

Advanced
**Synthesis &
Catalysis**

Supporting Information

© Copyright Wiley-VCH Verlag GmbH & Co. KGaA, 69451 Weinheim, 2008

Supporting Information

A Novel Enantiocomplementary C₂-Symmetric Chiral Bis(imidazoline) Ligands: Highly Enantioselective Friedel-Crafts Alkylation of Indoles with Ethyl 3,3,3-Trifluoropyruvate

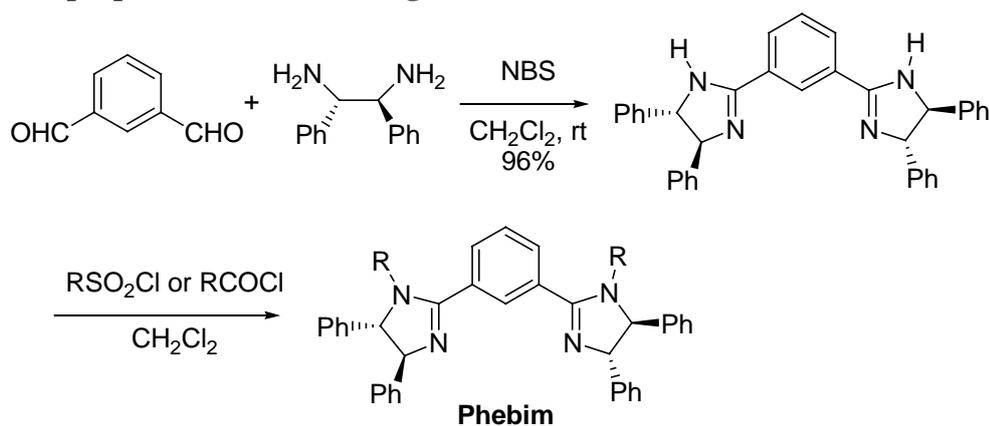
Shuichi Nakamura, Kengo Hyodo, Yuko Nakamura, Norio Shibata, and Takeshi Toru**

Department of Applied Chemistry, Graduate School of Engineering,

Nagoya Institute of Technology, Gokiso, Showa-ku, Nagoya 466-8555, Japan

General. All reactions were performed in oven-dried glassware under a positive pressure of nitrogen. Solvents were transferred via syringe and were introduced into the reaction vessels through a rubber septum. All of the reactions were monitored by thin-layer chromatography (TLC) carried out on 0.25 mm Merck silicagel (60-F254). The TLC plates were visualized with UV light and 7% phosphomolybdic acid or p-anisaldehyde in ethanol/ heat. Column chromatography was carried out on a column packed with silicagel 60N spherical neutral size 63-210 μ m. The ¹H-NMR (200 MHz), ¹⁹F-NMR (188 MHz), and ¹³C-NMR (151 MHz) spectra for solution in CDCl₃, were recorded on a Varian Gemini-200 or Bruker AVANCE600. Chemical shifts (δ) are expressed in ppm downfield from internal TMS, CFC₃ or CHCl₃. HPLC analyses were performed on a JASCO PU-2080 Plus or SHIMADZU LC-2010A HT using 4.6 x 250 mm CHIRALCEL OJ-H, OD-H, AD-H, CHIRALCEL IC column. Mass spectra were recorded on a SHIMADZU GCMS-QP5050A. APCI Mass spectra were recorded on a SHIMADZU LCMS-2050EV. Optical rotations were measured on a HORIBA SEPA-300. Infrared spectra were recorded on a JASCO FT/IR-200 spectrometer.

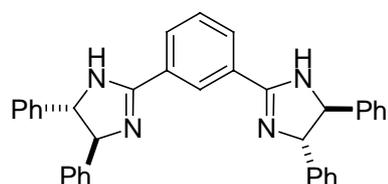
The preparation of Phebim ligand.



2c: R = <i>p</i> -TolSO ₂ : 93%	2j: R = CH ₃ SO ₂ : 56%
2d: R = PhSO ₂ : 90%	2k: R = CF ₃ SO ₂ : 92%
2e: R = 2,4,6-Me ₃ C ₆ H ₂ : 83%	2l: R = PhCO: 94%
2f: R = <i>p</i> -MeOC ₆ H ₄ SO ₂ : 78%	2m: R = <i>p</i> -MeOC ₆ H ₄ CO: 88%
2g: R = <i>p</i> -NO ₂ C ₆ H ₄ SO ₂ : 91%	2n: R = <i>p</i> -NO ₂ C ₆ H ₄ CO: 91%
2h: R = <i>p</i> -CF ₃ C ₆ H ₄ SO ₂ : 78%	2o: R = <i>p</i> -CF ₃ C ₆ H ₄ CO: 96%
2i: R = 3,5-(CF ₃) ₂ C ₆ H ₃ SO ₂ : 86%	2p: R = CH ₃ CO: 88%

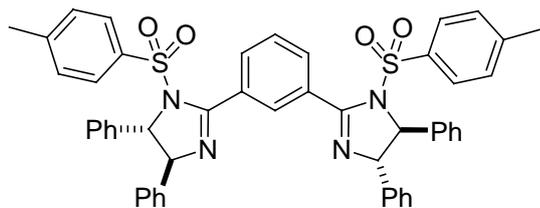
Typical Procedure for the preparation of Phebim ligand.

1,5-Bis[(4*S*,5*S*)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene

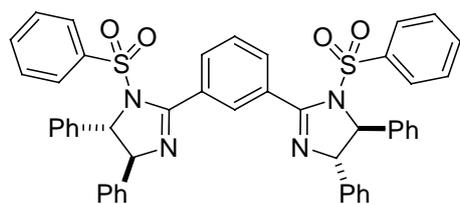


To a solution of (–)-diphenylethylenediamine (6.3 g, 29.8 mmol) and benzene-1,3-dicarbaldehyde (2.0 g, 14.9 mmol) in CH₂Cl₂ (280 mL) was slowly added a solution of *N*-bromosuccinimide (5.3 mg, 29.8 mmol) in CH₂Cl₂ (150 mL) at 0 °C, and the mixture was stirred overnight at room temperature. Saturated aqueous

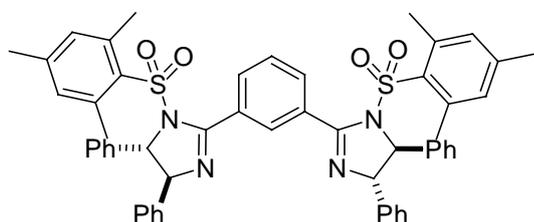
NaHCO₃ solution (300 mL) was added, and the mixture was extracted with CH₂Cl₂. The combined organic extracts were washed with brine, dried over Na₂SO₄, and concentrated under reduced pressure to leave a residue that was purified by recrystallization from ethyl acetate to give the crude (5.21 g). The purification of the mother liquid by column chromatography (hexane/ethyl acetate 20:80 with 1% of Et₃N) gave further crop of 1,5-bis[(4*S*,5*S*)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2.18 g, total 96%) as a white solid. $[\alpha]_{21}^D -39.5$ (*c* 0.70, CHCl₃); ¹H NMR (CDCl₃) δ 4.92 (br, 4H, CH), 5.58 (br, 2H, NH), 7.16-7.37 (m, 20H, Ar), 7.55 (t, *J* = 8.0 Hz, 1H, ArH), 8.10 (dd, *J* = 1.6, 7.8 Hz, 2H, ArH), 8.48 (s, 1H, ArH); ¹³C NMR (CDCl₃) δ 126.1, 126.6, 127.5, 128.7, 128.9, 129.9, 130.5, 143.2, 162.3; IR(KBr) 3030, 1507, 1490, 1456, 761, 698, 552 cm⁻¹; MS (APCI): 519.35 (M⁺).

1,5-Bis[(4*S*,5*S*)-1-(*p*-toluenesulfonyl)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2c).

To a solution of 1,5-bis[(4*S*,5*S*)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (500 mg, 0.97 mmol), DMAP (353 mg, 2.89 mmol) in CH₂Cl₂ (30 mL) was added *p*-toluenesulfonyl chloride (552 mg, 2.89 mmol) at 0 °C, and the mixture was stirred for 3 h. Saturated aqueous NH₄Cl was added, and the mixture was extracted with CH₂Cl₂. The combined organic extracts were washed with brine, dried over Na₂SO₄, and concentrated under reduced pressure to leave a residue that was purified by column chromatography (hexane/ethyl acetate 70:30) to give **2c** (744 mg, 93%) as a white solid. $[\alpha]_{21}^D$ 149.8 (*c* 0.75, CHCl₃); ¹H NMR (CDCl₃) δ 2.37 (s, 6H, CH₃), 4.97 (d, *J* = 4.8 Hz, 2H, CH), 5.11 (d, *J* = 4.8 Hz, 2H, CH), 6.65 (d, *J* = 8.0 Hz, 4H, Ar), 7.11–7.47 (m, 24H, Ar), 7.61 (t, *J* = 7.8 Hz, 1H, Ar), 8.12 (d, *J* = 7.8 Hz, 2H, Ar), 8.53 (s, 1H, Ar); ¹³C NMR (CDCl₃) δ 21.5, 72.7, 77.8, 125.8, 126.1, 127.2, 127.2, 128.0, 128.2, 128.6, 129.2, 130.0, 130.4, 131.1, 133.1, 133.8, 141.4, 142.3, 144.8, 159.1; IR (KBr): 3759, 3720, 1363, 1170, 699, 662 cm⁻¹; MS (APCI): 827.8 (M⁺); Anal. Calcd for C₅₀H₄₂N₄O₄S₂: C, 72.61; H, 5.12; N, 6.77. Found: C, 72.77; H, 5.02; N, 6.52.

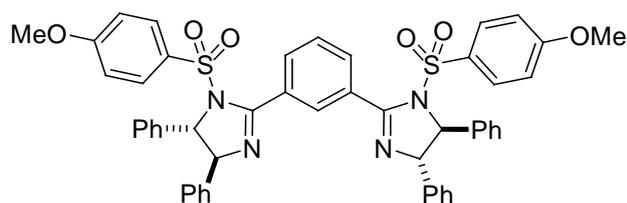
1,5-Bis[(4*S*,5*S*)-1-(benzenesulfonyl)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2d)

$[\alpha]_{22}^D$ +145.9 (*c* 0.71, CHCl₃); ¹H NMR (CDCl₃) δ 4.97 (d, *J* = 5.0 Hz, 2H, CH), 5.10 (d, *J* = 5.0 Hz, 2H, CH), 6.64 (d, *J* = 6.8 Hz, 4H, ArH), 7.08–7.24 (m, 6H, ArH), 7.34–7.64 (m, 21H, ArH), 8.10 (d, *J* = 7.6 Hz, 2H, ArH), 8.50 (s, 1H, ArH); ¹³C NMR (CDCl₃) δ 72.7, 78.0, 125.8, 126.1, 127.3, 127.5, 128.0, 128.3, 128.8, 129.2, 129.5, 130.4, 131.0, 133.0, 133.8, 136.9, 141.4, 142.1, 158.9; IR (KBr) 3061, 3032, 1366, 1173, 698, 606 cm⁻¹; MS (APCI): 799.75 (M⁺).

1,5-Bis[(4*S*,5*S*)-1-(2,4,6-trimethylbenzenesulfonyl)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2e)

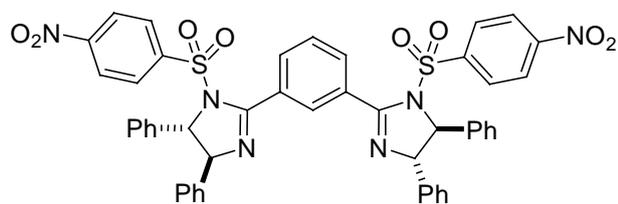
$[\alpha]_{22}^D$ +104.9 (*c* 0.65, CHCl₃); ¹H NMR (CDCl₃) δ 2.16 (s, 18H), 5.11 (d, *J* = 4.6 Hz, 2H), 5.20 (d, *J* = 4.6 Hz, 2H), 6.68 (s, 4H), 7.16–7.21 (m, 4H), 7.25–7.39 (m, 16H), 7.67–7.73 (m, 3H); ¹³C NMR (CDCl₃) δ 21.0, 22.4, 71.2, 79.1, 126.5, 126.7, 127.6, 127.9, 128.0, 128.8, 128.8, 130.0, 130.3, 131.7, 131.9, 131.9, 140.5, 141.2, 142.1, 143.8, 159.4; IR (KBr): 3903, 3871, 3855, 3735, 3650, 3035, 1339, 1160, 698, 661 cm⁻¹; MS (APCI): 883.57 (M⁺); Anal. Calcd for C₅₄H₅₀N₄O₄S₂: C, 72.36; H, 5.74; N, 6.05. Found: C, 72.18; H, 5.79; N, 6.01.

1,5-Bis[(4*S*,5*S*)-1-(4-methoxybenzenesulfonyl)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2f)



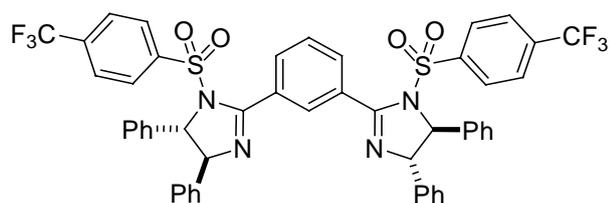
$[\alpha]_D^{22} +150.0$ (*c* 0.72, CHCl₃); ¹H NMR (CDCl₃) δ 3.80 (s, 6H, CH₃), 4.97 (d, *J* = 4.7 Hz, 2H, CH), 5.11 (d, *J* = 4.7 Hz, 2H, CH), 6.70-6.83 (m, 8H, ArH), 7.10-7.20 (m, 6H, ArH), 7.36-7.52 (m, 14H, ArH), 7.60 (t, *J* = 7.7 Hz, 1H, ArH), 8.11 (d, *J* = 7.8 Hz, 2H, ArH), 8.49 (s, 1H, ArH); ¹³C NMR (CDCl₃) δ 55.6, 72.7, 77.7, 114.6, 125.8, 126.2, 127.3, 127.3, 128.2, 128.4, 128.6, 129.2, 130.2, 130.5, 131.0, 133.0, 141.6, 142.3, 159.3, 163.8; IR (KBr): 3642, 3851, 3075, 3039, 1594, 1496, 1367, 1264, 1166, 699 cm⁻¹; MS (APCI): 861.46 (M⁺).

1,5-Bis[(4*S*,5*S*)-1-(4-nitrobenzenesulfonyl)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2g)



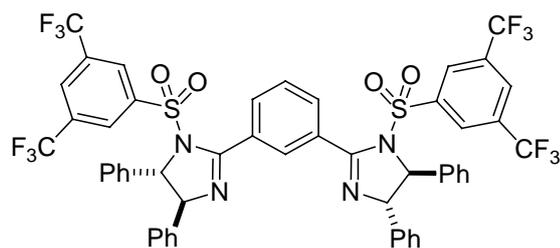
$[\alpha]_D^{23} +125.1$ (*c* 0.69, CHCl₃); ¹H NMR (CDCl₃) δ 5.10 (d, *J* = 3.8 Hz, 2H, CH), 5.27 (d, *J* = 3.8 Hz, 2H, CH), 6.81-6.86 (m, 4H, Ar), 7.10-7.19 (m, 6H, Ar), 7.35-7.48 (m, 10H, Ar), 7.61 (d, *J* = 8.8 Hz, 4H, ArH), 7.69 (t, *J* = 7.9 Hz, 1H, ArH), 8.01 (d, *J* = 8.6 Hz, 4H, ArH), 8.17 (d, *J* = 8.0 Hz, 2H, ArH), 8.46 (s, 1H, ArH); ¹³C NMR (CDCl₃) δ 73.0, 76.9, 124.4, 125.2, 126.0, 127.6, 128.0, 128.3, 128.7, 128.8, 128.9, 129.5, 130.0, 133.3, 141.1, 141.4, 142.1, 150.6, 157.9; IR (KBr): 3641, 3108, 3068, 3038, 1532, 1374, 1349, 1177, 739, 698, 685, 626 cm⁻¹; MS (APCI): 889.30 (M⁺).

1,5-Bis[(4*S*,5*S*)-1-(4-trifluoromethylbenzenesulfonyl)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2h)



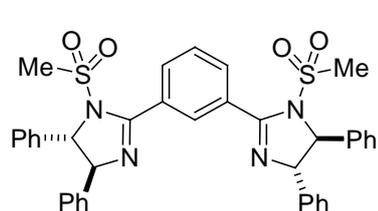
$[\alpha]_D^{22} +129.5$ (*c* 0.70, CHCl₃); ¹H NMR (CDCl₃) δ 5.01 (d, *J* = 4.2 Hz, 2H, CH), 5.21 (d, *J* = 4.2 Hz, 2H, CH), 6.71 (d, *J* = 6.4 Hz, 4H, Ar), 7.13-7.20 (m, 6H, Ar), 7.41-7.68 (m, 19H, Ar), 8.17 (dd, *J* = 1.6, 7.8 Hz, 2H, ArH), 8.57 (s, 1H, ArH); ¹³C NMR (CDCl₃) δ 73.0, 77.4, 122.9 (q, *J* = 273 Hz), 125.3, 126.1, 126.5 (q, *J* = 3.5 Hz), 127.7, 127.7, 128.4, 128.6, 128.8, 129.4, 130.2, 131.2, 133.3, 135.4 (q, *J* = 33.0 Hz), 140.2, 141.3, 141.5, 158.3; IR (KBr): 3910, 3730, 3562, 3039, 3065, 1646, 1322, 1175, 1063, 714, 698, 626 cm⁻¹; MS (APCI): 936.32 (M⁺).

1,5-Bis[(4*S*,5*S*)-1-[3,5-bis(trifluoromethyl)benzenesulfonyl]-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2i)



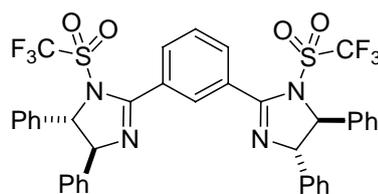
$[\alpha]_D^{22} +109.8$ (c 0.71, CHCl_3); $^1\text{H NMR}$ (CDCl_3) δ 5.17 (d, $J = 4.0$ Hz, 2H, CH), 5.28 (d, $J = 4.0$ Hz, 2H, CH), 6.96-7.01 (m, 4H, Ar), 7.21-7.24 (m, 6H, Ar), 7.38-7.47 (m, 10H, Ar), 7.58 (t, $J = 7.8$ Hz, 1H, ArH), 7.81-7.94 (m, 8H, ArH), 8.52 (s, 1H, ArH); $^{19}\text{F NMR}$ (CDCl_3) δ -62.9 (s); $^{13}\text{C NMR}$ (CDCl_3) δ 72.8, 77.2, 122.6 (q, $J = 274$ Hz), 125.2, 126.1, 127.1 (t, $J = 3.5$ Hz), 127.81, 127.85, 128.0, 128.9, 128.9, 129.5, 130.4, 131.3, 132.1, 132.9 (q, $J = 34.7$ Hz), 140.5, 140.8, 141.1, 157.4; IR (KBr): 3078, 3040, 1281, 1175, 1144, 697, 637 cm^{-1} ; MS (APCI): 1071.32 (M^+).

1,5-Bis[(4*S*,5*S*)-1-(methanesulfonyl)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2j)



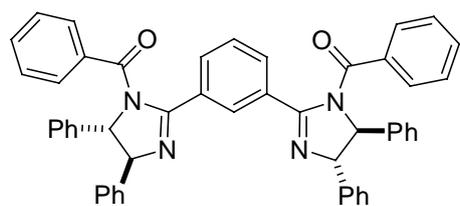
$[\alpha]_D^{23} +75.3$ (c 0.72, CHCl_3); $^1\text{H NMR}$ (CDCl_3) δ 2.74 (s, 6H, CH_3), 5.26 (d, $J = 3.0$ Hz, 4H, CH), 7.23-7.43 (m, 20H, Ar), 7.61 (t, $J = 7.8$ Hz, 1H, ArH), 8.09 (d, $J = 7.8$ Hz, 2H, ArH), 8.40 (s, 1H, Ar); $^{13}\text{C NMR}$ (CDCl_3) δ 41.3, 72.6, 77.5, 126.1, 127.9, 128.2, 128.6, 129.2, 129.3, 129.4, 129.5, 130.1, 130.4, 132.3, 141.2, 141.6, 158.2; IR (KBr): 3850, 3735, 3651, 3562, 3038, 3005, 2920, 1640, 1357, 1166, 763, 699 cm^{-1} ; MS (APCI): 674.95 (M^+); Anal. Calcd for $\text{C}_{38}\text{H}_{34}\text{N}_4\text{O}_4\text{S}_2$: C, 67.63; H, 5.08; N, 8.30. Found: C, 67.4; H, 5.26; N, 8.04.

1,5-Bis[(4*S*,5*S*)-1-[trifluoromethanesulfonyl]-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2k)



$[\alpha]_D^{22} +48.4$ (c 0.70, CHCl_3); $^1\text{H NMR}$ (CDCl_3) δ 5.30 (d, $J = 3.8$ Hz, 2H, CH), 5.40 (d, $J = 3.8$ Hz, 2H, CH), 7.24-7.44 (m, 20H, Ar), 7.63 (t, $J = 7.8$ Hz, 1H, ArH), 8.11 (dd, $J = 1.4, 7.8$ Hz, 2H, ArH), 8.35 (s, 1H, ArH); $^{19}\text{F NMR}$ (CDCl_3) δ -73.7 (s); $^{13}\text{C NMR}$ (CDCl_3) δ 73.4, 78.1, 119.3 (q, $J = 324$ Hz), 125.8, 125.9, 128.1, 128.5, 129.0, 129.0, 129.2, 129.5, 130.0, 132.7, 139.6, 140.5, 155.7; IR (KBr): 3738, 3643, 3039, 1654, 1406, 1217, 1202, 1145, 698, 634 cm^{-1} ; MS (APCI): 783.32 (M^+).

1,5-Bis[(4*S*,5*S*)-1-benzoyl-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2l)

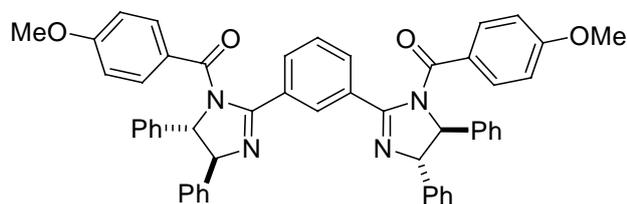


$[\alpha]_D^{23} +35.7$ (c 0.71, CHCl_3); $^1\text{H NMR}$ (CDCl_3) δ 5.09 (d, $J = 3.1$ Hz, 2H, CH), 5.19 (d, $J = 3.1$ Hz, 2H, CH), 7.09-7.38 (m, 31H, Ar), 7.72 (d, $J = 7.8$ Hz, 2H, ArH), 8.23 (s, 1H, ArH); $^{13}\text{C NMR}$ (CDCl_3) δ 72.3, 78.4, 125.6, 126.3, 127.9, 128.0, 128.1, 128.5, 128.8, 129.0, 129.2, 130.0, 130.6, 131.2, 131.8, 134.5, 140.3, 141.9, 160.6, 170.3; IR (KBr): 3641, 3038, 3075, 1671, 1321, 1262, 697 cm^{-1} ; MS

(APCI): 728.56 (M^+).

