Supporting Information

Iron-Catalyzed Asymmetric Epoxidation of β,β-Disubstituted Enones

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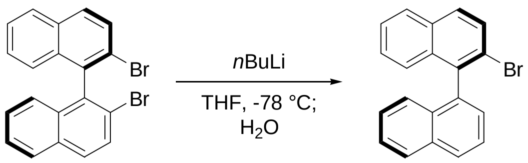
1. General Information. All reactions were carried out in flame-dried glassware under nitrogen atmosphere and stirred via magnetic stir-plates. All reactions were monitored by analytical thin-layer chromatography using Whatman pre-coated silica gel plates with F254 indicator. Visualization was accomplished by UV light (256 nm), phosphomolybdic acid, iodine, or anisaldehyde. Flash column chromatography was performed using Biotage Isolera one with Biotage SNAP cartridge KP-sil or KP-NH. All reactions were carried out with anhydrous solvents unless otherwise noted. Anhydrous acetonitrile was dried with M BRAUN solvent purification system (A2 Alumina). $FeCl_2$ and peracetic acid were purchased from Aldrich and used as received. $Fe(OTf)_2$ was purchased from Strem and used as received. All other reagents and starting materials, unless otherwise noted, were purchased from commercial vendors. Infrared spectra were recorded as thin films on sodium chloride plates using a Nicolet 20 SXB FTIR. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance 500 (500 MHz ^{1}H, 126 MHz ^{13}C). Chemical shift values (δ) are reported using tetramethylsilane in $CDCl_3$ or residual CD_2HOD (δH 3.31) and CD_3OD (δC 49.15) in CD_3OD or residual DMSO-d_5 (δH 2.50) and DMSO-d_6 (δC 39.51). The ^{1}H NMR spectra are reported as follows δ (number of protons, multiplicity, coupling constant *J*). Multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublet), m (multiplet) and br (broad).

2. Experimental procedure for preparation of ligands L1-L6 and characterization data.

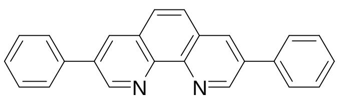
2.1. Experimental procedure for preparation of SI-1 and characterization data.



SI-1: **SI-1** was synthesized by modified literature procedure.[1] To a solution of (*R*)-2,2'-dibromo-1,1'-binaphthyl[2] (2.51 g, 6.09 mmol) in THF (30 mL) was added *n*-BuLi (1.6M solution in Hex, 3.8 mL, 6.1 mmol) at -78 ^{o}C. After stirring at that temperature for 1 h, the reaction was quenched by adding saturated aqueous NH_4Cl, warmed up to room temperature and then diluted with EtOAc. The organic layer was separated, washed with brine, dried with Na_2SO_4 and evaporated to give the crude product which was purified by a short pad of silica gel. The obtained oil was crystallized from hexane to furnish the title compound (87%) concomitant with a small amount of 1,1'-binaphthyl as white-pink crystal. ^{1}H-NMR ($CDCl_3$) 8.00 (1H, d, *J*=8.5), 7.97 (1H, d, *J*=8.5), 7.91 (1H, d, *J*=8.0), 7.83 (1H, d, *J*=8.5), 7.80 (1H, d, *J*=8.5), 7.64 (1H, dd, *J*=8.3, 7.3), 7.49 (2H, m), 7.41 (1H, dd, *J*=7.0, 1.0), 7.31 (1H, ddd, *J*=8.3, 7.0, 1.0), 7.27 (1H, m), 7.22 (1H, d, *J*=8.5), 7.18 (1H, d, *J*=8.5). ^{13}C NMR ($CDCl_3$) δ 138.1, 137.3, 134.4, 133.6, 132.3, 132.0, 129.9, 129.3, 12 8.3, 127.95, 127.92, 126.9, 126.8, 126.3, 126.1, 126.0, 125.7, 125.5, 122.7.

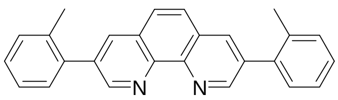
2.2. General procedure for preparation of SI-2~5 and characterization data.

To a mixture of 3,8-dibromophenanthroline[3] (676 mg, 2.0 mmol), $Pd(PPh_3)_4$ (347 mg, 0.3 mmol), and a corresponding boronic acid (5.0 mmol) were added a degassed mixture of THF (14 mL) and toluene (14 mL) at room temperature under nitrogen atmosphere. After the addition of a degassed aq. Na_2CO_3 (1 M solution, 5.0 mL, 5.0 mmol), the reaction mixture was heated to reflux for 18 h. After cooling to room temperature, the reaction was poured into 1 M aqueous NaOH (20 mL) and extracted three times with CH_2Cl_2. The organic layer was dried with Na_2SO_4 and evaporated to give the crude product which was purified by silica gel chromatography. The obtained oil was crystallized from hexane/EtOAc to furnish a desired product.



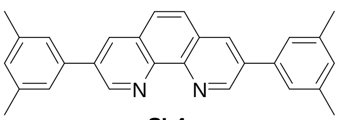
SI-2

SI-2[4]: 81% yield as a white solid, m.p. 186-190 °C (CH_2Cl_2), lit. [4] m.p. 190-191 °C (benzene-petroleum ether); ^{1}H NMR ($CDCl_3$) δ 9.45 (2H, d, *J* = 2.0 Hz), 8.41 (2H, t, *J* = 1.9 Hz), 7.89 (2H, d, *J* = 1.6 Hz), 7.80 (4H, d, *J* = 8.2 Hz), 7.57 (4H, t, *J* = 7.6 Hz), 7.48 (2H, t, *J* = 7.1 Hz); ^{13}C NMR ($CDCl_3$) δ 149.5, 145.1, 137.6, 135.8, 133.4, 129.2, 128.5, 128.4, 127.6, 127.1; IR (neat) 2362, 2338, 1653, 1600, 1558, 1443, 1426, 1274, 908, 761, 724, 699 cm^{-1}; MS (ESI$^+$) m/z (%): 665 ([2M+H]$^+$, 30), 333 ([M+H]$^+$, 100).



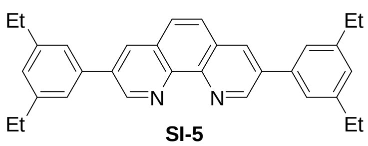
SI-3

SI-3: 61 % as white crystal, m.p. 157-160 °C; ^{1}H NMR ($CDCl_3$) δ 9.22 (2H, d, *J* = 2.2 Hz), 8.22 (2H, d, *J* = 2.2 Hz), 7.88 (2H, s), 7.36-7.45 (8H, m), 2.38 (6H, s); ^{13}C NMR ($CDCl_3$)δ 151.2, 144.8, 137.9, 136.8, 135.9, 135.5, 130.7, 130.2, 128.4, 128.1, 126.9, 126.2, 20.5; IR (neat) 3017, 2959, 1559, 1457, 1423, 918, 757 cm^{-1}; MS (ESI$^+$) m/z (%): 361 ([M+H]$^+$, 100).



SI-4

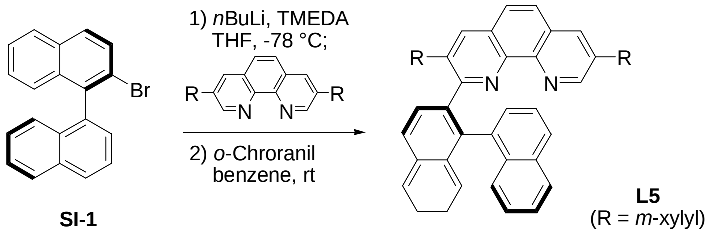
SI-4: 66% yield as yellow solid, m.p. 208-212 °C; ^{1}H NMR ($CDCl_3$) δ 9.42 (2H, d, *J* = 2.3 Hz), 8.39 (2H, d, *J* = 2.2 Hz), 7.88 (2H, s), 7.40 (4H, s), 7.12 (2H, s), 2.46 (12H, s); ^{13}C NMR ($CDCl_3$)δ 149.6, 145.0, 138.8, 137.5, 135. 9, 133.3, 130.0, 128.4, 127.0, 125.4, 21.4; IR (neat) 3030, 2917, 2860, 1603, 1435, 1366, 1221, 1038, 849, 736, 699 cm^{-1}; MS (ESI$^+$) m/z (%): 777 ([2M+H]$^+$, 50), 389 ([M+H]$^+$, 100).



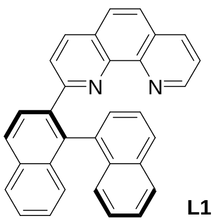
SI-5

SI-5: 54 % yield as a brown solid, m.p. 132-134 °C; ^{1}H NMR ($CDCl_3$) δ 9.43 (2H, d, *J* = 2.2 Hz), 8.38 (2H, d, *J* = 2.1 Hz), 7.87 (2H, s), 7.43 (4H, s), 7.16 (2H, s), 2.77 (8H, q, *J* = 7.6), 1.34 (12H, t, *J* = 7.6 Hz); ^{13}C NMR ($CDCl_3$) δ 149.7, 145.3, 145.0, 137.7, 136.1, 133.3, 128.4, 127.6, 127.0, 1 24.6, 28.9, 15.7; IR (neat) 3023, 2963, 2931, 2871, 1600, 1423, 1370, 868, 736, 705 cm^{-1}; MS (ESI$^+$) m/z (%): 445 ([M+H]$^+$, 100).

2.3. Representative procedure for the preparation of L1-6 and characterization data.

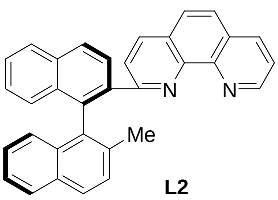


To a solution of **SI-1** (858 mg, 2.57 mmol) in THF (13 mL) was added TMEDA (0.44 mL, 2.9 mmol) at room temperature. After cooling to -78 °C, *n*-BuLi (1.6 M solution in Hex, 1.8 mL, 2.9 mmol) was added dropwise to the mixture which was stirred at that temperature for 1 h. To this solution was added a THF solution (26 mL in 2 portions) of **SI-4** (1.50 g, 3.86 mmol) which was pre-dried by azeotropic removal of water with toluene. Warmed up to room temperature overnight, the reaction was further stirred for 12 h at room temperature. The reaction was quenched by adding water and extracted three times with CH_2Cl_2 and the combined organic layers were dried with Na_2SO_4 and evaporated. To the obtained residue in benzene (20 mL) was added a solution of *o*-chloranil (696 mg, 2.83 mmol) in benzene (6 mL) at room temperature. After stirring at the same temperature for 2 h, the reaction was quenched by adding 1 M aqueous NaOH solution. The mixture was filtered with celite, and the filtrate was extracted three times with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4 and evaporated. The resulting crude product was purified by silica gel chromatography (EtOAc/*n*-Hex) to give **L5** (1.03 g) which was further purified by recrystallization from *n*-hexane/EtOAc.



L1

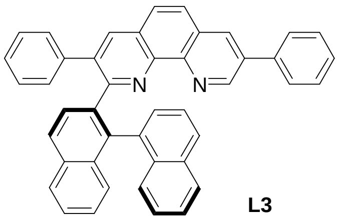
L1 (86% yield): White solid, m.p. 195-200 °C; ^{1}H NMR (CD_3OD) δ 9.06 (1H, d, *J* = 2.0 Hz), 8.39 (1H, dd, *J* = 8.1, 1.5 Hz), 8.27 (1H, d, *J* = 8.5 Hz), 8.06 (1H, d, *J* = 8.2 Hz), 7.91 (1H, s), 7.90 (1H, d, *J* = 5.8 Hz), 7.86 (1H, d, *J* = 8.0 Hz), 7.78 (1H, dt, *J* = 8.7, 1.6 Hz), 7.71-7.76 (2H, m), 7.70 (1H, dt, *J* = 8.4, 1.7 Hz), 7.65 (1H, dt, *J* = 8.8, 1.7 Hz), 7.54 (1H, t, *J* = 7.4 Hz), 7.46 (1H, d, *J* = 8.5 Hz), 7.41 (1H, dd, *J* = 8.1, 7.0 Hz), 7.24-7.39 (4H, m), 7.07-7.12 (1H, m), ; ^{13}C NMR (CD_3OD) δ 161.3, 150.8, 146.8, 146.5, 140.0, 138.2, 137.93, 137.90, 136.3, 135.3, 135.1, 134.9, 134.5, 131.0, 130.6, 129.9, 129.7, 129.4, 128.4, 128.2, 127.8, 127.67, 127.65, 127.59, 127.55, 127.1, 126.6, 125.7, 124.6; IR (neat) 2361, 2337, 1653, 1559, 1540, 1506, 752 cm^{-1}; MS (ESI$^+$) m/z (%): 433 ([M+H]$^+$, 100); $[\alpha]_D^{24}$ +316 (MeOH, *c* 0.36)



L2

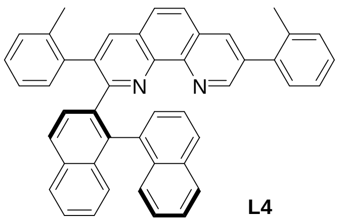
L2 (53% yield): Pale brown solid, m.p. 124-127 °C (decomp.); ^{1}H NMR ($CDCl_3$) δ 9.24 (1H, dd, *J* = 4.3, 1.6 Hz), 8.48 (1H, d, *J* = 8.6 Hz), 8.17 (1H, dd, *J* = 8.1, 1.5 Hz), 8.16 (1H, d, *J* = 8.9 Hz), 8.00 (1H, d, *J* = 8.2 Hz), 7.85 (1H, d, *J* = 7.8 Hz), 7.80 (1H, d, *J* = 8.4 Hz), 7.65 (1H, d, *J* = 8.7 Hz), 7.60 (1H, dd, *J* = 7.9, 4.6 Hz), 7.59 (1H, d, *J* = 8.4 Hz), 7.55 (1H, d, *J* = 8.7 Hz), 7.50 (1H, t, *J* = 7.4 Hz), 7.34-7.39 (2H, m), 7.28 (1H, d, *J* = 8.4 Hz), 7.21-7.27 (2H, m), 7.18 (1H, d, *J* = 8.5 Hz), 7.01 (1H, d, *J* = 8.4

Hz), 1.98 (3H, s); ^{13}C NMR ($CDCl_3$) δ 159.1, 150.3, 146.5, 146.0, 138.4, 135.9, 135.7, 135.2, 134.8, 134.5, 134.0, 133.8, 132.6, 131.8, 129.1, 128.78, 128.75, 128.6, 128.1, 127.9, 127.8, 126.8, 126.7, 126.45, 126.40, 126.3, 126.1, 124.8, 123.4, 122.6, 20.9; IR (neat) 2361, 1588, 1507, 1487, 853, 814, 751 cm^{-1}; MS (ESI$^+$) m/z (%): 447 ([M+H]$^+$, 100); $[\alpha]_D^{26}$ +299 ($CHCl_3$, *c* 0.95)



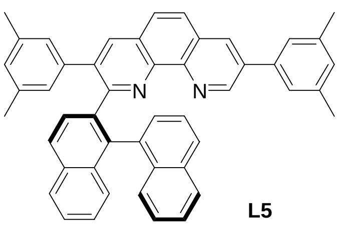
L3

L3 (78% yield): White semisolid; ^{1}H NMR (DMSO-d_6, VT80) δ 9.39 (1H, d, *J* = 2.0 Hz), 8.68 (1H, d, *J* = 2.0 Hz), 8.17 (1H, d, *J* = 8.4 Hz), 8.09 (1H, s), 8.08 (1H, d, *J* = 7.4 Hz), 7.97 (1H, d, *J* = 8.8 Hz), 7.92-7.97 (4H, m), 7.88 (1H, d, *J* = 8.8 Hz), 7.72 (1H, br-d, *J* = 8.0 Hz), 7.69 (1H, d, *J* = 8.5 Hz), 7.61 (2H, t, *J* = 7.7 Hz), 7.48-7.55 (2H, m), 7.27 (1H, ddd, *J* = 8.2, 7.1, 1.1 Hz), 7.22 (1H, br-t, *J* = 7.2 Hz), 7.15-7.20 (1H, m), 7.14 (1H, t, *J* = 7.6 Hz), 7.04-7.12 (1H, m), 7.01 (1H, d, *J* = 8.5 Hz), 6.74-6.98 (5H, m); ^{13}C NMR (DMSO-d_6, VT80)δ 157.6, 148.0, 144.2, 143.0, 141.6, 138.5, 138.08, 138.05, 136.6, 136.1, 135.4, 135.2, 134.3, 134.1, 132.6, 132.3, 132.2, 132.0, 131.6, 128.8, 128.7, 128.1, 127.9, 127.6, 127.3, 127.1, 127.0, 126.8, 126.7, 126.6, 126.3, 125.9, 125.7, 125.6, 1 24.8, 124.5, 124.1; IR (neat) 2361, 1506, 1410, 803, 779, 751, 697 cm^{-1} ; MS (ESI$^+$) m/z (%): 585 ([M+H]$^+$, 100); $[\alpha]_D^{22}$ +248 ($CHCl_3$, *c* 0.5)



L4

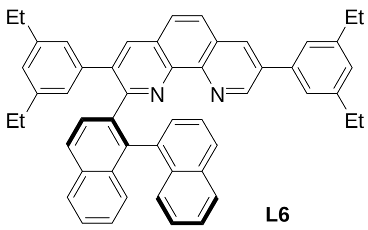
L4 (58% yield): White solid, m.p. 250-252 °C; ^{1}H NMR (DMSO-d_6, VT80) δ 9.08 (1H, d, *J* = 1.6 Hz), 8.42 (1H, d, *J* = 2.1 Hz), 8.07-8.12 (2H, m), 8.03 (1H, d, *J* = 8.2 Hz), 7.97 (1H, d, *J* = 8.8 Hz), 7.90 (1H, d, *J* = 8.8 Hz), 7.79-7.87 (1H, br-s), 7.80 (1H, d, *J* = 8.2 Hz), 7.71-7.76 (1H, m), 7.48 (1H, t, *J* = 7.5 Hz), 7.36-7.46 (4H, m), 7.34 (1H, t, *J* = 7.4 Hz), 7.22 (3H, t, *J* = 7.2 Hz), 7.04 (1H, br-t, *J* = 7.4 Hz), 6.93 (1H, d, *J* = 8.4 Hz), 6.75-7.10 (5H, m), 3.06 (3H, s), 2.38 (3H, s); ^{13}C NMR (DMSO-d_6, VT80) δ 149.7, 143.9, 143.0, 138.4, 137.3, 137.1, 137.0, 135.6, 135.3, 135.0, 134.9, 134.5, 132.5 , 132..1, 132.0, 131.9, 130.20, 130.15, 129.9, 129.6, 127.77, 127.74, 127..6, 127.14, 127.05, 126.8, 126.46, 126. 43, 126.3, 126.1, 125.9, 125.8, 125.7, 125.5, 125.0, 124.7, 124.24, 124.18, 78.7, 19.6; IR (neat) 3055, 2957, 1494, 1455, 1430, 1408, 1216, 1074, 924, 803, 752 cm^{-1}; MS (ESI$^+$) m/z (%): 613 ([M+H]$^+$, 100); $[\alpha]_D^{22}$ +507 ($CHCl_3$, *c* 0.93)



L5

L5 (62% yield): Yellow solid, m.p. 145-148 °C (decomp.); ^{1}H NMR (DMSO-d_6, VT80) δ 9.37 (1H, d, *J* = 2.0 Hz), 8.64 (1H, d, *J* = 1.9 Hz), 8.18 (1H, d, *J* = 8.4 Hz), 8.08 (1H, d, *J* = 8.2 Hz), 8.03 (2H, s), 7.97 (1H, d, *J* = 8.4 Hz) 7.95 (1H, d, *J* = 10.0 Hz), 7.85 (1H, d, *J* = 8.8 Hz), 7.72 (1H, br-d, *J* = 7.5 Hz), 7.68 (1H, d, *J* = 8.7 Hz), 7.53 (2H, s), 7.51 (1H, ddd, *J* = 8.1, 6.9, 1.2 Hz), 7.25 (1H, ddd, *J* = 8.3, 7.0, 1.2 Hz), 7.19-7.28 (1H, m), 7.13 (1H, br-s), 7.11

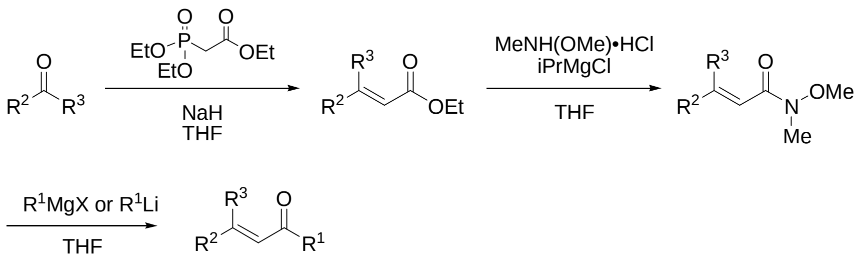
(1H, d, J = 7.8 Hz), 6.99 (1H, d, J = 8.5 Hz), 6.91 (1H, t, J = 7.2 Hz), 6.74 (1H, br-s), 6.48 (2H, br-s), 2.43 (6H, s), 2.02 (6H, br-s); ^{13}C NMR (DMSO-d_6, VT80) δ 157.7, 148.0, 144.1, 142.9, 138.7, 138.0, 137.8, 136.5, 13 6.2, 135.8, 135.6, 135.0, 134.3, 134.2, 132.5, 132.2, 132.0, 131.6, 129.4, 128.8, 127.6, 127.0, 126.8, 126.72, 12 6.66, 126.5, 126.21, 125.81, 125.77, 125.5, 124.6, 124.5, 123.9, 20.5, 20.3 ; IR (neat) 1602, 1407, 1218, 849, 802, 747 cm^{-1}; MS (ESI$^+$) m/z (%): 641 ([M+H]$^+$, 100); $[\alpha]_D^{23}$ +432 (CHCl$_3$, *c* 1.00)



L6

L6 (24% yield): Yellow solid, m.p. 153-158 °C (decomp.); ^{1}H NMR (DMSO-d_6, VT80) δ 9.38 (1H, d, J = 2.0 Hz), 8.65 (1H, br-s), 8.20 (1H, d, J = 8.4 Hz), 8.05-8.13 (3H, m), 7.99 (1H, d, J = 8.4 Hz), 7.96 (1H, d, J = 8.9 Hz), 7.87 (1H, d, J = 8.7 Hz), 7.65-7.73 (2H, m), 7.57 (2H, br-s), 7.51 (1H, ddd, J = 8.0, 7.1, 0.9 Hz), 7.24 (1H, br-t, J = 7.2 Hz), 7.19 (1H, br-s), 7.11 (1H, br-t, J = 7.6 Hz), 6.98 (1H, d, J = 8.4 Hz), 6.88 (1H, br-t, J = 7.3 Hz), 6.82 (2H, br-s), 6.59 (2H, br-s), 2.76 (4H, q, J = 7.6 Hz), 2.28-2.42 (4H, m), 1.31 (6H, t, J = 7.6 Hz), 0.95 (6H, t, J = 7.6 Hz); IR (neat) 1598, 1458, 1408, 1216, 869, 745, 709 cm^{-1}; MS (ESI$^+$) m/z (%): 697 ([M+H]$^+$, 100); $[\alpha]_D^{24}$ +331 (CHCl$_3$, *c* 0.57)

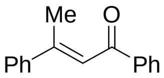
3. Experimental procedure for preparation of starting material 2a-d and characterization data.



General procedure[5]: To a suspension of NaH (60 % dispersion in oil, 12.0 mmol) in THF (10 mL) was added a solution of triethyl phosphonoacetate (12.0 mmol) in THF (5 mL) under an nitrogen atmosphere at 0 °C. After being stirred at room temperature for 30 min, a solution of a corresponding ketone (10 mmol) in THF (5 mL) was added to the reaction mixture at 0 °C. After being further stirred at room temperature for 16 h, the reaction was quenched by adding sat. aq. NaHCO$_3$ and diluted with EtOAc. The organic layer was separated and washed with brine, dried over MgSO$_4$ and concentrated at reduced pressure. The residue was purified by silica gel column chromatography (Et$_2$O:*n*-Hex=0:100 to 10:90) to afford the corresponding α,β-unsaturated ester.

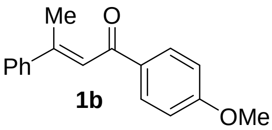
To a suspension of the α,β-unsaturated ester (3.0 mmol) obtained above and *N,O*-dimethylhydroxylamine hydrochloride (6.0 mmol) in THF (6.0 mL) was added dropwise *i*PrMgCl (2.0 M solution in THF, 6.0 mmol) under nitrogen atmosphere at -5 to -10 °C. After being stirring at 0 °C for 30 min, the reaction was quenched by adding sat. aq. NH_4Cl and diluted with EtOAc. The organic layer was separated and washed with brine, dried over $MgSO_4$ and concentrated at reduced pressure. The residue was purified by silica gel column chromatography (EtOAc:*n*-Hex=10:90 to 40:60) to afford the corresponding Weinreb amide.

To a solution of the Weinreb amide (1.0 mmol) obtained above in THF (2.0 mL) was added dropwise a solution of the corresponding Grignard reagent or lithium reagent (1.5 mmol) under nitrogen atmosphere at -30 °C. After being stirring at 0 °C for 30 min, the reaction was quenched by adding sat. aq. NH_4Cl and diluted with EtOAc. The organic layer was separated and washed with brine, dried over $MgSO_4$ and concentrated at reduced pressure. The residue was purified by silica gel column chromatography (EtOAc:*n*-Hex=0:100 to 10:90) to afford the corresponding β,β-disubstituted enone.



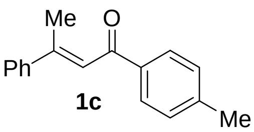
1a

1a (43% yield) [5]: yellow oil; ^{1}H NMR ($CDCl_3$) δ 7.98-8.02 (2H, m), 7.53-7.59 (3H, m), 7.45-7.50 (2H, m), 7.38-7.45 (3H, m), 7.17 (1H, d, *J* = 1.3 Hz), 2.60 (3H, d, *J* = 1.3 Hz); ^{13}C NMR ($CDCl_3$) δ 191.8, 155.0, 142.7, 139.3, 132.5, 129.1, 128.6, 128.5, 128.2, 126.4, 122.1, 18.8; IR (neat) 3058, 1658, 1599, 1493, 1448, 1275, 1214, 1047, 1025, 951, 854, 753 cm^{-1}; MS (ESI$^+$) m/z (%): 223 ([M+H]$^+$, 100).



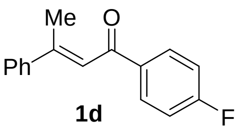
1b

1b (26% yield): yellow oil; ^{1}H NMR ($CDCl_3$) δ 8.00 (2H, d, *J* = 8.9 Hz), 7.55-7.59 (2H, m), 7.37-7.45 (3H, m), 7.13 (1H, d, *J* = 1.2 Hz), 6.96 (2H, d, *J* = 8.9 Hz), 3.88 (3H, s), 2.57 (3H, d, *J* = 1.2 Hz); ^{13}C NMR ($CDCl_3$) δ 190.7, 163.2, 153.7, 142.9, 132.2, 130.6, 128.9, 128.6, 126.4, 122.3, 113.7, 55.5, 18.8; IR (neat) 3062, 3030, 2975, 2938, 2879, 1691, 1598, 1450, 1402, 1228, 1001, 946, 845, 751; MS (ESI$^+$) m/z (%): 253 ([M+H]$^+$, 100).



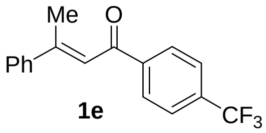
1c

1c (45% yield): yellow oil; ^{1}H NMR ($CDCl_3$) δ 7.91 (2H, d, *J* = 8.2 Hz), 7.57 (1H, dd, *J* = 8.0, 1.5 Hz), 7.37-7.45 (3H, m), 7.27 (2H, d, *J* = 8.0 Hz), 7.15 (1H, d, *J* = 1.0 Hz), 2.58 (3H, d, *J* = 1.1 Hz), 2.42 (3H, s); ^{13}C NMR ($CDCl_3$) δ 191.6, 154.4, 143.3, 142.9, 136.8, 129.2, 129.0, 128.6, 128.4, 126.5, 122.3, 21.6, 18.8; IR (neat) 3056, 3029, 2920, 1653, 1608, 1573, 1446, 1276, 1221, 1206, 1179, 1049, 945, 822 cm^{-1}; MS (ESI$^+$) m/z (%): 237 ([M+H]$^+$, 100).



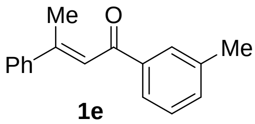
1d

1d (46% yield): yellow oil; ^{1}H NMR ($CDCl_3$) δ 8.02 (2H, m), 7.55-7.59 (2H, m), 7.38-7.45 (3H, m), 7.15 (2H, t, *J* =8.6 Hz), 7.12 (1H, s), 2.59 (3H, s); ^{13}C NMR ($CDCl_3$) δ 190.3, 165.4 (d, *J* = 254 Hz), 155.4, 142.6, 135.7 (d, *J* =2.4 Hz), 130.8 (d, *J* = 9.2 Hz), 129.2, 128.6, 121.7, 115.6 (d, *J* = 21.8 Hz), 18.9; IR (neat) 3061, 1656, 1599, 1504, 1446, 1276, 1213, 1155, 841, 766 cm^{-1}; MS (ESI^{+}) m/z (%): 241 ([M+H]$^{+}$, 100).



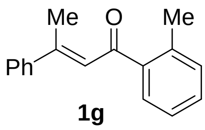
1e

1e (39% yield): yellow solid; ^{1}H NMR ($CDCl_3$) δ 8.08 (2H, d, J = 8.16 Hz), 7.74 (1H, d, *J* = 8.12 Hz), 7.55-7.62 (2H, m), 7.40-7.47 (3H, m), 7.15 (1H, s), 2.64 (3H, s).; ^{13}C NMR ($CDCl_3$) δ 190.4, 157.2, 142.5, 142.2, 133.8 (q, *J* = 32.6 Hz), 129.5, 128.7, 128.5, 126.5, 125.8 (q, *J* = 5.2 Hz), 123.7 (q, *J* = 272 Hz), 121.2, 19.1.; IR (neat) 1661 1596, 1575, 1447, 1410, 1325, 1276, 1213, 1129, 1112, 1067, 1015, 950, 844, 768, 746, 694 cm^{-1}; MS (ESI^{+}) m/z (%): 291 ([M+H]$^{+}$, 100).



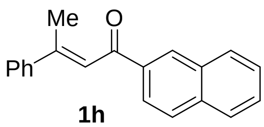
1e

1f (29% yield): yellow oil; ^{1}H NMR ($CDCl_3$) δ 7.77-7.81 (2H, m), 7.56-7.59 (2H, m), 7.39-7.45 (3H, m), 7.39-7.33 (2H, m), 7.14 (1H, d, *J* = 1.1 Hz), 2.59 (3H, d, *J* = 1.1 Hz), 2.42 (3H, s); ^{13}C NMR ($CDCl_3$) δ 192.1, 154.7, 142.8, 139.4, 138.3, 133.3, 129.1, 128.8, 128.6, 128.4, 126.5, 125.5, 122.3, 21.4, 18.8; IR (neat) 3058, 2920, 1656, 1594, 1446, 1378, 1278, 1241, 1164, 761 cm^{-1}; MS (ESI^{+}) m/z (%): 237 ([M+H]$^{+}$, 100).



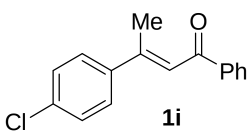
1g

1g (28% yield): yellow oil; ^{1}H NMR ($CDCl_3$) δ 7.58-7.61 (1H, m), 7.53-7.56 (2H, m), 7.35-7.42 (4H, m), 7.25-7.28 (2H, m), 6.89 (1H, d, *J* = 1.3 Hz), 2.58 (3H, d, *J* = 1.2 Hz), 2.53 (3H, s); ^{13}C NMR ($CDCl_3$) δ 196.1, 154.4 , 142.5, 140.6, 137.3, 131.5, 130.7, 129. 2, 128.6, 128.4, 126.5, 125.6, 125.3, 20.6, 18.6; IR (neat) 1659, 1593, 1573, 1446, 1212, 1040, 942, 756, 695 cm^{-1}; MS (ESI^{+}) m/z (%): 237 ([M+H]$^{+}$, 100).



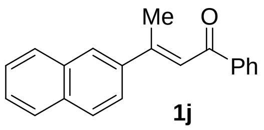
1h

1h (43% yield)[6]: ; ^{1}H NMR ($CDCl_3$) δ 8.50 (1H, br-s), 8.09 (1H, dd, *J* = 8.6, 1.7 Hz), 7.97 (1H, d, *J* = 8.1 Hz), 7.93 (1H, d, *J* = 8.6 Hz), 7.89 (1H, d, *J* = 7.9 Hz), 7.63 (2H, m), 7.60 (1H, ddd, *J* = 8.1, 6.8, 1.3 Hz), 7.55 (1H, ddd, *J* = 8.1, 6.8, 1.3 Hz), 7.40-7.49 (2H, m), 7.32 (1H, d, *J* = 1.3 Hz), 2.64 (3H, d, *J* = 1.3 Hz); ^{13}C NMR ($CDCl_3$) δ 191.8, 155.0, 142.8, 136.6, 135.3, 132.6, 129.7, 129.5, 129.1, 128.6, 128.4, 128.2, 127.8, 126.7, 126.5, 124.3, 122.2, 18.9; IR (neat) 3056, 1650, 1626, 1595, 1572, 1446, 1380, 1281, 1183, 1124, 856, 821, 763 cm^{-1}; MS (ESI^{+}) m/z (%): 273 ([M+H]$^{+}$, 100).



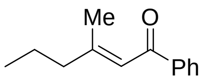
1i

1i (31% yield): yellow oil; ^{1}H NMR ($CDCl_3$) δ 7.96-8.00 (2H, m), 7.56 (1H, t, *J* = 7.4 Hz), 7.46-7.53 (4H, m), 7.39 (2H, d, *J* = 8.6 Hz), 7.14 (1H, d, *J* = 1.2 Hz), 2.56 (3H, d, *J* = 1.1 Hz); ^{13}C NMR ($CDCl_3$) δ 191.7, 153.4, 141.0, 139.1, 135.1, 132.7, 128.8, 128.6, 128.2, 127.8, 122.4, 18.7; IR (neat) 1655, 1600, 1489, 1447, 1276, 1214, 1095, 1046, 1011, 825, 780, 691 cm^{-1}; MS (ESI^{+}) m/z (%): 259 ([M+H+2]+, 30), 257 ([M+H]$^{+}$, 100).



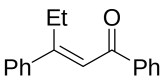
1j

1j (54% yield): yellow oil; ^{1}H NMR ($CDCl_3$) δ 8.00-8.05 (3H, m), 7.84-7.92 (3H, m), 7.70 (1H, dd, *J* = 8.6 Hz), 7.57 (1H, tt, *J* = 7.4, 1.3 Hz), 7.47-7.54 (4H, m), 7.32 (1H, d, *J* = 1.2 Hz), 2.71 (3H, d, *J* = 1.2 Hz); ^{13}C NMR ($CDCl_3$) δ 191.9, 154.8, 139.9, 139.4, 133.5, 133.2, 132.5, 128.6, 128.5, 128.29, 128.25, 127.6, 126.8, 126.6, 126.2, 124.1, 122.5, 18.9; IR (neat) 3057, 1655, 1600, 1447, 1281, 1268, 1237, 1211, 855, 818, 737, 698 cm^{-1}; MS (ESI^{+}) m/z (%): 273 ([M+H]$^{+}$, 100).



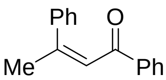
1k

1k (45% yield): colorless oil; ^{1}H NMR ($CDCl_3$) δ 7.92 (2H, d, *J* = 7.3 Hz), 7.52 (1H, t, *J* = 7.3 Hz), 7.45 (2H, t, *J* = 7.5 Hz), 6.73 (1H, s), 2.24 (2H, t, *J* = 7.6 Hz), 2.19 (3H, s), 1.61 (2H, sext, *J* = 7.5 Hz), 0.97 (3H, t, *J* = 7.4 Hz); ^{13}C NMR ($CDCl_3$) δ 191.7, 160.2, 139.4, 132.2, 128.4, 128.1, 120.6, 43.5, 20.8, 19.7, 13.7; IR (neat) 1660, 1611, 1448, 1239, 1179, 1037, 1022, 775, 700, 667 cm^{-1}; MS (ESI^{+}) m/z (%): 189 ([M+H]$^{+}$, 100).



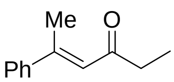
1l

1l (21% yield)[5]: yellow oil; ^{1}H NMR ($CDCl_3$) δ 7.99 (2H, d, *J* = 7.8 Hz), 7.52-7.57 (3H, m), 7.47 (2H, t, *J* = 7.6 Hz), 7.37-7.45 (3H, m), 7.04 (1H, s), 3.08 (2H, q, *J* = 7.5 Hz), 1.13 (3H, t, *J* = 7.5 Hz); ^{13}C NMR ($CDCl_3$) δ 191.5, 161.4, 141.6, 139.3, 132.5, 129.0, 128.6, 128.5, 128.3, 126.8, 121.9, 25.1, 13.6; IR (neat) 3058, 2970, 2933, 2873, 1657, 1599, 1493, 1448, 1377, 1290, 1240, 1213, 1178, 1046, 1002, 993, 761, 696 cm^{-1}; MS (ESI^{+}) m/z (%): 237 ([M+H]$^{+}$, 100).



1m

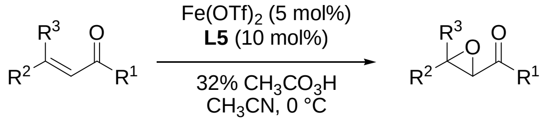
1m (18% yield): yellow oil; ^{1}H NMR ($CDCl_3$) δ 7.83 (2H, d, *J* = 8.4 Hz), 7.44 (1H, t, *J* = 7.4 Hz), 7.34 (2H, t, *J* = 7.7 Hz), 7.16-7.26 (5H, m), 6.69 (1H, s), 2.31 (3H, s); ^{13}C NMR ($CDCl_3$) δ 193.1, 152.4, 140.6, 138.0, 132.5, 128.7, 128.2, 128.04, 127.95, 127.4, 124.2, 26.5; IR (neat) 1663, 1653, 1636, 1616, 1597, 1577, 1492, 1448, 1240, 1201, 1176, 1025, 977, 848, 778, 761, 736, 696, 668 cm^{-1}; MS (ESI^{+}) m/z (%): 223 ([M+H]$^{+}$, 100).



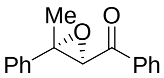
1o

1o (43% yield)[7]: colorless oil; ^{1}H NMR ($CDCl_3$) δ 7.47-7.51 (2H, m), 7.35-7.41 (3H, m), 6.50 (1H, d, *J* = 1.2 Hz), 2.57 (2H, q, *J* = 7.3), 2.55 (3H, d, *J* = 1.1 Hz), 1.13 (3H, t, *J* = 7.3); ^{13}C NMR ($CDCl_3$) δ 201.9, 153.5, 142.7, 129.0, 128.5, 126.4, 124.0, 38.0, 18.4, 8.2; IR (neat) 1684, 1604, 1447, 1377, 1124, 1045, 951, 756, 695 cm^{-1}.

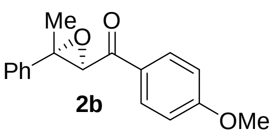
4. Experimental procedure for Iron-catalyzed asymmetric epoxidations and characterization data.



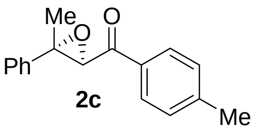
General procedure: A solution of $Fe(OTf)_2$ (0.025 M solution in CH_3CN, 0.31 mL, 7.8 μmol) was added to **L5** (10 mg, 15.6 μmol) under nitrogen atmosphere at room temperature. After rinsed with additional CH_3CN (0.31 mL), the reaction mixture was stirred at room temperature for 3 h. To the solution of iron complex was added a solution of a corresponding enone (0.156 mmol) in CH_3CN (0.1 mL) and the mixture was cooled in ice bath. To the reaction was rapidly added CH_3CO_3H (32 wt% solution in CH_3CO_2H, 50 μL, 0.234 mmol). After being stirred in ice bath for 30 min, the reaction was quenched by adding a mixture of 10 % aq. $Na_2S_2O_3$ and sat. aq. $NaHCO_3$, and diluted with EtOAc. The organic layer was separated and washed with brine, dried over $MgSO_4$ and concentrated at reduced pressure. The residue was purified by silica gel column chromatography and NH-silica gel column chromatography (EtOAc:*n*-Hex=0:100 to 15:85) to afford the corresponding α,β-epoxyketone.


2a

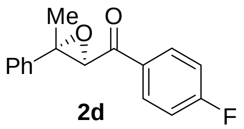
2a (80 % yield, 91 % ee)[8]: white solid; ^{1}H NMR ($CDCl_3$) δ 7.96 (2H, d, *J* = 7.8 Hz), 7.62 (1H, t, *J* = 7.4 Hz), 7.46-7.52 (4H, m), 7.43 (2H, t, *J* = 7.5 Hz), 7.37 (1H, t, *J* = 7.2 Hz), 4.16 (1H, s), 1.64 (3H, s); ^{13}C NMR ($CDCl_3$) δ 193.0, 140.4, 135.6, 134.0, 128.9, 128.8, 128.2, 125.1, 66.9, 62.8, 16.9; IR (neat) 1691, 1598, 1449, 1383, 1229, 957, 848, 751 cm^{-1}; MS (ESI$^+$) m/z (%): 499 ($[2M+Na]^+$, 30), 239 ($[M+H]^+$, 100); $[\alpha]_D^{23}$ -137 ($CHCl_3$, *c* 0.66); lit.[7b] for (2*S*, 3*R*)-isomer: $[\alpha]_D^{25}$ +147 ($CHCl_3$, c 1.3); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=5/95, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 12.4 min, t_r (major) = 29.3 min.



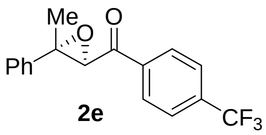
2b (78% yield, 90 % ee): yellowish oil; ^{1}H NMR ($CDCl_3$) δ 7.96 (2H, d, *J* = 8.9 Hz), 7.46-7.50 (2H, m), 7.43 (2H, t, *J* = 7.6 Hz), 7.34-7.39 (1H, m), 6.96 (2H, d, *J* = 8.9 Hz), 4.11 (1H, s), 3.88 (3H, s), 1.62 (3H, s); ^{13}C NMR ($CDCl_3$) δ 191.3, 164.1, 140.6, 130.6, 128.7, 128.1, 125.1, 114.1, 66.8, 62.5, 55.5, 17.0; IR (neat) 1682, 1600, 1261, 1239, 1171, 1025, 956, 857, 765, 699; MS (ESI$^+$) m/z (%): 559 ($[2M+Na]^+$, 30), 269 ($[M+H]^+$, 50), 135 (100); $[\alpha]_D^{23}$ -115 ($CHCl_3$, *c* 0.90); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 18.7 min, t_r (major) = 46.1 min.



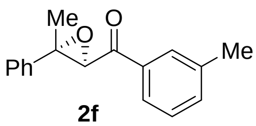
2c (77 % yield, 92 % ee): white solid; ^{1}H NMR ($CDCl_3$) δ 7.82 (2H, d, *J* = 8.2 Hz), 7.49 (2H, d, *J* = 7.2 Hz), 7.43 (2H, t, *J* = 7.4 Hz), 7.37 (1H, t, *J* = 7.2 Hz), 7.29 (2H, d, *J* = 8.1 Hz), 4.14 (1H, s), 2.43 (3H, s), 1.62 (3H, s); ^{13}C NMR ($CDCl_3$) δ 192.5, 145.0, 140.5, 133.1, 129.6, 128.7, 128.3, 128.2, 125.1, 66.8, 62.7, 21.8, 17.0; IR (neat) 1688, 1606, 1382, 1232, 1181, 955, 761, 699 cm^{-1}; MS (ESI^+) m/z (%): 527 ($[2M+Na]^+$, 30), 253 ($[M+H]^+$, 70), 119 (100); $[\alpha]_D^{24}$ -128 ($CHCl_3$, *c* 0.72); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 12.7 min, t_r (major) = 34.6 min.


2d

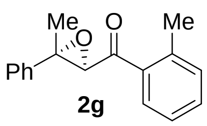
2d (78 % yield, 92 % ee): white solid; ^1H NMR ($CDCl_3$) δ 8.02 (2H, m), 7.46-7.51 (2H, m), 7.43 (2H, t, *J* = 7.4 Hz), 7.35-7.40 (1H, m), 7.17 (2H, t, *J* = 8.6 Hz), 4.11 (1H, s), 1.63 (3H, s); ^{13}C NMR ($CDCl_3$) δ 191.5, 166.2 (d, *J* = 257 Hz), 140.2, 132.1, 131.0 (d, *J* = 9.6 Hz), 128.8, 128.3, 125.1, 116.2 (d, *J* = 22.1 Hz), 66.7, 62.3, 17.0; IR (neat) 1691, 1598, 1506, 1231, 1156, 958, 860, 763, 699 cm^{-1}; MS (ESI^+) m/z (%): 535 ($[2M+Na]^+$, 30), 257 ($[M+H]^+$, 70), 123 (100); $[\alpha]_D^{22}$ -129 ($CHCl_3$, *c* 0.58); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 13.5 min, t_r (major) = 34.0 min.


2e

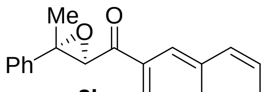
2e (70 % yield, 89 % ee): white solid; ^1H NMR ($CDCl_3$) δ 8.09 (2H, d, *J* = 8.1 Hz), 7.77 (2H, d, *J* = 8.2 Hz), 7.46-7.50 (2H, m), 7.44 (2H, t, *J* = 7.5 Hz), 7.37-7.41 (1H, m), 4.15 (1H, s), 1.64 (3H, s).; ^{13}C NMR ($CDCl_3$) δ 192.3, 140.0, 138.1, 135.1 (q, *J* = 32.9 Hz), 128.8, 128.7, 128.4, 126.0 (q, *J* = 3.7 Hz), 125.1, 123.4 (q, *J* = 273 Hz), 66.8, 63.1, 17.0.; IR (neat) 1699, 1414, 1326, 1227, 1171, 1131, 1107, 1067, 1015, 958, 863, 766, 698 cm^{-1}; MS (ESI^+) m/z (%): 307 ($[M+H]^+$, 20), 173 (100); $[\alpha]_D^{23}$ -115 ($CHCl_3$, *c* 0.78); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 7.2 min, t_r (major) = 10.9 min.


2f

2f (67 % yield, 90 % ee): white solid; ^1H NMR ($CDCl_3$) δ 7.80 (1H, s), 7.74 (1H, d, *J* = 7.6 Hz), 7.47-7.53 (2H, m), 7.41-7.47 (3H, m), 7.37 (2H, t, *J* = 7.7 Hz), 2.42 (3H, s), 1.63 (3H, s); ^{13}C NMR ($CDCl_3$) δ 193.2, 140.5, 138.9, 135.6, 134.8, 128.74, 128.73, 128.6, 128.2, 125.5, 125.1, 66.9, 62.8, 21.3, 17.0; IR (neat) 1691, 1382, 1254, 1169, 934, 759, 699 cm^{-1}; MS (ESI^+) m/z (%): 253 ($[M+H]^+$, 60), 119 (100); $[\alpha]_D^{23}$ -139 ($CHCl_3$, *c* 0.93); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 8.5 min, t_r (major) = 19.6 min.

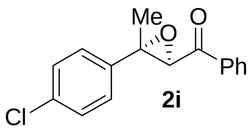

2g

2g (61 % yield, 92 % ee): white solid; ^1H NMR ($CDCl_3$) δ 7.65 (1H, d, *J* = 7.8 Hz), 7.46-7.50 (2H, m), 7.39-7.46 (3H, m), 7.34-7.38 (1H, m), 7.29 (2H, t, *J* = 8.2 Hz), 3.99 (1H, s), 2.59 (3H, s), 1.69 (3H, s); ^{13}C NMR ($CDCl_3$) δ 195.8, 140.5, 139.4, 135.2, 132.4, 132.3, 129.3, 128.7, 128.2, 125.9, 125.1, 67.9, 63.2, 21.2, 16.7; IR (neat) 1691, 1455, 1381, 1221, 956, 752, 731, 698, 656 cm^{-1}; MS (ESI^+) m/z (%): 527 ($[2M+Na]^+$, 10), 253 ($[M+H]^+$, 50), 119 (100); $[\alpha]_D^{23}$ -154 ($CHCl_3$, *c* 0.80); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 8.1 min, t_r (major) = 13.3 min.



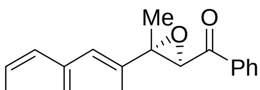
2h

2h (88 % yield, 92 % ee): yellow oil; ^{1}H NMR ($CDCl_3$) δ 8.49 (1H, s), 8.03 (1H, dd, *J* = 8.6 Hz), 7.94 (2H, t, *J* = 7.4 Hz), 7.89 (1H, d, *J* = 8.2 Hz), 7.63 (1H, t, *J* = 7.5 Hz), 7.52-7.59 (3H, m), 7.47 (2H, t, *J* = 7.5 Hz), 7.40 (1H, br-t, *J* = 7.3 Hz), 4.29 (1H, s), 1.67 (3H, s); ^{13}C NMR ($CDCl_3$) δ 192.9, 140.5, 135.9, 132.9, 132.4, 130.4, 129.7, 129.0, 128.9, 128.8, 128.2, 127.9, 127.0, 125.1, 123.5, 66.9, 62.9, 17.1; IR (neat) 2361, 2337, 1685, 1394, 1276, 1181, 942, 762, 699 cm^{-1}; MS (ESI^{+}) m/z (%): 601 ([2M+Na]$^{+}$, 10), 289 ([M+H]$^{+}$, 70), 155 (100); $[\alpha]_D^{25}$ -72.0 ($CHCl_3$, *c* 1.70); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 12.4 min, t_r (major) = 29.3 min.



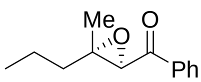
2i

2i (88 % yield, 92 % ee): white solid; ^{1}H NMR ($CDCl_3$) δ 7.95 (2H, d, *J* = 7.3 Hz), 7.63 (1H, td, *J* = 7.5, 0.8 Hz), 7.50 (2H, t, *J* = 7.7 Hz), 7.38-7.45 (4H, m), 4.11 (1H, s), 1.62 (3H, s); ^{13}C NMR ($CDCl_3$) δ 192.6, 139.0, 135.5, 134.2, 134.0, 129.0, 128.2, 126.6, 66.8, 62.3, 16.8; IR (neat) 1692, 1598, 1495, 1450, 1383, 1229, 1093, 1013, 957, 700, 667 cm^{-1}; MS (ESI^{+}) m/z (%): 567 ([2M+Na]$^{+}$, 40), 275 ([M+H+2]$^{+}$, 60), 273 ([M+H]$^{+}$, 95), 105 (100); $[\alpha]_D^{23}$ -184 ($CHCl_3$, *c* 0.82); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 10.1 min, t_r (major) = 30.8 min.



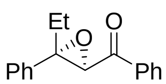
2j

2j (45 % yield, 92 % ee): yellow oil; ^{1}H NMR ($CDCl_3$) δ 7.96-7.99 (3H, m), 7.91 (1H, d, *J* = 8.7 Hz), 7.86-7.90 (2H, m), 7.62 (1H, tt, *J* = 7.4, 1.2 Hz), 7.56 (1H, dd, *J* = 8.6, 1.9 Hz), 7.51-7.55 (2H, m), 7.49 (2H, t, *J* = 7.8 Hz), 4.25 (1H, s), 1.75 (3H, s); ^{13}C NMR ($CDCl_3$) δ 192.9, 137.8, 135.6, 134.0, 133.1, 133.0, 128.9, 128.7, 128.2, 128.1, 127.7, 126.6, 126.5, 124.6, 122.5, 67.0, 62.9, 16.9; IR (neat) 2361, 2337, 1685, 1449, 1386, 1228, 1181, 962, 820, 707, 669 cm^{-1}; MS (ESI^{+}) m/z (%): 289 ([M+H]$^{+}$, 100), 105 (60); $[\alpha]_D^{24}$ -185 ($CHCl_3$, *c* 1.47); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 12.6 min, t_r (major) = 26.8 min.



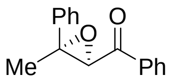
2k

2k (20 % yield, 51 % ee): colorless oil; ^{1}H NMR ($CDCl_3$) δ 7.99 (2H, dd, *J* = 7.4, 1.0 Hz), 7.62 (1H, td, *J* = 7.4, 1.1 Hz), 7.51 (2H, t, *J* = 7.7 Hz), 4.06 (1H, s), 1.79 (2H, m), 1.57 (2H, sext, *J* = 7.6 Hz), 1.23 (3H, s), 1.02 (3H, t, *J* = 7.3 Hz); ^{13}C NMR ($CDCl_3$) δ 194.4, 135.8, 133.7, 128.8, 128.2, 63.9, 63.5, 40.1, 18.4, 16.3, 14.1; IR (neat) 1691, 1598, 1450, 1401, 1385, 1229, 928, 693 cm^{-1}; MS (ESI^{+}) m/z (%): 187 ([M+H]$^{+}$, 20), 105 (100); $[\alpha]_D^{23}$ -7.7 ($CHCl_3$, *c* 0.19); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 7.1 min, t_r (major) = 38.0 min.



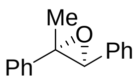
2l

2l (72 % yield, 92 % ee)[9]: white solid, m.p. 49-51 °C; ^{1}H NMR ($CDCl_3$) δ 7.99-8.05 (2H, m), 7.63 (1H, tt, *J* = 7.4, 1.3 Hz), 7.48-7.54 (4H, m), 7.44 (2H, t, *J* = 7.5 Hz), 7.37 (1H, tt, *J* = 7.6, 1.3 Hz), 4.15 (1H, s), 2.13 (1H, sext, J = 7.4 Hz), 1.62 (1H, sext, J = 7.3 Hz), 0.91 (3H, t, J = 7.4 Hz); ^{13}C NMR ($CDCl_3$) δ 193.1, 138.7, 135.6, 133.9, 128.9, 128.7, 128.2, 128.0, 125.8, 67.4, 67.1, 23.7, 9.4; IR (neat) 3062, 2975, 2938, 2879, 1691, 1598, 1450, 1228, 946, 845, 751 cm^{-1}; MS (ESI^+) m/z (%): 253 ($[M+H]^+$, 100), 105 (80); $[\alpha]_D^{23}$ -82 ($CHCl_3$, c 1.3); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 8.8 min, t_r (major) = 16.9 min.



2m

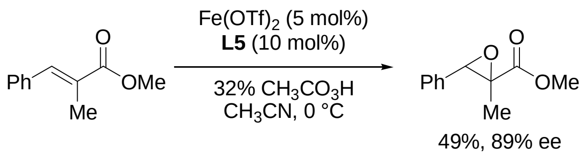
2m (33 % yield, 6 % ee): yellow solid; ^{1}H NMR ($CDCl_3$) δ 7.82 (2H, d, J = 8.4 Hz), 7.54 (1H, t, J = 7.4 Hz), 7.41 (2H, t, J = 7.8 Hz), 7.31 (2H, d, J = 8.2 Hz), 7.13-7.21 (3H, m), 4.34 (1H, s), 1.93 (3H, s); ^{13}C NMR ($CDCl_3$) δ 192.5, 136.6, 135.5, 133.5, 128.6, 128.0, 127.9, 127.8, 126.2, 66.1, 64.5, 24.4; IR (neat) 1684, 1228, 9757, 763, 700, 689 cm^{-1}; MS (ESI^+) m/z (%): 239 ($[M+H]^+$, 100), 261 ($[M+Na]^+$, 50); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 7.9 min, t_r (major) = 33.0 min.



2n

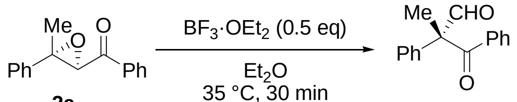
2n (50 % yield, 87 % ee)[10]: colorless oil; ^{1}H NMR ($CDCl_3$) δ 7.44-7.48 (2H, m), 7.35-7.41 (6H, m), 7.30-7.34 (2H, m), 3.98 (1H, s), 1.47 (3H, s); ^{13}C NMR ($CDCl_3$) δ 142.3, 135.9, 128.4, 128.2, 127.7, 127.5, 126.5, 125.1, 67.1, 63.1, 16.7; IR (neat) 1496, 1449, 1381, 1279, 1065, 1027, 763, 741, 698, 605 cm^{-1}; MS (ESI^+) m/z (%): 211 ($[M+H]^+$, 100); $[\alpha]_D^{24}$ -107 (EtOH, c 0.50); lit.[10b] for (R, R)-isomer (95.5% ee): $[\alpha]_D^{25}$ +113.9 (EtOH, c 0.9); chiral HPLC analysis (Chiralcel OD-H, *i*PrOH:*n*-Hex=3/97, flow rate = 1.0 mL/min, λ = 225 nm), t_r (minor) = 14.1 min, t_r (major) = 5.5 min.

