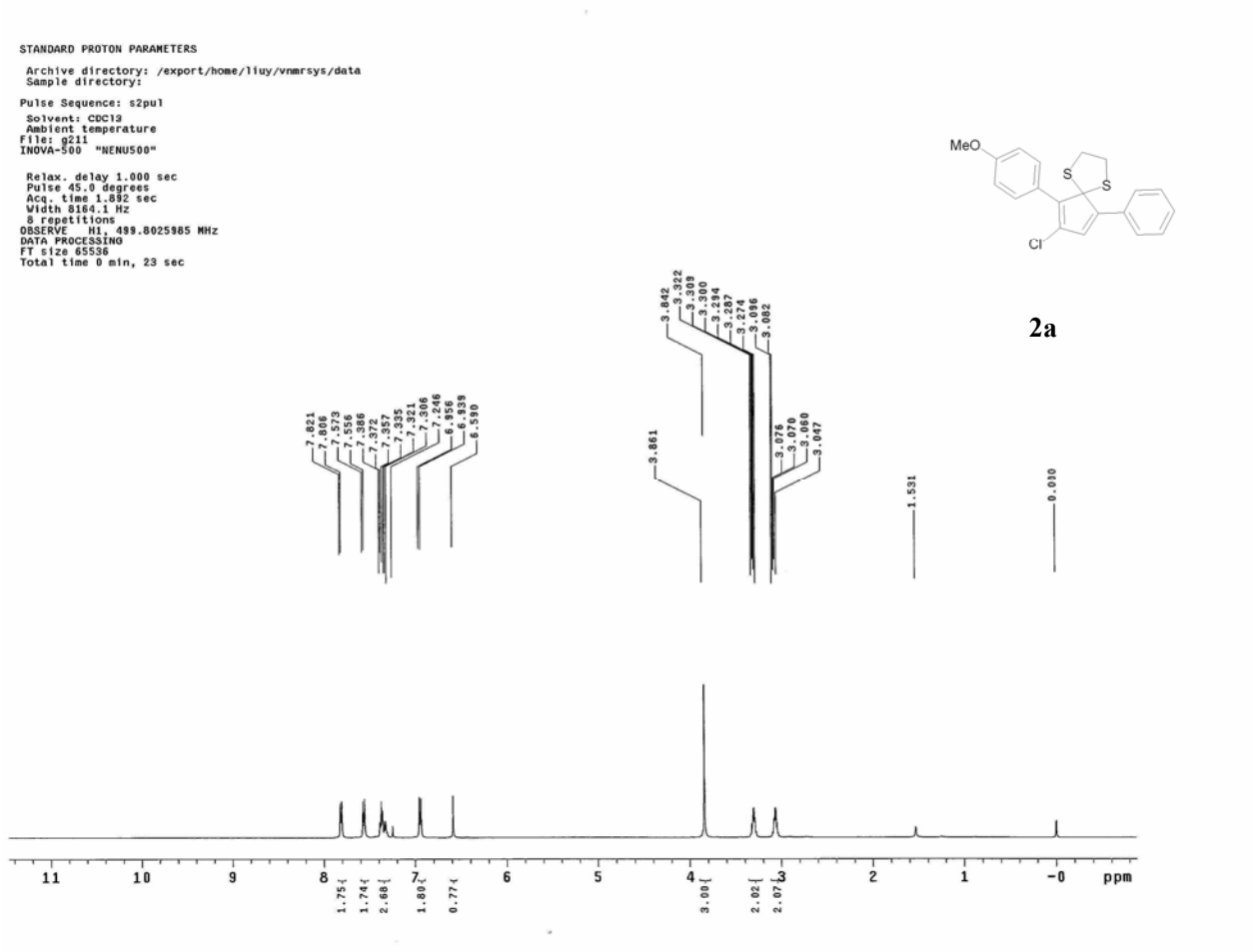
1′b

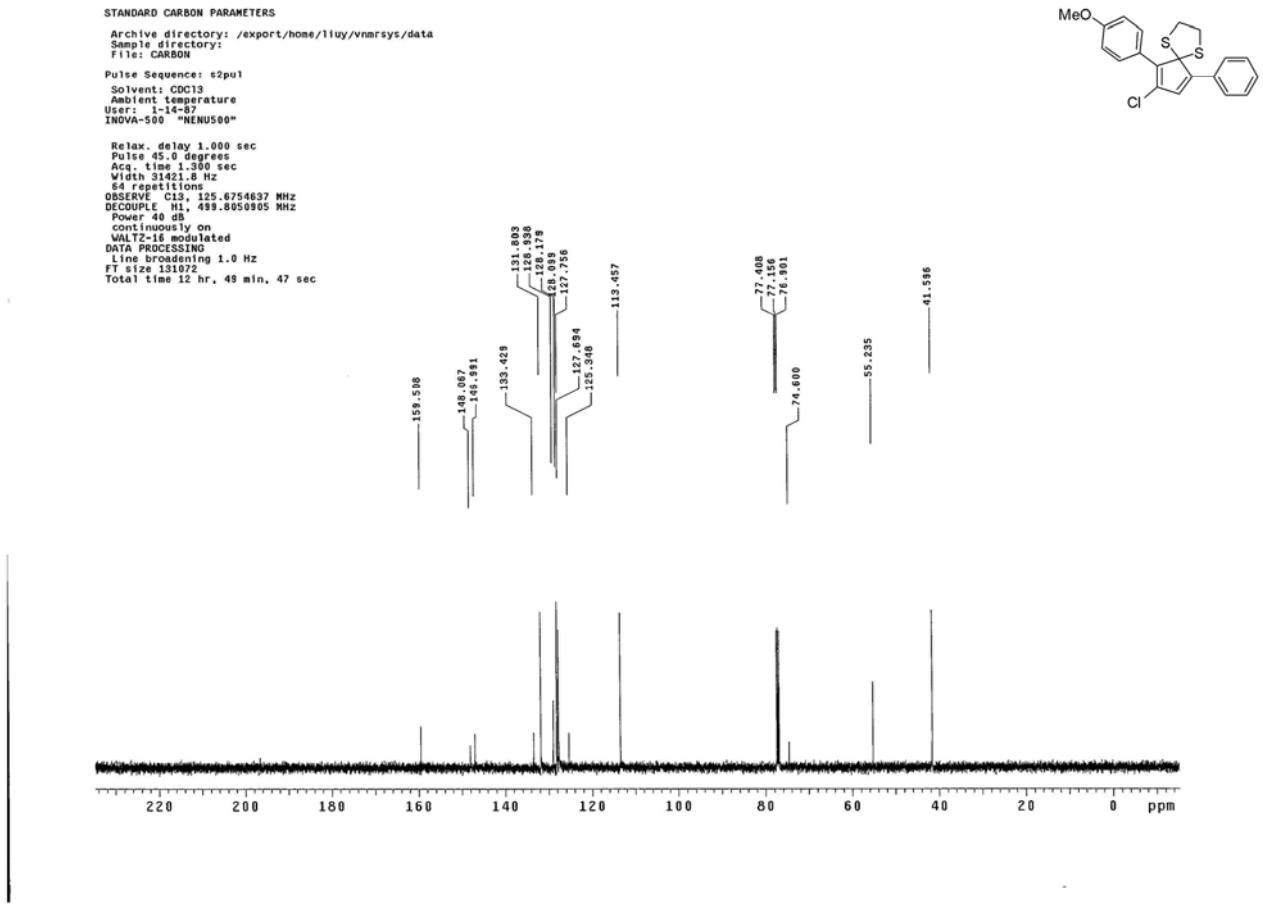

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: f217
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754661 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
190.899
161.351
160.181
159.173
143.050
138.073
130.026
132.608
131.075
130.079
129.526
127.855
121.232
114.805
114.210
113.705
77.252
77.000
76.744
55.315
55.220
55.182
220
200
180
160
140
120
100
80
60
40
20
0
ppm

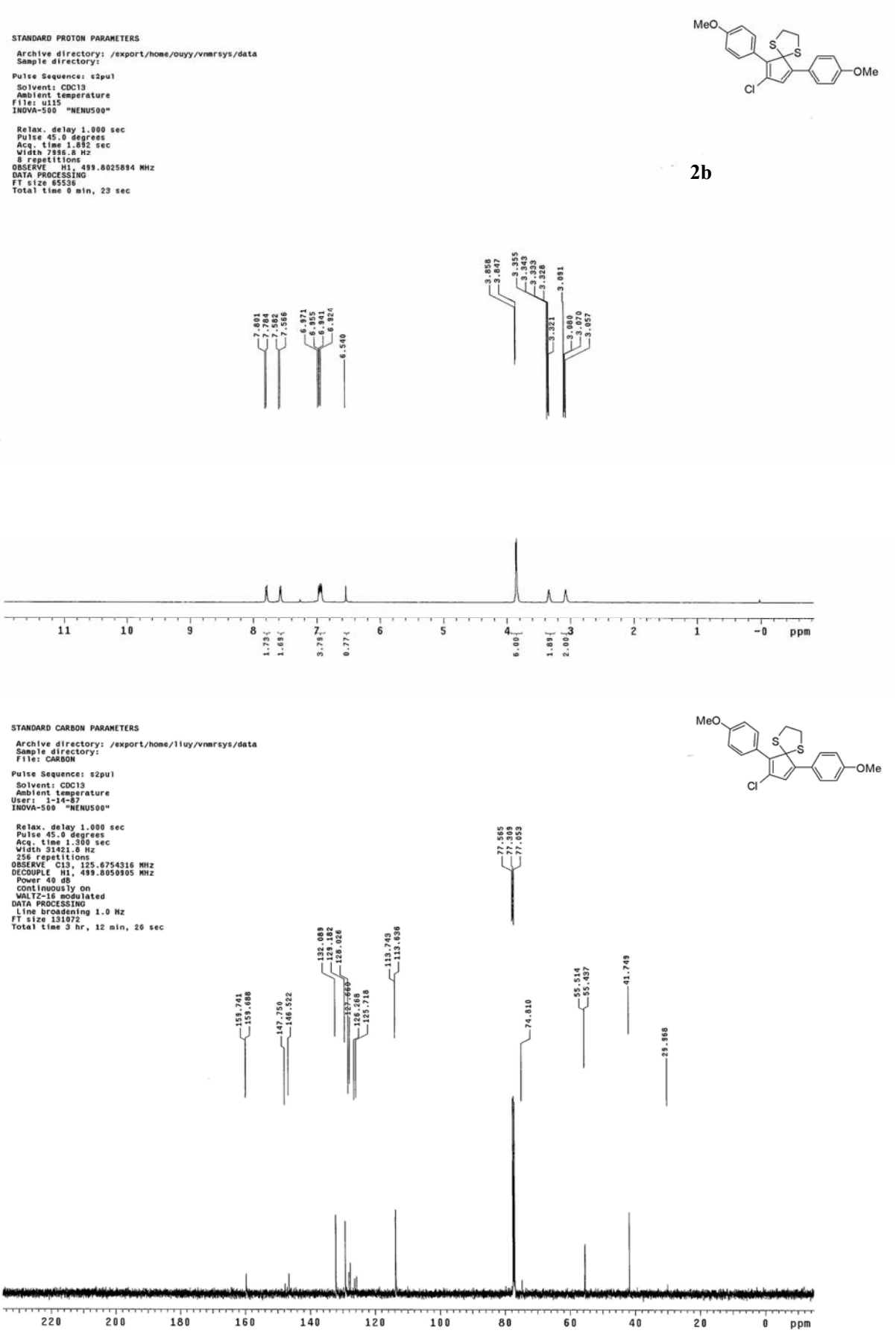

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f585
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.692 sec
Width 8164.1 Hz
8 repetitions
OBSERVE H1, 499.8025918 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.707
7.676
7.652
7.449
7.438
7.432
7.421
7.260
7.157
7.140
7.127
7.116
7.110
7.083
7.067
7.030
7.012
6.882
6.865
6.898
6.880
6.863
6.847
6.815
3.881
-0.000
9
8
7
6
5
4
3
2
1
-0
ppm
1.98
2.13
4.16
2.33
2.22
0.63
3.00

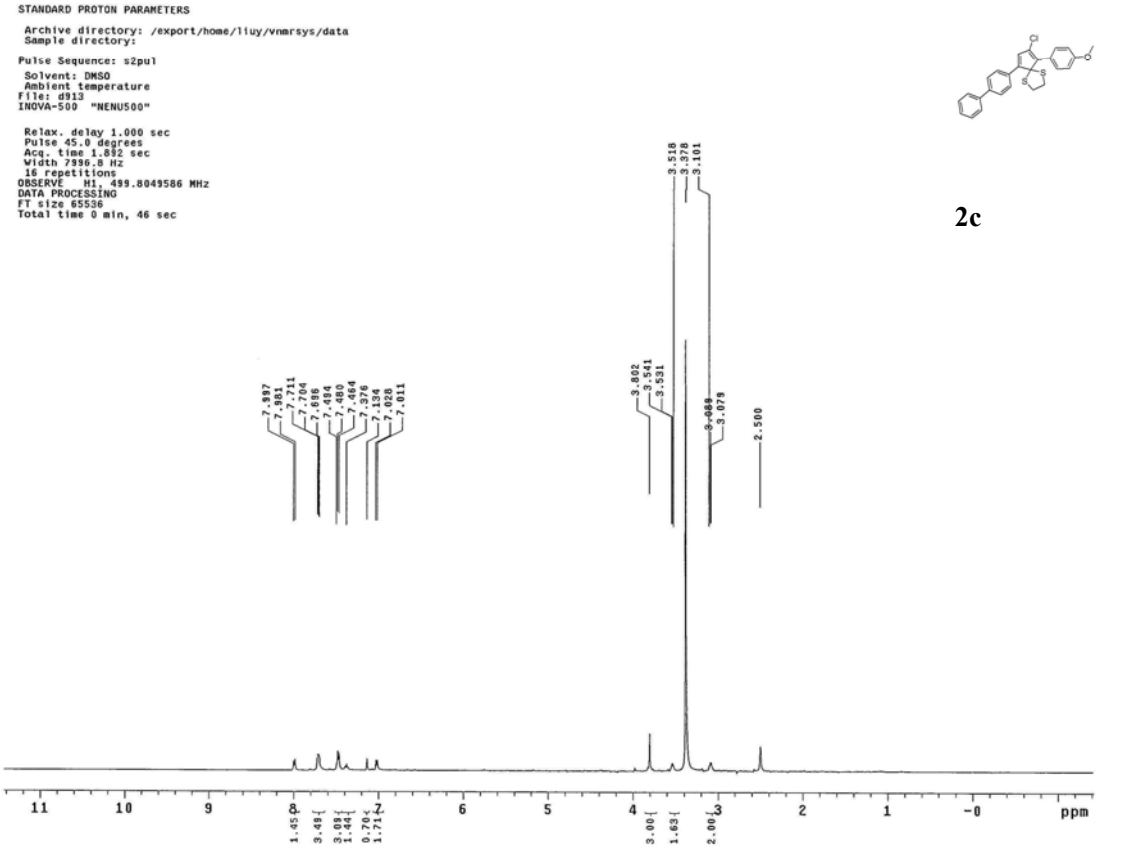
1′c

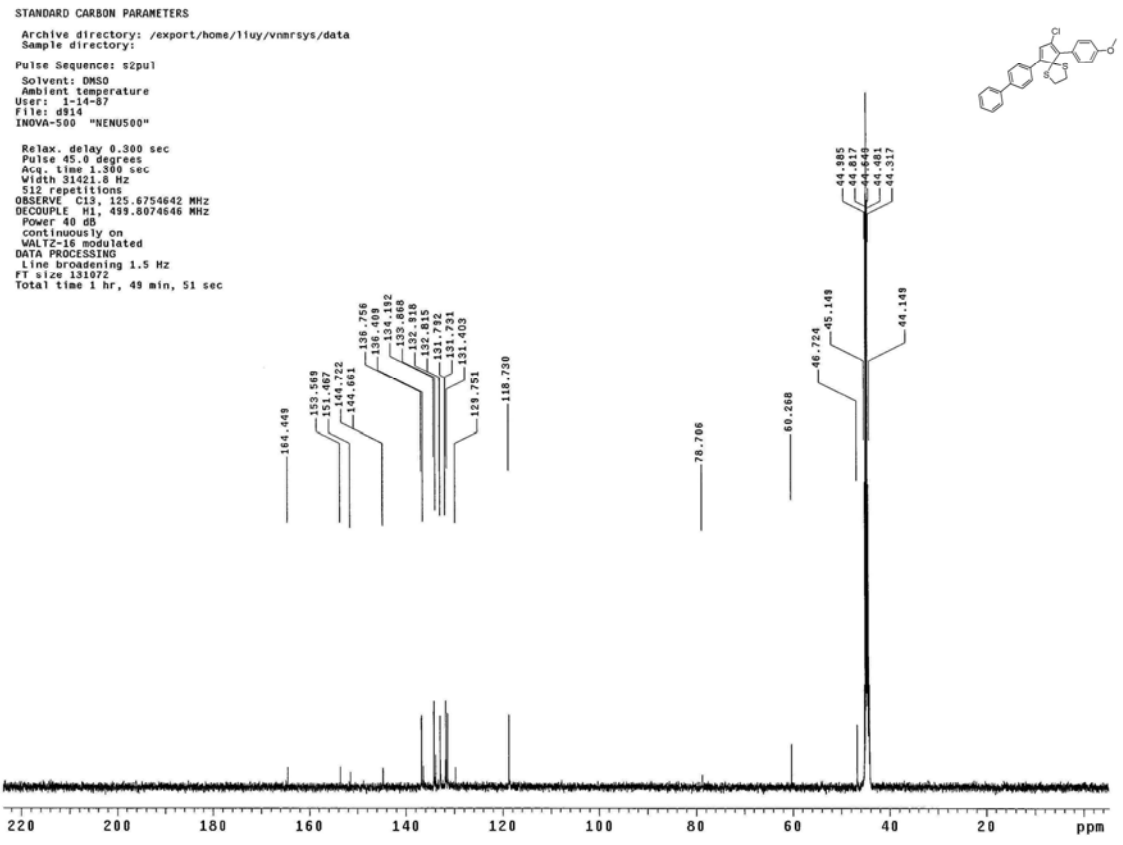

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: f586
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
640 repetitions
OBSERVE C13, 125.6754623 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
190.834
163.831
162.915
161.832
159.466
142.428
140.460
137.102
132.757
132.668
131.188
130.983
130.331
130.266
128.534
122.826
116.059
115.887
115.471
115.218
114.754
77.252
77.000
76.744
55.266
220
200
180
160
140
120
100
80
60
40
20
0
ppm

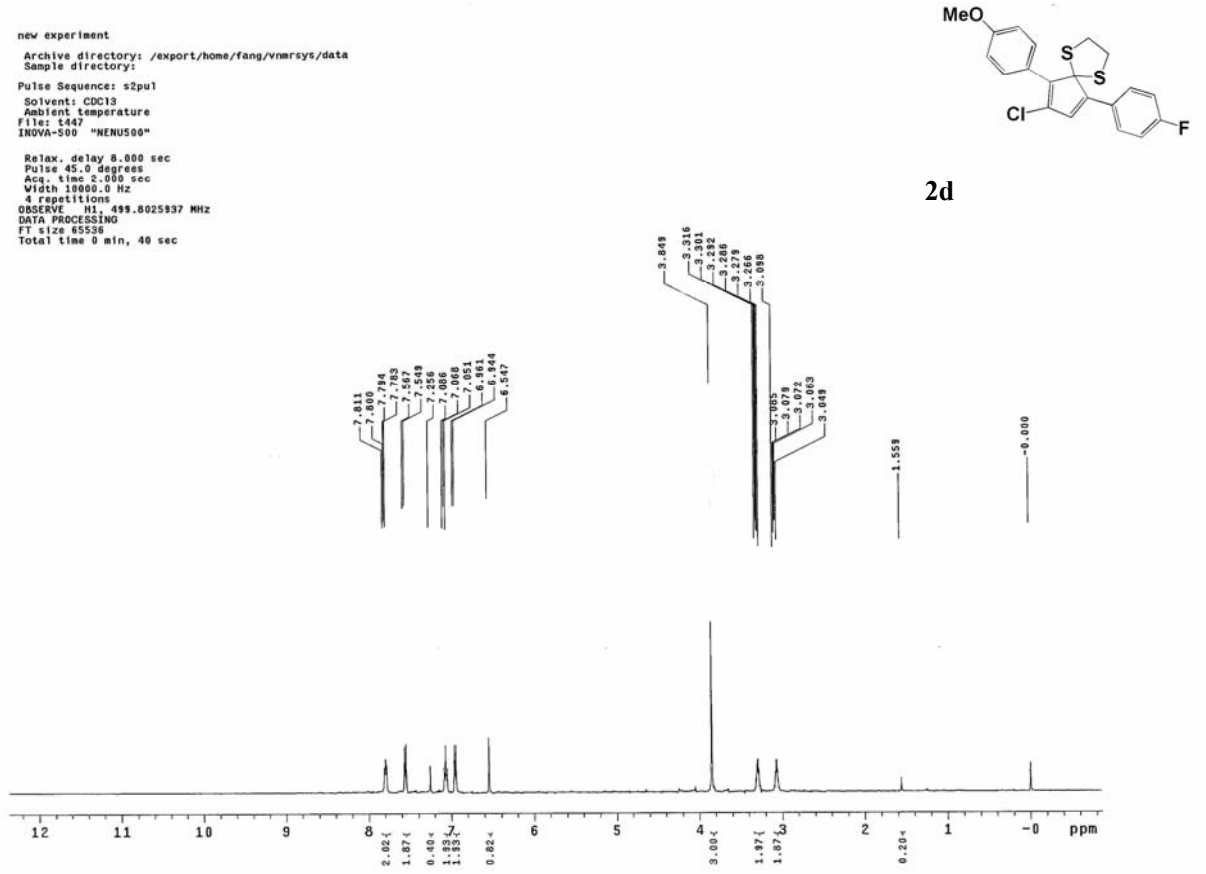

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: g211
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8164.1 Hz
8 repetitions
OBSERVE H1, 499.8025985 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
S
S
Cl
2a
7.821
7.806
7.573
7.556
7.386
7.372
7.357
7.351
7.331
7.306
7.246
6.956
6.839
6.590
3.842
3.322
3.309
3.300
3.294
3.287
3.274
3.096
3.082
3.861
3.076
3.070
3.060
3.047
1.531
0.010
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.75
1.74
2.68
1.80
0.77
3.00
2.02
2.07