1,5-Bis[(4*S*,5*S*)-1-(4-methoxybenzoyl)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene

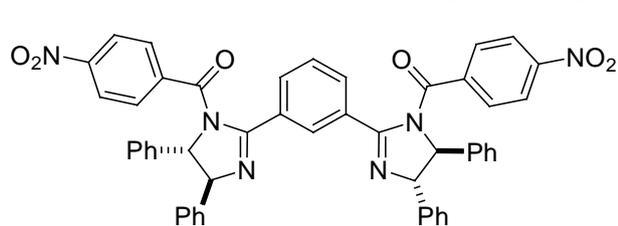
(2m)



$[\alpha]_D^{22} +81.6$ (*c* 0.70, $CHCl_3$); 1H NMR ($CDCl_3$) δ 3.74 (s, 6H, CH_3), 5.09 (d, *J* = 2.8 Hz, 2H, CH), 5.16 (d, *J* = 2.8 Hz, 2H, CH), 6.63 (d, *J* = 8.6 Hz, 4H, Ar), 7.24-7.39 (m, 25H, Ar), 7.75 (d, *J* = 7.8 Hz, 2H, ArH), 8.32 (s, 1H, ArH); ^{13}C

NMR ($CDCl_3$) δ 55.3, 72.5, 78.6, 113.3, 125.5, 126.4, 126.6, 127.9, 128.0, 128.8, 129.0, 129.2, 130.5, 130.8, 131.4, 140.4, 142.2, 161.2, 162.5, 170.1; IR (KBr): 3645, 3075, 3037, 2840, 1665, 1604, 1310, 1257, 1171, 698 cm^{-1} ; MS (APCI): 788.51 (M^+).

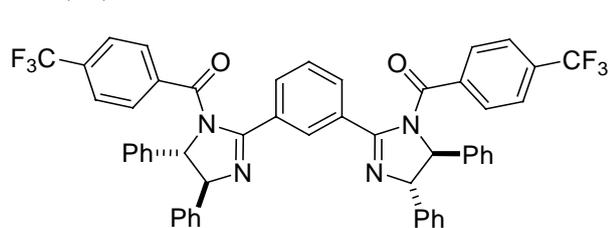
1,5-Bis [(4*S*,5*S*)-1-(4-nitrobenzoyl)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2n)



$[\alpha]_D^{22} +17.6$ (*c* 0.71, $CHCl_3$); 1H NMR ($CDCl_3$) δ 5.11 (d, *J* = 3.2 Hz, 2H, CH), 5.25 (d, *J* = 3.2 Hz, 2H, CH), 7.23-7.47 (m, 25H, Ar), 7.72 (dd, *J* = 1.6, 7.8 Hz, 2H, ArH), 7.98-8.03 (m, 5H, ArH); ^{13}C NMR ($CDCl_3$) δ 72.2, 78.2, 123.3, 125.6,

126.1, 128.3, 128.4, 128.6, 128.8, 129.2, 129.3, 129.5, 130.7, 131.1, 139.9, 140.0, 141.0, 149.3, 159.2, 167.7; IR (KBr): 3730, 3065, 3038, 1676, 1524, 1342, 698 cm^{-1} ; MS (APCI): 818.49 (M^+); Anal. Calcd for $C_{50}H_{36}N_6O_6$: C, 73.52; H, 4.44; N, 10.29. Found: C, 73.23; H, 4.53; N, 10.00.

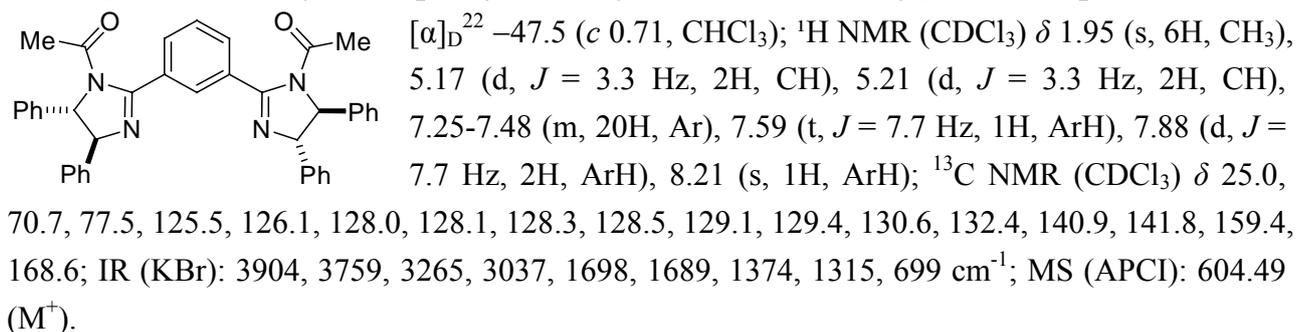
1,5-Bis[(4*S*,5*S*)-1-(4-trifluoromethylbenzoyl)-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2o)



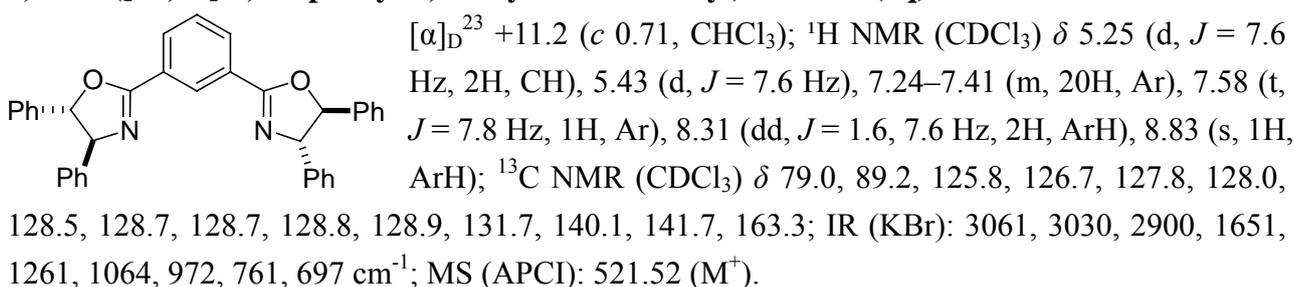
$[\alpha]_D^{22} +58.4$ (*c* 0.71, $CHCl_3$); 1H NMR ($CDCl_3$) δ 5.07 (d, *J* = 3.1 Hz, 2H, CH), 5.25 (d, *J* = 3.1 Hz, 2H, CH), 7.17-7.42 (m, 29H, Ar), 7.66 (d, *J* = 7.8 Hz, 2H, ArH), 8.29 (s, 1H, ArH); ^{19}F NMR ($CDCl_3$) δ -63.2 (s); ^{13}C NMR ($CDCl_3$) δ 72.3,

78.4, 123.4 (q, *J* = 273 Hz), 125.1 (q, *J* = 3.8 Hz), 125.6, 126.2, 128.0, 128.2, 128.4, 128.7, 129.0, 129.1, 129.4, 130.7, 131.0, 133.1 (q, *J* = 32.7 Hz), 137.8, 140.0, 141.4, 160.0, 168.7; IR (KBr): 3077, 3035, 1675, 1322, 1130, 1066, 698 cm^{-1} ; MS (APCI): 864.52 (M^+).

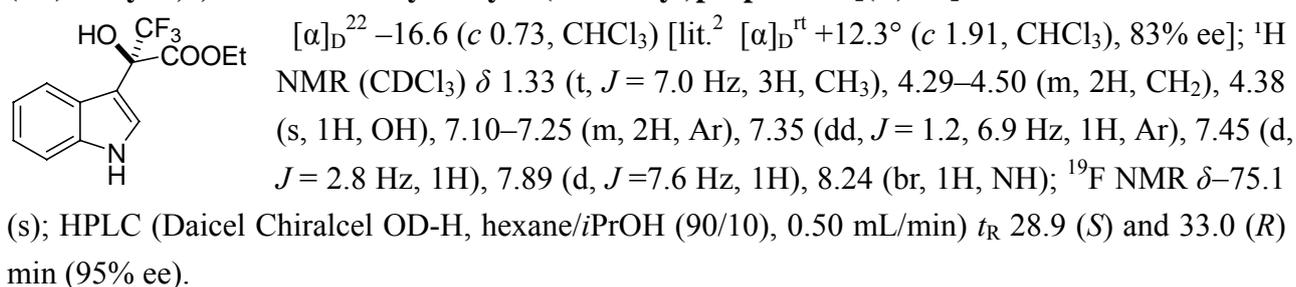
1,5-Bis[(4*S*,5*S*)-1-acetyl-4,5-diphenyl-4,5-dihydro-1*H*-imidazol-2-yl]benzene (2p)



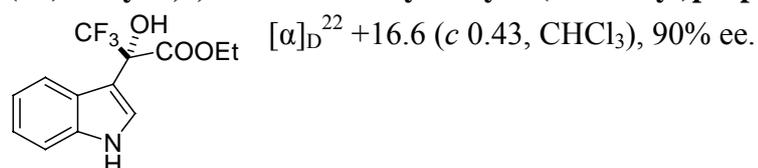
1,5-Bis-([4*S*,5*S*]-4,5-diphenyl-4,5-dihydro-oxazol-2-yl)-benzene (2q)¹



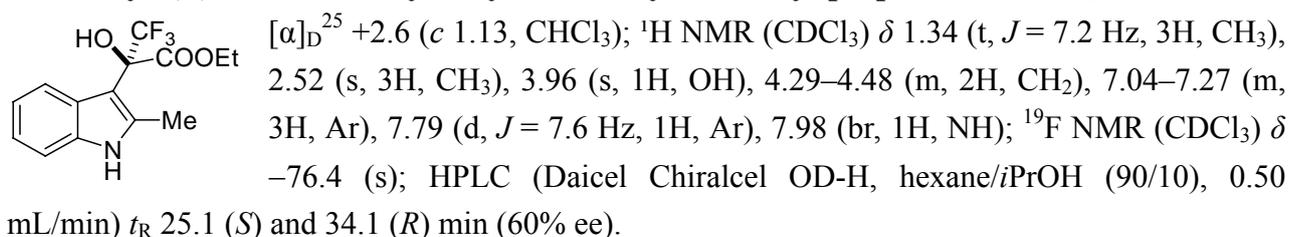
(2*R*)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(indol-3-yl)propionate [(*R*)-3a]



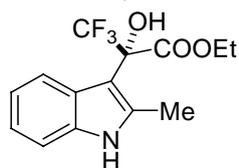
(2*S*)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(indol-3-yl)propionate [(*S*)-3a]



(2*R*)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(2-methyl-indol-3-yl)propionate [(*R*)-3b]



(2S)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(2-methyl-indol-3-yl)propionate [(S)-3b]



$[\alpha]_D^{25} -1.0$ (*c* 0.40, CHCl₃), 67% ee.

(2R)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-methyl-indol-3-yl)propionate [(R)-3c]



$[\alpha]_D^{25} -3.5$ (*c* 0.80, CHCl₃); ¹H NMR (CDCl₃) δ 1.35 (t, *J* = 7.2 Hz, 3H, CH₃), 2.44 (s, 3H, CH₃), 4.29–4.50 (m, 2H, CH₂), 4.37 (s, 1H, OH), 7.03 (dd, *J* = 1.0, 8.2 Hz, 1H, Ar), 7.22–7.26 (m, 1H, Ar), 7.40 (d, *J* = 2.8 Hz, 1H, Ar), 7.66 (s, 1H, Ar), 8.17 (br, 1H, NH); ¹⁹F NMR (CDCl₃) δ -76.5

(s); HPLC (Daicel Chiralcel OJ-H, hexane/*i*PrOH (80/20), 1.0 mL/min) *t*_R 20.0 (*S*) and 23.8 (*R*) min (90% ee).

(2S)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-methyl-indol-3-yl)propionate [(S)-3c]



$[\alpha]_D^{25} +5.9$ (*c* 0.51, CHCl₃), 93% ee.

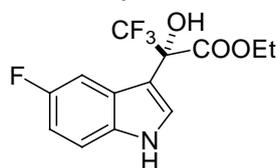
(2R)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-fluoro-indol-3-yl)propionate [(R)-3d]



$[\alpha]_D^{25} -16.0$ (*c* 0.36, CHCl₃); ¹H NMR (CDCl₃) δ 1.36 (t, *J* = 7.0 Hz, 3H, CH₃), 4.32–4.51 (m, 2H, CH₂), 4.38 (s, 1H, OH), 6.96 (dt, *J* = 2.4, 8.8 Hz, 1H, Ar), 7.24–7.31 (m, 1H, Ar), 7.50 (d, *J* = 2.8 Hz), 7.57 (dd, *J* = 2.2, 10.3 Hz), 8.27 (br, 1H, NH); ¹⁹F NMR (CDCl₃) δ -76.9 (s, 3F, CF₃), -122.6 (dt, *J* = 5.6, 9.2 Hz, 1F, CF); HPLC (Daicel Chiralcel OJ-H, hexane/*i*PrOH (90/10), 0.50 mL/min) *t*_R

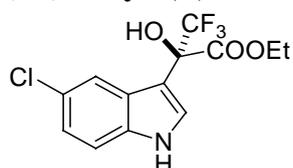
68.5 (*S*) and 73.6 (*R*) min (86% ee).

(2S)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-fluoro-indol-3-yl)propionate [(S)-3d]



$[\alpha]_D^{25} +18.3$ (*c* 0.78, CHCl₃), 91% ee.

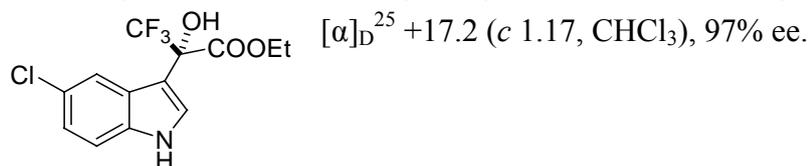
(2R)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-chloro-indol-3-yl)propionate [(R)-3e]



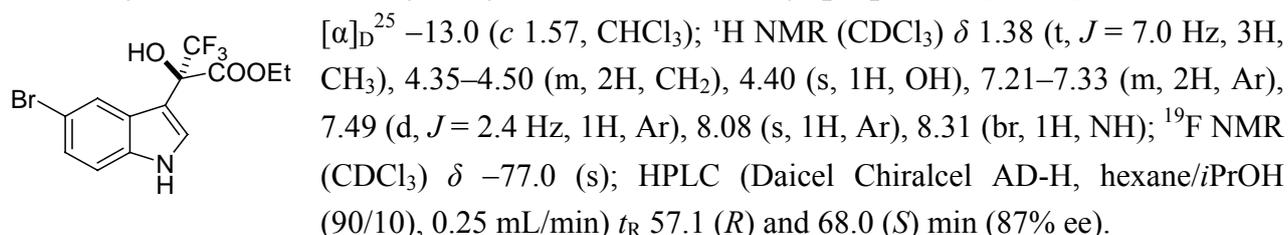
$[\alpha]_D^{25} -14.4$ (*c* 1.37, CHCl₃); ¹H NMR (CDCl₃) δ 1.38 (t, *J* = 7.2 Hz, 3H, CH₃), 4.33–4.51 (m, 2H, CH₂), 4.39 (s, 1H, OH), 7.14–7.31 (m, 2H, Ar), 7.50 (d, *J* = 2.8 Hz, 1H, Ar), 7.91 (s, 1H, Ar), 8.30 (br, 1H, NH); ¹⁹F NMR (CDCl₃) δ -77.0 (s); HPLC (Daicel Chiralcel OD-H, hexane/*i*PrOH (95/5),

0.50 mL/min) t_R 51.2 (*S*) and 59.7 (*R*) min (93% ee).

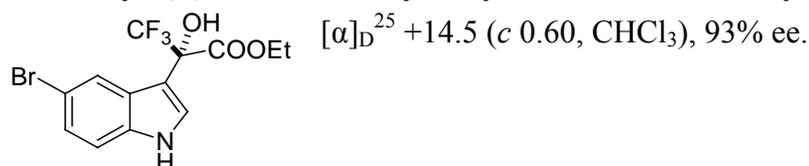
(2*S*)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-chloro-indol-3-yl)propionate [(*S*)-3e]



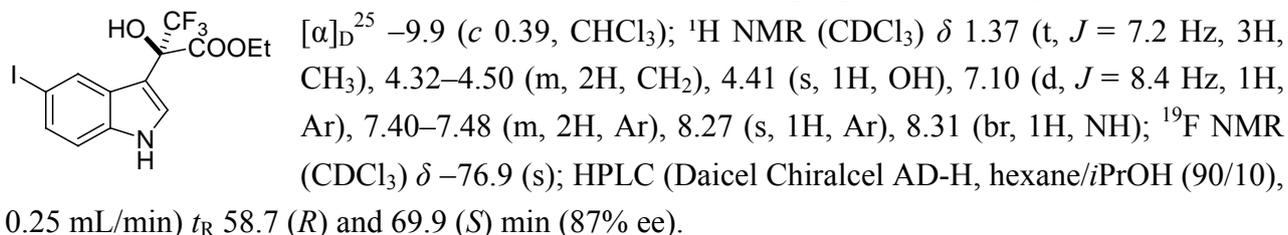
(2*R*)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-bromo-indol-3-yl)propionate [(*R*)-3f]



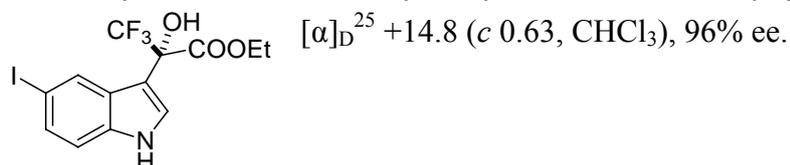
(2*S*)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-bromo-indol-3-yl)propionate [(*S*)-3f]



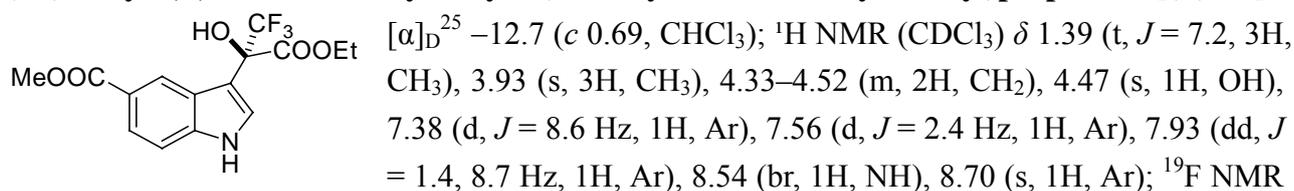
(2*R*)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-iodo-indol-3-yl)propionate [(*R*)-3g]



(2*S*)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-iodo-indol-3-yl)propionate [(*S*)-3g]

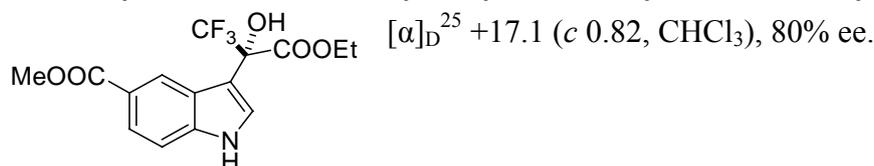


(2*R*)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-methyl-indol-carboxylate-3-yl)propionate [(*R*)-3h]

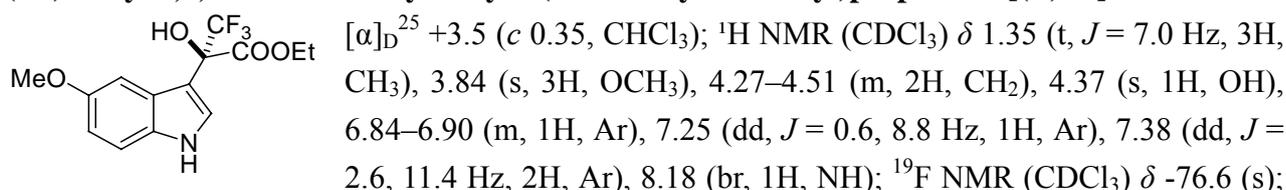


(CDCl₃) δ -76.9 (s); HPLC (Daicel Chiralcel OJ-H, hexane/*i*PrOH (85/15), 1.0 mL/min) t_R 30.9 (S) and 36.0 (R) min (51% ee).

(2S)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-methyl-indol-3-yl)propionate [(S)-3h]

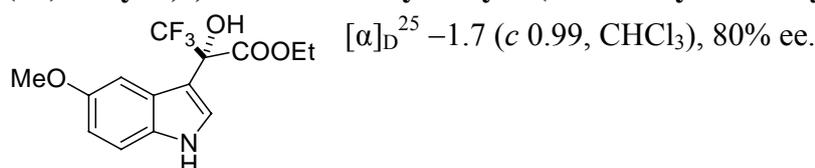


(2R)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-methoxy-indol-3-yl)propionate [(R)-3i]

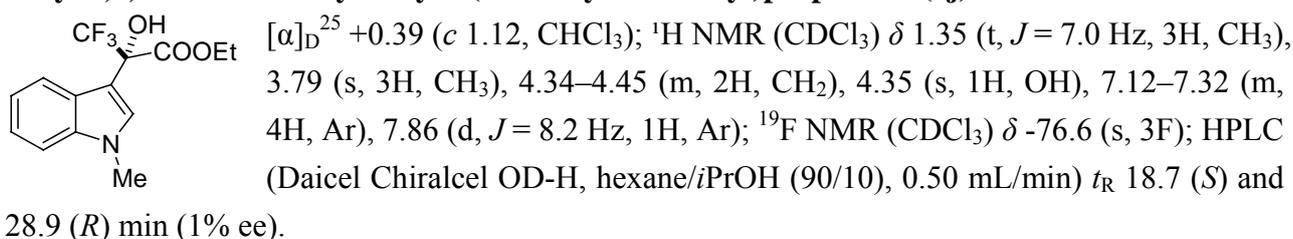


HPLC (Daicel Chiralcel OD-H, hexane/*i*PrOH (90/10), 1.0 mL/min) t_R 17.6 (S) and 23.8 (R) min (57% ee).