4.1. Another example of the iron-catalyzed asymmetric epoxidation.

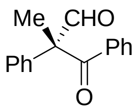


Some of α,β-unsaturated esters can be epoxidized. For example, α-methyl *trans*-cinnamic acid methyl ester was converted to the corresponding epoxide in 49% and 89% ee.

5. Representative procedure for Lewis acid mediated rearrangement and characterization data.

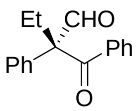


To a solution of **2a** (16 mg, 0.067 mmol, 91% ee) in Et_2O (0.7 mL) was added $BF_3 \cdot OEt_2$ (4 μL, 0.033 mmol). After being stirred at 35 °C for 30 min, the reaction was quenched by adding sat. aq. $NaHCO_3$ and diluted with EtOAc. The organic layer was separated and washed with brine, dried over $MgSO_4$ and concentrated at reduced pressure. The residue was purified by silica gel column chromatography (EtOAc:*n*-Hex=0:100 to 15:85) to afford **3a** (15.3 mg).



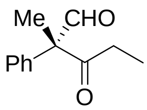
3a

3a (95 % yield, 90 % ee)[8b]: colorless oil; ^{1}H NMR ($CDCl_3$) δ 9.93 (1H, s), 7.62 (2H, d, J = 8.1 Hz), 7.41-7.48 (3H, m), 7.32-7.40 (3H, m), 7.29 (2H, t, J = 7.8 Hz), 1.87 (3H, s); ^{13}C NMR ($CDCl_3$) δ 200.6, 196.6, 136.0, 134.4, 133.1, 129.9, 129.6, 128.4, 128.3, 127.0, 65.5, 17.5; IR (neat) 1729, 1684, 1666, 1597, 1578, 1494, 1447, 1260, 972, 760, 700 cm^{-1}; MS (ESI$^+$) m/z (%): 260 ([M+Na]$^+$, 20), 239 ([M+H]$^+$, 10), 225 (90), 163 (100); $[\alpha]_D^{24}$ +425 ($CHCl_3$, *c* 1.06); lit.[7b] for (*S*)-isomer: $[\alpha]_D^{25}$ -411 ($CHCl_3$, c 1.1); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=5/95, flow rate = 1.0 mL/min, λ = 254 nm), t_r (major) = 10.4 min, t_r (minor) = 12.1 min.



3l

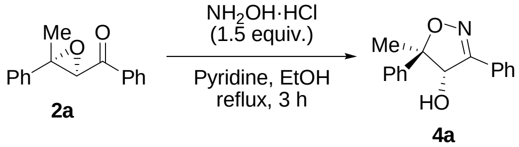
3l (58 % yield, 90 % ee): colorless oil ; ^{1}H NMR ($CDCl_3$) δ 9.97 (1H, d, J = 1.6 Hz), 7.62-7.66 (2H, m), 7.446 (1H, tt, J = 7.4, 1.2 Hz), 7.38-7.43 (2H, m), 7.27-7.37 (5H, m), 2.82 (1H, sext, J = 7.2 Hz), 2.31 (1H, sextd, J = 7.1, 1.6 Hz), 0.84 (3H, t, J = 7.4 Hz); ^{13}C NMR ($CDCl_3$) δ 199.8, 196.3, 135.8, 135.1, 133.1, 129.6, 128.4, 128.2, 127.3, 69.5, 24.6, 9.5; IR (neat) 1727, 1663, 1596, 1578, 1447, 1236, 758, 715, 700 cm^{-1}; MS (ESI$^+$) m/z (%): 275 ([M+Na]$^+$, 10), 253 ([M+H]$^+$, 20); $[\alpha]_D^{23}$ +411 ($CHCl_3$, *c* 0.63); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=5/95, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 7.2 min, t_r (major) = 8.6 min.



3o

3o (35 % yield, 77 % ee): colorless oil ; ^{1}H NMR ($CDCl_3$) δ 9.92 (1H, s), 7.44 (2H, t, J = 7.6 Hz), 7.37 (1H, t, J = 7.3 Hz), 7.24-7.28 (2H, m), 2.36 (2H, q, J = 7.2 Hz), 1.70 (3H, s), 1.01 (3H, t, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$) δ 210.5, 198.9, 135.7, 129.4, 128.3, 127.0, 66.9, 32.2, 16.0, 7.9; IR (neat) 1729, 1702, 1494, 1459, 1446, 1377, 1344, 1187, 1091, 1026, 971, 759, 701 cm^{-1}; MS (ESI$^+$) m/z (%): 191 ([M+H]$^+$, 100); $[\alpha]_D^{23}$ +156 ($CHCl_3$, *c* 0.34); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=5/95, flow rate = 1.0 mL/min, λ = 210 nm), t_r (minor) = 22.9 min, t_r (major) = 9.3 min.

6. Experimental procedure for the synthesis of isoxazoline 4a and characterization data.



To a solution of **2a** (25 mg, 0.10 mmol, 91% ee) in EtOH (0.66 mL) was added $NH_2OH\cdot$ HCl (11 mg, 0.16 mmol) and pyridine (73 mL, 0.90 mmol). After refluxing for 3 h, the reaction was quenched by adding 0.5 M aq. HCl and diluted with Et_2O. The organic layer was separated and dried over $MgSO_4$ and concentrated at reduced pressure. The obtained residue was dissolved in $CHCl_3$, and the solution was filtered through short pad of silica gel. The filtrate was concentrated until white solid was precipitated which was collected to afford the oxime **4a** (17 mg, 99% ee).

4a (65 % yield, 99 % ee): white crystal ; ^{1}H NMR (DMSO-d_6) δ 7.71-7.76 (2H, m), 7.39-7.43 (5H, m), 7.36 (2H, t, J = 7.6 Hz), 7.26 (1H, t, J = 7.2 Hz), 6.34 (1H, d, J = 8.1 Hz), 5.18 (1H, d, J = 8.1), 1.63 (3H, s); ^{13}C NMR (DMSO-d_6) δ 157.7, 144.2, 130.0, 128.9, 128.8, 128.4, 127.3, 126.8, 124.7, 90.2, 82.1, 20.6; IR (neat) 3295, 1568, 1496, 1448, 1350, 1049, 928, 919, 757, 697 cm^{-1}; MS (ESI^+) m/z (%): 254 ($[M+H]^+$, 100); $[\alpha]_D^{23}$ -35 ($CHCl_3$, *c* 0.12); chiral HPLC analysis (Chiralcel AS-H, *i*PrOH:*n*-Hex=10/90, flow rate = 1.0 mL/min, λ = 254 nm), t_r (minor) = 8.8 min, t_r (major) = 11.8 min.

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9. X-ray crystallographic analysis of [Fe(L3)$_2$(CH$_3$CN)(OTf)](OTf)

[Fe(L3)$_2$(CH$_3$CN)(OTf)](OTf): To *rac*-**L3** (30 mg, 0.051 mmol) was added Fe(OTf)$_2$ (0.025 M solution in CH$_3$CN, 1.0 mL) and CH$_3$CN (0.5 mL) at room temperature under nitrogen atmosphere. After being stirred for 3 h at the same temperature, the solution was placed without stirring. Crystals were observed after 1 week, which were collected by filtration, washed with cold ether, and dried under nitrogen stream to yield the title compound as crystal (9.1 mg, 25% yield). MS (ESI$^+$) m/z (%): 612 ([Fe(**L3**)$_2$]$^{2+}$, 20).

The obtained crystal was tested for the epoxidation as follows: A suspension of the obtained crystal (6.1 mg, 4.3 μmol) and **1a** (19 mg, 86.2 μmol) in CH$_3$CN (0.34 mL) was cooled in ice bath. To the suspension, CH$_3$CO$_3$H (32 wt% solution in CH$_3$CO$_2$H, 27 μL, 0.129 mmol) was rapidly added. After being stirred in ice bath for 30 min, the reaction was quenched and purified by the same way as described in general procedure of the epoxidation to afford the α,β-epoxyketone **2a** (12 mg. 58% yield).

Crystal Structure Report for Nish02

$C_{88}H_{56}FeN_4 + 2C_2H_3N + 2CF_3O_3S + 2O$

Report Prepared for:

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September, 2010

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Crystallographic Experimental Section

Data Collection

A well formed rhomb (0.16 x 0.12 x 0.10 mm) was selected under a stereo-microscope while immersed in Fluorolube oil to avoid possible reaction with air. The crystal was removed from the oil using a tapered glass fiber that also served to hold the crystal for data collection. The crystal was mounted and centered on a Bruker SMART APEX system at 100 K. Rotation and still images showed the diffractions to be sharp. Frames separated in reciprocal space were obtained and provided an orientation matrix and initial cell parameters. Final cell parameters were obtained from the full data set.

A "full sphere" data set was obtained which samples approximately all of reciprocal space to a resolution of 0.75 Å using 0.3^{o} steps in ω using 10 second integration times for each frame. Data collection was made at 100 K. Integration of intensities and refinement of cell parameters were done using SAINT [1]. Absorption corrections were applied using SADABS [1] based on redundant diffractions.

Structure solution and refinement

The space group was determined as P1(bar) based on systematic absences and intensity statistics. Direct methods were used to locate the Fe and some C atoms from the E-map. Repeated difference Fourier maps allowed recognition of all expected C and N atoms. In addition two C_2H_3N and CF_3O_3S molecules were present, one of the former bonded to Fe through its N atom and one of the latter bonded to Fe through an O atom. In addition two isolated atoms are present which were assigned as O with occupancies of 0.73 and 0.44; presumably these are H_2O molecules. Following anisotropic refinement of all non-H atoms, ideal H-atom positions were calculated except for the two isolated O atoms. Final refinement was anisotropic for all non-H atoms, and isotropic-riding for H atoms. The C_2H_3N and CF_3O_3S molecules bonded to Fe showed moderate disorder while those not bonded molecules showed severe disorder. No other anomalous bond lengths or thermal parameters were noted. All ORTEP diagrams have been drawn with 50% probability ellipsoids.

Equations of interest:

$R_{int} = \Sigma \mid F_o^2 - \langle F_o^2 \rangle \mid / \Sigma \mid F_o^2 \mid$

$R1 = \Sigma \mid \mid F_o \mid - \mid F_c \mid \mid / \Sigma \mid F_o \mid$

$wR2 = [\Sigma [w (F_o^2 - F_c^2)^2] / \Sigma [w (F_o^2)^2]]^{1/2}$

where: $w = q / \sigma^2 (F_o^2) + (aP)^2 + bP$;

q, a, b, P as defined in [1]

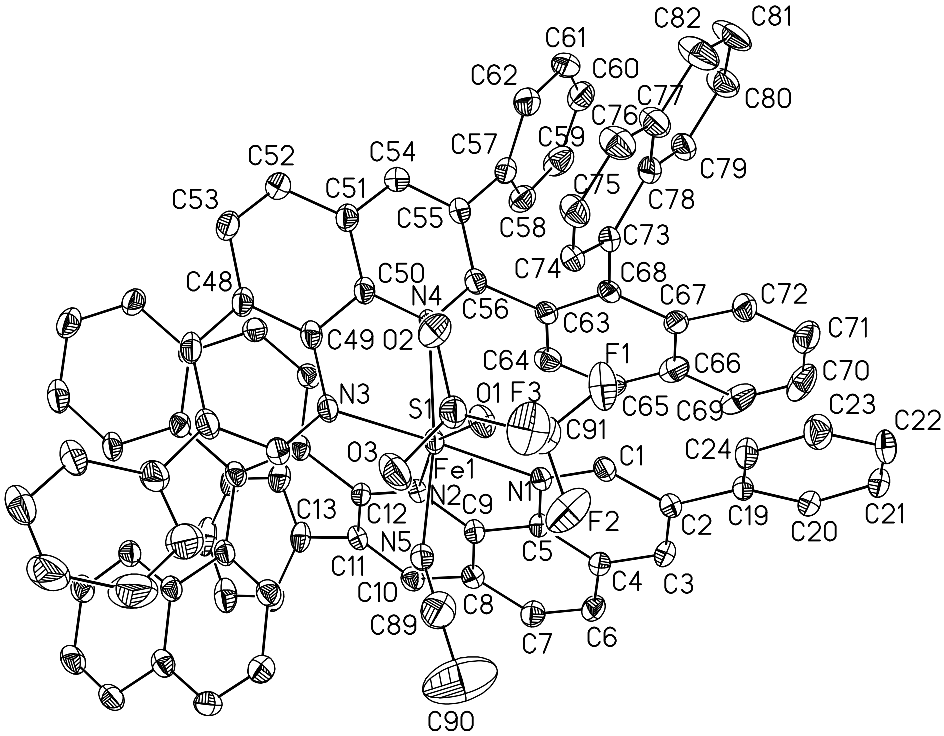
$GooF = S = [\Sigma [w (F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$

n = number of independent reflections;

p = number of parameters refined.

References

[1] All software and sources of scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).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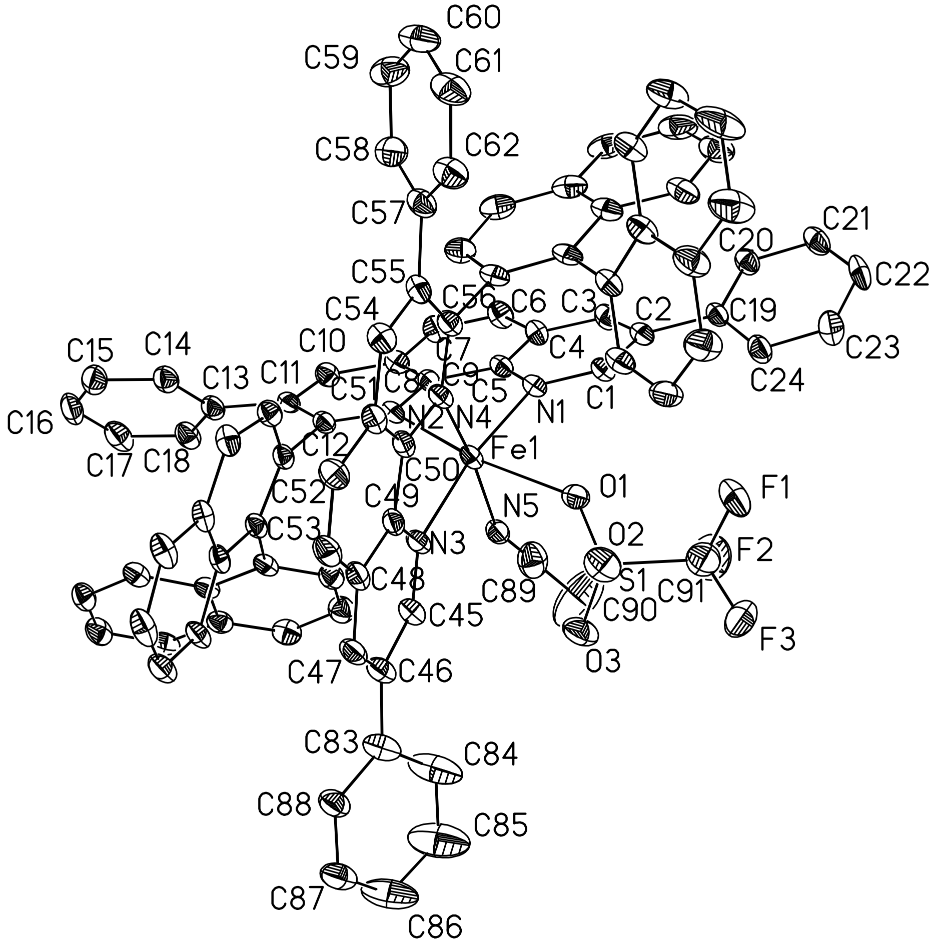
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O1
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C53
N3
O2
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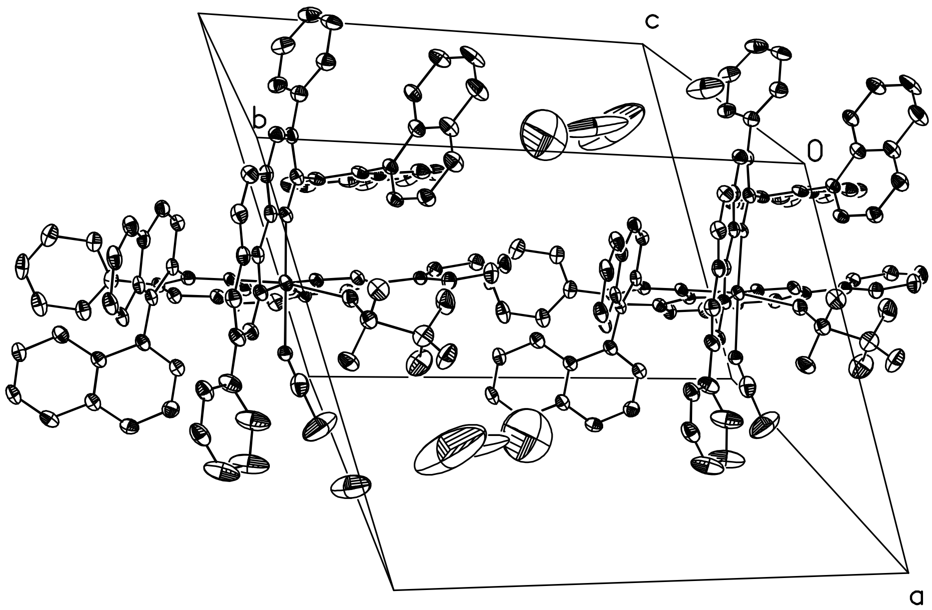
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Table 1. Crystal and structure refinement for Nish02.

Identification Code	Nish02	
Empirical formula	$C_{88}H_{56}FeN_4$ + $2C_2H_3N$ + $2CF_3O_3S$ + 2O	
Formula weight	1621.47	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space Group	P1(bar)	
Unit cell dimensions	*a* = 12.581(2) Å	α = 108.511(3) °
	b = 16.682(3) Å	β = 90.726(3) °
	c = 21.338(4) Å	γ = 112.000(3) °
Volume	3892.9(12) $Å^3$	
Z	2	
Density (calculated)	1.383 Mg/m^3	
Absorption coefficient	0.325 mm^{-1}	
F(000)	1672	
Crystal size, color, habit	0.16 x 0.12 x 0.10 mm, pale yellow, rhomb	
Theta range for data collection	1.76 – 28.35 °	
Index ranges	$-16 \le h \le 16$, $-22 \le k \le 22$, $-28 \le l \le 27$	
Reflections collected	47,447	
Independent reflections	18,748 (R_{int} = 0.0288)	
Reflections with $I > 4\sigma(F_o)$	10,808	
Absorption correction	SADABS based on redundant diffractions	
Max. and min. transmission	1.0, 0.849	
Refinement method	Full-matrix least squares on F^2	
Weighting scheme	$w = q [\sigma^2 (F_o^2) + (aP)^2 + bP]^{-1}$ where:	
	$P = (F_o^2 + 2 F_c^2)/3$, a = 0.0783, b = 0.0, q =1	
Data / restraints / parameters	18748 / 0 / 1058	
Goodness-of-fit on F^2	0.897	
Final R indices [I > 2 sigma(I)]	R1 = 0.0632, wR2 = 0.1515	
R indices (all data)	R1 = 0.1084, wR2 = 0.1684	
Largest diff. peak and hole	1.338, -0.579 e$Å^{-3}$	

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for Nish02. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
C(1)	6278(3)	-1576(2)	7885(2)	21(1)	
C(2)	6604(3)	-1948(2)	8325(2)	21(1)	
C(3)	7134(3)	-1340(2)	8963(2)	23(1)	
C(4)	7311(3)	-410(2)	9153(2)	22(1)	
C(5)	6932(3)	-114(2)	8677(2)	20(1)	
C(6)	7820(3)	236(2)	9811(2)	26(1)	
C(7)	7948(3)	1123(2)	9974(2)	25(1)	
C(8)	7564(3)	1437(2)	9501(2)	23(1)	
C(9)	7046(3)	819(2)	8853(2)	20(1)	
C(10)	7626(3)	2338(2)	9662(2)	23(1)	
C(11)	7155(3)	2590(2)	9210(2)	20(1)	
C(12)	6672(3)	1925(2)	8561(2)	19(1)	
C(13)	7126(3)	3519(2)	9427(2)	25(1)	
C(14)	6090(3)	3614(2)	9345(2)	27(1)	
C(15)	6051(3)	4476(2)	9596(2)	33(1)	
C(16)	7041(4)	5247(2)	9926(2)	37(1)	
C(17)	8079(3)	5160(2)	10012(2)	36(1)	
C(18)	8120(3)	4298(2)	9766(2)	28(1)	
C(19)	6337(3)	-2946(2)	8112(2)	20(1)	
C(20)	6284(3)	-3371(2)	8582(2)	24(1)	
C(21)	5967(3)	-4318(2)	8374(2)	28(1)	
C(22)	5717(3)	-4849(2)	7707(2)	33(1)	
C(23)	5797(3)	-4425(2)	7236(2)	34(1)	
C(24)	6111(3)	-3485(2)	7435(2)	27(1)	
C(25)	6168(3)	2166(2)	8041(1)	20(1)	
C(26)	4946(3)	1787(2)	7888(2)	22(1)	
C(27)	4417(3)	1996(2)	7438(2)	25(1)	
C(28)	5086(3)	2544(2)	7085(2)	23(1)	
C(29)	6318(3)	2918(2)	7229(2)	21(1)	
C(30)	6857(3)	2765(2)	7747(2)	20(1)	
C(31)	4556(3)	2698(2)	6569(2)	28(1)	
C(32)	5214(3)	3182(2)	6198(2)	31(1)	
C(33)	6424(3)	3541(2)	6336(2)	29(1)	
C(34)	6969(3)	3424(2)	6837(2)	24(1)	
C(35)	8142(3)	3226(2)	7954(2)	21(1)	
C(36)	8778(3)	2707(2)	7908(2)	24(1)	
C(37)	9981(3)	3101(2)	8106(2)	29(1)	
C(38)	10559(3)	4041(2)	8352(2)	28(1)	
C(39)	9946(3)	4612(2)	8418(2)	24(1)	
C(40)	8727(3)	4213(2)	8226(2)	21(1)	
C(41)	10528(3)	5586(2)	8684(2)	28(1)	
C(42)	9925(3)	6134(2)	8771(2)	29(1)	
C(43)	8723(3)	5738(2)	8595(2)	27(1)	
C(44)	8131(3)	4803(2)	8332(2)	25(1)	
C(45)	7099(3)	1010(2)	6380(2)	24(1)	
C(46)	7005(3)	1311(2)	5842(2)	26(1)	
C(47)	5910(3)	1157(2)	5591(2)	25(1)	
C(48)	4938(3)	707(2)	5848(2)	23(1)	

C(49)	5121(3)	404(2)	6371(2)	21(1)
C(50)	4147(3)	-106(2)	6626(2)	20(1)
C(51)	3035(3)	-251(2)	6373(2)	23(1)
C(52)	2879(3)	93(2)	5861(2)	26(1)
C(53)	3792(3)	543(2)	5605(2)	26(1)
C(54)	2103(3)	-712(2)	6654(2)	25(1)
C(55)	2277(3)	-1028(2)	7150(2)	23(1)
C(56)	3424(3)	-903(2)	7349(2)	21(1)
C(57)	1270(3)	-1469(2)	7460(2)	26(1)
C(58)	1276(3)	-1123(2)	8147(2)	30(1)
C(59)	303(3)	-1503(3)	8423(2)	37(1)
C(60)	-671(3)	-2232(3)	8023(2)	38(1)
C(61)	-689(3)	-2571(3)	7338(2)	36(1)
C(62)	275(3)	-2185(2)	7055(2)	32(1)
C(63)	3624(3)	-1331(2)	7833(2)	22(1)
C(64)	4057(3)	-765(2)	8507(2)	26(1)
C(65)	4209(3)	-1144(2)	8967(2)	30(1)
C(66)	3962(3)	-2090(3)	8777(2)	30(1)
C(67)	3523(3)	-2667(2)	8103(2)	25(1)
C(68)	3331(3)	-2270(2)	7626(2)	22(1)
C(69)	4168(3)	-2485(3)	9243(2)	38(1)
C(70)	3925(3)	-3405(3)	9046(2)	43(1)
C(71)	3512(3)	-3966(3)	8381(2)	38(1)
C(72)	3333(3)	-3607(2)	7918(2)	31(1)
C(73)	2894(3)	-2874(2)	6910(2)	22(1)
C(74)	3583(3)	-2721(2)	6431(2)	25(1)
C(75)	3260(3)	-3307(2)	5758(2)	30(1)
C(76)	2233(3)	-4062(2)	5568(2)	35(1)
C(77)	1482(3)	-4242(2)	6032(2)	33(1)
C(78)	1786(3)	-3643(2)	6712(2)	26(1)
C(79)	994(3)	-3832(2)	7160(2)	28(1)
C(80)	-33(3)	-4595(2)	6956(2)	36(1)
C(81)	-314(3)	-5203(3)	6287(2)	47(1)
C(82)	416(3)	-5029(3)	5838(2)	44(1)
C(83)	8041(3)	1725(2)	5550(2)	33(1)
C(84)	9004(3)	1507(3)	5588(2)	60(1)
C(85)	9942(4)	1844(4)	5270(2)	74(2)
C(86)	9919(4)	2399(3)	4904(2)	61(1)
C(87)	8968(3)	2584(2)	4844(2)	36(1)
C(88)	8034(3)	2248(2)	5158(2)	31(1)
C(89)	9000(4)	333(3)	7441(2)	41(1)
C(90)	10087(5)	183(5)	7379(3)	103(2)
C(91)	6887(3)	-2414(3)	5791(2)	39(1)
C(92)	1514(7)	2463(15)	6764(3)	227(11)
C(93)	1813(11)	3630(8)	6786(5)	242(8)
C(94)	1136(4)	1644(3)	9398(2)	58(1)
F(1)	6022(2)	-3091(1)	5887(1)	48(1)
F(2)	7802(2)	-2209(2)	6227(1)	60(1)
F(3)	7135(2)	-2711(2)	5179(1)	51(1)
F(4)	357(3)	832(3)	9108(2)	161(2)
F(5)	699(3)	2167(4)	9258(3)	169(2)
F(6)	1148(3)	1819(4)	10023(2)	144(2)
Fe(1)	6250(1)	-18(1)	7383(1)	20(1)
N(1)	6436(2)	-701(2)	8045(1)	19(1)
N(2)	6622(2)	1069(2)	8386(1)	19(1)
N(3)	6202(2)	571(2)	6637(1)	21(1)
N(4)	4344(2)	-431(2)	7111(1)	20(1)

N(5)	8158(2)	430(2)	7463(1)	23(1)	
N(6)	1521(14)	2031(14)	6880(9)	348(11)	
O(1)	6290(2)	-1202(1)	6598(1)	27(1)	
O(2)	5542(2)	-1746(2)	5431(1)	36(1)	
O(3)	7587(2)	-739(2)	5817(1)	39(1)	
O(4)	2306(4)	1594(5)	8460(2)	196(3)	
O(5)	2831(4)	1165(3)	9310(2)	107(2)	
O(6)	3217(3)	2695(2)	9513(3)	180(3)	
O(7)	3163(5)	3627(4)	8278(3)	95(3)	0.73
O(8)	9971(7)	9968(7)	5681(5)	103(5)	0.44
S(1)	6542(1)	-1409(1)	5917(1)	28(1)	
S(2)	2522(1)	1790(1)	9141(1)	50(1)	

Table 3. Bond lengths [Å] and angles [°] for Nish02.

C(1)-N(1)	1.324(4)	C(45)-N(3)	1.328(4)
C(1)-C(2)	1.407(4)	C(45)-C(46)	1.409(4)
C(2)-C(3)	1.385(4)	C(46)-C(47)	1.371(4)
C(2)-C(19)	1.480(4)	C(46)-C(83)	1.478(4)
C(3)-C(4)	1.401(4)	C(47)-C(48)	1.397(4)
C(4)-C(5)	1.409(4)	C(48)-C(49)	1.411(4)
C(4)-C(6)	1.429(4)	C(48)-C(53)	1.420(4)
C(5)-N(1)	1.359(4)	C(49)-N(3)	1.358(4)
C(5)-C(9)	1.429(4)	C(49)-C(50)	1.432(4)
C(6)-C(7)	1.352(4)	C(50)-N(4)	1.370(4)
C(7)-C(8)	1.430(4)	C(50)-C(51)	1.397(4)
C(8)-C(10)	1.401(4)	C(51)-C(54)	1.400(4)
C(8)-C(9)	1.402(4)	C(51)-C(52)	1.430(4)
C(9)-N(2)	1.367(4)	C(52)-C(53)	1.345(4)
C(10)-C(11)	1.375(4)	C(54)-C(55)	1.372(4)
C(11)-C(12)	1.419(4)	C(55)-C(56)	1.418(4)
C(11)-C(13)	1.485(4)	C(55)-C(57)	1.488(4)
C(12)-N(2)	1.332(4)	C(56)-N(4)	1.343(4)
C(12)-C(25)	1.502(4)	C(56)-C(63)	1.495(4)
C(13)-C(14)	1.387(5)	C(57)-C(62)	1.392(4)
C(13)-C(18)	1.392(4)	C(57)-C(58)	1.395(5)
C(14)-C(15)	1.386(4)	C(58)-C(59)	1.385(5)
C(15)-C(16)	1.380(5)	C(59)-C(60)	1.381(5)
C(16)-C(17)	1.383(5)	C(60)-C(61)	1.386(5)
C(17)-C(18)	1.386(5)	C(61)-C(62)	1.389(5)
C(19)-C(20)	1.390(4)	C(63)-C(68)	1.382(4)
C(19)-C(24)	1.397(4)	C(63)-C(64)	1.410(4)
C(20)-C(21)	1.389(4)	C(64)-C(65)	1.371(4)
C(21)-C(22)	1.375(5)	C(65)-C(66)	1.405(5)
C(22)-C(23)	1.385(5)	C(66)-C(67)	1.415(5)
C(23)-C(24)	1.379(4)	C(66)-C(69)	1.424(4)
C(25)-C(30)	1.381(4)	C(67)-C(72)	1.412(4)
C(25)-C(26)	1.413(4)	C(67)-C(68)	1.441(4)
C(26)-C(27)	1.361(4)	C(68)-C(73)	1.496(4)
C(27)-C(28)	1.411(4)	C(69)-C(70)	1.365(5)
C(28)-C(31)	1.418(4)	C(70)-C(71)	1.391(5)
C(28)-C(29)	1.424(4)	C(71)-C(72)	1.365(4)
C(29)-C(34)	1.424(4)	C(73)-C(74)	1.372(4)
C(29)-C(30)	1.426(4)	C(73)-C(78)	1.438(4)
C(30)-C(35)	1.495(4)	C(74)-C(75)	1.406(4)
C(31)-C(32)	1.373(5)	C(75)-C(76)	1.365(5)
C(32)-C(33)	1.399(5)	C(76)-C(77)	1.402(5)
C(33)-C(34)	1.367(4)	C(77)-C(82)	1.420(5)
C(35)-C(36)	1.367(4)	C(77)-C(78)	1.425(5)
C(35)-C(40)	1.436(4)	C(78)-C(79)	1.409(4)
C(36)-C(37)	1.401(4)	C(79)-C(80)	1.371(4)
C(37)-C(38)	1.370(5)	C(80)-C(81)	1.411(5)
C(38)-C(39)	1.411(4)	C(81)-C(82)	1.358(5)
C(39)-C(40)	1.418(4)	C(83)-C(88)	1.389(5)
C(39)-C(41)	1.418(4)	C(83)-C(84)	1.398(5)
C(40)-C(44)	1.413(4)	C(84)-C(85)	1.390(6)
C(41)-C(42)	1.363(5)	C(85)-C(86)	1.395(6)
C(42)-C(43)	1.395(4)	C(86)-C(87)	1.358(6)
C(43)-C(44)	1.366(4)	C(87)-C(88)	1.380(5)

C(89)-N(5)	1.129(4)	Fe(1)-N(3)	2.132(2)
C(89)-C(90)	1.480(6)	Fe(1)-N(1)	2.136(2)
C(91)-F(1)	1.317(4)	Fe(1)-O(1)	2.166(2)
C(91)-F(3)	1.328(4)	Fe(1)-N(5)	2.217(3)
C(91)-F(2)	1.336(4)	Fe(1)-N(2)	2.227(2)
C(91)-S(1)	1.826(4)	Fe(1)-N(4)	2.245(3)
C(92)-N(6)	0.84(3)	O(1)-S(1)	1.451(2)
C(92)-C(93)	1.82(2)	O(2)-S(1)	1.429(2)
C(94)-F(6)	1.270(5)	O(3)-S(1)	1.442(2)
C(94)-F(4)	1.279(5)	O(4)-S(2)	1.384(4)
C(94)-F(5)	1.294(5)	O(5)-S(2)	1.381(4)
C(94)-S(2)	1.787(5)	O(6)-S(2)	1.381(4)

N(1)-C(1)-C(2)	124.4(3)	C(31)-C(28)-C(29)	119.3(3)
C(3)-C(2)-C(1)	116.6(3)	C(28)-C(29)-C(34)	118.1(3)
C(3)-C(2)-C(19)	122.8(3)	C(28)-C(29)-C(30)	119.6(3)
C(1)-C(2)-C(19)	120.6(3)	C(34)-C(29)-C(30)	122.3(3)
C(2)-C(3)-C(4)	120.8(3)	C(25)-C(30)-C(29)	118.5(3)
C(3)-C(4)-C(5)	118.0(3)	C(25)-C(30)-C(35)	120.8(3)
C(3)-C(4)-C(6)	122.8(3)	C(29)-C(30)-C(35)	120.7(3)
C(5)-C(4)-C(6)	119.1(3)	C(32)-C(31)-C(28)	120.9(3)
N(1)-C(5)-C(4)	121.6(3)	C(31)-C(32)-C(33)	119.7(3)
N(1)-C(5)-C(9)	118.1(3)	C(34)-C(33)-C(32)	121.2(3)
C(4)-C(5)-C(9)	120.3(3)	C(33)-C(34)-C(29)	120.8(3)
C(7)-C(6)-C(4)	120.6(3)	C(36)-C(35)-C(40)	118.7(3)
C(6)-C(7)-C(8)	121.2(3)	C(36)-C(35)-C(30)	120.0(3)
C(10)-C(8)-C(9)	117.4(3)	C(40)-C(35)-C(30)	121.3(3)
C(10)-C(8)-C(7)	122.8(3)	C(35)-C(36)-C(37)	122.5(3)
C(9)-C(8)-C(7)	119.7(3)	C(38)-C(37)-C(36)	119.7(3)
N(2)-C(9)-C(8)	122.4(3)	C(37)-C(38)-C(39)	120.3(3)
N(2)-C(9)-C(5)	118.6(3)	C(38)-C(39)-C(40)	120.0(3)
C(8)-C(9)-C(5)	119.0(3)	C(38)-C(39)-C(41)	121.0(3)
C(11)-C(10)-C(8)	121.0(3)	C(40)-C(39)-C(41)	119.0(3)
C(10)-C(11)-C(12)	117.6(3)	C(44)-C(40)-C(39)	118.5(3)
C(10)-C(11)-C(13)	119.1(3)	C(44)-C(40)-C(35)	122.6(3)
C(12)-C(11)-C(13)	123.2(3)	C(39)-C(40)-C(35)	118.8(3)
N(2)-C(12)-C(11)	122.9(3)	C(42)-C(41)-C(39)	120.8(3)
N(2)-C(12)-C(25)	117.0(3)	C(41)-C(42)-C(43)	120.0(3)
C(11)-C(12)-C(25)	120.1(3)	C(44)-C(43)-C(42)	121.0(3)
C(14)-C(13)-C(18)	119.1(3)	C(43)-C(44)-C(40)	120.7(3)
C(14)-C(13)-C(11)	120.4(3)	N(3)-C(45)-C(46)	124.2(3)
C(18)-C(13)-C(11)	120.3(3)	C(47)-C(46)-C(45)	116.7(3)
C(15)-C(14)-C(13)	120.1(3)	C(47)-C(46)-C(83)	122.5(3)
C(16)-C(15)-C(14)	120.4(3)	C(45)-C(46)-C(83)	120.7(3)
C(15)-C(16)-C(17)	119.9(3)	C(46)-C(47)-C(48)	121.3(3)
C(16)-C(17)-C(18)	119.8(3)	C(47)-C(48)-C(49)	117.6(3)
C(17)-C(18)-C(13)	120.6(3)	C(47)-C(48)-C(53)	123.0(3)
C(20)-C(19)-C(24)	118.6(3)	C(49)-C(48)-C(53)	119.4(3)
C(20)-C(19)-C(2)	120.7(3)	N(3)-C(49)-C(48)	121.8(3)
C(24)-C(19)-C(2)	120.7(3)	N(3)-C(49)-C(50)	118.5(3)
C(21)-C(20)-C(19)	119.9(3)	C(48)-C(49)-C(50)	119.7(3)
C(22)-C(21)-C(20)	121.2(3)	N(4)-C(50)-C(51)	122.4(3)
C(21)-C(22)-C(23)	119.0(3)	N(4)-C(50)-C(49)	118.6(3)
C(24)-C(23)-C(22)	120.4(3)	C(51)-C(50)-C(49)	119.0(3)
C(23)-C(24)-C(19)	120.7(3)	C(50)-C(51)-C(54)	117.8(3)
C(30)-C(25)-C(26)	121.1(3)	C(50)-C(51)-C(52)	120.1(3)
C(30)-C(25)-C(12)	121.9(3)	C(54)-C(51)-C(52)	122.0(3)
C(26)-C(25)-C(12)	116.9(3)	C(53)-C(52)-C(51)	120.8(3)
C(27)-C(26)-C(25)	120.7(3)	C(52)-C(53)-C(48)	121.0(3)
C(26)-C(27)-C(28)	120.2(3)	C(55)-C(54)-C(51)	120.9(3)
C(27)-C(28)-C(31)	121.2(3)	C(54)-C(55)-C(56)	117.7(3)
C(27)-C(28)-C(29)	119.4(3)	C(54)-C(55)-C(57)	119.1(3)

C(56)-C(55)-C(57)	123.2(3)	F(1)-C(91)-F(3)	109.1(3)
N(4)-C(56)-C(55)	122.8(3)	F(1)-C(91)-F(2)	106.5(3)
N(4)-C(56)-C(63)	118.2(3)	F(3)-C(91)-F(2)	108.0(3)
C(55)-C(56)-C(63)	119.0(3)	F(1)-C(91)-S(1)	111.4(3)
C(62)-C(57)-C(58)	119.4(3)	F(3)-C(91)-S(1)	110.9(3)
C(62)-C(57)-C(55)	119.8(3)	F(2)-C(91)-S(1)	110.8(3)
C(58)-C(57)-C(55)	120.6(3)	N(6)-C(92)-C(93)	160.4(19)
C(59)-C(58)-C(57)	120.0(3)	F(6)-C(94)-F(4)	106.9(5)
C(60)-C(59)-C(58)	120.5(3)	F(6)-C(94)-F(5)	105.2(5)
C(59)-C(60)-C(61)	119.9(3)	F(4)-C(94)-F(5)	103.3(5)
C(60)-C(61)-C(62)	120.1(3)	F(6)-C(94)-S(2)	113.6(3)
C(61)-C(62)-C(57)	120.1(3)	F(4)-C(94)-S(2)	113.4(4)
C(68)-C(63)-C(64)	121.2(3)	F(5)-C(94)-S(2)	113.6(4)
C(68)-C(63)-C(56)	120.1(3)	N(3)-Fe(1)-N(1)	171.91(10)
C(64)-C(63)-C(56)	118.6(3)	N(3)-Fe(1)-O(1)	87.68(9)
C(65)-C(64)-C(63)	119.9(3)	N(1)-Fe(1)-O(1)	85.36(9)
C(64)-C(65)-C(66)	121.1(3)	N(3)-Fe(1)-N(5)	92.12(10)
C(65)-C(66)-C(67)	119.6(3)	N(1)-Fe(1)-N(5)	82.84(9)
C(65)-C(66)-C(69)	121.7(3)	O(1)-Fe(1)-N(5)	81.89(9)
C(67)-C(66)-C(69)	118.7(3)	N(3)-Fe(1)-N(2)	108.77(9)
C(72)-C(67)-C(66)	118.6(3)	N(1)-Fe(1)-N(2)	77.22(9)
C(72)-C(67)-C(68)	122.3(3)	O(1)-Fe(1)-N(2)	159.52(9)
C(66)-C(67)-C(68)	119.1(3)	N(5)-Fe(1)-N(2)	85.29(9)
C(63)-C(68)-C(67)	119.0(3)	N(3)-Fe(1)-N(4)	77.36(9)
C(63)-C(68)-C(73)	121.6(3)	N(1)-Fe(1)-N(4)	107.18(9)
C(67)-C(68)-C(73)	119.2(3)	O(1)-Fe(1)-N(4)	93.84(9)
C(70)-C(69)-C(66)	120.7(4)	N(5)-Fe(1)-N(4)	168.85(9)
C(69)-C(70)-C(71)	120.4(3)	N(2)-Fe(1)-N(4)	101.48(9)
C(72)-C(71)-C(70)	120.5(4)	C(1)-N(1)-C(5)	118.6(3)
C(71)-C(72)-C(67)	121.1(4)	C(1)-N(1)-Fe(1)	127.4(2)
C(74)-C(73)-C(78)	118.8(3)	C(5)-N(1)-Fe(1)	113.26(19)
C(74)-C(73)-C(68)	119.6(3)	C(12)-N(2)-C(9)	118.6(3)
C(78)-C(73)-C(68)	121.5(3)	C(12)-N(2)-Fe(1)	130.7(2)
C(73)-C(74)-C(75)	122.3(3)	C(9)-N(2)-Fe(1)	109.65(19)
C(76)-C(75)-C(74)	119.6(3)	C(45)-N(3)-C(49)	118.3(3)
C(75)-C(76)-C(77)	120.6(3)	C(45)-N(3)-Fe(1)	126.7(2)
C(76)-C(77)-C(82)	120.9(3)	C(49)-N(3)-Fe(1)	114.84(19)
C(76)-C(77)-C(78)	120.4(3)	C(56)-N(4)-C(50)	118.1(3)
C(82)-C(77)-C(78)	118.8(3)	C(56)-N(4)-Fe(1)	131.3(2)
C(79)-C(78)-C(77)	118.7(3)	C(50)-N(4)-Fe(1)	110.65(19)
C(79)-C(78)-C(73)	123.1(3)	C(89)-N(5)-Fe(1)	155.7(3)
C(77)-C(78)-C(73)	118.2(3)	S(1)-O(1)-Fe(1)	136.65(13)
C(80)-C(79)-C(78)	121.1(3)	O(2)-S(1)-O(3)	116.29(15)
C(79)-C(80)-C(81)	120.0(3)	O(2)-S(1)-O(1)	113.14(15)
C(82)-C(81)-C(80)	120.5(3)	O(3)-S(1)-O(1)	115.37(14)
C(81)-C(82)-C(77)	120.9(3)	O(2)-S(1)-C(91)	103.77(17)
C(88)-C(83)-C(84)	117.5(3)	O(3)-S(1)-C(91)	103.51(17)
C(88)-C(83)-C(46)	121.1(3)	O(1)-S(1)-C(91)	102.35(15)
C(84)-C(83)-C(46)	120.9(3)	O(6)-S(2)-O(5)	113.0(3)
C(85)-C(84)-C(83)	120.8(4)	O(6)-S(2)-O(4)	115.6(4)
C(84)-C(85)-C(86)	119.7(4)	O(5)-S(2)-O(4)	114.2(4)
C(87)-C(86)-C(85)	119.8(4)	O(6)-S(2)-C(94)	103.6(3)
C(86)-C(87)-C(88)	120.5(4)	O(5)-S(2)-C(94)	105.5(2)
C(87)-C(88)-C(83)	121.6(4)	O(4)-S(2)-C(94)	103.2(2)
N(5)-C(89)-C(90)	177.4(4)		

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for Nish02. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	21(2)	17(2)	22(2)	8(1)	2(1)	4(1)
C(2)	19(2)	19(2)	25(2)	11(1)	3(1)	6(1)
C(3)	25(2)	23(2)	27(2)	14(1)	2(1)	12(1)
C(4)	22(2)	19(2)	24(2)	9(1)	0(1)	7(1)
C(5)	20(2)	19(2)	22(2)	11(1)	3(1)	7(1)
C(6)	30(2)	27(2)	23(2)	11(1)	-3(1)	10(2)
C(7)	30(2)	22(2)	22(2)	6(1)	-1(1)	10(1)
C(8)	21(2)	22(2)	24(2)	10(1)	3(1)	7(1)
C(9)	21(2)	18(2)	22(2)	9(1)	4(1)	7(1)
C(10)	25(2)	19(2)	20(2)	6(1)	1(1)	6(1)
C(11)	22(2)	16(2)	21(2)	8(1)	6(1)	5(1)
C(12)	20(2)	19(2)	23(2)	11(1)	4(1)	8(1)
C(13)	39(2)	22(2)	19(2)	10(1)	7(1)	14(2)
C(14)	38(2)	25(2)	21(2)	10(1)	5(1)	14(2)
C(15)	52(2)	36(2)	24(2)	15(2)	11(2)	28(2)
C(16)	69(3)	24(2)	28(2)	12(2)	13(2)	26(2)
C(17)	57(3)	22(2)	23(2)	8(2)	2(2)	10(2)
C(18)	39(2)	23(2)	22(2)	9(1)	1(2)	10(2)
C(19)	18(2)	18(2)	30(2)	12(1)	4(1)	7(1)
C(20)	22(2)	23(2)	30(2)	13(1)	2(1)	10(1)
C(21)	28(2)	25(2)	38(2)	19(2)	3(2)	11(2)
C(22)	38(2)	16(2)	46(2)	12(2)	1(2)	11(2)
C(23)	42(2)	24(2)	34(2)	8(2)	1(2)	14(2)
C(24)	31(2)	22(2)	33(2)	14(2)	5(2)	12(2)
C(25)	26(2)	14(1)	18(2)	4(1)	2(1)	7(1)
C(26)	24(2)	18(2)	23(2)	7(1)	2(1)	6(1)
C(27)	23(2)	19(2)	31(2)	8(1)	-1(1)	8(1)
C(28)	28(2)	16(2)	26(2)	7(1)	-2(1)	10(1)
C(29)	26(2)	12(1)	21(2)	4(1)	-2(1)	6(1)
C(30)	24(2)	13(1)	20(2)	5(1)	2(1)	5(1)
C(31)	33(2)	20(2)	30(2)	10(2)	-6(2)	9(2)
C(32)	44(2)	19(2)	26(2)	7(1)	-8(2)	11(2)
C(33)	42(2)	19(2)	24(2)	10(1)	4(2)	8(2)
C(34)	32(2)	15(2)	23(2)	9(1)	1(1)	5(1)
C(35)	25(2)	18(2)	19(2)	10(1)	3(1)	5(1)
C(36)	25(2)	20(2)	23(2)	7(1)	2(1)	6(1)
C(37)	27(2)	30(2)	33(2)	14(2)	5(2)	13(2)
C(38)	21(2)	32(2)	30(2)	13(2)	1(1)	7(2)
C(39)	28(2)	21(2)	18(2)	7(1)	3(1)	5(1)
C(40)	24(2)	21(2)	18(2)	9(1)	4(1)	6(1)
C(41)	25(2)	28(2)	24(2)	11(2)	2(1)	1(2)
C(42)	33(2)	16(2)	29(2)	5(1)	1(2)	2(1)
C(43)	31(2)	18(2)	28(2)	7(1)	1(1)	7(1)
C(44)	27(2)	23(2)	23(2)	11(1)	2(1)	7(1)
C(45)	24(2)	20(2)	25(2)	11(1)	1(1)	6(1)
C(46)	28(2)	21(2)	25(2)	11(1)	3(1)	4(1)
C(47)	31(2)	22(2)	26(2)	15(1)	6(1)	10(1)
C(48)	30(2)	17(2)	22(2)	8(1)	2(1)	10(1)

C(49)	28(2)	14(2)	21(2)	6(1)	4(1)	8(1)
C(50)	27(2)	15(2)	20(2)	6(1)	2(1)	8(1)
C(51)	28(2)	19(2)	24(2)	9(1)	2(1)	10(1)
C(52)	27(2)	25(2)	26(2)	12(1)	0(1)	9(1)
C(53)	32(2)	25(2)	25(2)	13(1)	1(1)	12(2)
C(54)	22(2)	23(2)	29(2)	10(1)	0(1)	7(1)
C(55)	24(2)	17(2)	27(2)	9(1)	2(1)	7(1)
C(56)	25(2)	17(2)	20(2)	7(1)	2(1)	5(1)
C(57)	25(2)	26(2)	32(2)	17(2)	4(1)	10(1)
C(58)	26(2)	38(2)	35(2)	19(2)	6(2)	16(2)
C(59)	33(2)	56(3)	37(2)	27(2)	11(2)	23(2)
C(60)	24(2)	53(2)	53(2)	38(2)	12(2)	15(2)
C(61)	19(2)	38(2)	50(2)	23(2)	1(2)	4(2)
C(62)	28(2)	34(2)	36(2)	18(2)	4(2)	10(2)
C(63)	17(2)	25(2)	24(2)	14(1)	4(1)	4(1)
C(64)	22(2)	29(2)	25(2)	10(2)	4(1)	6(1)
C(65)	22(2)	44(2)	20(2)	13(2)	3(1)	11(2)
C(66)	23(2)	47(2)	27(2)	22(2)	9(1)	15(2)
C(67)	17(2)	32(2)	30(2)	20(2)	4(1)	7(1)
C(68)	15(2)	24(2)	26(2)	15(1)	4(1)	2(1)
C(69)	34(2)	72(3)	27(2)	30(2)	14(2)	29(2)
C(70)	41(2)	71(3)	52(3)	49(2)	26(2)	37(2)
C(71)	29(2)	47(2)	56(3)	37(2)	15(2)	19(2)
C(72)	23(2)	33(2)	44(2)	26(2)	7(2)	9(2)
C(73)	22(2)	19(2)	26(2)	12(1)	1(1)	5(1)
C(74)	20(2)	22(2)	31(2)	14(1)	3(1)	4(1)
C(75)	31(2)	28(2)	27(2)	13(2)	9(2)	6(2)
C(76)	38(2)	33(2)	23(2)	6(2)	3(2)	5(2)
C(77)	32(2)	28(2)	29(2)	11(2)	1(2)	1(2)
C(78)	27(2)	23(2)	26(2)	14(1)	1(1)	6(1)
C(79)	28(2)	24(2)	27(2)	13(2)	1(1)	3(1)
C(80)	30(2)	36(2)	33(2)	16(2)	3(2)	-1(2)
C(81)	35(2)	38(2)	39(2)	15(2)	-3(2)	-16(2)
C(82)	42(2)	37(2)	27(2)	6(2)	1(2)	-7(2)
C(83)	29(2)	38(2)	31(2)	20(2)	3(2)	4(2)
C(84)	34(2)	93(4)	66(3)	60(3)	8(2)	12(2)
C(85)	30(2)	126(5)	75(3)	66(4)	7(2)	16(3)
C(86)	32(2)	91(4)	57(3)	48(3)	7(2)	2(2)
C(87)	44(2)	28(2)	26(2)	11(2)	7(2)	2(2)
C(88)	37(2)	23(2)	29(2)	11(2)	6(2)	5(2)
C(89)	49(3)	33(2)	36(2)	6(2)	-2(2)	16(2)
C(90)	79(4)	146(6)	87(4)	4(4)	6(3)	79(4)
C(91)	43(2)	42(2)	35(2)	16(2)	6(2)	19(2)
C(92)	56(5)	560(30)	22(3)	40(8)	9(3)	122(10)
C(93)	273(17)	252(14)	102(8)	-15(9)	43(8)	62(12)
C(94)	51(3)	62(3)	61(3)	36(3)	-3(2)	10(2)
F(1)	65(2)	31(1)	58(2)	25(1)	14(1)	19(1)
F(2)	63(2)	74(2)	62(2)	29(1)	2(1)	44(2)
F(3)	70(2)	52(2)	44(1)	16(1)	24(1)	40(1)
F(4)	99(3)	113(3)	152(4)	-11(3)	70(3)	-39(2)
F(5)	83(3)	229(5)	307(6)	200(5)	46(3)	93(3)
F(6)	117(3)	311(6)	53(2)	55(3)	27(2)	143(4)
Fe(1)	24(1)	16(1)	19(1)	9(1)	1(1)	6(1)
N(1)	18(1)	18(1)	21(1)	10(1)	2(1)	5(1)
N(2)	20(1)	15(1)	23(1)	10(1)	3(1)	4(1)
N(3)	24(1)	18(1)	24(1)	11(1)	5(1)	6(1)
N(4)	22(1)	15(1)	20(1)	8(1)	2(1)	4(1)

N(5)	23(2)	21(1)	28(2)	12(1)	5(1)	9(1)
N(6)	333(18)	520(20)	500(30)	340(20)	266(18)	348(19)
O(1)	32(1)	22(1)	23(1)	9(1)	8(1)	8(1)
O(2)	39(2)	41(2)	30(1)	17(1)	-2(1)	15(1)
O(3)	34(1)	33(1)	46(2)	21(1)	11(1)	4(1)
O(4)	61(3)	424(10)	101(4)	152(5)	25(3)	43(4)
O(5)	128(4)	93(3)	169(4)	90(3)	76(3)	79(3)
O(6)	47(2)	33(2)	385(9)	-15(3)	27(4)	12(2)
O(7)	92(4)	104(5)	129(5)	67(4)	32(4)	61(4)
O(8)	55(6)	141(10)	134(9)	65(7)	35(5)	48(6)
S(1)	31(1)	26(1)	28(1)	13(1)	5(1)	10(1)
S(2)	47(1)	40(1)	57(1)	25(1)	4(1)	5(1)

Table 5. Hydrogen coordinates [x 10^4] and isotropic displacement parameters [$Å^2$ x 10^3] for Nish02.

	x	y	z	U(eq)
H(1)	5921	-1981	7444	25
H(3)	7381	-1557	9275	28
H(6)	8070	41	10136	32
H(7)	8300	1545	10412	30
H(10)	7999	2780	10091	27
H(14)	5405	3088	9115	32
H(15)	5338	4537	9540	40
H(16)	7010	5836	10095	45
H(17)	8763	5690	10238	43
H(18)	8833	4239	9829	34
H(20)	6465	-3015	9046	28
H(21)	5921	-4604	8699	34
H(22)	5494	-5497	7570	39
H(23)	5634	-4783	6773	40
H(24)	6174	-3201	7107	32
H(26)	4489	1383	8100	27
H(27)	3596	1772	7362	30
H(31)	3734	2462	6479	34
H(32)	4849	3273	5850	37
H(33)	6875	3872	6076	35
H(34)	7792	3682	6925	29
H(36)	8390	2054	7736	29
H(37)	10394	2718	8071	35
H(38)	11377	4310	8478	34
H(41)	11347	5860	8804	33
H(42)	10325	6786	8950	35
H(43)	8309	6125	8659	32
H(44)	7311	4547	8219	30
H(45)	7853	1129	6570	28
H(47)	5811	1359	5234	30
H(52)	2121	4	5699	31
H(53)	3669	753	5257	31
H(54)	1339	-808	6498	30
H(58)	1947	-628	8427	37
H(59)	306	-1260	8891	45
H(60)	-1328	-2500	8216	46
H(61)	-1361	-3068	7062	43
H(62)	255	-2411	6585	38
H(64)	4244	-122	8642	32
H(65)	4486	-760	9422	35
H(69)	4477	-2104	9695	46
H(70)	4038	-3664	9366	52
H(71)	3354	-4605	8248	45
H(72)	3076	-3995	7463	37
H(74)	4303	-2201	6558	29
H(75)	3753	-3178	5438	35
H(76)	2025	-4469	5116	42
H(79)	1175	-3425	7611	33
H(80)	-555	-4713	7265	44

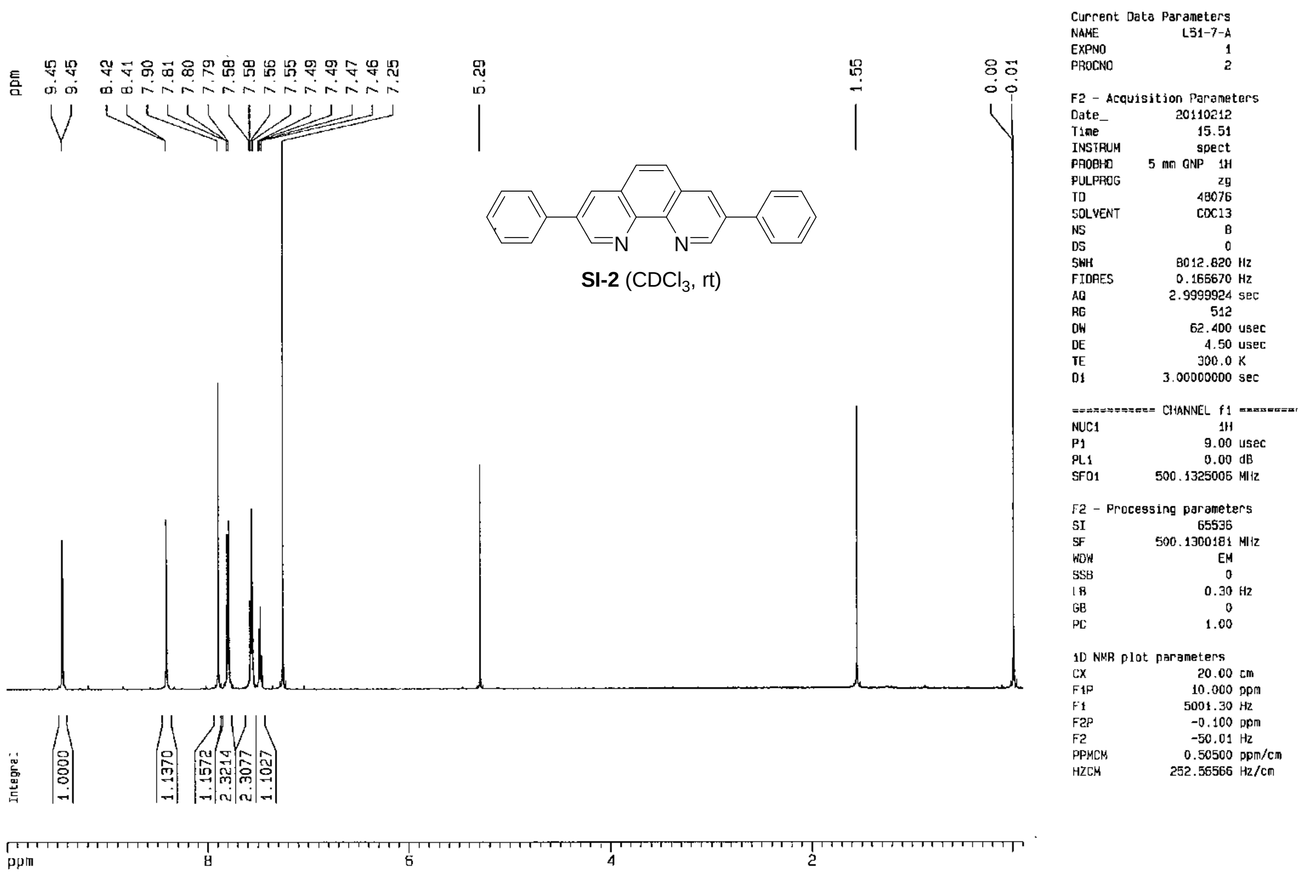
H(81)	-1019	-5737	6150	56
H(82)	210	-5440	5388	53
H(84)	9017	1124	5833	72
H(85)	10596	1697	5302	89
H(86)	10567	2646	4697	74
H(87)	8944	2948	4585	44
H(88)	7372	2377	5104	37
H(90A)	9984	-386	7460	155
H(90B)	10717	705	7709	155
H(90C)	10280	131	6929	155
H(93A)	1085	3675	6674	362
H(93B)	2330	3773	6461	362
H(93C)	2186	4070	7235	362

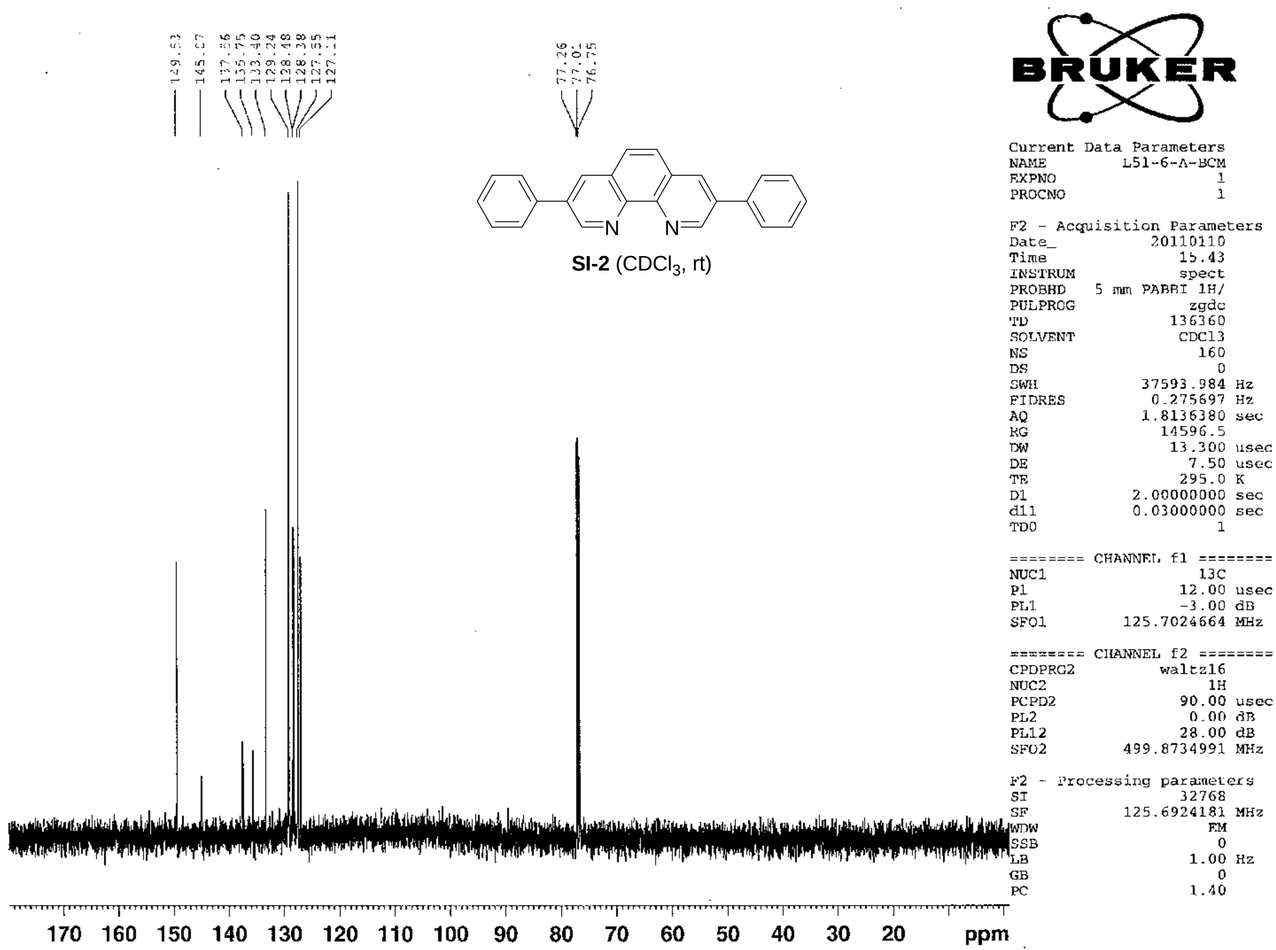
Table 6. Torsion angles [°] for Nish02.