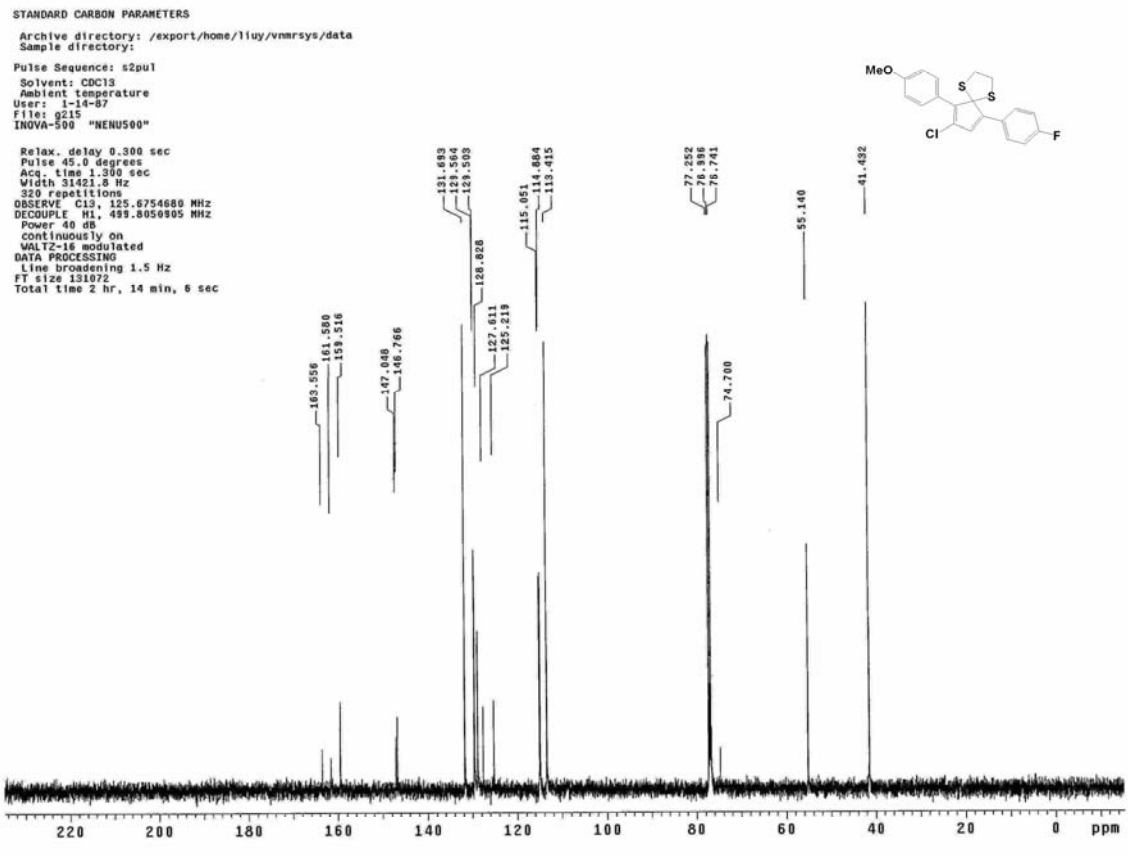

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754637 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 12 hr, 48 min, 47 sec
MeO
S
S
Cl
159.508
148.067
146.891
133.428
131.803
128.938
128.179
126.098
127.756
127.694
125.348
113.457
77.408
77.156
76.901
74.600
55.235
41.586
220
200
180
160
140
120
100
80
60
40
20
0
ppm

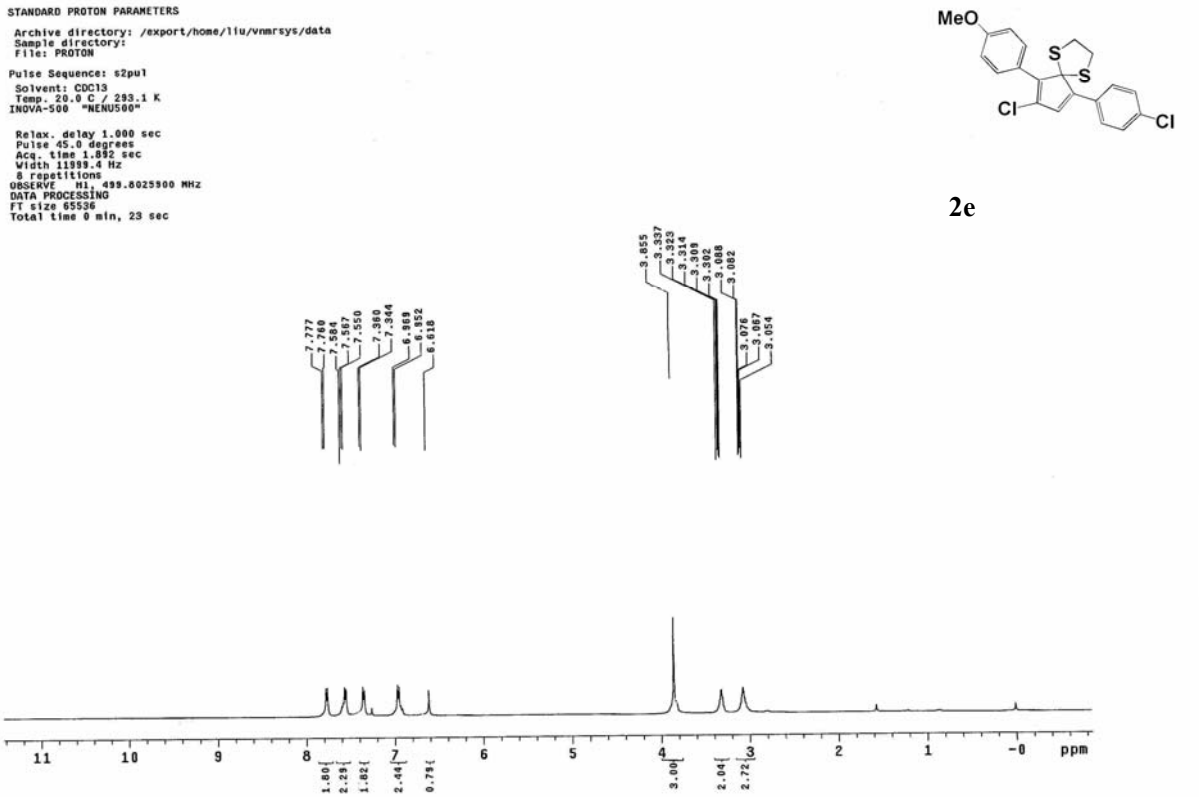

STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: u115
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
OMe
Cl
S
S
2b
7.801
7.784
7.582
7.566
6.971
6.955
6.941
6.924
6.549
3.858
3.847
3.355
3.343
3.328
3.321
3.091
3.080
3.070
3.057
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.79
1.68
3.78
0.77
6.00
1.89
2.00
STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/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 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
MeO
OMe
Cl
S
S
77.565
77.309
77.053
159.741
159.688
147.750
146.522
132.089
129.182
128.026
127.660
126.168
125.718
113.743
113.636
74.810
55.514
55.437
41.749
29.968
220
200
180
160
140
120
100
80
60
40
20
0
ppm

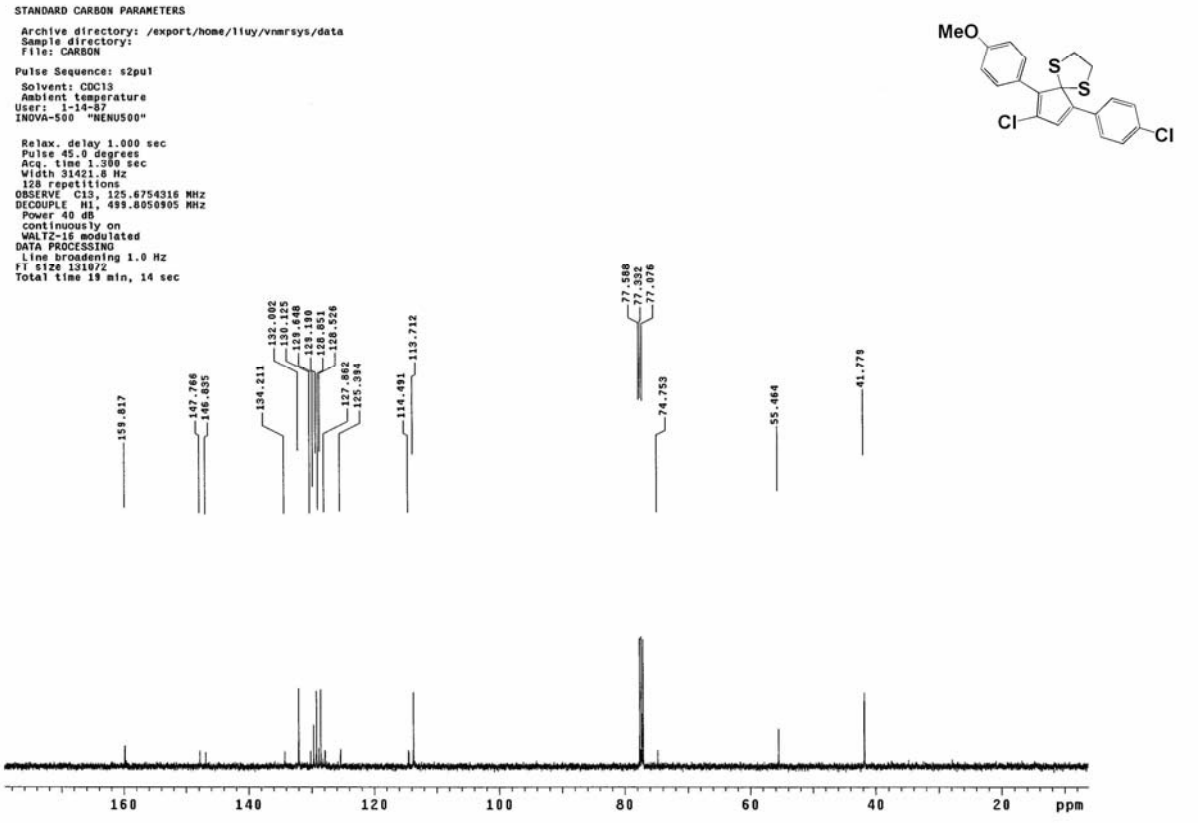

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: d913
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
16 repetitions
OBSERVE H1, 499.8049586 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 46 sec
2c
7.997
7.981
7.711
7.704
7.696
7.464
7.480
7.464
7.376
7.134
7.028
7.011
3.802
3.541
3.531
3.518
3.378
3.101
3.089
3.079
2.500
ppm

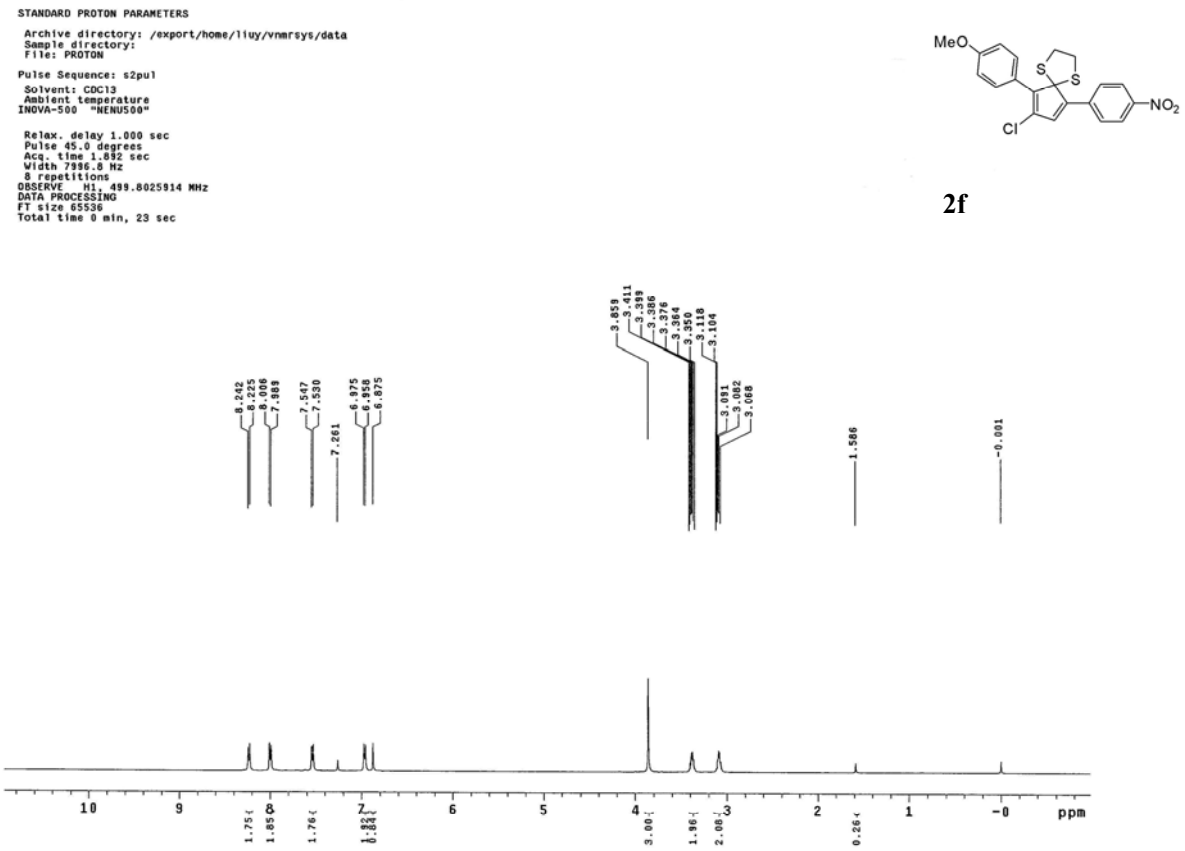

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
File: d914
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
512 repetitions
OBSERVE C13, 125.6754642 MHz
DECOUPLE H1, 499.8074646 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
164.449
153.569
151.467
144.722
144.661
136.756
136.409
134.192
133.868
132.418
132.015
131.782
131.731
131.403
129.751
118.730
78.706
60.268
46.724
45.149
44.985
44.817
44.649
44.481
44.317
44.149
ppm

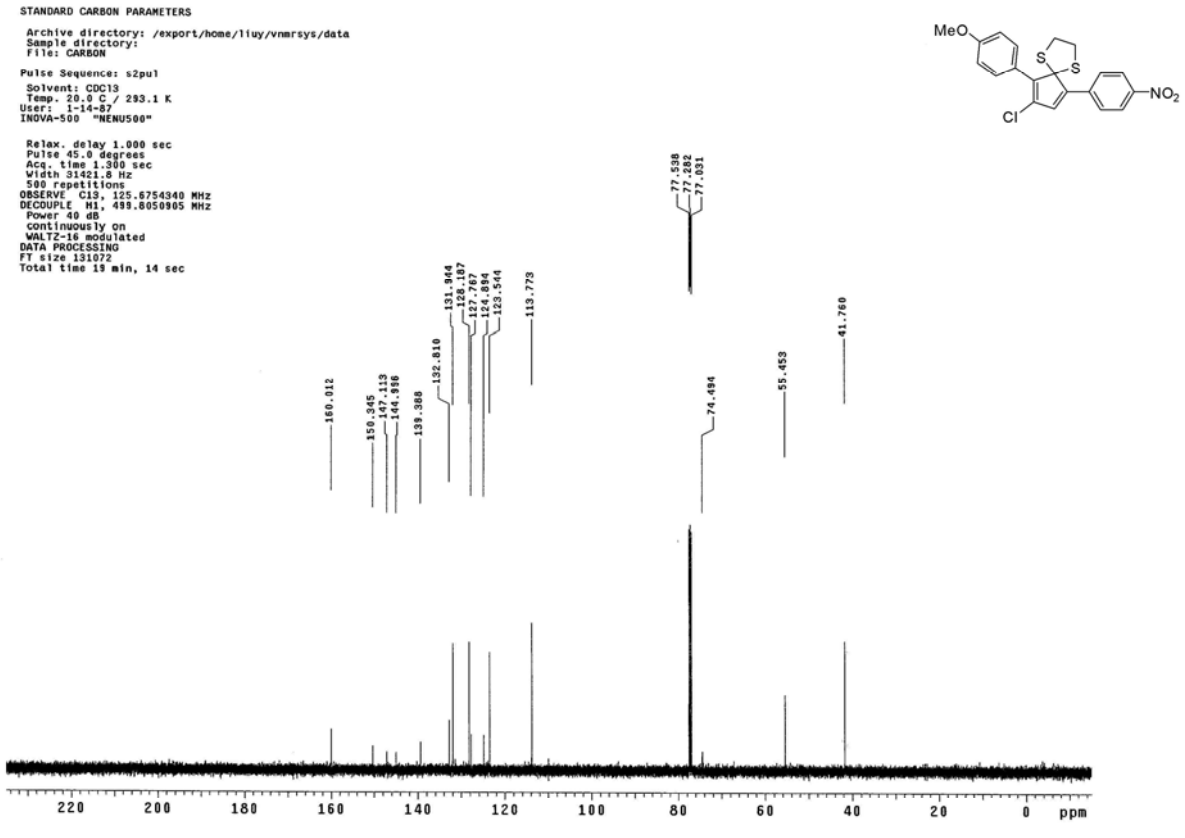

new experiment
Archive directory: /export/home/fang/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: t447
INOVA-500 "NENU500"
Relax. delay 8.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 10000.0 Hz
4 repetitions
OBSERVE H1, 499.8025937 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 40 sec
MeO
S
S
Cl
F
2d
ppm

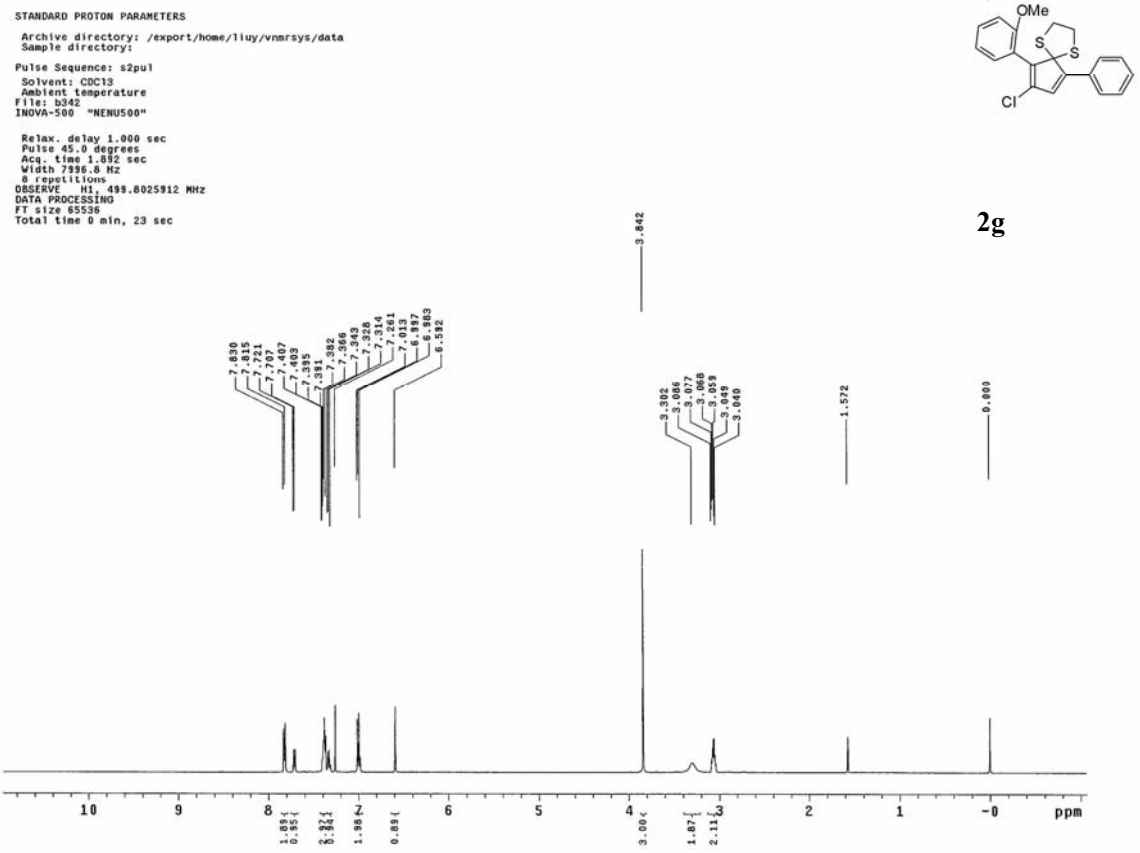

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: g215
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754680 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 14 min, 8 sec
MeO
S
S
Cl
F
ppm

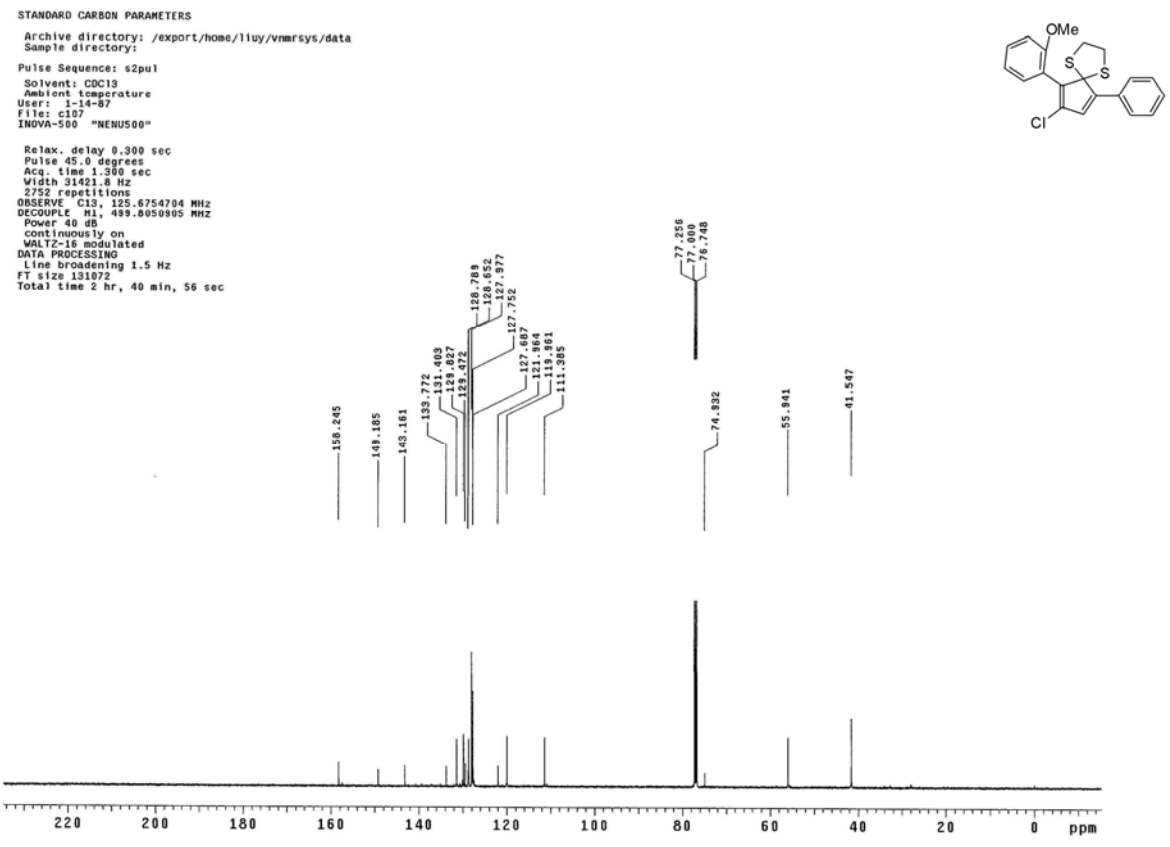

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 11999.4 Hz
8 repetitions
OBSERVE H1, 499.8025900 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
S
S
Cl
Cl
2e
7.777
7.760
7.584
7.567
7.550
7.380
7.364
6.868
6.852
6.618
3.855
3.337
3.323
3.314
3.309
3.302
3.088
3.082
3.076
3.067
3.054
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.86
2.29
1.82
2.44
0.79
3.00
2.04
2.72