(2S)-Ethyl 3,3,3-trifluoro-2-hydroxy-2-(5-methoxy-indol-3-yl)propionate [(S)-3i]

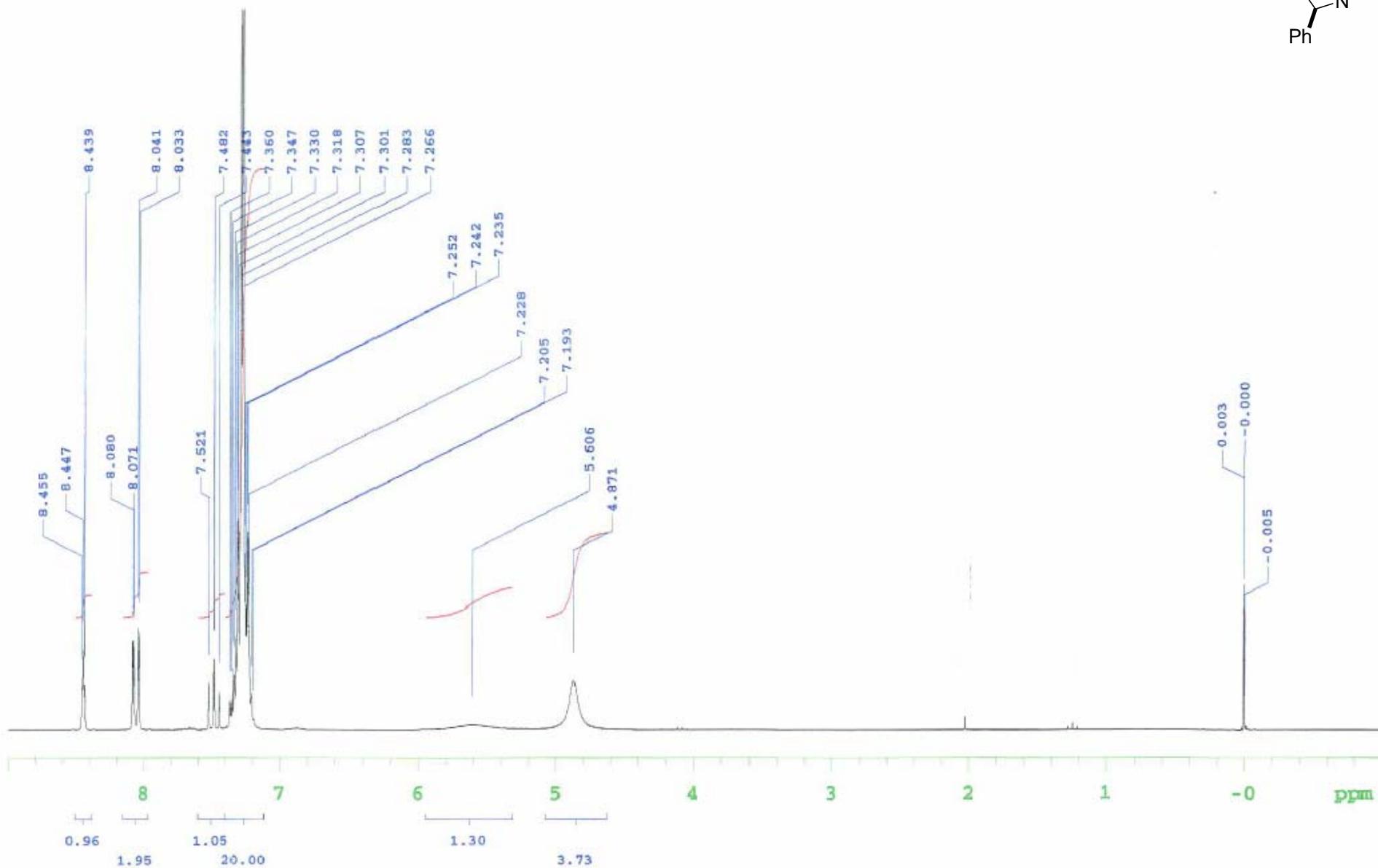
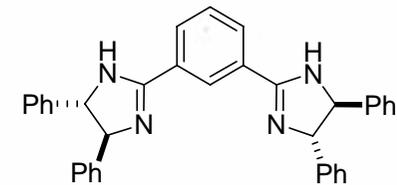


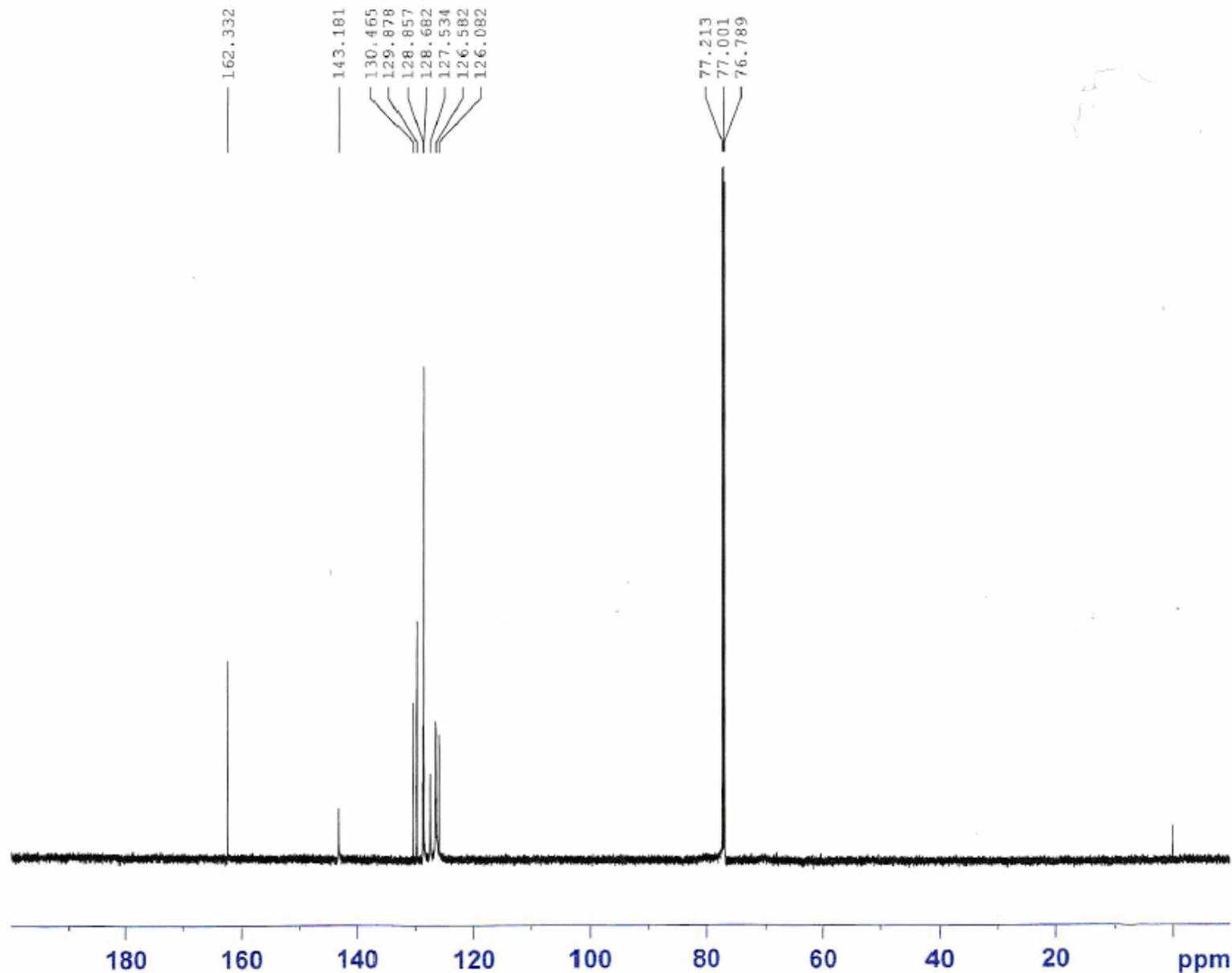
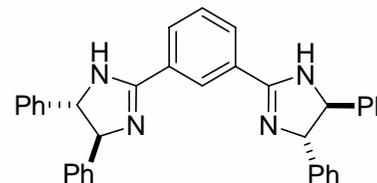
Ethyl 3,3,3-trifluoro-2-hydroxy-2-(N-methyl-indol-3-yl)propionate (3j)



¹ Y. Kanazawa, Y. Tsuchiya, K. Kobayashi, T. Shiomi, J. Itoh, M. Kikuchi, Y. Yamamoto, H. Nishiyama, *Chem. Eur. J.* **2006**, *12*, 63.

² W. Zhuang, N. Gathergood, R. G. Hazell, K. A. Jørgensen, *J. Org. Chem.* **2001**, *66*, 1009.





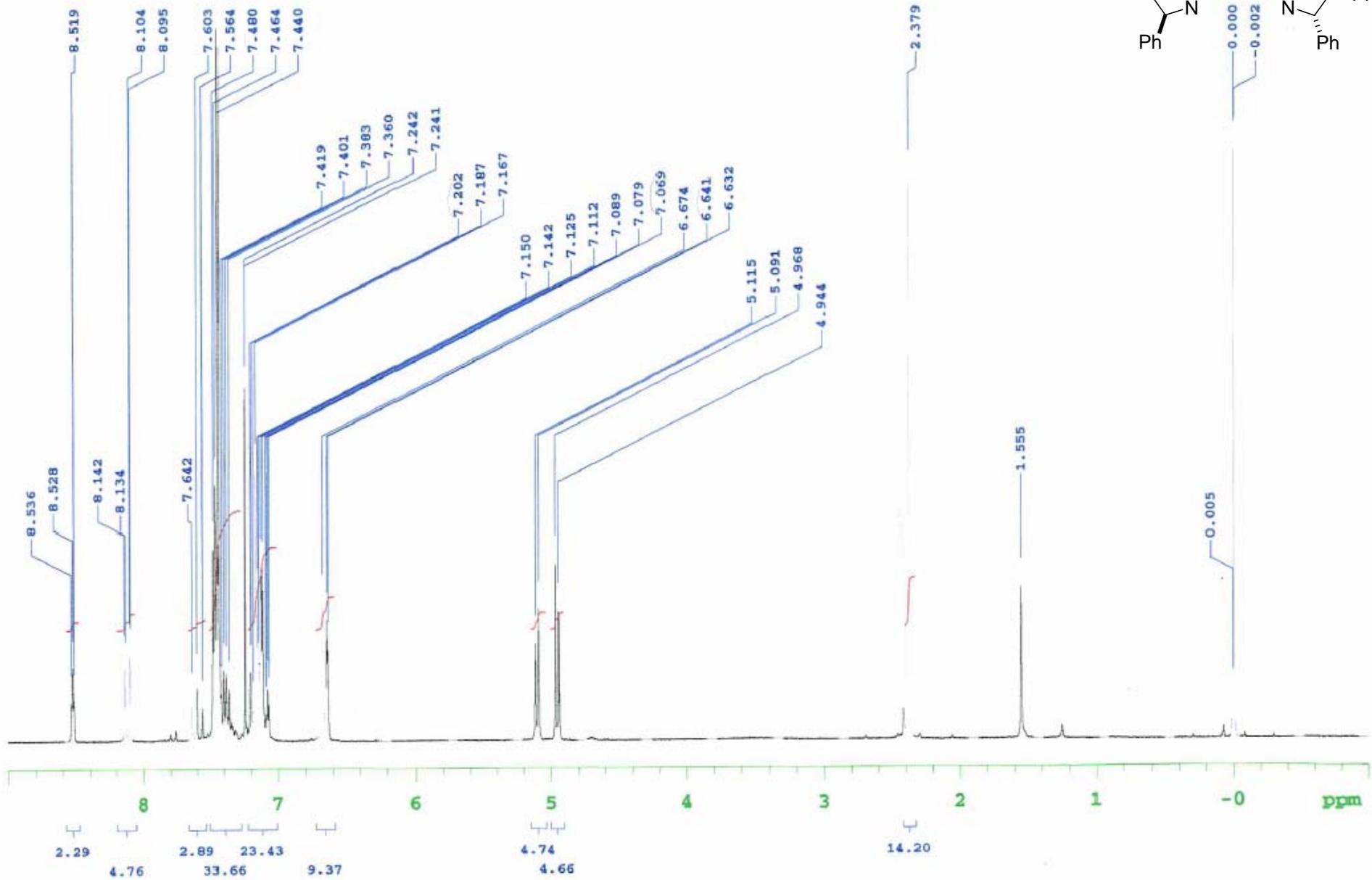
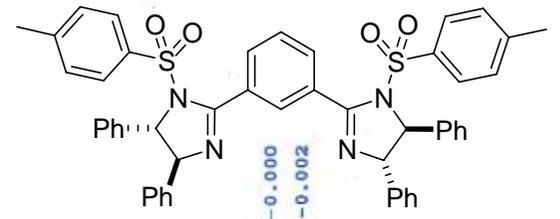
Current Data Parameters
 NAME NY-298
 EXPNO 10
 PROCNO 1

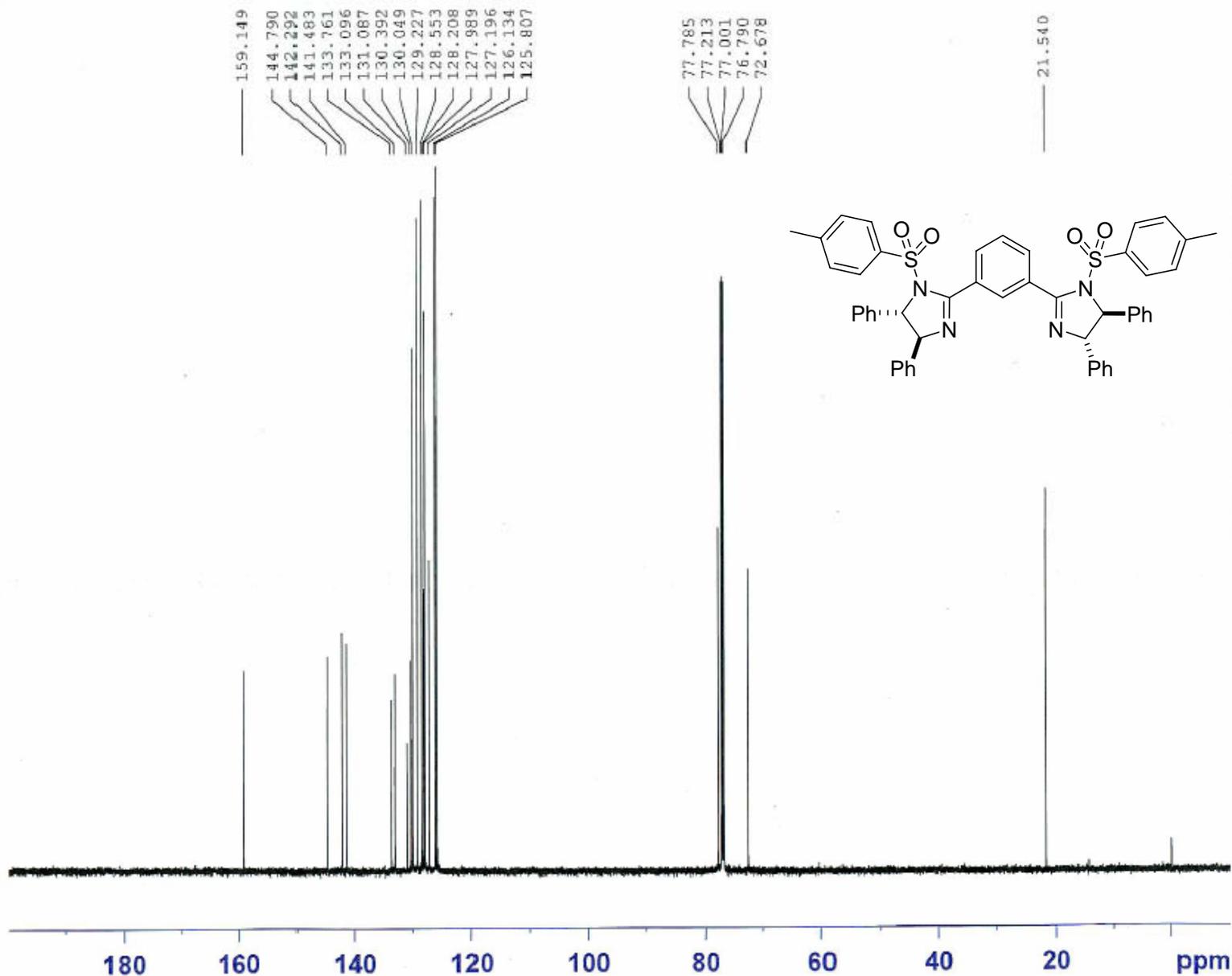
F2 - Acquisition Parameters
 Date_ 20071223
 Time_ 12.59
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 7298.2
 DW 11.000 usec
 DE 6.00 usec
 TE 297.6 K
 D1 0.60000002 sec
 d11 0.03000000 sec
 DELTA 0.50000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SF01 150.9223664 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SF02 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028169 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





Current Data Parameters
 NAME NY-311-2
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters

Date 20071223
 Time 20.01
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 6502
 DW 11.000 usec
 DE 6.00 usec
 TE 297.7 K
 D1 0.60000002 sec
 d11 0.03000000 sec
 DELTA 0.50000000 sec
 TD0 1

===== CHANNEL f1 =====

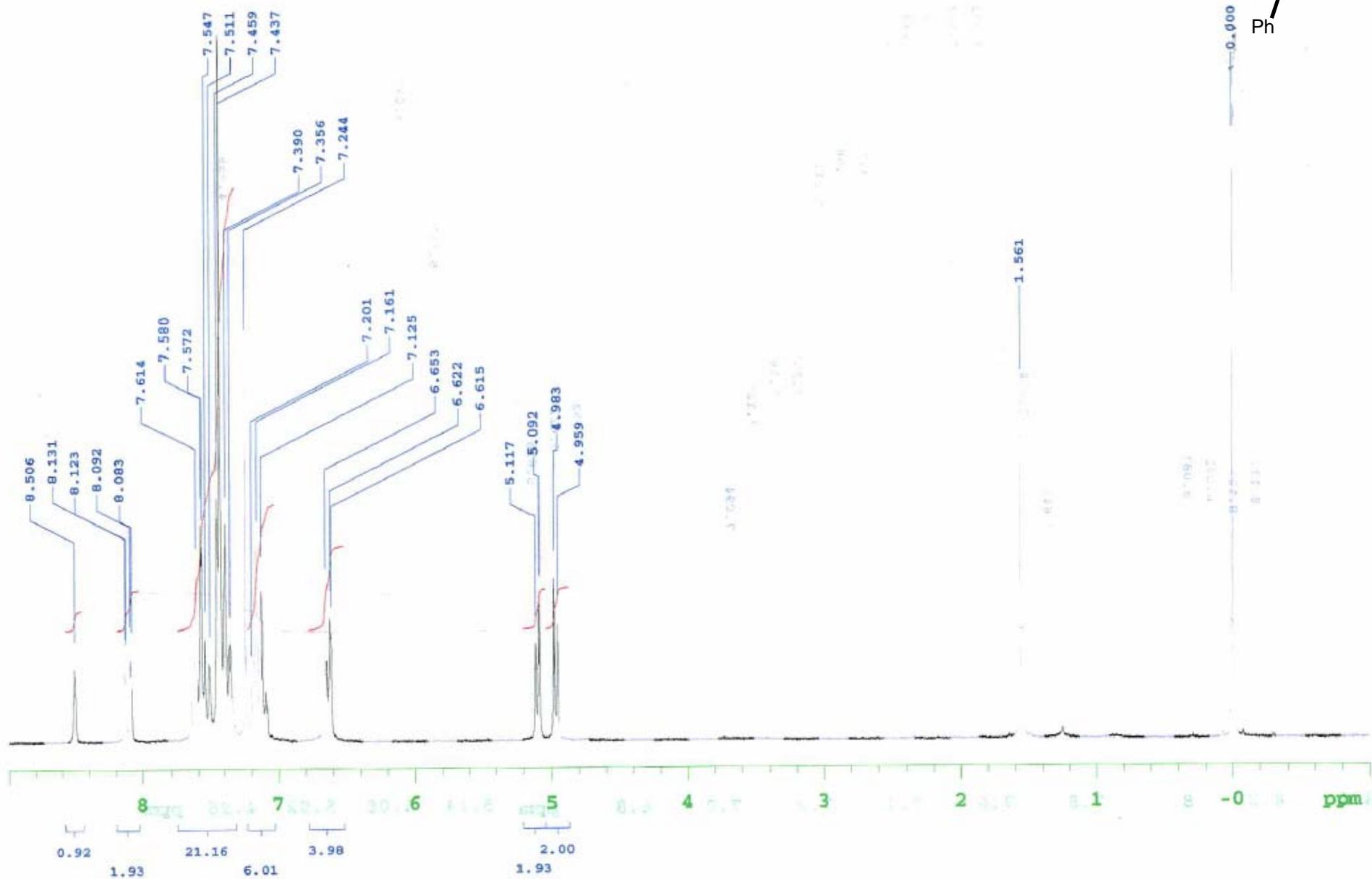
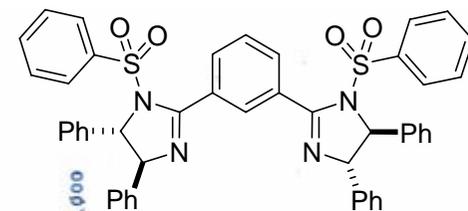
NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

===== CHANNEL f2 =====

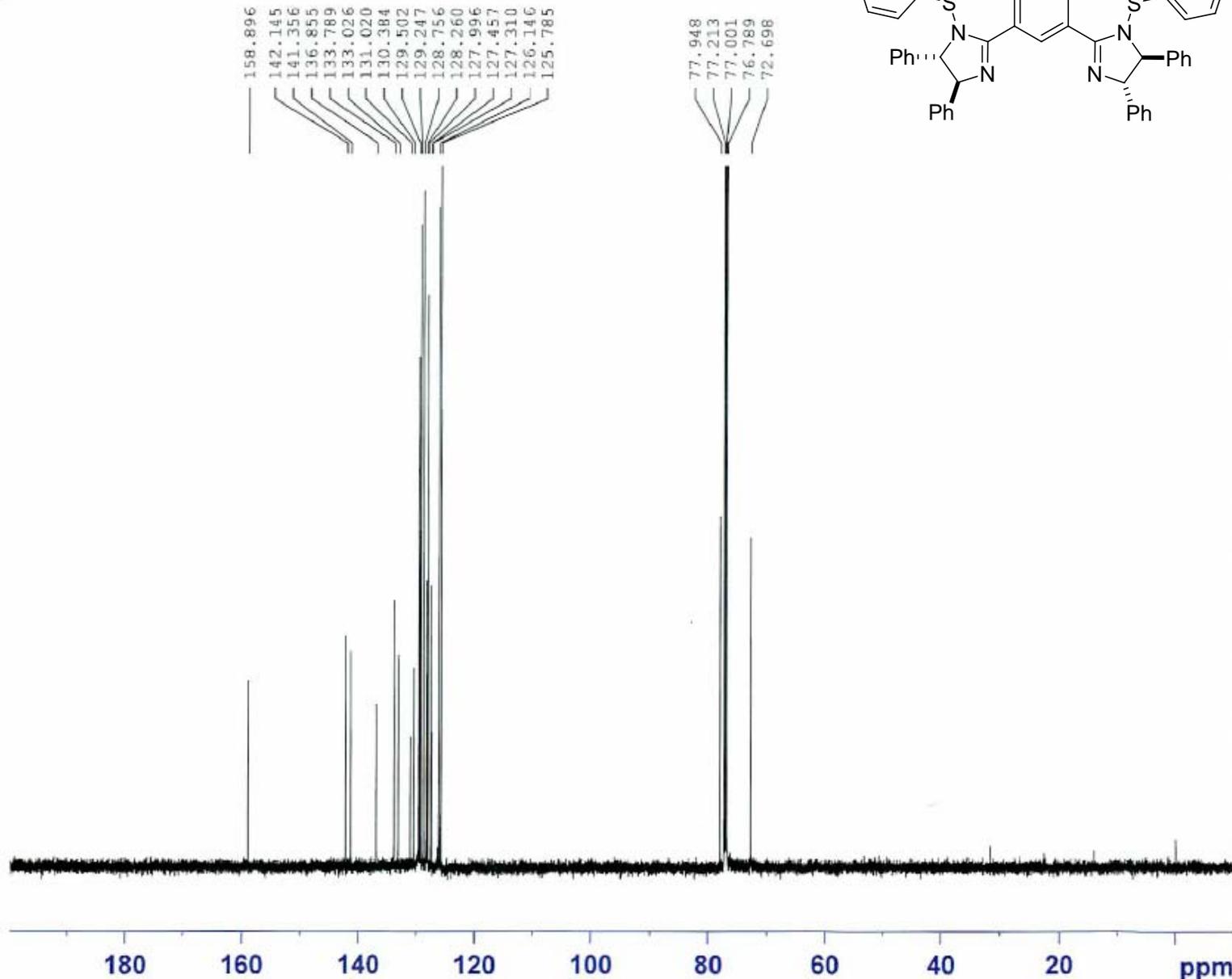
CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters