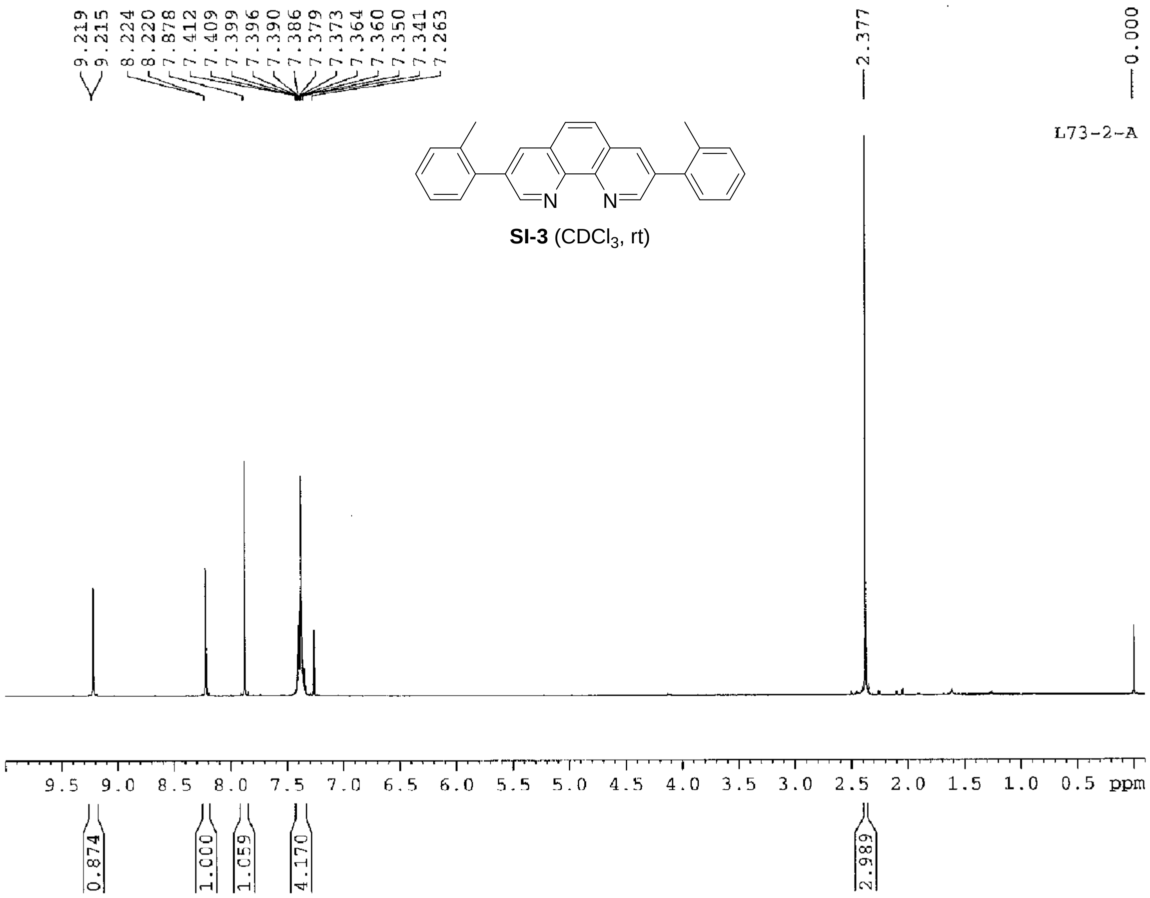
N(1)-C(1)-C(2)-C(3)	-0.7(5)	C(31)-C(28)-C(29)-C(30)	-179.9(3)
N(1)-C(1)-C(2)-C(19)	176.8(3)	C(26)-C(25)-C(30)-C(29)	-6.7(4)
C(1)-C(2)-C(3)-C(4)	1.0(4)	C(12)-C(25)-C(30)-C(29)	176.4(3)
C(19)-C(2)-C(3)-C(4)	-176.4(3)	C(26)-C(25)-C(30)-C(35)	174.7(3)
C(2)-C(3)-C(4)-C(5)	0.1(5)	C(12)-C(25)-C(30)-C(35)	-2.1(4)
C(2)-C(3)-C(4)-C(6)	177.9(3)	C(28)-C(29)-C(30)-C(25)	7.5(4)
C(3)-C(4)-C(5)-N(1)	-1.7(4)	C(34)-C(29)-C(30)-C(25)	-171.4(3)
C(6)-C(4)-C(5)-N(1)	-179.6(3)	C(28)-C(29)-C(30)-C(35)	-173.9(3)
C(3)-C(4)-C(5)-C(9)	177.3(3)	C(34)-C(29)-C(30)-C(35)	7.2(4)
C(6)-C(4)-C(5)-C(9)	-0.6(5)	C(27)-C(28)-C(31)-C(32)	-175.7(3)
C(3)-C(4)-C(6)-C(7)	-178.5(3)	C(29)-C(28)-C(31)-C(32)	1.6(5)
C(5)-C(4)-C(6)-C(7)	-0.7(5)	C(28)-C(31)-C(32)-C(33)	-0.9(5)
C(4)-C(6)-C(7)-C(8)	1.0(5)	C(31)-C(32)-C(33)-C(34)	-0.4(5)
C(6)-C(7)-C(8)-C(10)	176.5(3)	C(32)-C(33)-C(34)-C(29)	1.0(5)
C(6)-C(7)-C(8)-C(9)	-0.1(5)	C(28)-C(29)-C(34)-C(33)	-0.3(4)
C(10)-C(8)-C(9)-N(2)	0.7(4)	C(30)-C(29)-C(34)-C(33)	178.6(3)
C(7)-C(8)-C(9)-N(2)	177.5(3)	C(25)-C(30)-C(35)-C(36)	57.9(4)
C(10)-C(8)-C(9)-C(5)	-178.0(3)	C(29)-C(30)-C(35)-C(36)	-120.7(3)
C(7)-C(8)-C(9)-C(5)	-1.2(4)	C(25)-C(30)-C(35)-C(40)	-119.9(3)
N(1)-C(5)-C(9)-N(2)	1.8(4)	C(29)-C(30)-C(35)-C(40)	61.6(4)
C(4)-C(5)-C(9)-N(2)	-177.2(3)	C(40)-C(35)-C(36)-C(37)	-1.0(5)
N(1)-C(5)-C(9)-C(8)	-179.5(3)	C(30)-C(35)-C(36)-C(37)	-178.8(3)
C(4)-C(5)-C(9)-C(8)	1.5(4)	C(35)-C(36)-C(37)-C(38)	-0.5(5)
C(9)-C(8)-C(10)-C(11)	2.5(5)	C(36)-C(37)-C(38)-C(39)	1.2(5)
C(7)-C(8)-C(10)-C(11)	-174.2(3)	C(37)-C(38)-C(39)-C(40)	-0.4(5)
C(8)-C(10)-C(11)-C(12)	-3.9(4)	C(37)-C(38)-C(39)-C(41)	178.7(3)
C(8)-C(10)-C(11)-C(13)	173.3(3)	C(38)-C(39)-C(40)-C(44)	176.7(3)
C(10)-C(11)-C(12)-N(2)	2.4(4)	C(41)-C(39)-C(40)-C(44)	-2.4(4)
C(13)-C(11)-C(12)-N(2)	-174.7(3)	C(38)-C(39)-C(40)-C(35)	-1.1(4)
C(10)-C(11)-C(12)-C(25)	-178.7(3)	C(41)-C(39)-C(40)-C(35)	179.8(3)
C(13)-C(11)-C(12)-C(25)	4.2(4)	C(36)-C(35)-C(40)-C(44)	-175.9(3)
C(10)-C(11)-C(13)-C(14)	-125.6(3)	C(30)-C(35)-C(40)-C(44)	1.9(4)
C(12)-C(11)-C(13)-C(14)	51.5(4)	C(36)-C(35)-C(40)-C(39)	1.8(4)
C(10)-C(11)-C(13)-C(18)	49.1(4)	C(30)-C(35)-C(40)-C(39)	179.6(3)
C(12)-C(11)-C(13)-C(18)	-133.8(3)	C(38)-C(39)-C(41)-C(42)	-177.5(3)
C(18)-C(13)-C(14)-C(15)	0.1(5)	C(40)-C(39)-C(41)-C(42)	1.6(5)
C(11)-C(13)-C(14)-C(15)	174.9(3)	C(39)-C(41)-C(42)-C(43)	-0.1(5)
C(13)-C(14)-C(15)-C(16)	0.3(5)	C(41)-C(42)-C(43)-C(44)	-0.4(5)
C(14)-C(15)-C(16)-C(17)	-0.3(5)	C(42)-C(43)-C(44)-C(40)	-0.5(5)
C(15)-C(16)-C(17)-C(18)	-0.2(5)	C(39)-C(40)-C(44)-C(43)	2.0(4)
C(16)-C(17)-C(18)-C(13)	0.6(5)	C(35)-C(40)-C(44)-C(43)	179.6(3)
C(14)-C(13)-C(18)-C(17)	-0.6(5)	N(3)-C(45)-C(46)-C(47)	2.1(5)
C(11)-C(13)-C(18)-C(17)	-175.4(3)	N(3)-C(45)-C(46)-C(83)	-174.8(3)
C(3)-C(2)-C(19)-C(20)	19.4(5)	C(45)-C(46)-C(47)-C(48)	-1.2(5)
C(1)-C(2)-C(19)-C(20)	-158.0(3)	C(83)-C(46)-C(47)-C(48)	175.6(3)
C(3)-C(2)-C(19)-C(24)	-161.9(3)	C(46)-C(47)-C(48)-C(49)	-0.9(5)
C(1)-C(2)-C(19)-C(24)	20.7(4)	C(46)-C(47)-C(48)-C(53)	179.4(3)
C(24)-C(19)-C(20)-C(21)	-2.5(5)	C(47)-C(48)-C(49)-N(3)	2.5(4)
C(2)-C(19)-C(20)-C(21)	176.2(3)	C(53)-C(48)-C(49)-N(3)	-177.8(3)
C(19)-C(20)-C(21)-C(22)	1.0(5)	C(47)-C(48)-C(49)-C(50)	-176.5(3)
C(20)-C(21)-C(22)-C(23)	0.6(5)	C(53)-C(48)-C(49)-C(50)	3.2(4)
C(21)-C(22)-C(23)-C(24)	-0.6(5)	N(3)-C(49)-C(50)-N(4)	-2.2(4)
C(22)-C(23)-C(24)-C(19)	-0.9(5)	C(48)-C(49)-C(50)-N(4)	176.9(3)
C(20)-C(19)-C(24)-C(23)	2.5(5)	N(3)-C(49)-C(50)-C(51)	177.7(3)
C(2)-C(19)-C(24)-C(23)	-176.2(3)	C(48)-C(49)-C(50)-C(51)	-3.3(4)
N(2)-C(12)-C(25)-C(30)	-110.1(3)	N(4)-C(50)-C(51)-C(54)	2.9(5)
C(11)-C(12)-C(25)-C(30)	70.9(4)	C(49)-C(50)-C(51)-C(54)	-176.9(3)
N(2)-C(12)-C(25)-C(26)	72.9(4)	N(4)-C(50)-C(51)-C(52)	-179.2(3)
C(11)-C(12)-C(25)-C(26)	-106.1(3)	C(49)-C(50)-C(51)-C(52)	1.0(4)
C(30)-C(25)-C(26)-C(27)	0.8(5)	C(50)-C(51)-C(52)-C(53)	1.5(5)
C(12)-C(25)-C(26)-C(27)	177.8(3)	C(54)-C(51)-C(52)-C(53)	179.3(3)
C(25)-C(26)-C(27)-C(28)	4.4(5)	C(51)-C(52)-C(53)-C(48)	-1.6(5)
C(26)-C(27)-C(28)-C(31)	173.9(3)	C(47)-C(48)-C(53)-C(52)	178.9(3)
C(26)-C(27)-C(28)-C(29)	-3.4(5)	C(49)-C(48)-C(53)-C(52)	-0.8(5)
C(27)-C(28)-C(29)-C(34)	176.4(3)	C(50)-C(51)-C(54)-C(55)	-1.4(5)
C(31)-C(28)-C(29)-C(34)	-1.0(4)	C(52)-C(51)-C(54)-C(55)	-179.3(3)
C(27)-C(28)-C(29)-C(30)	-2.6(4)	C(51)-C(54)-C(55)-C(56)	-2.5(5)

C(51)-C(54)-C(55)-C(57)	176.7(3)	C(45)-C(46)-C(83)-C(88)	-162.1(3)
C(54)-C(55)-C(56)-N(4)	5.3(5)	C(47)-C(46)-C(83)-C(84)	-150.5(4)
C(57)-C(55)-C(56)-N(4)	-173.8(3)	C(45)-C(46)-C(83)-C(84)	26.2(5)
C(54)-C(55)-C(56)-C(63)	-173.2(3)	C(88)-C(83)-C(84)-C(85)	3.2(7)
C(57)-C(55)-C(56)-C(63)	7.6(5)	C(46)-C(83)-C(84)-C(85)	175.2(4)
C(54)-C(55)-C(57)-C(62)	51.9(4)	C(83)-C(84)-C(85)-C(86)	-0.6(8)
C(56)-C(55)-C(57)-C(62)	-128.9(3)	C(84)-C(85)-C(86)-C(87)	-1.9(8)
C(54)-C(55)-C(57)-C(58)	-122.9(3)	C(85)-C(86)-C(87)-C(88)	1.7(7)
C(56)-C(55)-C(57)-C(58)	56.2(4)	C(86)-C(87)-C(88)-C(83)	1.0(6)
C(62)-C(57)-C(58)-C(59)	0.9(5)	C(84)-C(83)-C(88)-C(87)	-3.5(5)
C(55)-C(57)-C(58)-C(59)	175.8(3)	C(46)-C(83)-C(88)-C(87)	-175.4(3)
C(57)-C(58)-C(59)-C(60)	0.9(5)	C(2)-C(1)-N(1)-C(5)	-0.8(4)
C(58)-C(59)-C(60)-C(61)	-1.7(5)	C(2)-C(1)-N(1)-Fe(1)	168.5(2)
C(59)-C(60)-C(61)-C(62)	0.6(5)	C(4)-C(5)-N(1)-C(1)	2.0(4)
C(60)-C(61)-C(62)-C(57)	1.3(5)	C(9)-C(5)-N(1)-C(1)	-177.0(3)
C(58)-C(57)-C(62)-C(61)	-2.0(5)	C(4)-C(5)-N(1)-Fe(1)	-168.8(2)
C(55)-C(57)-C(62)-C(61)	-176.9(3)	C(9)-C(5)-N(1)-Fe(1)	12.3(3)
N(4)-C(56)-C(63)-C(68)	-104.1(3)	N(3)-Fe(1)-N(1)-C(1)	-45.8(8)
C(55)-C(56)-C(63)-C(68)	74.5(4)	O(1)-Fe(1)-N(1)-C(1)	-15.2(2)
N(4)-C(56)-C(63)-C(64)	79.1(4)	N(5)-Fe(1)-N(1)-C(1)	-97.6(3)
C(55)-C(56)-C(63)-C(64)	-102.3(3)	N(2)-Fe(1)-N(1)-C(1)	175.6(3)
C(68)-C(63)-C(64)-C(65)	0.9(5)	N(4)-Fe(1)-N(1)-C(1)	77.4(3)
C(56)-C(63)-C(64)-C(65)	177.6(3)	N(3)-Fe(1)-N(1)-C(5)	124.0(7)
C(63)-C(64)-C(65)-C(66)	1.3(5)	O(1)-Fe(1)-N(1)-C(5)	154.6(2)
C(64)-C(65)-C(66)-C(67)	-1.5(5)	N(5)-Fe(1)-N(1)-C(5)	72.2(2)
C(64)-C(65)-C(66)-C(69)	177.0(3)	N(2)-Fe(1)-N(1)-C(5)	-14.6(2)
C(65)-C(66)-C(67)-C(72)	177.1(3)	N(4)-Fe(1)-N(1)-C(5)	-112.8(2)
C(69)-C(66)-C(67)-C(72)	-1.5(5)	C(11)-C(12)-N(2)-C(9)	0.6(4)
C(65)-C(66)-C(67)-C(68)	-0.4(5)	C(25)-C(12)-N(2)-C(9)	-178.4(3)
C(69)-C(66)-C(67)-C(68)	-178.9(3)	C(11)-C(12)-N(2)-Fe(1)	-166.4(2)
C(64)-C(63)-C(68)-C(67)	-2.8(5)	C(25)-C(12)-N(2)-Fe(1)	14.6(4)
C(56)-C(63)-C(68)-C(67)	-179.5(3)	C(8)-C(9)-N(2)-C(12)	-2.2(4)
C(64)-C(63)-C(68)-C(73)	-178.7(3)	C(5)-C(9)-N(2)-C(12)	176.5(3)
C(56)-C(63)-C(68)-C(73)	4.6(4)	C(8)-C(9)-N(2)-Fe(1)	167.4(2)
C(72)-C(67)-C(68)-C(63)	-174.8(3)	C(5)-C(9)-N(2)-Fe(1)	-13.9(3)
C(66)-C(67)-C(68)-C(63)	2.5(4)	N(3)-Fe(1)-N(2)-C(12)	8.6(3)
C(72)-C(67)-C(68)-C(73)	1.2(4)	N(1)-Fe(1)-N(2)-C(12)	-177.1(3)
C(66)-C(67)-C(68)-C(73)	178.5(3)	O(1)-Fe(1)-N(2)-C(12)	150.5(3)
C(65)-C(66)-C(69)-C(70)	-179.8(3)	N(5)-Fe(1)-N(2)-C(12)	99.2(3)
C(67)-C(66)-C(69)-C(70)	-1.2(5)	N(4)-Fe(1)-N(2)-C(12)	-71.8(3)
C(66)-C(69)-C(70)-C(71)	2.4(5)	N(3)-Fe(1)-N(2)-C(9)	-159.34(19)
C(69)-C(70)-C(71)-C(72)	-0.7(5)	N(1)-Fe(1)-N(2)-C(9)	15.01(19)
C(70)-C(71)-C(72)-C(67)	-2.1(5)	O(1)-Fe(1)-N(2)-C(9)	-17.4(4)
C(66)-C(67)-C(72)-C(71)	3.2(5)	N(5)-Fe(1)-N(2)-C(9)	-68.7(2)
C(68)-C(67)-C(72)-C(71)	-179.5(3)	N(4)-Fe(1)-N(2)-C(9)	120.26(19)
C(63)-C(68)-C(73)-C(74)	60.7(4)	C(46)-C(45)-N(3)-C(49)	-0.5(5)
C(67)-C(68)-C(73)-C(74)	-115.2(3)	C(46)-C(45)-N(3)-Fe(1)	173.8(2)
C(63)-C(68)-C(73)-C(78)	-122.2(3)	C(48)-C(49)-N(3)-C(45)	-1.8(4)
C(67)-C(68)-C(73)-C(78)	61.9(4)	C(50)-C(49)-N(3)-C(45)	177.2(3)
C(78)-C(73)-C(74)-C(75)	-2.2(5)	C(48)-C(49)-N(3)-Fe(1)	-176.8(2)
C(68)-C(73)-C(74)-C(75)	175.0(3)	C(50)-C(49)-N(3)-Fe(1)	2.2(3)
C(73)-C(74)-C(75)-C(76)	-0.6(5)	N(1)-Fe(1)-N(3)-C(45)	-50.8(8)
C(74)-C(75)-C(76)-C(77)	2.0(5)	O(1)-Fe(1)-N(3)-C(45)	-81.3(3)
C(75)-C(76)-C(77)-C(82)	-179.7(4)	N(5)-Fe(1)-N(3)-C(45)	0.5(3)
C(75)-C(76)-C(77)-C(78)	-0.5(6)	N(2)-Fe(1)-N(3)-C(45)	86.3(3)
C(76)-C(77)-C(78)-C(79)	178.2(3)	N(4)-Fe(1)-N(3)-C(45)	-175.7(3)
C(82)-C(77)-C(78)-C(79)	-2.6(5)	N(1)-Fe(1)-N(3)-C(49)	123.8(7)
C(76)-C(77)-C(78)-C(73)	-2.3(5)	O(1)-Fe(1)-N(3)-C(49)	93.2(2)
C(82)-C(77)-C(78)-C(73)	176.9(3)	N(5)-Fe(1)-N(3)-C(49)	175.0(2)
C(74)-C(73)-C(78)-C(79)	-176.9(3)	N(2)-Fe(1)-N(3)-C(49)	-99.2(2)
C(68)-C(73)-C(78)-C(79)	5.9(5)	N(4)-Fe(1)-N(3)-C(49)	-1.2(2)
C(74)-C(73)-C(78)-C(77)	3.6(5)	C(55)-C(56)-N(4)-C(50)	-4.0(4)
C(68)-C(73)-C(78)-C(77)	-173.5(3)	C(63)-C(56)-N(4)-C(50)	174.6(3)
C(77)-C(78)-C(79)-C(80)	2.2(5)	C(55)-C(56)-N(4)-Fe(1)	174.3(2)
C(73)-C(78)-C(79)-C(80)	-177.3(3)	C(63)-C(56)-N(4)-Fe(1)	-7.2(4)
C(78)-C(79)-C(80)-C(81)	-0.3(5)	C(51)-C(50)-N(4)-C(56)	-0.3(4)
C(79)-C(80)-C(81)-C(82)	-1.3(6)	C(49)-C(50)-N(4)-C(56)	179.6(3)
C(80)-C(81)-C(82)-C(77)	0.9(7)	C(51)-C(50)-N(4)-Fe(1)	-178.8(2)
C(76)-C(77)-C(82)-C(81)	-179.7(4)	C(49)-C(50)-N(4)-Fe(1)	1.0(3)
C(78)-C(77)-C(82)-C(81)	1.0(6)	N(3)-Fe(1)-N(4)-C(56)	-178.2(3)
C(47)-C(46)-C(83)-C(88)	21.2(5)	N(1)-Fe(1)-N(4)-C(56)	8.7(3)

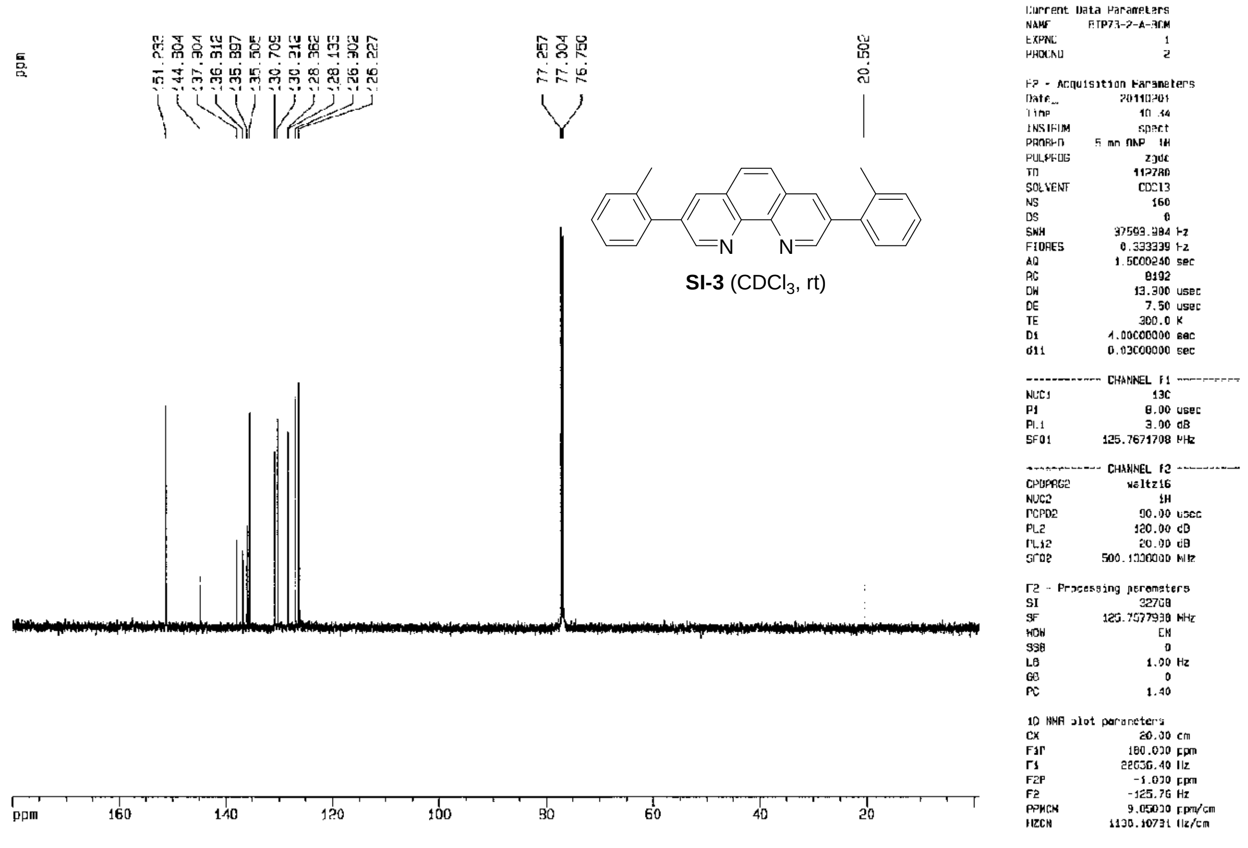
O(1)-Fe(1)-N(4)-C(56)	95.0(3)	Fe(1)-O(1)-S(1)-O(3)	47.1(3)
N(5)-Fe(1)-N(4)-C(56)	162.1(4)	Fe(1)-O(1)-S(1)-C(91)	158.8(2)
N(2)-Fe(1)-N(4)-C(56)	-71.3(3)	F(1)-C(91)-S(1)-O(2)	-60.2(3)
N(3)-Fe(1)-N(4)-C(50)	0.10(19)	F(3)-C(91)-S(1)-O(2)	61.5(3)
N(1)-Fe(1)-N(4)-C(50)	-172.97(19)	F(2)-C(91)-S(1)-O(2)	-178.6(3)
O(1)-Fe(1)-N(4)-C(50)	-86.6(2)	F(1)-C(91)-S(1)-O(3)	178.0(2)
N(5)-Fe(1)-N(4)-C(50)	-19.6(6)	F(3)-C(91)-S(1)-O(3)	-60.4(3)
N(2)-Fe(1)-N(4)-C(50)	107.02(19)	F(2)-C(91)-S(1)-O(3)	59.5(3)
C(90)-C(89)-N(5)-Fe(1)	66(11)	F(1)-C(91)-S(1)-O(1)	57.7(3)
N(3)-Fe(1)-N(5)-C(89)	-120.9(7)	F(3)-C(91)-S(1)-O(1)	179.4(3)
N(1)-Fe(1)-N(5)-C(89)	52.7(7)	F(2)-C(91)-S(1)-O(1)	-60.7(3)
O(1)-Fe(1)-N(5)-C(89)	-33.6(7)	F(6)-C(94)-S(2)-O(6)	-57.3(5)
N(2)-Fe(1)-N(5)-C(89)	130.4(7)	F(4)-C(94)-S(2)-O(6)	-179.6(5)
N(4)-Fe(1)-N(5)-C(89)	-101.7(8)	F(5)-C(94)-S(2)-O(6)	62.9(5)
N(3)-Fe(1)-O(1)-S(1)	17.2(2)	F(6)-C(94)-S(2)-O(5)	61.7(5)
N(1)-Fe(1)-O(1)-S(1)	-158.6(2)	F(4)-C(94)-S(2)-O(5)	-60.7(5)
N(5)-Fe(1)-O(1)-S(1)	-75.2(2)	F(5)-C(94)-S(2)-O(5)	-178.2(5)
N(2)-Fe(1)-O(1)-S(1)	-127.0(2)	F(6)-C(94)-S(2)-O(4)	-178.2(5)
N(4)-Fe(1)-O(1)-S(1)	94.4(2)	F(4)-C(94)-S(2)-O(4)	59.5(5)
Fe(1)-O(1)-S(1)-O(2)	-90.2(2)	F(5)-C(94)-S(2)-O(4)	-58.0(6)

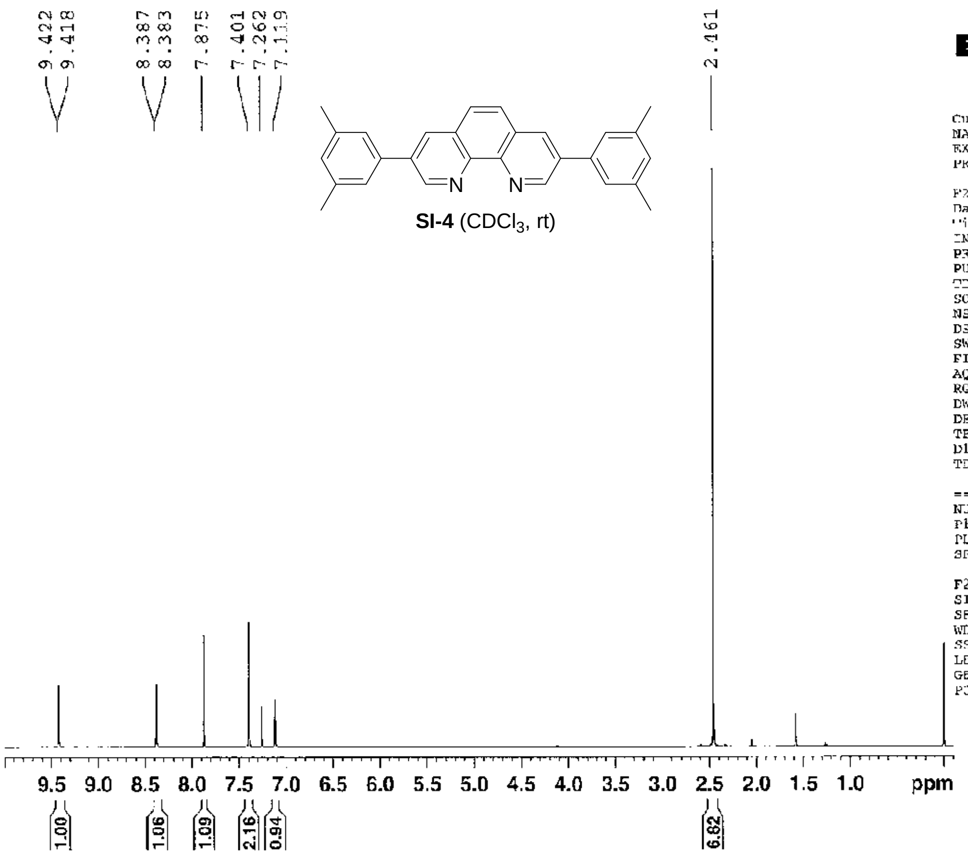

ppm
9.45
9.45
8.42
8.41
7.90
7.81
7.80
7.79
7.58
7.56
7.55
7.49
7.49
7.47
7.46
7.25
5.29
1.55
0.00
-0.01
SI-2 (CDCl3, rt)
Integral
1.0000
1.1370
1.1572
2.3214
2.3077
1.1027
ppm
8
6
4
2
Current Data Parameters
NAME L51-7-A
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20110212
Time 15.51
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 512
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325005 MHz
F2 - Processing parameters
SI 65536
SF 500.1300181 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P -0.100 ppm
F2 -50.01 Hz
PPMCM 0.50500 ppm/cm
HZCM 252.56566 Hz/cm


149.53
145.07
137.56
135.75
133.40
129.24
128.48
128.38
127.55
127.11
77.26
77.01
76.75
SI-2 (CDCl3, rt)
BRUKER
Current Data Parameters
NAME L51-6-A-BCM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110110
Time 15.43
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT CDCl3
NS 160
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136380 sec
RG 14596.5
DW 13.300 usec
DE 7.50 usec
TE 295.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7024664 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6924181 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



97/10/27, #5, 122.5 mg





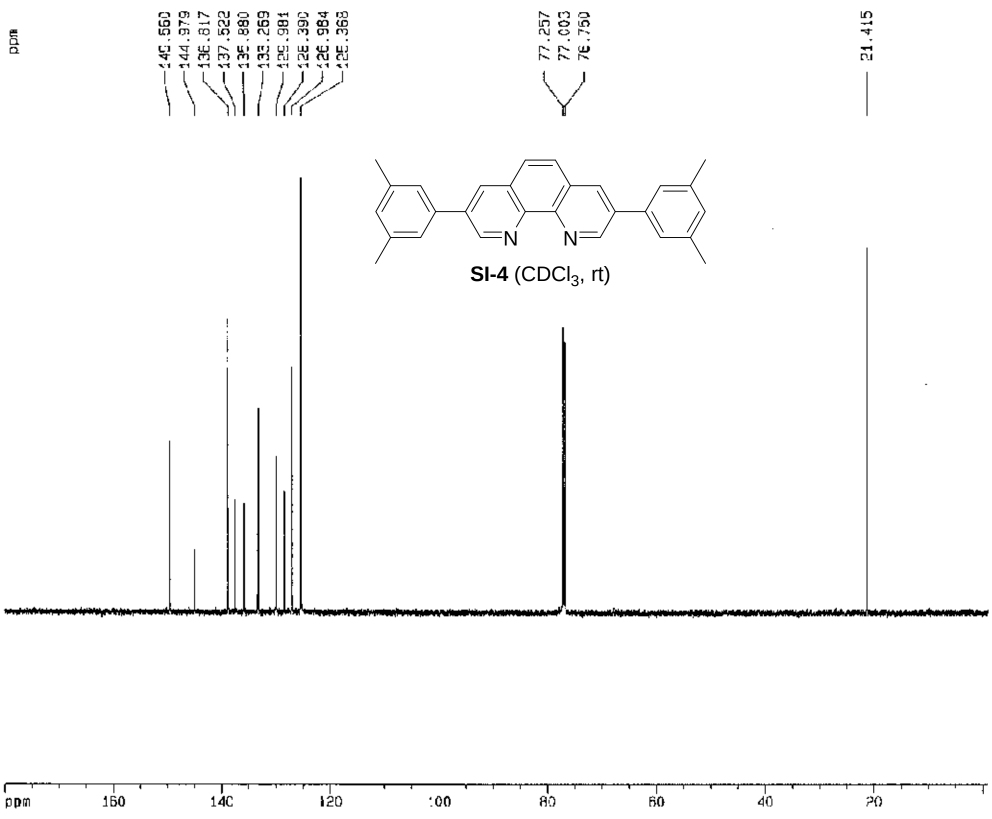
Current Data Parameters
NAME L86-6-A2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100910
Time 17.43
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 6
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 181
DW 50.000 usec
DE 7.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8722992 MHz

F2 - Processing parameters
SI 32768
SF 499.8700170 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

97/10/17, #5, 122.5 mg



Current Data Parameters
NAME L86-TH-BCM
EXPNO 1
PROCNO 2

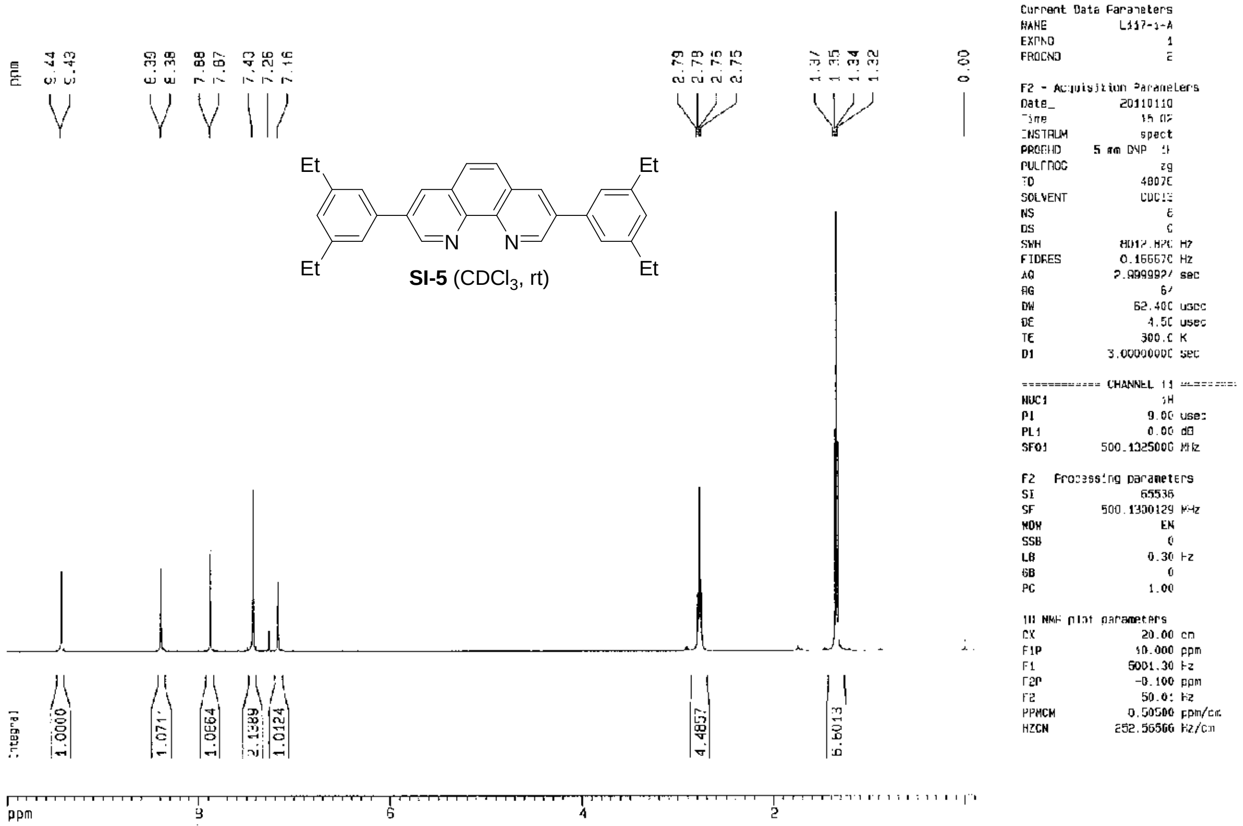
F2 - Acquisition Parameters
Date_ 20101230
Time 10.02
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zgdc
TD 112760
SOLVENT CDCl3
NS 200
DS 0
SWH 37593.984 Hz
FIDRES 0.333339 Hz
AQ 1.5000240 sec
RG 8192
DW 13.300 usec
DE 7.50 usec
TE 300.0 K
D1 4.00000000 sec
d11 0.03000000 sec

============ CHANNEL f1 ============
NUC1 13C
P1 8.00 usec
PL1 3.00 dB
SFO1 125.7671708 MHz

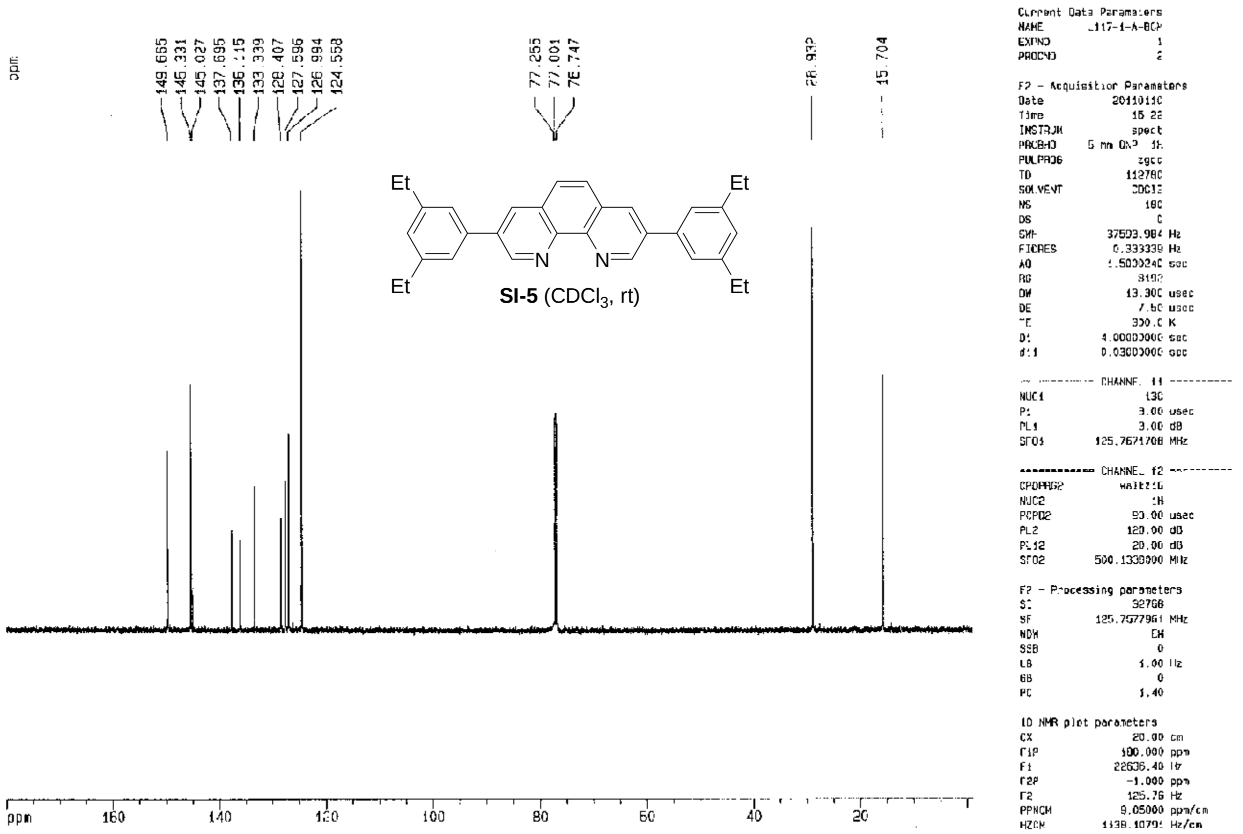
============ CHANNEL f2 ============
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 120.00 dB
PL12 20.00 dB
SFO2 500.1338000 MHz

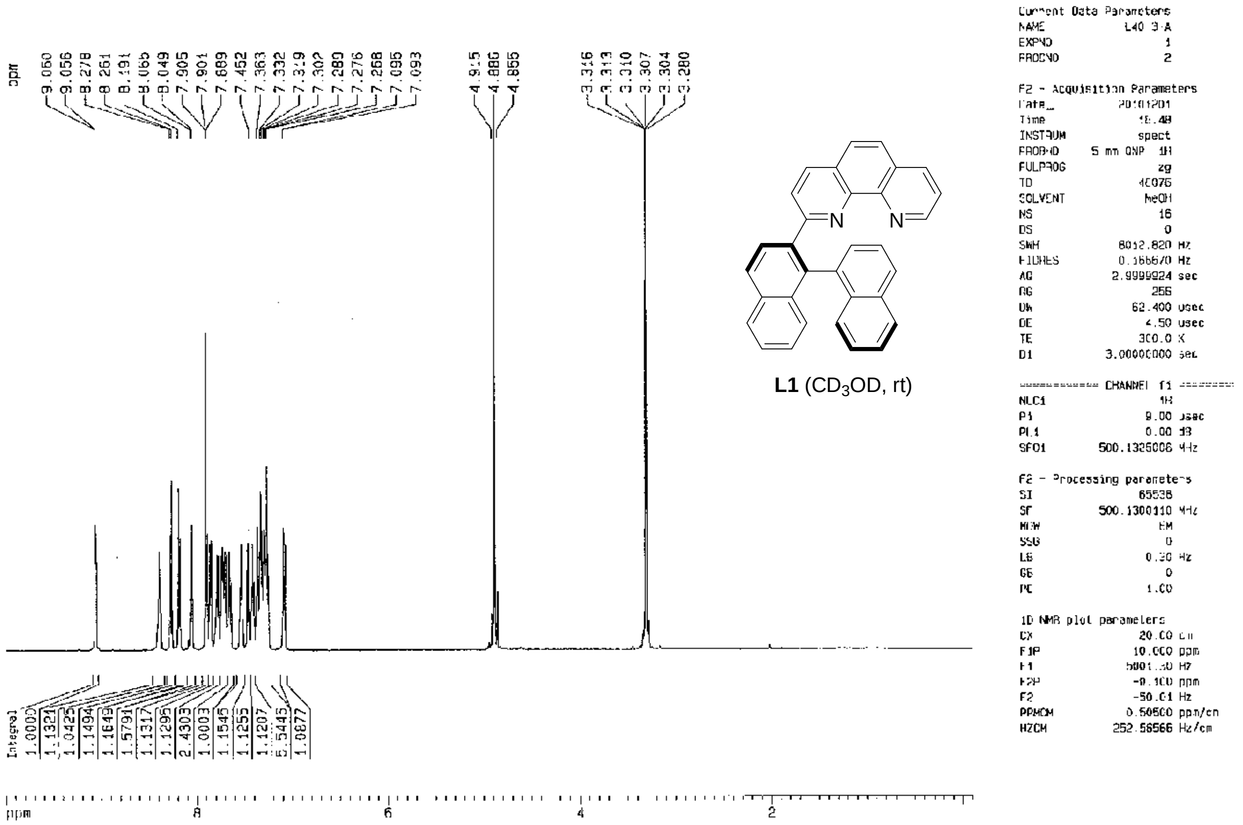
F2 - Processing parameters
SI 32768
SF 125.7577901 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 180.000 ppm
F1 22636.40 Hz
F2P -1.000 ppm
F2 -125.76 Hz
PPMCM 9.05000 ppm/cm
HZCM 1138.10791 Hz/cm