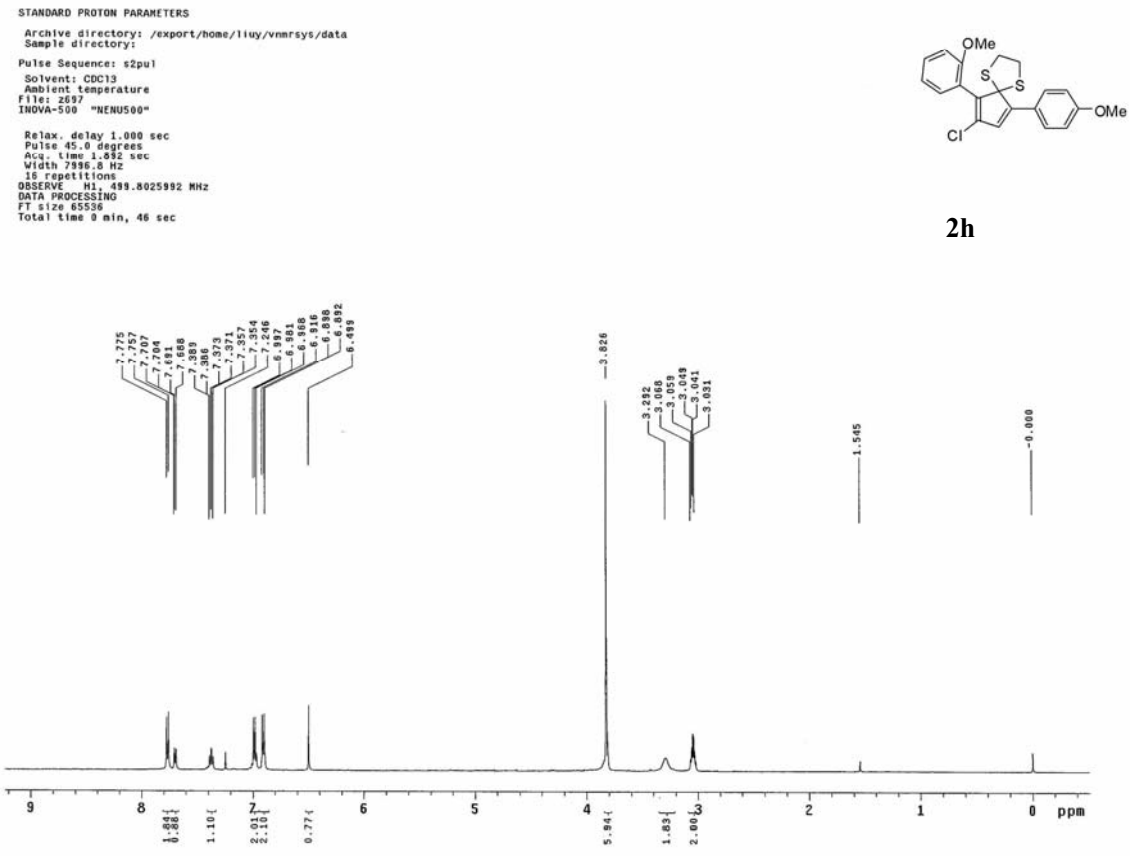

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 19 min, 14 sec
MeO
S
S
Cl
Cl
159.817
147.766
146.835
134.211
132.002
130.125
129.648
128.510
128.351
128.526
127.862
125.384
114.491
113.712
77.588
77.332
77.076
74.753
55.464
41.779
160
140
120
100
80
60
40
20
ppm

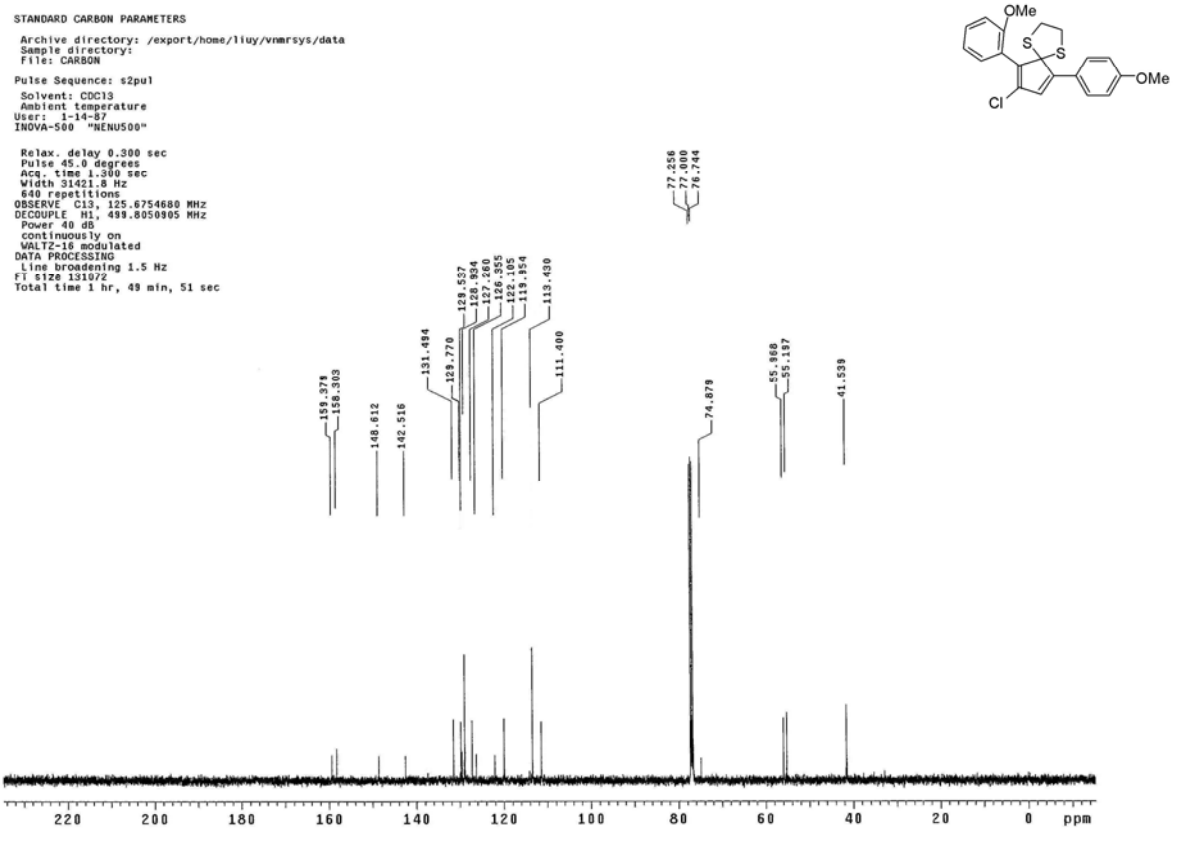

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7896.8 Hz
8 repetitions
OBSERVE H1, 499.8025914 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
S
S
NO2
Cl
2f
8.242
8.225
8.006
7.988
7.547
7.530
7.261
6.975
6.958
6.875
3.859
3.411
3.399
3.386
3.376
3.364
3.351
3.338
3.104
3.091
3.082
3.068
1.586
-0.001
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.75
1.85
1.76
1.82
0.84
3.00
1.96
2.08
0.26

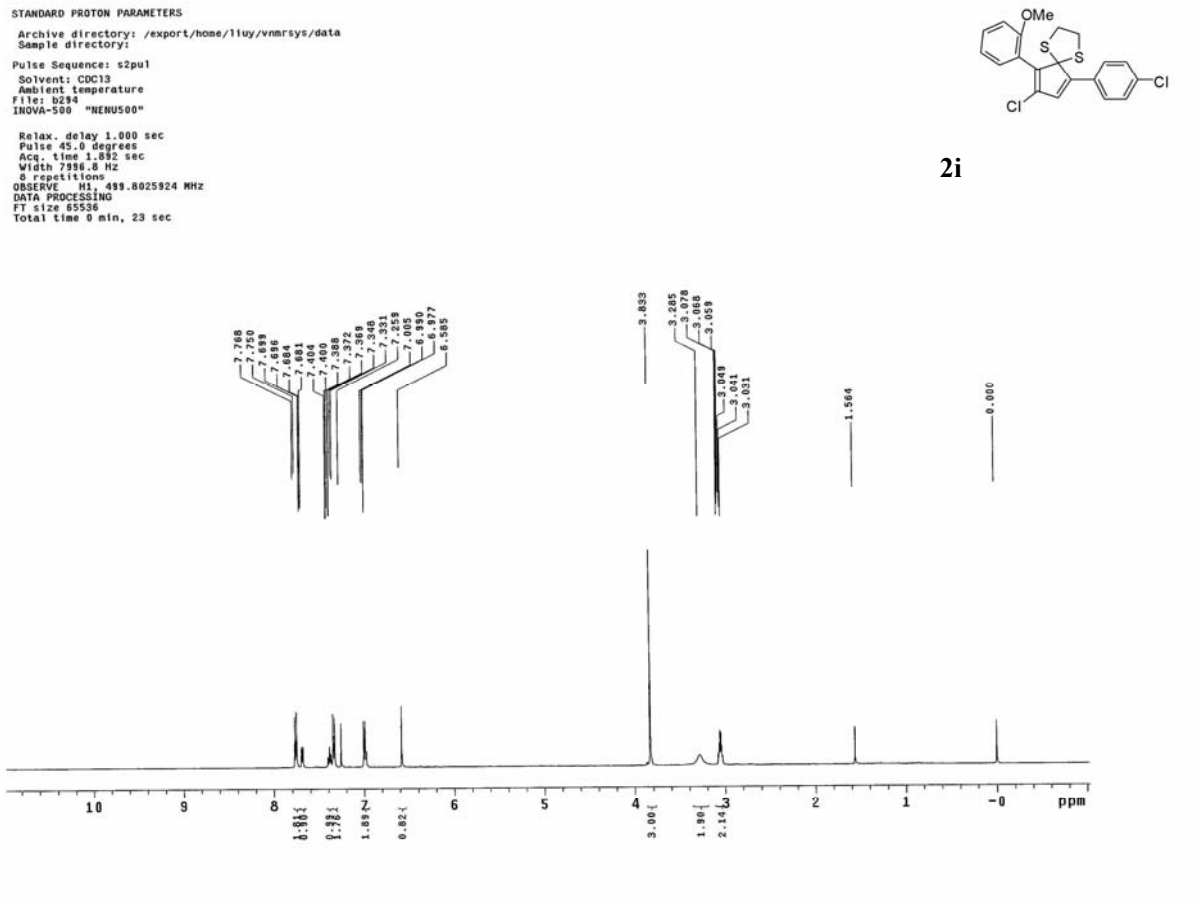

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
500 repetitions
OBSERVE C13, 125.6754340 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 131072
Total time 19 min, 14 sec
MeO
S
S
NO2
Cl
77.538
77.282
77.031
160.012
150.345
147.113
144.996
139.388
132.810
131.944
128.187
127.767
124.884
123.544
113.773
74.494
55.453
41.760
220
200
180
160
140
120
100
80
60
40
20
0
ppm

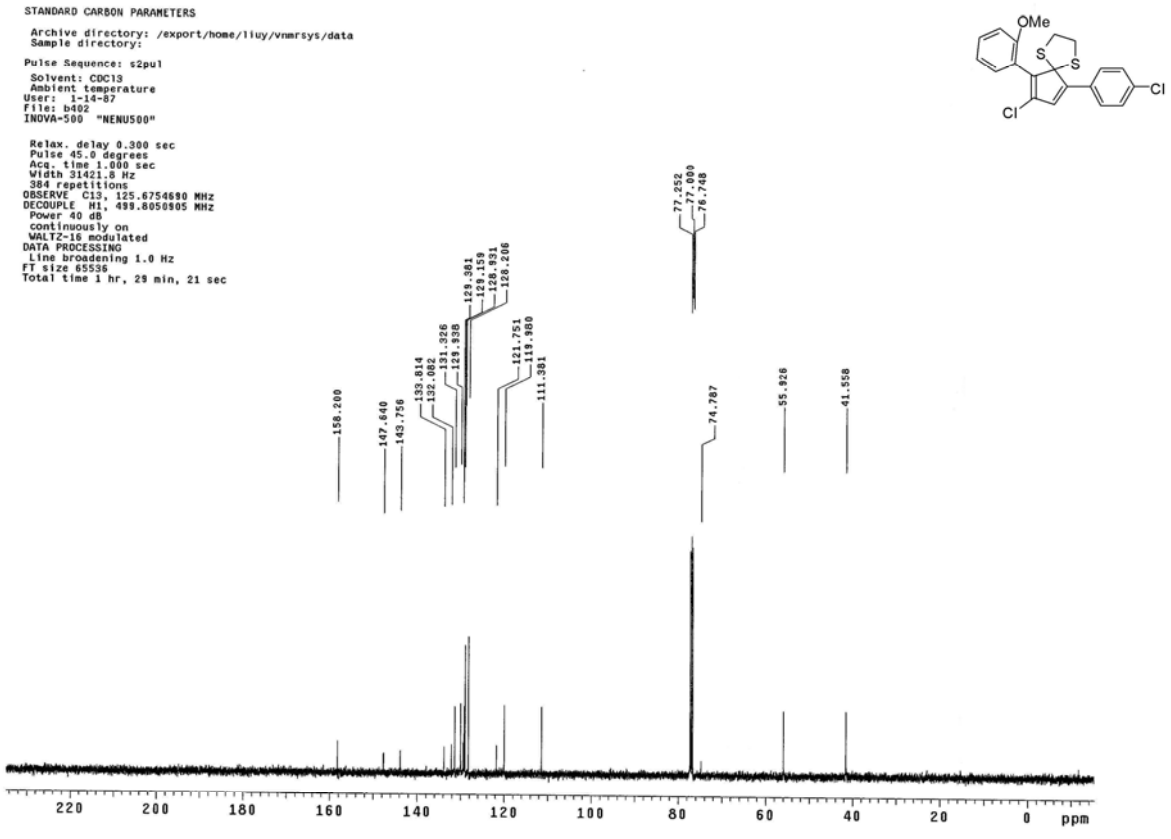

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b342
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.692 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025912 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
OMe
S
S
Cl
2g
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: c107
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
2752 repetitions
OBSERVE C13, 125.6754704 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec
OMe
S
S
Cl
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: z697
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, 499.8025992 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 46 sec
OMe
S
S
OMe
Cl
2h
7.775
7.757
7.707
7.704
7.691
7.688
7.389
7.386
7.373
7.371
7.357
7.354
7.246
6.997
6.881
6.866
6.818
6.816
6.892
6.499
3.826
3.282
3.068
3.059
3.049
3.041
3.031
1.545
0.000
9
8
7
6
5
4
3
2
1
0
ppm
1.84
0.88
1.10
2.01
2.10
0.77
5.94
1.83
2.00


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
640 repetitions
OBSERVE C13, 125.6754680 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
OMe
S
S
OMe
Cl
77.256
77.000
76.744
159.379
158.303
148.612
142.516
131.494
129.770
129.537
128.934
127.260
126.355
122.165
119.954
113.430
111.400
74.879
55.968
55.187
41.539
220
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b294
INOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025924 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
OMe
S
S
Cl
Cl
2i
7.768
7.750
7.699
7.686
7.684
7.681
7.404
7.400
7.392
7.369
7.348
7.331
7.259
7.005
6.597
6.585
3.833
3.085
3.078
3.068
3.059
3.049
3.041
3.031
1.564
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm

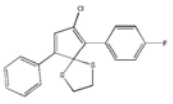

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: b402
INOVA-500 "NENUS00"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 31421.8 Hz
384 repetitions
OBSERVE C13, 125.6754690 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 29 min, 21 sec
OMe
S
S
Cl
Cl
77.252
77.000
76.748
129.381
129.159
128.931
128.206
133.814
132.082
131.326
129.938
121.751
119.980
111.381
158.200
147.640
143.756
74.787
55.926
41.558
220
200
180
160
140
120
100
80
60
40
20
0
ppm

STANDARD PROTON PARAMETERS

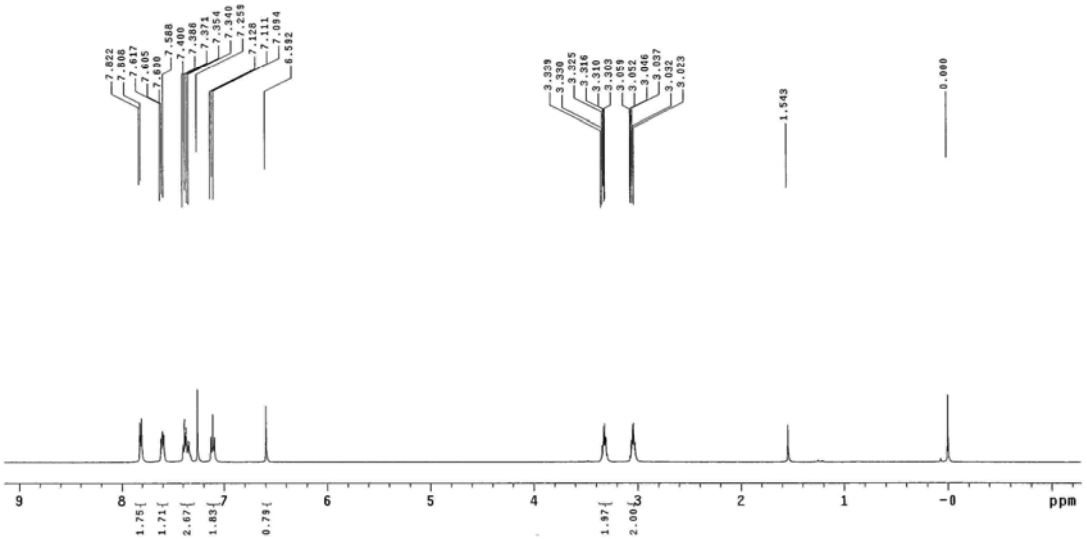
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1886
INOVA-500 "NENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025921 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec



2j

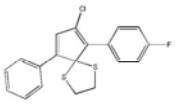


STANDARD CARBON PARAMETERS

Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1902
INOVA-500 "NENU500"

Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
6000 repetitions
OBSERVE C13, 125.6754627 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec



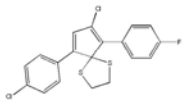
STANDARD PROTON PARAMETERS

Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:

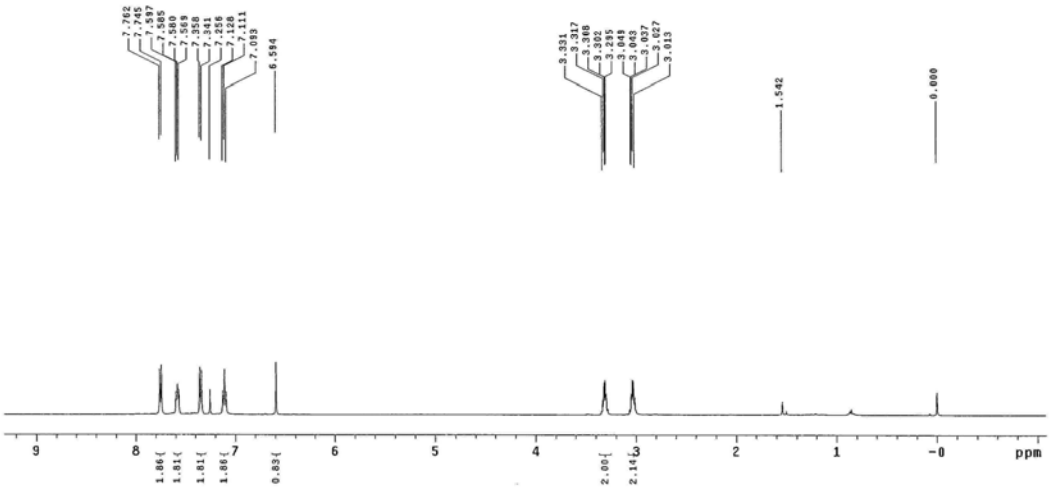
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 1885
INOVA-500 "NENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025936 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec



2k



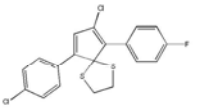
STANDARD CARBON PARAMETERS

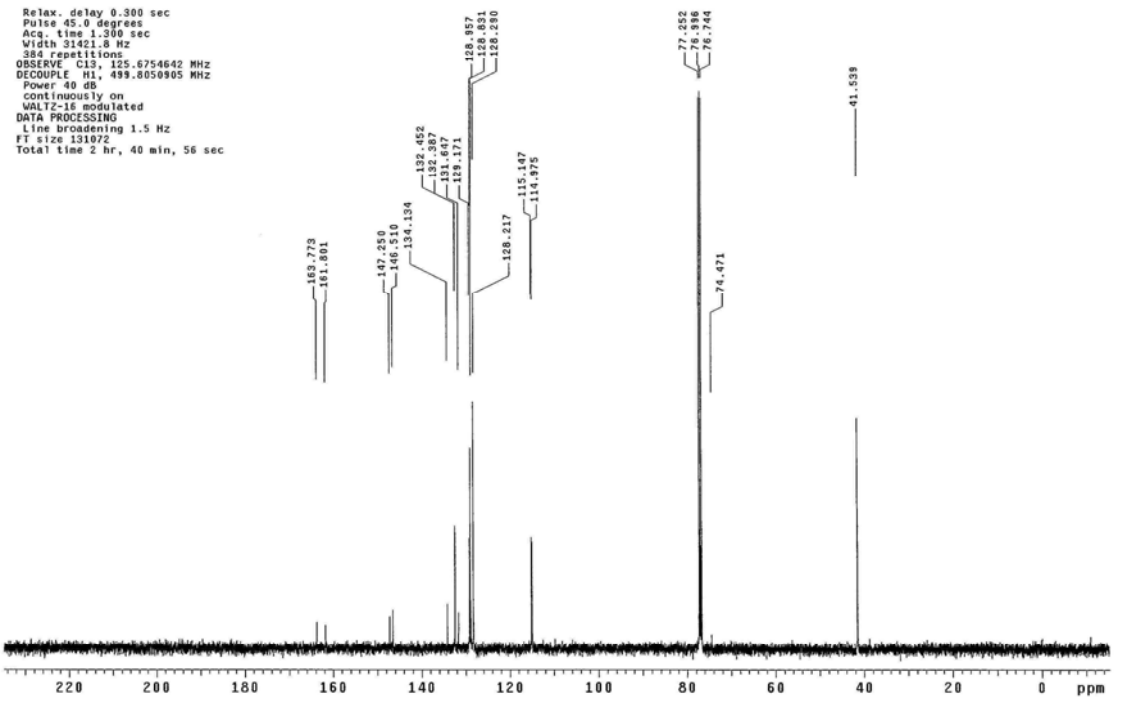
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:

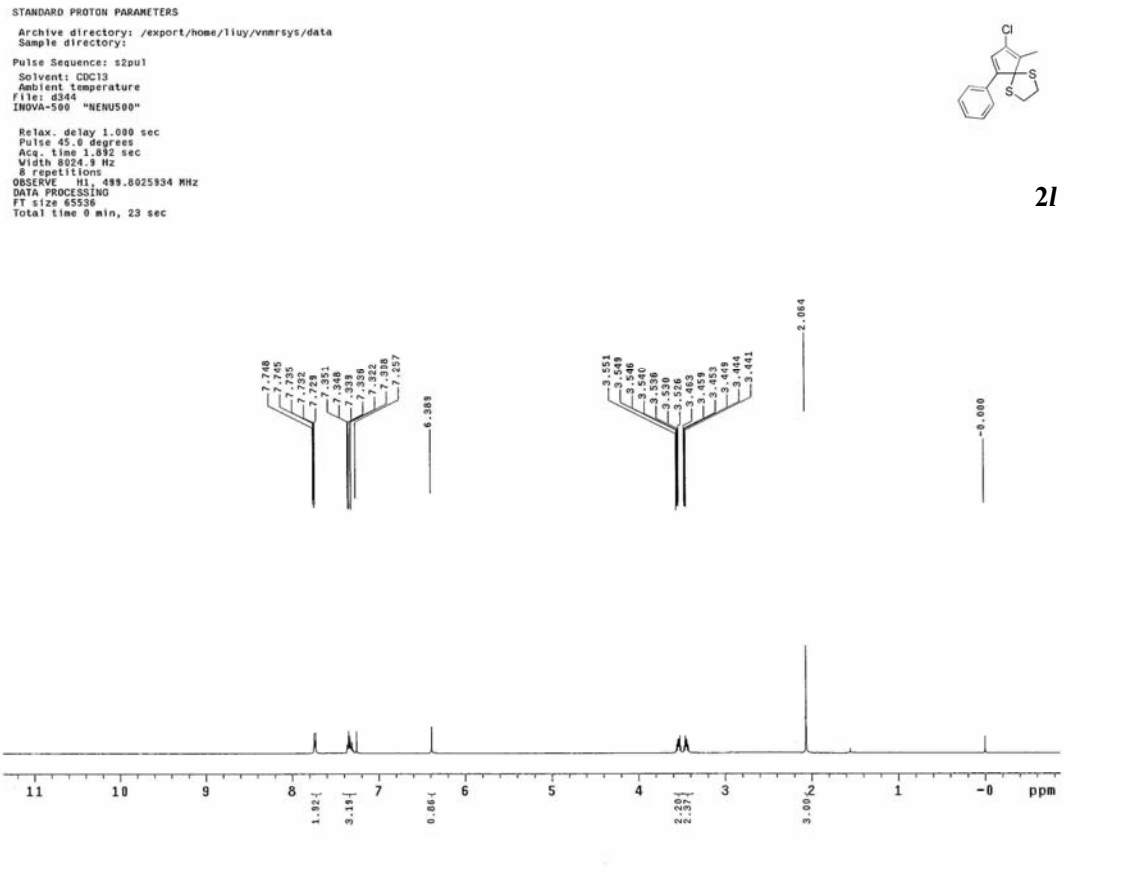
Pulse Sequence: s2pul

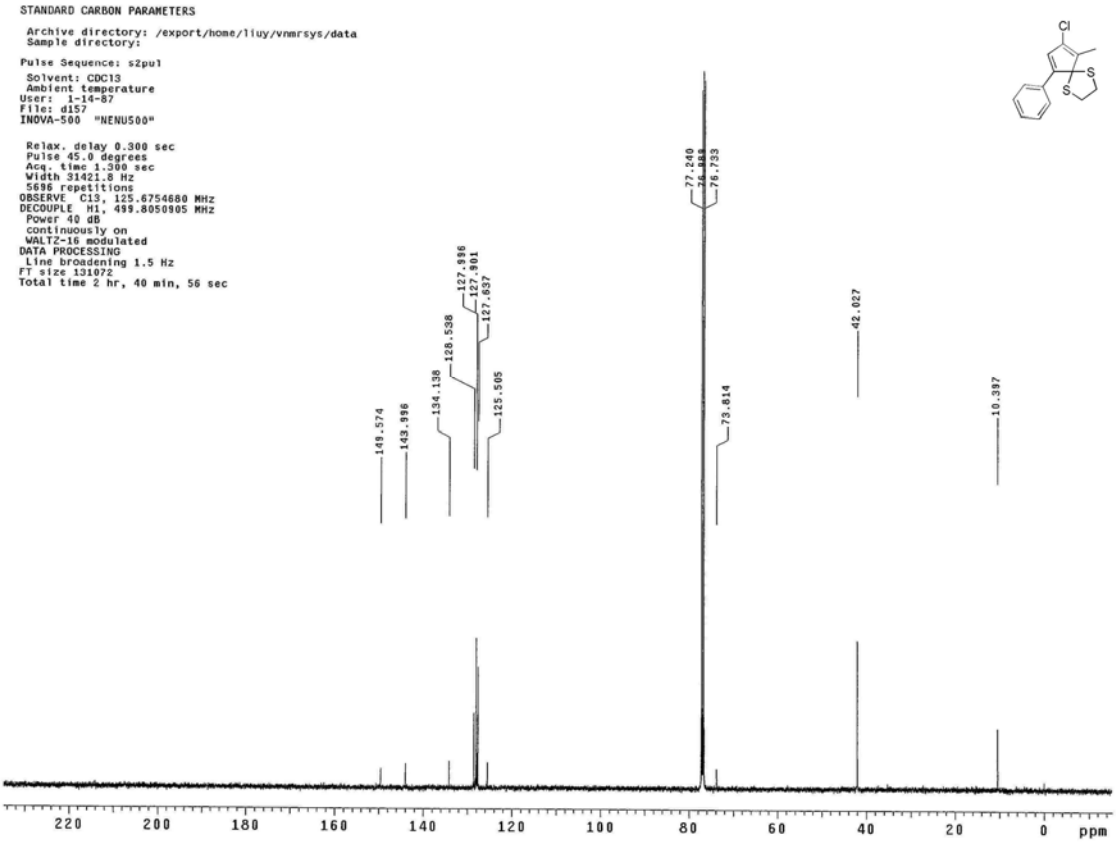
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1901
INOVA-500 "NENU500"

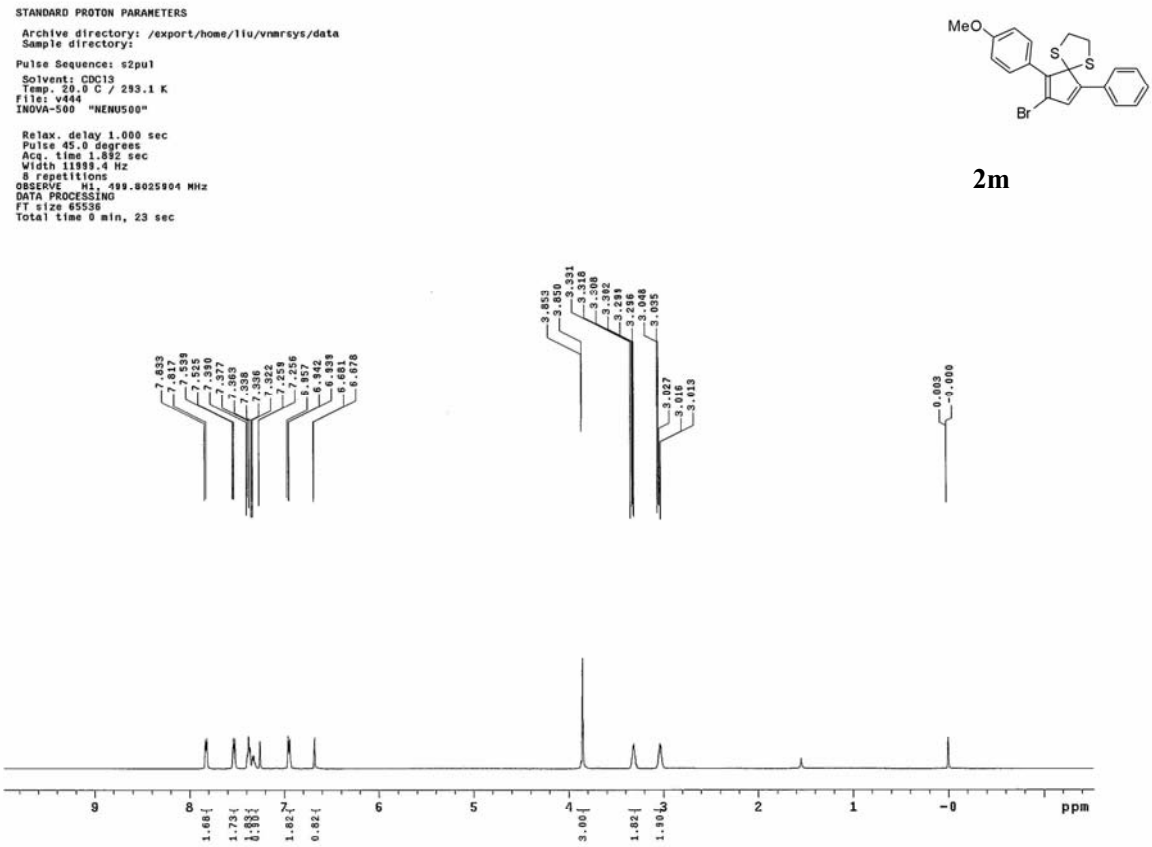
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
384 repetitions
OBSERVE C13, 125.6754642 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec

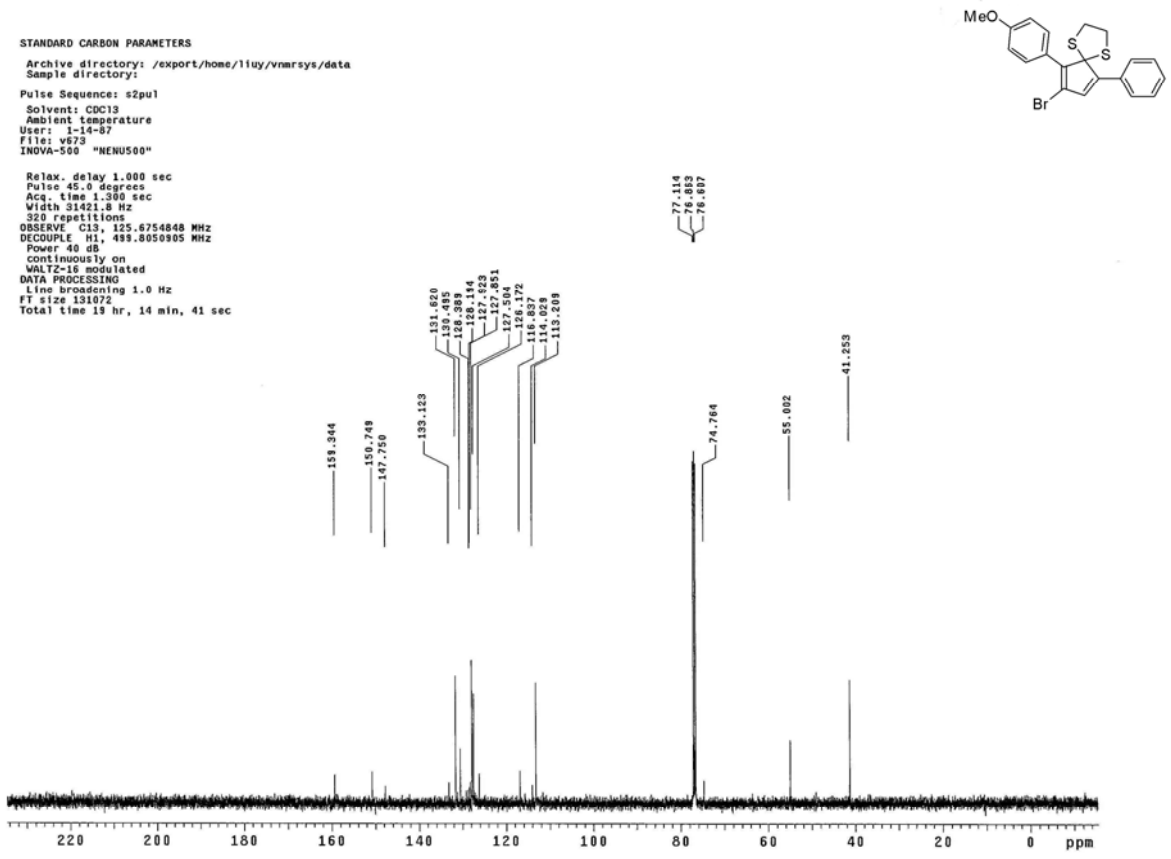


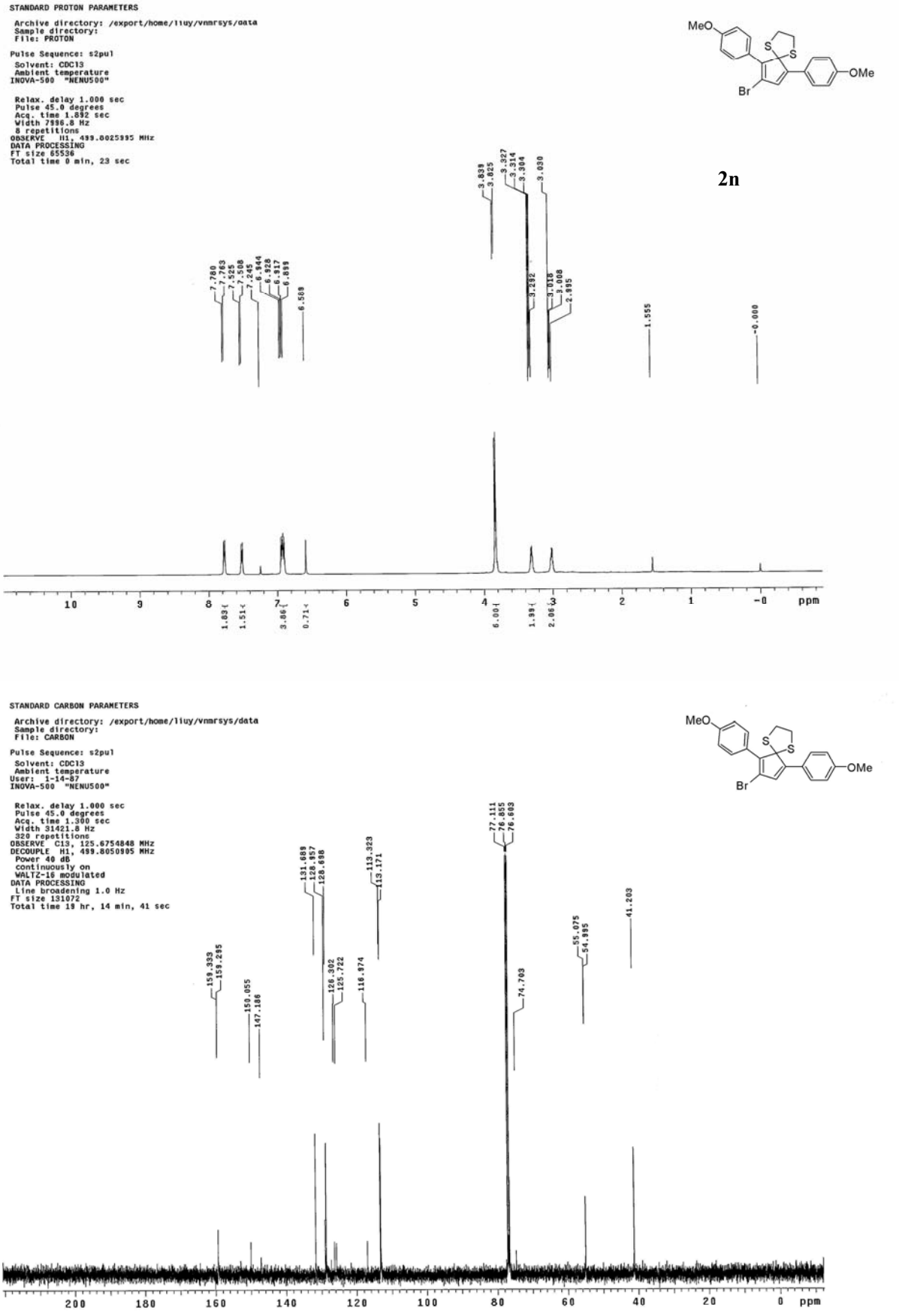


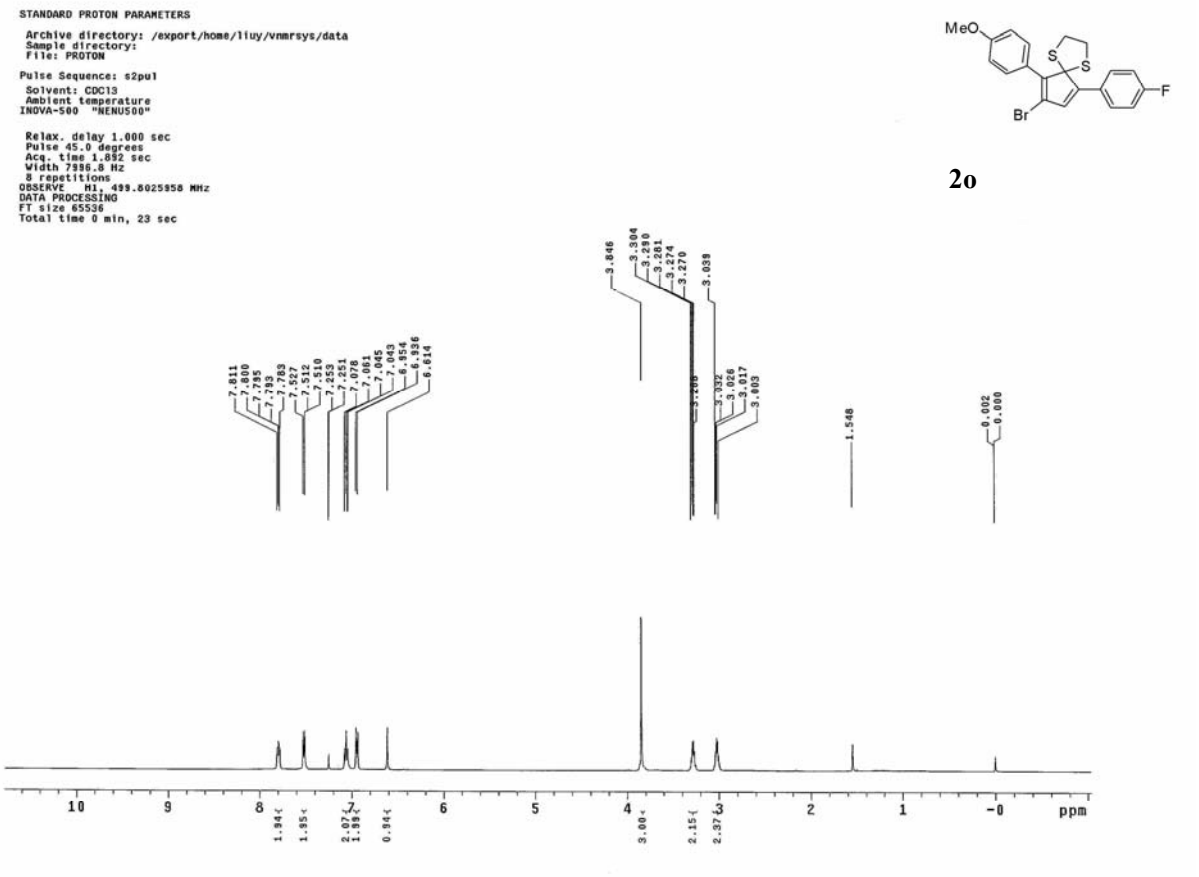

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d344
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8024.9 Hz
8 repetitions
OBSERVE H1, 499.8025934 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
S
S
2l
7.748
7.745
7.735
7.732
7.728
7.351
7.348
7.339
7.338
7.322
7.318
7.257
6.389
3.551
3.548
3.546
3.540
3.536
3.530
3.526
3.463
3.459
3.453
3.449
3.444
3.441
2.064
0.000
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.92
3.19
0.86
2.20
2.37
3.00

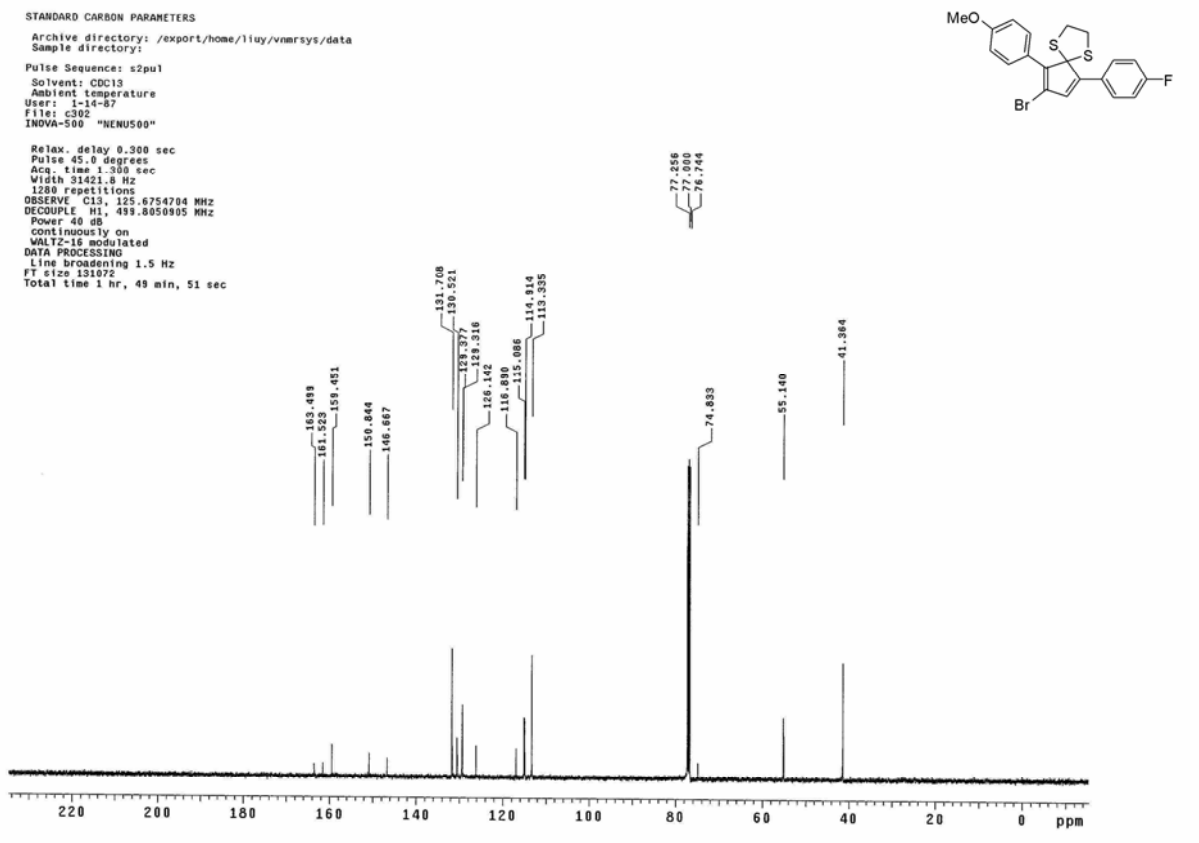

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: d157
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
5696 repetitions
OBSERVE C13, 125.6754680 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec
Cl
S
S
149.574
143.996
134.138
128.538
127.996
127.901
127.637
125.565
77.240
76.733
73.614
42.027
10.397
220
200
180
160
140
120
100
80
60
40
20
0
ppm