SI 131072
 SF 150.9028205 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



13C



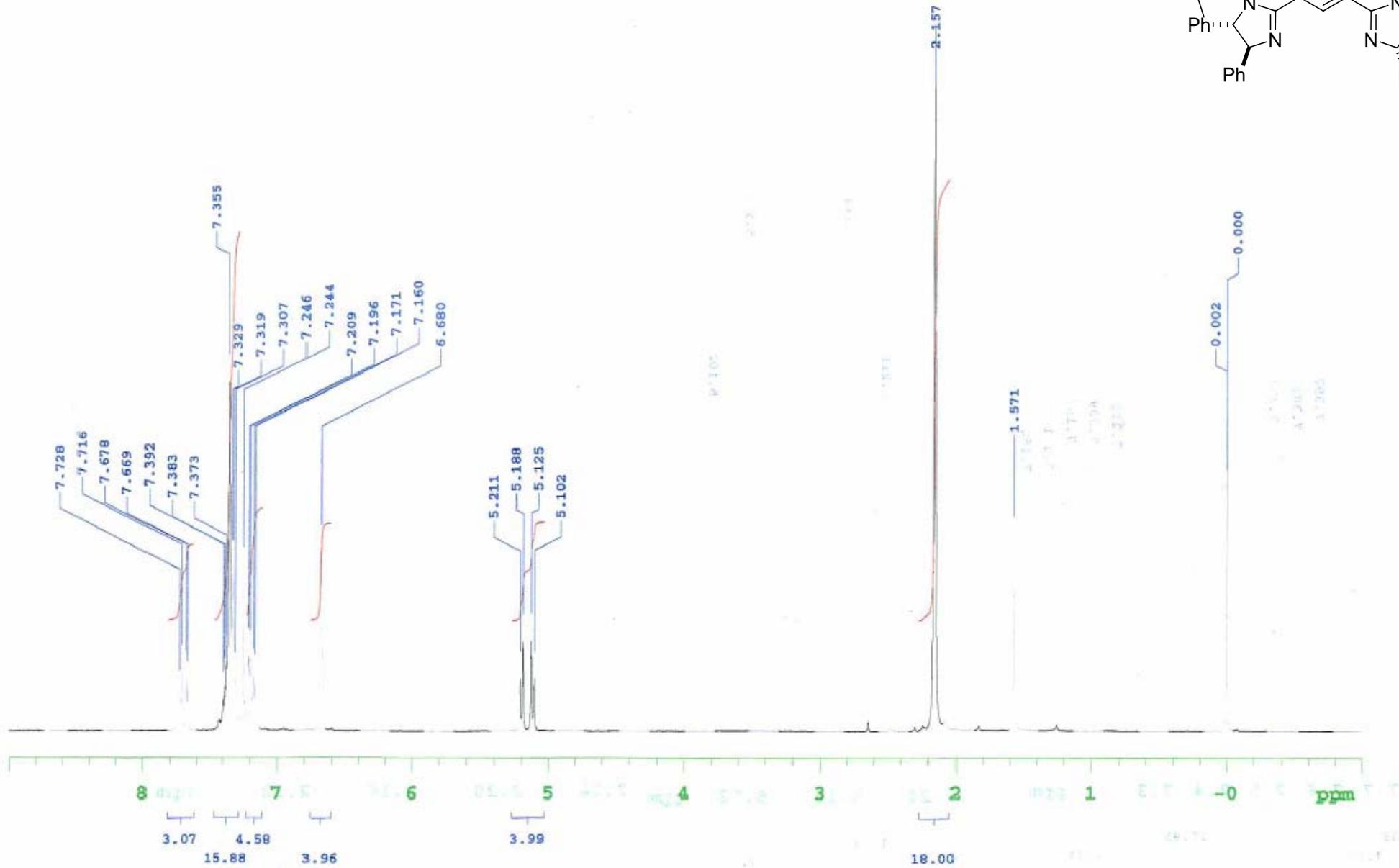
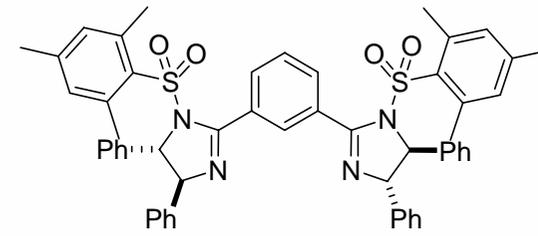
Current Data Parameters
 NAME NY-305
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071217
 Time 22.36
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 218
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 5160.6
 DW 11.000 usec
 DE 6.00 usec
 TE 297.5 K
 D1 0.60000002 sec
 d11 0.03000000 sec
 DELTA 0.50000000 sec
 TD0 1

----- CHANNEL f1 -----
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028188 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





Current Data Parameters
 NAME HK-421
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071223
 Time_ 18.11
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDC13
 NS 512
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 7298.2
 DW 11.000 usec
 DE 6.00 usec
 TE 297.8 K
 D1 0.60000002 sec
 d11 0.03000000 sec
 DELTA 0.50000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

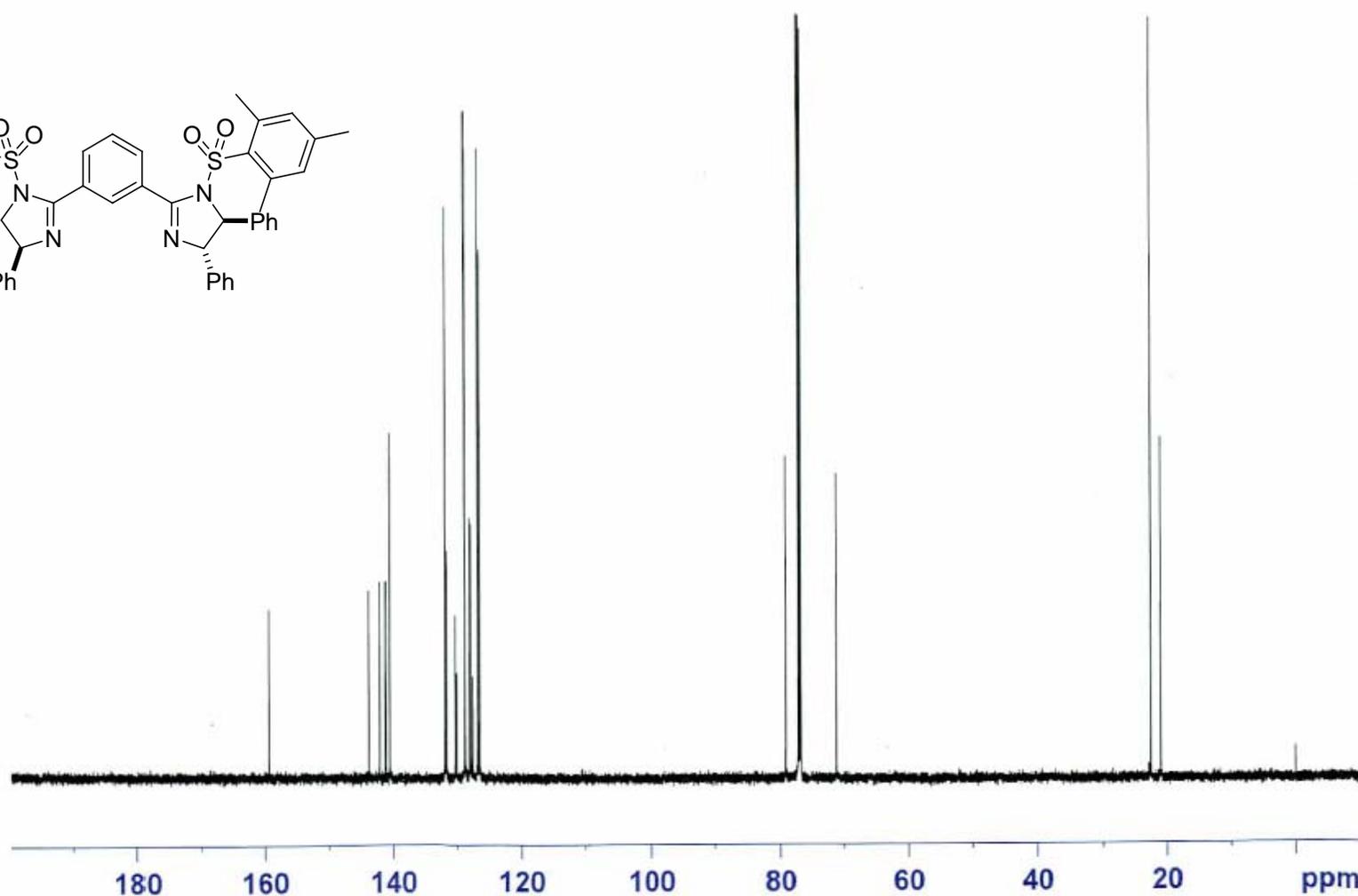
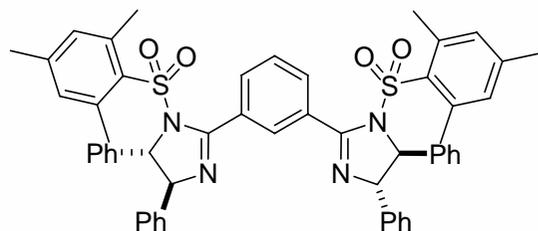
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028173 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

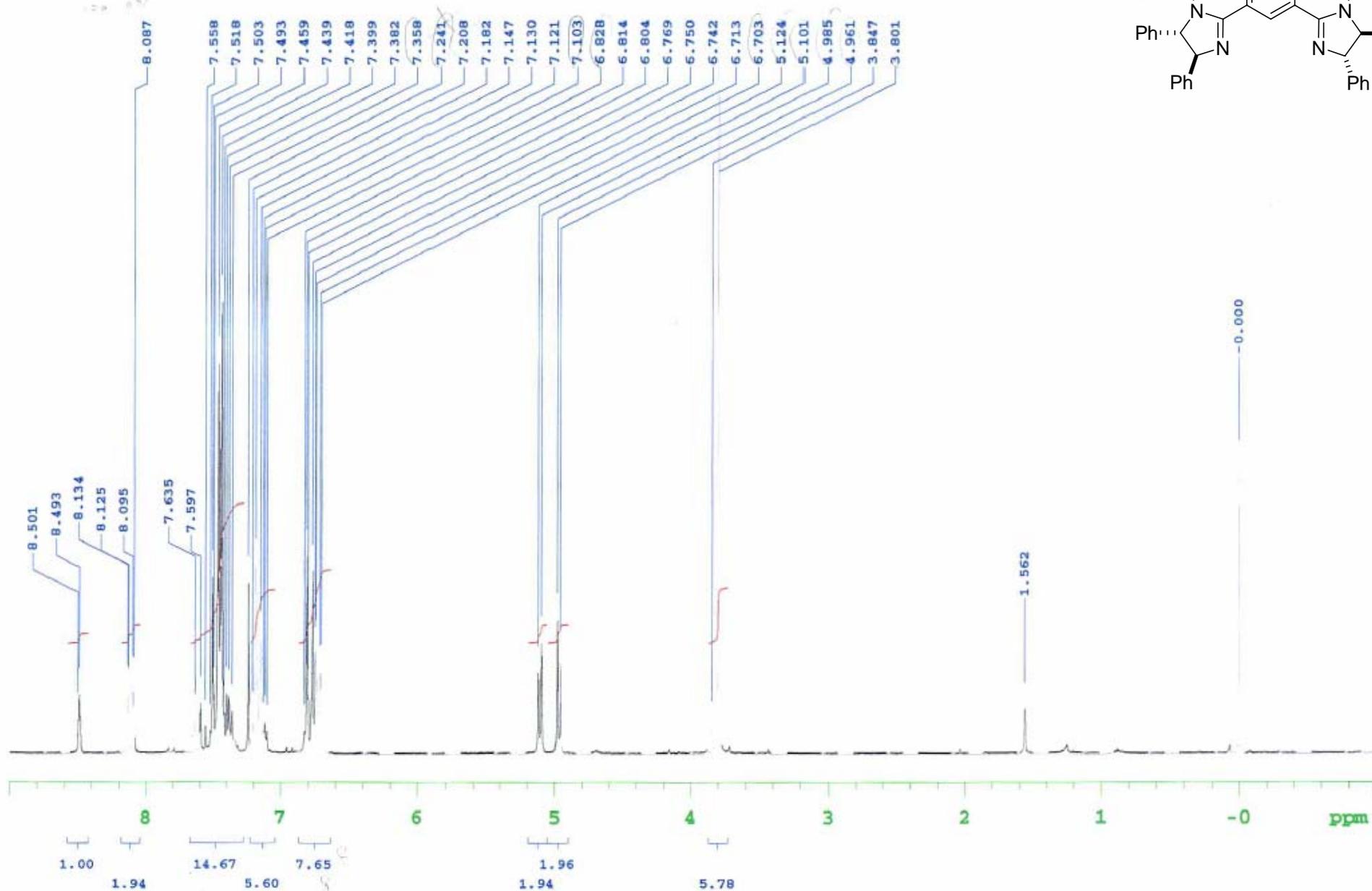
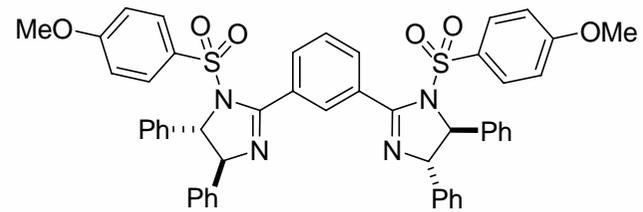
159.417
 143.824
 142.061
 141.152
 140.547
 131.888
 131.869
 131.856
 130.259
 130.014
 128.842
 128.813
 128.048
 127.879
 127.557
 126.748
 126.451

79.114
 77.213
 77.001
 76.789
 71.171

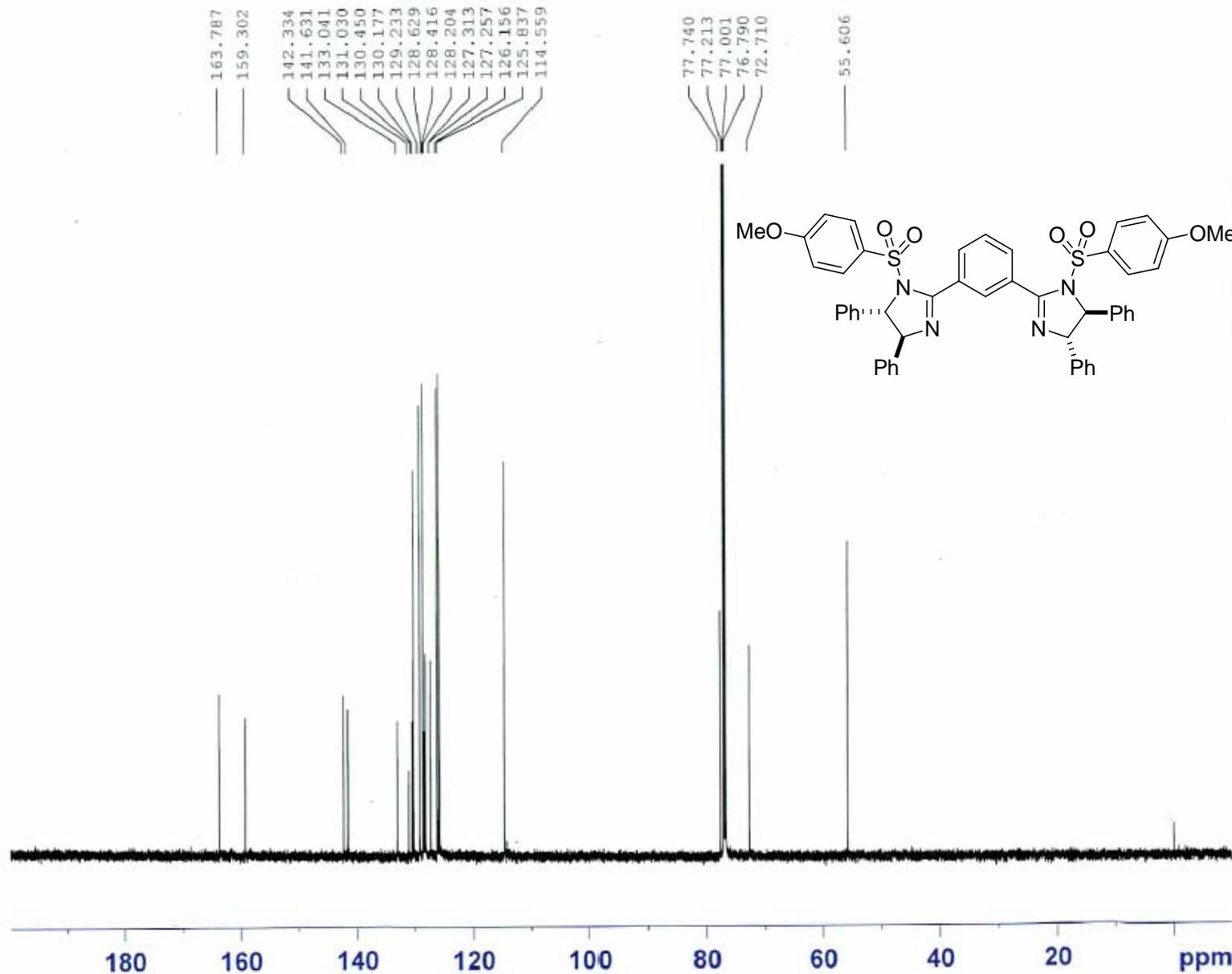
22.438
 20.954



180 160 140 120 100 80 60 40 20 ppm



p-OMe Ph SO₂



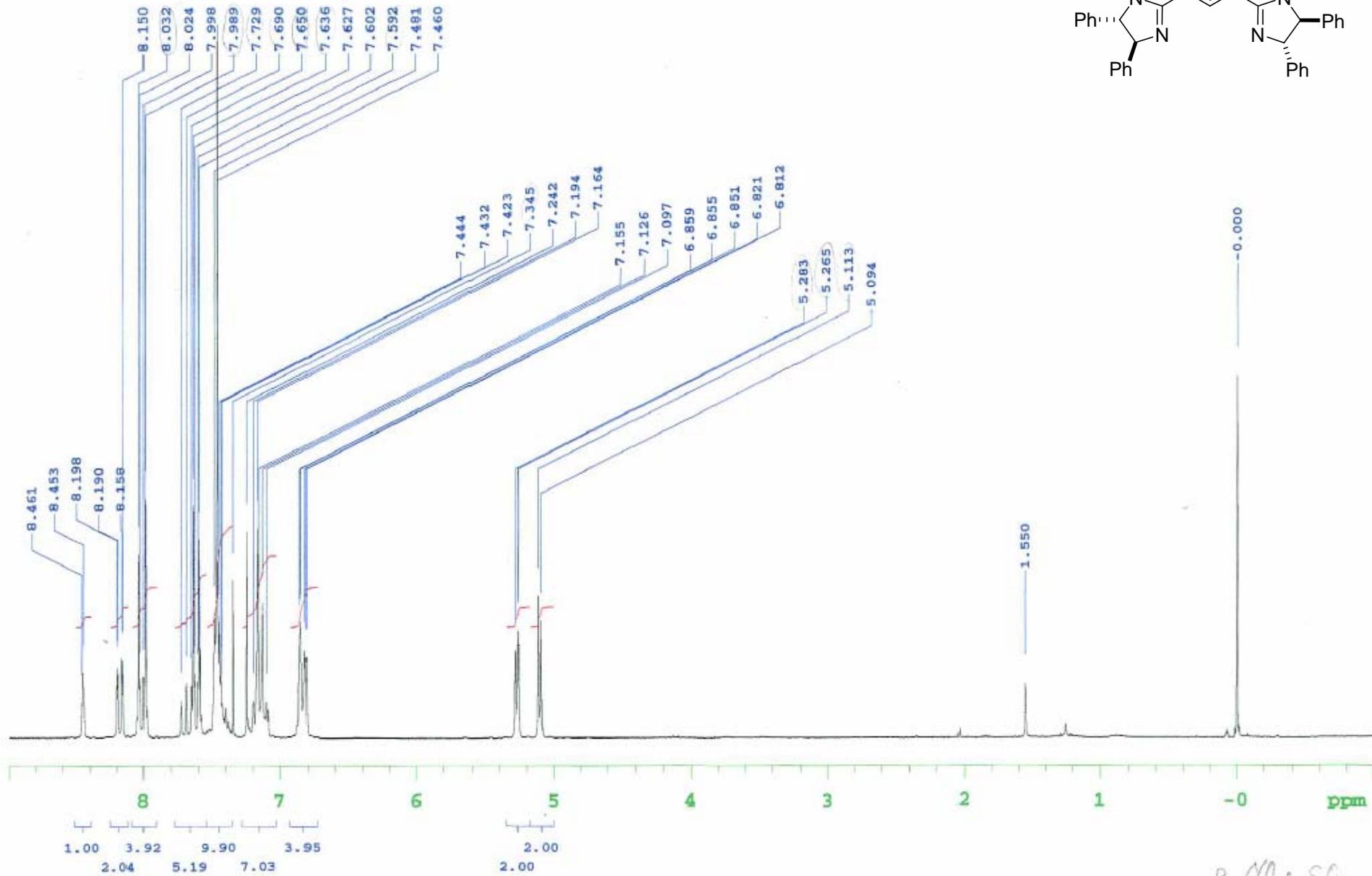
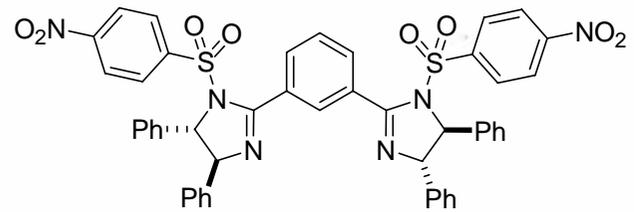
Current Data Parameters
 NAME NY-306
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071223
 Time 15.30
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDC13
 NS 512
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 3649.1
 DW 11.000 usec
 DE 6.00 usec
 TE 297.8 K
 D1 0.60000002 sec
 d11 0.03000000 sec
 DELTA 0.50000000 sec
 TD0 1

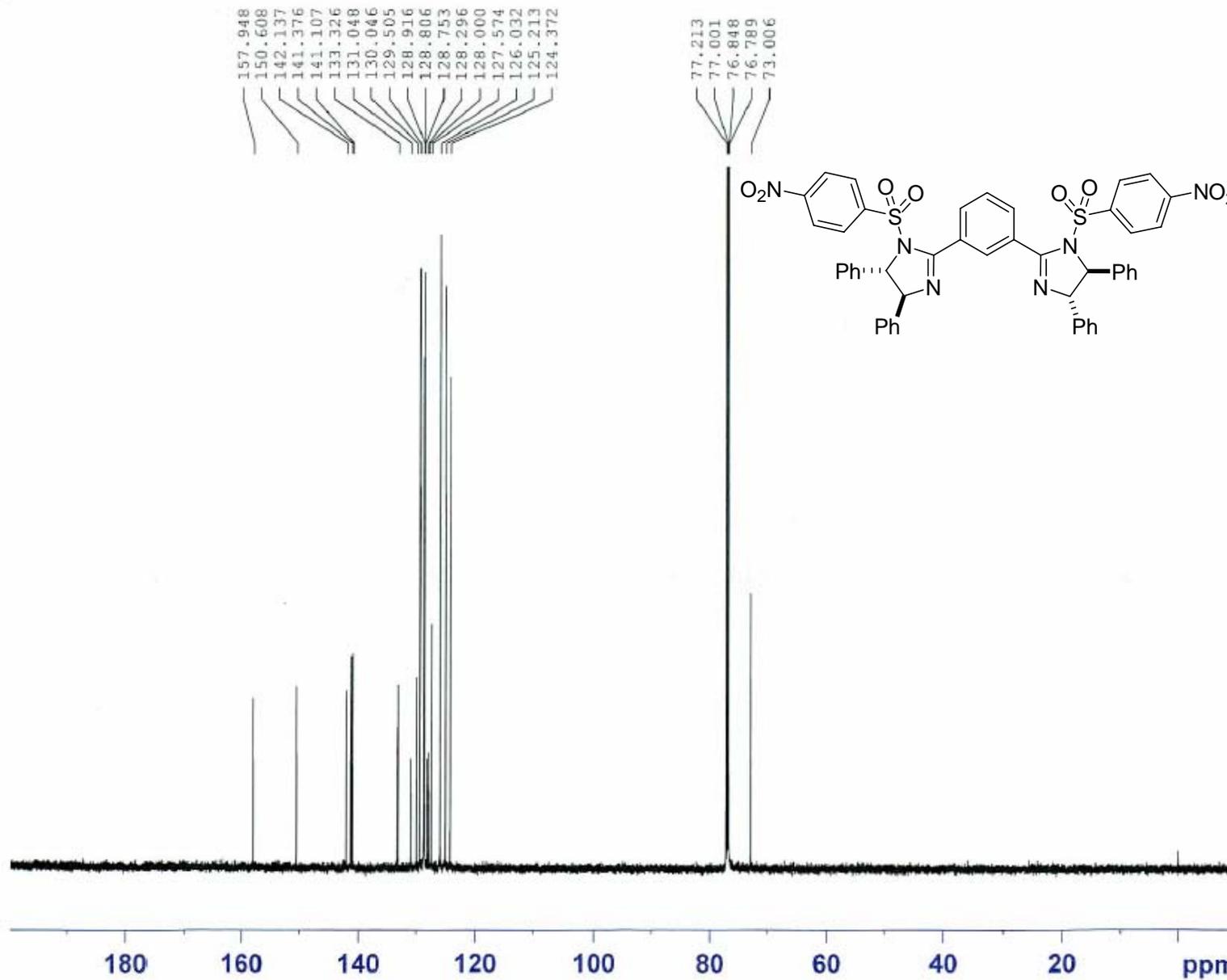
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028165 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