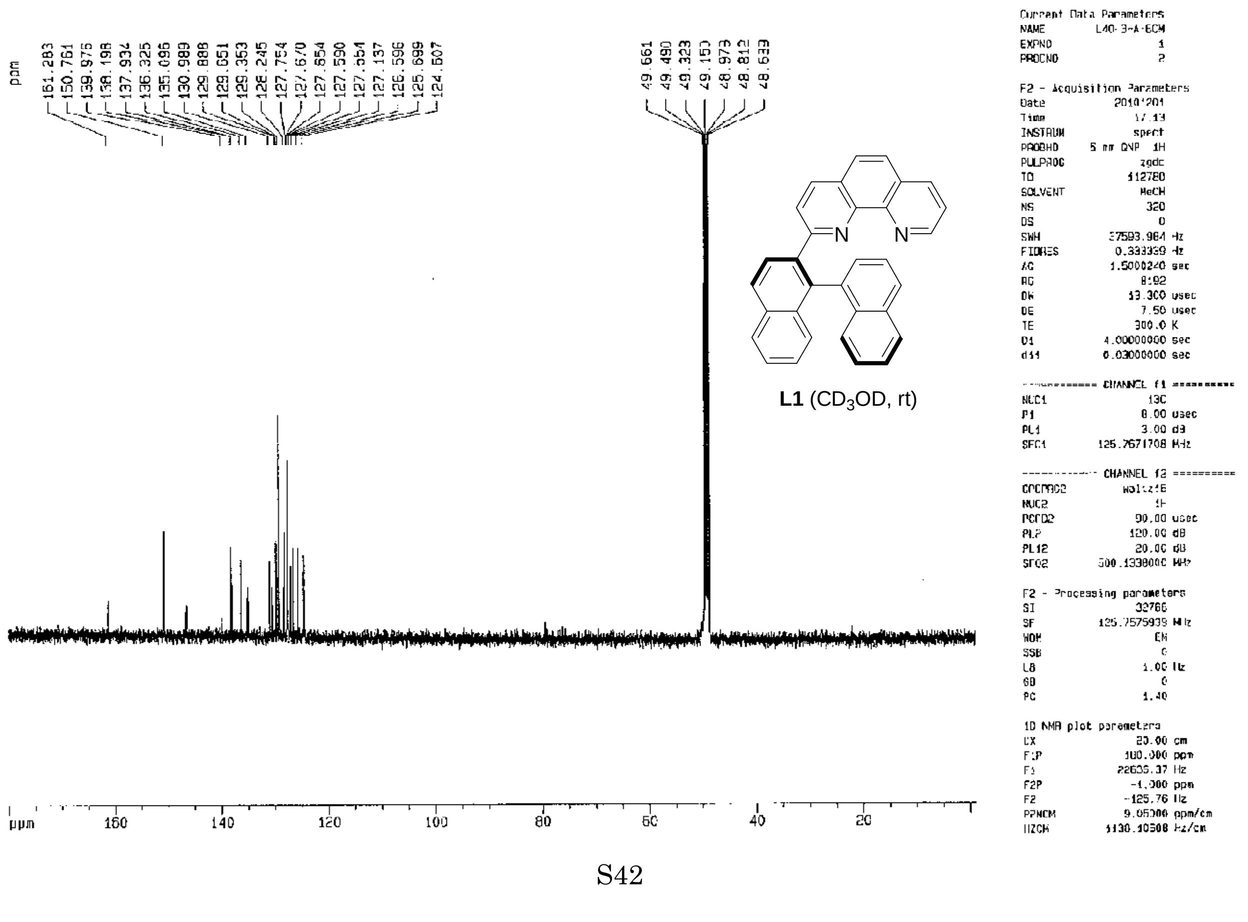
07/10/17, #5, 122.5 mg



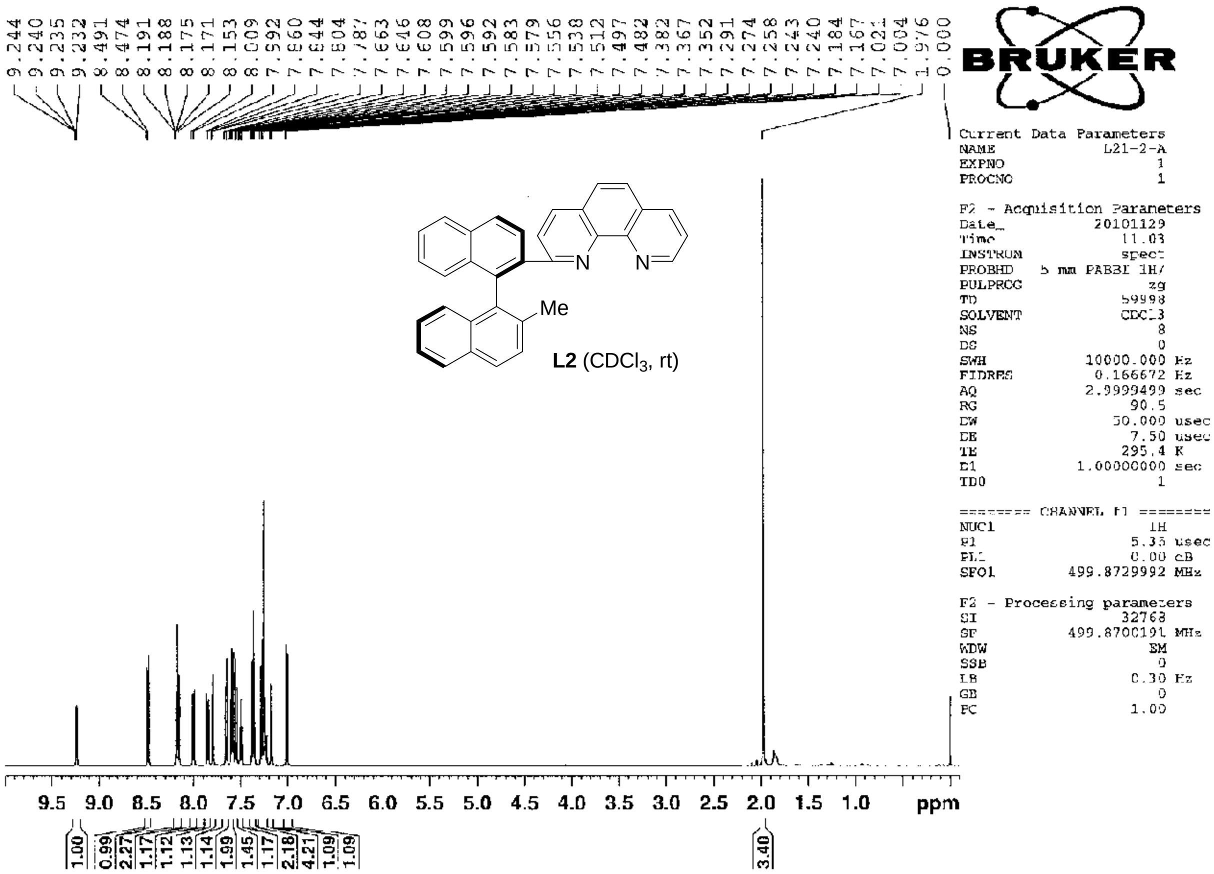


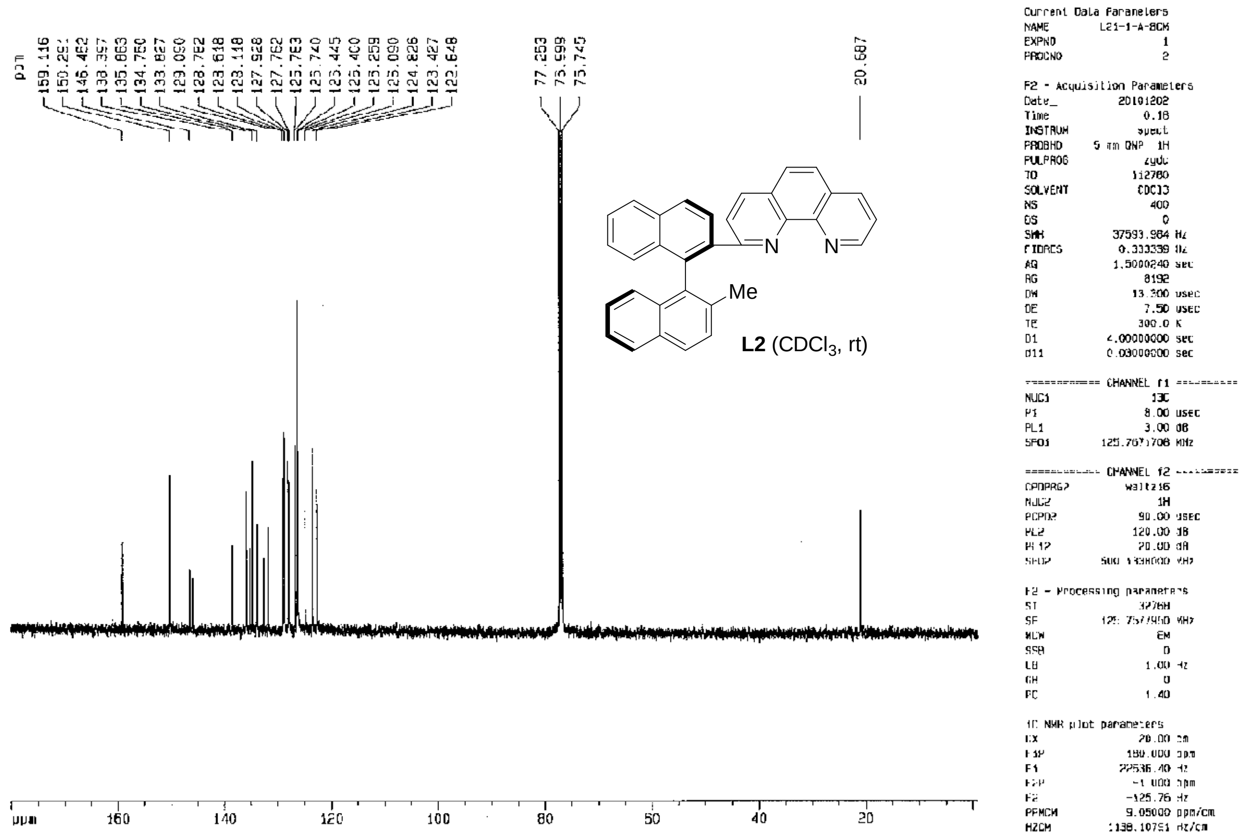
L1 (CD_3OD, rt)

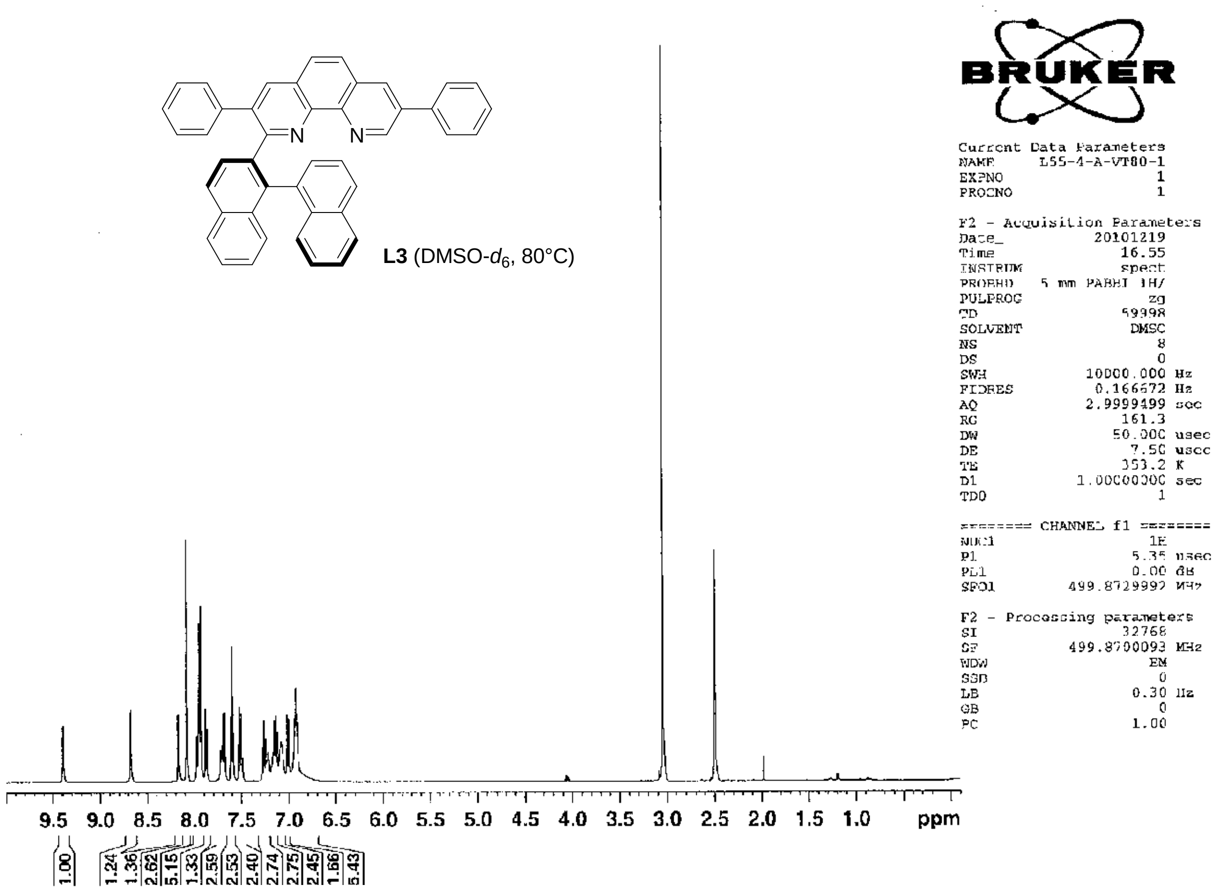
07/10/17, #5, 122.5 mg



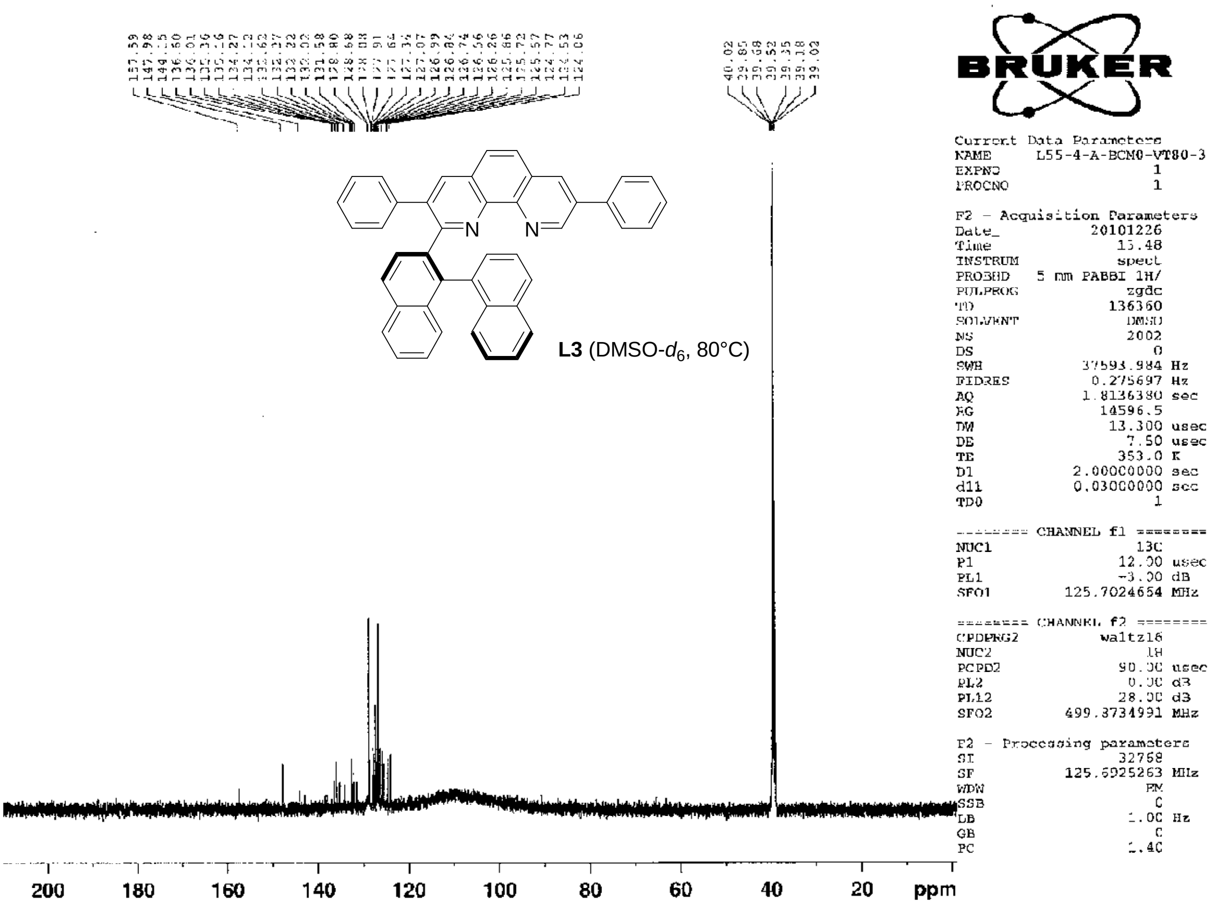
L1 (CD_3OD, rt)



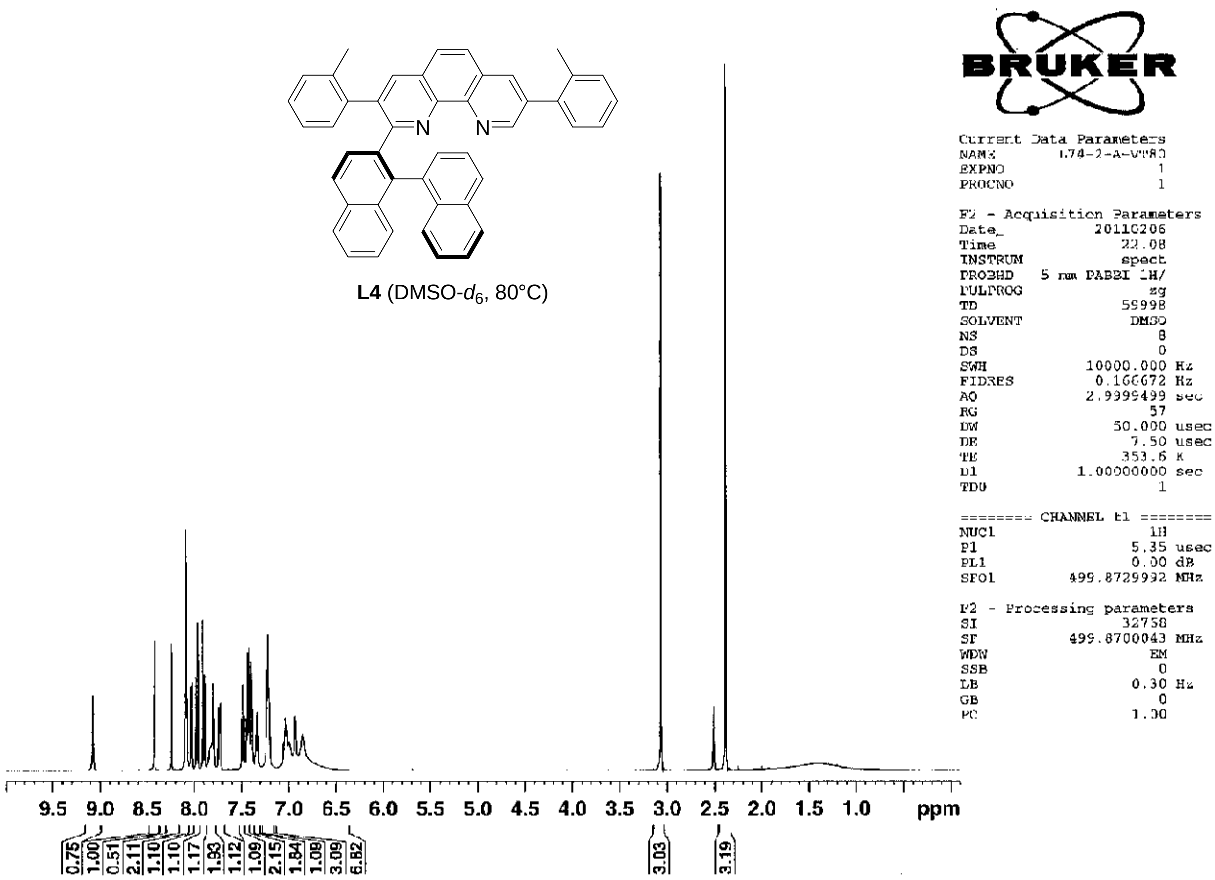


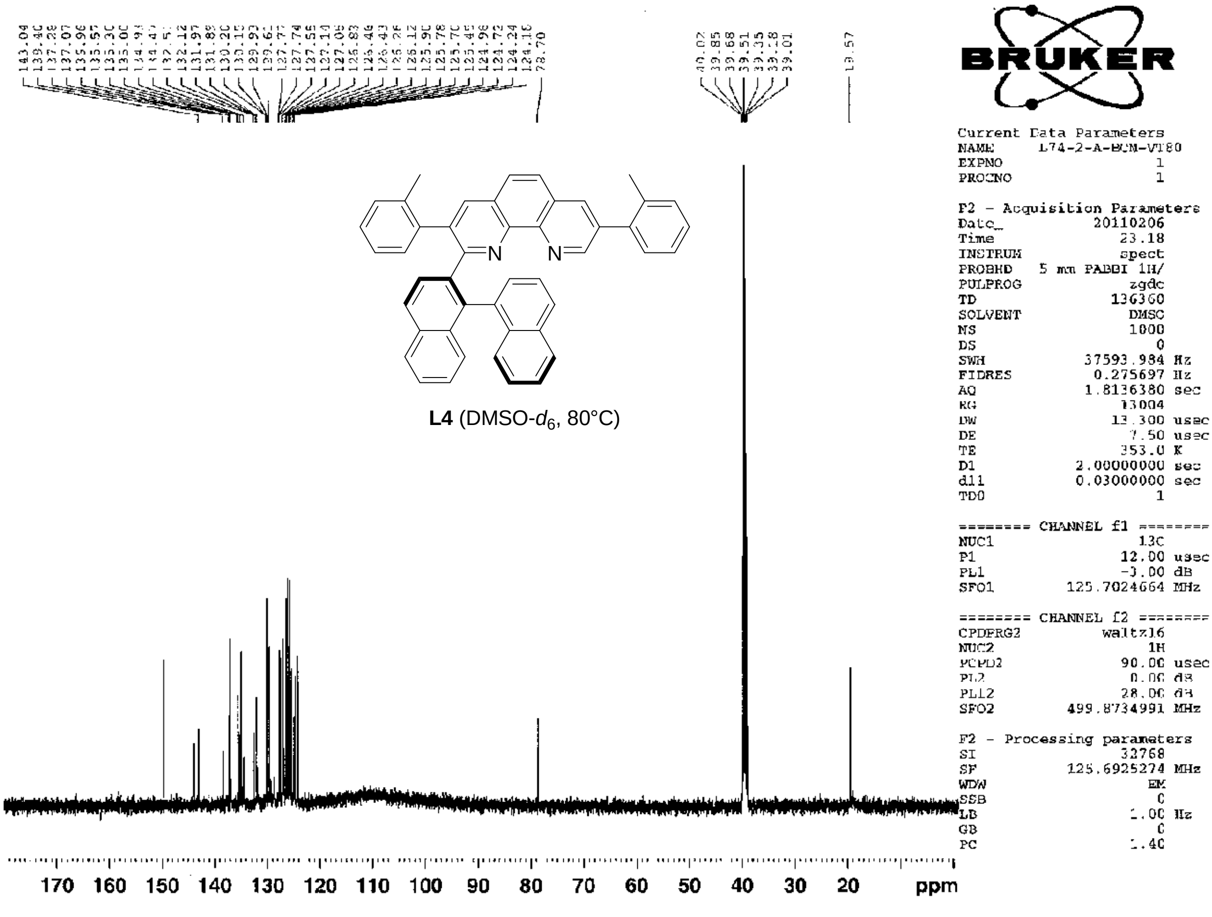


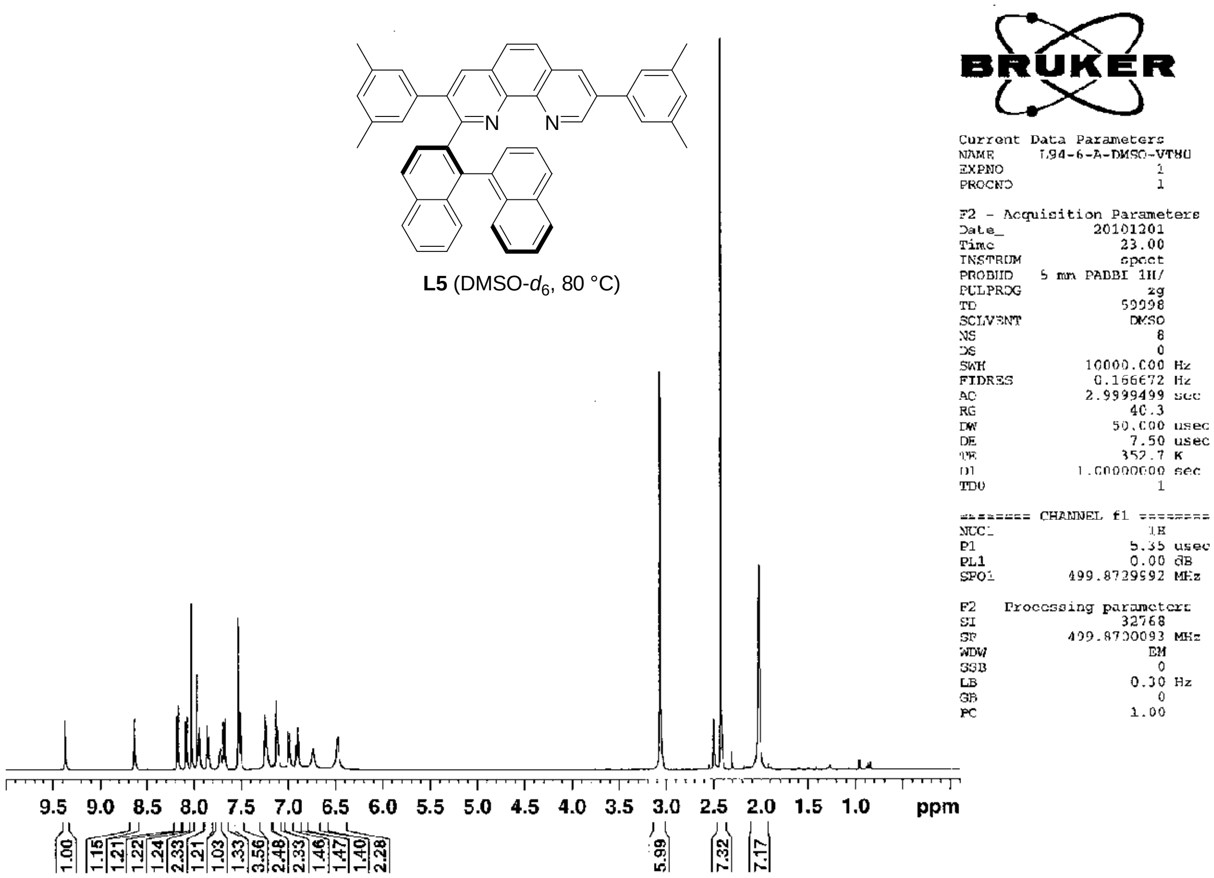
L3 (DMSO-d6, 80°C)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 1.24 1.36 2.62 5.15 1.33 2.59 2.53 2.40 2.74 2.75 2.45 1.86 5.43
BRUKER
Current Data Parameters
NAME L55-4-A-VT80-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101219
Time 16.55
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT DMSO
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 161.3
DW 50.000 usec
DE 7.50 usec
TE 353.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

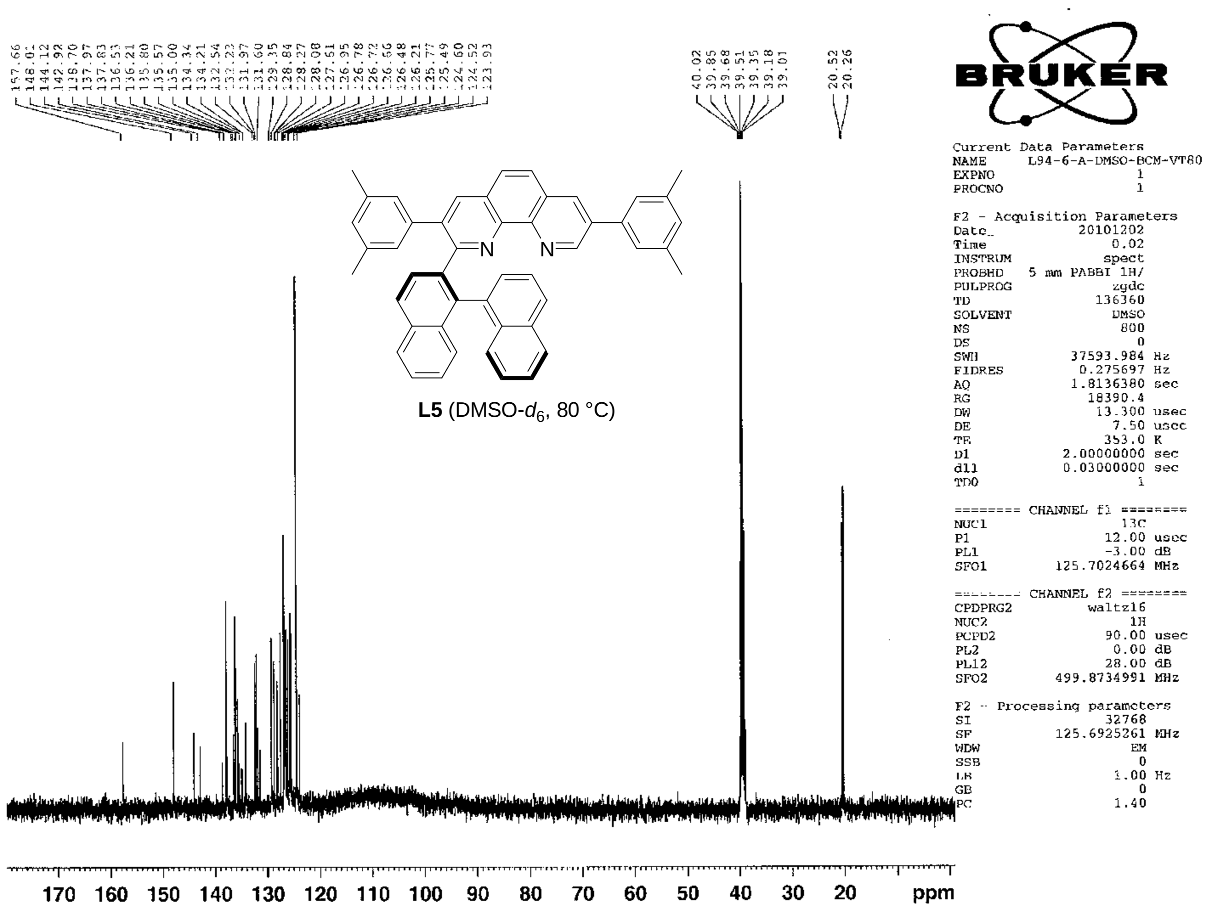


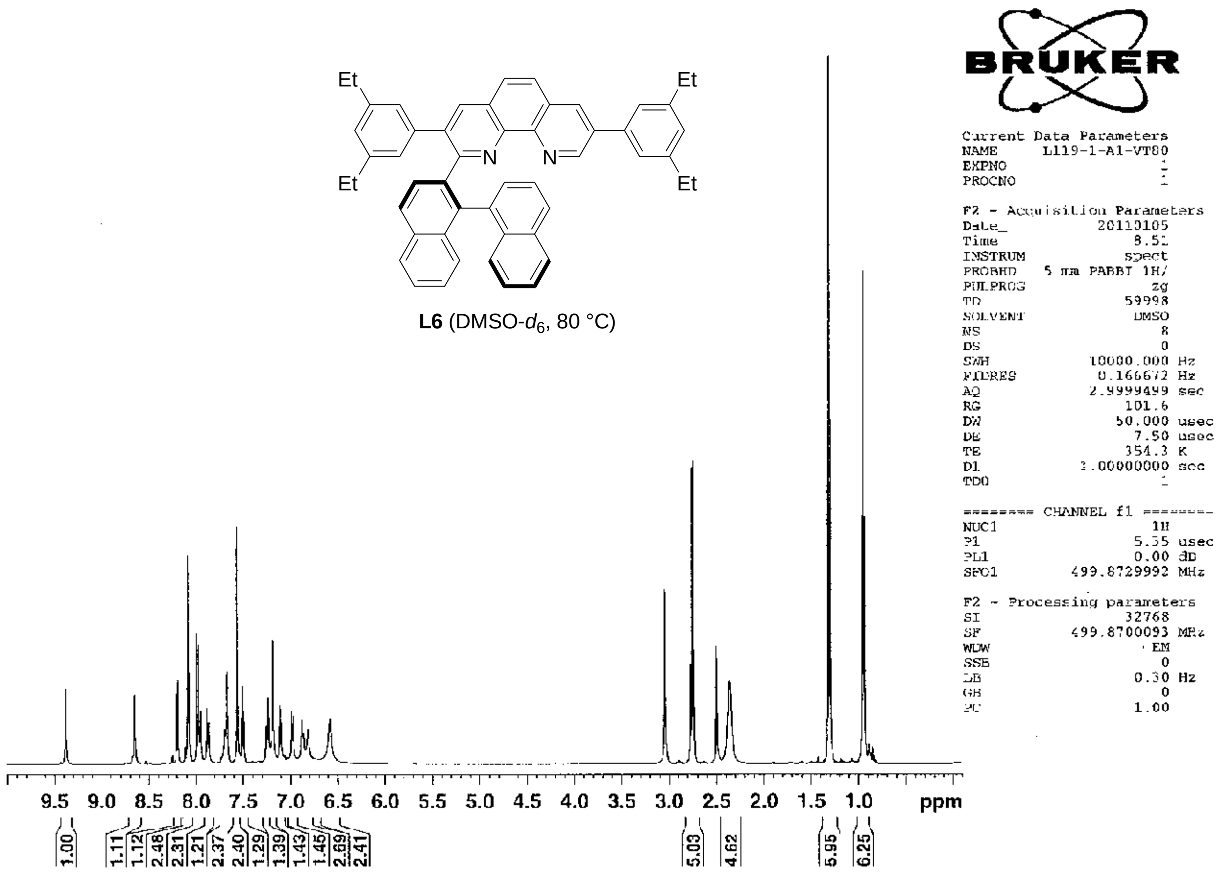
157.59 147.98 144.15 136.60 136.01 135.36 135.16 134.27 134.12 133.62 133.37 132.22 132.02 131.58 128.80 128.68 128.08 127.91 127.64 127.34 127.07 126.99 126.84 126.74 126.56 126.26 125.86 125.72 125.57 124.77 124.53 124.06
40.02 39.85 39.68 39.52 39.35 39.18 39.02
L3 (DMSO-d6, 80°C)
200 180 160 140 120 100 80 60 40 20 ppm
BRUKER
Current Data Parameters
NAME L55-4-A-BCM0-VT80-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101226
Time 13.48
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT DMSO
NS 2002
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136380 sec
RG 14596.5
DW 13.300 usec
DE 7.50 usec
TE 353.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7024654 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6925263 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


L4 (DMSO-d_6, 80°C)
BRUKER
ppm

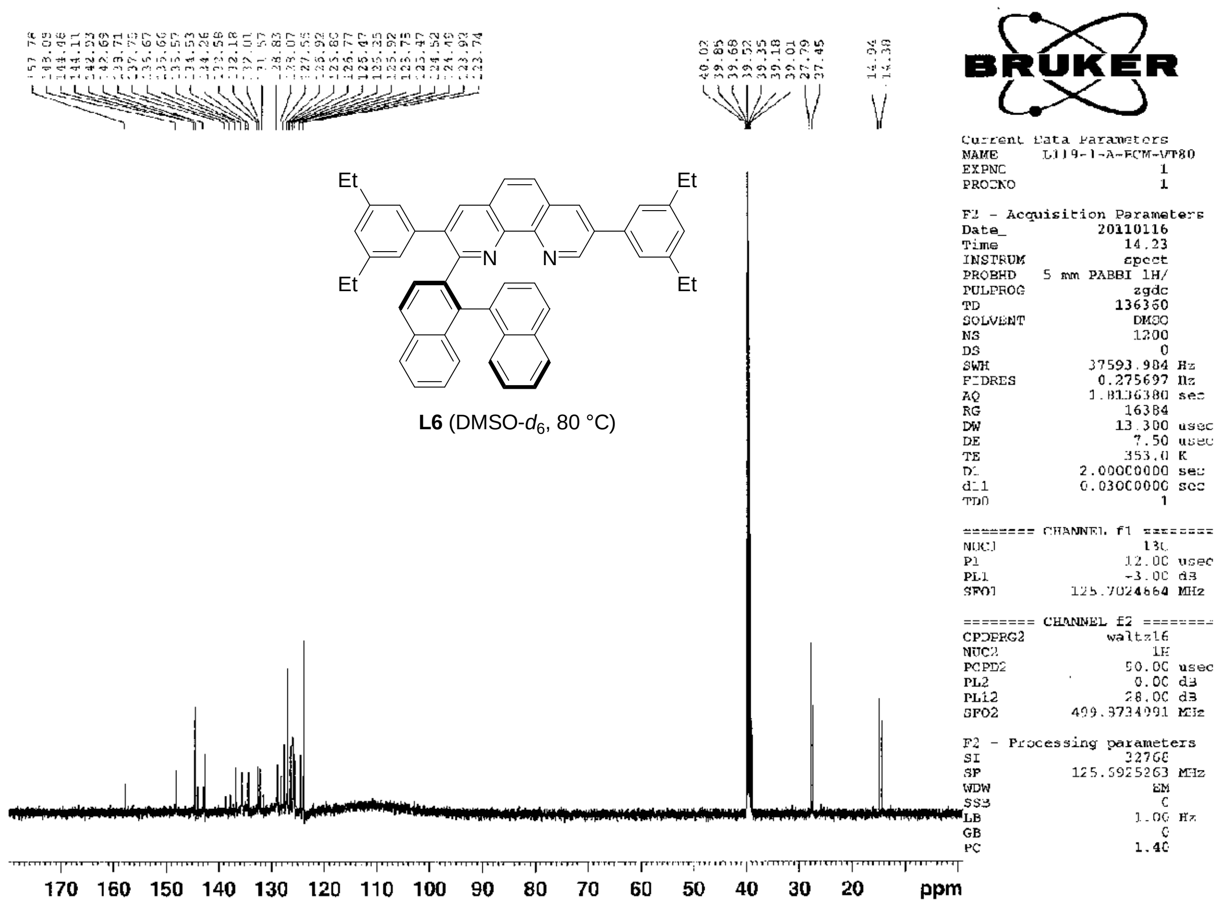

L4 (DMSO-d_6, 80°C)
BRUKER
ppm


L5 (DMSO-d_6, 80 °C)
Current Data Parameters
NAME L94-6-A-DMSO-VT80
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101201
Time 23.00
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT DMSO
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 40.3
DW 50.000 usec
DE 7.50 usec
TE 352.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729592 MHz
F2 - Processing parameters
SI 32768
SF 499.8700093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 1.15 1.21 1.22 1.24 2.33 1.21 1.03 1.33 3.56 2.48 2.33 1.46 1.47 1.40 2.28 5.99 7.32 7.17

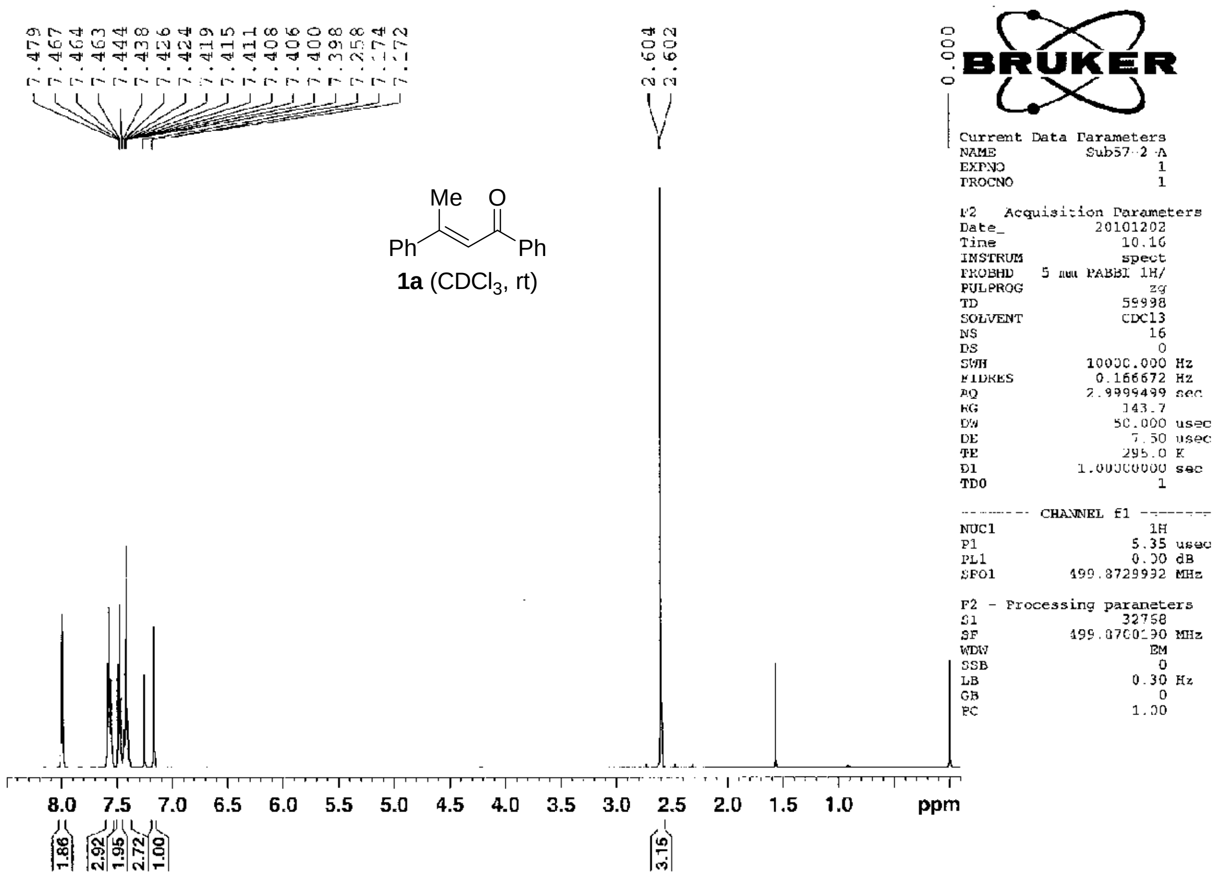

157.66 148.01 144.12 142.92 138.70 137.97 137.83 136.53 136.21 135.80 135.57 135.00 134.34 134.21 132.54 132.23 131.97 131.60 129.35 128.84 128.27 128.08 127.51 126.95 126.78 126.72 126.66 126.48 126.21 125.77 125.49 124.60 124.52 123.93
40.02 39.85 39.68 39.51 39.35 39.18 39.01
20.52 20.26
L5 (DMSO-d_6, 80 °C)
Current Data Parameters
NAME L94-6-A-DMSO-BCM-VT80
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101202
Time 0.02
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT DMSO
NS 800
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136380 sec
RG 18390.4
DW 13.300 usec
DE 7.50 usec
TE 353.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7024664 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6925261 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

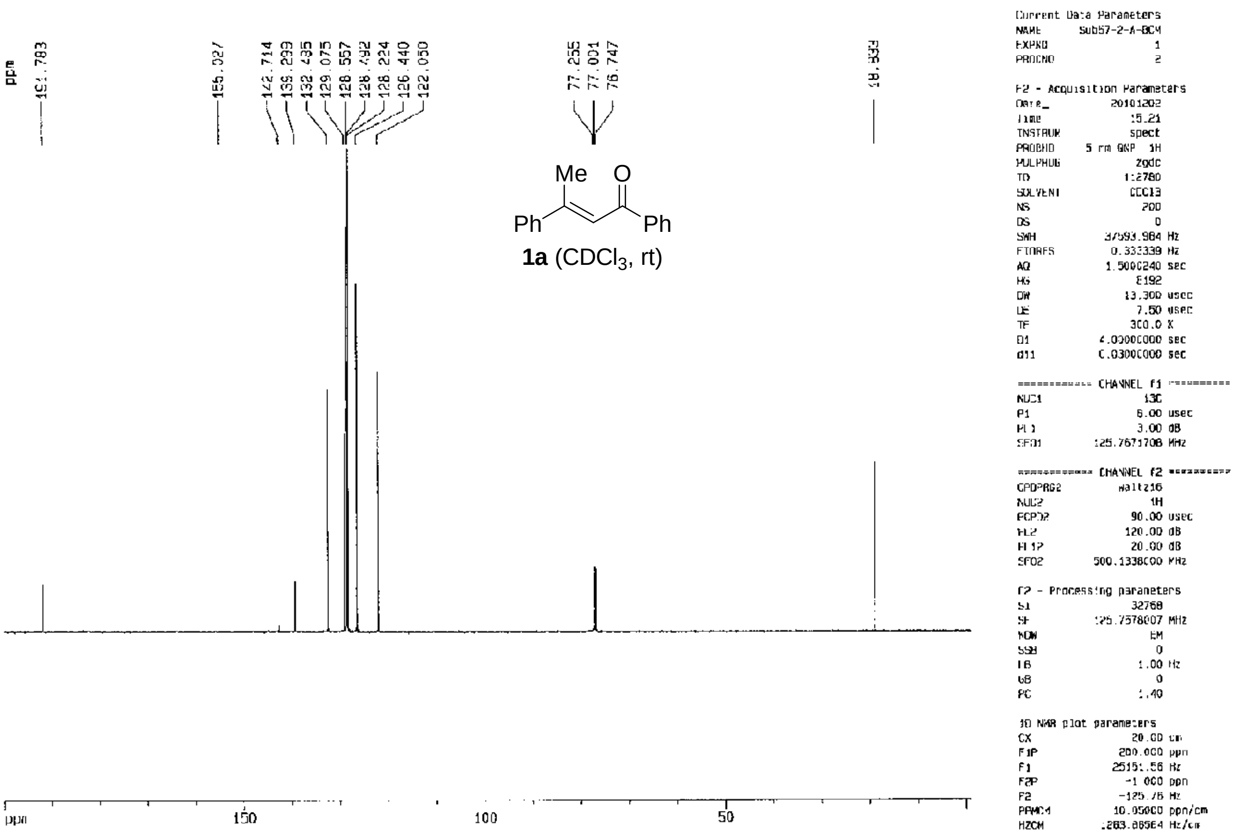


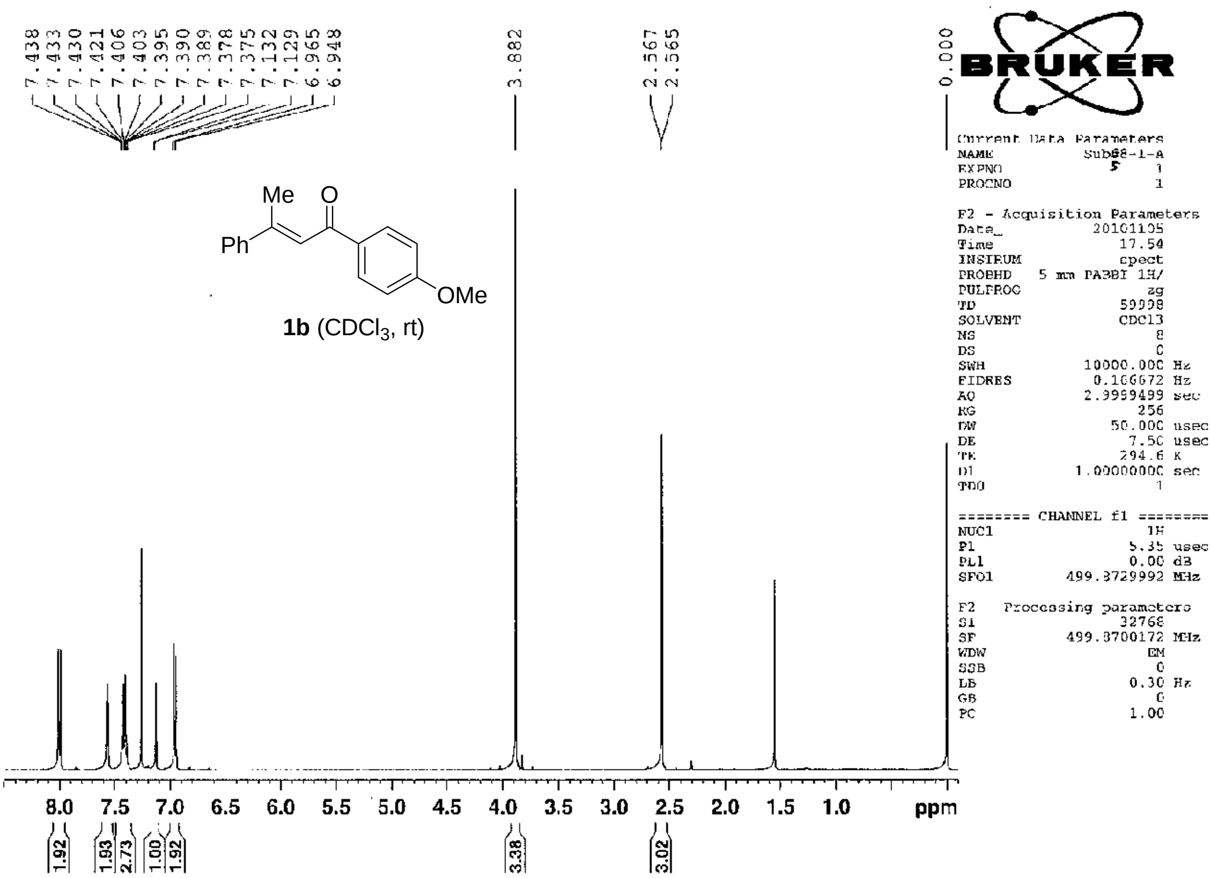
L6 (DMSO-d_6, 80 °C)

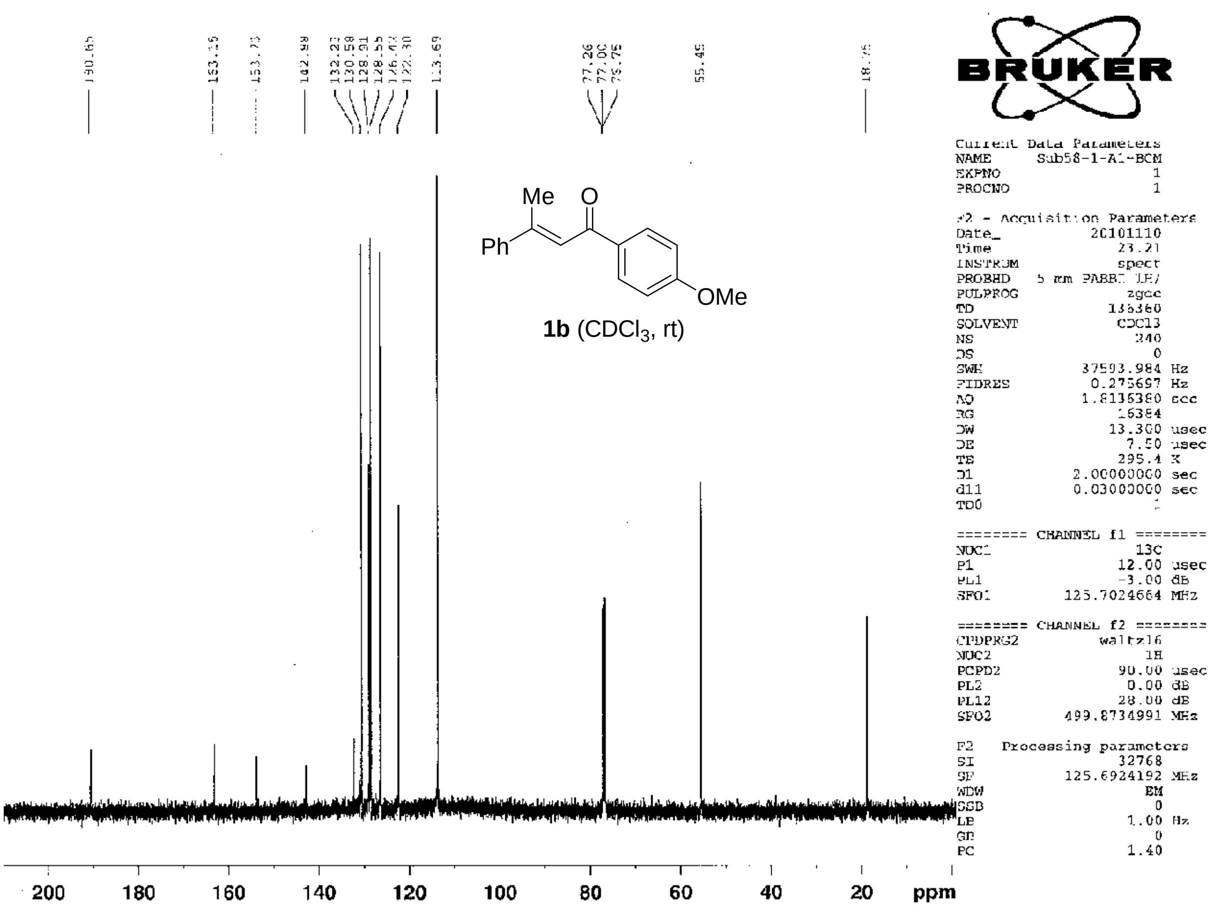


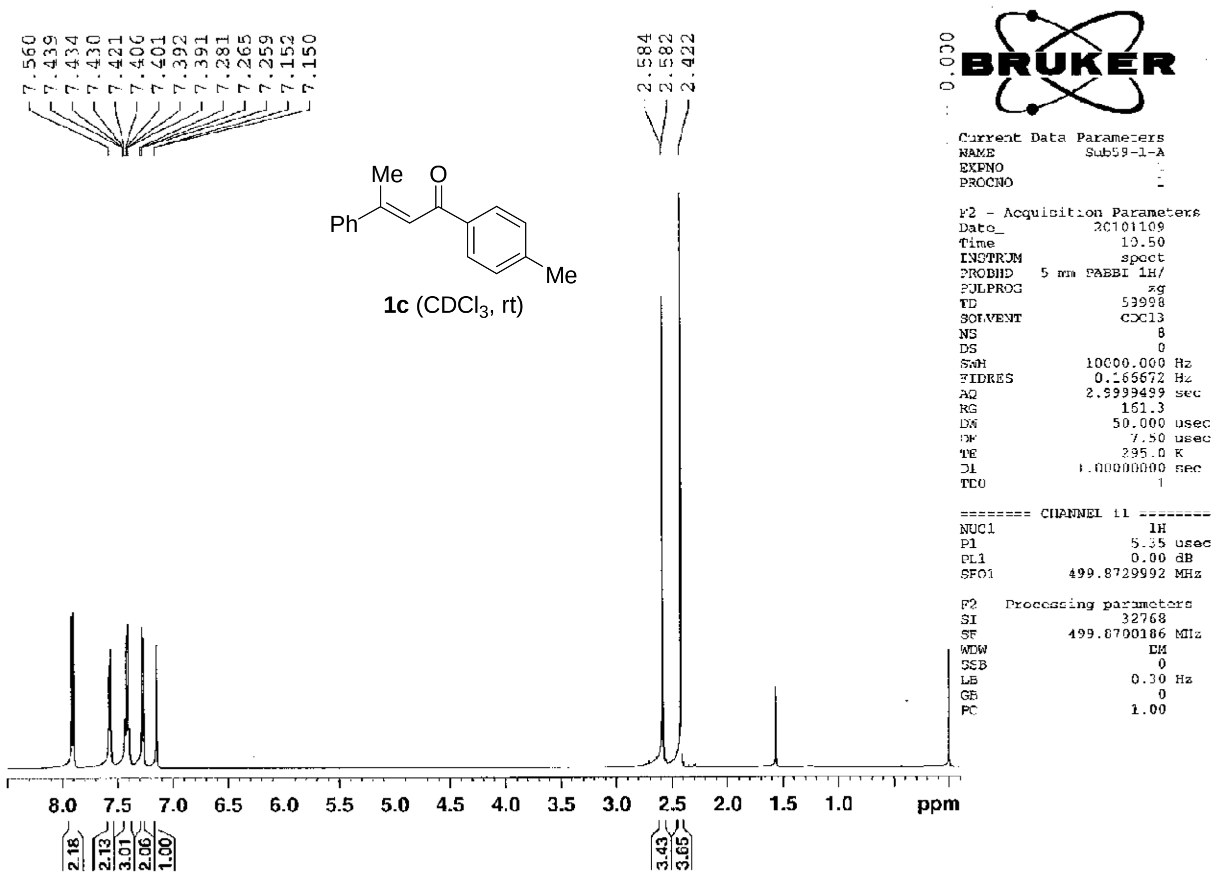
L6 (DMSO-d_6, 80 °C)

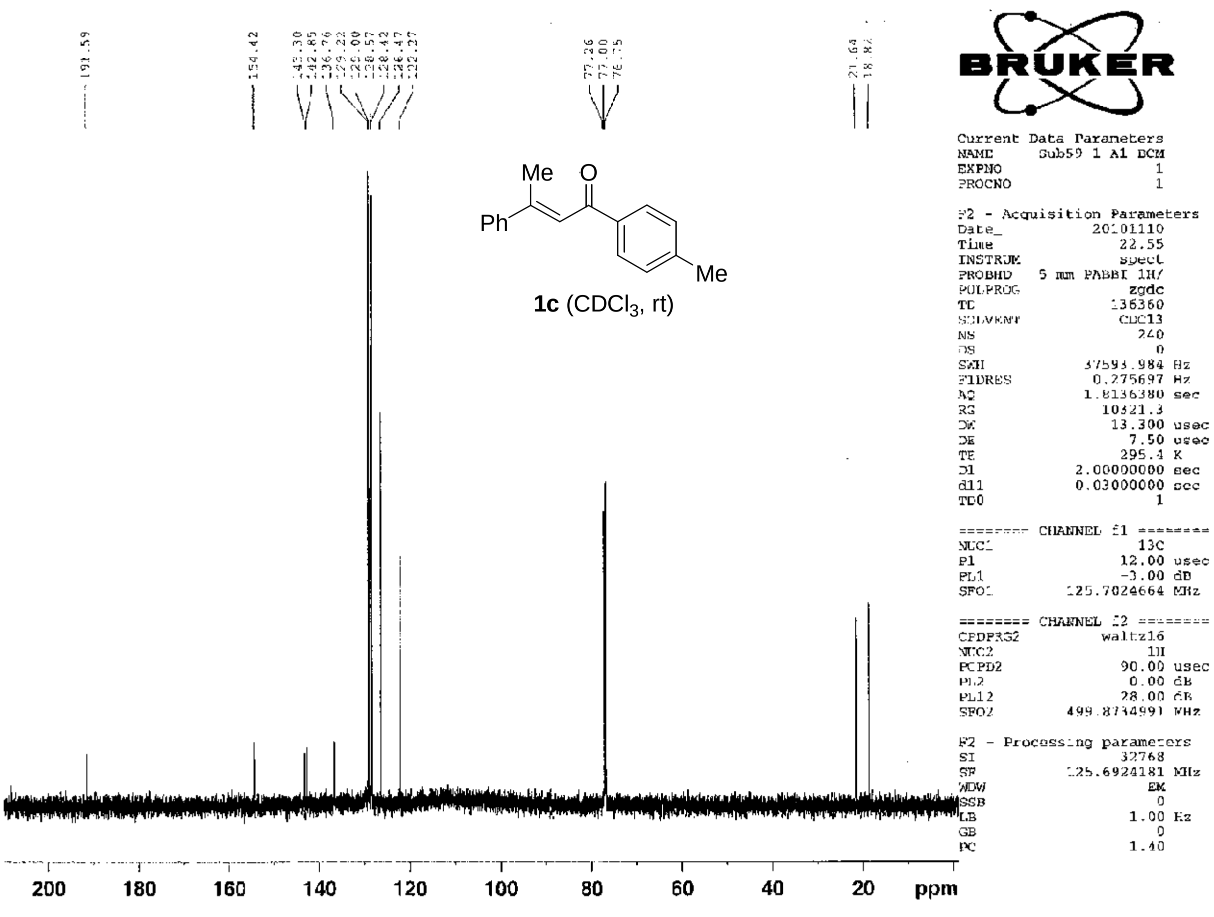

Me O
Ph Ph
1a (CDCl3, rt)
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
BRUKER
Current Data Parameters