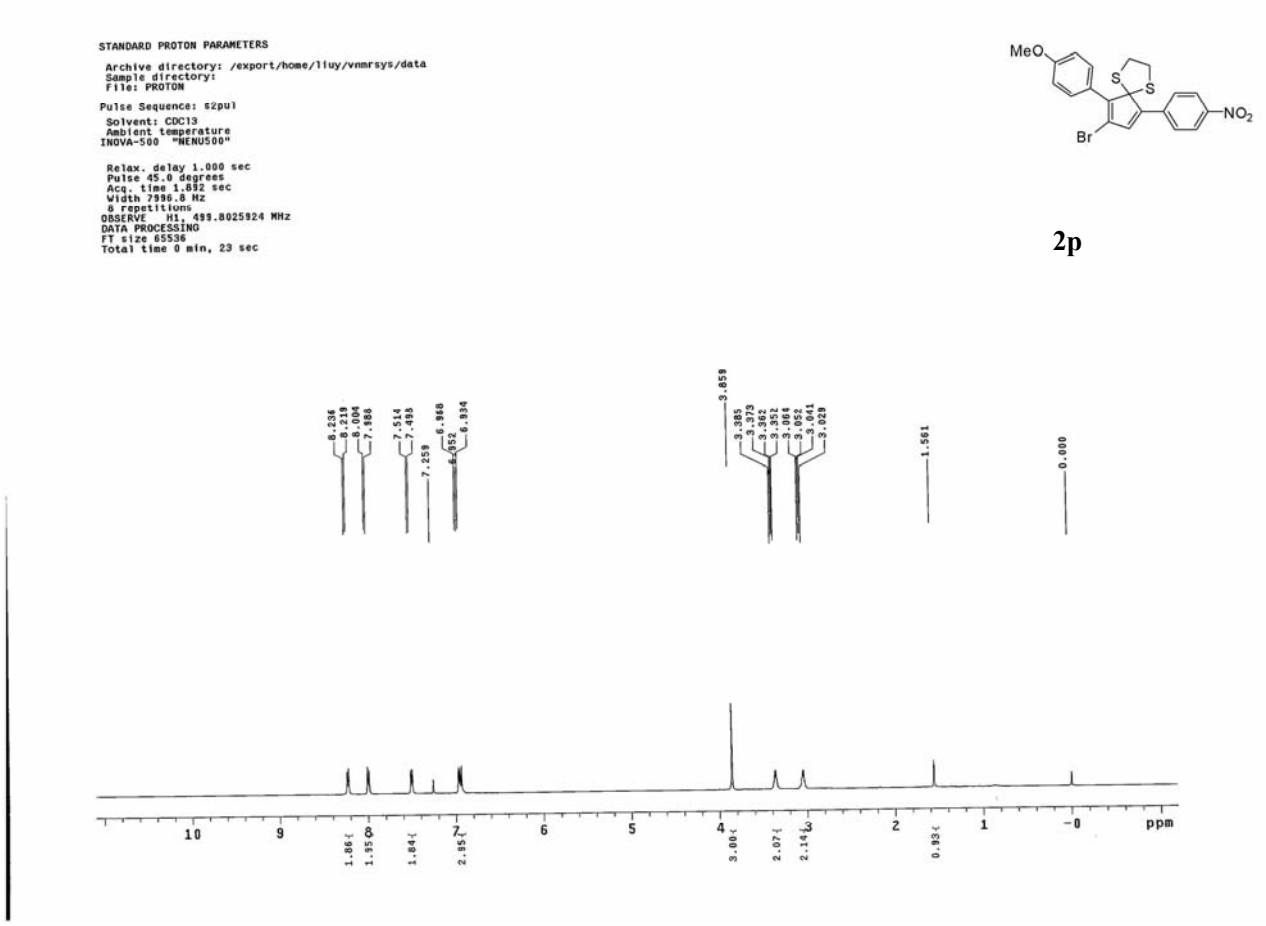

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liu/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
File: v444
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 11988.4 Hz
8 repetitions
OBSERVE H1, 499.8025904 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
S
S
Br
2m
7.833
7.817
7.539
7.525
7.390
7.377
7.363
7.336
7.322
7.259
7.256
6.937
6.923
6.438
6.681
6.678
3.853
3.851
3.331
3.316
3.308
3.302
3.291
3.286
3.048
3.035
3.027
3.016
3.013
0.003
-0.000
9
8
7
6
5
4
3
2
1
-0
ppm
1.68
1.73
1.83
1.90
1.82
0.82
3.00
1.82
1.90


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: v673
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, 125.6754848 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 19 hr, 14 min, 41 sec
MeO
S
S
Br
77.114
76.863
76.607
159.344
150.749
147.750
133.123
131.620
130.495
128.383
128.114
127.823
127.851
127.504
126.172
116.837
114.029
113.209
74.764
55.002
41.253
220
200
180
160
140
120
100
80
60
40
20
0
ppm

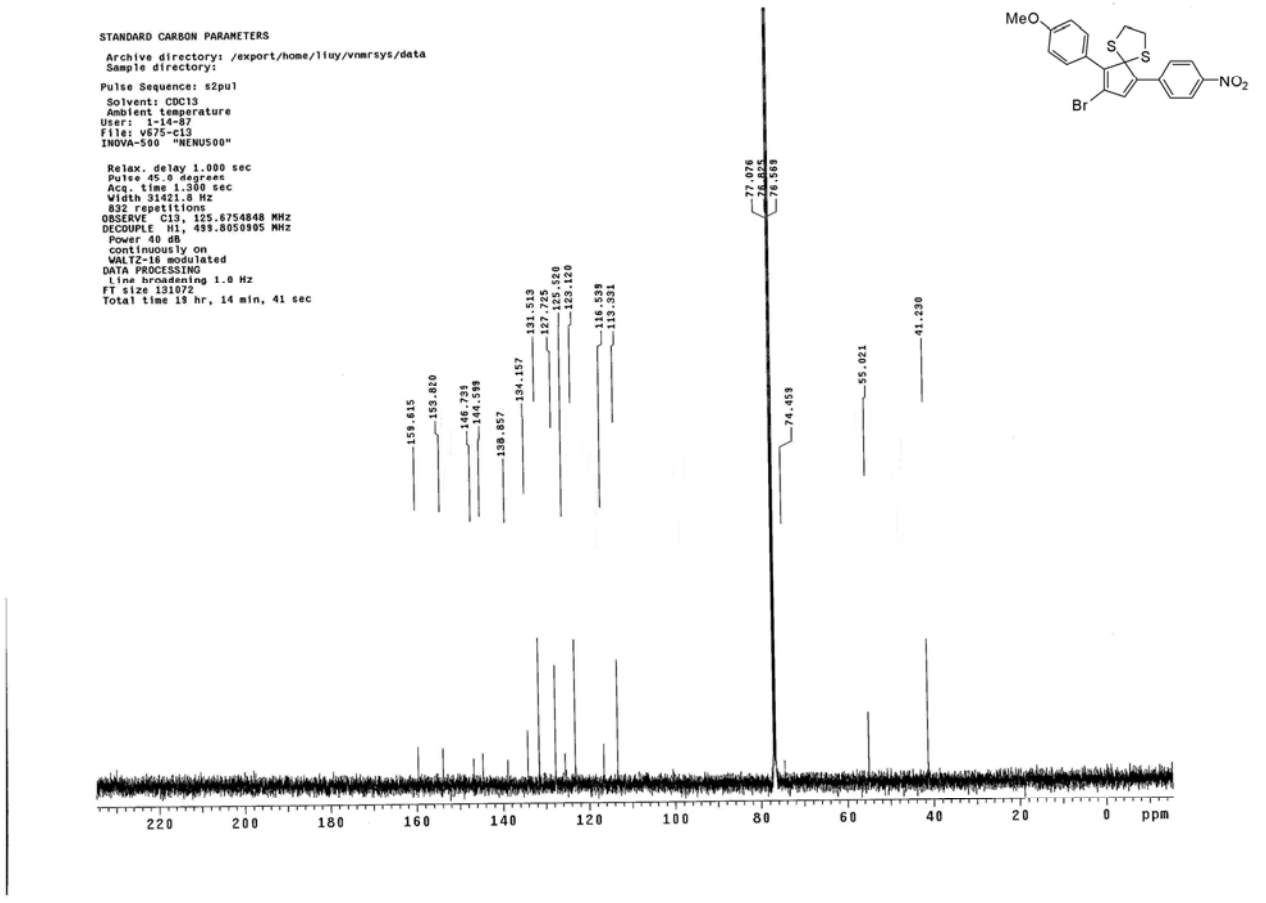

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025995 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
OMe
Br
2n
7.780
7.763
7.525
7.508
7.245
6.944
6.928
6.917
6.899
6.569
3.839
3.825
3.327
3.314
3.304
3.030
3.292
3.018
3.008
2.995
1.555
0.000
10
9
8
7
6
5
4
3
2
1
0
ppm
1.03
1.51
3.86
0.71
6.00
1.89
2.06
STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/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 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754848 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 19 hr, 14 min, 41 sec
MeO
OMe
Br
159.339
159.285
150.055
147.186
131.688
128.957
128.698
126.302
125.722
116.874
113.323
113.171
77.111
76.855
76.603
74.703
55.075
54.895
41.203
200
180
160
140
120
100
80
60
40
20
0
ppm

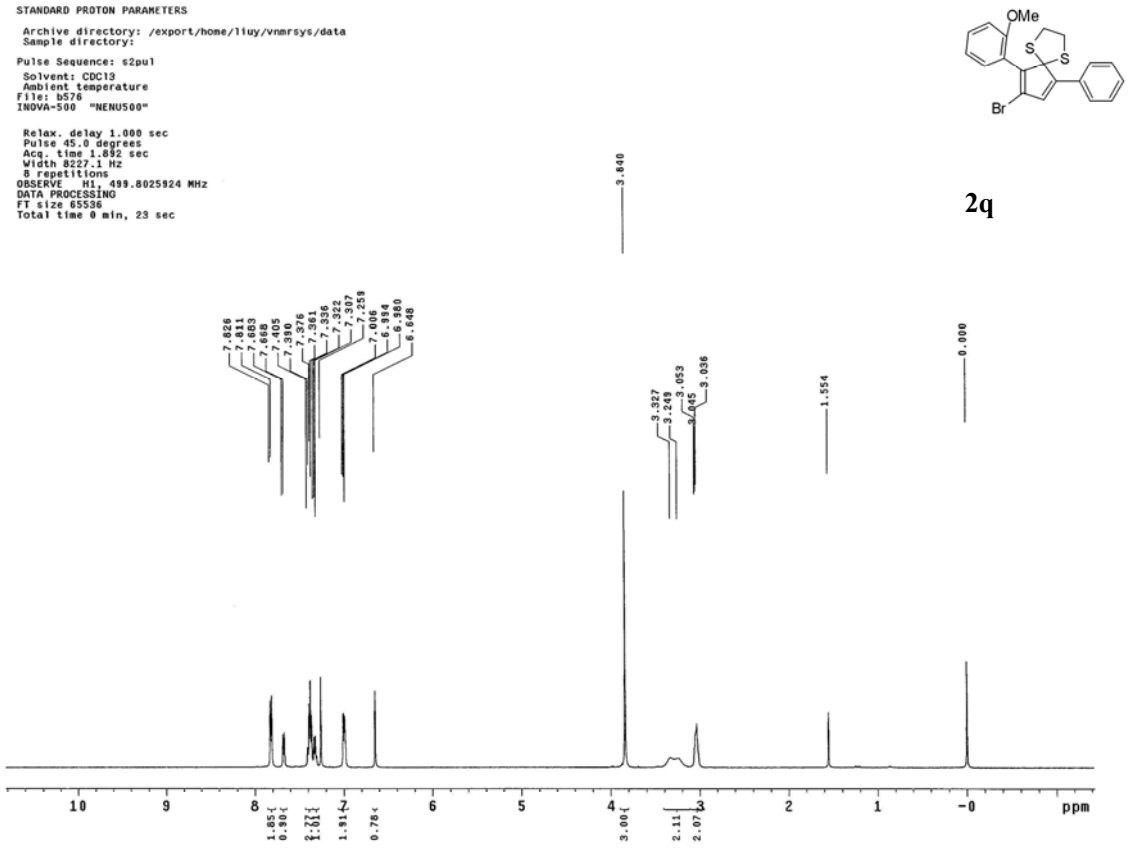

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025858 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
S
S
F
Br
2o
7.811
7.800
7.795
7.793
7.783
7.527
7.512
7.510
7.255
7.078
7.061
7.045
7.043
6.936
6.914
3.846
3.304
3.290
3.281
3.274
3.270
3.039
3.032
3.026
3.017
3.003
1.548
0.002
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.94
1.95
2.07
1.98
0.84
3.00
2.15
2.37

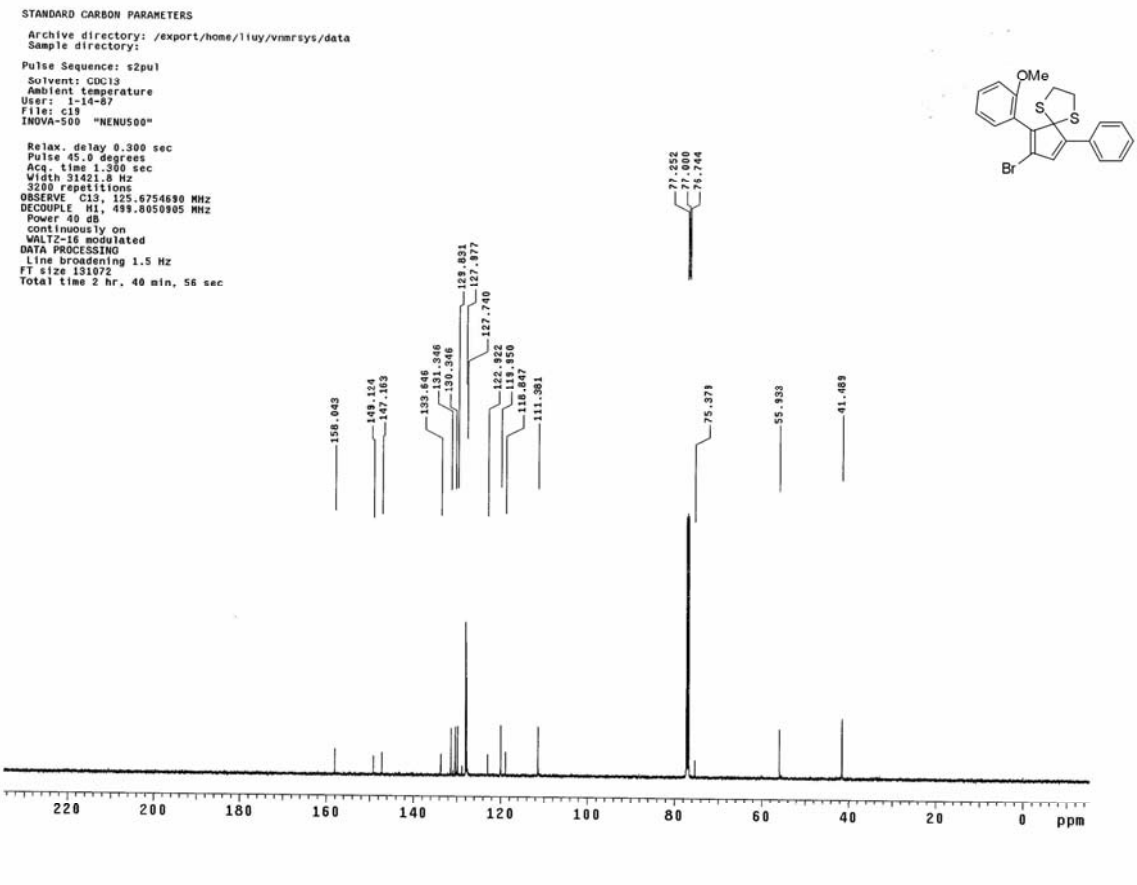

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: c302
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
1280 repetitions
OBSERVE C13, 125.6754704 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
MeO
S
S
F
Br
77.256
77.000
76.744
131.708
130.521
129.377
128.316
126.142
116.890
115.086
114.914
113.335
163.499
161.523
159.451
150.844
146.667
74.893
55.140
41.364
220
200
180
160
140
120
100
80
60
40
20
0
ppm

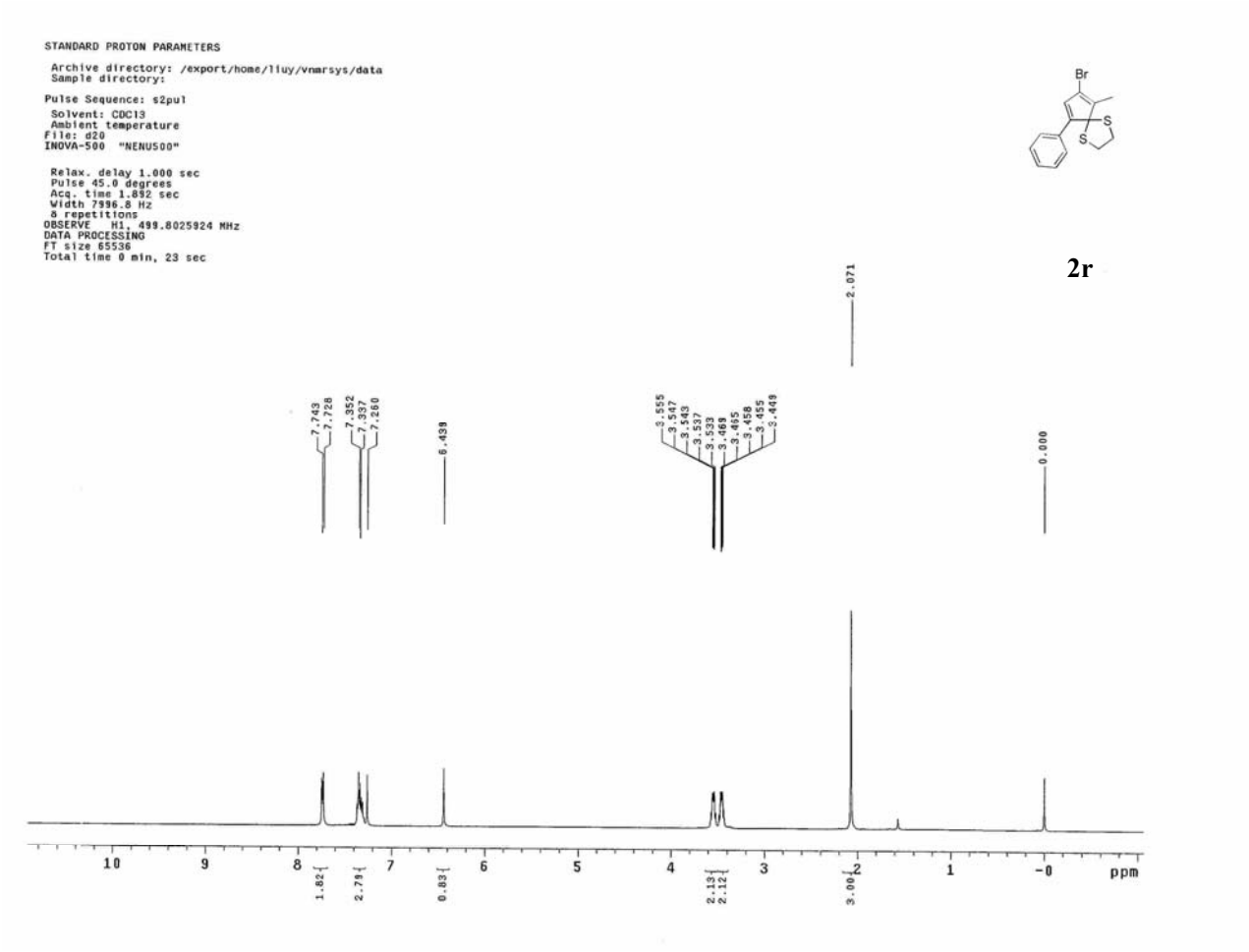

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025924 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
MeO
S
S
NO2
Br
8.236
8.219
8.004
7.988
7.514
7.495
7.259
6.968
6.834
3.859
3.385
3.373
3.362
3.351
3.341
3.052
3.041
3.029
1.561
0.000
10
9
7
6
5
4
3
2
1
0
ppm