P NO₂ SO₂
Ph



157.948
150.608
142.137
141.376
141.107
133.326
131.048
130.046
129.505
128.916
128.806
128.753
128.296
128.000
127.574
126.032
125.213
124.372

77.213
77.001
76.848
76.789
73.006

Current Data Parameters
 NAME NY-225
 EXPNO 10
 PROCNO 1

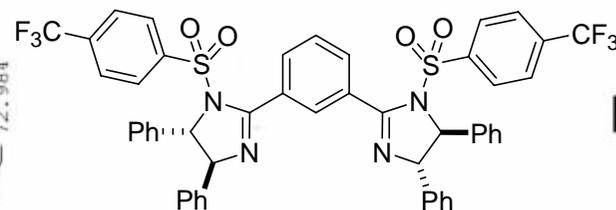
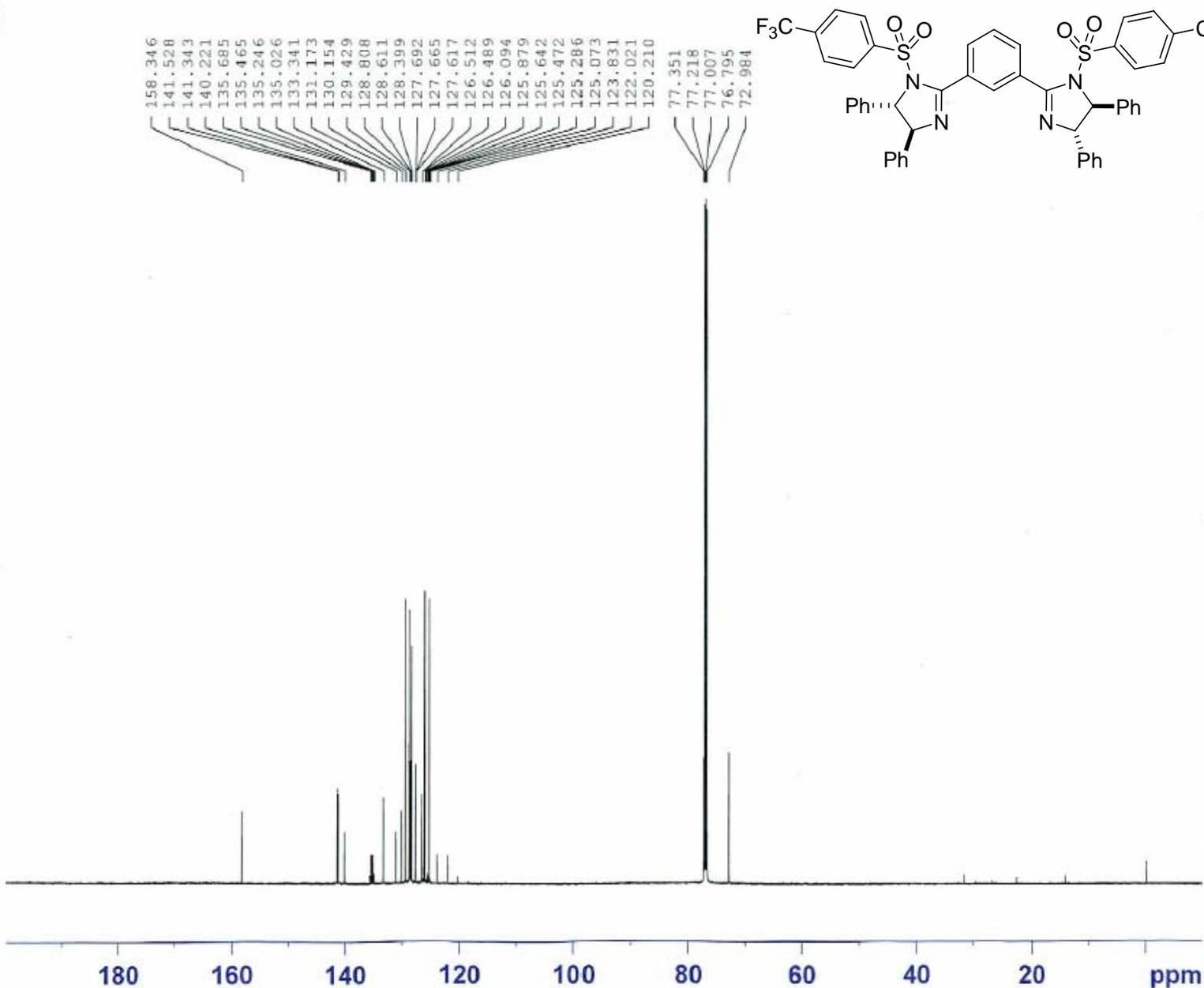
F2 - Acquisition Parameters
 Date_ 20071223
 Time 16.07
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 2580.3
 DW 11.000 usec
 DE 6.00 usec
 TE 297.8 K
 D1 0.60000002 sec
 d11 0.03000000 sec
 DELTA 0.50000000 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028169 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

13C



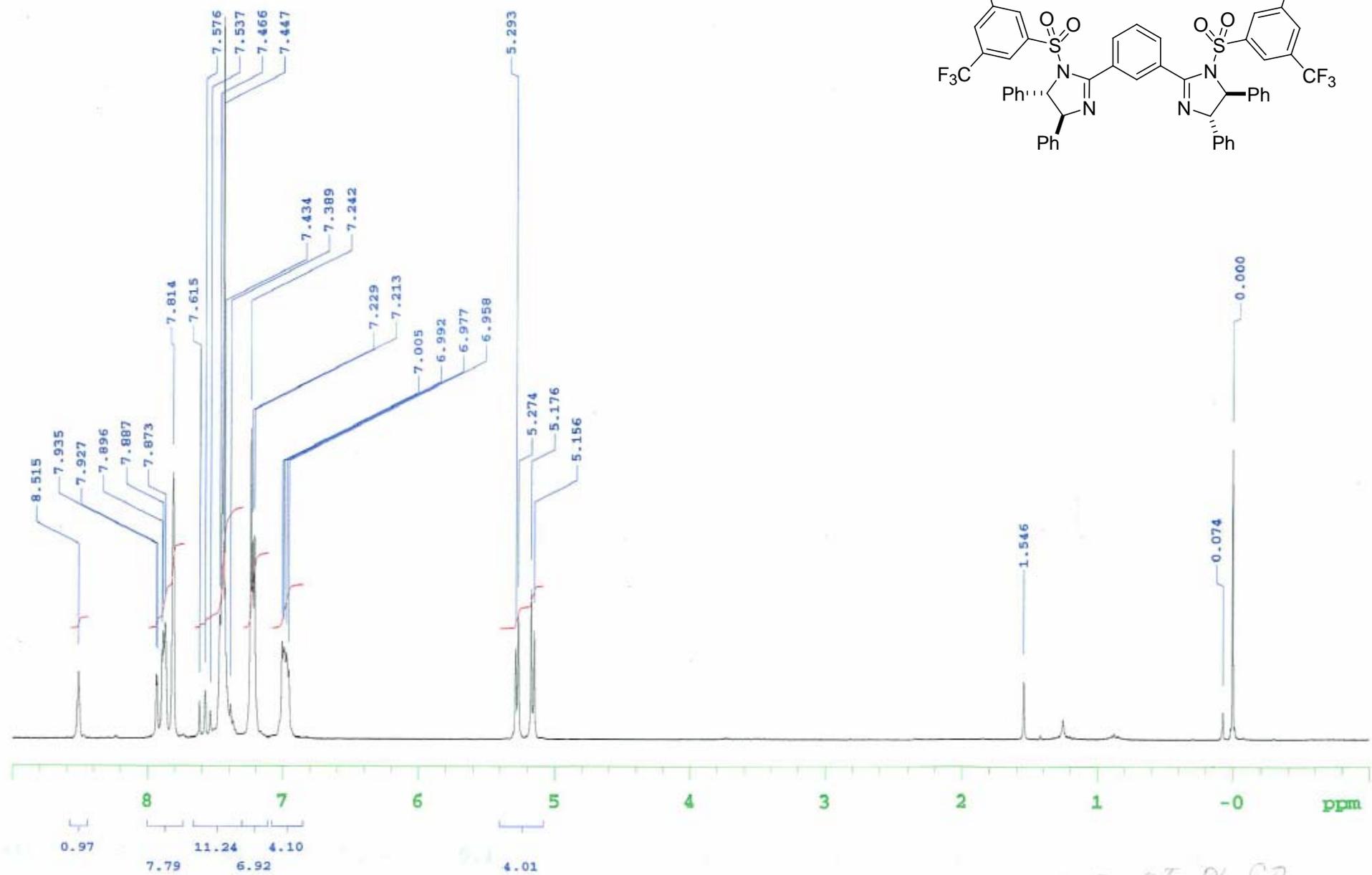
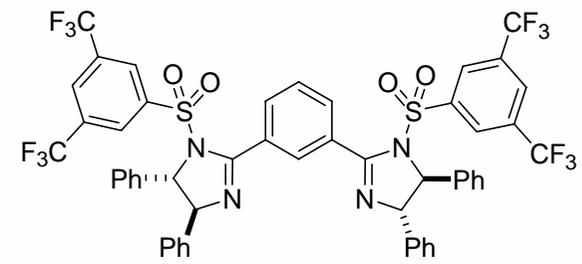
Current Data Parameters
NAME HK-406
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date 20071222
Time 13.05
INSTRUM drx600
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 1787
DS 4
SWH 45454.547 Hz
FIDRES 0.346791 Hz
AQ 1.4418530 sec
RG 6502
DW 11.000 usec
DE 6.00 usec
TE 297.6 K
D1 5.00000000 sec
d11 0.03000000 sec
DELTA 4.90000010 sec
TDO 1

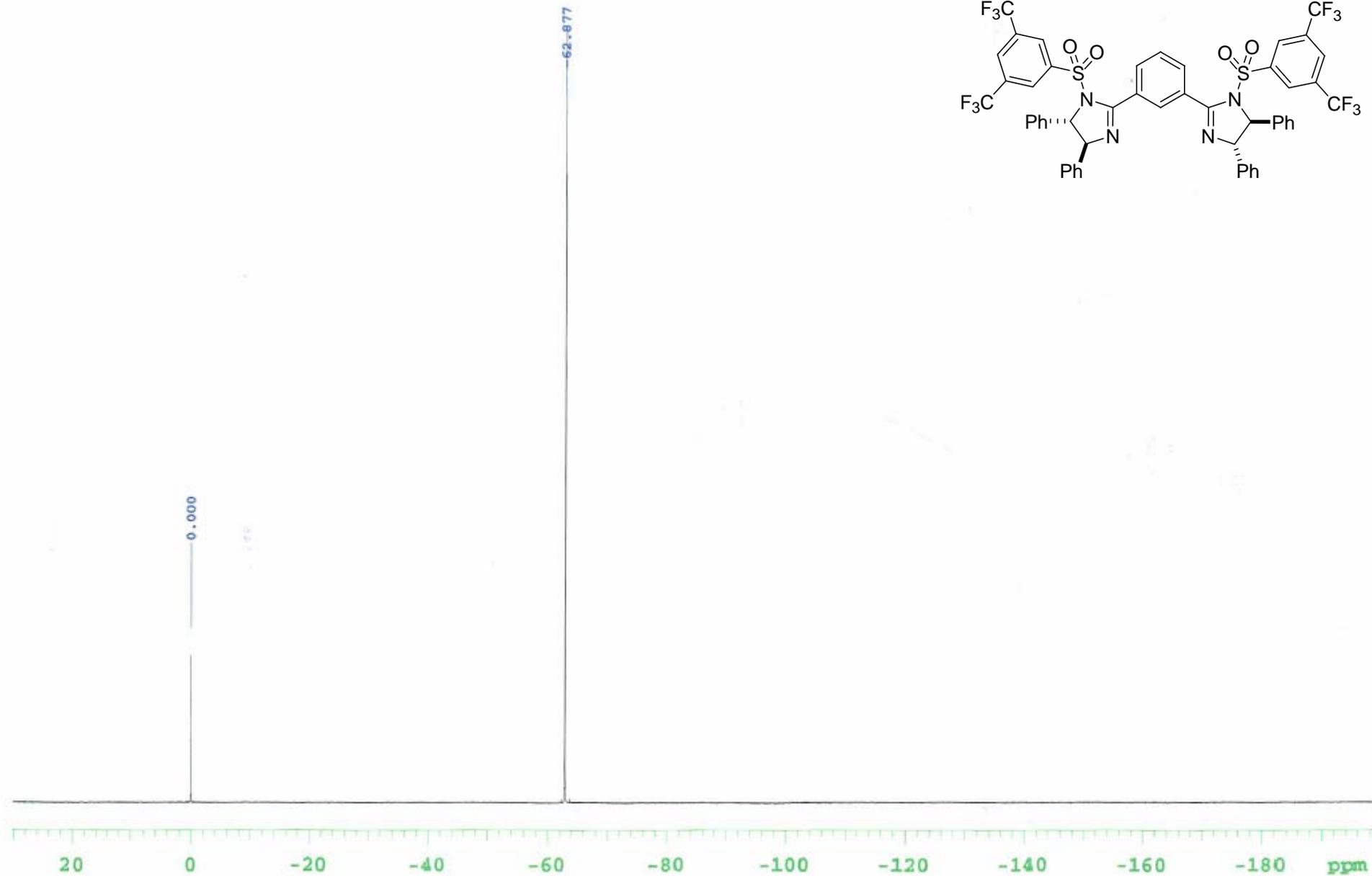
----- CHANNEL f1 -----
NUC1 13C
P1 8.20 usec
PL1 4.50 dB
SFO1 150.9223664 MHz

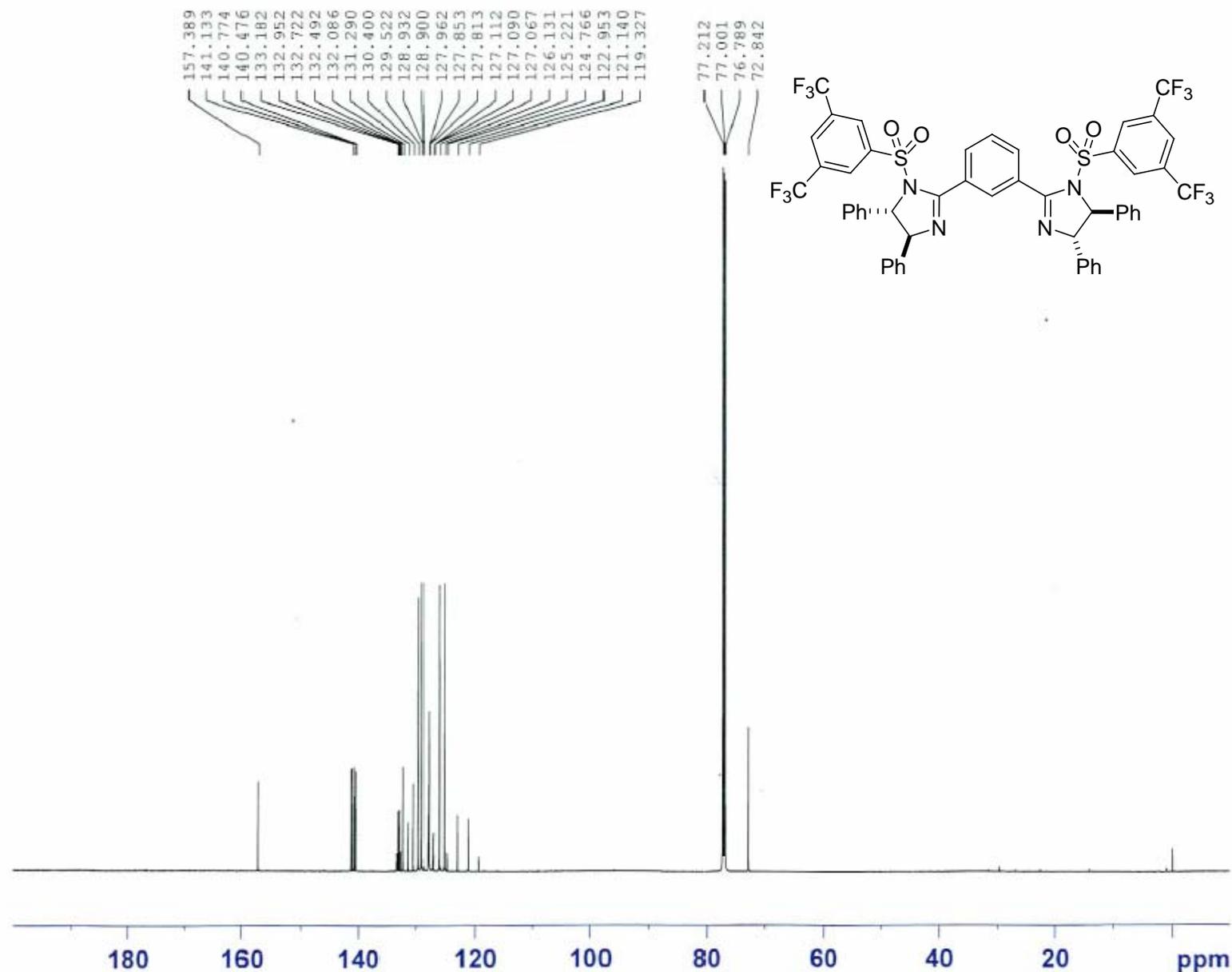
----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 82.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 131072
SF 150.9028148 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



3,5-(CF₃)₂Ph SO₂





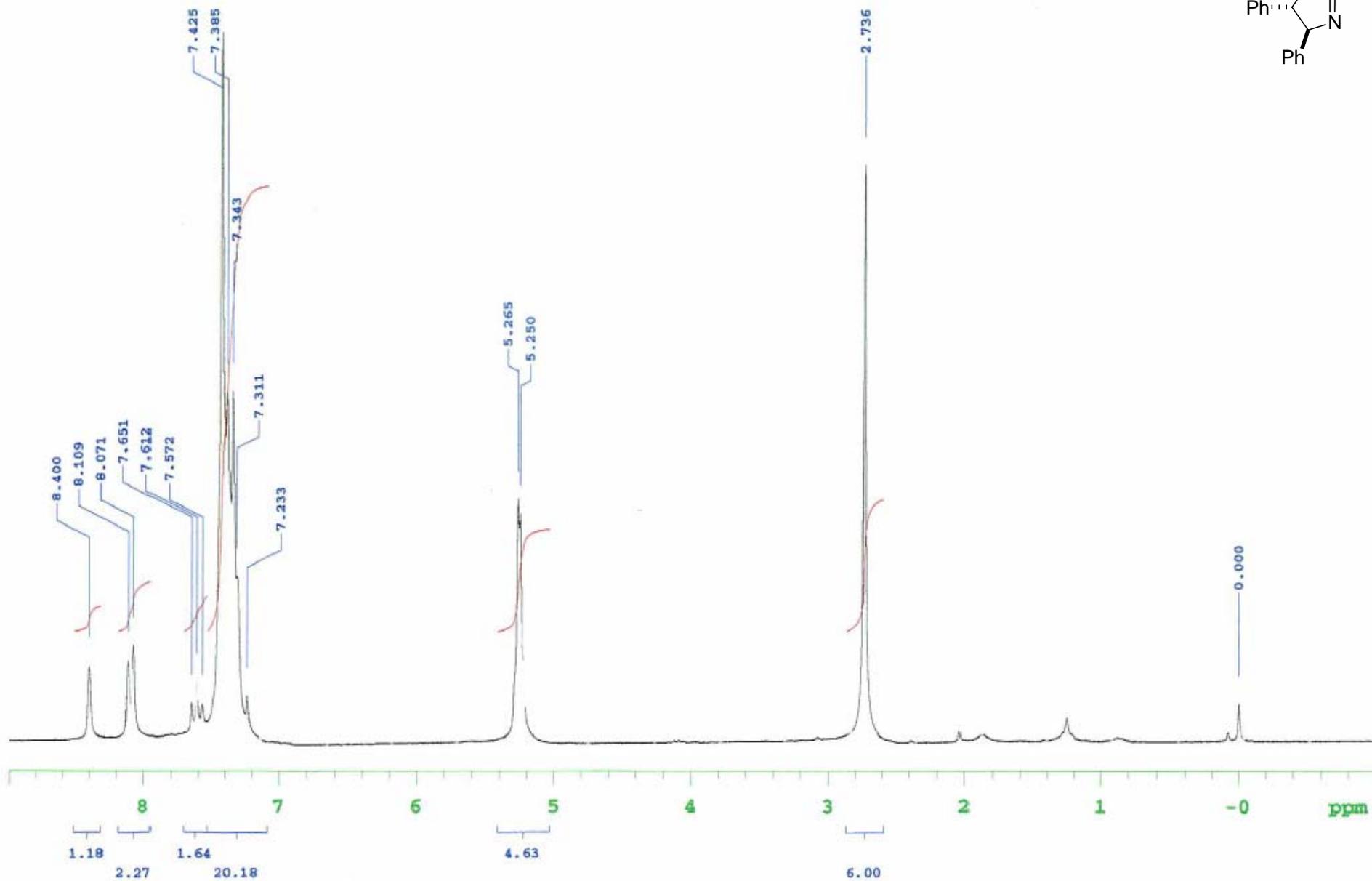
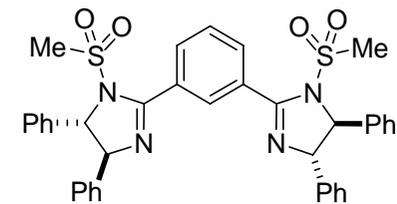
Current Data Parameters
 NAME NY-308
 EXPNO 10
 PROCNO 1