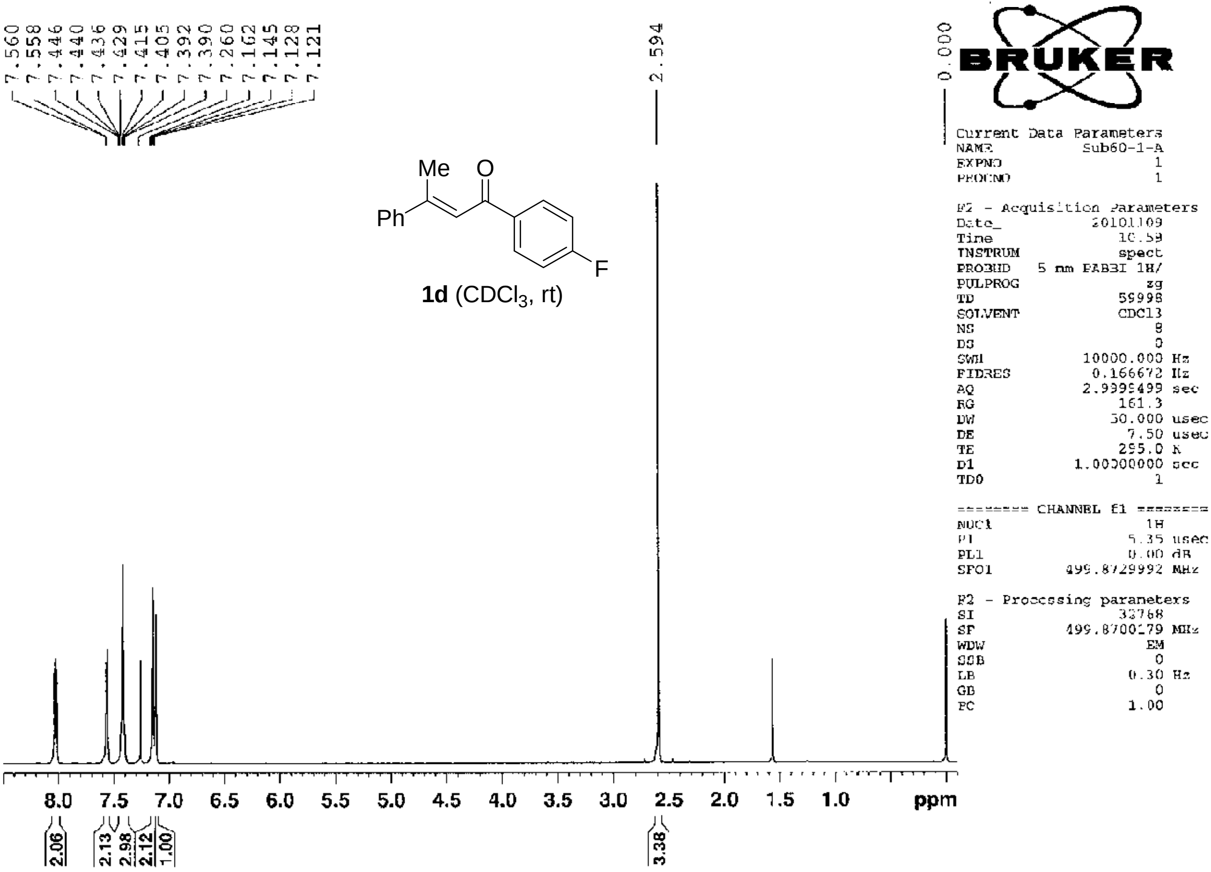

Me O
Ph Ph
1a (CDCl3, rt)
ppm 150 100 50
Current Data Parameters

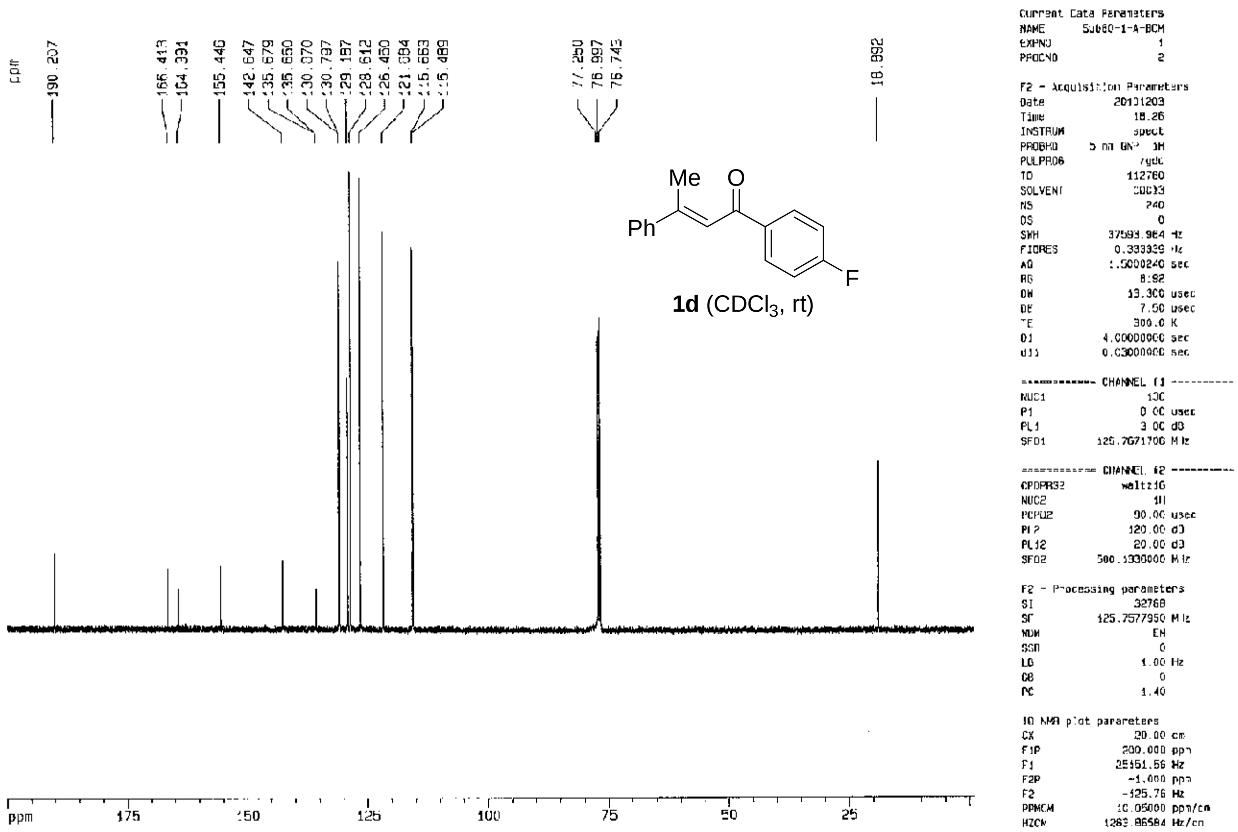

7.438
7.433
7.430
7.421
7.406
7.403
7.395
7.390
7.389
7.378
7.375
7.132
7.129
6.965
6.948
3.882
2.567
2.565
0.000
Me
O
Ph
OMe
1b (CDCl3, rt)
BRUKER
Current Data Parameters
NAME Sub58-1-A
EXPNO 5 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101105
Time 17.54
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 256
DW 50.000 usec
DE 7.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.92
1.93
2.73
1.00
1.92
3.38
3.02

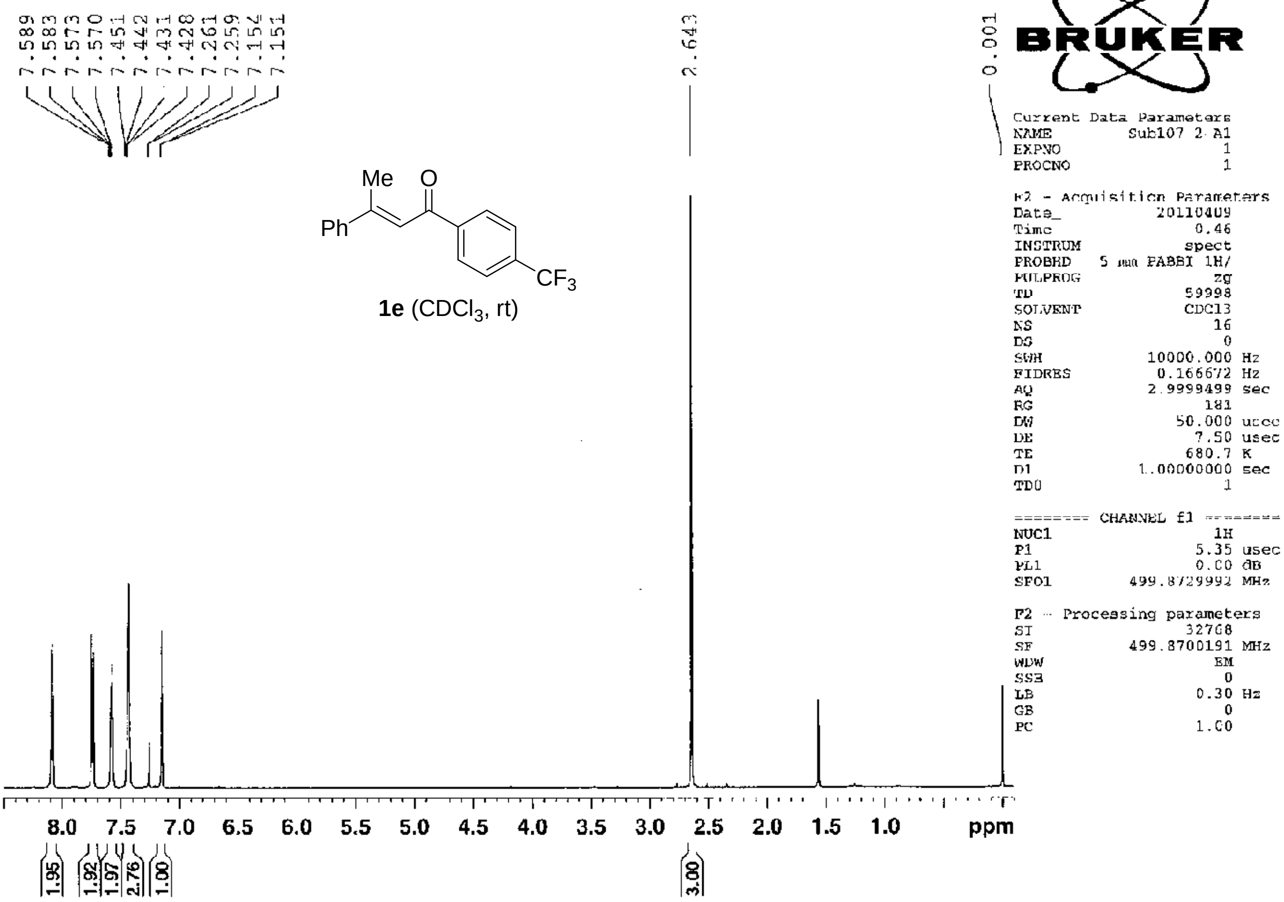

190.65
163.35
153.73
142.98
132.22
130.58
128.91
128.55
126.42
122.30
113.69
77.26
77.00
76.75
55.45
18.75
Me
O
Ph
OMe
1b (CDCl3, rt)
BRUKER
Current Data Parameters
NAME Sub58-1-A1-BCM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101110
Time 23.21
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg
TD 136360
SOLVENT CDCl3
NS 240
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136380 sec
RG 16384
DW 13.300 usec
DE 7.50 usec
TE 295.4 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7024664 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6924192 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm

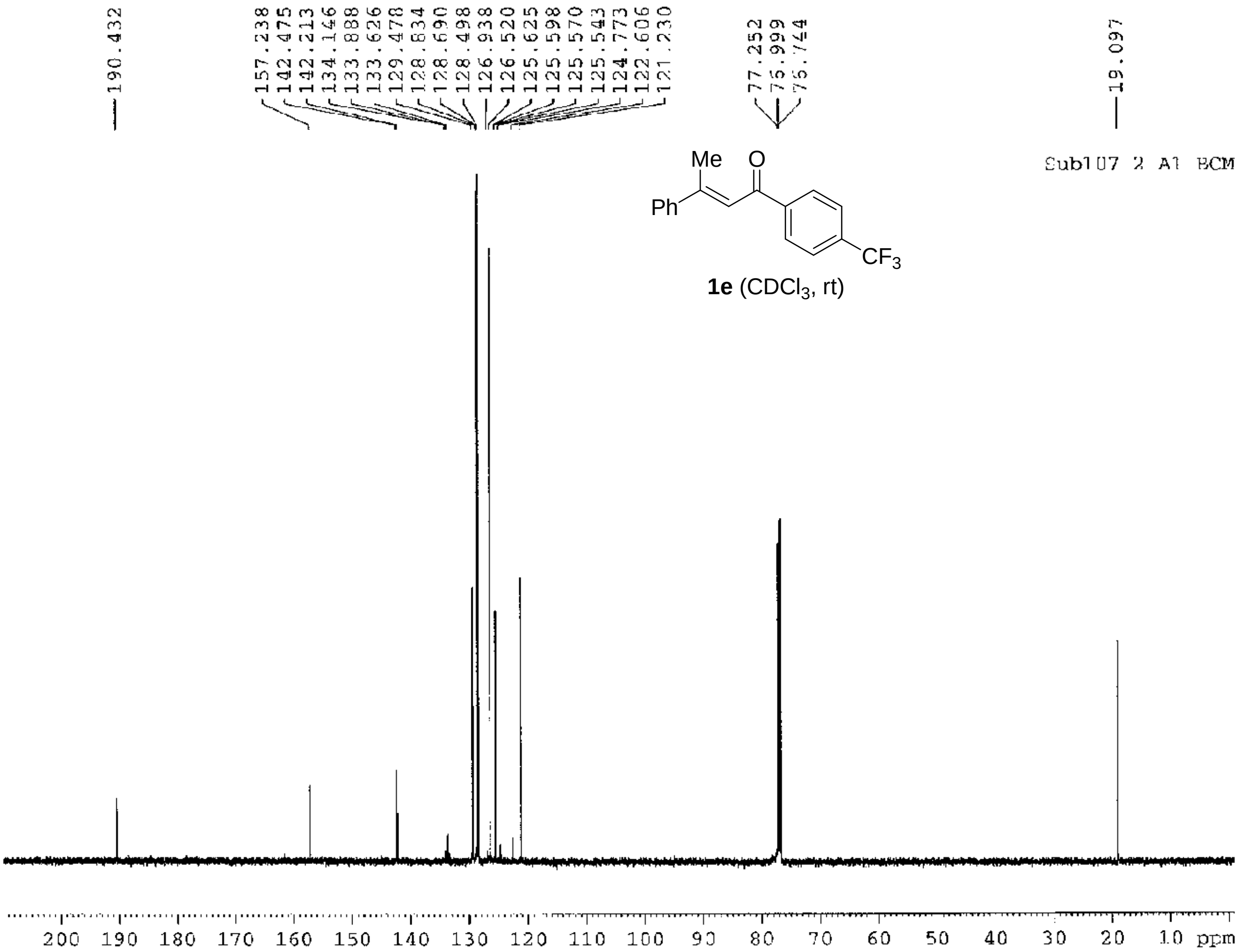

7.560
7.439
7.434
7.430
7.421
7.406
7.401
7.392
7.391
7.281
7.265
7.259
7.152
7.150
2.584
2.582
2.422
0.000
BRUKER
Me
O
Ph
Me
1c (CDCl3, rt)
Current Data Parameters
NAME Sub59-1-A
F2 - Acquisition Parameters
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 161.3
DW 50.000 usec
DE 7.50 usec
TE 295.0 K
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700186 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.18
2.13
3.01
2.06
1.00
3.43
3.65

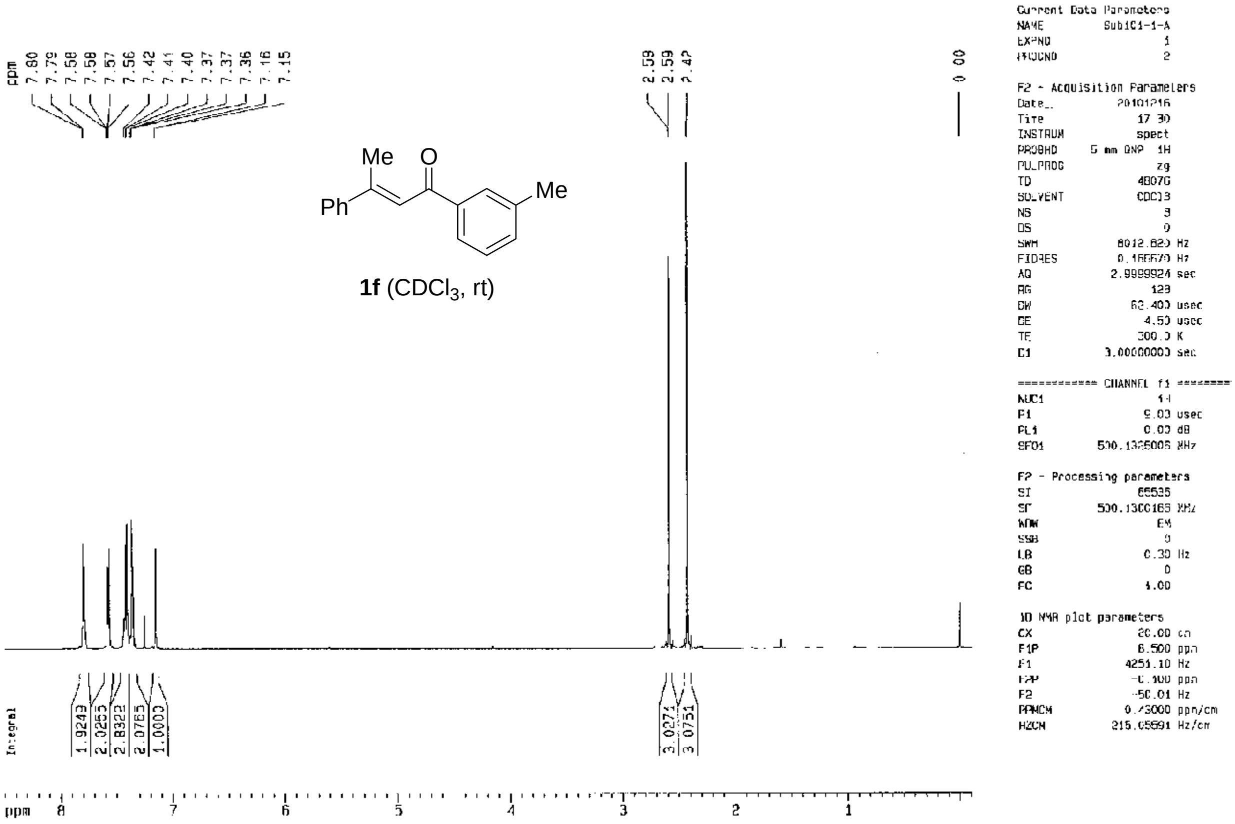

191.59
154.42
77.26
77.00
76.75
21.64
18.82
BRUKER
Me
O
Ph
Me
1c (CDCl3, rt)
Current Data Parameters
NAME Sub59 1 A1 DCM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101110
Time 22.55
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT CDCl3
NS 240
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136380 sec
RG 10321.3
DW 13.300 usec
DE 7.50 usec
TE 295.4 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7024664 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6924181 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

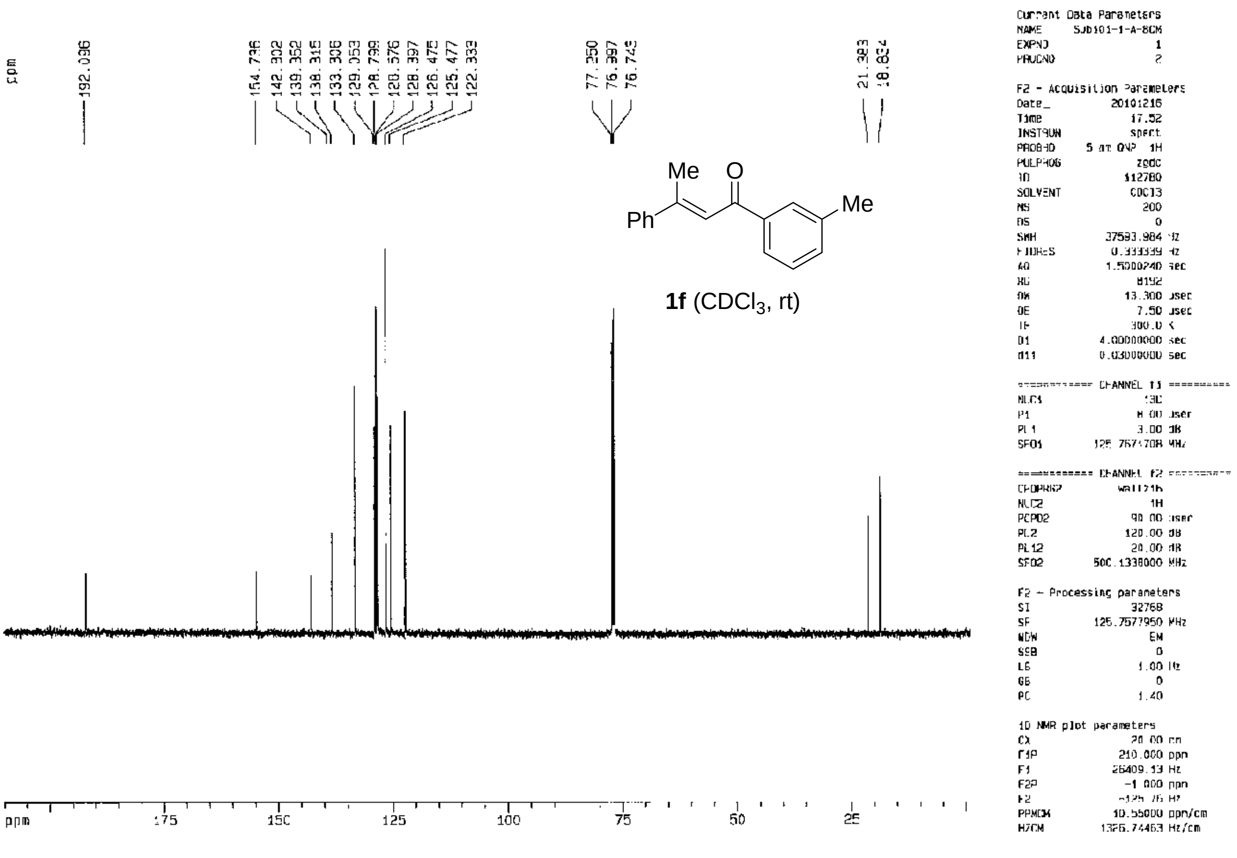

7.560
7.558
7.446
7.440
7.436
7.429
7.415
7.405
7.392
7.390
7.260
7.162
7.145
7.128
7.121
2.594
0.000
BRUKER
Me
O
Ph
F
1d (CDCl3, rt)
Current Data Parameters
NAME Sub60-1-A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101109
INSTRUM spect
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 161.3
DE 7.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.06
2.13
2.98
2.12
1.00
3.38

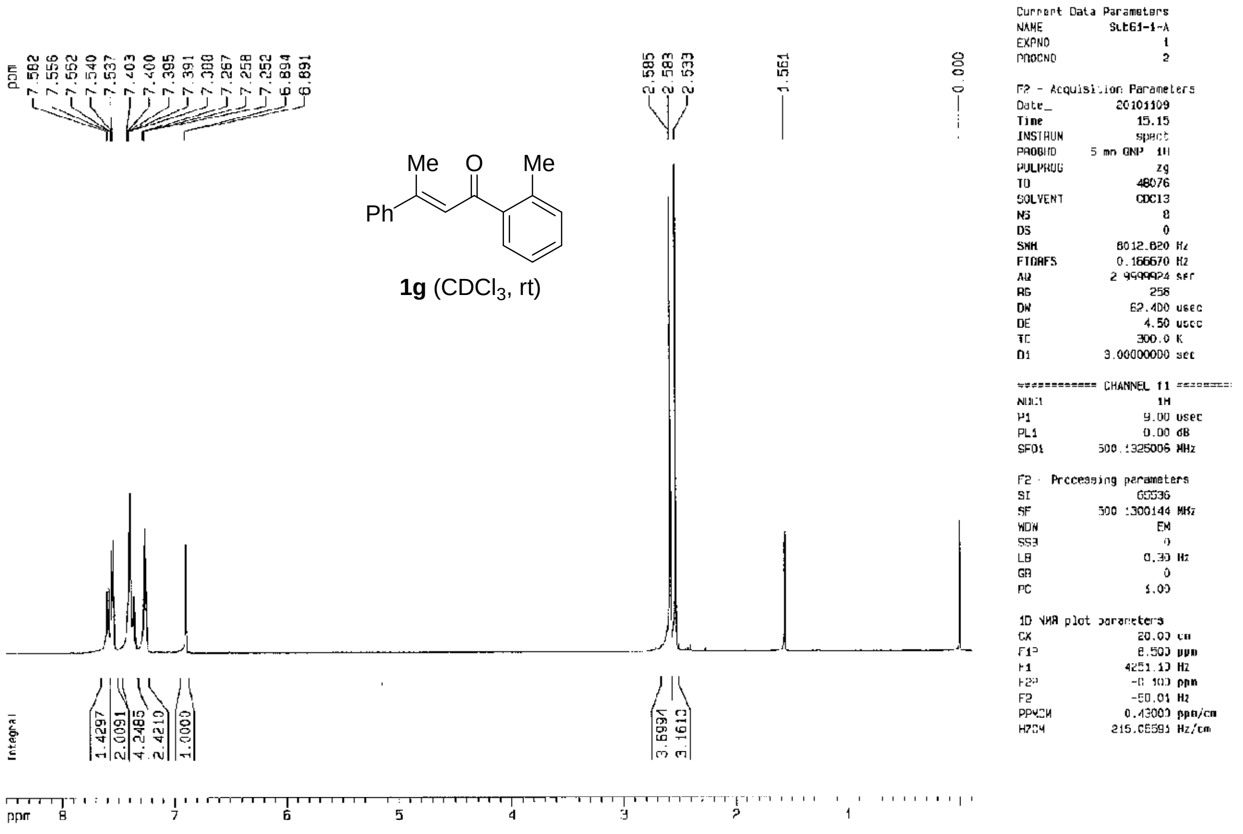

190.207
166.413
164.391
155.440
142.647
135.679
135.650
130.870
130.797
129.187
128.612
126.450
121.684
115.663
115.489
77.250
76.997
76.743
18.892
Me
O
Ph
F
1d (CDCl3, rt)
Current Data Parameters
NAME Sub60-1-A-BCM
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date 20101203
Time 18.26
INSTRUM spect
TD 112760
NS 240
DS 0
SWH 37593.984 Hz
FIDRES 0.333339 Hz
AQ 1.5000240 sec
RG 8.92
DW 13.300 usec
DE 7.50 usec
TE 300.0 K
D1 4.00000000 sec
CHANNEL f1
NUC1 13C
SFO1 125.7671700 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 120.00 dB
PL12 20.00 dB
SFO2 500.1330000 MHz
F2 - Processing parameters
SI 32768
SF 125.7577950 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 25151.56 Hz
F2P -1.000 ppm
F2 -125.76 Hz
PPMCM 10.05000 ppm/cm
HZCM 1263.86584 Hz/cm
ppm
175
150
125
100
75
50
25

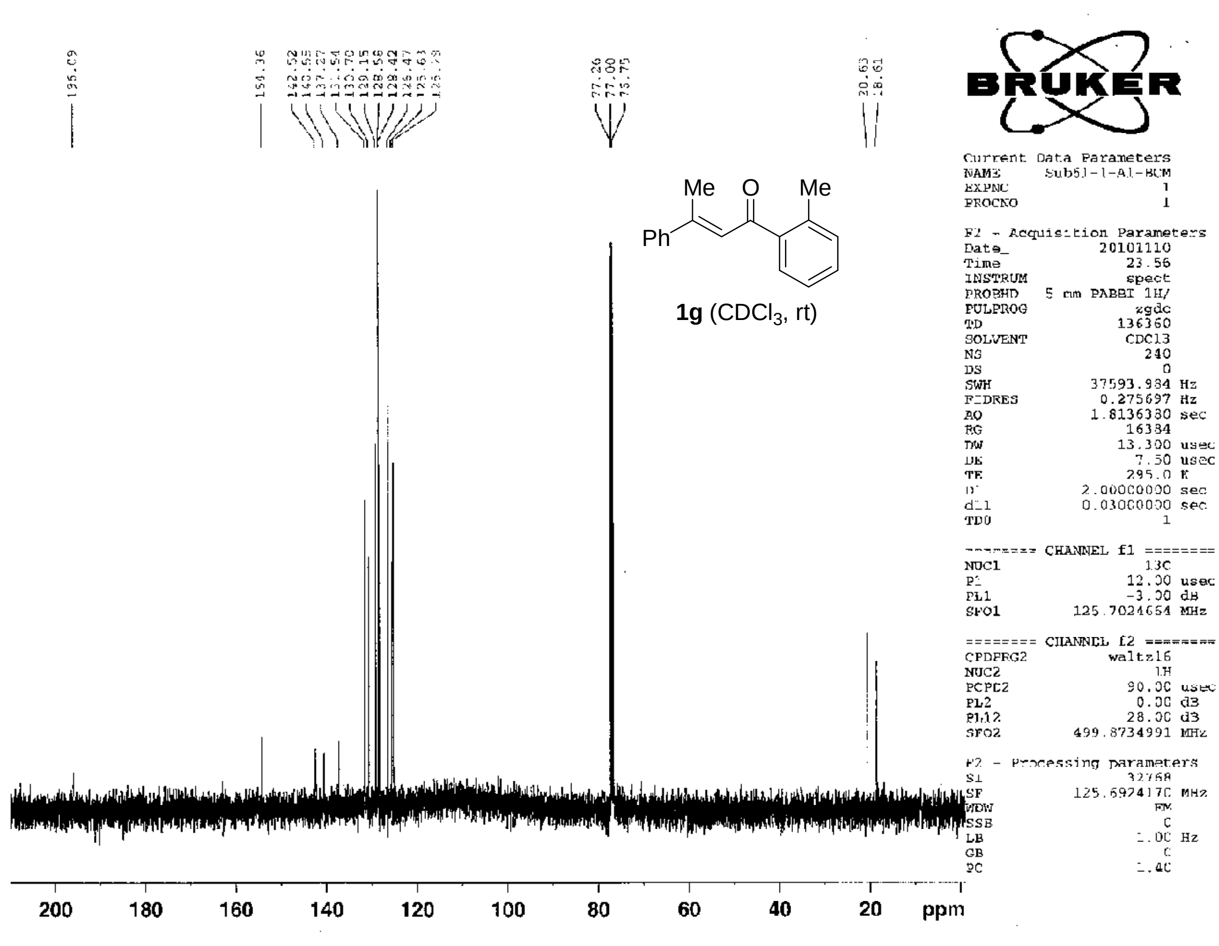

7.589
7.583
7.573
7.570
7.451
7.442
7.431
7.428
7.261
7.259
7.154
7.151
2.643
0.001
Me
O
Ph
CF3
1e (CDCl3, rt)
Current Data Parameters
NAME Sub107 2 A1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110409
Time 0.46
INSTRUM spect
PROBHD 5 mm FABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 181
DW 50.000 usec
DE 7.50 usec
TE 680.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700191 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.95
1.92
1.97
2.76
1.00
3.00

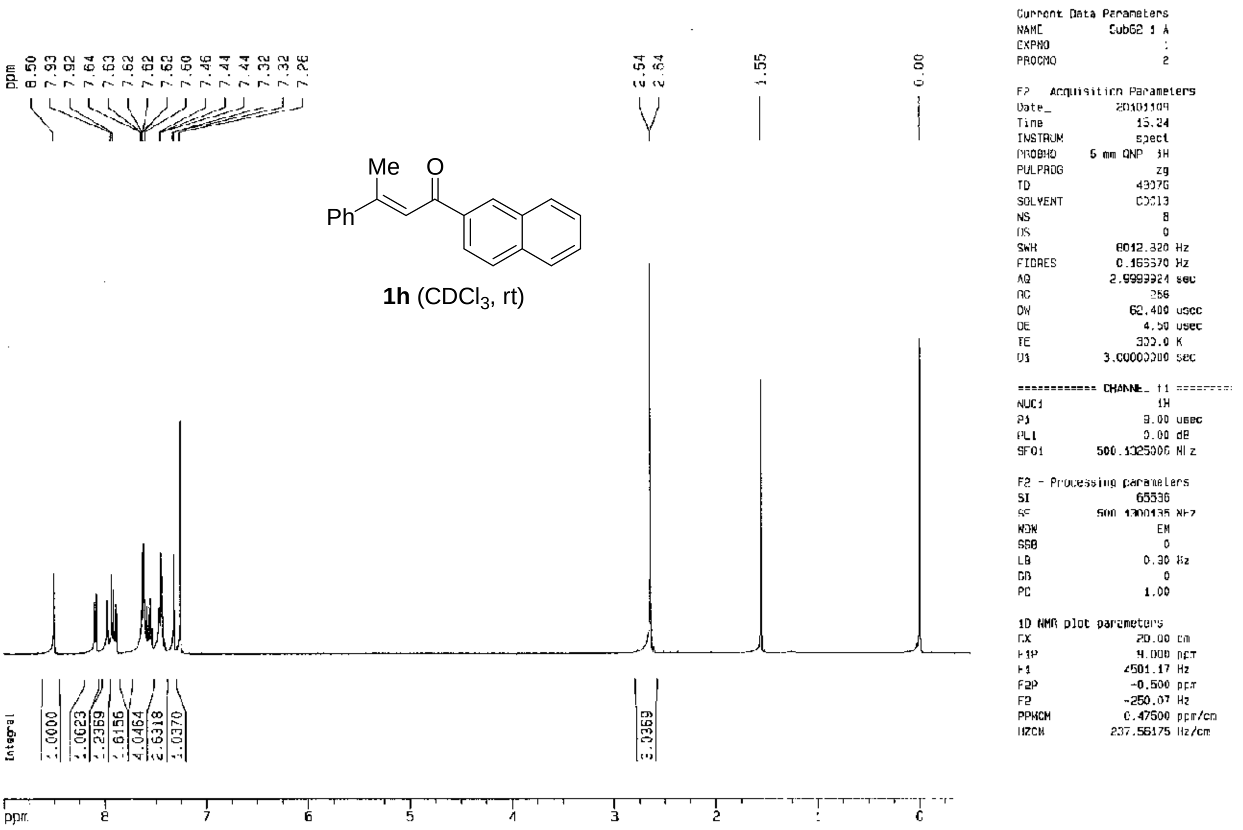

190.432
157.238
142.475
142.213
134.146
133.888
133.626
129.478
128.834
128.690
128.498
126.938
126.520
125.625
125.598
125.570
125.543
124.773
122.606
121.230
77.252
76.999
76.744
19.097
Sub107 2 A1 BCM
Me
O
Ph
CF3
1e (CDCl3, rt)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

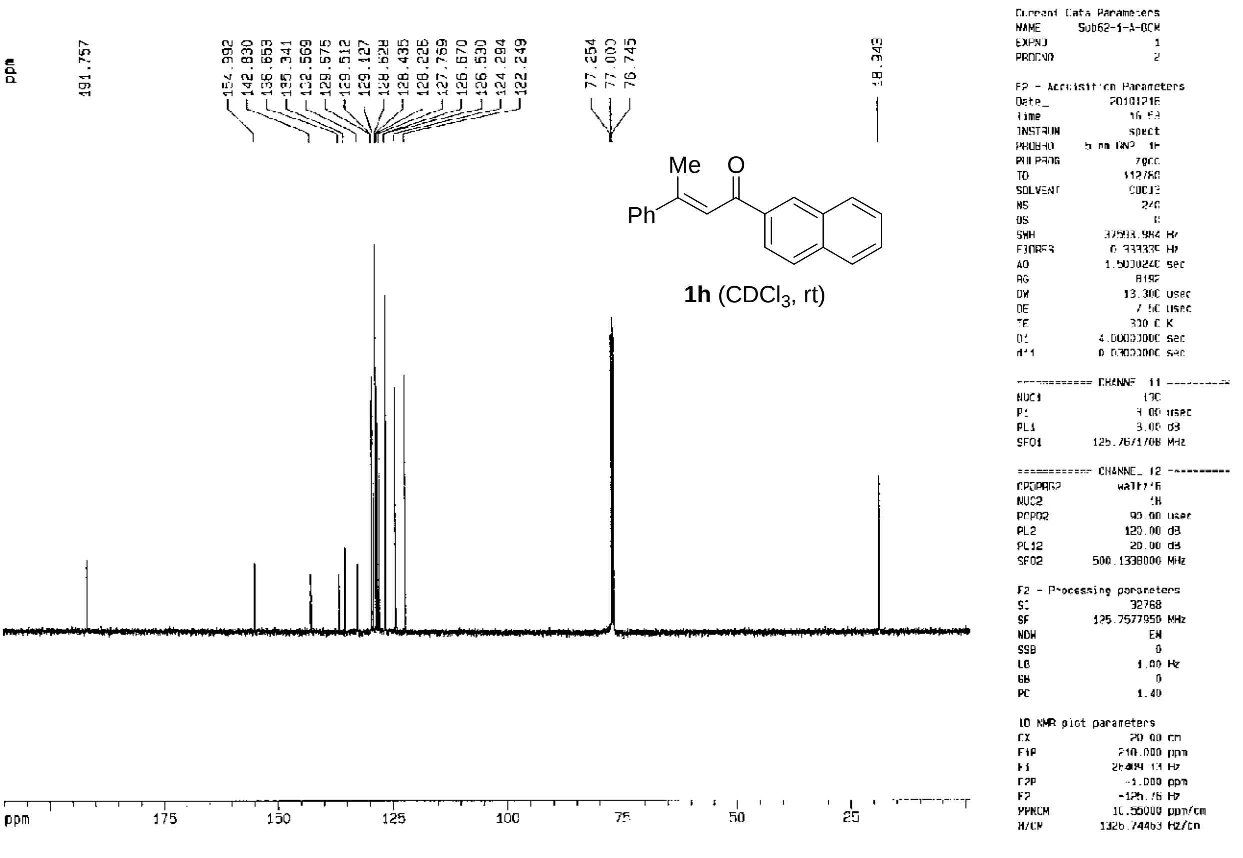

Me
O
Ph
Me
1f (CDCl3, rt)
7.80 7.79 7.58 7.58 7.57 7.56 7.42 7.41 7.40 7.37 7.37 7.36 7.16 7.15
2.59 2.59 2.42
0.00
Integral
1.9243 2.0265 2.8322 2.0765 1.0000
3.0271 3.0751
ppm 8 7 6 5 4 3 2 1

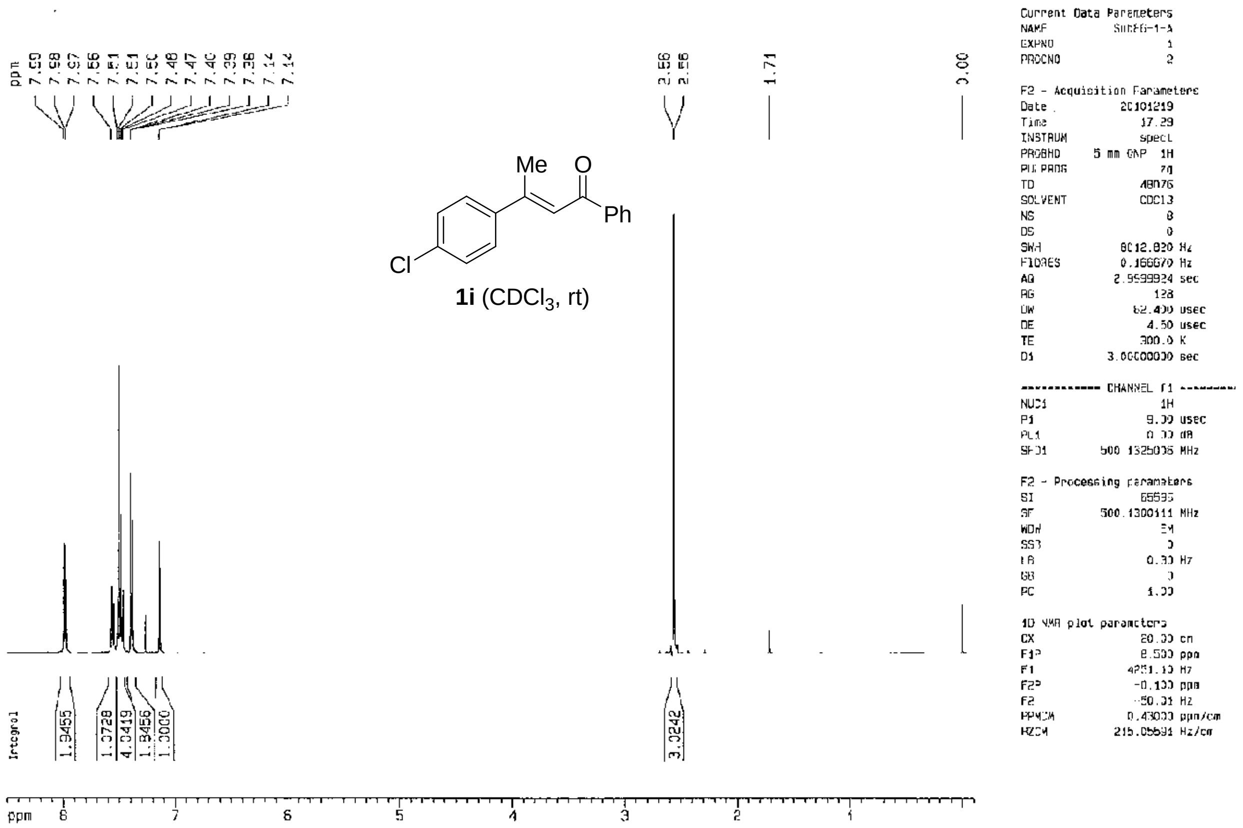

192.036
154.736 142.302 139.352 138.315 133.306 129.053 128.799 128.576 128.397 126.475 125.477 122.333
77.250 76.997 76.743
21.383 18.624
Me
O
Ph
Me
1f (CDCl3, rt)
ppm 175 150 125 100 75 50 25

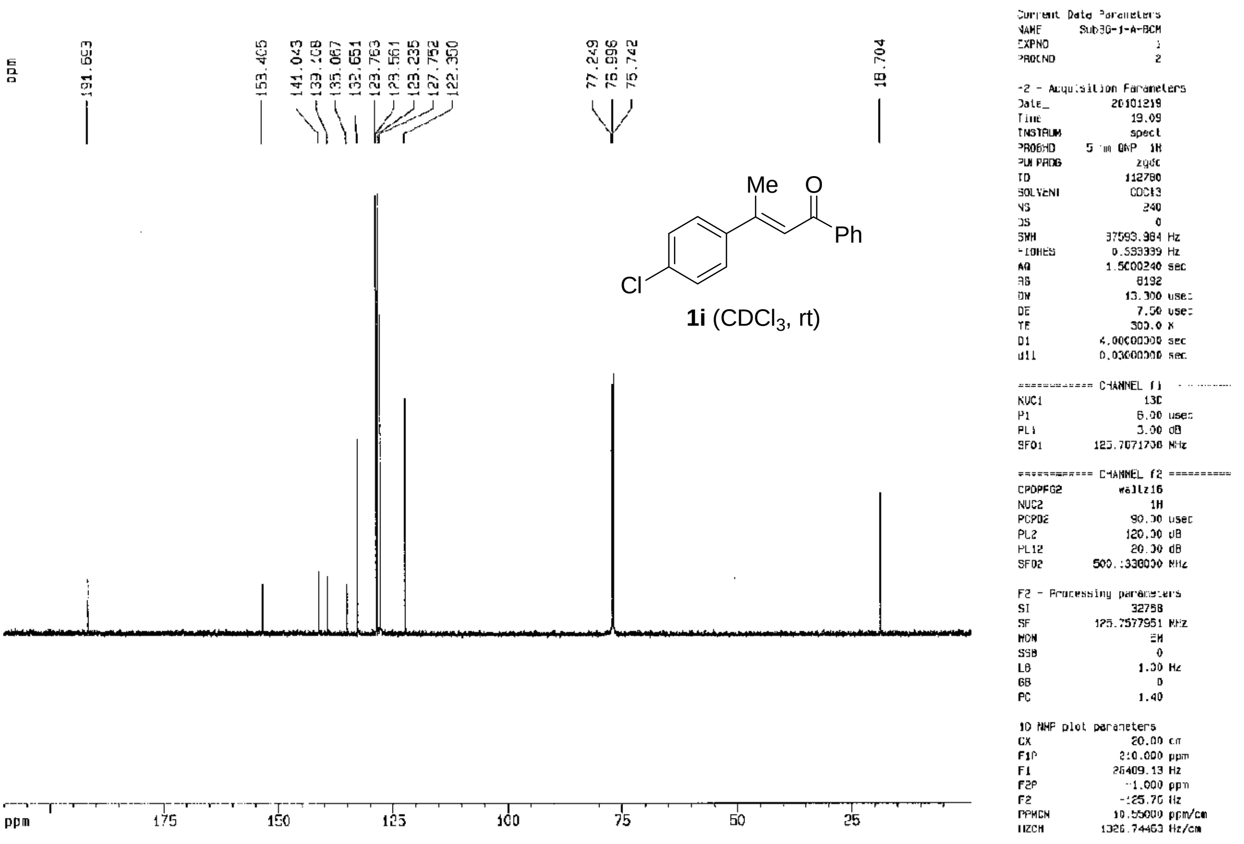

7.582 7.556 7.552 7.540 7.537 7.403 7.400 7.395 7.391 7.388 7.267 7.258 7.252 6.894 6.891
2.585 2.583 2.533
1.561
0.000
Me O Me
Ph
1g (CDCl3, rt)
Integral 1.4297 2.0091 4.2485 2.4210 1.0000 3.6991 3.1613
ppm 8 7 6 5 4 3 2 1
Current Data Parameters
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20101109
Time 15.15
INSTRUM spect
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
RG 258
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec
CHANNEL f1
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
F2 - Processing parameters
SI 65536
SF 500.1300144 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 8.500 ppm
F1 4251.13 Hz
PPMCM 0.43000 ppm/cm
HZCM 215.05591 Hz/cm

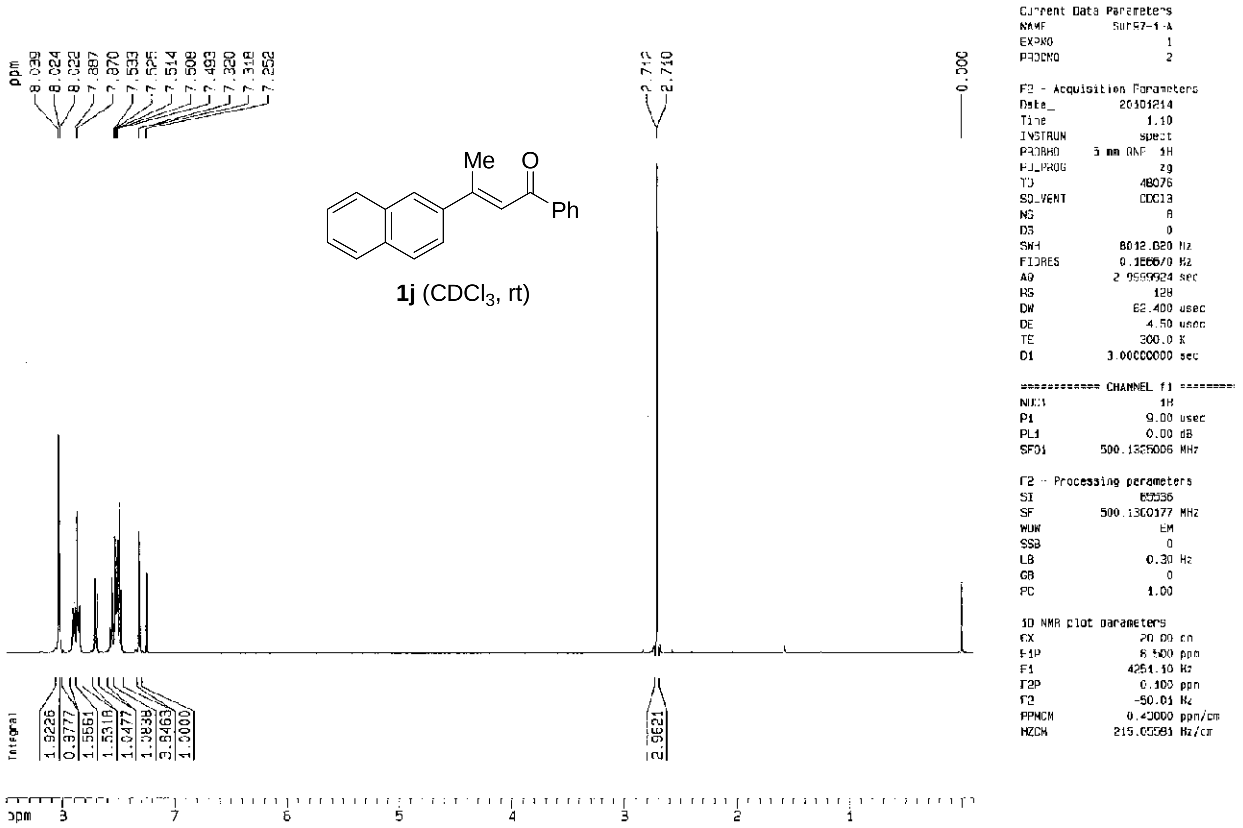

195.09
154.36 142.52 140.55 137.27 131.54 130.70 129.15 128.56 128.42 126.47 125.63 125.23
77.26 77.00 76.75
20.63 18.61
Me O Me
Ph
1g (CDCl3, rt)
BRUKER
Current Data Parameters
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101110
Time 23.56
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT CDCl3
NS 240
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136380 sec
RG 16384
DW 13.300 usec
DE 7.50 usec
TE 295.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7024654 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6924170 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

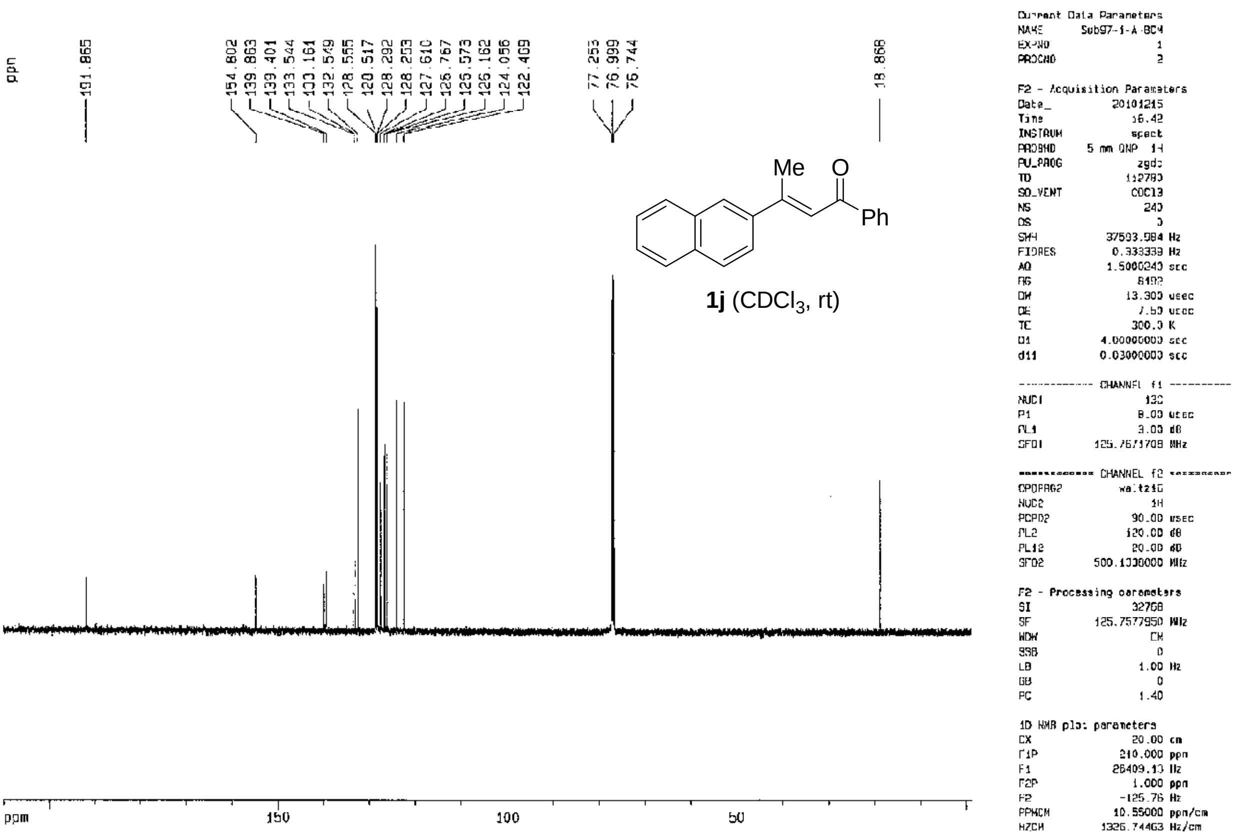

ppm
8.50 7.93 7.92 7.64 7.63 7.62 7.62 7.62 7.60 7.46 7.44 7.32 7.32 7.26
2.64 2.64
1.55
0.00
Me
O
Ph
1h (CDCl3, rt)
Integral
1.0000 1.0623 1.2369 1.6156 4.0464 2.6318 1.0370
3.0369
ppm 8 7 6 5 4 3 2 1 0
Current Data Parameters
NAME SubG2 1 A
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20101109
Time 15.24
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 256
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
F2 - Processing parameters
SI 65536
SF 500.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 9.000 ppm
F1 4501.17 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.47500 ppm/cm
HZCM 237.56175 Hz/cm

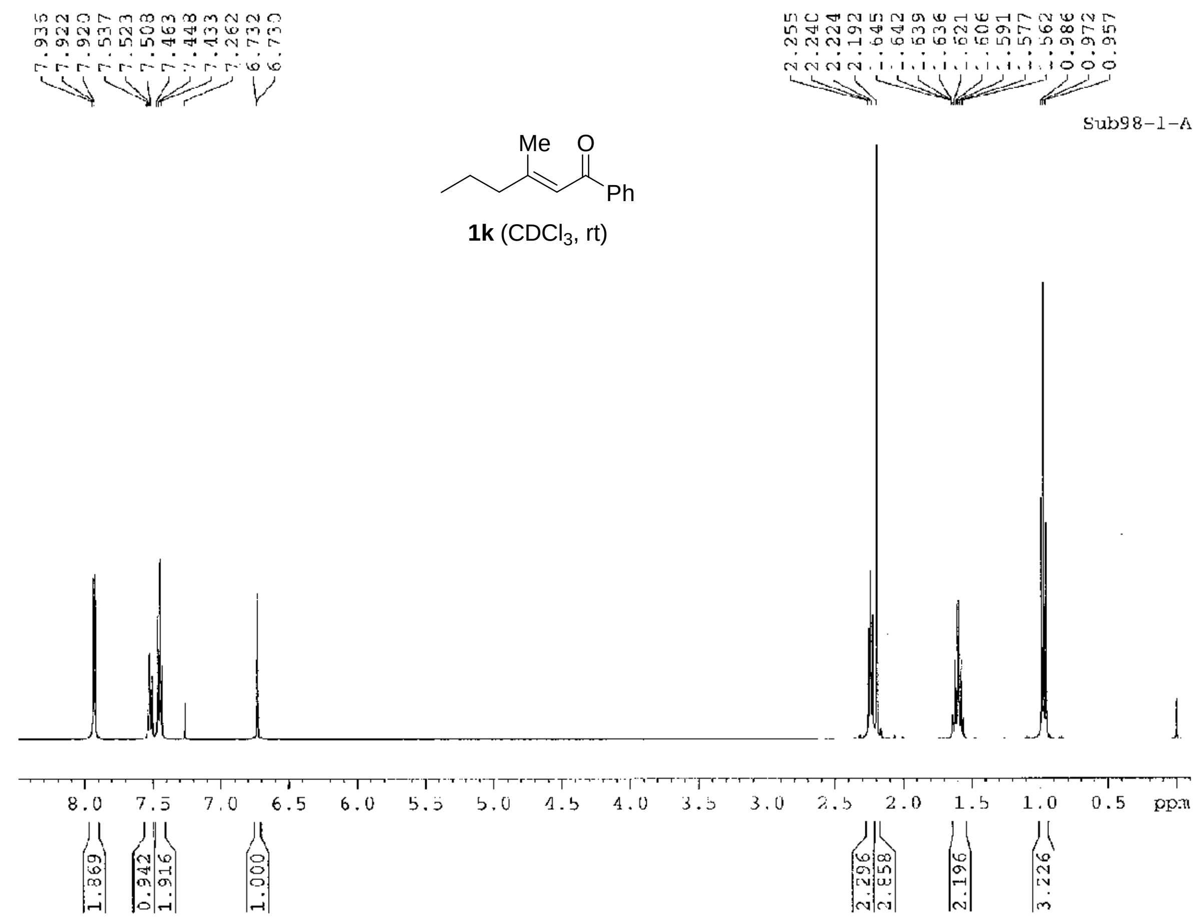

ppm
191.757
154.992 142.830 136.653 135.341 132.569 129.675 129.512 129.127 128.628 128.435 128.226 127.769 126.670 126.530 124.294 122.249
77.254 77.000 76.745
18.943
Me
O
Ph
1h (CDCl3, rt)
ppm 175 150 125 100 75 50 25
Current Data Parameters
NAME Sub62-1-A-BCM
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20101216
TD 112760
SOLVENT CDCl3
NS 240
DS 0
SWH 37593.984 Hz
FIDRES 0.333335 Hz
AQ 1.5000240 sec
DW 13.300 usec
DE 7.50 usec
TE 300.0 K
D1 4.00000000 sec
d11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 3.00 dB
SFO1 125.7671708 MHz
======== CHANNEL f2 ========
NUC2 1H
PCPD2 90.00 usec
PL2 120.00 dB
PL12 20.00 dB
SFO2 500.1338000 MHz
F2 - Processing parameters
SI 32768
SF 125.7577950 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 210.000 ppm
F2P -1.000 ppm
PPMCM 10.55000 ppm/cm