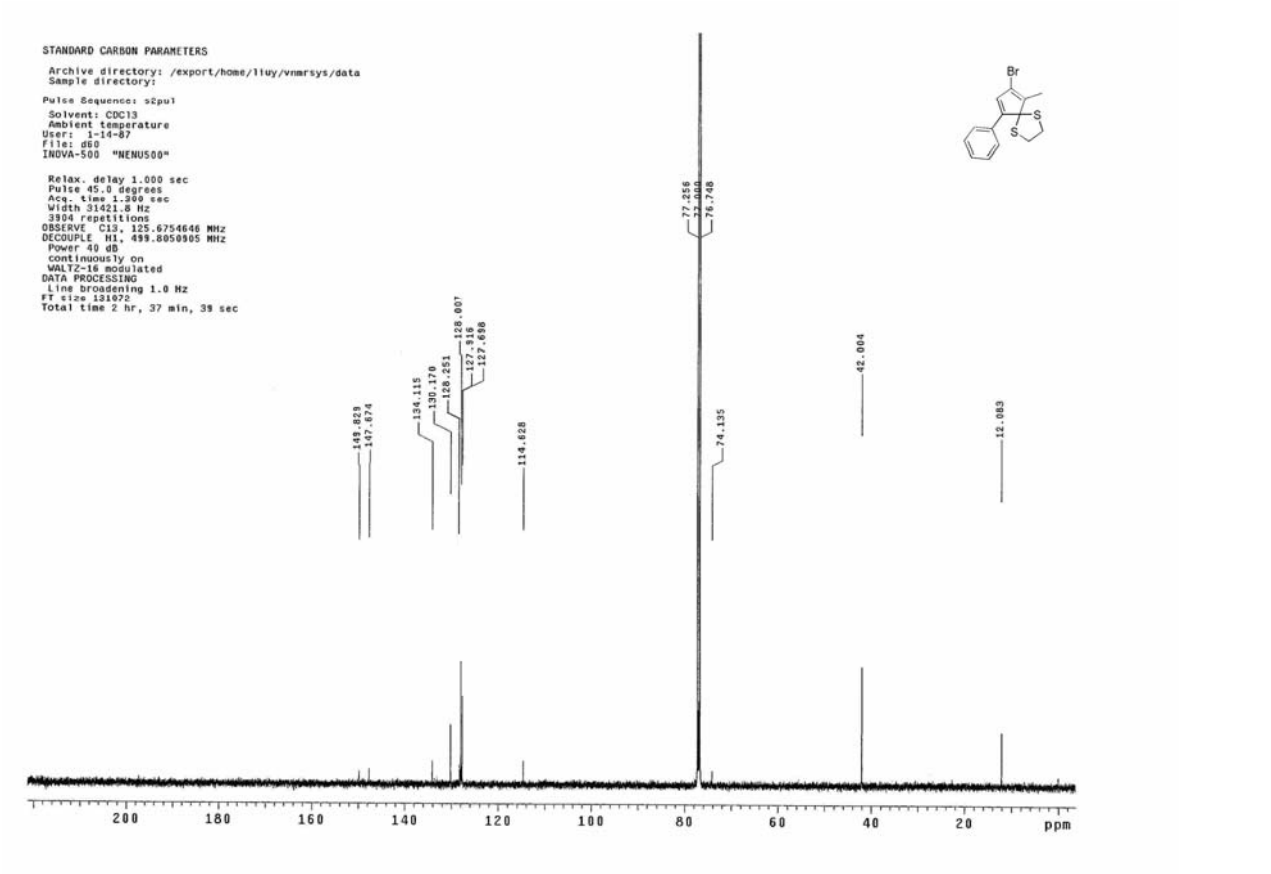
2p

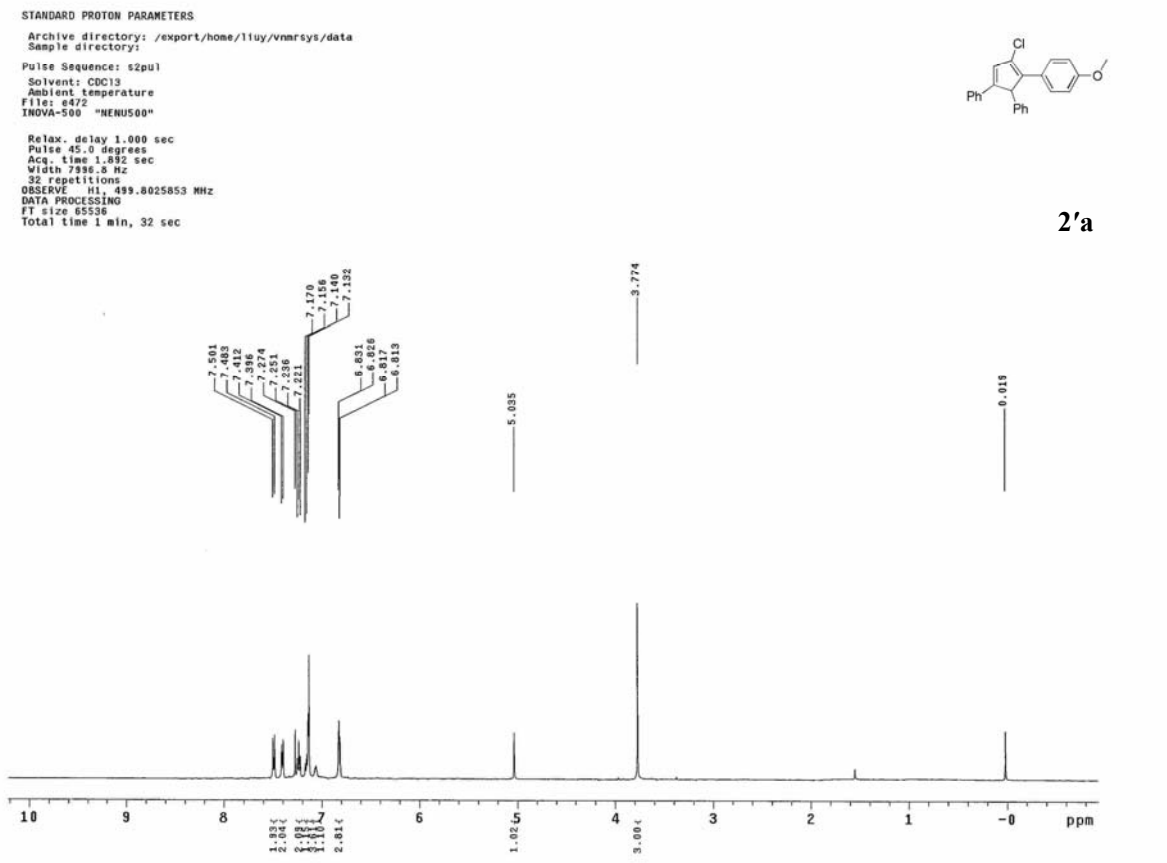

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: v675-c13
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
832 repetitions
OBSERVE C13, 125.6754848 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 19 hr, 14 min, 41 sec
MeO
S
S
NO2
Br
159.615
153.810
146.735
144.599
138.857
134.157
131.513
127.725
125.520
123.120
116.539
113.331
77.076
76.569
74.458
55.021
41.230
220
200
180
160
140
120
100
80
60
40
20
0
ppm

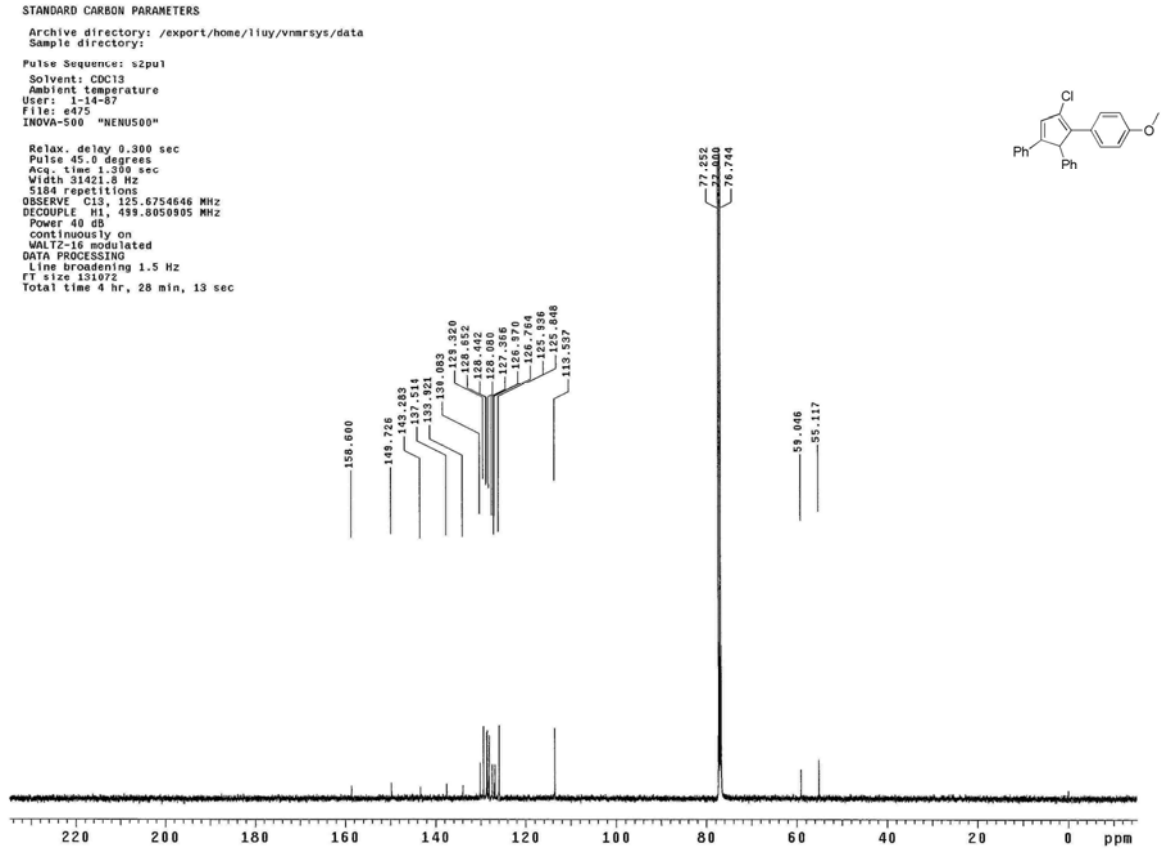

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b576
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8227.1 Hz
8 repetitions
OBSERVE H1, 499.8025924 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
OMe
S
S
Br
2q
3.640
7.826
7.811
7.683
7.660
7.405
7.390
7.376
7.361
7.336
7.322
7.307
7.259
7.006
6.984
6.380
6.648
3.327
3.249
3.053
3.045
3.036
1.554
0.000
10
9
8
6
5
4
3
2
1
-0
ppm

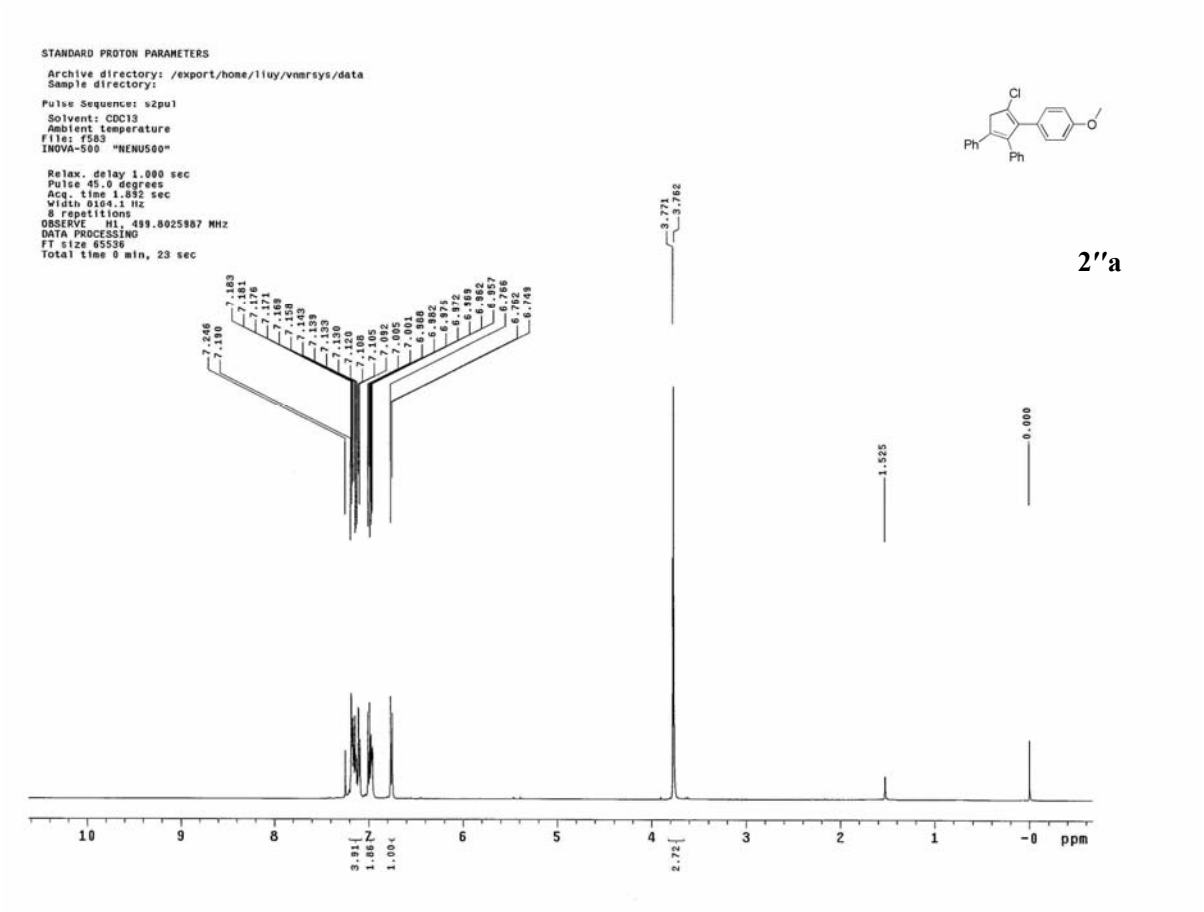

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: c19
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.360 sec
Width 31421.8 Hz
3200 repetitions
OBSERVE C13, 125.6754690 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec
OMe
S
S
Br
77.252
77.000
76.744
129.831
127.977
127.740
133.646
131.946
130.346
122.922
119.950
118.647
111.981
150.043
149.124
147.163
75.373
55.933
41.489
220
200
180
160
140
120
100
80
60
40
20
0
ppm

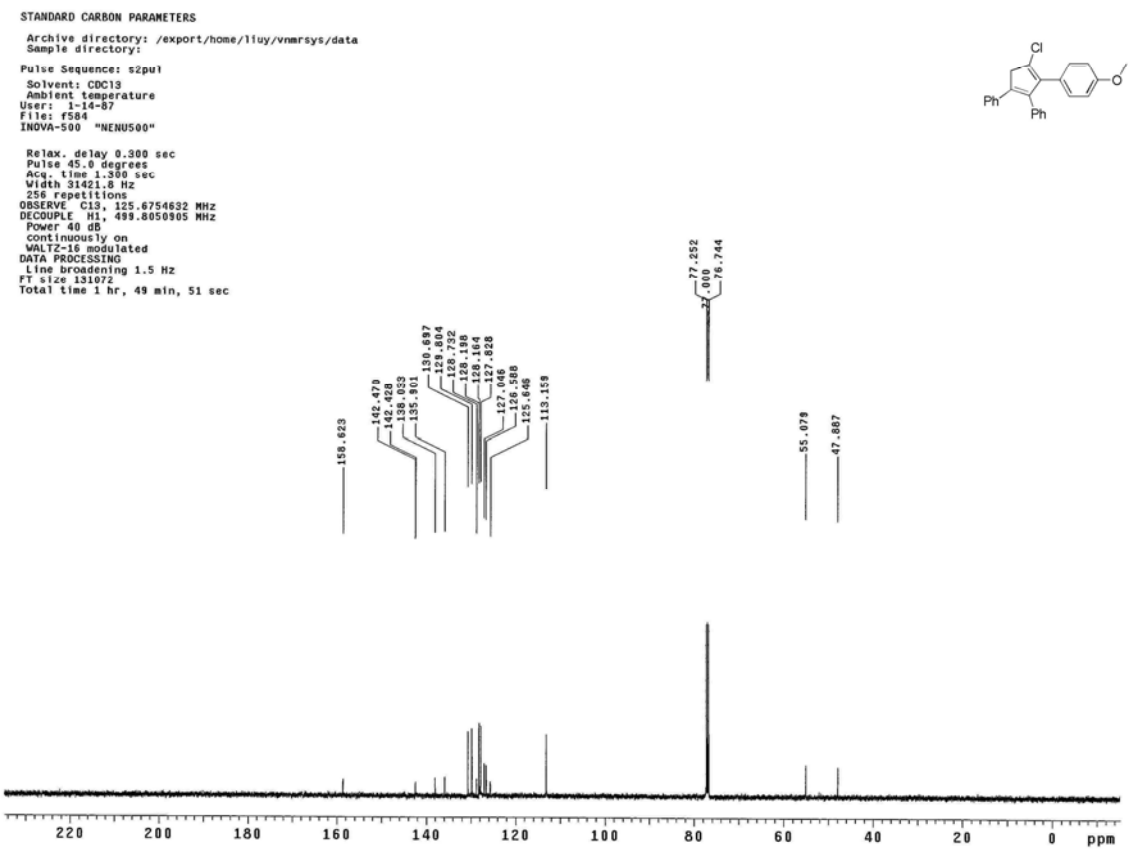

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d20
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025924 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Br
S
S
2r
2.071
7.743
7.728
7.352
7.337
7.260
6.439
3.555
3.547
3.543
3.537
3.533
3.469
3.465
3.458
3.455
3.449
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.82
2.79
0.83
2.13
2.12
3.00

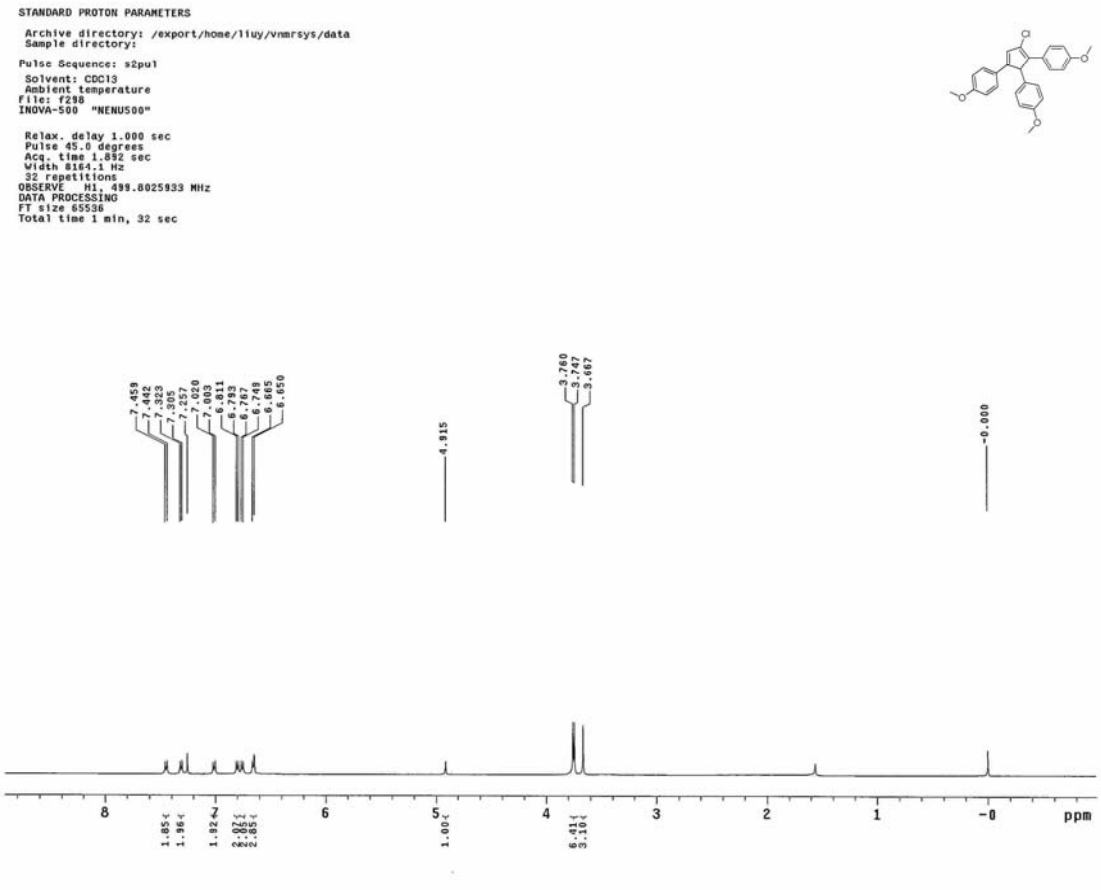

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: d60
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
3904 repetitions
OBSERVE C13, 125.6754646 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 37 min, 39 sec
Br
S
S
77.256
77.060
76.748
148.829
147.074
134.115
130.170
128.251
128.007
127.916
127.696
114.628
74.135
42.004
12.083
200
180
160
140
120
100
80
60
40
20
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: e472
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
32 repetitions
OBSERVE H1, 499.8025853 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 32 sec
Cl
Ph
Ph
O
2′a
7.501
7.403
7.412
7.396
7.274
7.251
7.236
7.221
7.179
7.158
7.140
7.132
6.831
6.826
6.817
6.813
5.035
3.774
0.018
1.93
2.04
2.08
3.01
1.10
2.01
1.02
3.00
10
9
8
7
6
5
4
3
2
1
-0
ppm

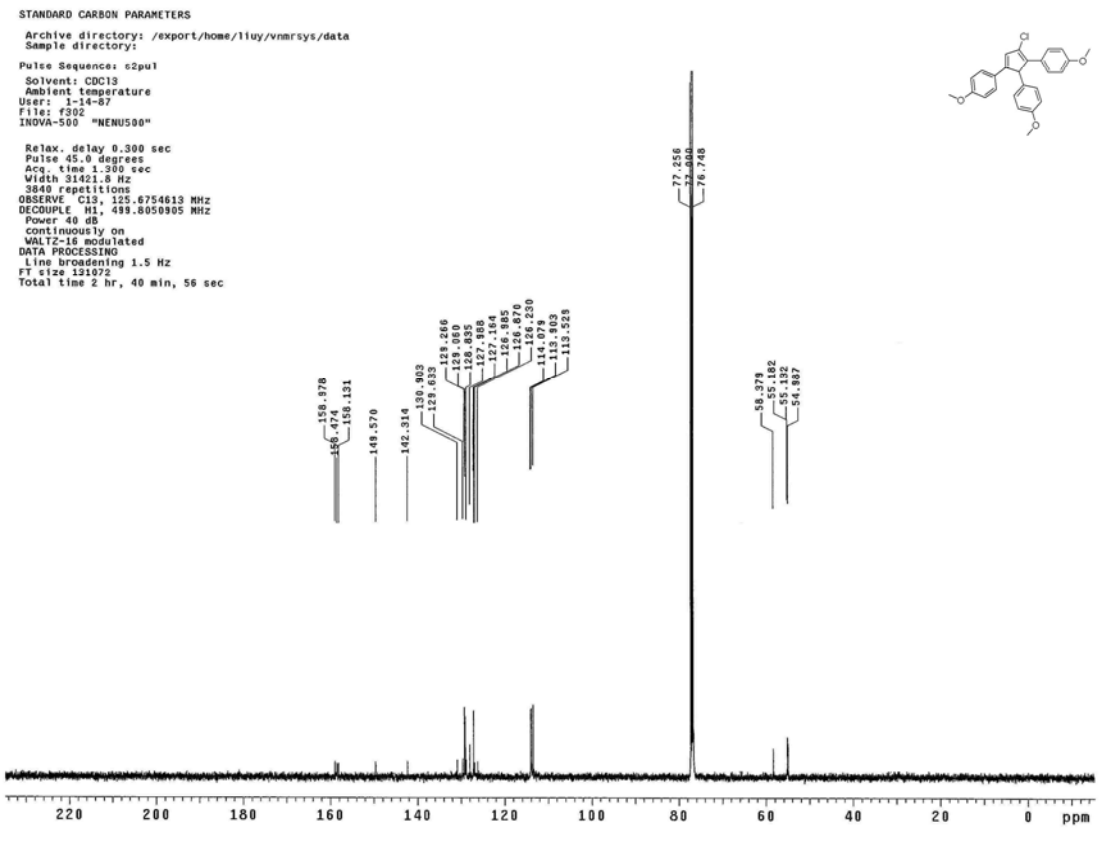

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: e475
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
5184 repetitions
OBSERVE C13, 125.6754646 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 4 hr, 28 min, 13 sec
Cl
Ph
Ph
O
158.600
149.726
143.283
137.514
133.921
130.083
129.320
128.652
128.442
128.080
127.855
126.970
126.764
125.936
125.848
113.537
77.252
77.000
76.744
59.046
55.117
220
200
180
160
140
120
100
80
60
40
20
0
ppm