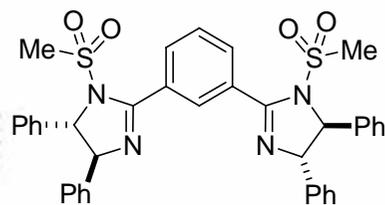
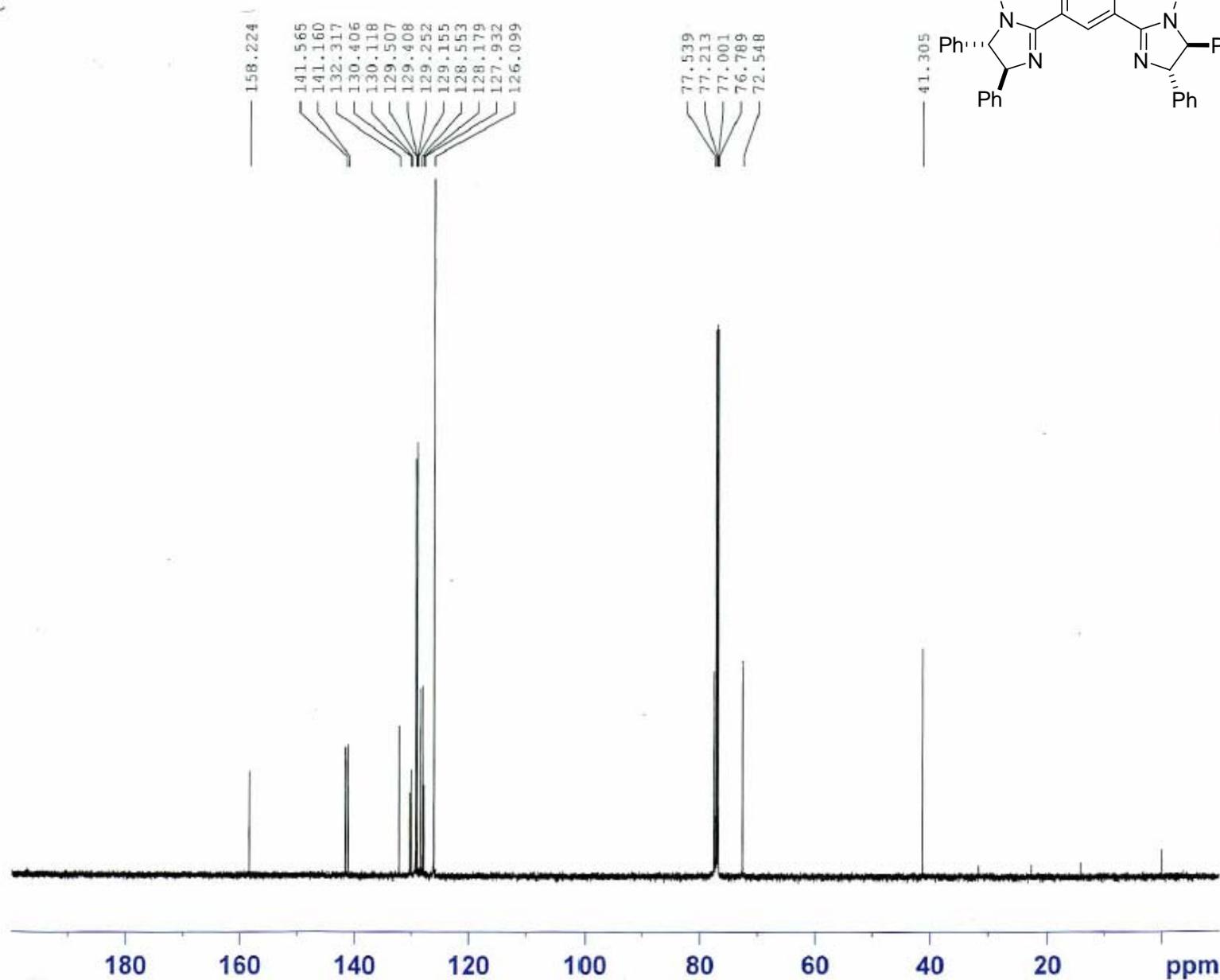
F2 - Acquisition Parameters
 Date_ 20071222
 Time 22.33
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDC13
 NS 7756
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 5792.6
 DW 11.000 usec
 DE 6.00 usec
 TE 297.7 K
 D1 5.00000000 sec
 d11 0.03000000 sec
 DELTA 4.90000010 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028134 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





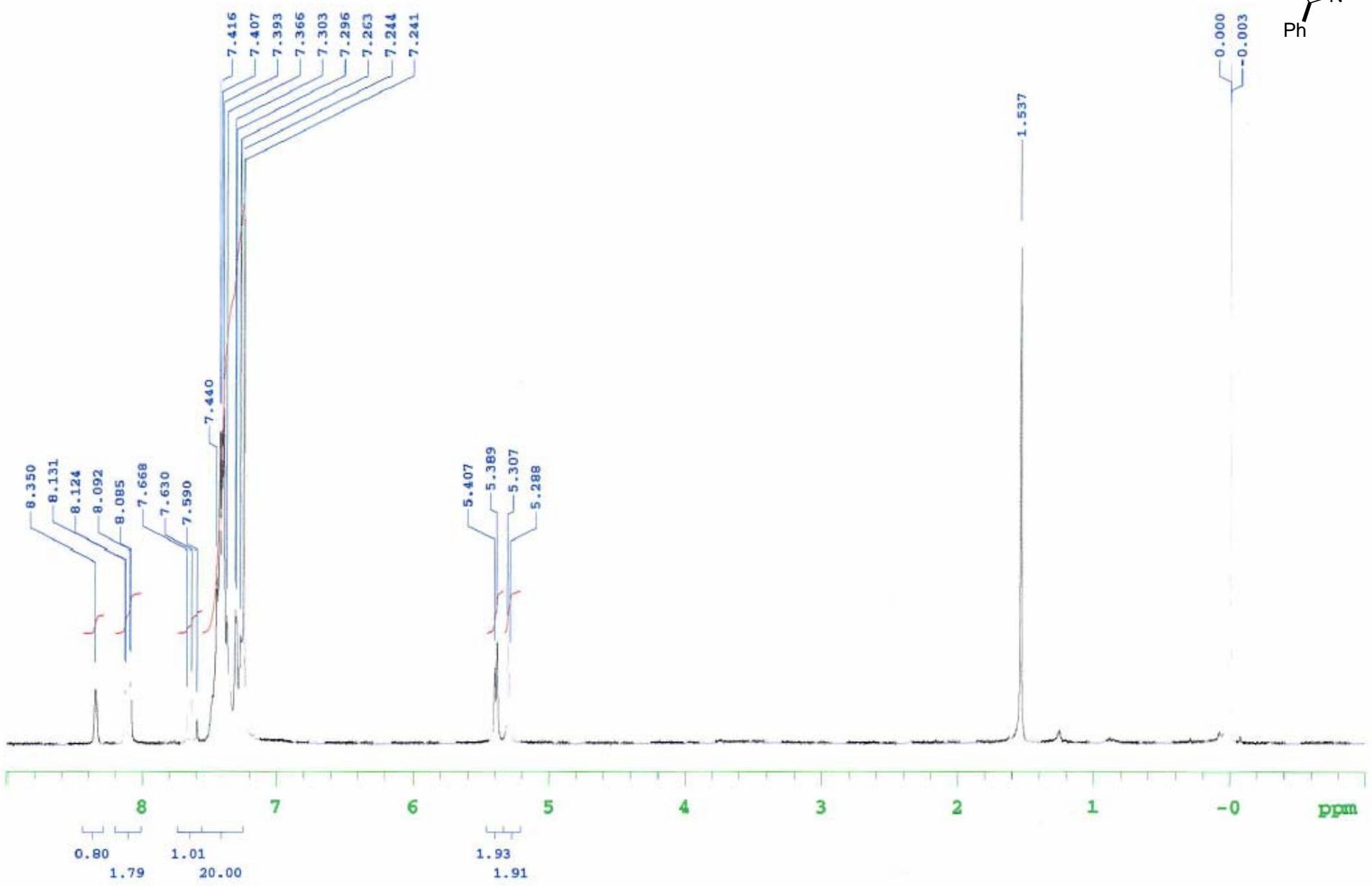
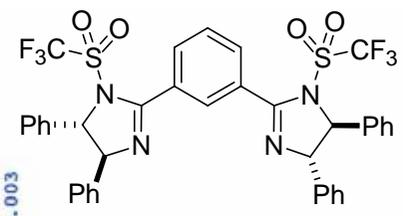
Current Data Parameters
NAME HK-347
EXPNO 10
PROCNO 1

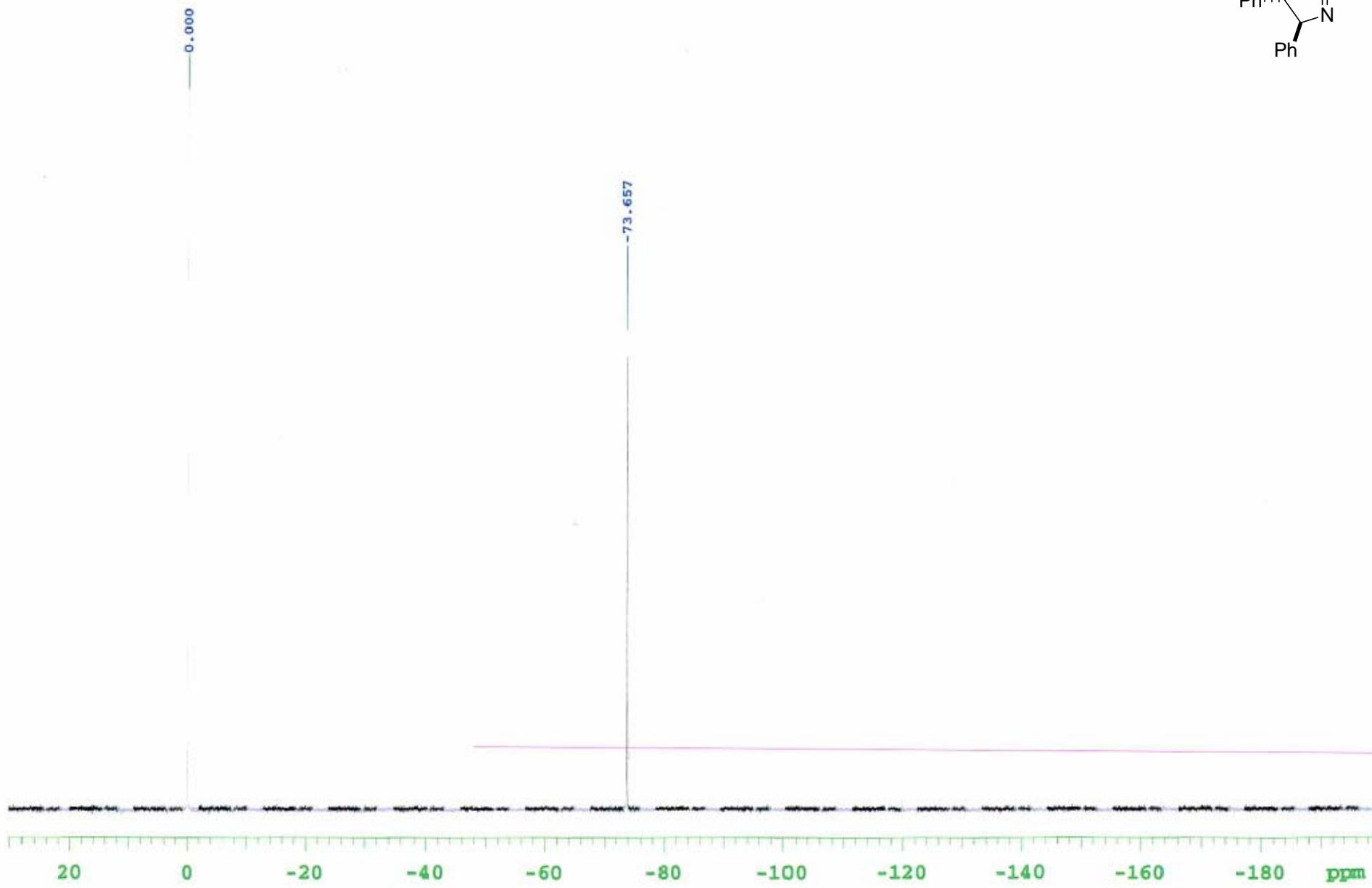
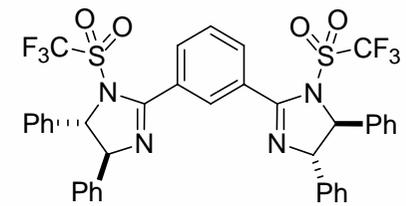
F2 - Acquisition Parameters
Date_ 20071222
Time_ 21.55
INSTRUM drx600
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 512
DS 4
SWH 45454.547 Hz
FIDRES 0.346791 Hz
AQ 1.4418530 sec
RG 3251
DW 11.000 usec
DE 6.00 usec
TE 297.6 K
D1 0.60000002 sec
d11 0.03000000 sec
DELTA 0.50000000 sec
TD0 1

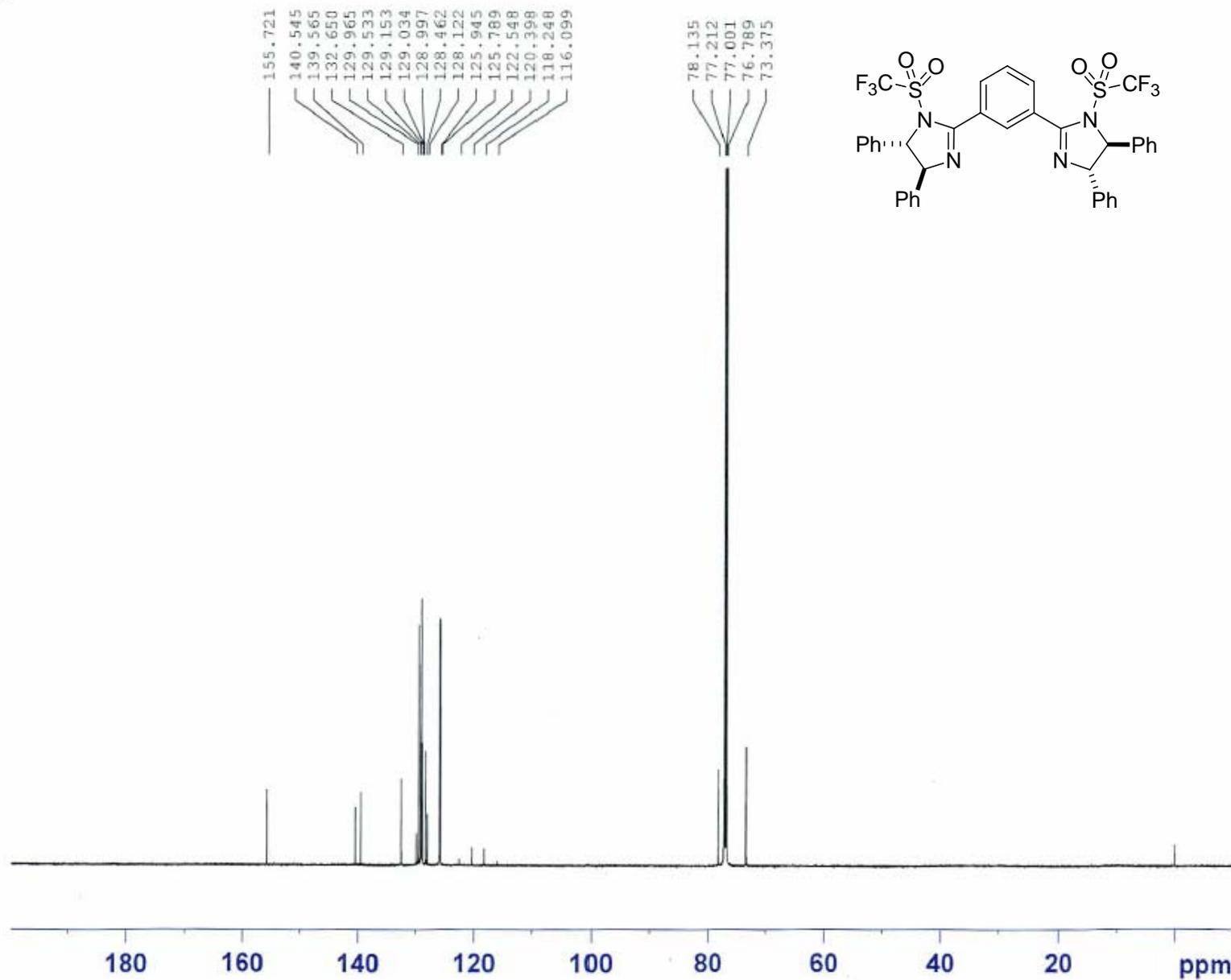
===== CHANNEL f1 =====
NUC1 13C
P1 8.20 usec
PL1 4.50 dB
SFO1 150.9223664 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 82.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 131072
SF 150.9028167 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







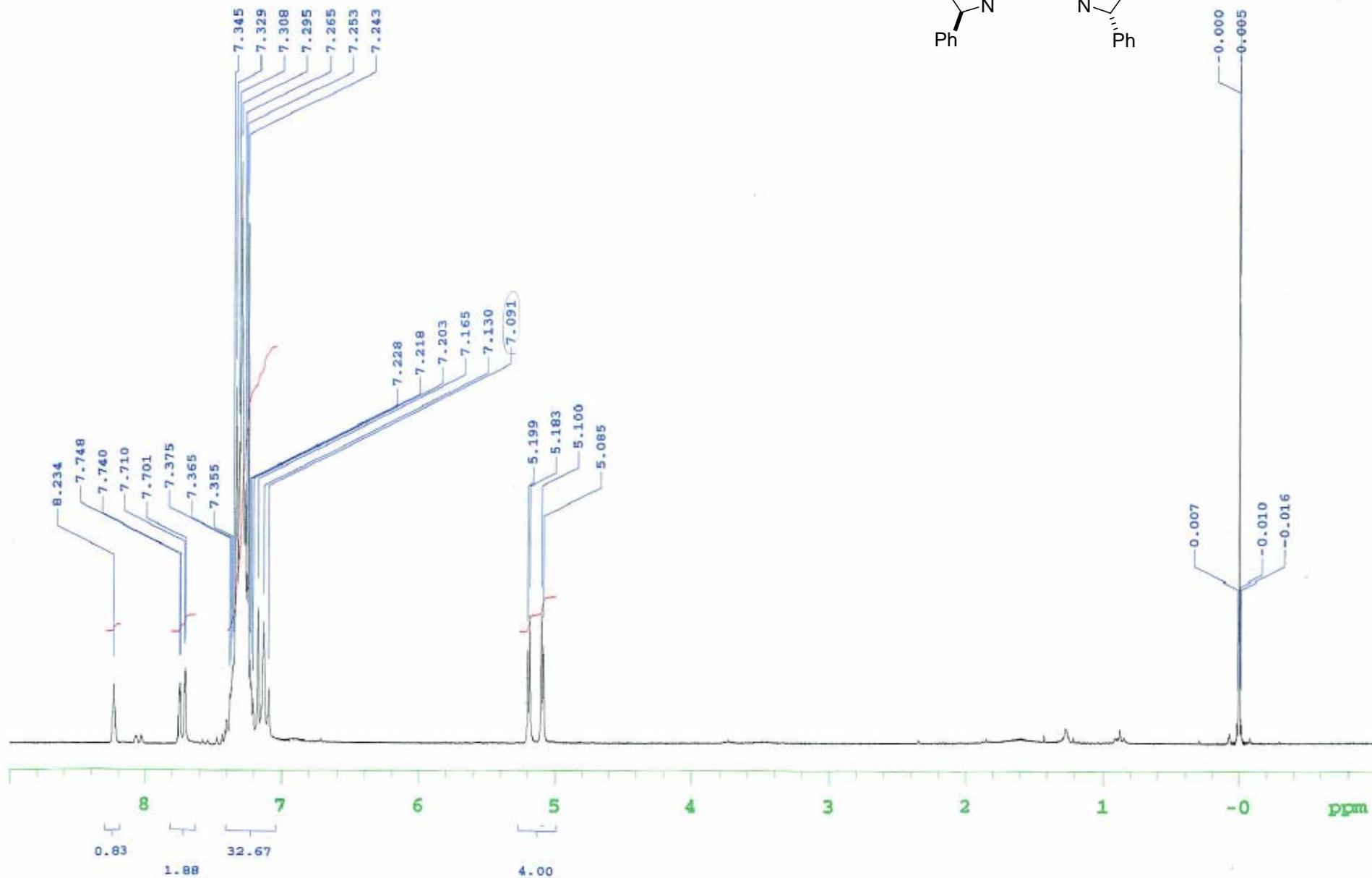
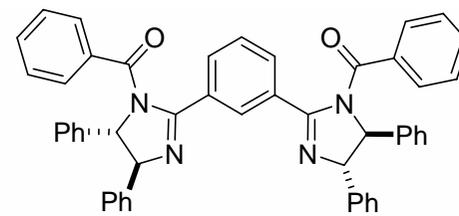
Current Data Parameters
 NAME HK-406
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071222
 Time_ 17.16
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 2351
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 6502
 DW 11.000 usec
 DE 6.00 usec
 TE 297.7 K
 D1 5.00000000 sec
 d11 0.03000000 sec
 DELTA 4.90000010 sec
 TDO 1

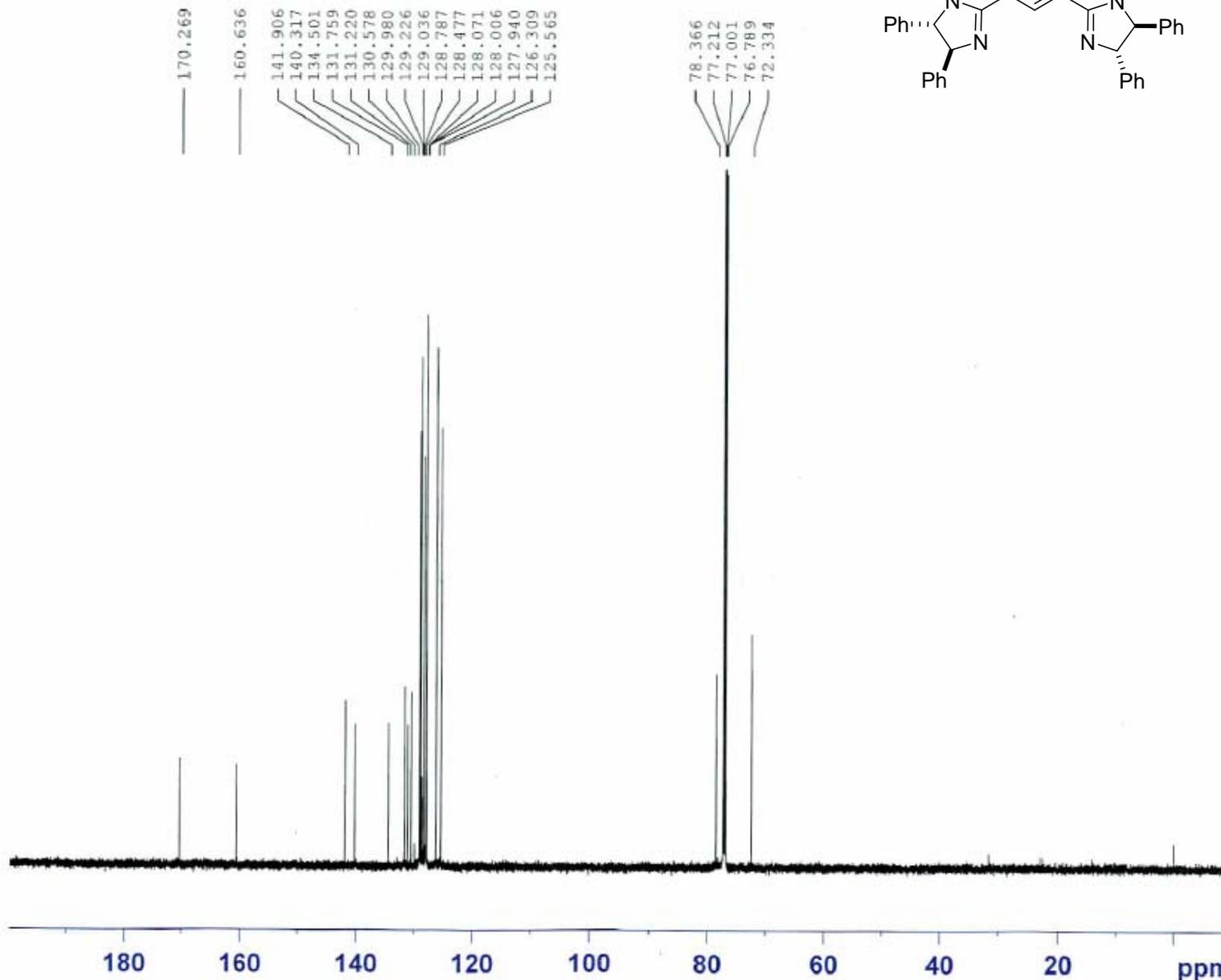
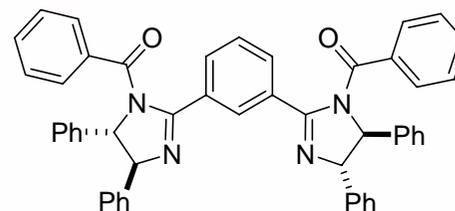
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028134 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