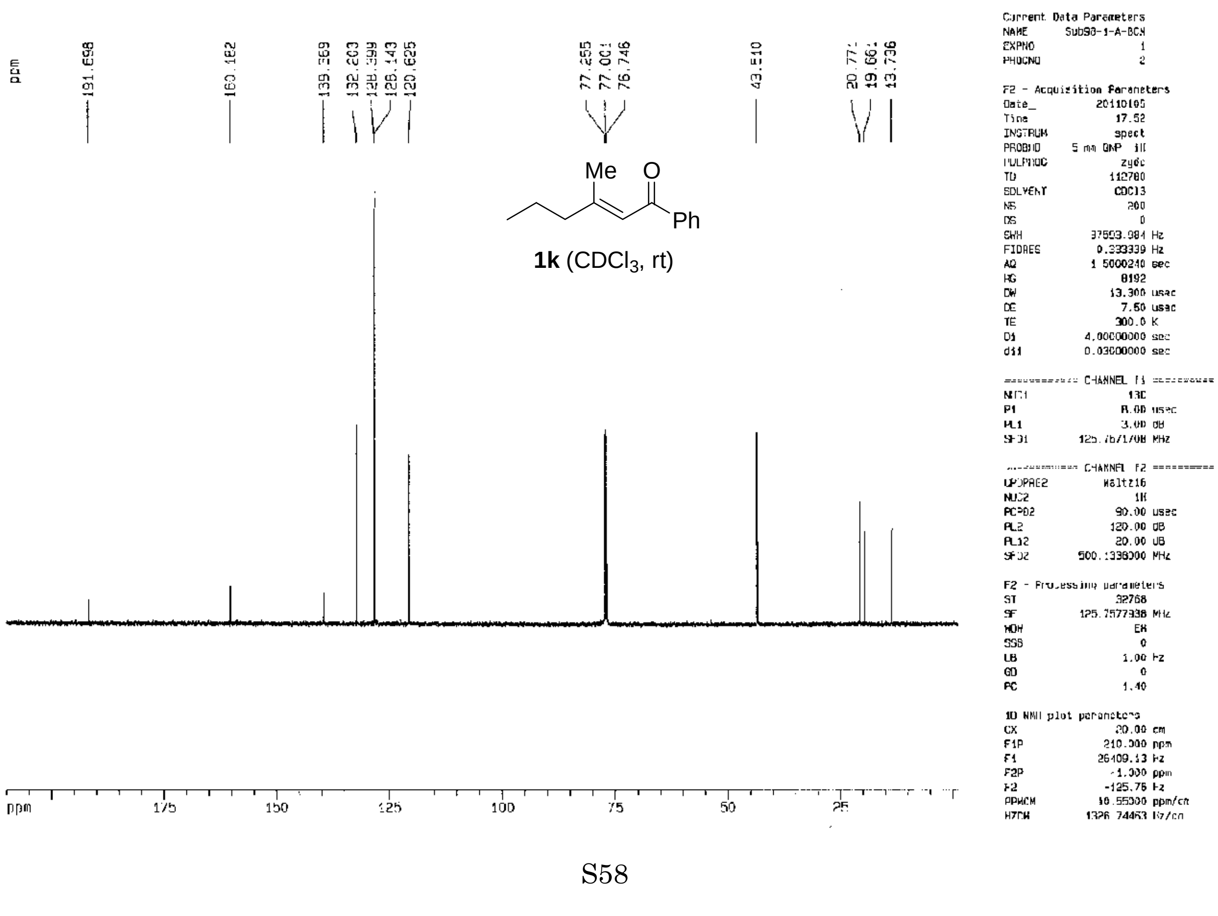

Me
O
Ph
Cl
1i (CDCl3, rt)

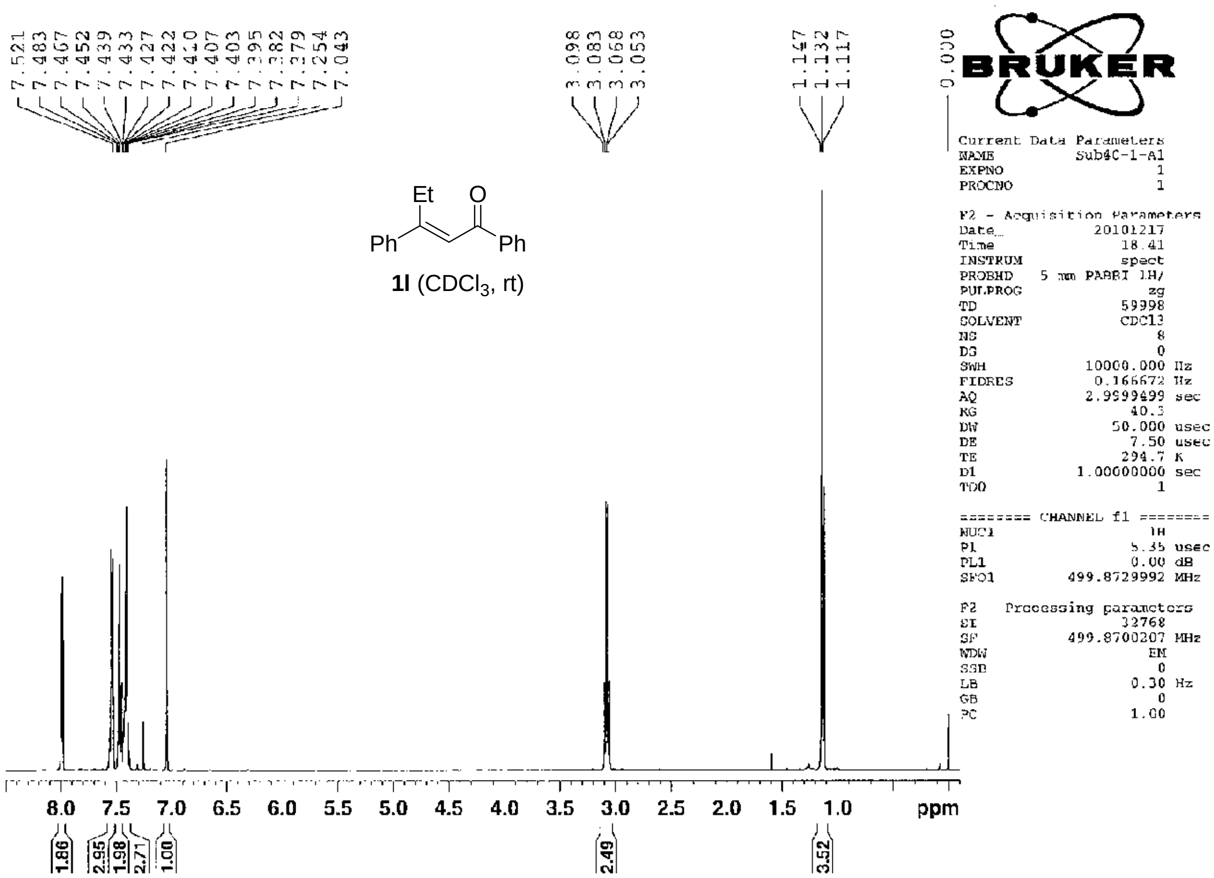

Me
O
Ph
Cl
1i (CDCl3, rt)

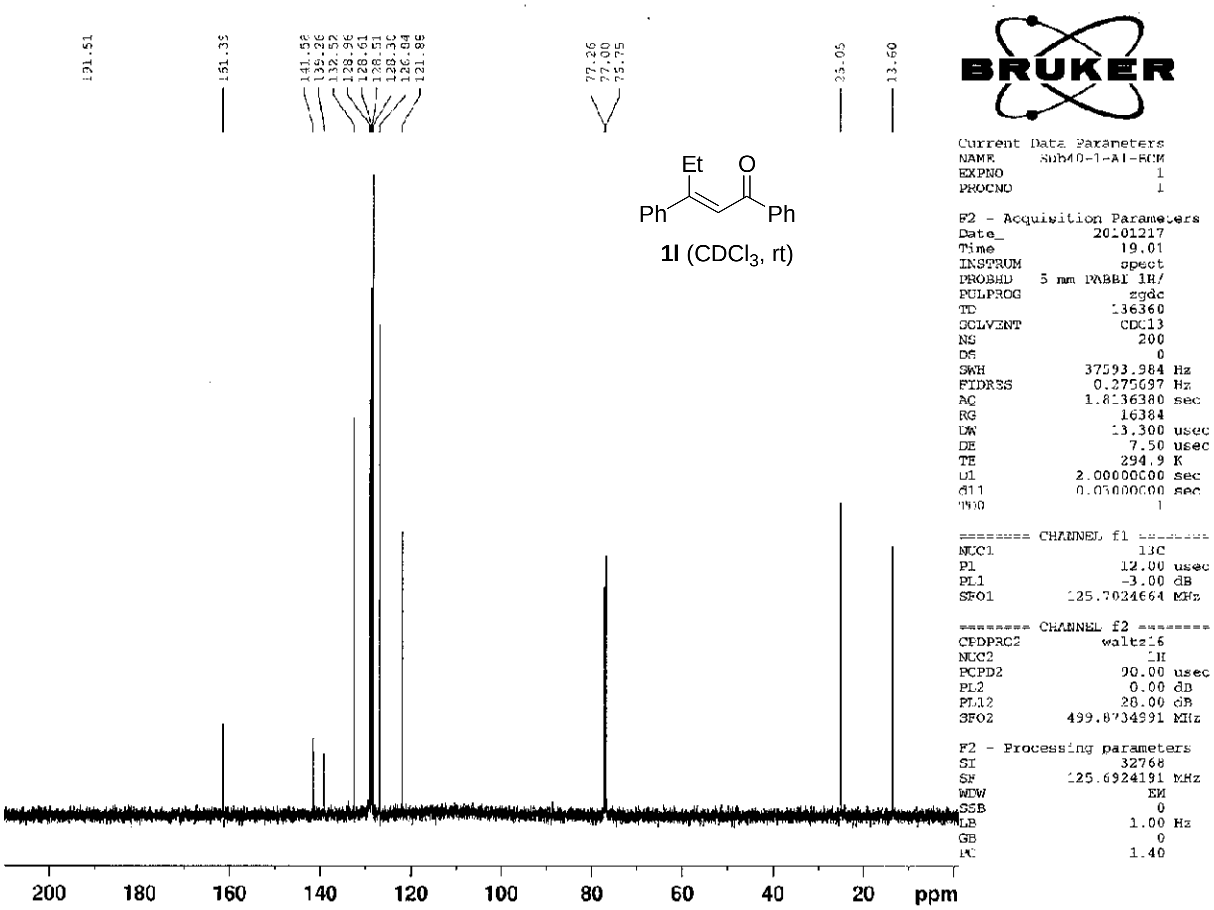

Me
O
Ph
1j (CDCl3, rt)
ppm
8.039
8.024
8.022
7.887
7.870
7.533
7.525
7.514
7.508
7.493
7.320
7.318
7.252
2.712
2.710
0.000
Integral
1.9226
0.3777
1.5561
1.5318
1.0477
1.0838
3.8463
1.0000
2.9621

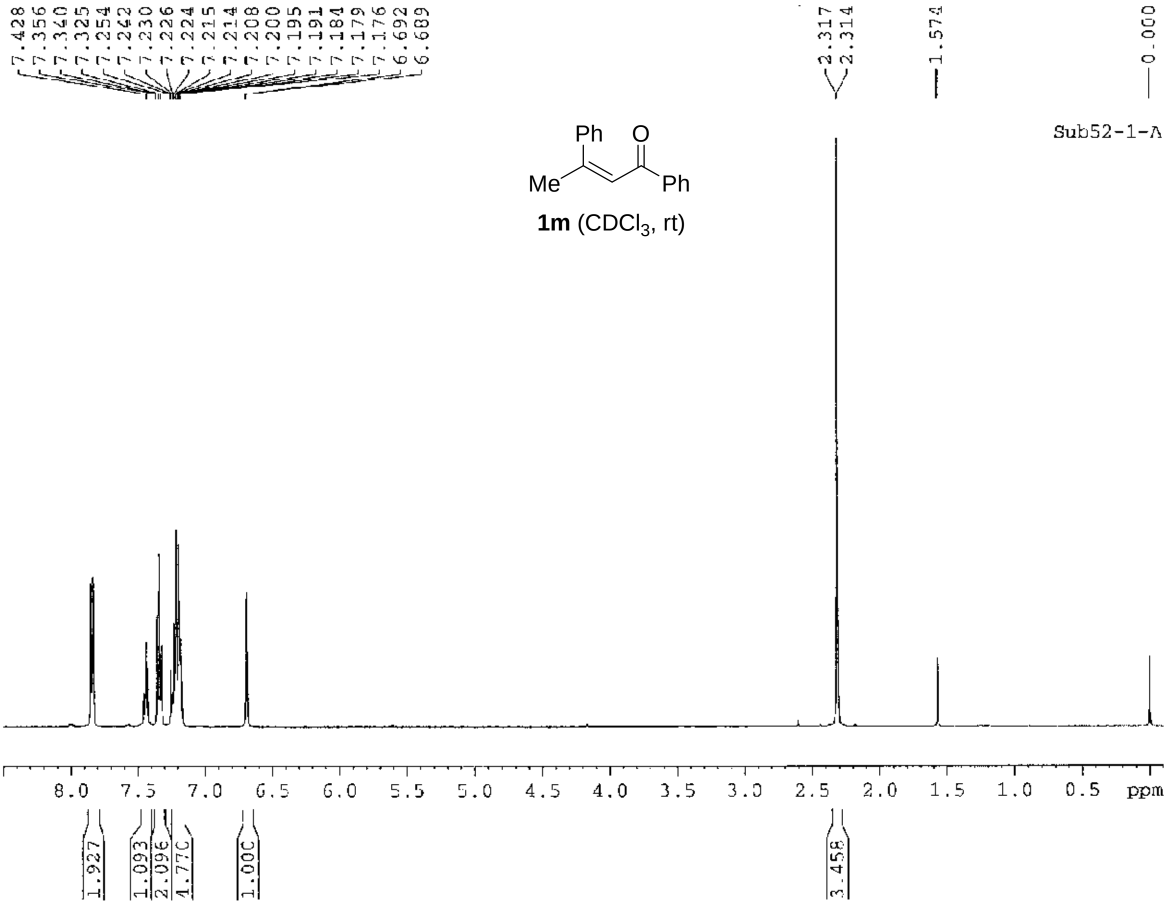

Me
O
Ph
1j (CDCl3, rt)
ppm
191.865
154.802
139.863
139.401
133.544
133.161
132.549
128.555
128.517
128.292
128.253
127.610
126.767
126.573
126.162
124.056
122.409
77.253
76.999
76.744
18.868
150
100
50

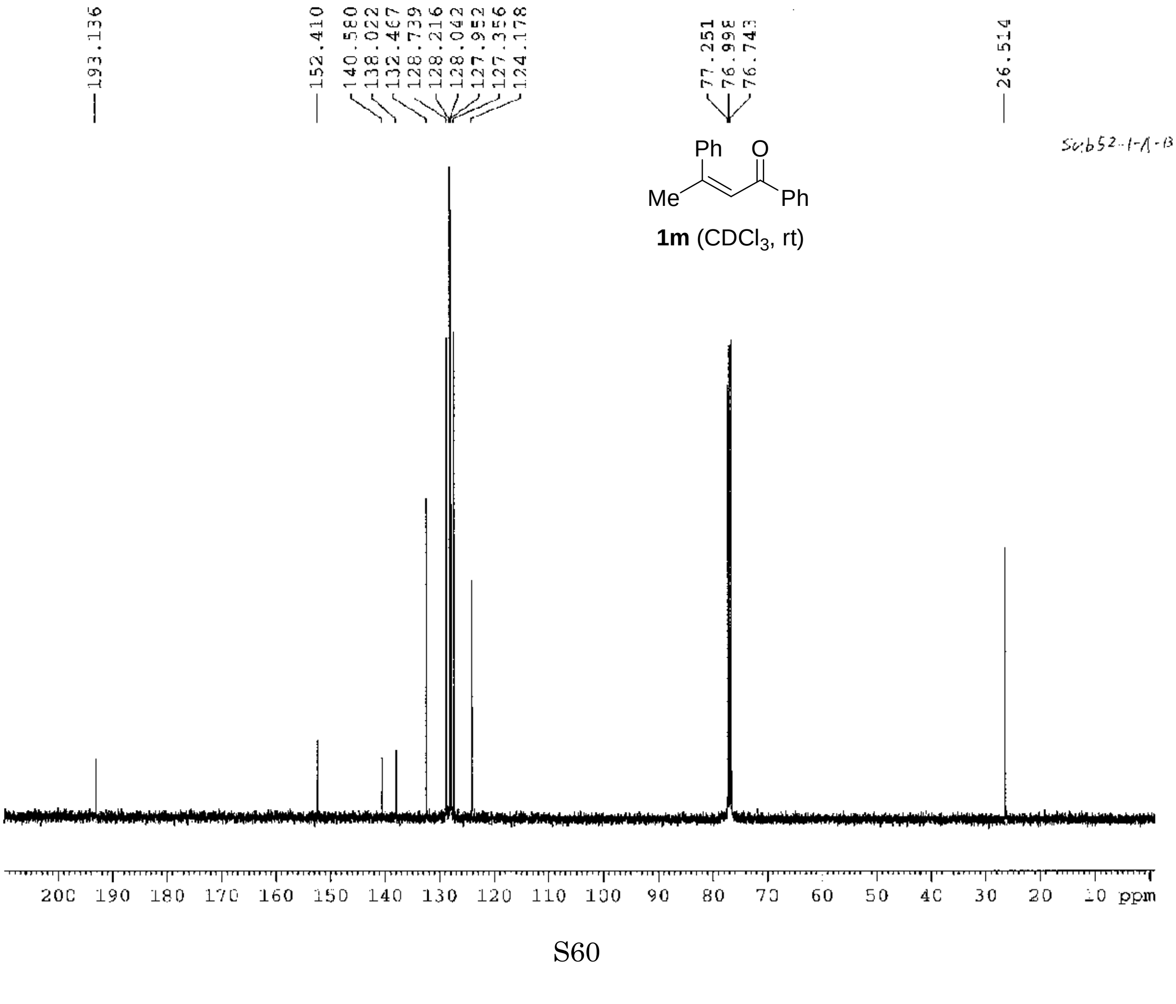

Sub98-1-A
Me
O
Ph
1k (CDCl3, rt)

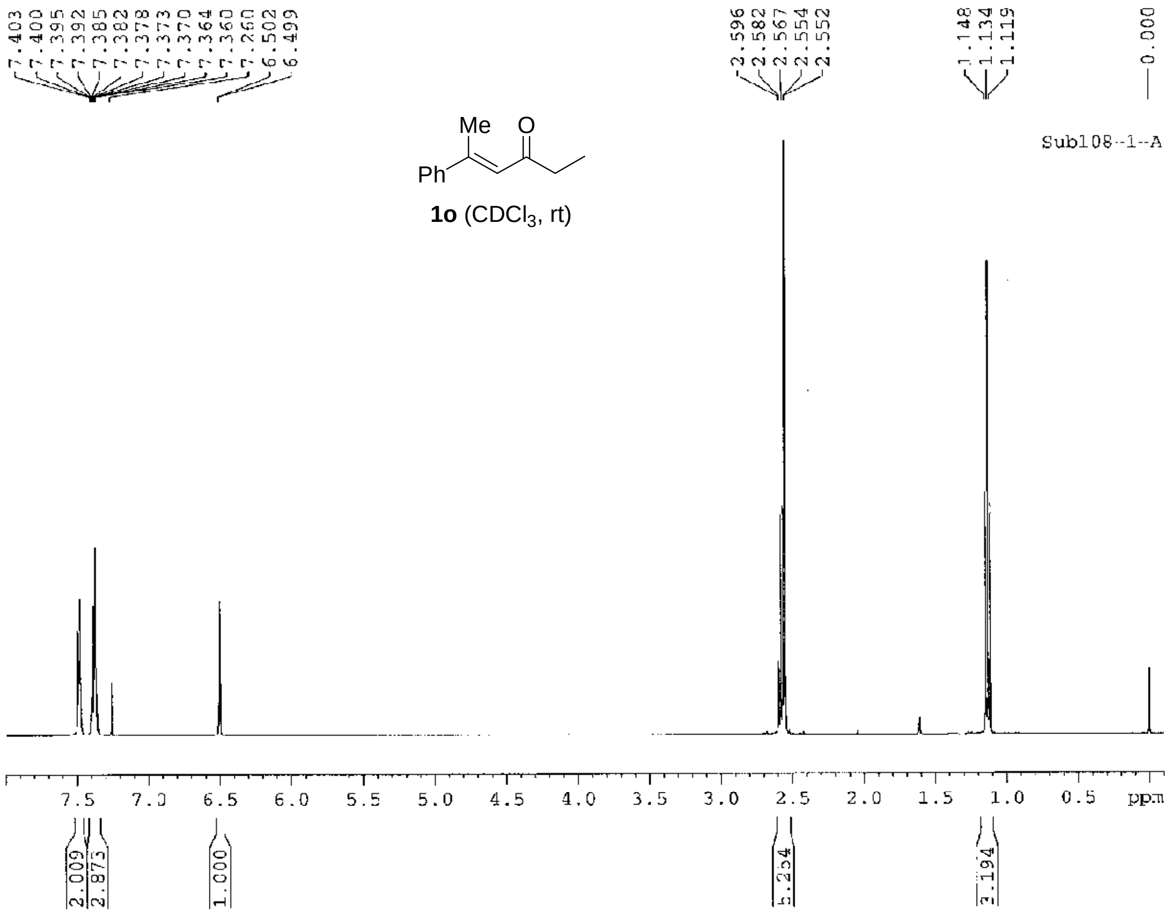

Me
O
Ph
1k (CDCl3, rt)
Current Data Parameters

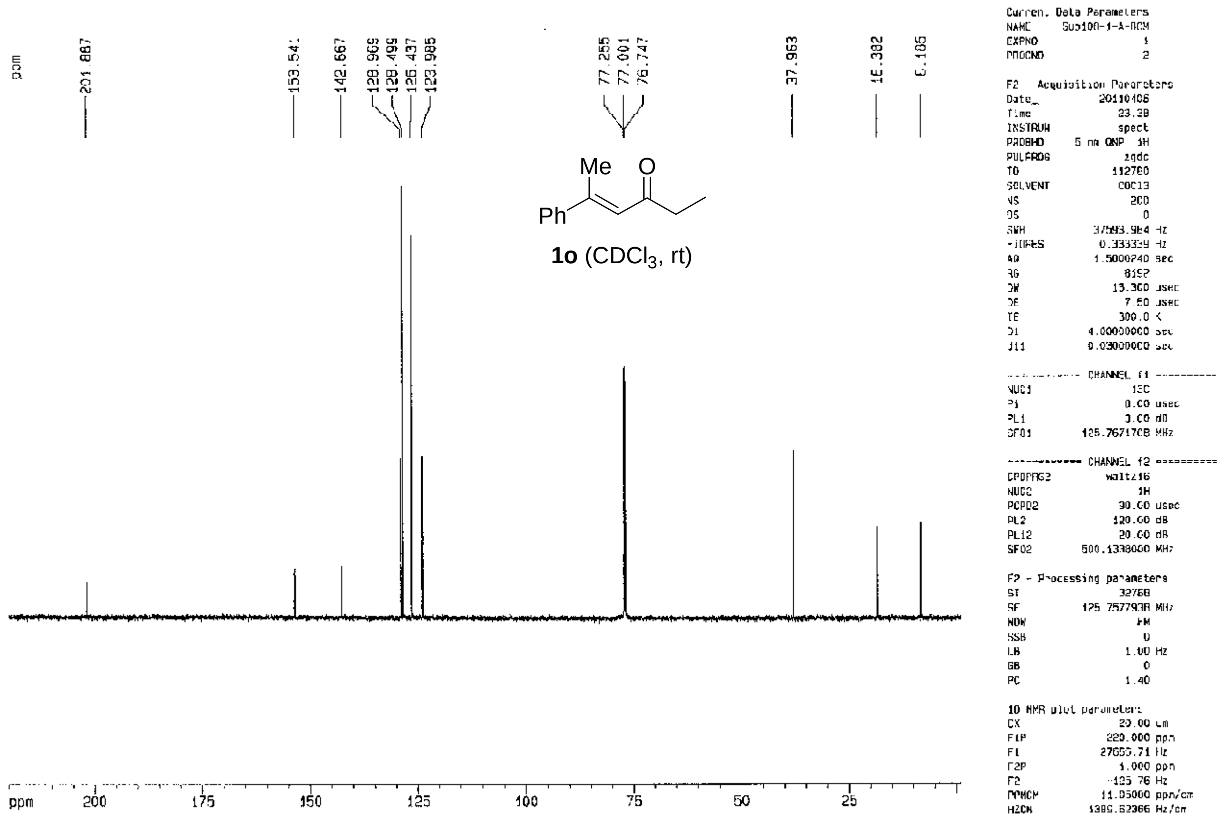

7.521 7.483 7.467 7.452 7.439 7.433 7.427 7.422 7.410 7.407 7.403 7.395 7.382 7.379 7.254 7.043
3.098 3.083 3.068 3.053
1.147 1.132 1.117
0.000
Et O
Ph Ph
1l (CDCl3, rt)
BRUKER
Current Data Parameters
NAME Sub4C-1-A1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101217
Time 18.41
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 40.3
DW 50.000 usec
DE 7.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 Processing parameters
SI 32768
SF 499.8700207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.86 2.95 1.98 2.71 1.00 2.49 3.52

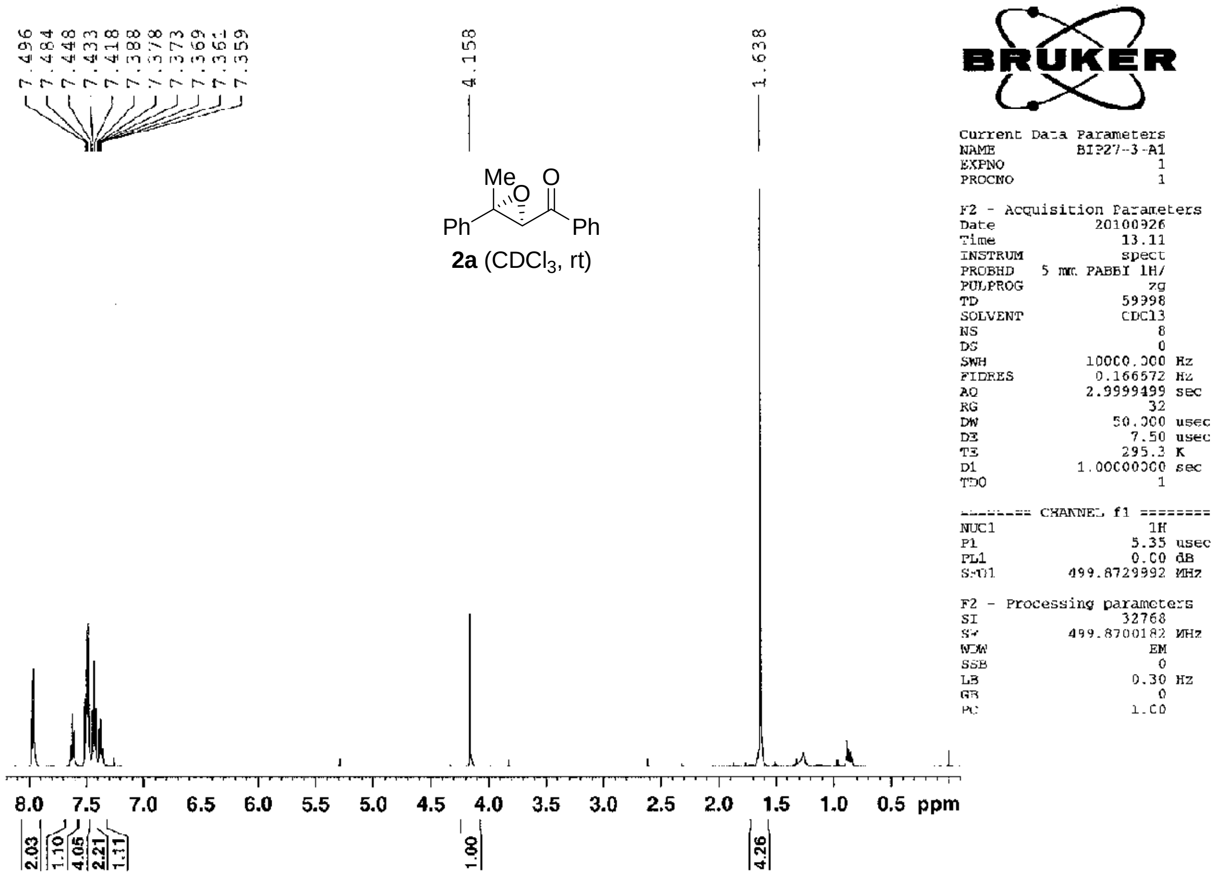

191.51
161.39
141.58 139.26 132.52 128.96 128.61 128.51 128.30 126.84 121.88
77.26 77.00 76.75
25.05
13.60
BRUKER
Et O
Ph Ph
1l (CDCl3, rt)
Current Data Parameters
NAME Sub40-1-A1-BCM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101217
Time 19.01
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT CDCl3
NS 200
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136380 sec
RG 16384
DW 13.300 usec
DE 7.50 usec
TE 294.9 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7024664 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6924191 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

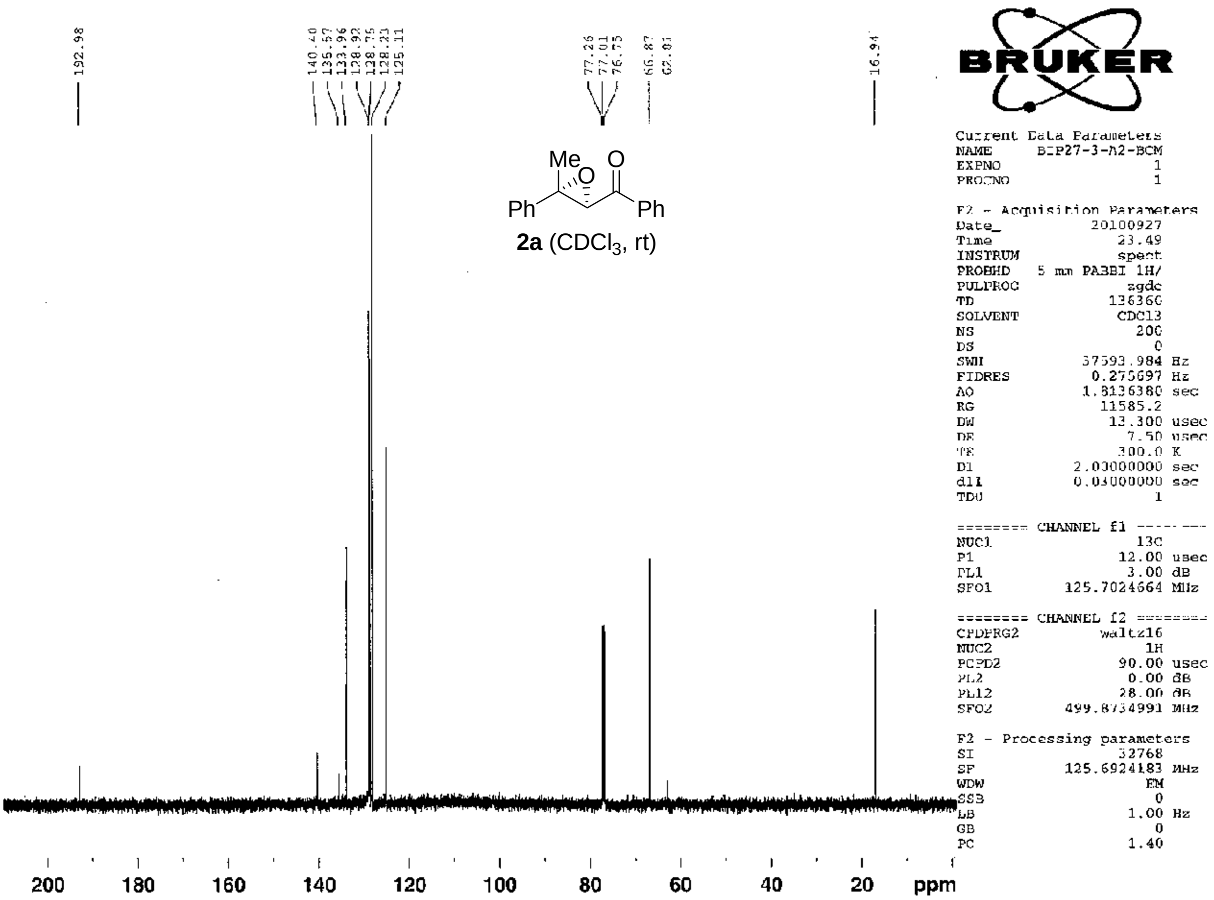

7.428
7.356
7.340
7.325
7.254
7.242
7.230
7.226
7.224
7.215
7.214
7.208
7.200
7.195
7.191
7.184
7.179
7.176
6.692
6.689
2.317
2.314
1.574
0.000
Sub52-1-A
Ph
O
Me
Ph
1m (CDCl3, rt)
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.927
1.093
2.096
1.770
1.000
3.458


193.136
152.410
140.580
138.022
132.467
128.739
128.216
128.042
127.952
127.356
124.178
77.251
76.998
76.743
26.514
Sub52-1-A-B
Ph
O
Me
Ph
1m (CDCl3, rt)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.403 7.400 7.395 7.392 7.385 7.382 7.378 7.373 7.370 7.364 7.360 7.260 6.502 6.499
2.596 2.582 2.567 2.554 2.552
1.148 1.134 1.119
0.000
Sub108-1-A
Me
O
Ph
1o (CDCl$_3$, rt)
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.009 2.873 1.000


ppm
201.887
153.541
142.667
128.969
128.499
126.437
123.985
77.255
77.001
76.747
37.963
18.382
8.185
Me
O
Ph
1o (CDCl$_3$, rt)
ppm 200 175 150 125 100 75 50 25


7.496
7.484
7.448
7.433
7.418
7.388
7.378
7.373
7.369
7.361
7.359
4.158
1.638
Me
O
O
Ph
Ph
2a (CDCl3, rt)
BRUKER
Current Data Parameters
NAME BIP27-3-A1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date 20100926
Time 13.11
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 32
DW 50.000 usec
DE 7.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700182 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.03
1.10
4.05
2.21
1.11
1.00
4.26

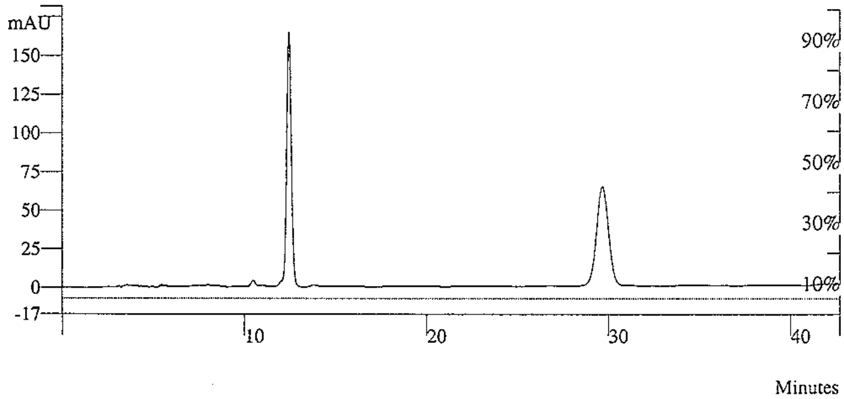

192.98
140.40
135.57
133.96
128.92
128.75
128.23
125.11
77.26
77.01
76.75
66.87
62.81
16.94
Me
O
O
Ph
Ph
2a (CDCl3, rt)
BRUKER
Current Data Parameters
NAME BIP27-3-A2-BCM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100927
Time 23.49
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT CDCl3
NS 200
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136380 sec
RG 11585.2
DW 13.300 usec
DE 7.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
SFO1 125.7024664 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6924183 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

Chiral HPLC analysis of compound **2a**

rac-**2a**

Data File:	c:\star\1-6-11 5;05;55 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP27-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 5/95 FR1.0	Calibration Level:	N/A
Injection Date:	01/06/11 05:05:55 PM	Run Time (min):	42.747

Injection Method: c:\star\yasuhiro\temporaly.mth

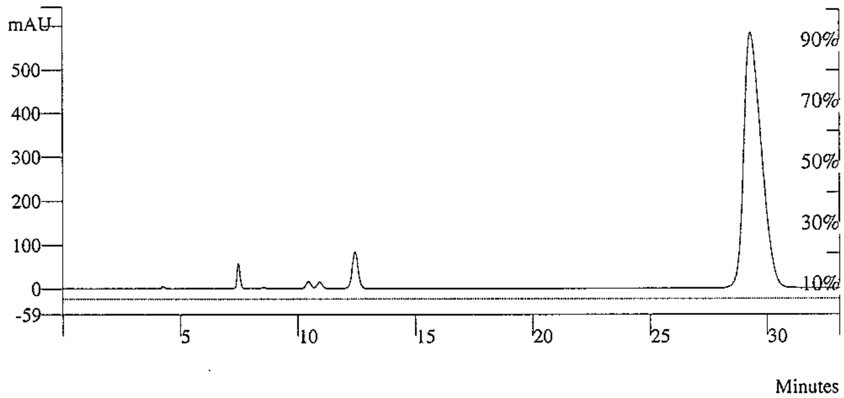


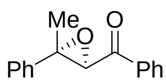
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	50.1862	12.440	0.000	15054974	0.00	BB	16.2
2	49.8138	29.640	0.000	14943252	0.00	BB	42.3
	100.0000		**0.000**	**29998226**			

chiral **2a**

Data File:	c:\star\1-8-11 7;16;47 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP27-27-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	01/08/11 07:16:47 PM	Run Time (min):	33.093

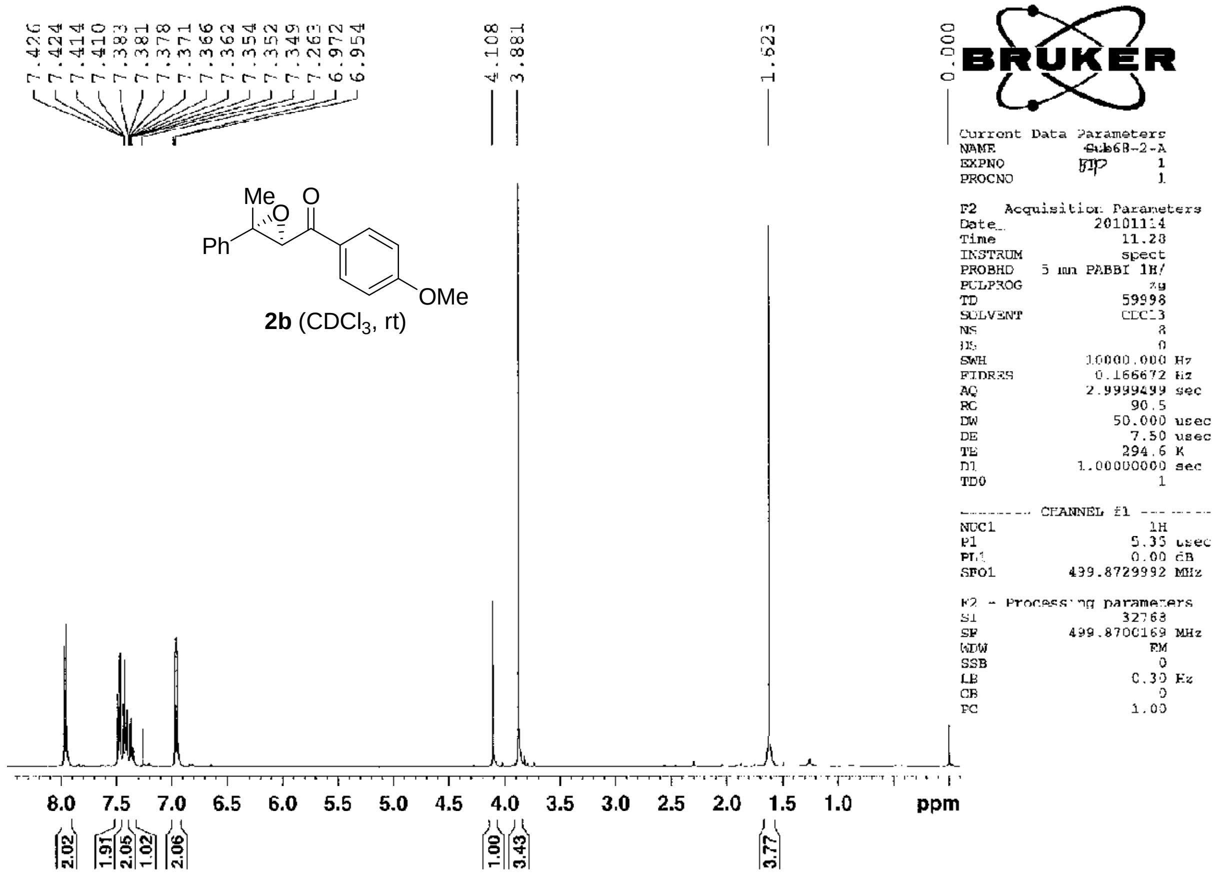
Injection Method: c:\star\yasuhiro\temporaly.mth

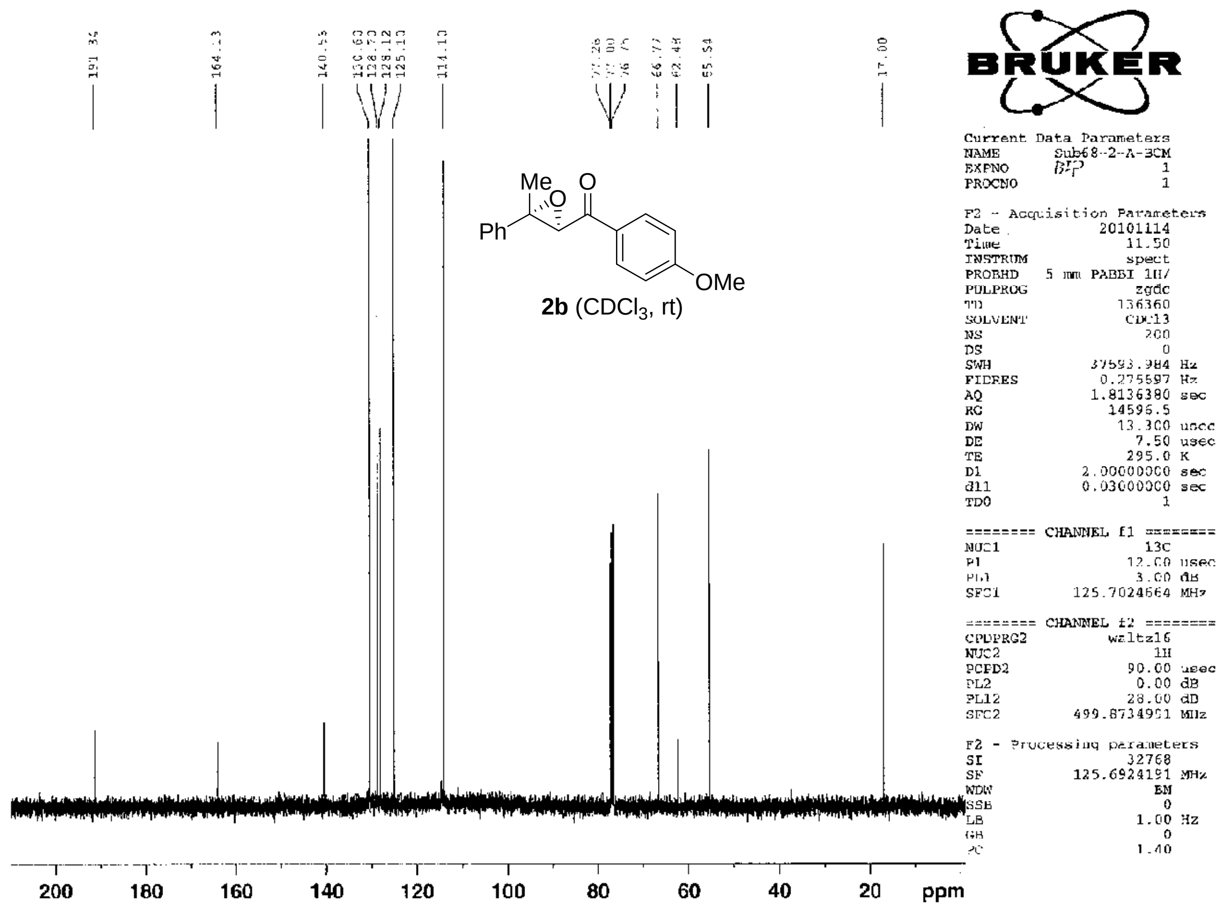




2a

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	4.4650	12.413	0.000	7346354	0.00	BB	15.9
2	95.5350	29.267	0.000	157187232	0.00	BB	49.2
	100.0000		**0.000**	**164533584**			



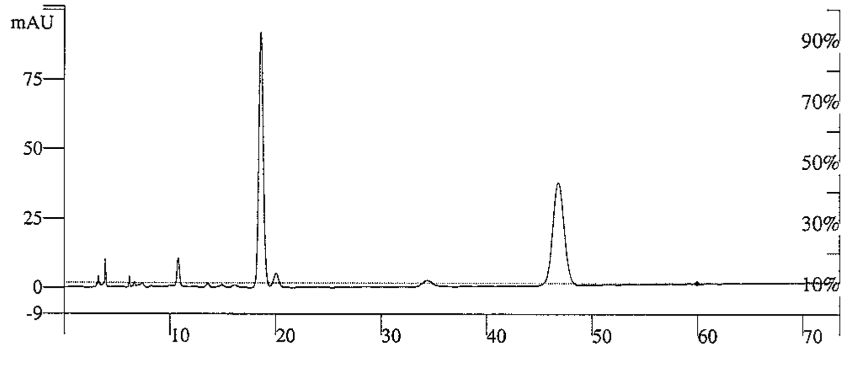


Chiral HPLC analysis of compound **2b**

rac-**2b**

Data File:	c:\star\11-10-10 10;30;23 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP68-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 10/90 FR1.0	Calibration Level:	N/A
Injection Date:	11/10/10 10:30:23 PM	Run Time (min):	73.547

Injection Method: c:\star\yasuhiro\temporaly.mth

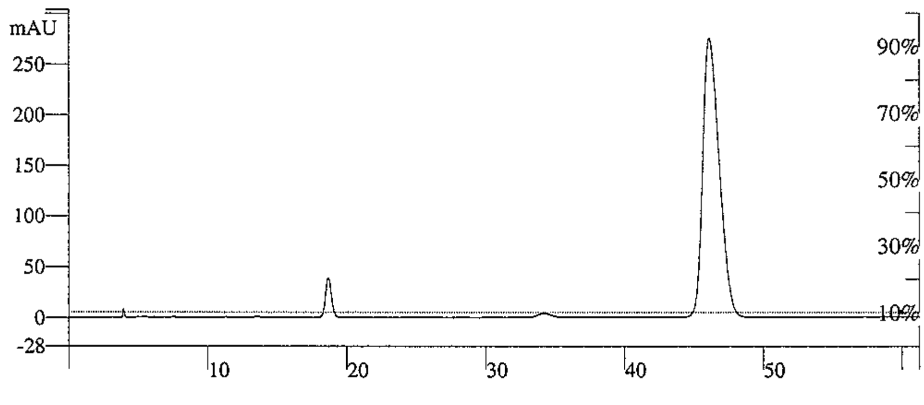


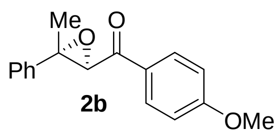
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	48.8328	18.600	0.000	13537579	0.00	BB	27.8
2	51.1672	46.787	0.000	14184718	0.00	BB	70.3
	100.0000		**0.000**	**27722296**			

chiral **2b**

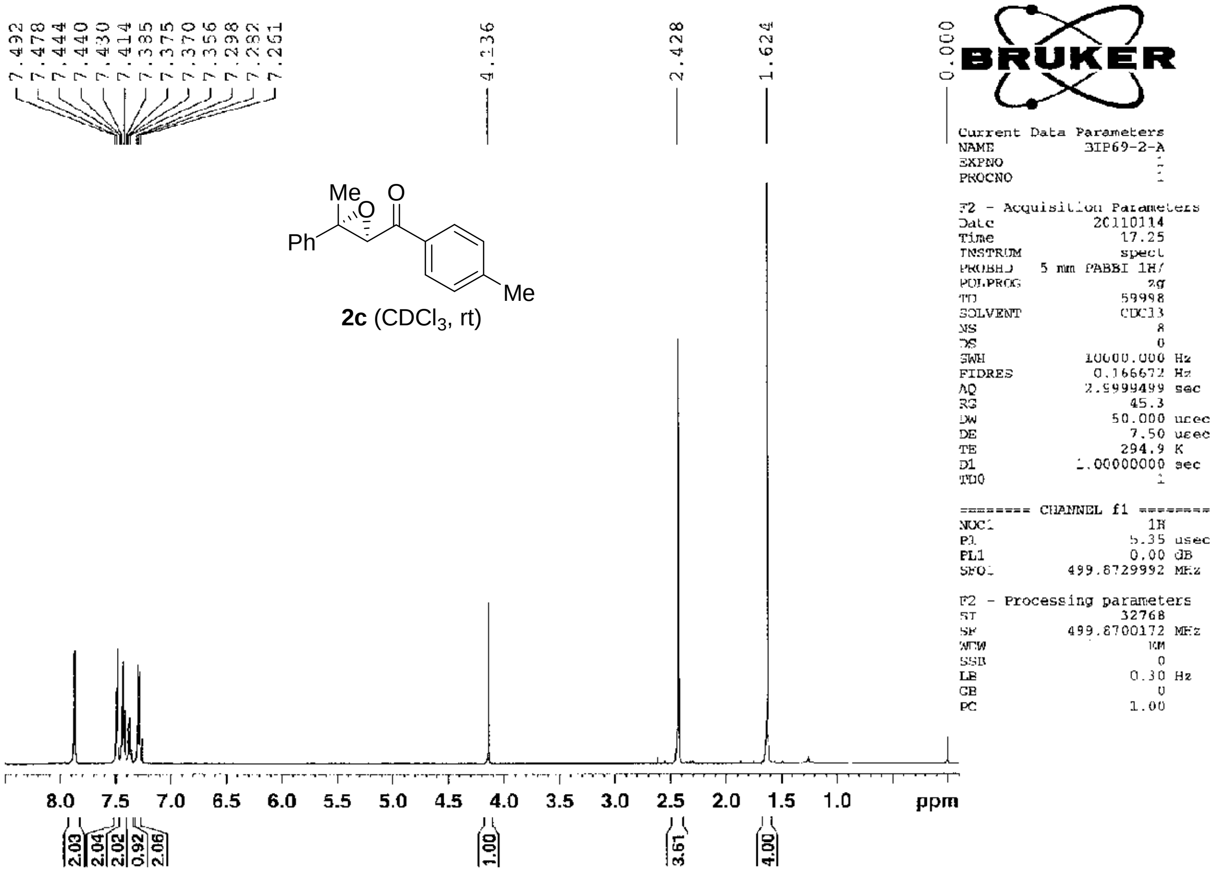
Data File:	c:\star\11-11-10 4;50;20 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP68-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	11/11/10 04:50:20 PM	Run Time (min):	61.280

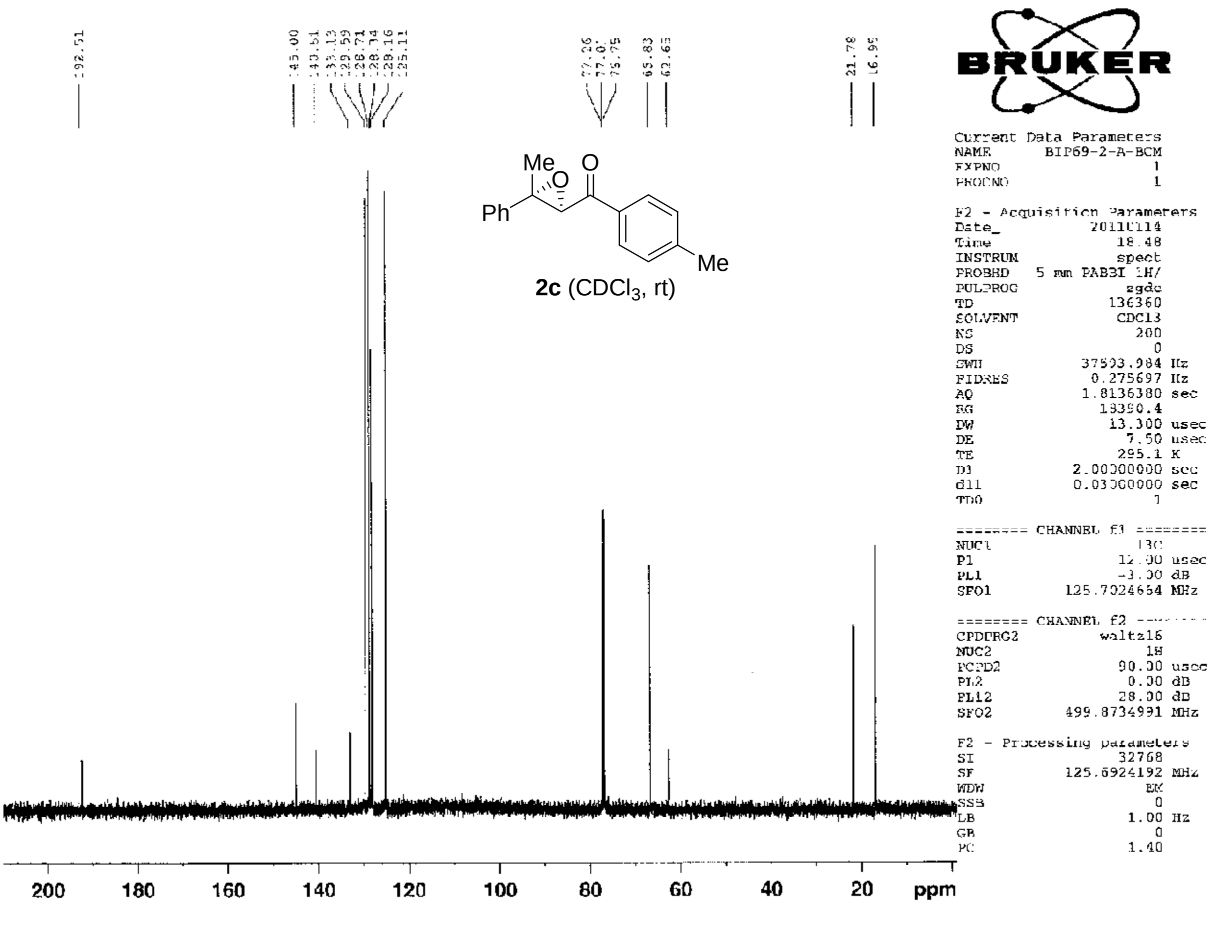
Injection Method: c:\star\yasuhiro\temporaly.mth





Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	4.8213	18.653	0.000	5893033	0.00	BB	27.5
2	95.1787	46.093	0.000	116335656	0.00	BB	77.5
	100.0000		**0.000**	**122228688**			


7.492
7.478
7.444
7.440
7.430
7.414
7.385
7.375
7.370
7.356
7.298
7.282
7.261
4.136
2.428
1.624
0.000
Me
O
O
Ph
Me
2c (CDCl3, rt)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.03
2.04
2.02
0.92
2.06
1.00
3.61
4.00
Current Data Parameters
NAME BIP69-2-A
F2 - Acquisition Parameters
Date 20110114
Time 17.25
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 45.3
DW 50.000 usec
DE 7.50 usec
TE 294.9 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