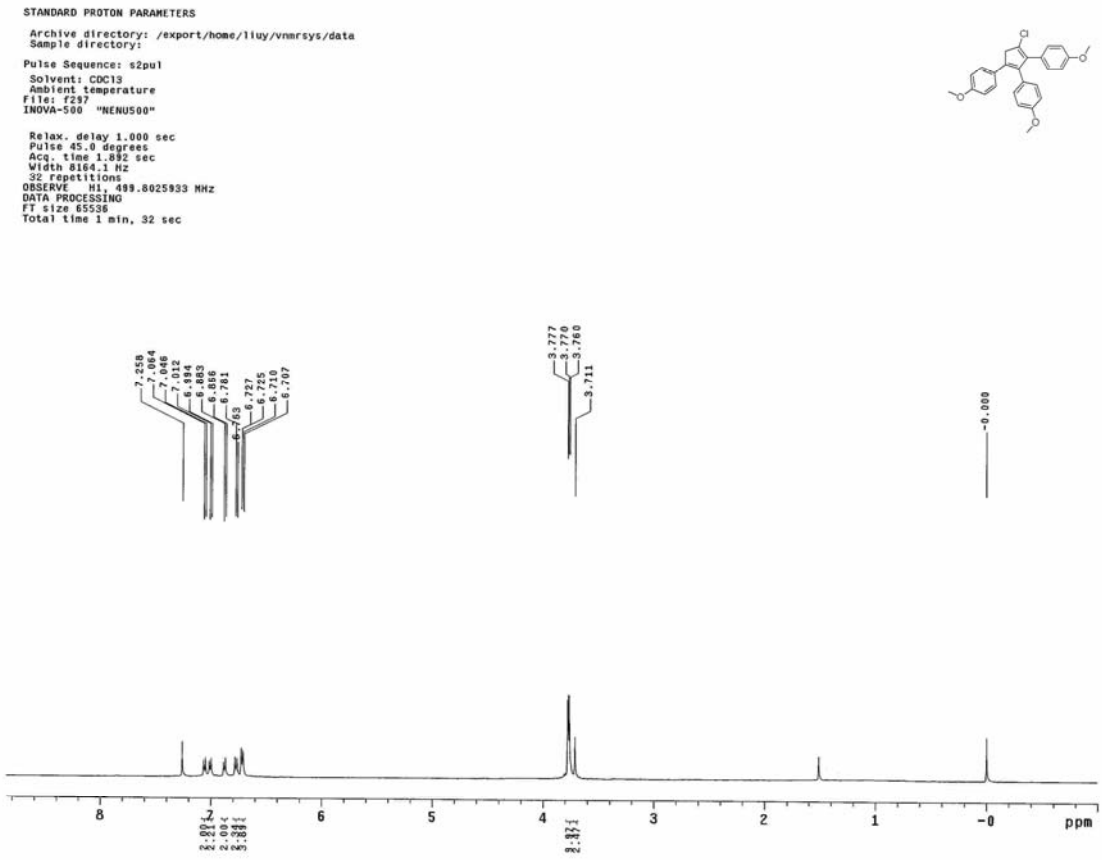

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f583
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 6164.1 Hz
8 repetitions
OBSERVE H1, 499.8025987 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
Ph
Ph
O
2''a
10
9
8
6
5
4
3
2
1
-0
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: f584
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754632 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
Cl
Ph
Ph
O
220
200
180
160
140
120
100
80
60
40
20
0
ppm

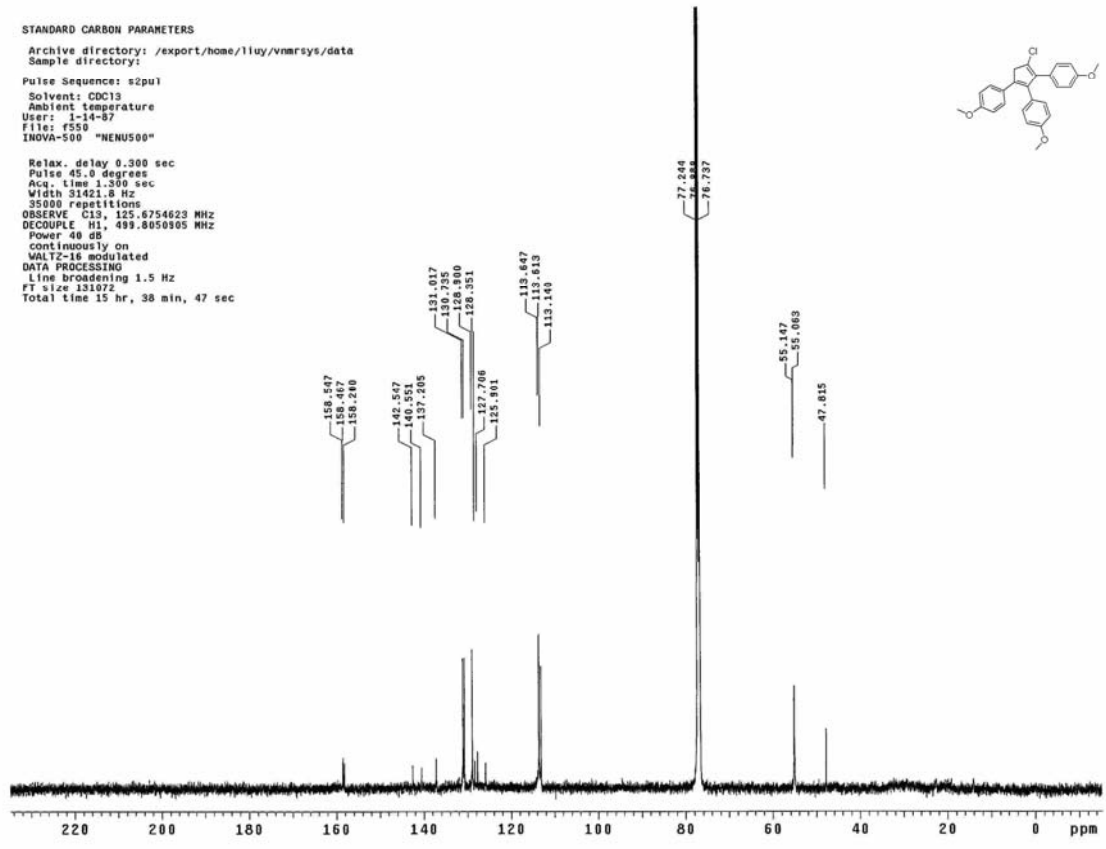

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f238
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8164.1 Hz
32 repetitions
OBSERVE H1, 499.8025933 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 32 sec
7.459
7.442
7.323
7.305
7.257
7.020
7.003
6.811
6.793
6.767
6.749
6.665
6.650
4.915
3.760
3.747
3.667
-0.000
8
7
6
5
4
3
2
1
-0
ppm

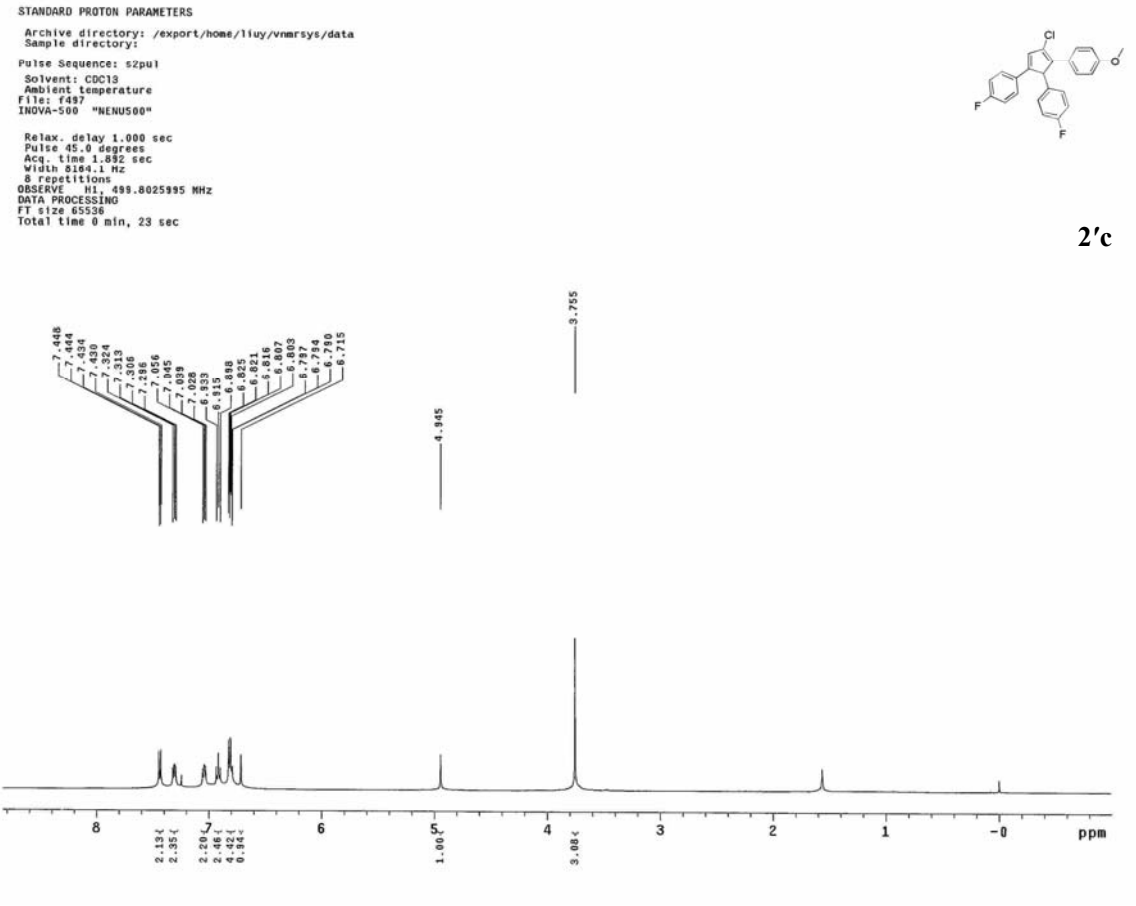
2′b

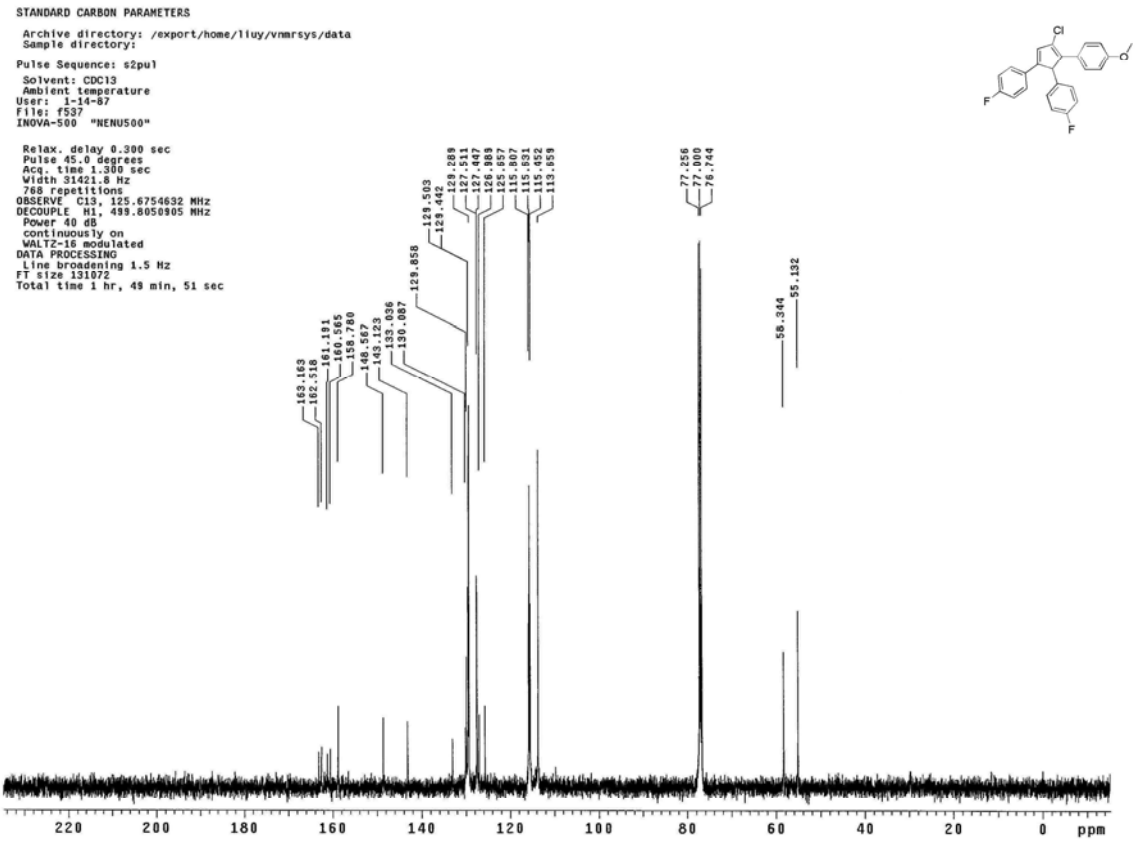

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: c2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: f302
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
3840 repetitions
OBSERVE C13, 125.6754613 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec
158.378
156.474
150.131
149.570
142.314
130.903
129.633
129.266
128.865
128.835
127.988
127.164
126.885
126.870
126.230
114.078
113.803
113.528
77.256
77.000
76.748
58.379
55.182
55.132
54.887
220
200
180
160
140
120
100
80
60
40
20
0
ppm

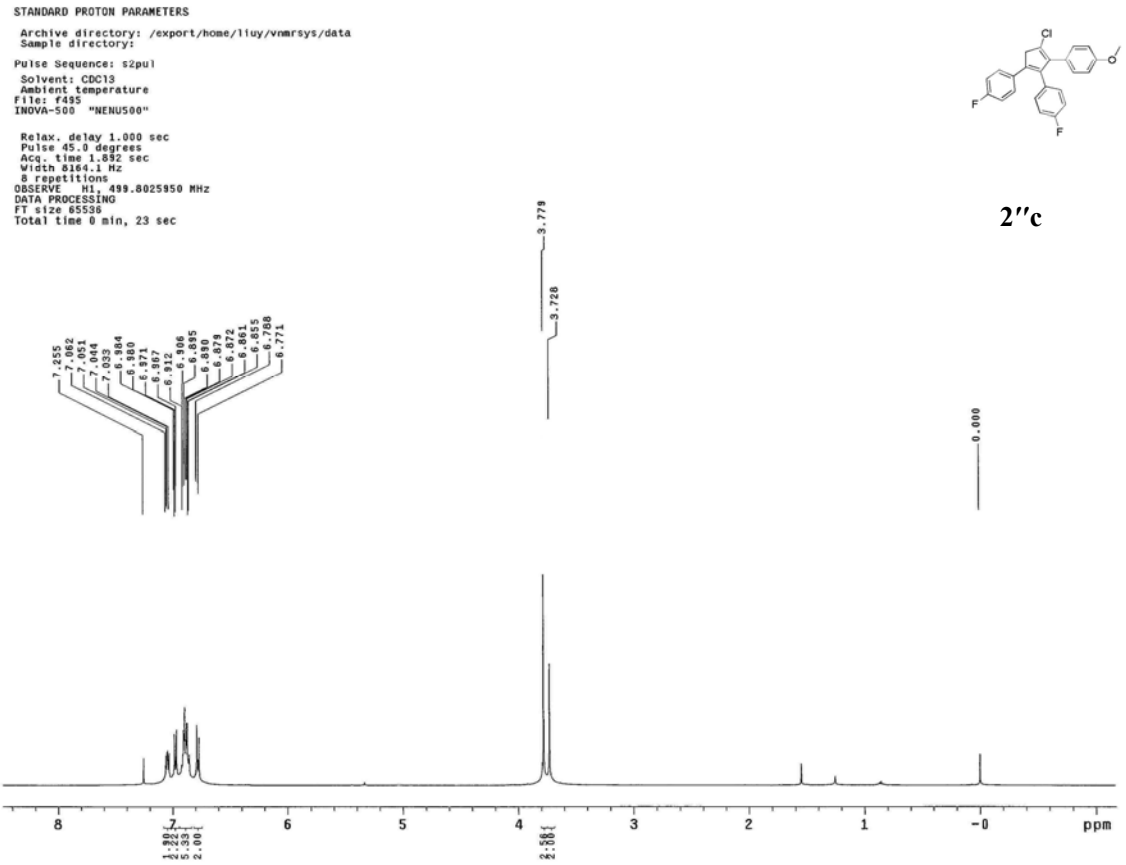

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f297
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8164.1 Hz
32 repetitions
OBSERVE H1, 499.8025933 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 32 sec
7.258
7.064
7.046
7.012
6.894
6.883
6.866
6.761
6.727
6.725
6.710
6.707
3.777
3.770
3.760
3.711
-0.000
ppm

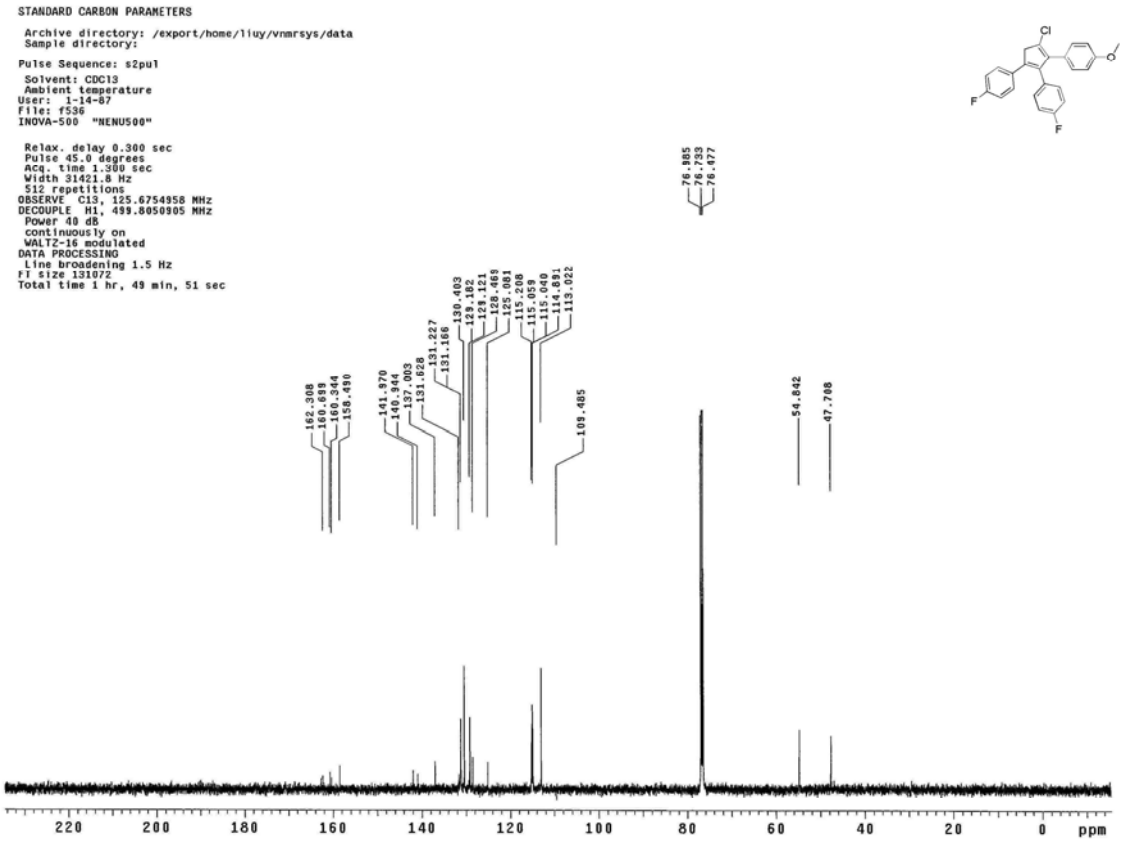
2″b

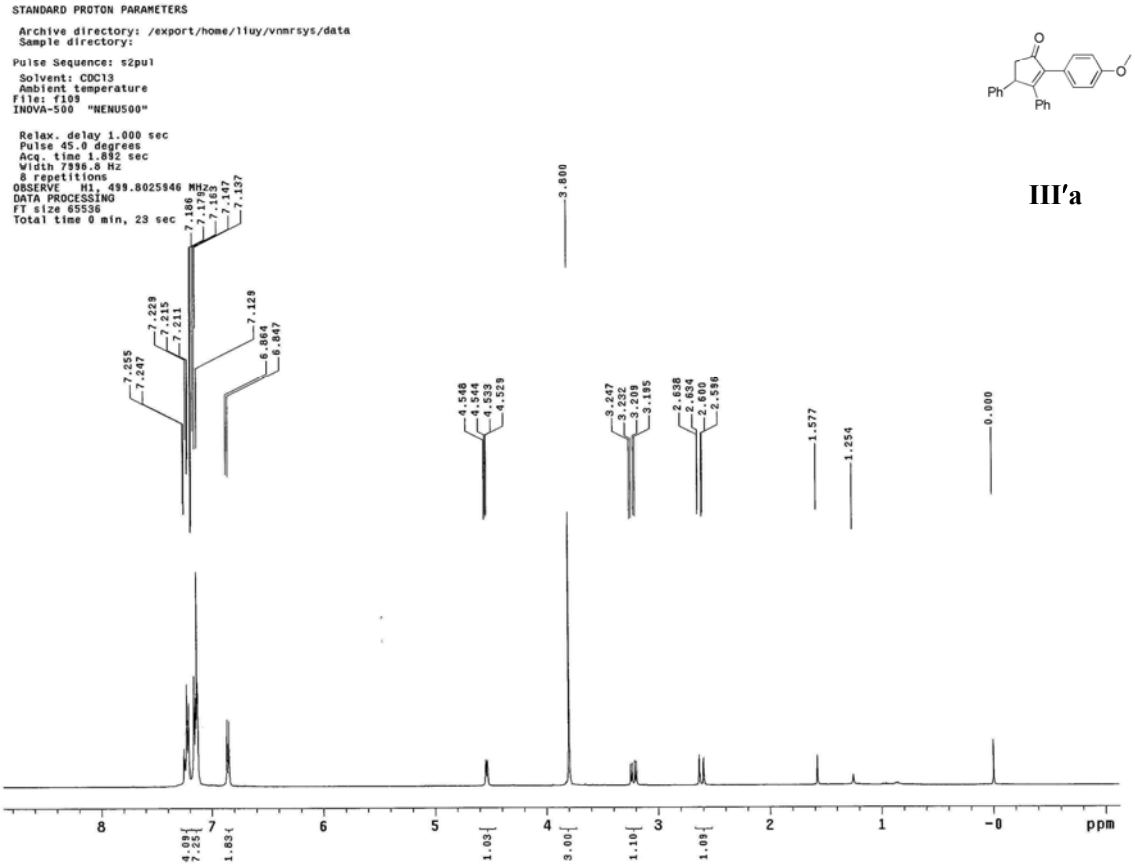

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: f550
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
35000 repetitions
OBSERVE C13, 125.6754623 MHz
DECOUPLE H1, 499.8050805 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 15 hr, 38 min, 47 sec
158.547
158.467
158.260
142.547
140.551
137.205
131.017
130.735
128.800
128.351
127.706
125.801
113.647
113.613
113.140
77.244
76.737
55.147
55.063
47.815
ppm