CO Ph



170.269
160.636
141.906
140.317
134.501
131.759
131.220
130.578
129.980
129.226
129.036
128.787
128.477
128.071
128.006
127.940
126.309
125.565
78.366
77.212
77.001
76.789
72.334

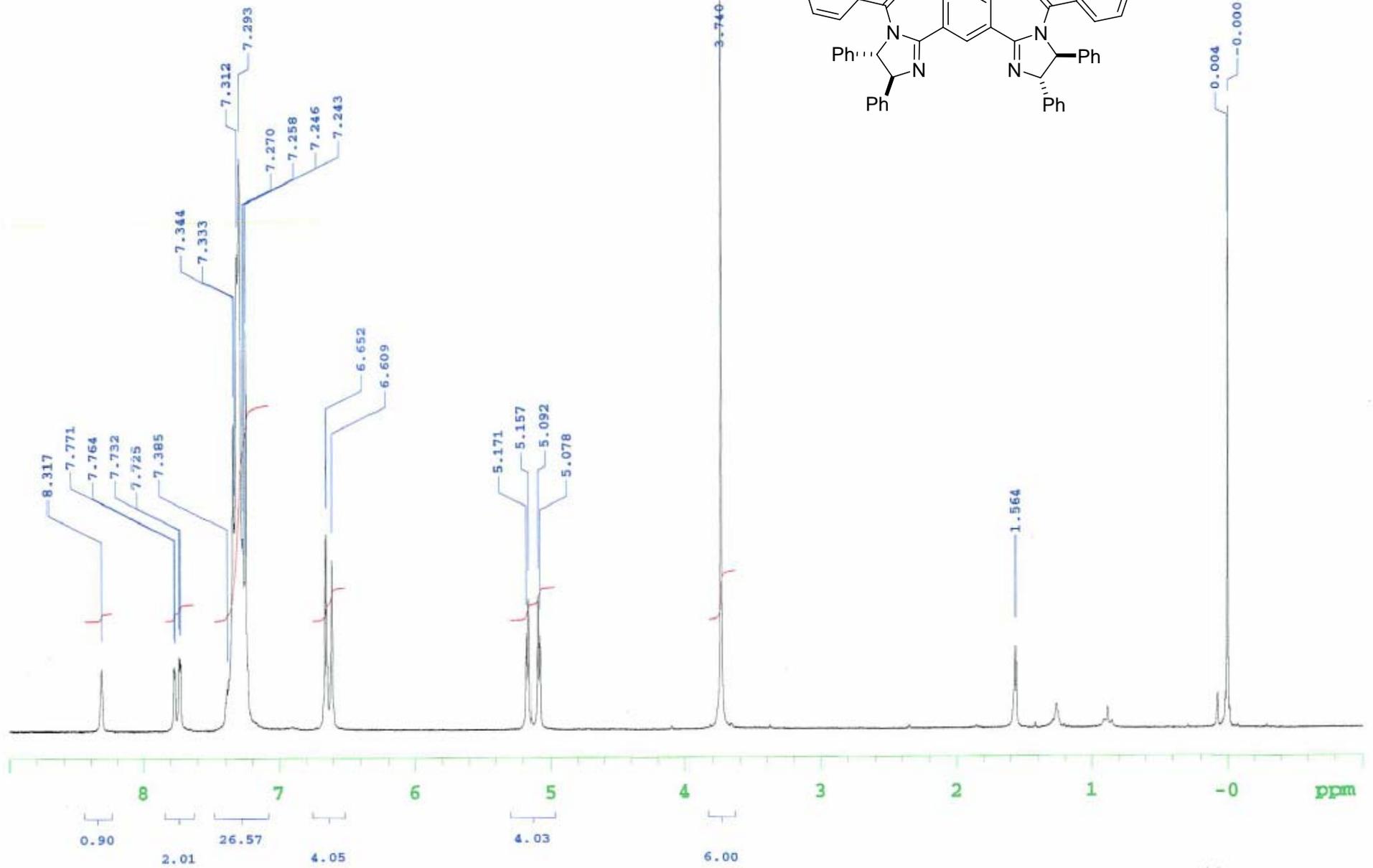
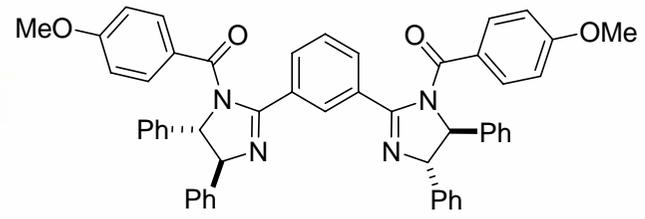
Current Data Parameters
NAME NY-307
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071223
Time 14.39
INSTRUM drx600
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDC13
NS 512
DS 4
SWH 45454.547 Hz
FIDRES 0.346791 Hz
AQ 1.4418530 sec
RG 4597.6
DW 11.000 usec
DE 6.00 usec
TE 297.7 K
D1 0.60000002 sec
d11 0.03000000 sec
DELTA 0.50000000 sec
TD0 1

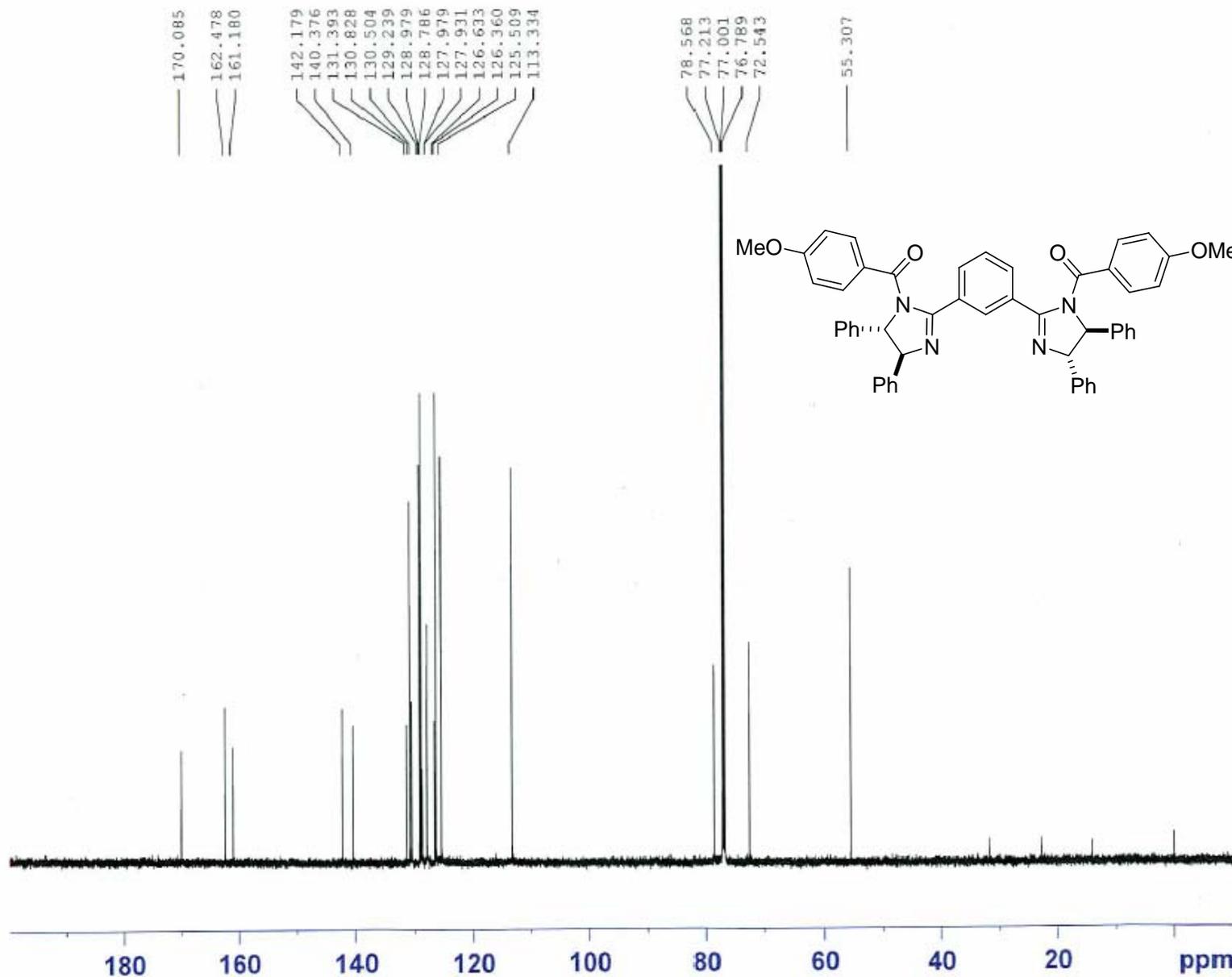
==== CHANNEL f1 =====
NUC1 13C
P1 8.20 usec
PL1 4.50 dB
SFO1 150.9223664 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 82.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 131072
SF 150.9028154 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



p-CMe
Pr. 8-11



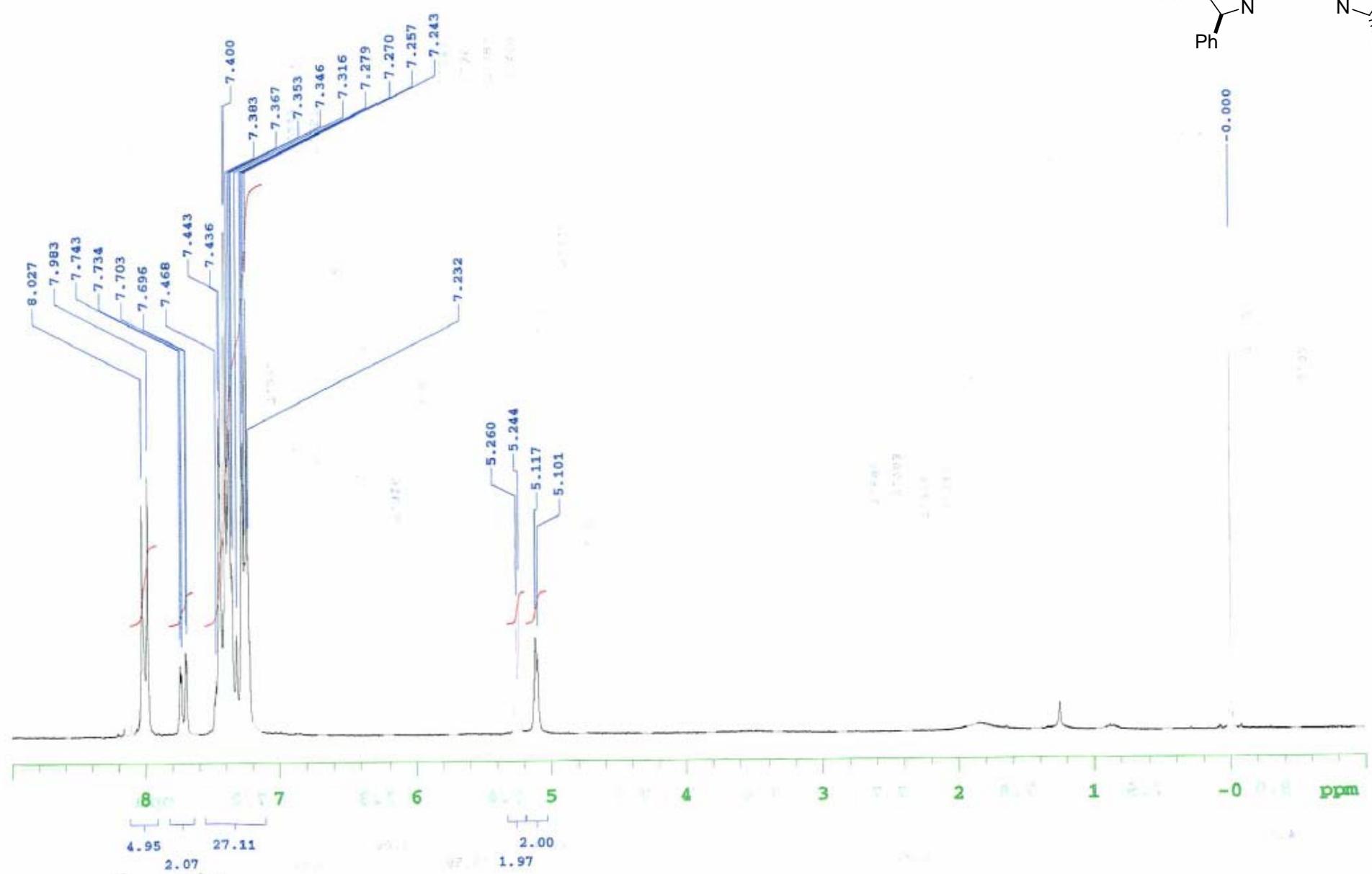
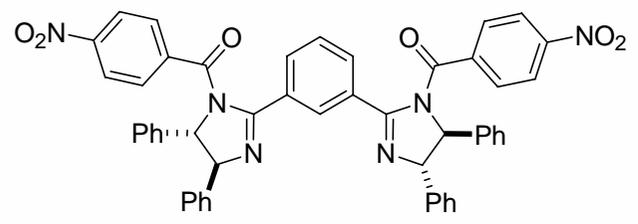
Current Data Parameters
 NAME NY-236
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date 20071223
 Time 16.48
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 6502
 DW 11.000 usec
 DE 6.00 usec
 TE 297.7 K
 D1 0.60000002 sec
 d11 0.03000000 sec
 DELTA 0.50000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028164 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

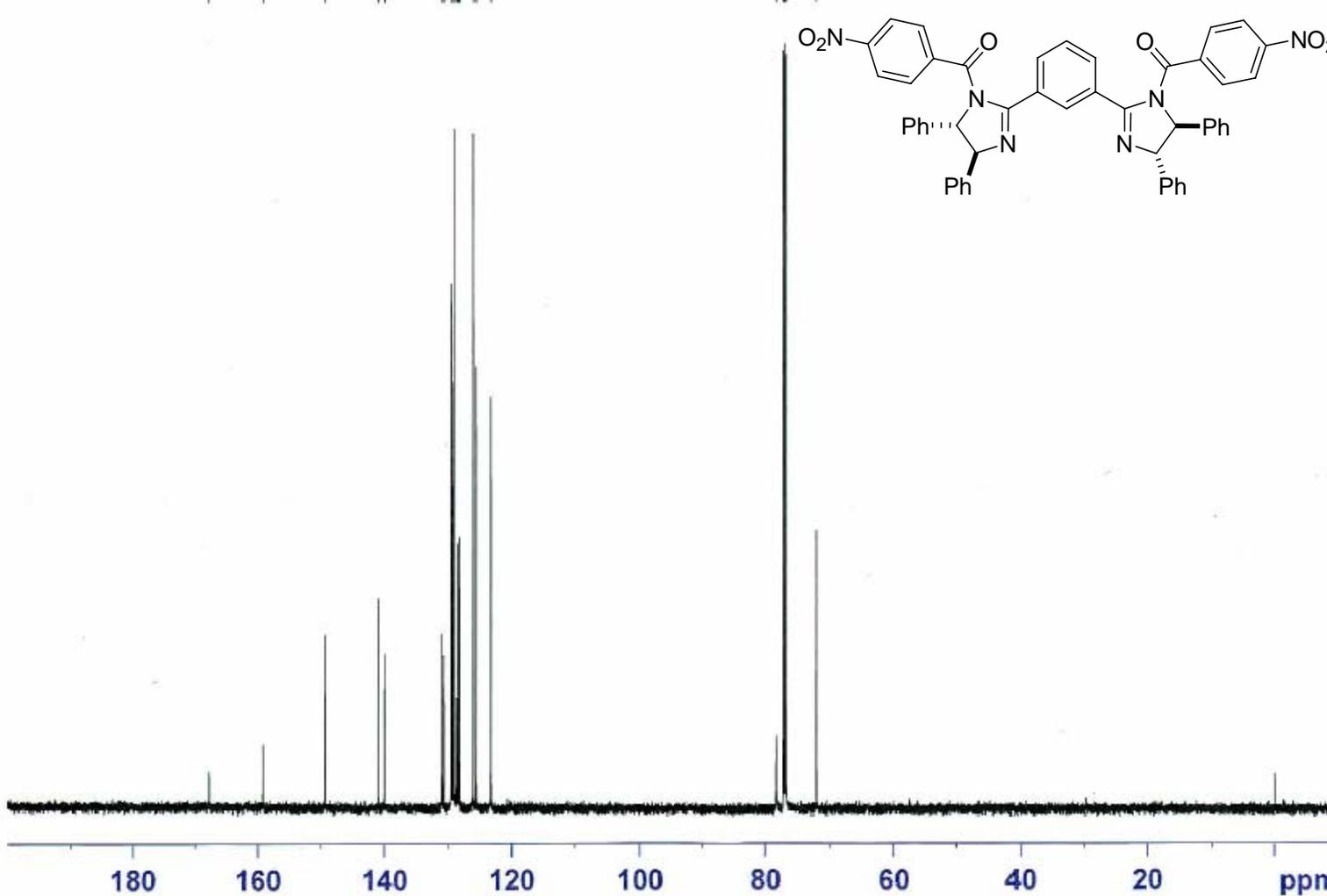
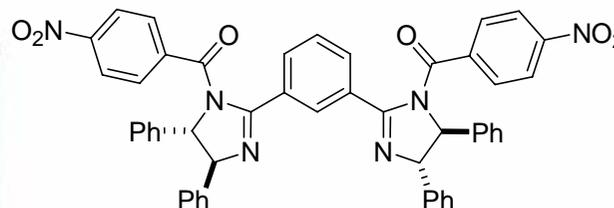


4.95
2.07
27.11
1.97
2.00



167.680
159.224
149.342
140.968
139.972
139.933
131.053
130.738
129.531
129.256
129.208
128.798
128.569
128.353
128.287
126.087
125.597
123.300

78.235
77.213
77.001
76.789
72.223



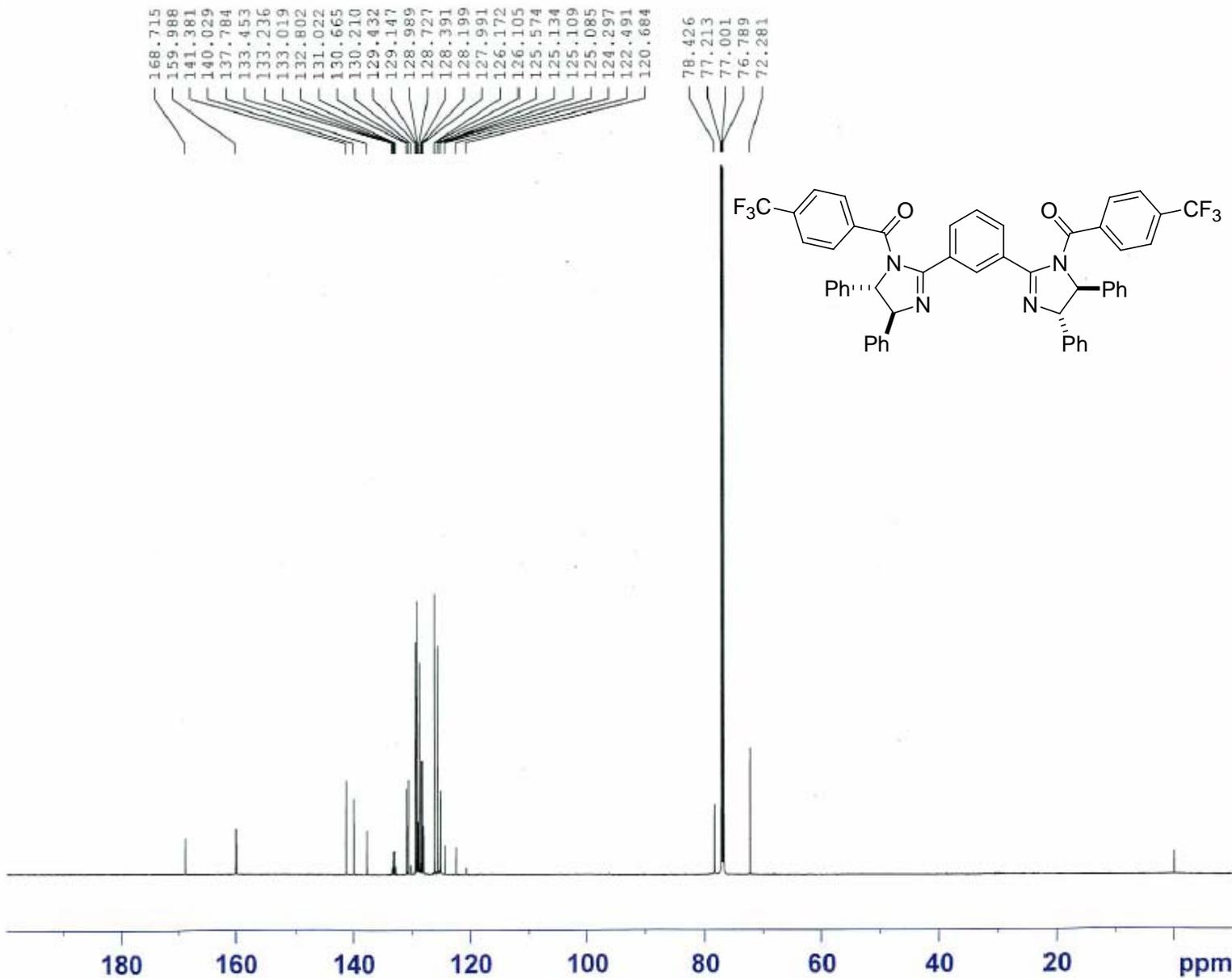
Current Data Parameters
NAME HK-471
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071223
Time_ 13.41
INSTRUM drx600
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 512
DS 4
SWH 45454.547 Hz
FIDRES 0.346791 Hz
AQ 1.4418530 sec
RG 4597.6
DW 11.000 usec
DE 6.00 usec
TE 297.7 K
D1 0.60000002 sec
d11 0.03000000 sec
DELTA 0.50000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.20 usec
PL1 4.50 dB
SFO1 150.9223664 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 82.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 131072
SF 150.9028183 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