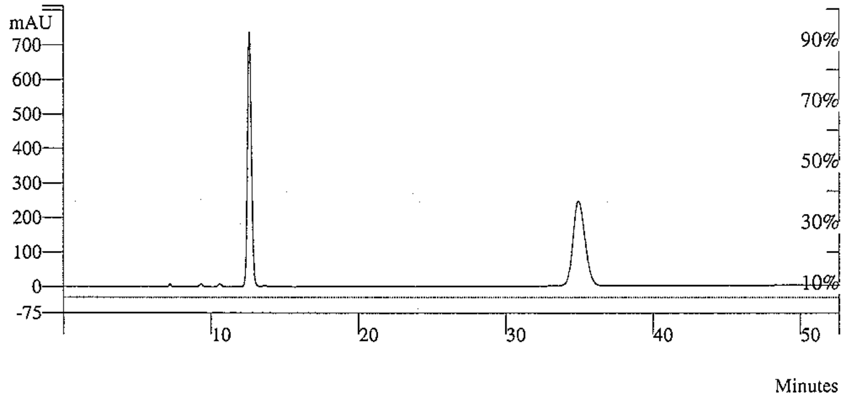

192.51
145.00
140.51
133.13
129.59
128.71
128.34
128.16
125.11
77.26
77.01
76.75
65.83
62.63
21.78
16.95
Me
O
O
Ph
Me
2c (CDCl3, rt)
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME BIP69-2-A-BCM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110114
Time 18.48
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT CDCl3
NS 200
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136380 sec
RG 13390.4
DW 13.300 usec
DE 7.50 usec
TE 295.1 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7024654 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6924192 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Chiral HPLC analysis of compound **2c**

rac-**2c**

Data File:	c:\star\11-10-10 1;53;24 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP69-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 5/95 FR1.0	Calibration Level:	N/A
Injection Date:	11/10/10 01:53:24 PM	Run Time (min):	52.667

Injection Method: c:\star\yasuhiro\temporaly.mth

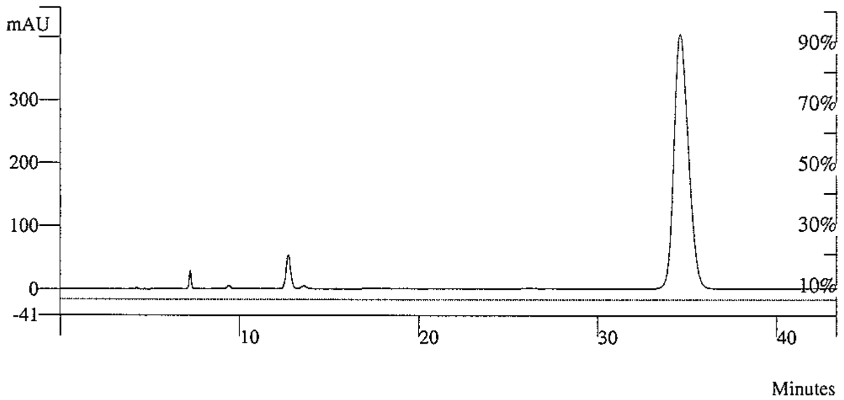


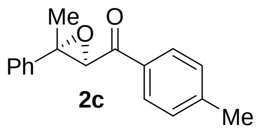
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	49.7482	12.600	0.000	69961256	0.00	BB	17.2
2	50.2518	34.920	0.000	70669520	0.00	BB	52.5
	100.0000		**0.000**	**140630784**			

chiral **2c**

Data File:	c:\star\11-11-10 11;05;17 am -1.run	Run Mode:	Analysis
Sample ID:	BIP69-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	11/11/10 11:05:17 AM	Run Time (min):	43.360

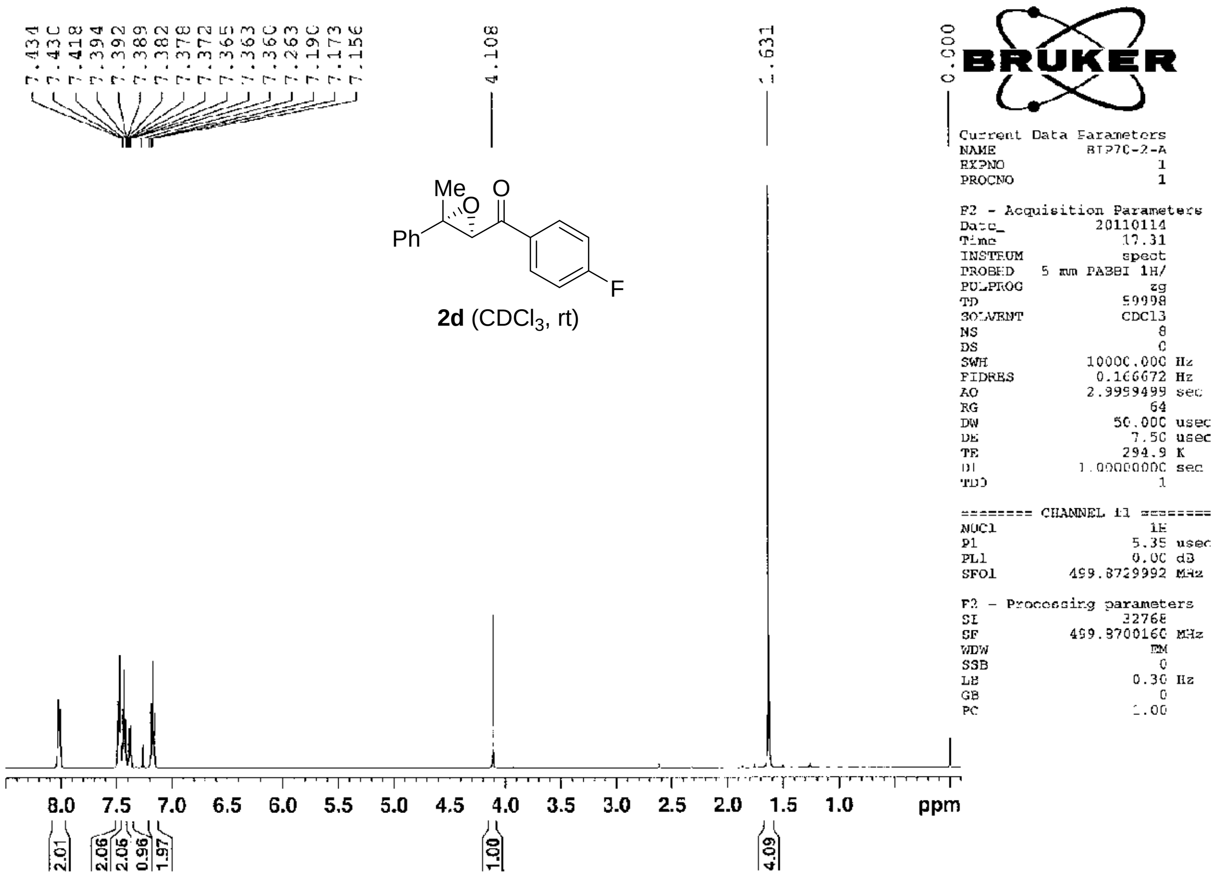
Injection Method: c:\star\yasuhiro\temporaly.mth

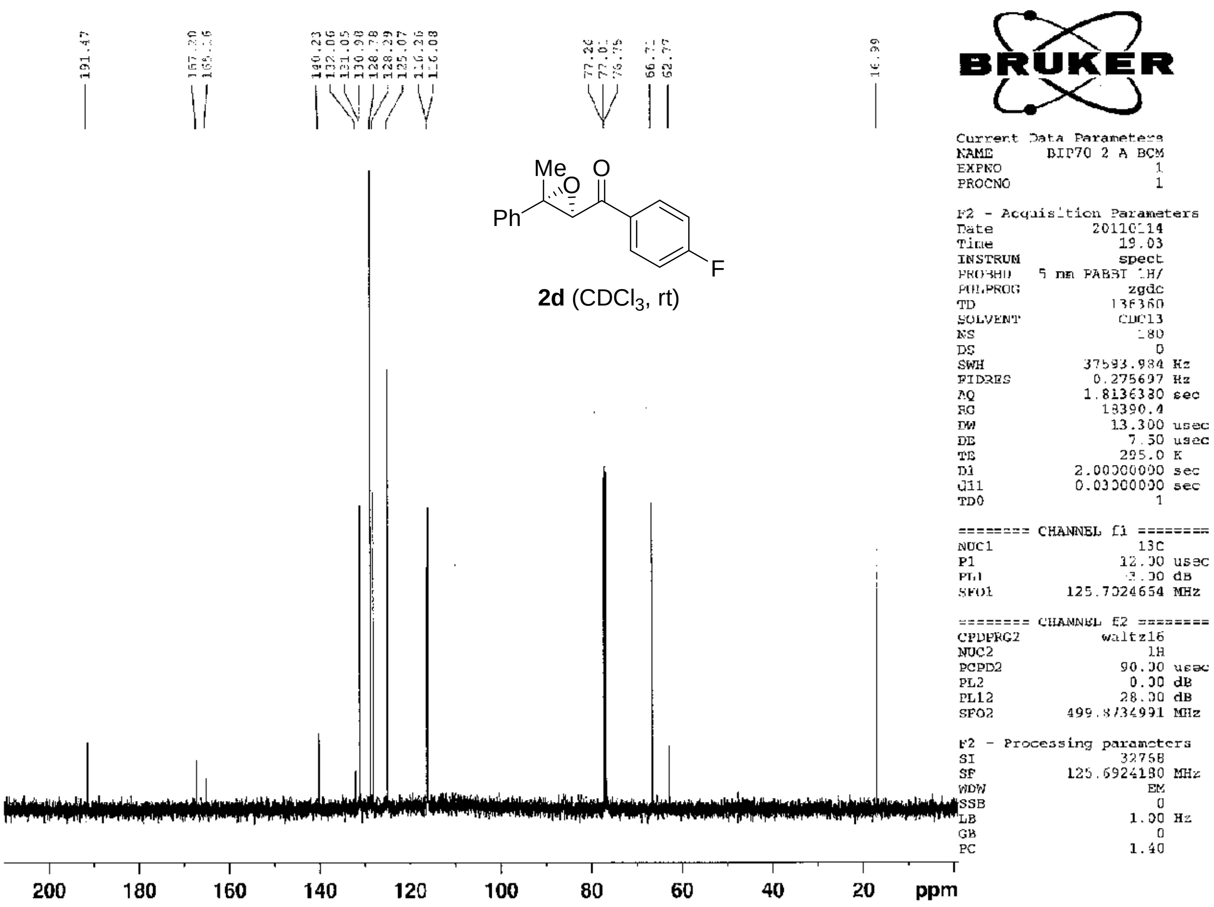




2c

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	4.0338	12.707	0.000	4843680	0.00	BB	16.6
2	95.9662	34.573	0.000	115235112	0.00	BB	52.2
	100.0000		**0.000**	**120078792**			


Me
O
O
Ph
F
2d (CDCl3, rt)
BRUKER
ppm

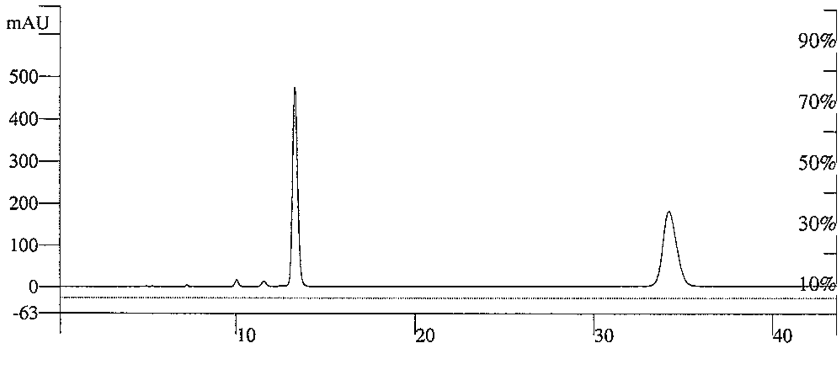

Me
O
O
Ph
F
2d (CDCl3, rt)
BRUKER
ppm

Chiral HPLC analysis of compound **2d**

rac-**2d**

Data File:	c:\star\11-10-10 2;50;08 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP70-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 5/95 FR1.0	Calibration Level:	N/A
Injection Date:	11/10/10 02:50:08 PM	Run Time (min):	52.293

Injection Method: c:\star\yasuhiro\temporaly.mth

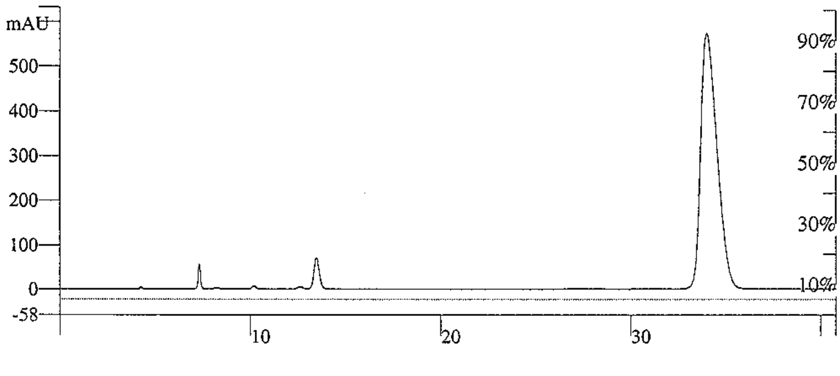


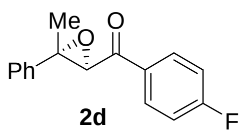
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	49.5796	13.267	0.000	48385256	0.00	BB	18.4
2	50.4204	34.200	0.000	49205740	0.00	BB	49.9
	100.0000		**0.000**	**97590992**			

chiral-**2d**

Data File:	c:\star\11-11-10 11;51;34 am -1.run	Run Mode:	Analysis
Sample ID:	BIP70-1-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	11/11/10 11:51:34 AM	Run Time (min):	40.827

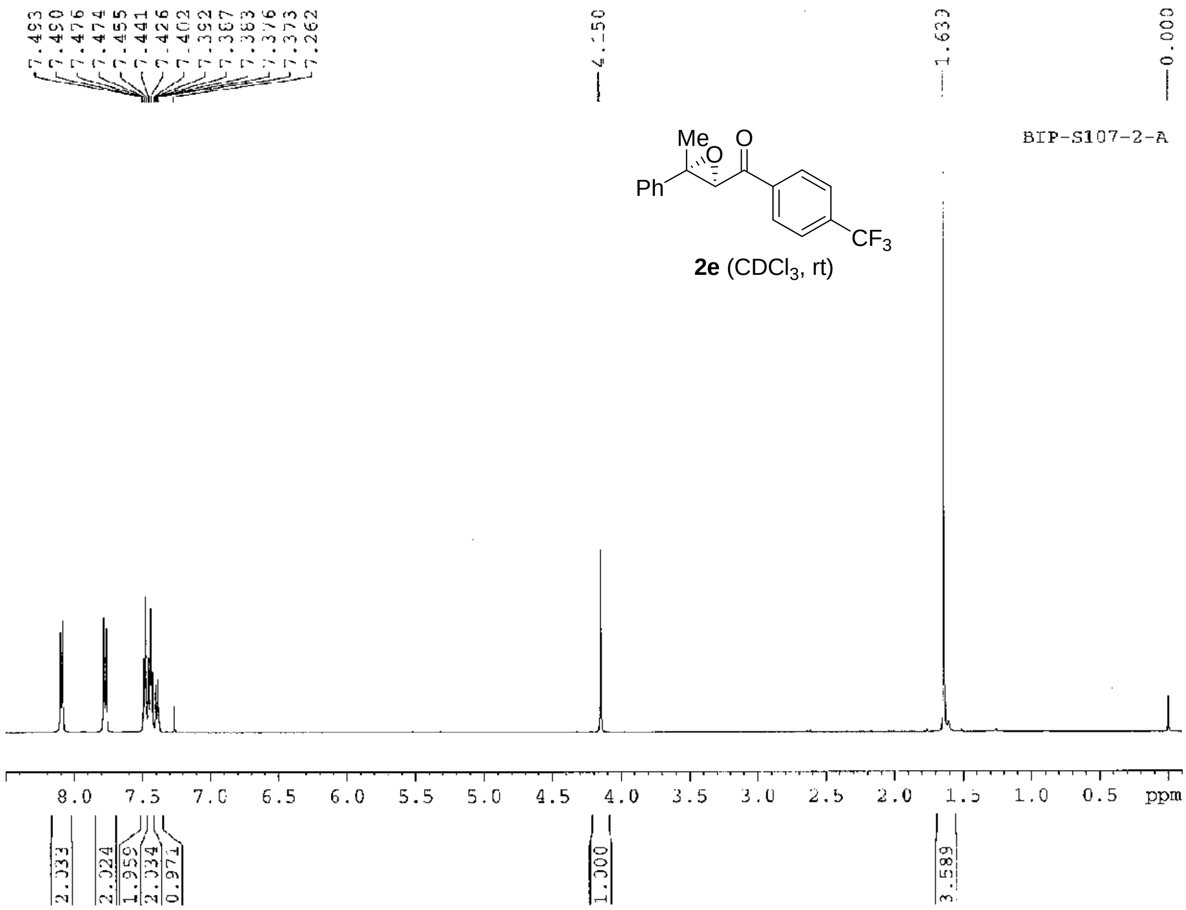
Injection Method: c:\star\yasuhiro\temporaly.mth

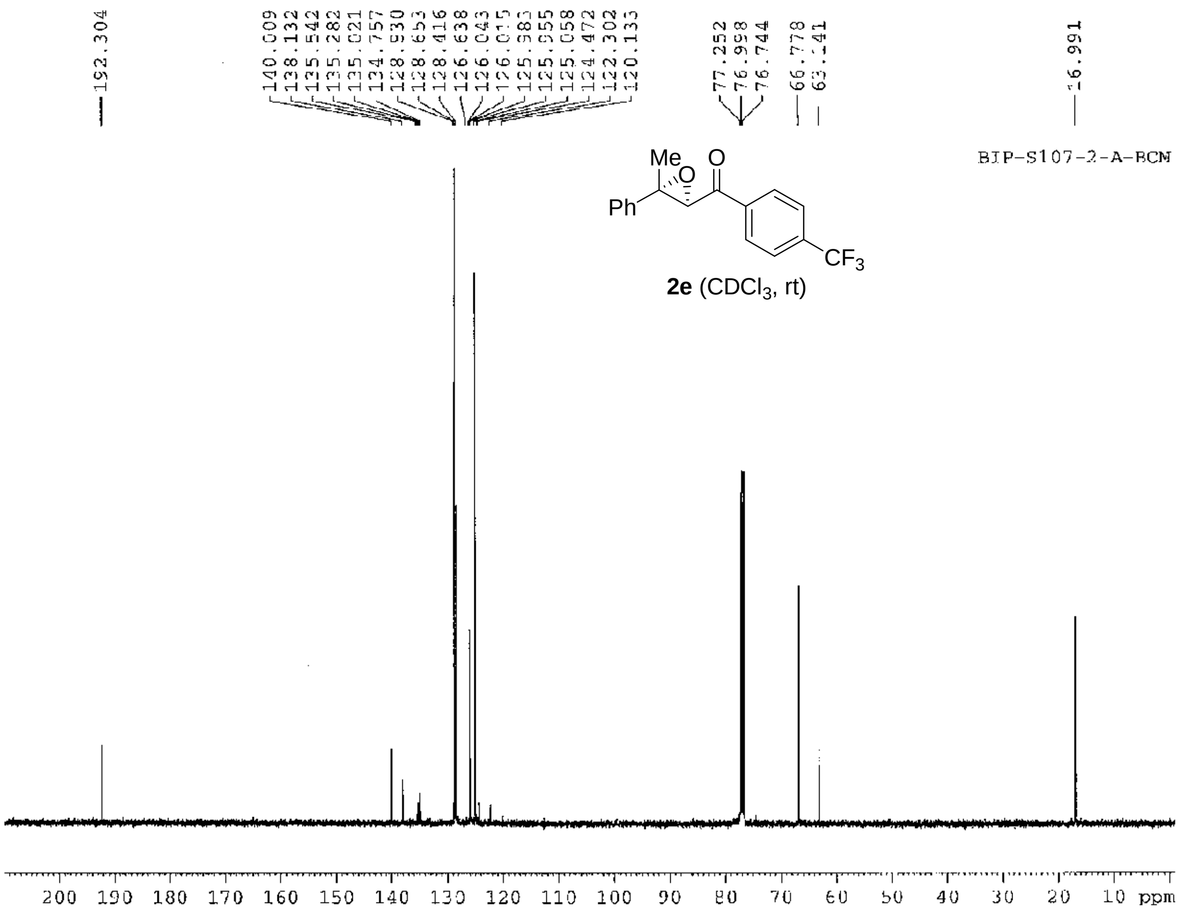




2d

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	3.8592	13.480	0.000	7048031	0.00	BB	18.1
2	96.1408	33.987	0.000	175581072	0.00	BB	56.2
	100.0000		**0.000**	**182629104**			


7.493
7.490
7.476
7.474
7.455
7.441
7.426
7.402
7.392
7.387
7.383
7.376
7.373
7.262
4.150
1.633
0.000
BIP-S107-2-A
Me
O
O
Ph
CF3
2e (CDCl3, rt)
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.033
2.024
1.959
2.034
0.971
1.000
3.589

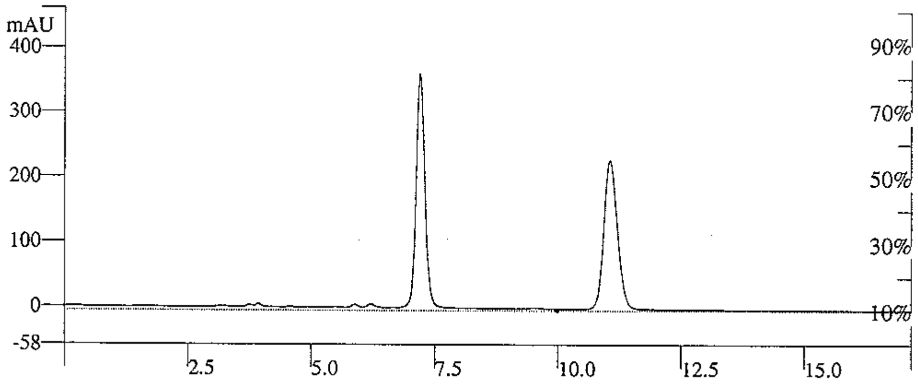

192.304
140.009
138.132
135.542
135.282
135.021
134.757
128.930
128.653
128.416
126.638
126.043
126.015
125.983
125.955
125.058
124.472
122.302
120.133
77.252
76.998
76.744
66.778
63.141
16.991
BIP-S107-2-A-BCM
Me
O
O
Ph
CF3
2e (CDCl3, rt)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Chiral HPLC analysis of compound **2e**

rac-**2e**

Data File:	c:\star\4-9-11 2;20;10 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP-S107-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 10/90 FR1.0	Calibration Level:	N/A
Injection Date:	04/09/11 02:20:10 PM	Run Time (min):	23.093

Injection Method: c:\star\yasuhiro\temporaly.mth

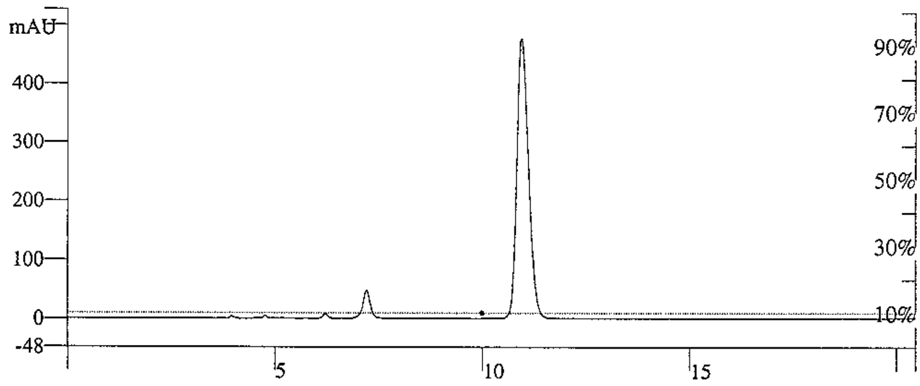


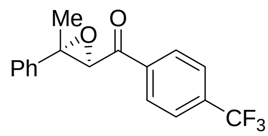
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	50.2934	7.187	0.000	22690444	0.00	BB	11.0
2	49.7066	11.053	0.000	22425706	0.00	BB	17.6
	100.0000		**0.000**	**45116152**			

chiral-**2e**

Data File:	c:\star\4-9-11 4;15;01 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP-S107-1-A	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	04/09/11 04:15:01 PM	Run Time (min):	20.480

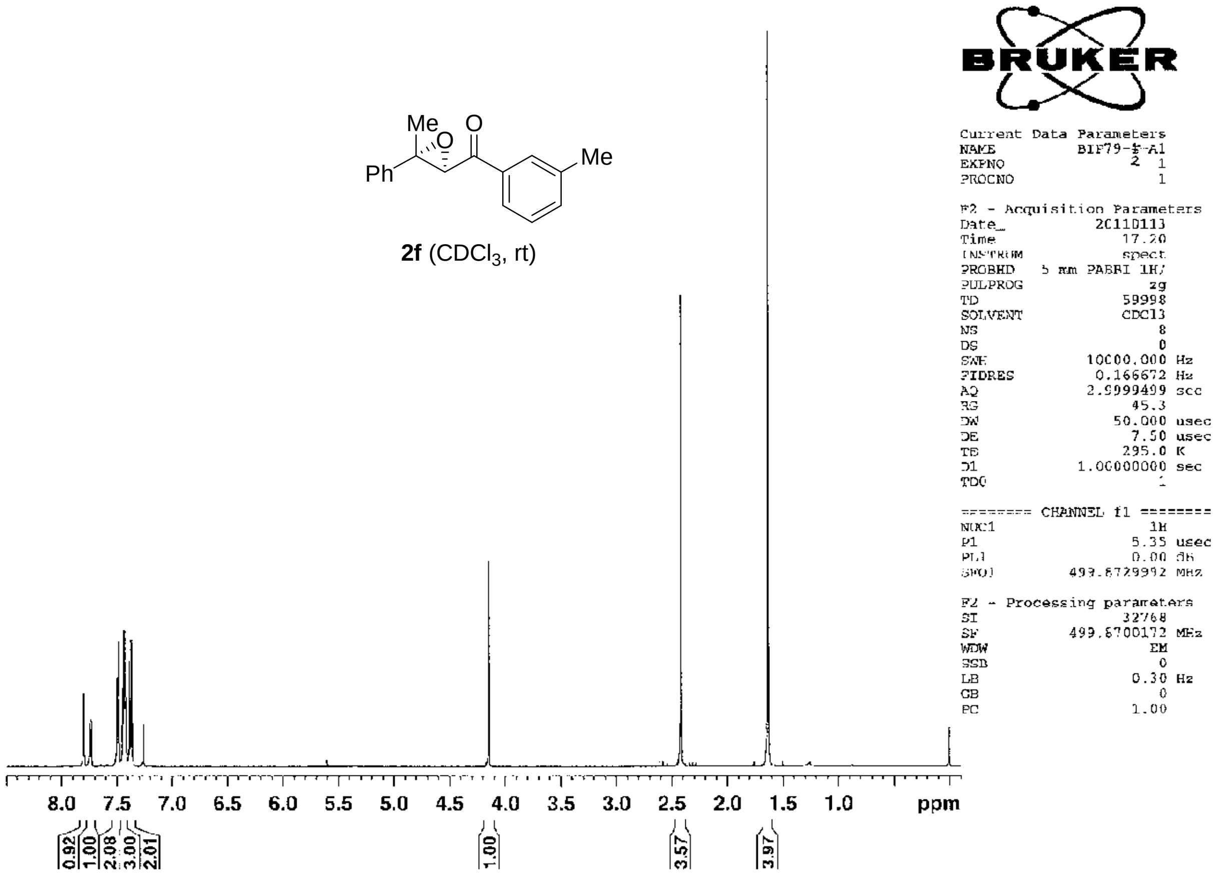
Injection Method: c:\star\yasuhiro\temporaly.mth

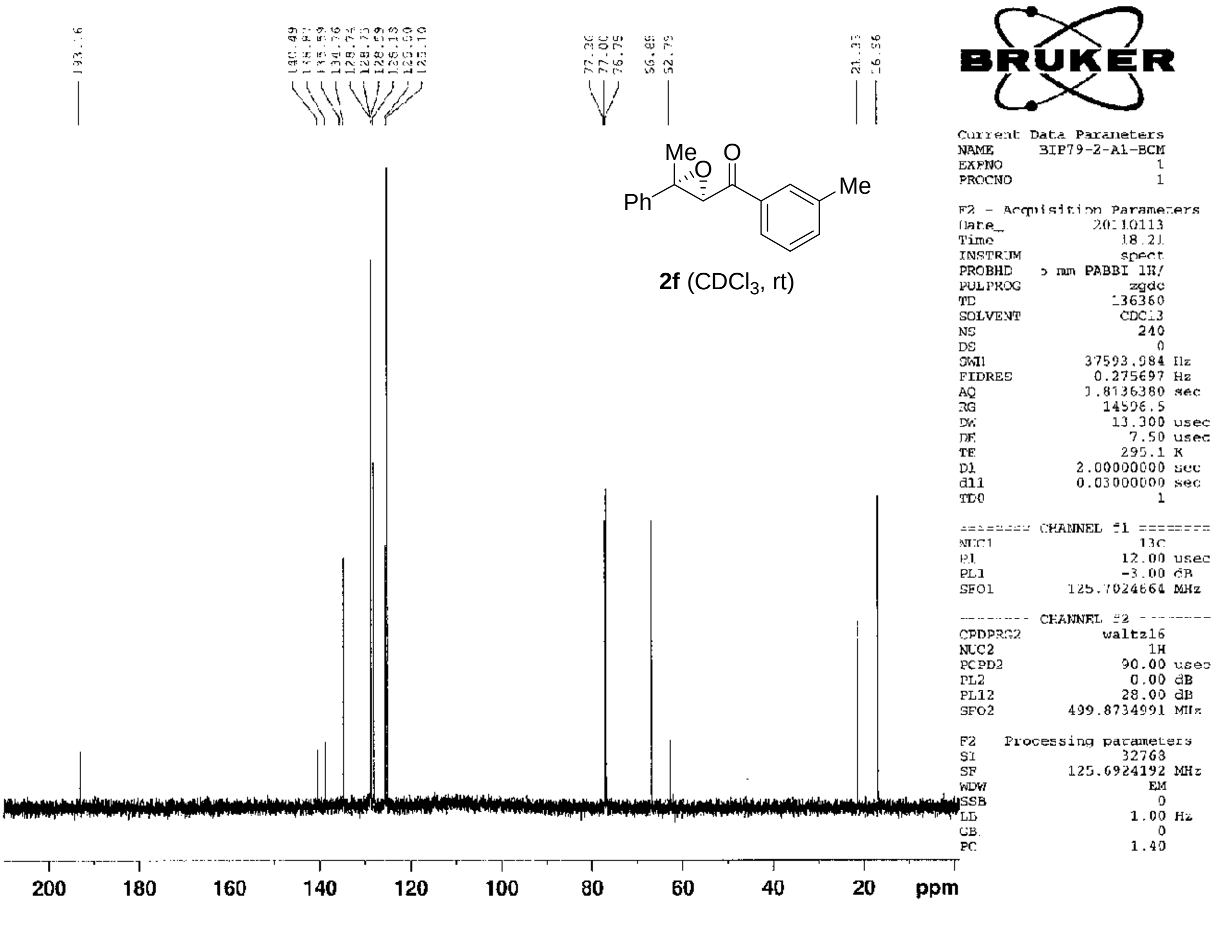




2e

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	5.6382	7.187	0.000	2848207	0.00	BB	10.6
2	94.3618	10.947	0.000	47668300	0.00	BB	18.1
	100.0000		**0.000**	**50516508**			


Me
O
O
Ph
Me
2f (CDCl$_3$, rt)
BRUKER
Current Data Parameters
NAME BIF79-2-A1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110113
Time 17.20
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 45.3
DW 50.000 usec
DE 7.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.92 1.00 2.08 3.00 2.01
1.00
3.57
3.97

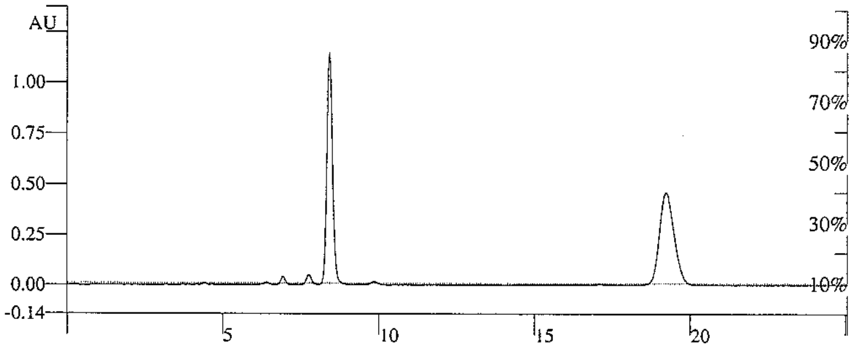

77.30
77.00
76.75
Me
O
O
Ph
Me
2f (CDCl$_3$, rt)
BRUKER
Current Data Parameters
NAME BIP79-2-A1-BCM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110113
Time 18.21
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT CDCl3
NS 240
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136380 sec
RG 14596.5
DW 13.300 usec
DE 7.50 usec
TE 295.1 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7024664 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6924192 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

Chiral HPLC analysis of compound **2f**

rac-**2f**

Data File:	c:\star\12-23-10 7;22;29 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP79-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 10/90 FR1.0	Calibration Level:	N/A
Injection Date:	12/23/10 07:22:29 PM	Run Time (min):	33.253

Injection Method: c:\star\yasuhiro\temporaly.mth

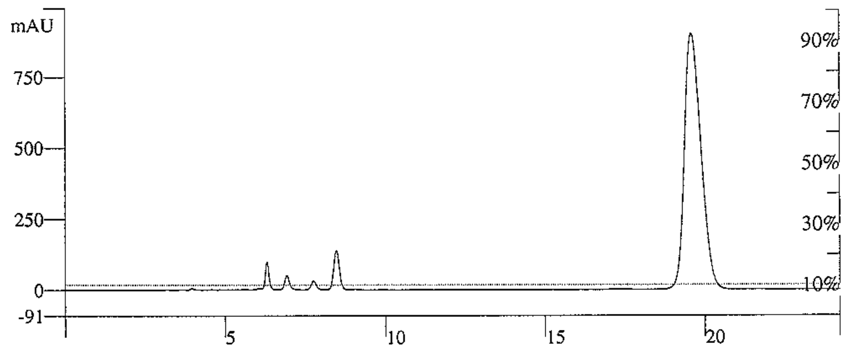


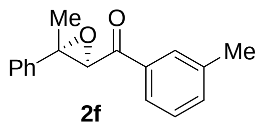
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	49.5917	8.413	0.000	75518336	0.00	BB	11.8
2	50.4083	19.213	0.000	76761848	0.00	BB	30.5
	100.0000		**0.000**	**152280192**			

chiral-**2f**

Data File:	c:\star\1-12-11 3;06;09 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP79-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	01/12/11 03:06:09 PM	Run Time (min):	24.240

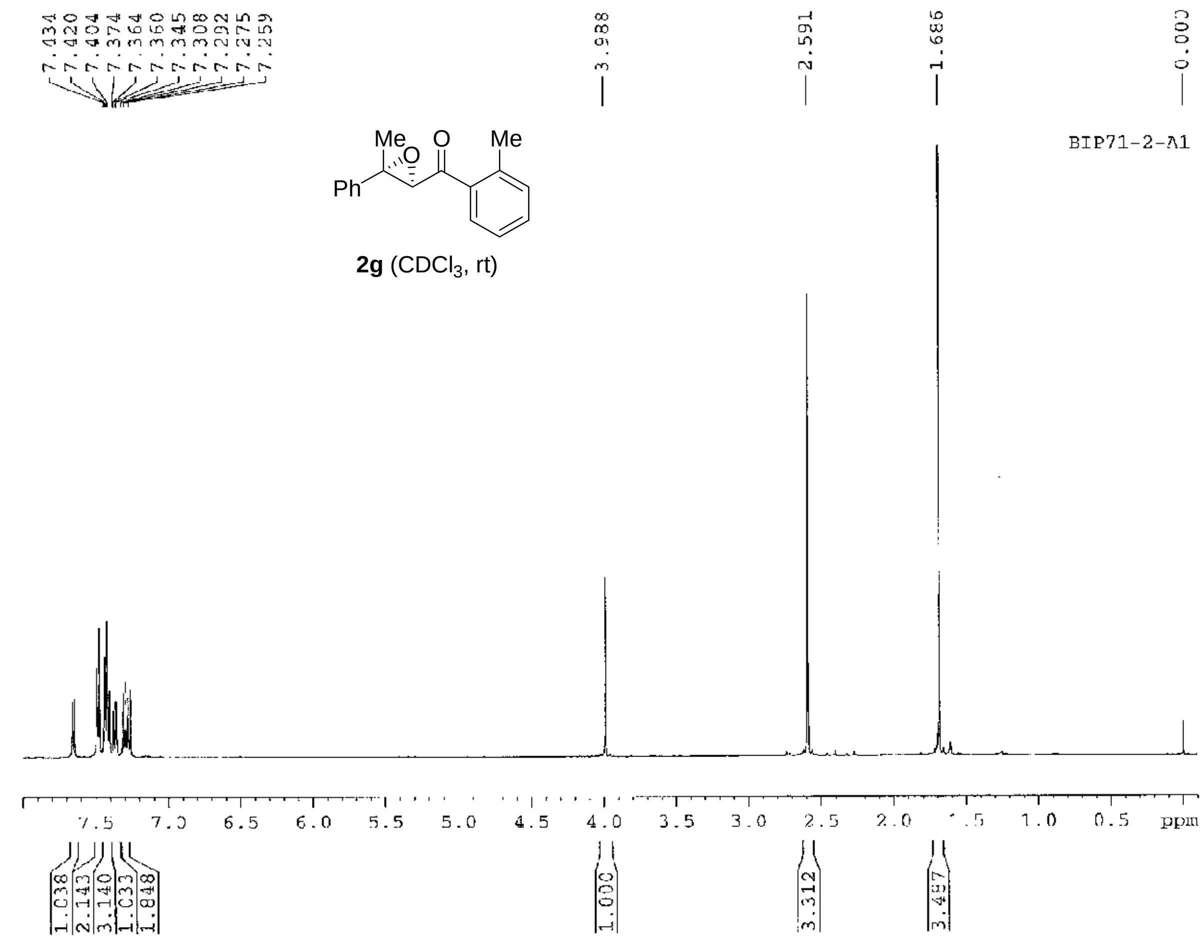
Injection Method: c:\star\yasuhiro\temporaly.mth

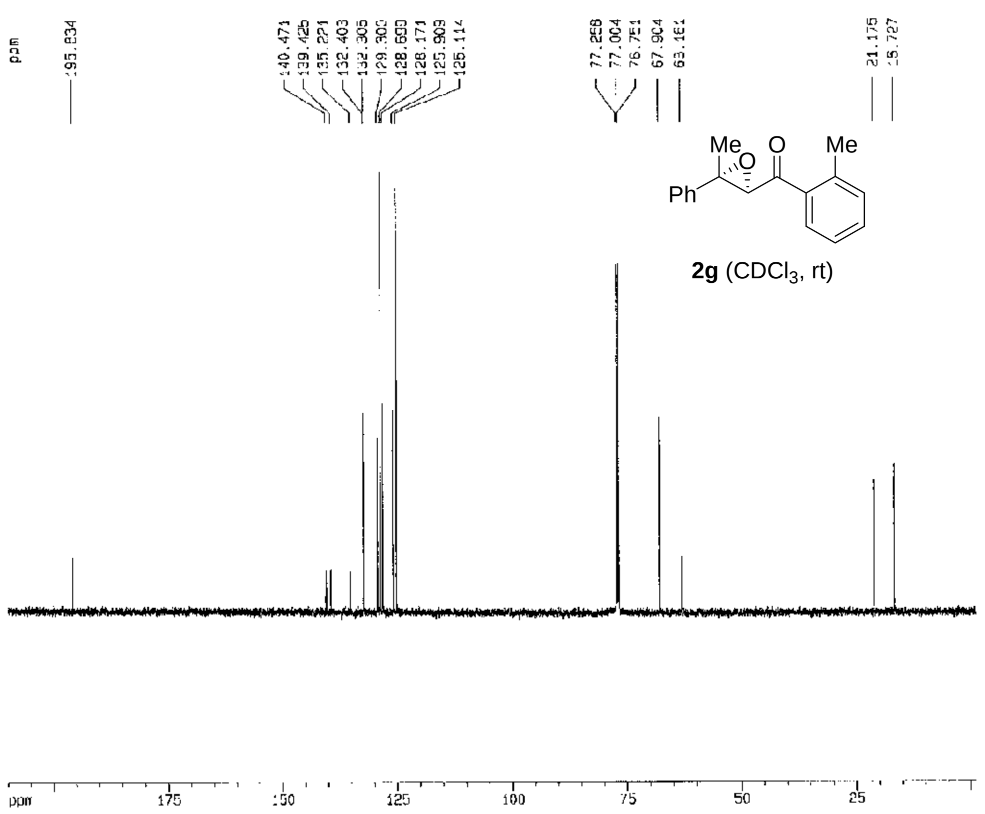




2f

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	5.0115	8.467	0.000	8734572	0.00	BB	11.5
2	94.9885	19.560	0.000	165556336	0.00	BB	33.5
	100.0000		**0.000**	**174290912**			



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Current Data Parameters
NAME      BIP71-2-A1-BCN
EXPNO              1
PROCNO             2

F2 - Acquisition Parameters
Date_          20110123
Time              17.24
INSTRUM           spect
PROBHD   5 mm BNP  1H
PULPROG          zgdc
TD               112700
SOLVENT           CDCl3
NS                  200
DS                    0
SWH          37593.984 Hz
FIDRES        0.333539 Hz
AQ           1.5000240 sec
RG                 8192
DW               13.300 usec
DE                 7.50 usec
TE                300.0 K
D1          4.00000000 sec
d11         0.03000000 sec

============ CHANNEL f1 ========
NUC1                13C
P1                 8.00 usec
PL1                3.00 dB
SFO1        125.7671708 MHz

============ CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2              120.00 dB
PL12              20.00 dB
SFO2        500.1330030 MHz

F2 - Processing parameters
SI                32768
SF          125.7577938 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