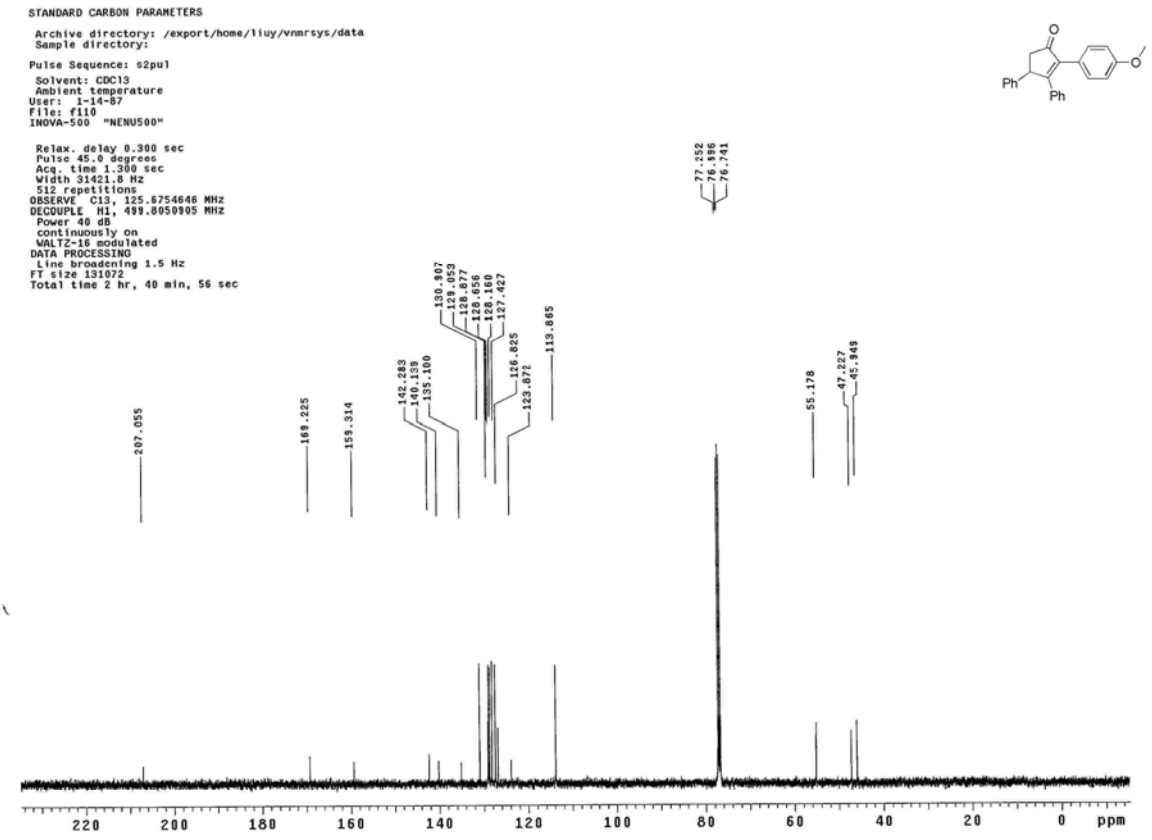

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f497
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8164.1 Hz
8 repetitions
OBSERVE H1, 499.8025995 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
O
F
F
2′c
7.448
7.444
7.434
7.430
7.324
7.313
7.306
7.286
7.056
7.045
7.039
7.028
6.833
6.815
6.808
6.825
6.821
6.816
6.807
6.803
6.797
6.794
6.780
6.715
4.945
3.755
8
7
6
5
4
3
2
1
-0
ppm
2.13
2.35
2.20
2.46
4.42
0.94
1.00
3.08

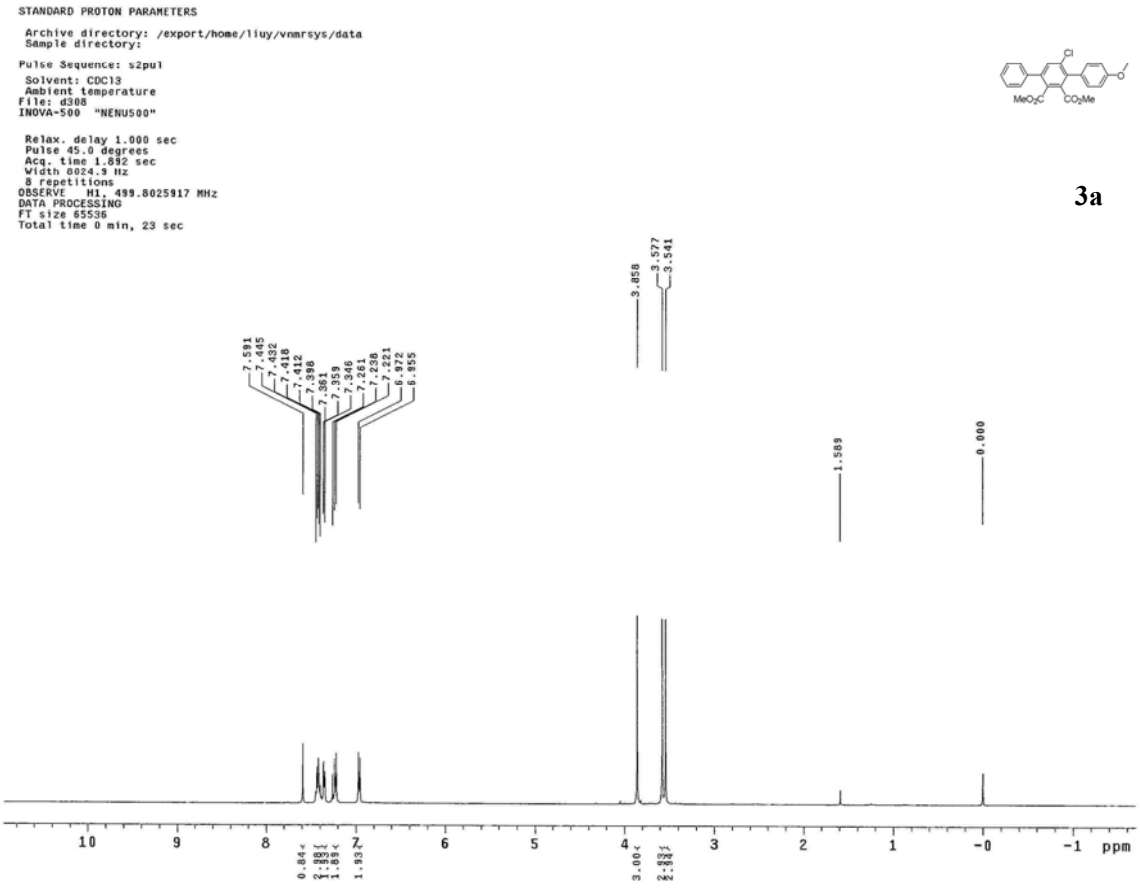

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: f537
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
768 repetitions
OBSERVE C13, 125.6754632 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
Cl
O
F
F
163.163
162.518
161.191
160.565
158.780
148.567
143.123
133.036
130.087
129.858
128.503
128.442
128.289
127.511
127.447
126.988
125.657
115.807
115.621
115.452
113.859
77.256
77.000
76.744
58.344
55.132
220
200
180
160
140
120
100
80
60
40
20
0
ppm

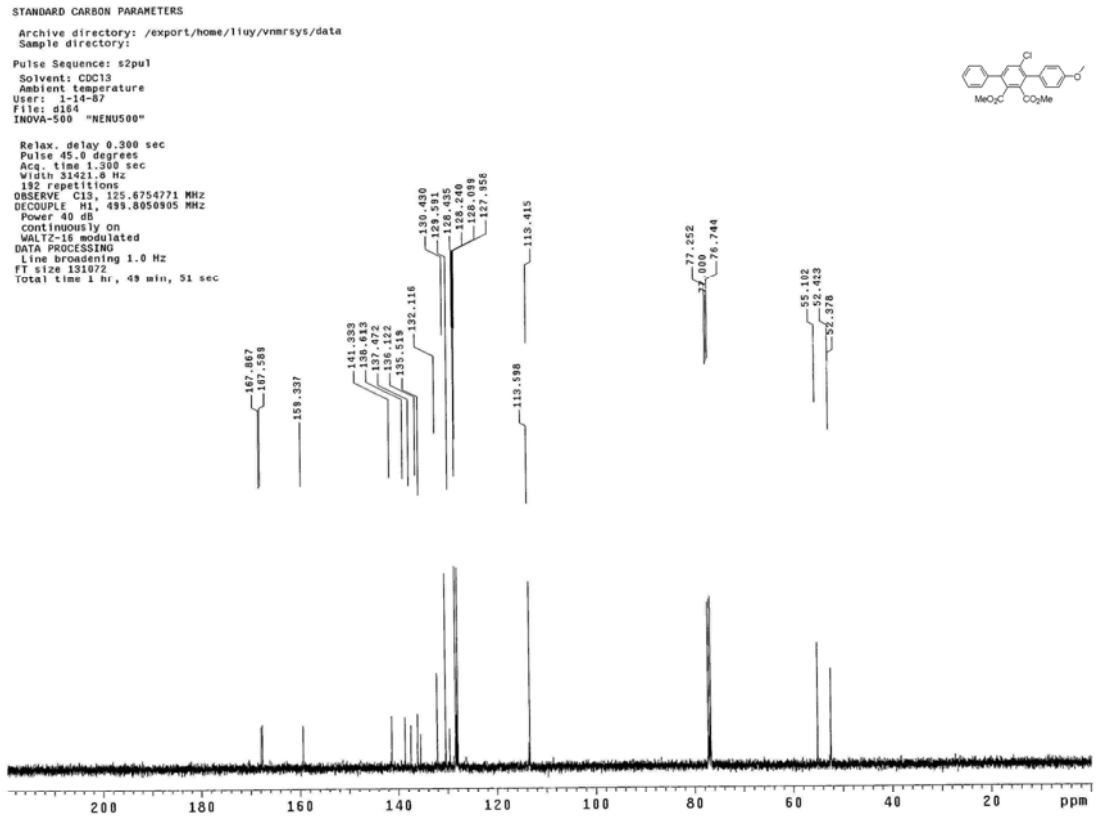

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f495
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8164.1 Hz
8 repetitions
OBSERVE H1, 499.8025950 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
2″c
7.255
7.062
7.051
7.044
7.033
6.984
6.980
6.971
6.957
6.912
6.906
6.895
6.890
6.879
6.872
6.861
6.855
6.788
6.771
3.779
3.728
0.000
8
7
6
5
4
3
2
1
-0
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: f536
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
512 repetitions
OBSERVE C13, 125.6754958 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
76.885
76.733
76.477
162.308
160.698
160.344
158.490
141.870
140.944
137.003
131.628
131.227
131.166
130.402
130.122
129.121
128.468
125.081
115.208
115.059
115.040
114.891
113.022
109.485
54.842
47.708
220
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: f109
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025946 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
III'a
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: f110
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
512 repetitions
OBSERVE C13, 125.6754646 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 40 min, 56 sec
ppm


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d308
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 6024.9 Hz
8 repetitions
OBSERVE H1, 499.8025917 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
3a
7.591
7.445
7.432
7.418
7.412
7.398
7.361
7.359
7.346
7.261
7.238
7.221
6.972
6.955
3.858
3.577
3.541
1.589
0.000
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: d164
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.0 Hz
192 repetitions
OBSERVE C13, 125.6754771 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
167.867
167.588
159.337
141.333
138.613
137.472
136.122
135.519
132.116
130.430
129.591
128.435
128.240
128.098
127.958
118.415
113.598
77.252
77.000
76.744
55.102
52.423
52.378
200
180
160
140
120
100
80
60
40
20
ppm

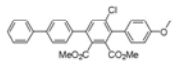
```
STANDARD PROTON PARAMETERS

 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:

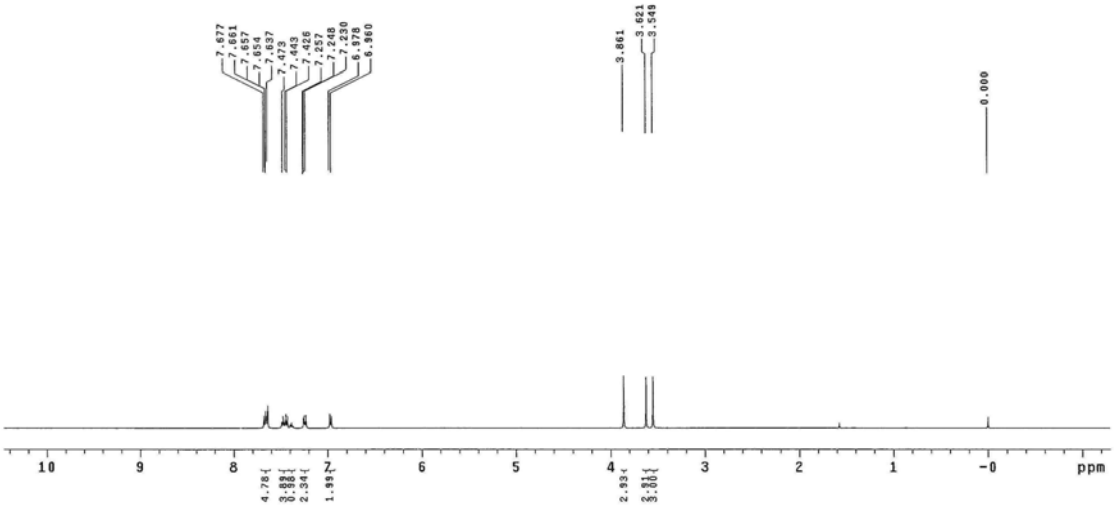
Pulse Sequence: s2pul

 Solvent: CDCl3
 Ambient temperature
File: e123
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, 499.8025938 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
```



3b



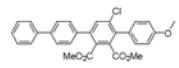

```
STANDARD CARBON PARAMETERS

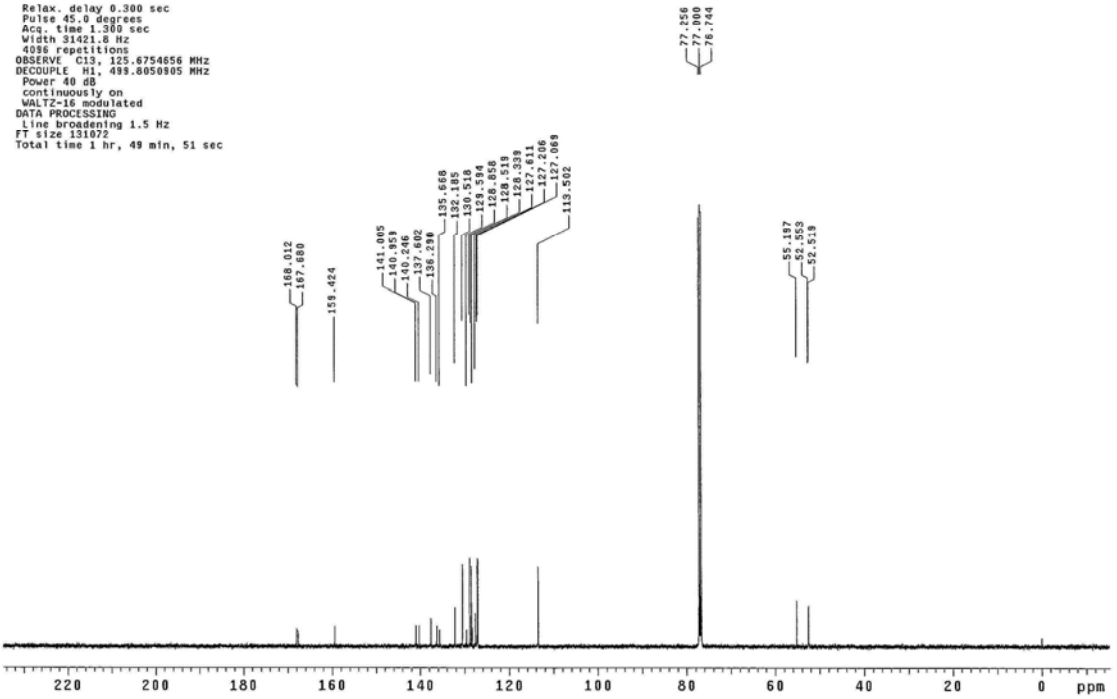
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:

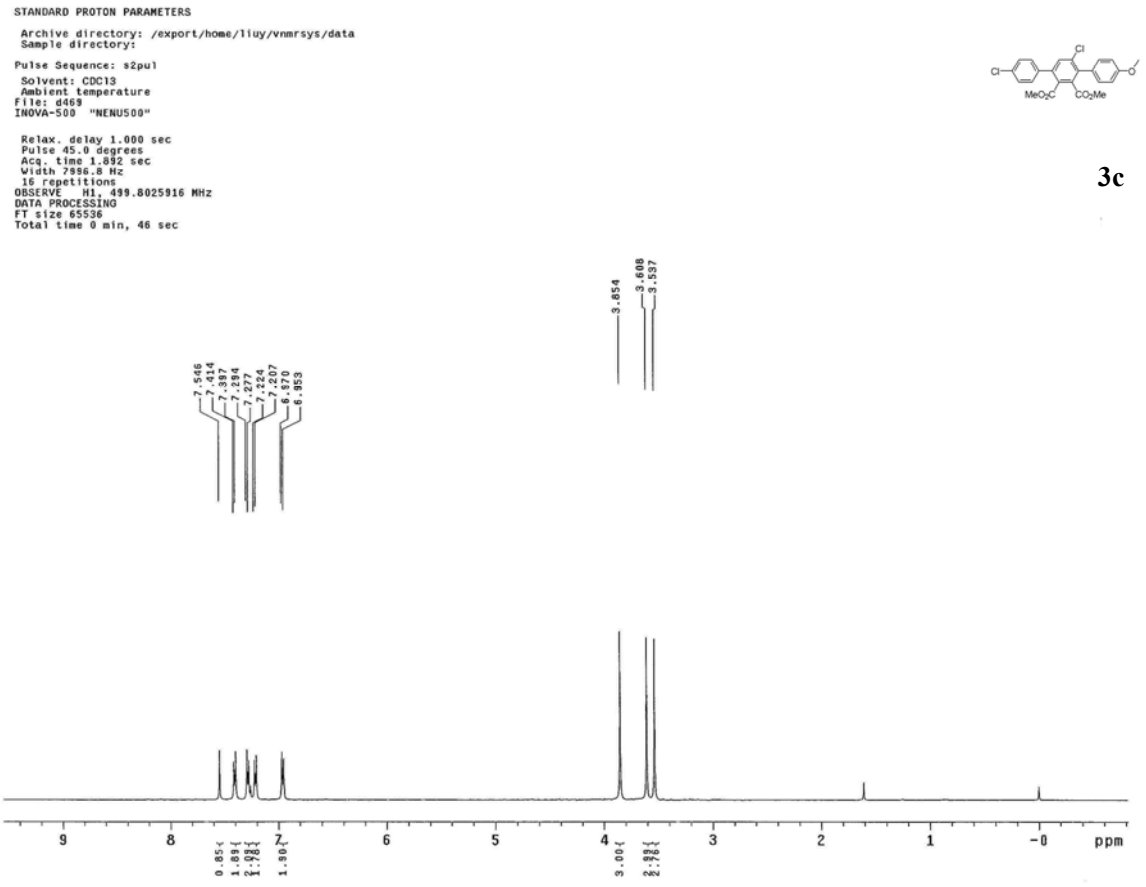
Pulse Sequence: s2pul

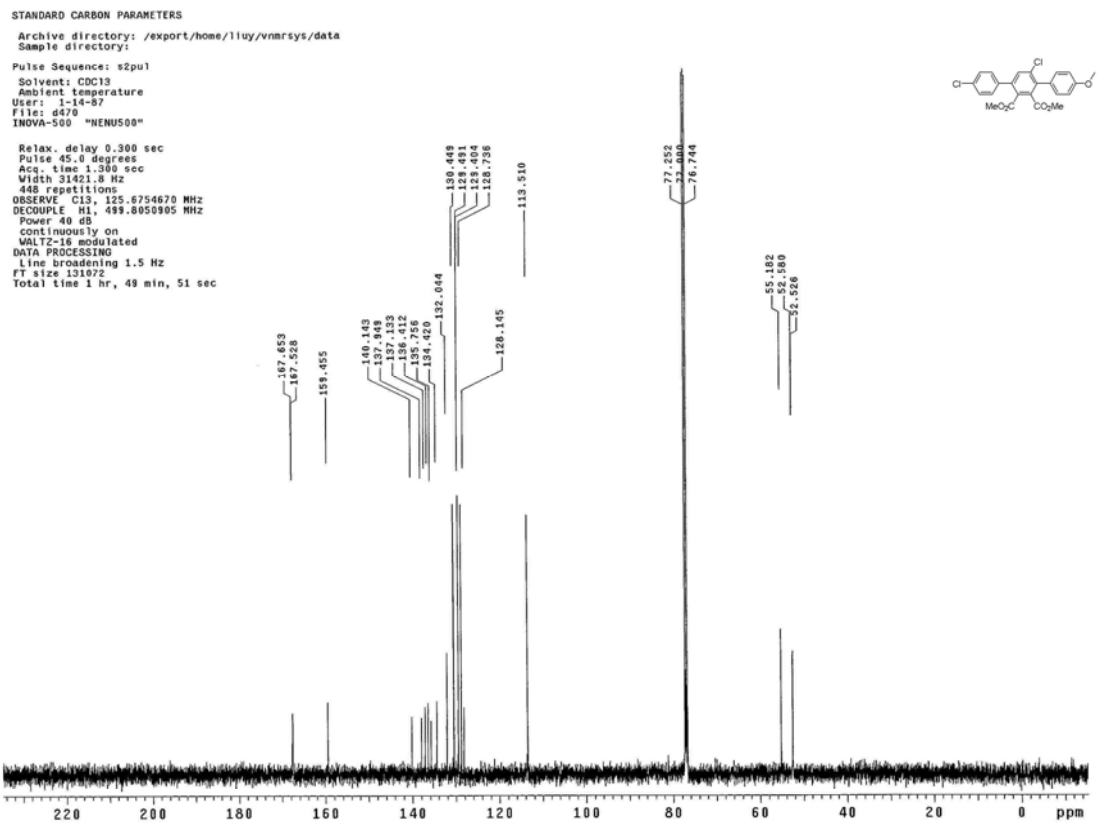
 Solvent: CDCl3
 Ambient temperature
User:  1-14-87
File: e141
INOVA-500  "NENU500"

 Relax. delay 0.300 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 4096 repetitions
OBSERVE  C13, 125.6754656 MHz
DECOUPLE  H1, 499.8050805 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
```






STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d469
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, 499.8025916 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 46 sec
3c
7.546
7.414
7.397
7.284
7.277
7.224
7.207
6.970
6.953
3.854
3.608
3.597
0.85
1.89
2.08
1.98
1.90
3.00
2.98
2.78
9
8
7
6
5
4
3
2
1
-0
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: d470
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
448 repetitions
OBSERVE C13, 125.6754670 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
167.653
167.528
159.455
140.143
137.949
137.133
136.612
135.756
134.426
132.044
130.449
129.491
129.404
128.736
128.145
113.510
77.252
77.000
76.744
55.182
52.580
52.526
220
200
180
160
140
120
100
80
60
40
20
0
ppm