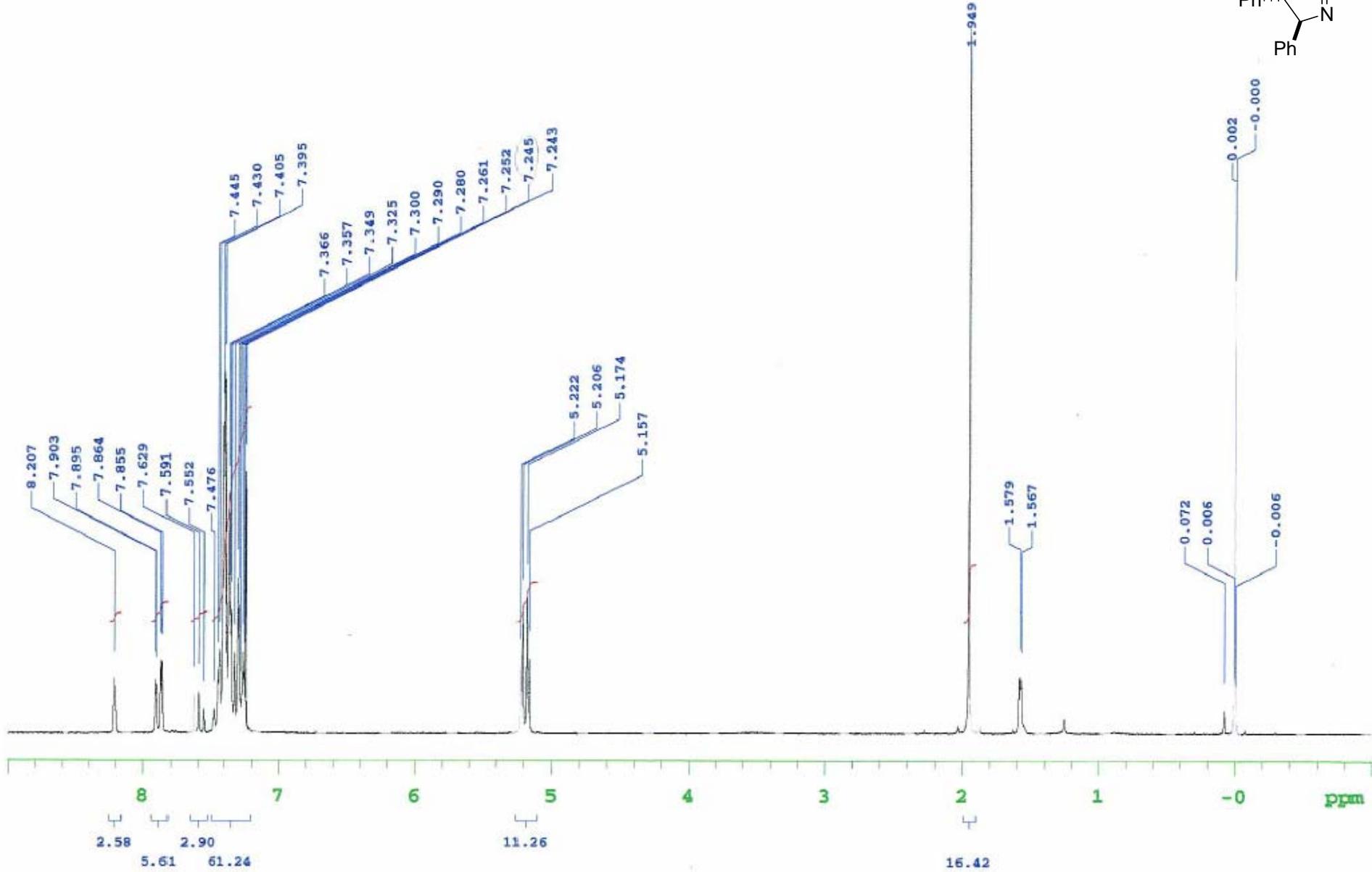
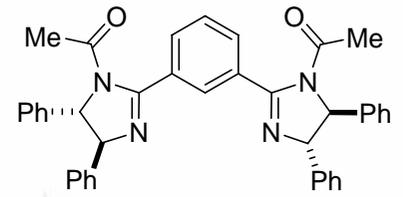
Current Data Parameters
 NAME NY-240
 EXPNO 10
 PROCNO 1

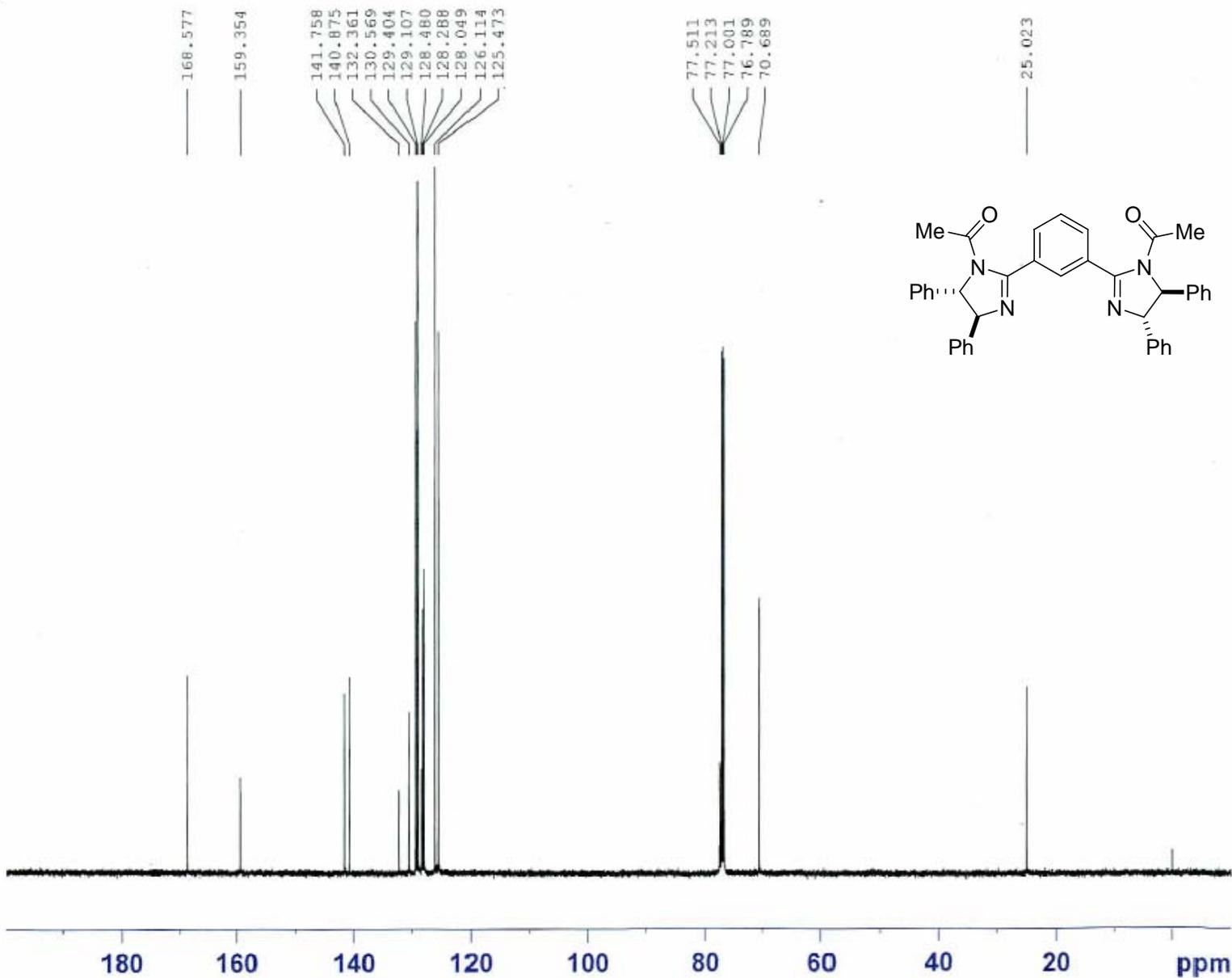
F2 - Acquisition Parameters
 Date_ 20071223
 Time_ 21.45
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 7808
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 4597.6
 DW 11.000 usec
 DE 6.00 usec
 TE 297.6 K
 D1 5.00000000 sec
 d11 0.03000000 sec
 DELTA 4.90000010 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028147 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





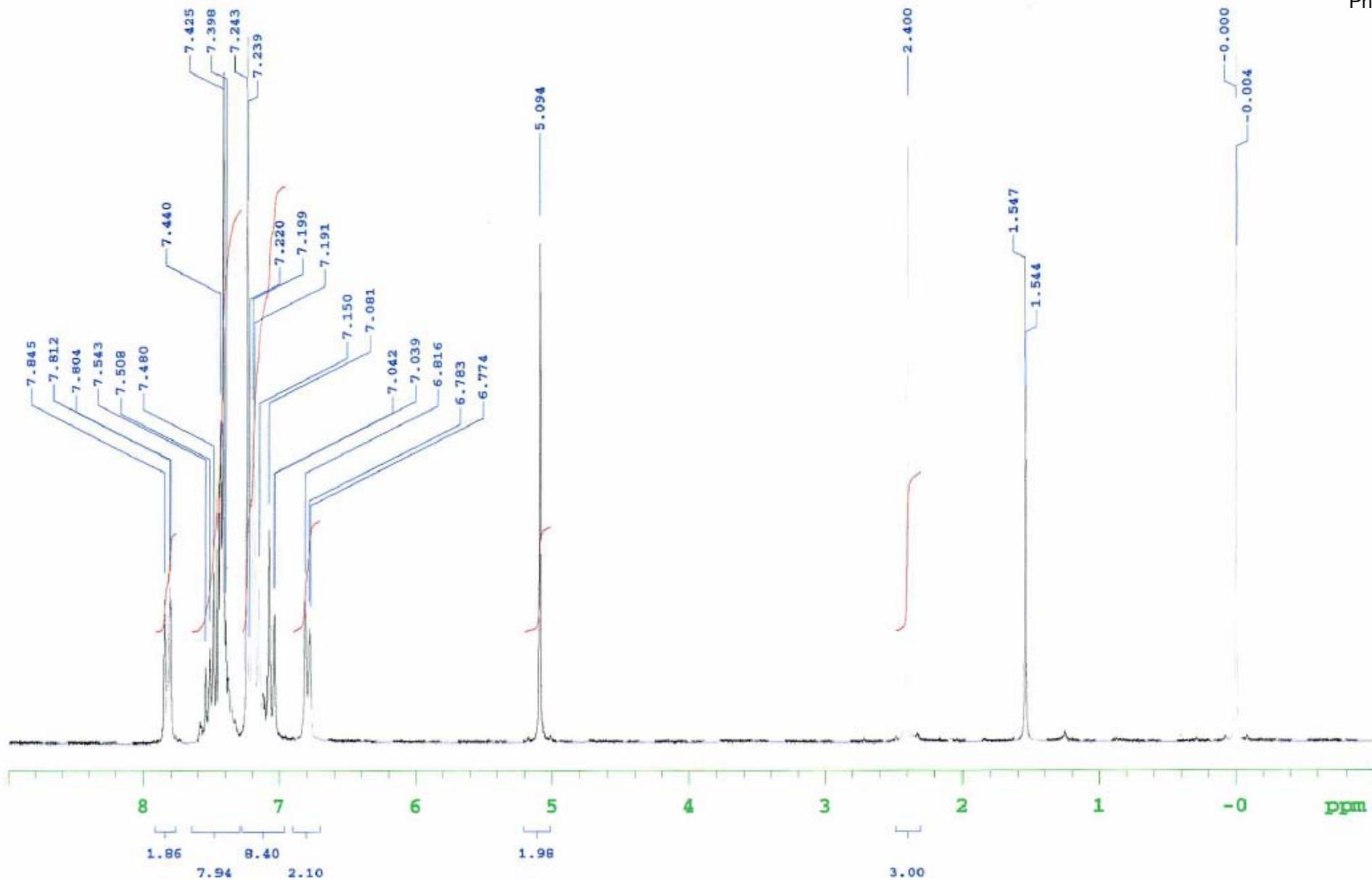
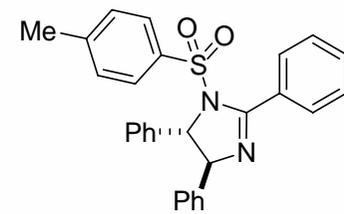
Current Data Parameters
 NAME NY-249-2
 EXPNO 10
 PROCNO 1

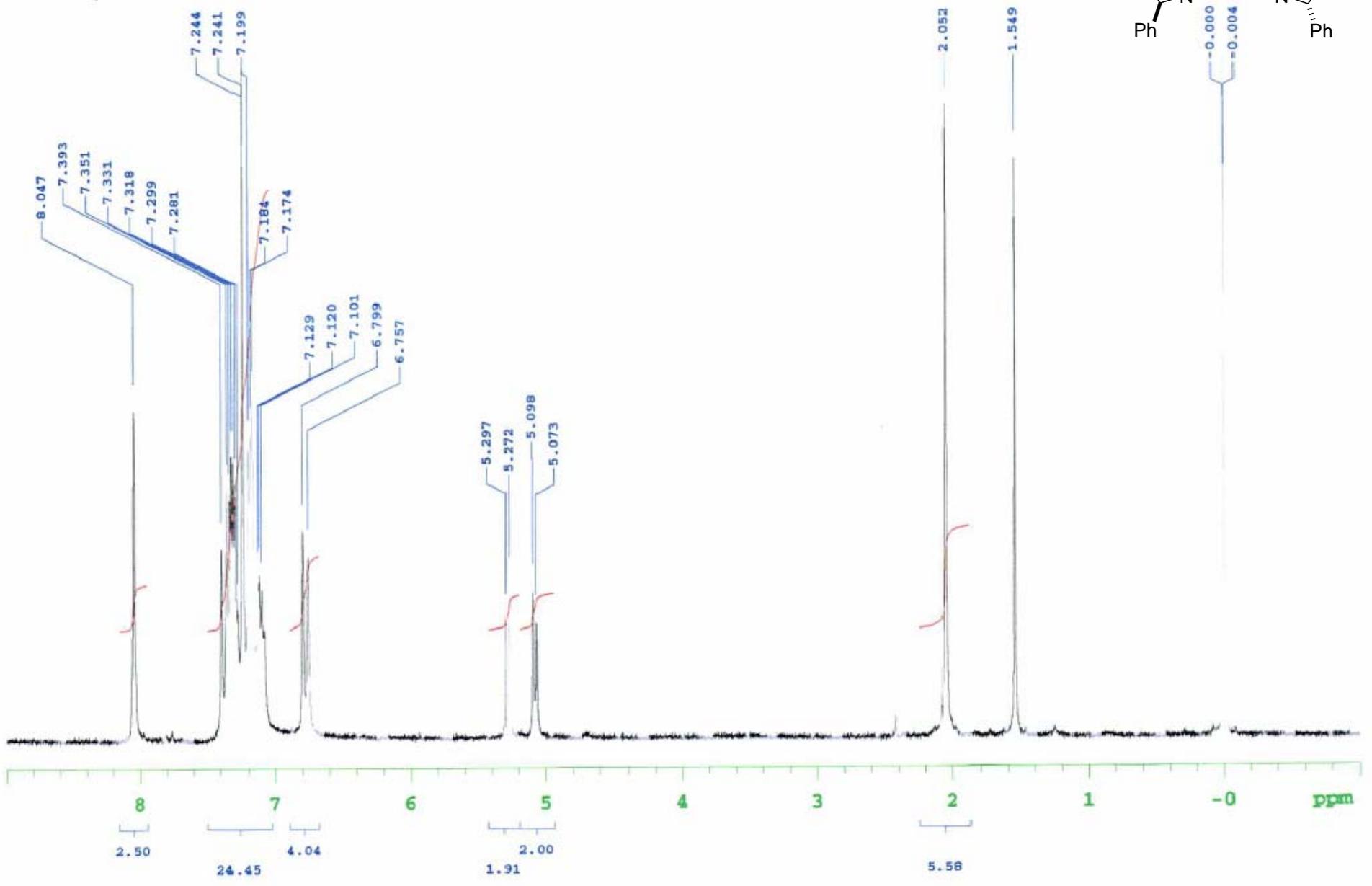
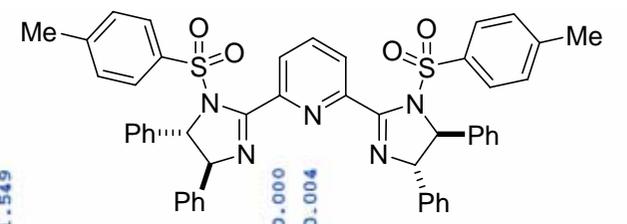
F2 - Acquisition Parameters
 Date_ 20071223
 Time_ 17.33
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 5160.6
 DW 11.000 usec
 DE 6.00 usec
 TE 297.7 K
 D1 0.60000002 sec
 d11 0.03000000 sec
 DELTA 0.50000000 sec
 TDO 1

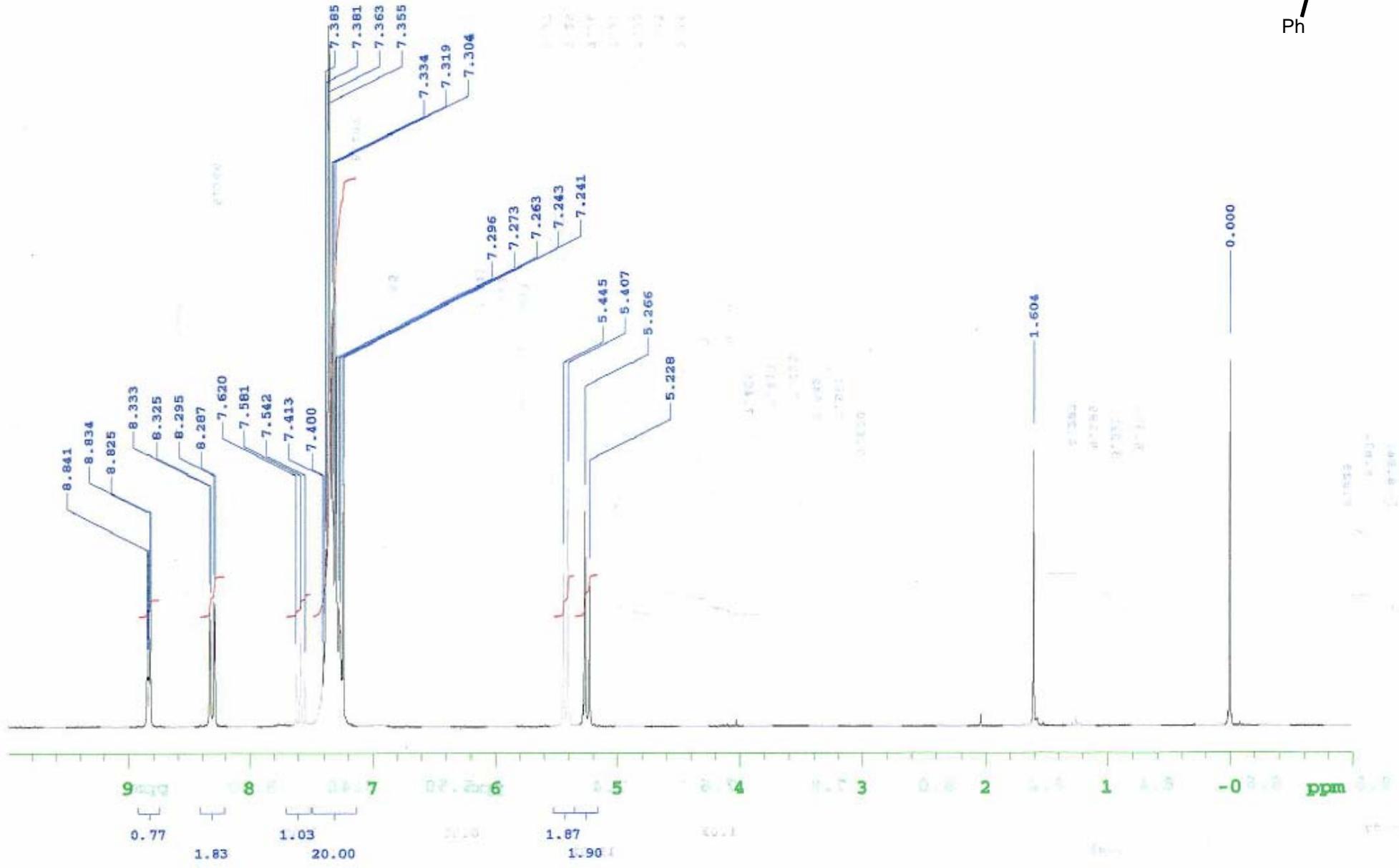
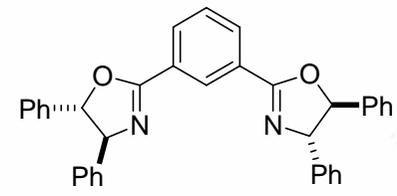
==== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028214 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

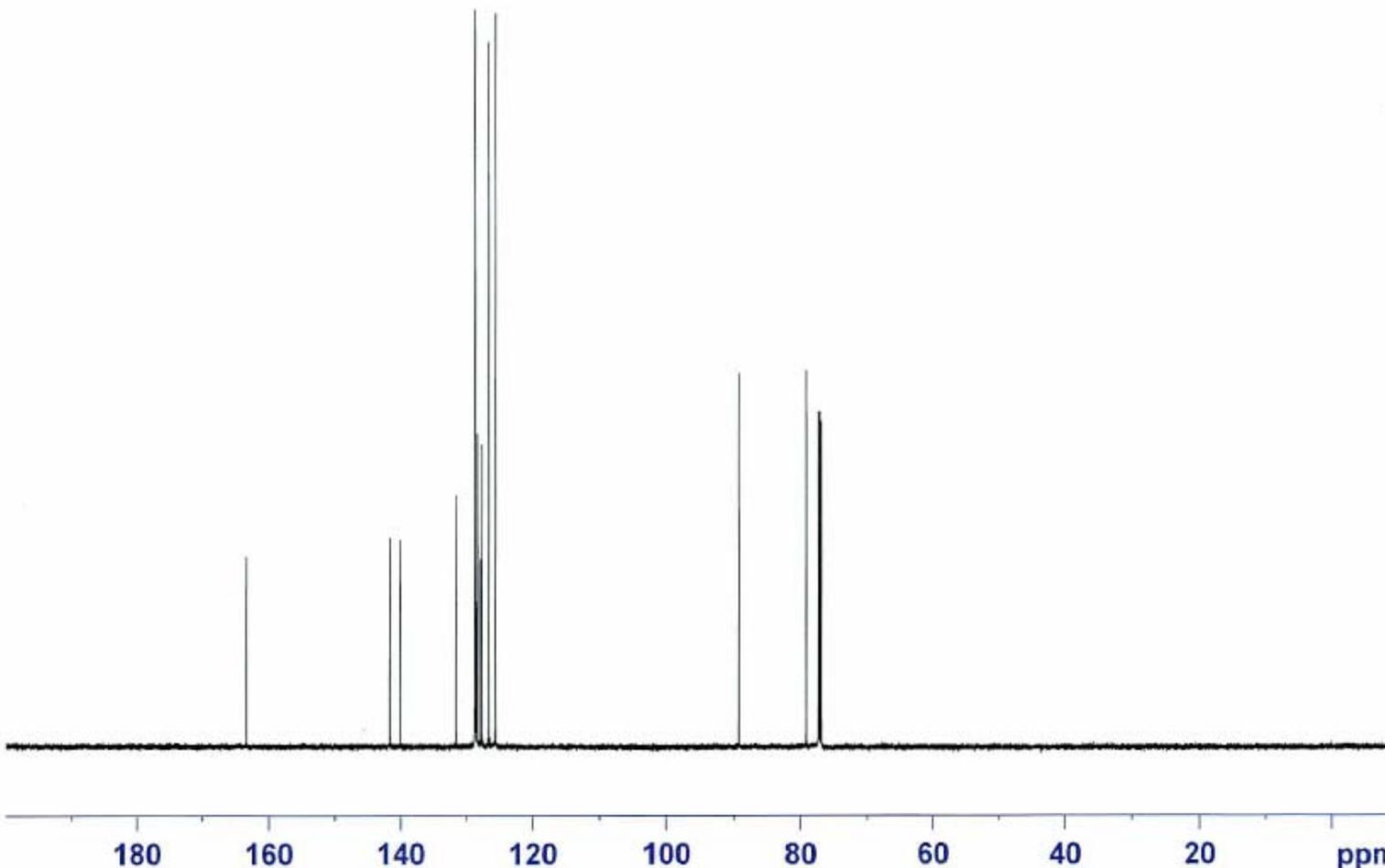
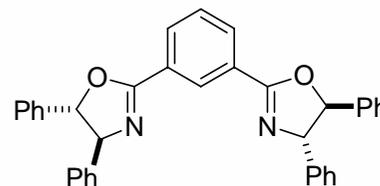






13C

163.327
 141.723
 140.145
 131.686
 128.883
 128.835
 128.738
 128.683
 128.487
 127.990
 127.796
 126.703
 125.758
 89.205
 78.968
 77.212
 77.001
 76.789



Current Data Parameters
 NAME HK-512
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date 20071217
 Time 21.59
 INSTRUM drx600
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 131072
 SOLVENT CDC13
 NS 206
 DS 4
 SWH 45454.547 Hz
 FIDRES 0.346791 Hz
 AQ 1.4418530 sec
 RG 5792.6
 DW 11.000 usec
 DE 6.00 usec
 TE 297.7 K
 D1 0.60000002 sec
 d11 0.03000000 sec
 DELTA 0.50000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 4.50 dB
 SFO1 150.9223664 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 82.00 usec
 PL2 -4.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SFO2 600.1324005 MHz

F2 - Processing parameters
 SI 131072
 SF 150.9028221 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