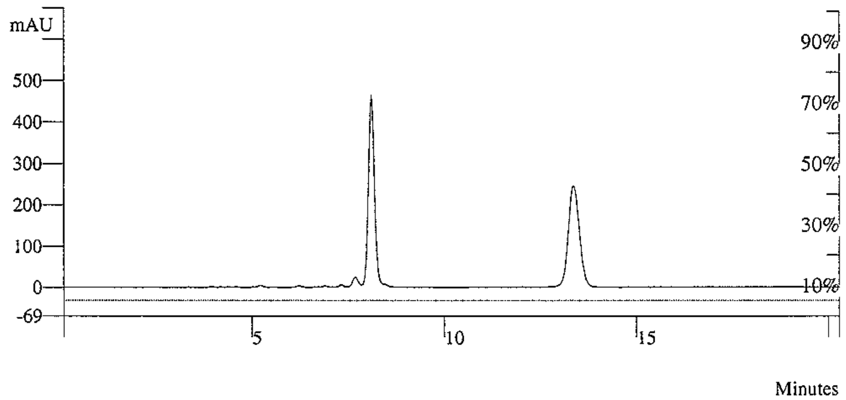
1D NMR plot parameters
CX                20.00 cm
F1P             210.000 ppm
F1             26409.13 Hz
F2P              -1.000 ppm
F2              -125.76 Hz
PPMCM          10.55000 ppm/cm
HZCM         1326.74463 Hz/cm
```

Chiral HPLC analysis of compound **2g**

rac-**2g**

Data File:	c:\star\11-10-10 3;45;52 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP71-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 5/95 FR1.0	Calibration Level:	N/A
Injection Date:	11/10/10 03:45:52 PM	Run Time (min):	35.893

Injection Method: c:\star\yasuhiro\temporaly.mth

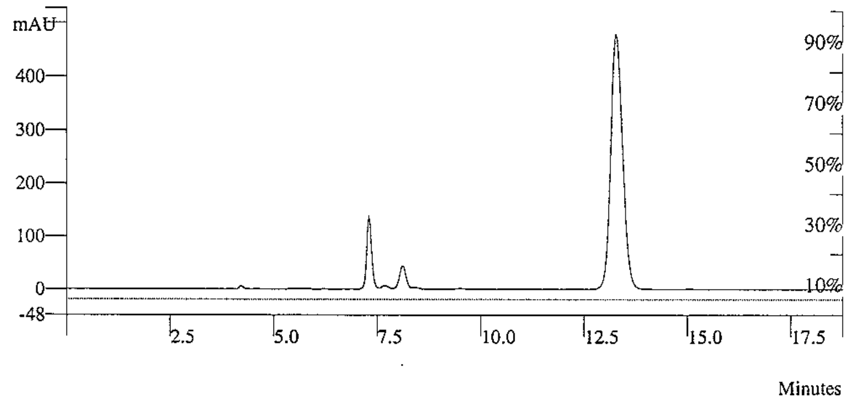


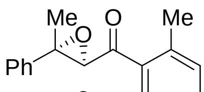
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	50.2860	8.093	0.000	25563956	0.00	BB	10.0
2	49.7140	13.373	0.000	25273168	0.00	BB	18.5
	100.0000		**0.000**	**50837124**			

chiral-**2g**

Data File:	c:\star\11-11-10 1;18;13 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP71-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	11/11/10 01:18:13 PM	Run Time (min):	18.773

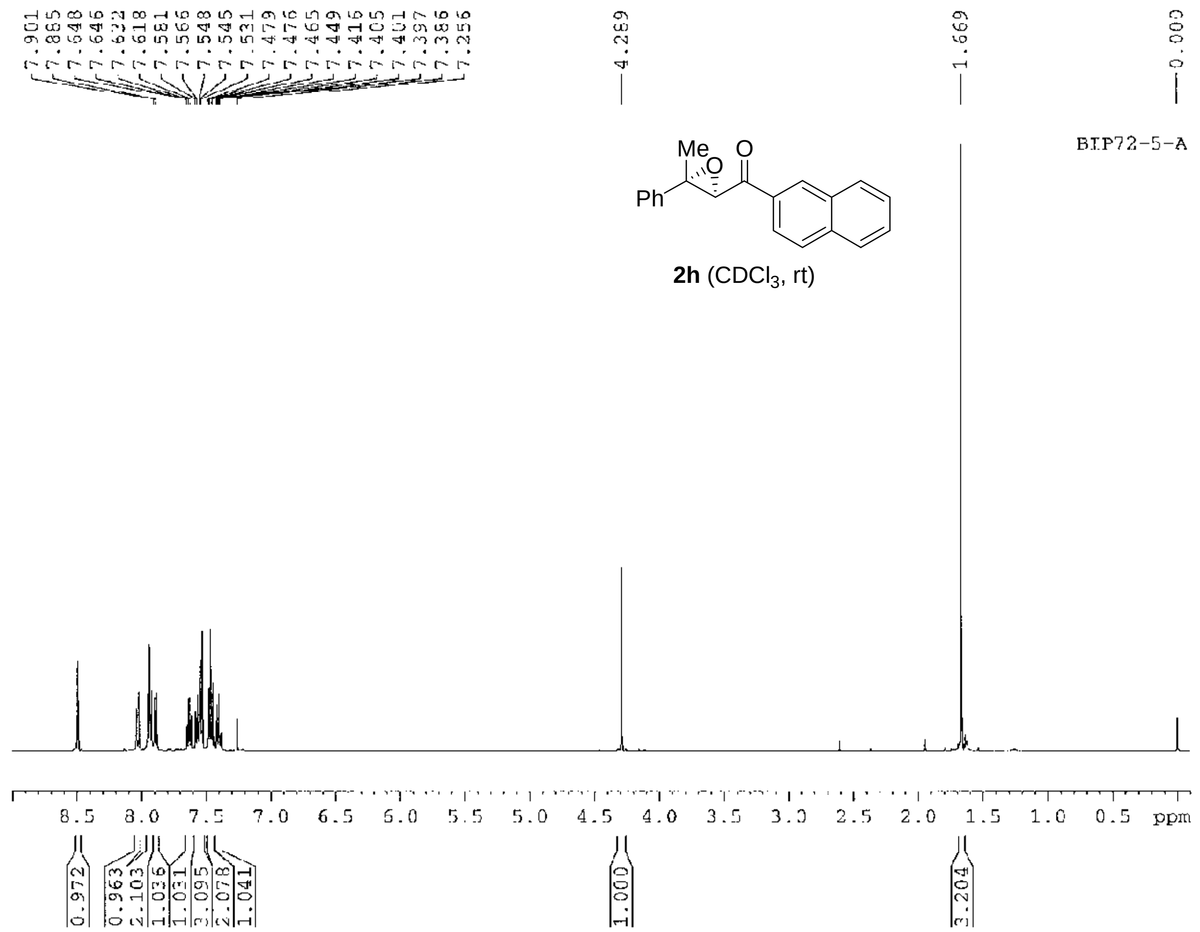
Injection Method: c:\star\yasuhiro\temporaly.mth



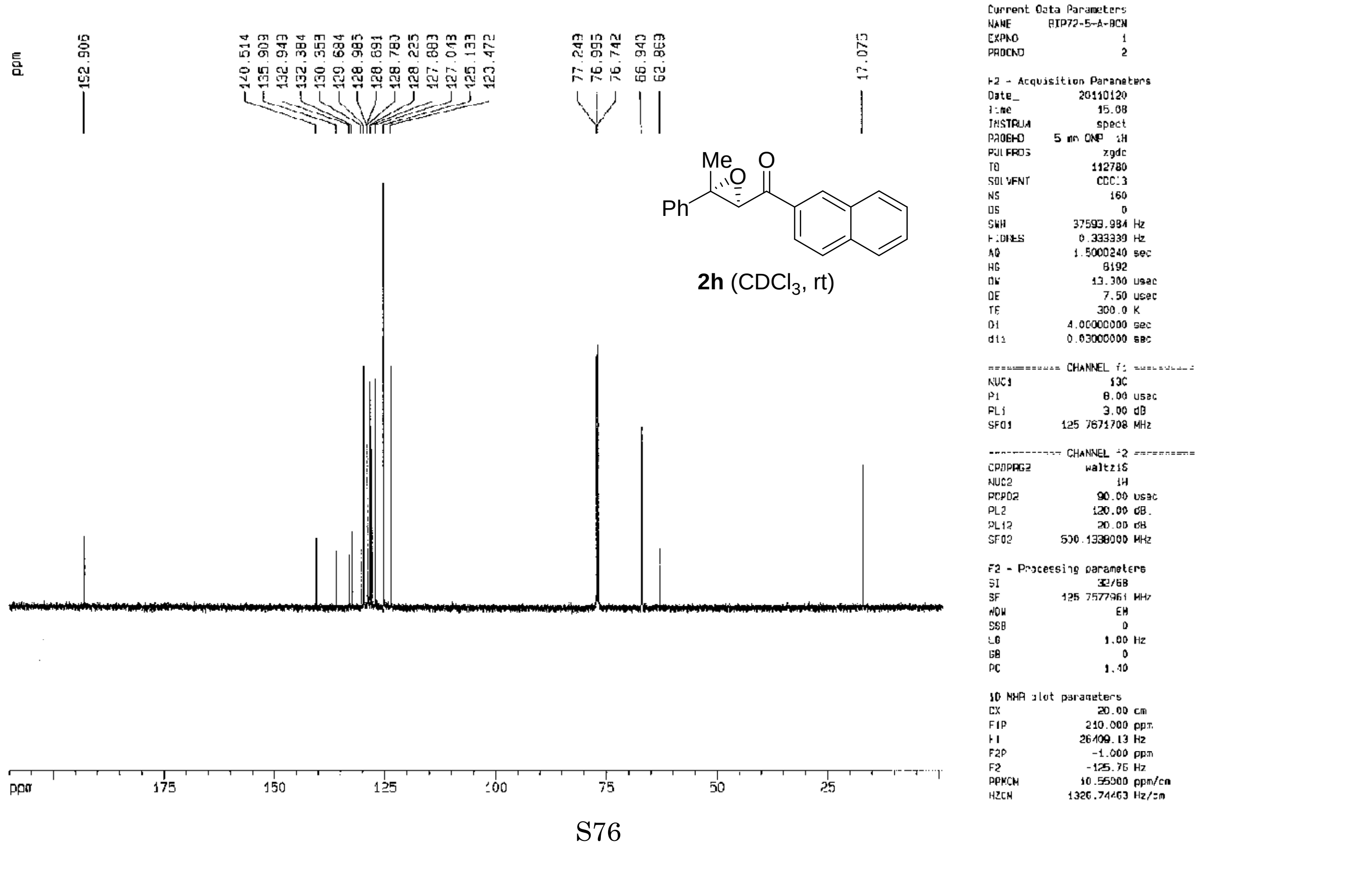


2g

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	4.1941	8.093	0.000	2147622	0.00	BB	9.6
2	95.8059	13.267	0.000	49058432	0.00	BB	18.6
	100.0000		**0.000**	**51206056**			



2h ($CDCl_3$, rt)



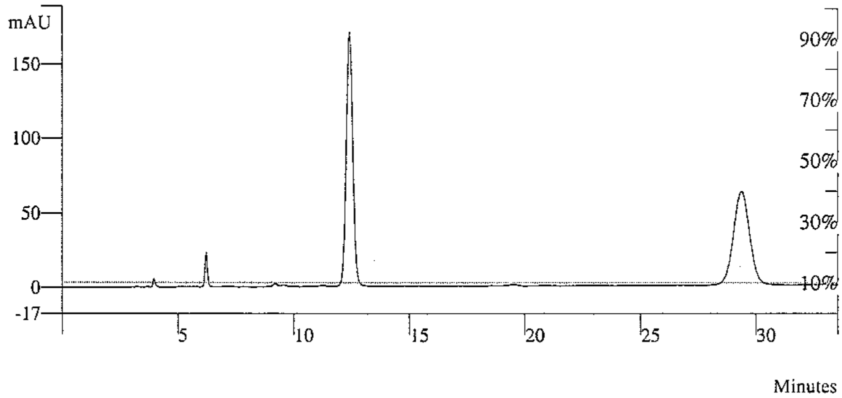
2h ($CDCl_3$, rt)

Chiral HPLC analysis of compound **2h**

rac-**2h**

Data File:	c:\star\11-18-10 7;05;20 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP72-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	11/18/10 07:05:20 PM	Run Time (min):	33.573

Injection Method: c:\star\yasuhiro\temporaly.mth

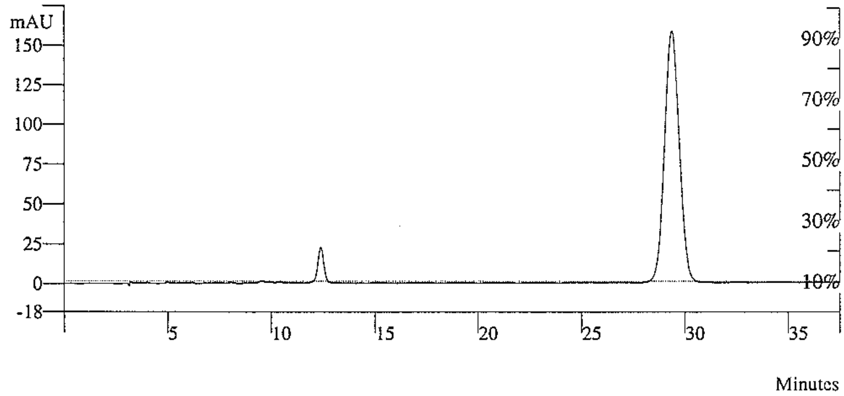


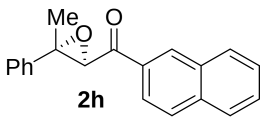
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	52.3713	12.413	0.000	17317630	0.00	BB	18.3
2	47.6287	29.373	0.000	15749393	0.00	BB	45.8
	100.0000		**0.000**	**33067024**			

chiral-**2h**

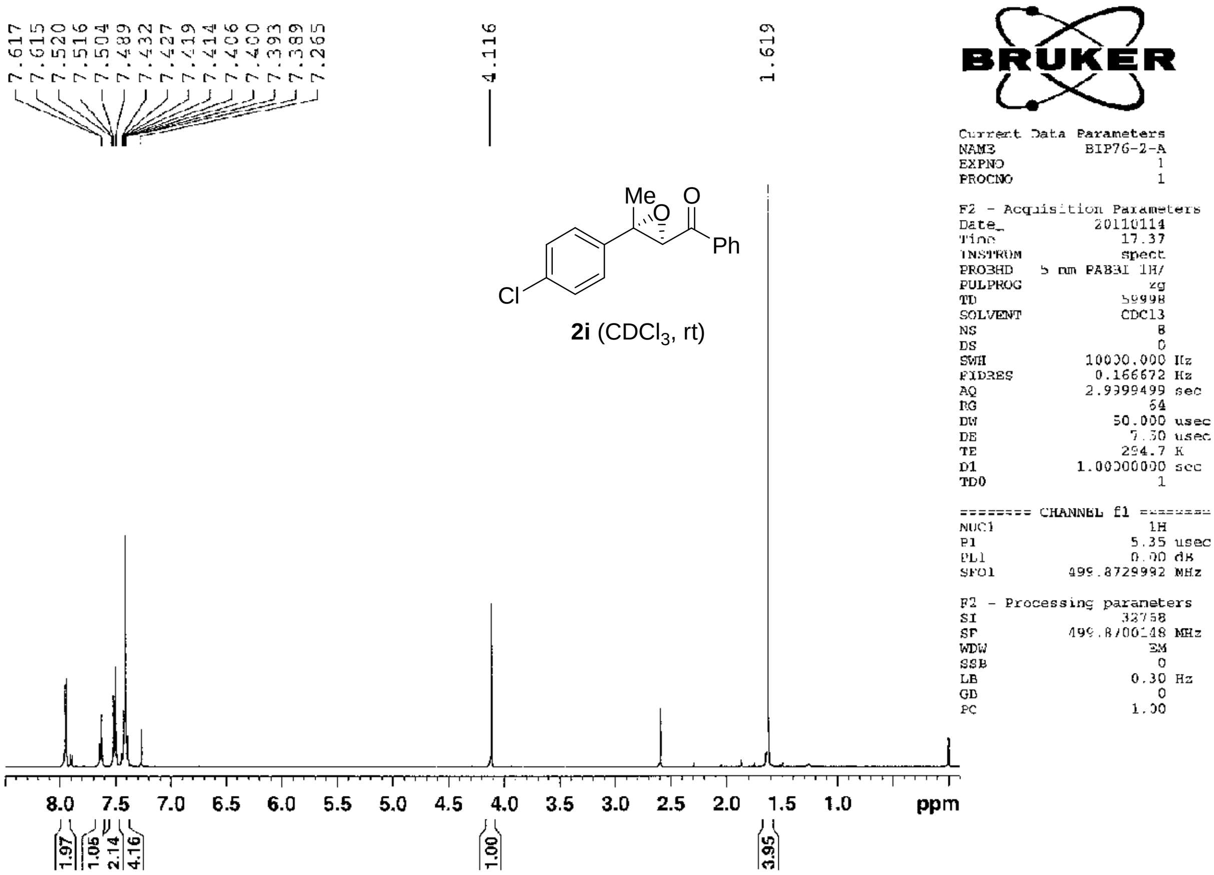
Data File:	c:\star\11-19-10 2;52;49 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP72-5-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	11/19/10 02:52:49 PM	Run Time (min):	37.520

Injection Method: c:\star\yasuhiro\temporaly.mth





Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	5.0530	12.387	0.000	2156610	0.00	BB	17.7
2	94.9470	29.347	0.000	40523036	0.00	BB	46.3
	100.0000		**0.000**	**42679648**			



7.617
7.615
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7.516
7.504
7.489
7.432
7.427
7.419
7.414
7.406
7.400
7.393
7.389
7.265
4.116
1.619
Me
O
O
Ph
Cl
2i (CDCl3, rt)
Current Data Parameters
NAME BIP76-2-A
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110114
Time 17.37
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 64
DW 50.000 usec
DE 7.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700148 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.97
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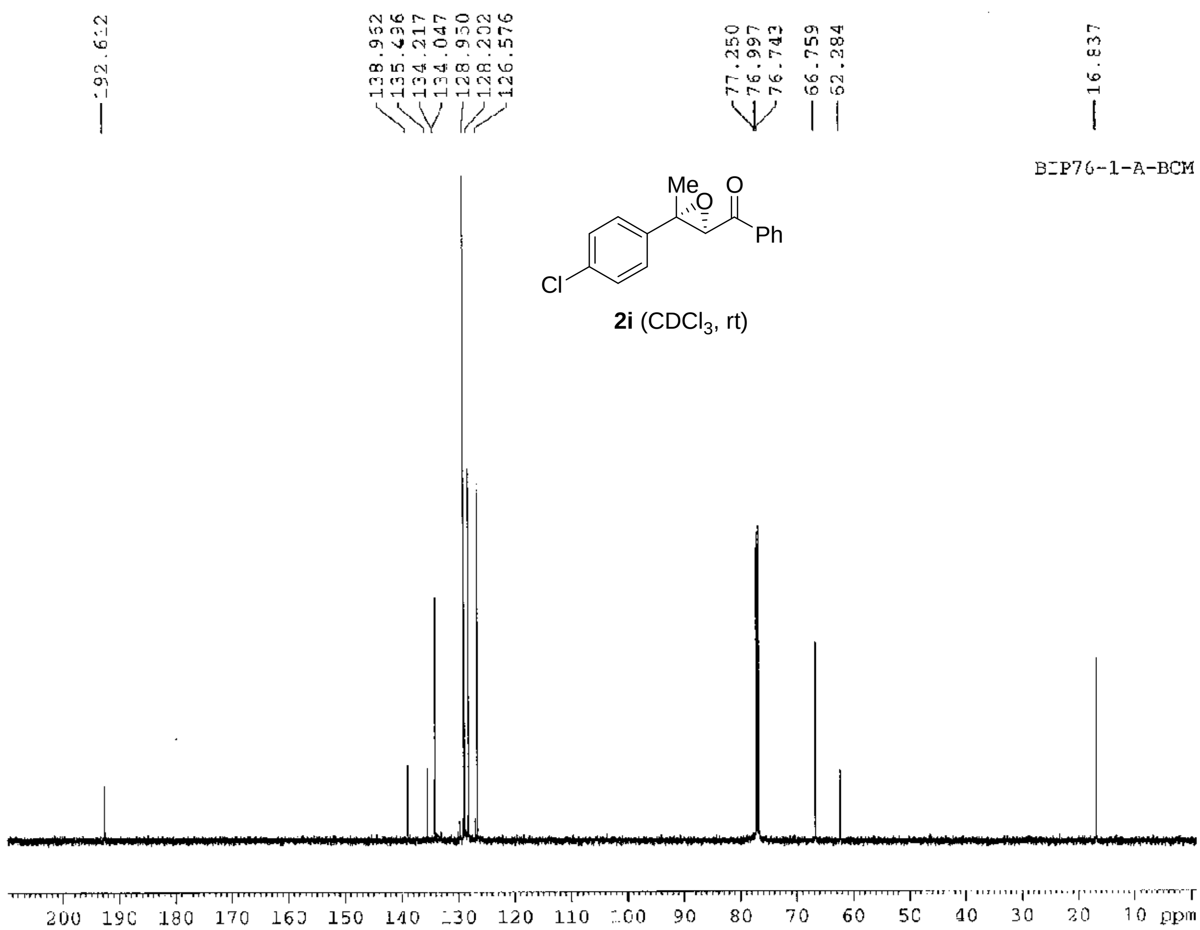
4.16
1.00
3.95



192.612
138.952
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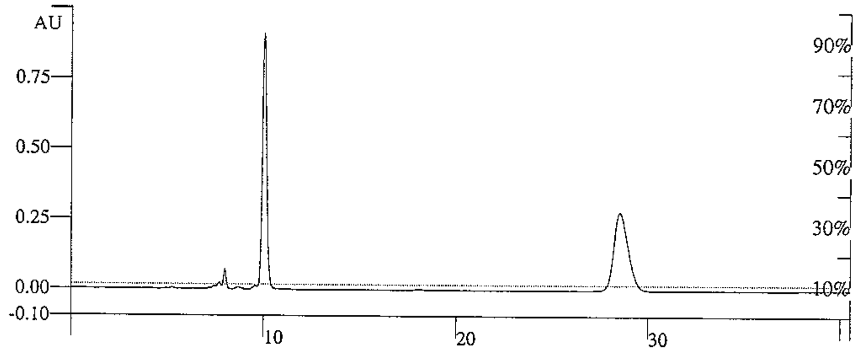
134.217
134.047
128.950
128.202
126.576
77.250
76.997
76.743
66.759
62.284
16.837
BIP76-1-A-BCM
Me
O
O
Ph
Cl
2i (CDCl3, rt)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Chiral HPLC analysis of compound **2i**

rac-**2i**

Data File:	c:\star\12-23-10 5;20;33 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP76-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 10/90 FR1.0	Calibration Level:	N/A
Injection Date:	12/23/10 05:20:33 PM	Run Time (min):	40.560

Injection Method: c:\star\yasuhiro\temporaly.mth

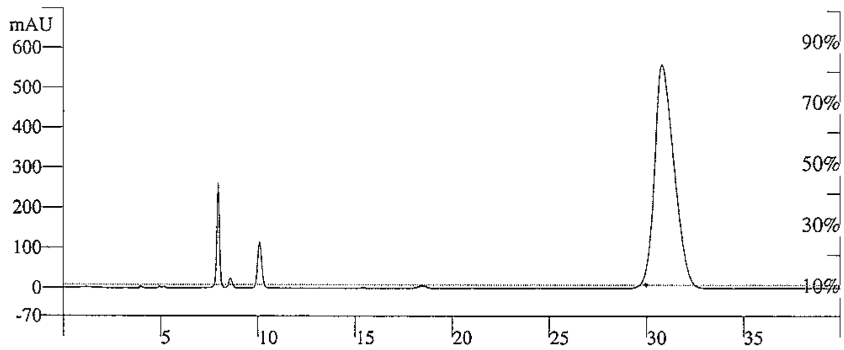


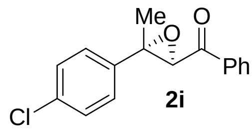
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	49.1877	10.013	0.000	71183960	0.00	BB	14.3
2	50.8123	28.520	0.000	73535112	0.00	BB	48.0
	100.0000		**0.000**	**144719072**			

chiral-**2i**

Data File:	c:\star\1-14-11 2;21;52 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP76-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	01/14/11 02:21:52 PM	Run Time (min):	50.880

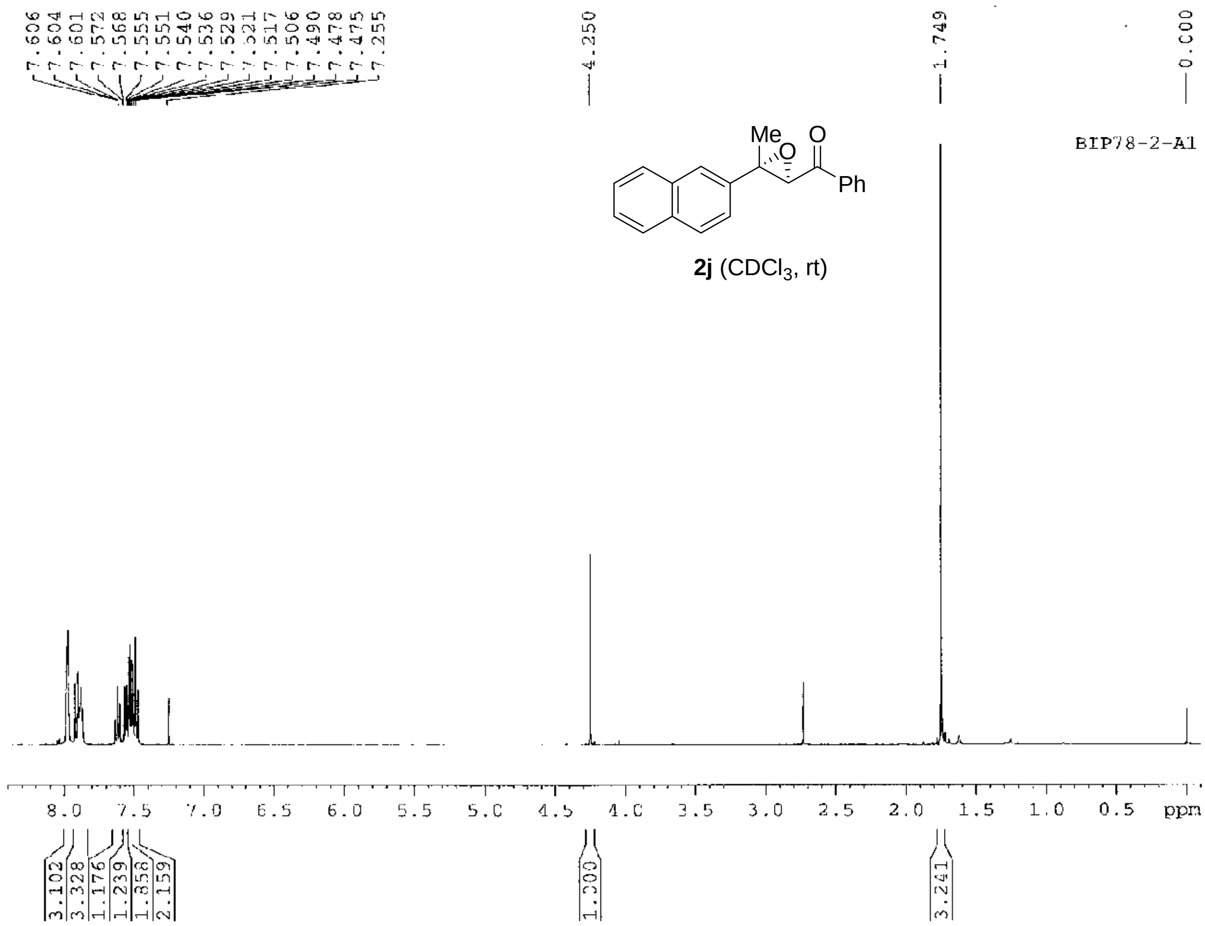
Injection Method: c:\star\yasuhiro\temporaly.mth

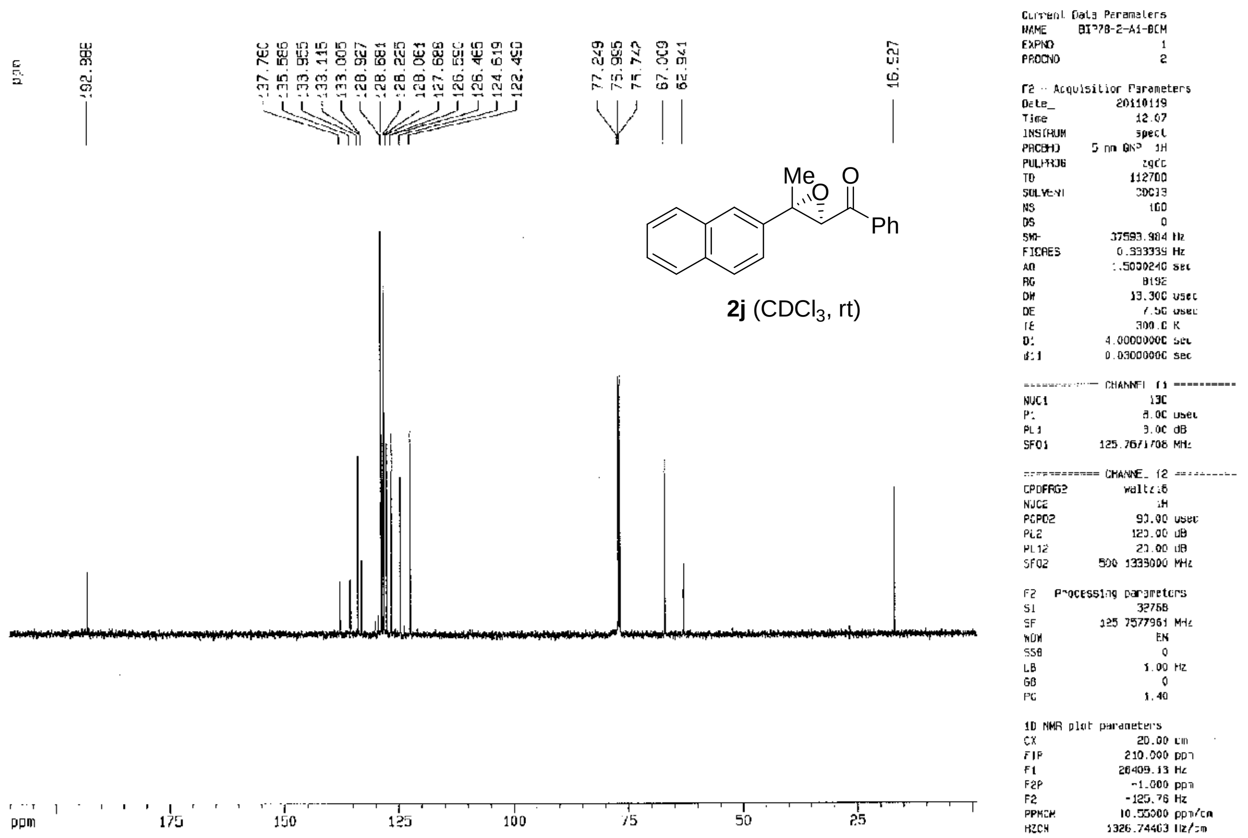




2i

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	4.2120	10.067	0.000	8787672	0.00	BB	13.9
2	95.7880	30.787	0.000	199847744	0.00	BB	64.2
	100.0000		**0.000**	**208635424**			


BIP78-2-A1
Me
O
O
Ph
2j (CDCl3, rt)
ppm

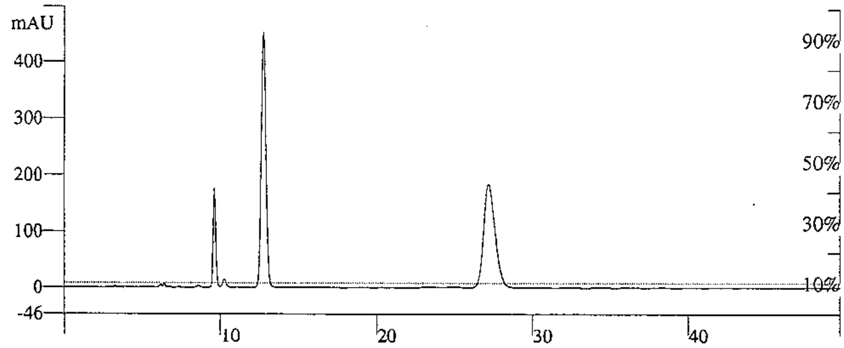

Me
O
O
Ph
2j (CDCl3, rt)
ppm

Chiral HPLC analysis of compound **2j**

rac-**2j**

Data File:	c:\star\1-18-11 12;18;59 am -1.run	Run Mode:	Analysis
Sample ID:	BIP78-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 10/90 FR1.0	Calibration Level:	N/A
Injection Date:	01/18/11 12:18:59 AM	Run Time (min):	49.787

Injection Method: c:\star\yasuhiro\temporaly.mth

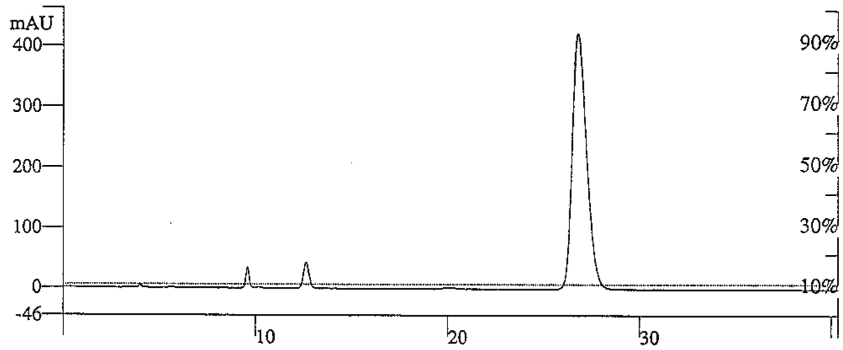


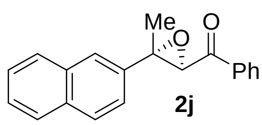
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	50.0059	12.733	0.000	47659984	0.00	BB	19.1
2	49.9941	27.187	0.000	47648672	0.00	BB	47.0
	100.0000		**0.000**	**95308656**			

chiral-**2j**

Data File:	c:\star\1-18-11 10;34;27 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP78-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	01/18/11 10:34:27 PM	Run Time (min):	40.373

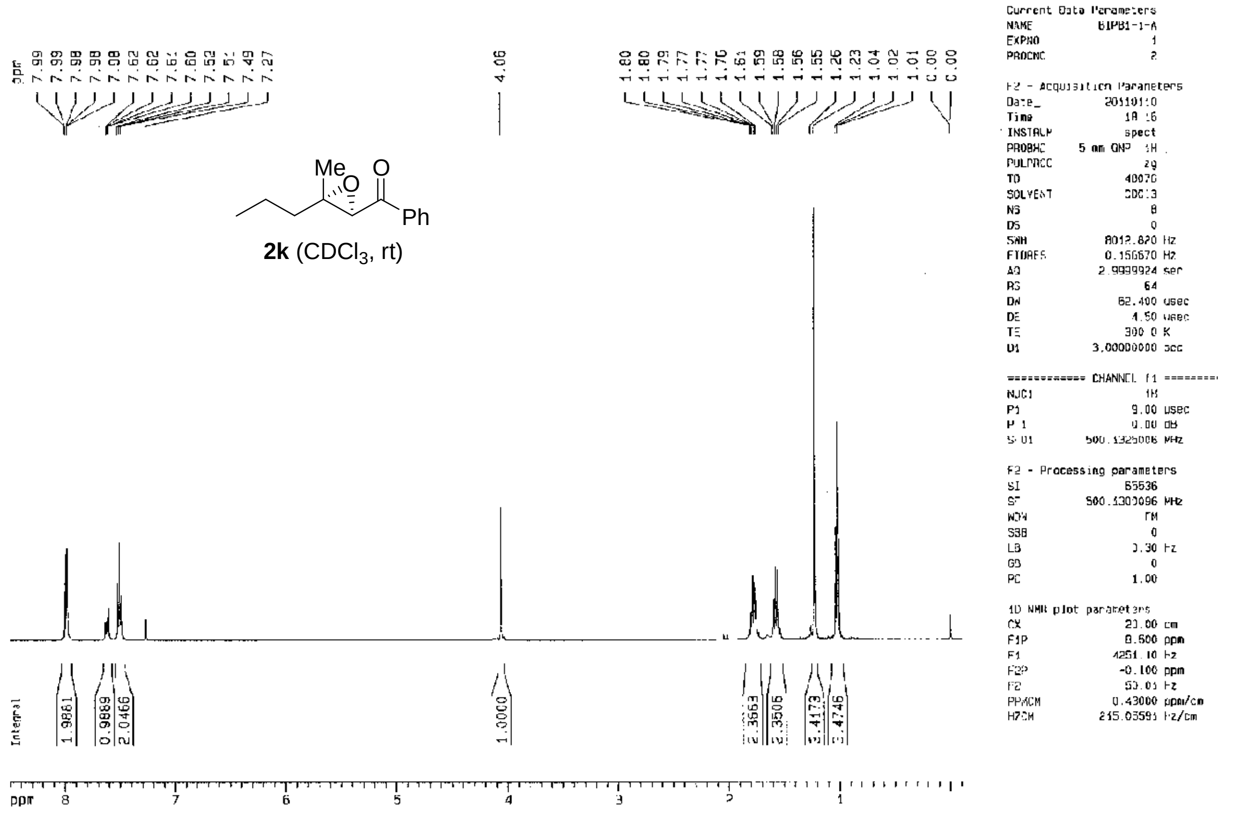
Injection Method: c:\star\yasuhiro\temporaly.mth

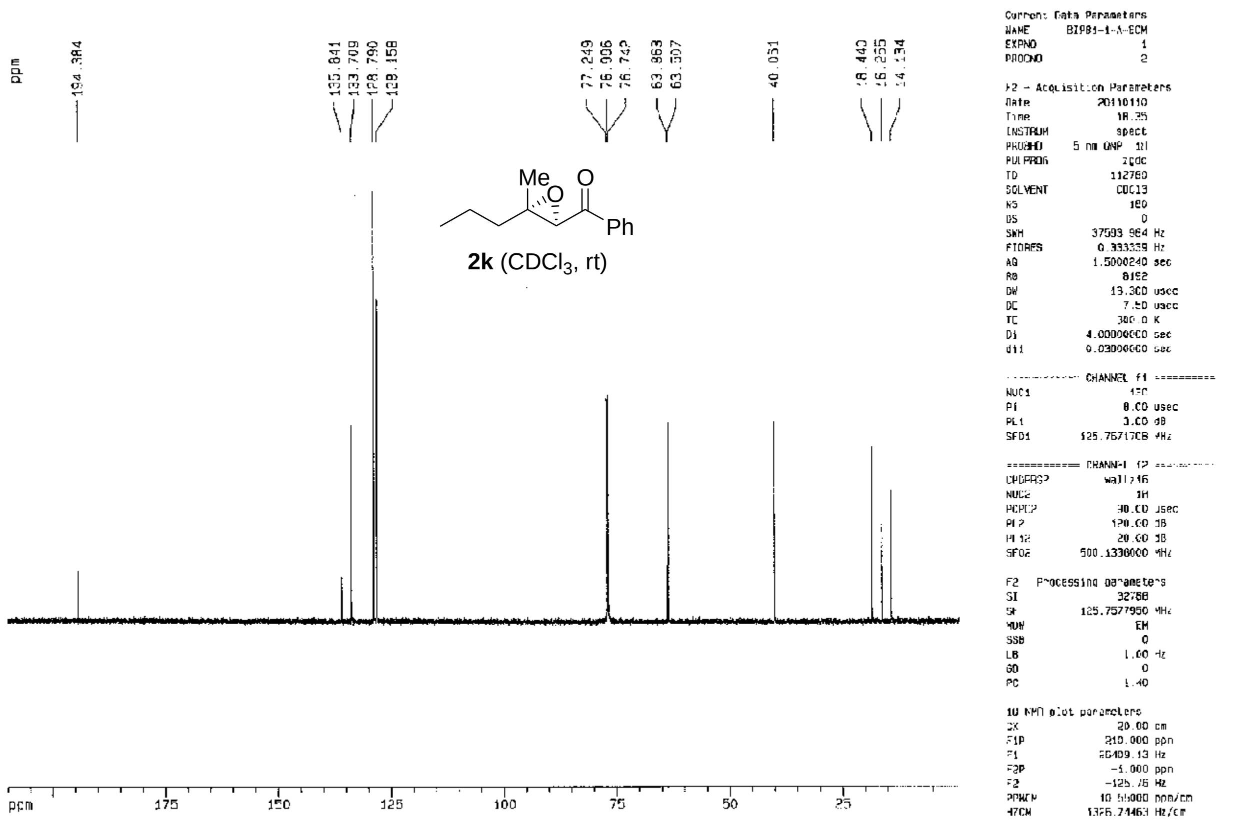




2j

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	3.7732	12.627	0.000	4420293	0.00	BB	18.6
2	96.2268	26.787	0.000	112730496	0.00	BB	48.4
	100.0000		**0.000**	**117150792**			


2k (CDCl3, rt)
Current Data Parameters
NAME BIPB1-1-A
F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters
Integral
ppm

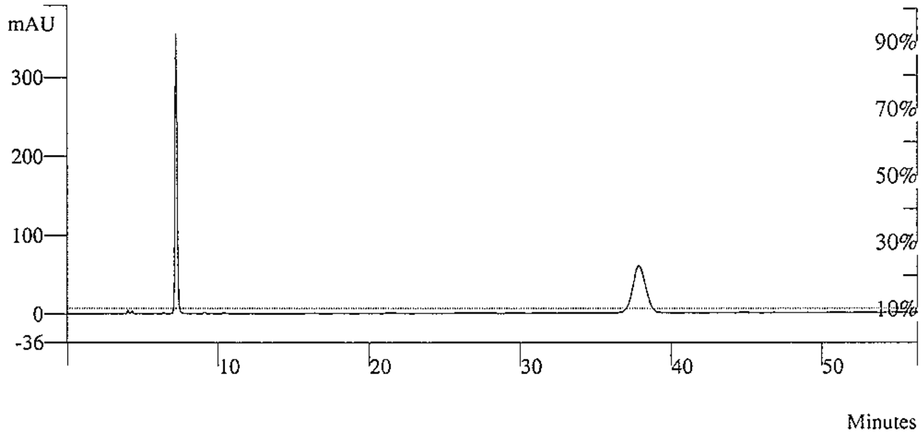

2k (CDCl3, rt)
Current Data Parameters
F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters
ppm

Chiral HPLC analysis of compound **2k**

rac- **2k**

Data File:	c:\star\1-6-11 6;17;32 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP81-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 10/90 FR1.0	Calibration Level:	N/A
Injection Date:	01/06/11 06:17:32 PM	Run Time (min):	56.373

Injection Method: c:\star\yasuhiro\temporaly.mth

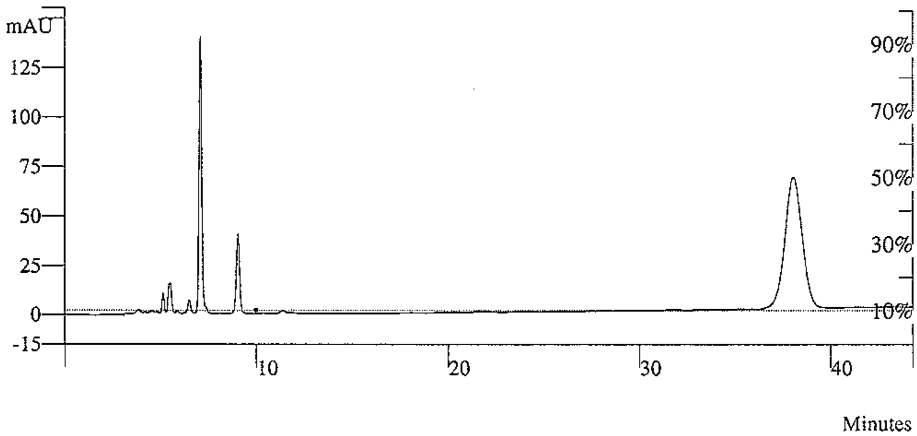


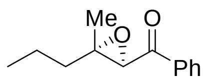
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	49.7091	7.187	0.000	17879268	0.00	BB	9.0
2	50.2909	37.853	0.000	18088532	0.00	BB	55.1
	100.0000		**0.000**	**35967800**			

chiral-**2k**

Data File:	c:\star\4-21-11 10;22;16 am -1.run	Run Mode:	Analysis
Sample ID:	BIP81-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	04/21/11 10:22:16 AM	Run Time (min):	44.320

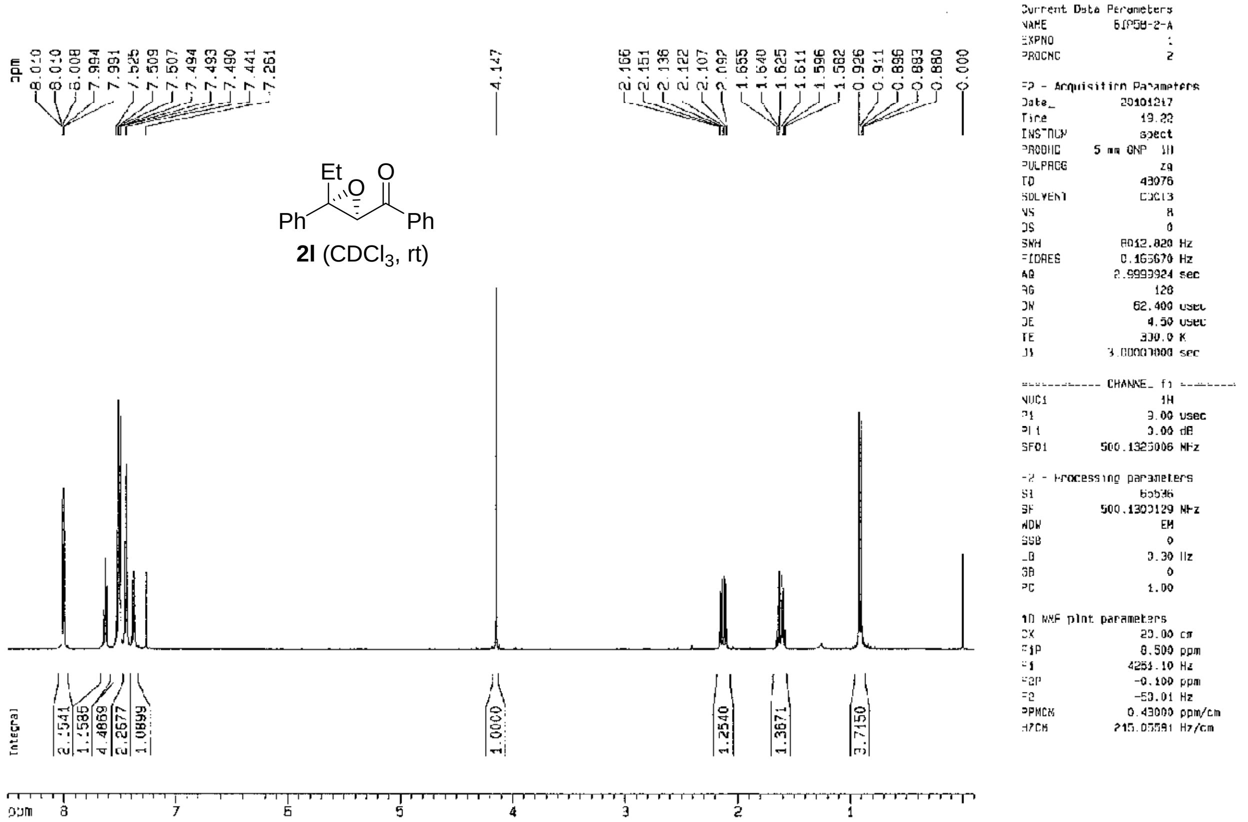
Injection Method: c:\star\yasuhiro\temporaly.mth

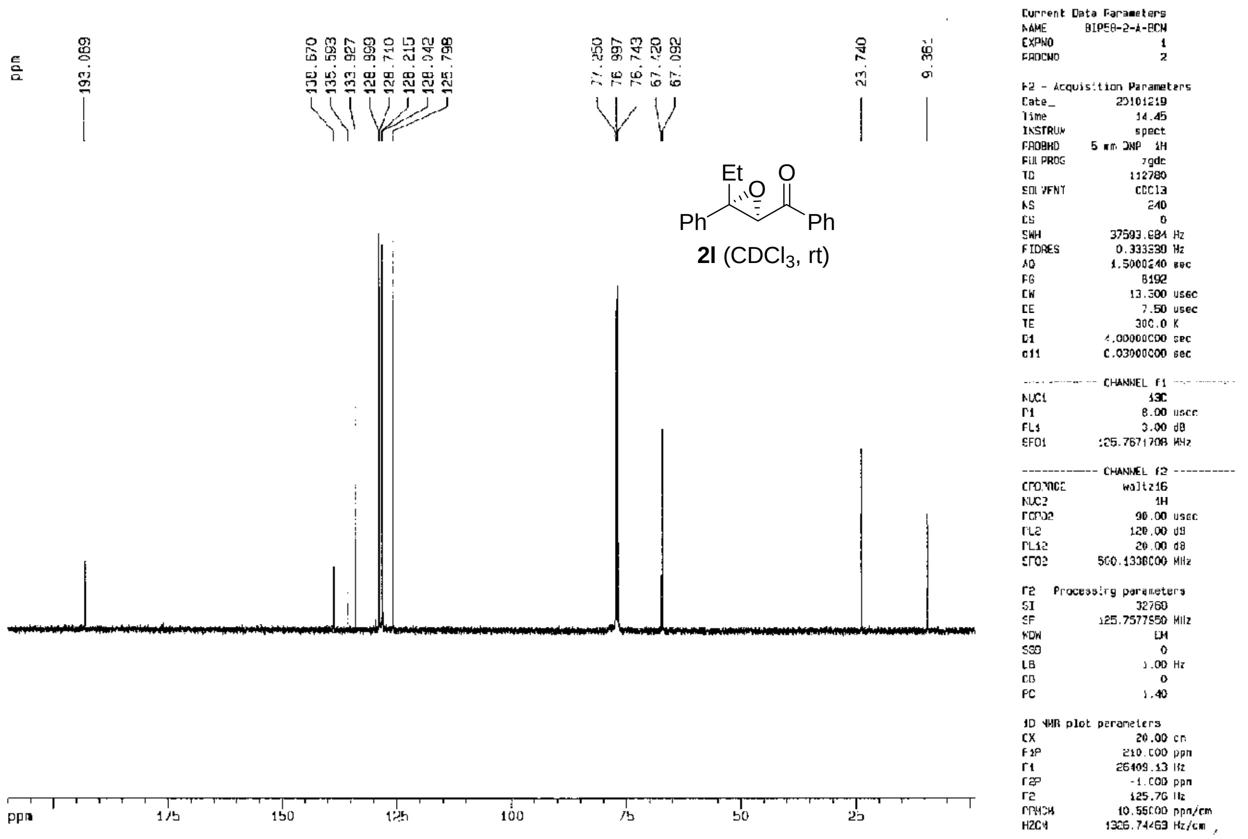




2k

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	24.6830	7.080	0.000	7055874	0.00	BB	8.8
2	75.3170	38.013	0.000	21530118	0.00	BB	58.4
	100.0000		**0.000**	**28585992**			


2l (CDCl3, rt)
8.010 8.010 8.008 7.994 7.991 7.525 7.509 7.507 7.494 7.493 7.490 7.441 7.261
4.147
2.166 2.151 2.136 2.122 2.107 2.092 1.655 1.640 1.625 1.611 1.596 1.582 0.926 0.911 0.896 0.883 0.880 0.000
Integral
2.1541 1.1585 4.4869 2.2677 1.0899 1.0000 1.2540 1.3671 3.7150
ppm

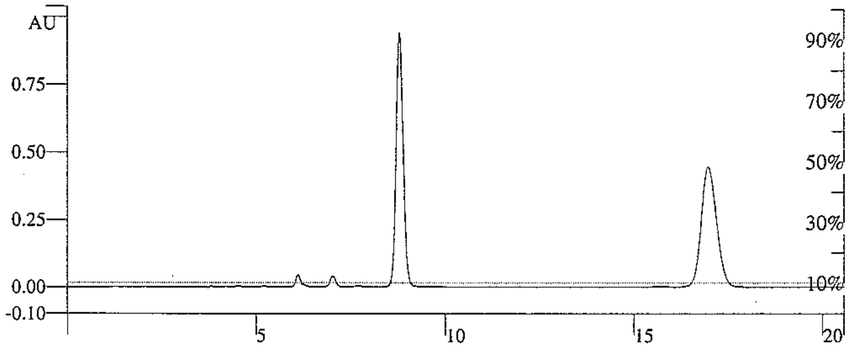

2l (CDCl3, rt)
193.069
136.670 135.593 133.927 128.889 128.710 128.215 128.042 125.798
77.250 76.997 76.743 67.420 67.092
23.740
9.361
ppm

Chiral HPLC analysis of compound **2l**

rac-**2l**

Data File:	c:\star\10-14-10 6;00;09 am -1.run	Run Mode:	Analysis
Sample ID:	BIP58-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 10/90 FR1.0	Calibration Level:	N/A
Injection Date:	10/14/10 06:00:09 AM	Run Time (min):	20.587

Injection Method: c:\star\yasuhiro\temporaly.mth

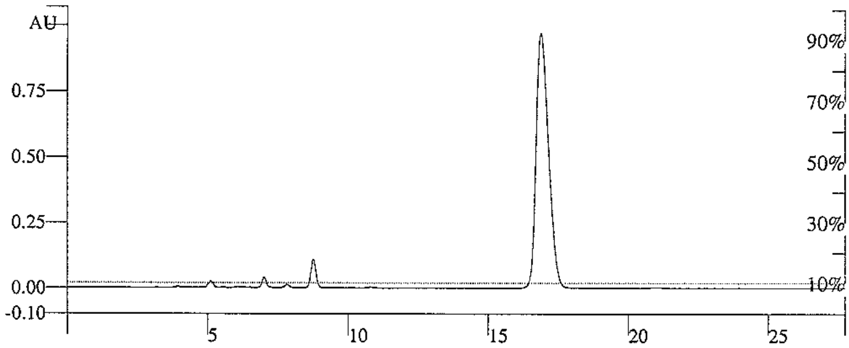


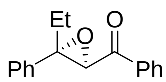
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	49.5016	8.787	0.000	62768716	0.00	BB	12.0
2	50.4984	16.947	0.000	64032712	0.00	BB	26.2
	100.0000		**0.000**	**126801424**			

chiral-**2l**

Data File:	c:\star\10-14-10 7;54;13 am -1.run	Run Mode:	Analysis
Sample ID:	BIP58-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	10/14/10 07:54:13 AM	Run Time (min):	27.760

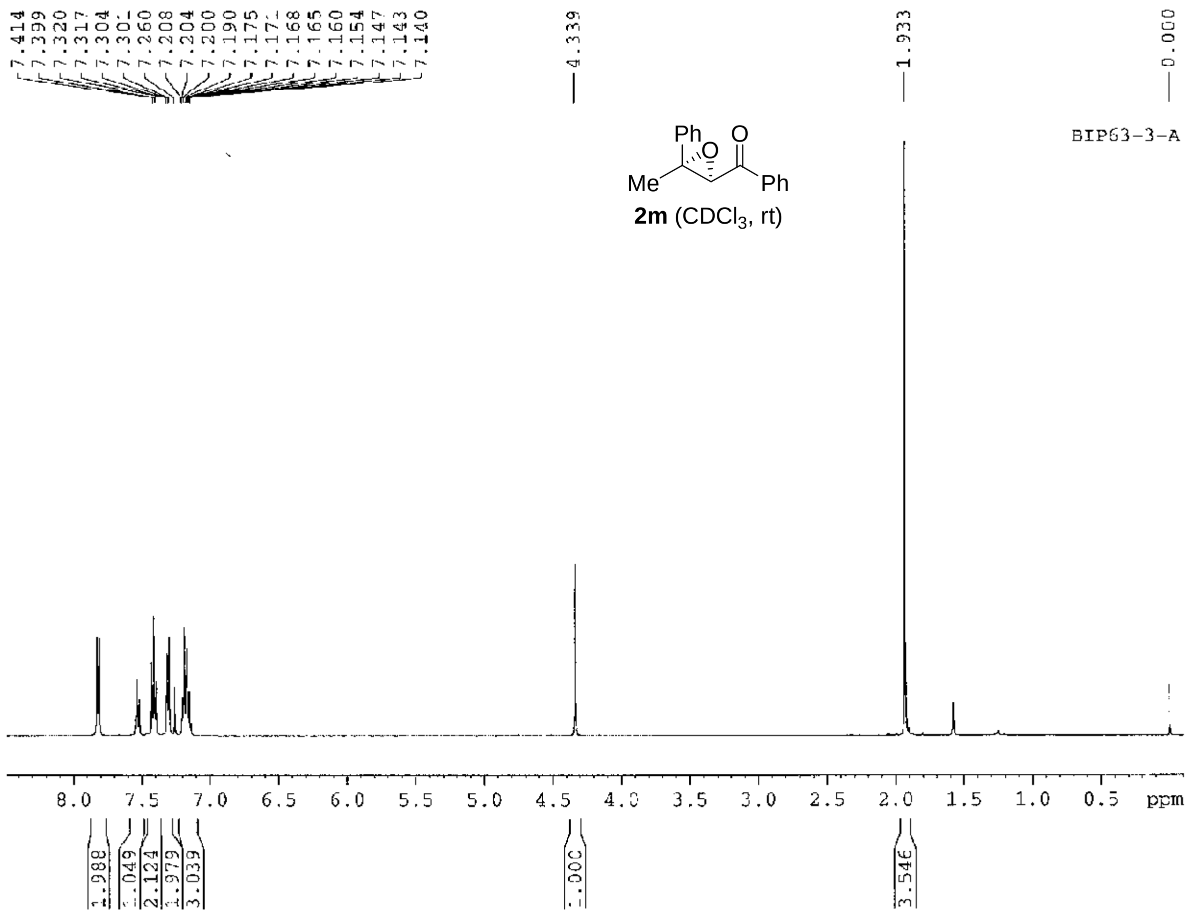
Injection Method: c:\star\yasuhiro\temporaly.mth

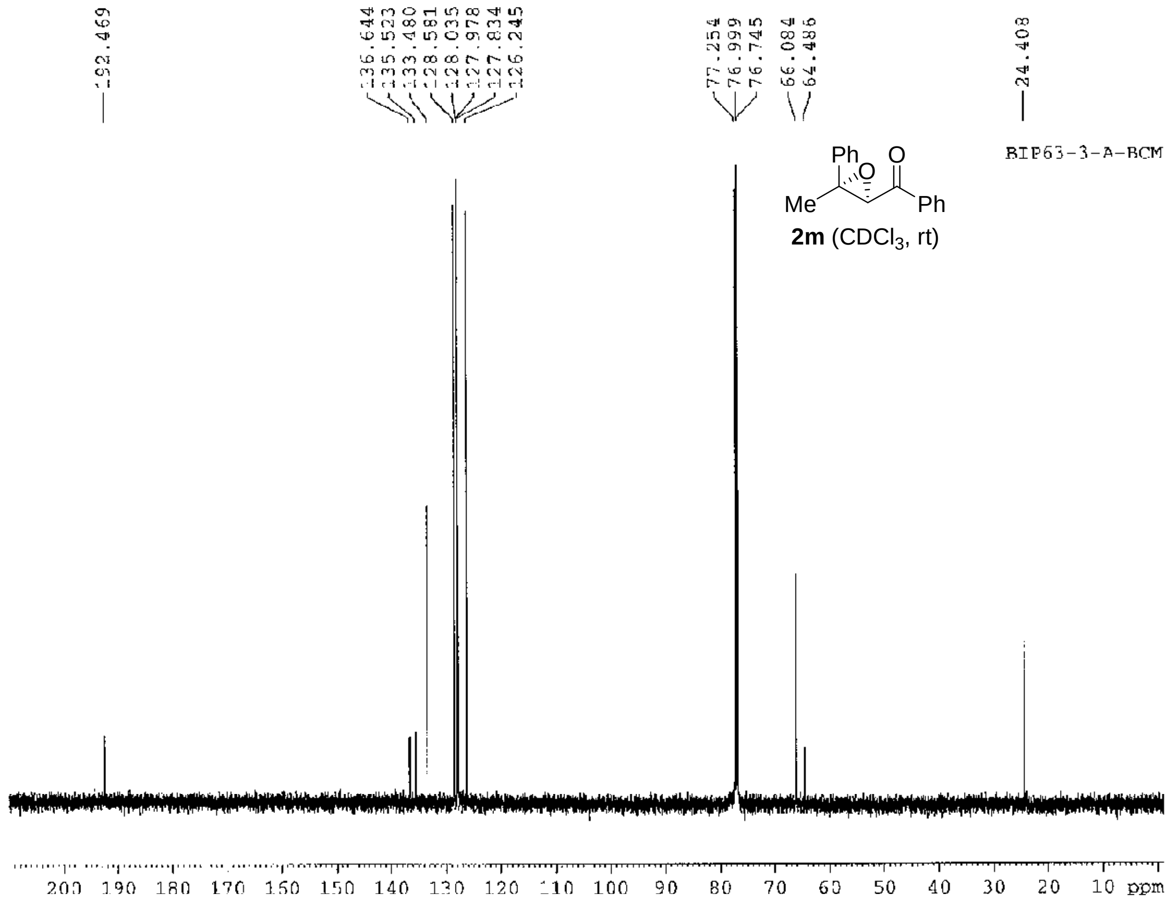




2l

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	4.1924	8.760	0.000	6712514	0.00	BB	11.4
2	95.8076	16.867	0.000	153400800	0.00	BB	29.0
	100.0000		**0.000**	**160113312**			


BIP63-3-A
2m (CDCl3, rt)
ppm

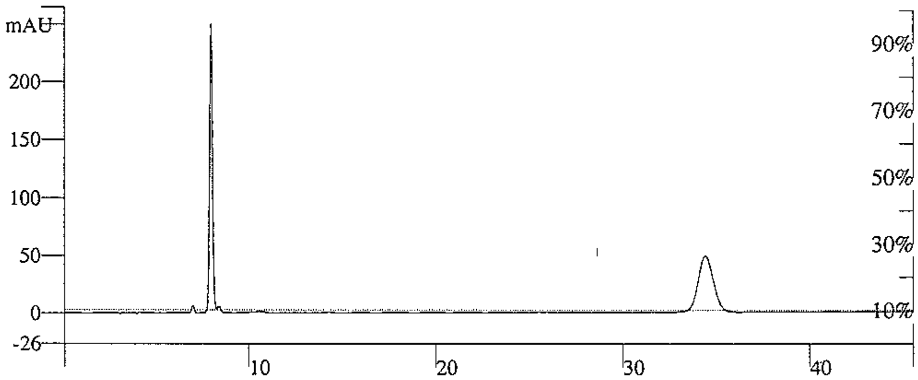

BIP63-3-A-BCM
2m (CDCl3, rt)
ppm

Chiral HPLC analysis of compound **2m**

rac-**2m**

Data File:	c:\star\10-26-10 5;31;13 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP63-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 10/90 FR1.0	Calibration Level:	N/A
Injection Date:	10/26/10 05:31:13 PM	Run Time (min):	55.707

Injection Method: c:\star\yasuhiro\temporaly.mth

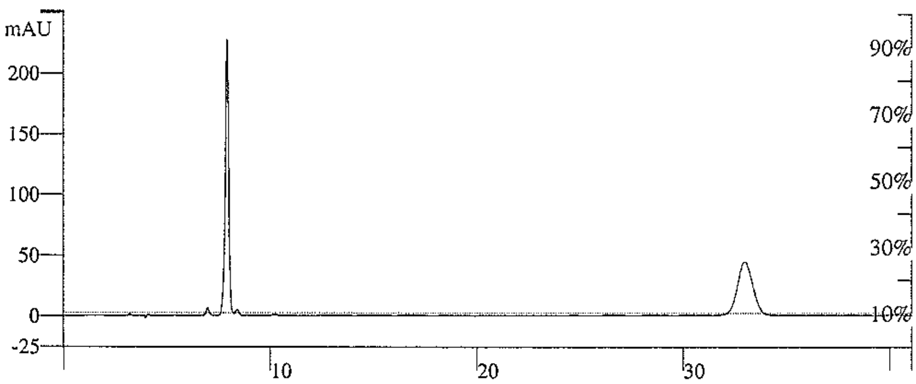


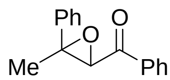
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	50.5427	7.987	0.000	14354109	0.00	BB	10.1
2	49.4573	34.387	0.000	14045867	0.00	BB	52.6
	100.0000		**0.000**	**28399976**			

chiral-**2m**

Data File:	c:\star\10-28-10 3;19;07 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP63-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	10/28/10 03:19:07 PM	Run Time (min):	41.093

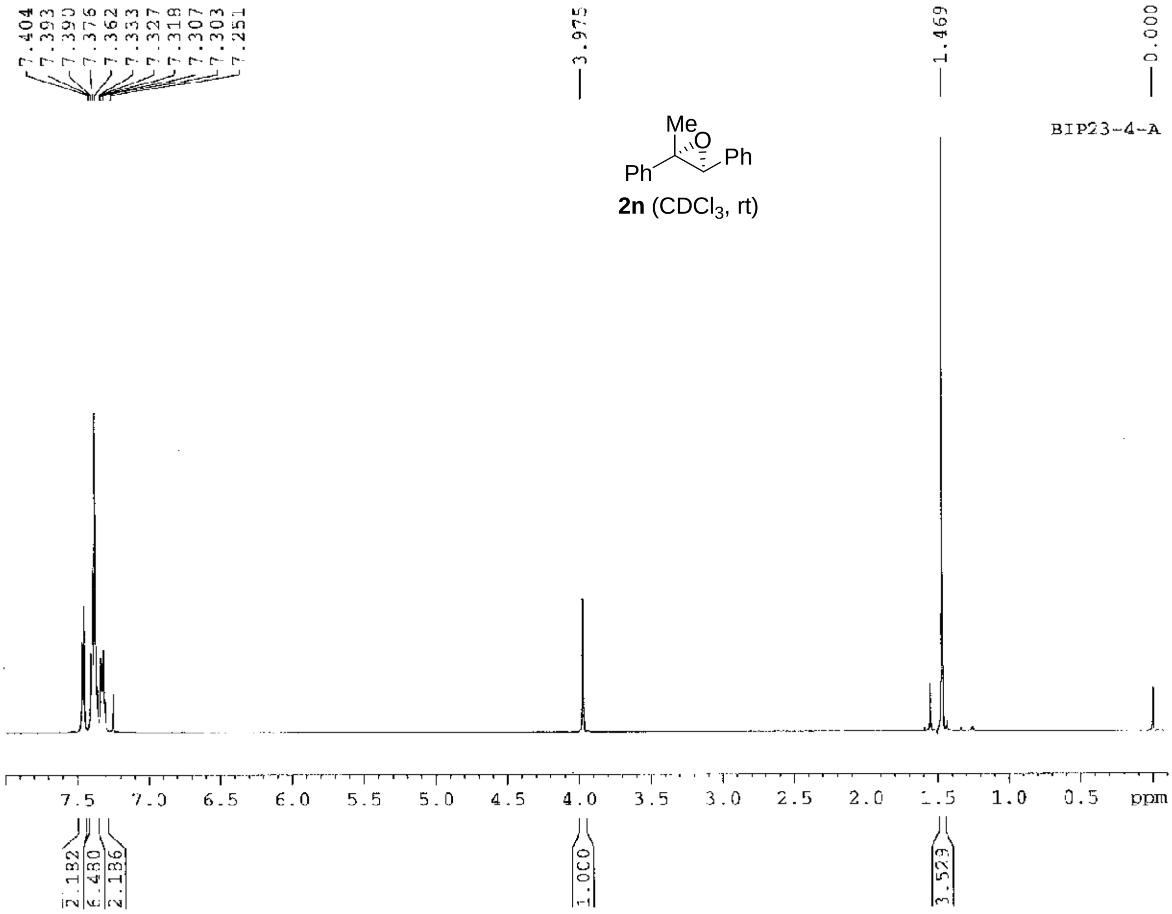
Injection Method: c:\star\yasuhiro\temporaly.mth

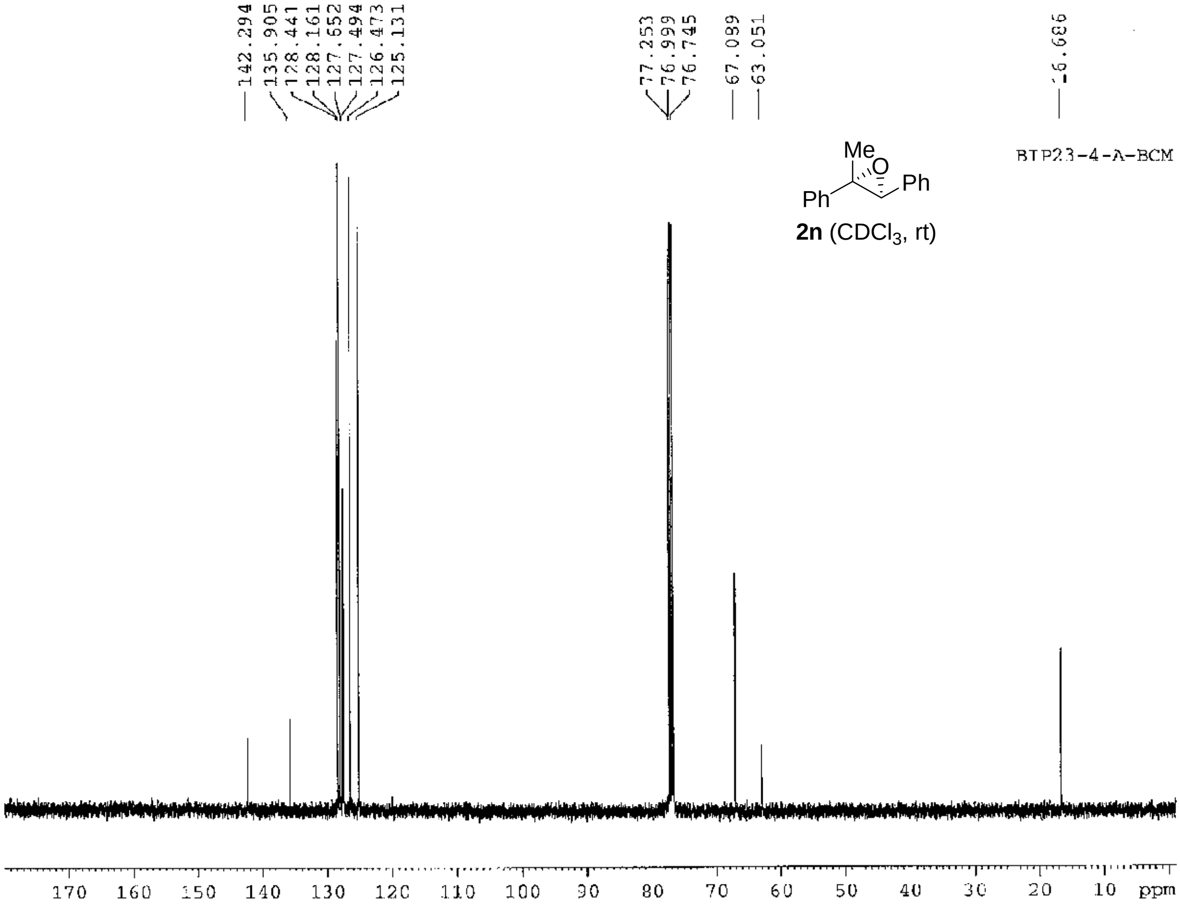




2m

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	52.8417	7.907	0.000	13132324	0.00	BB	10.1
2	47.1582	32.973	0.000	11719851	0.00	BB	48.3
	99.9999		**0.000**	**24852176**			


7.404
7.393
7.390
7.376
7.362
7.333
7.327
7.318
7.307
7.303
7.251
3.975
1.469
0.000
BIP23-4-A
Me
O
Ph
Ph
2n (CDCl3, rt)
ppm
2.182
6.480
2.136
1.000
3.529

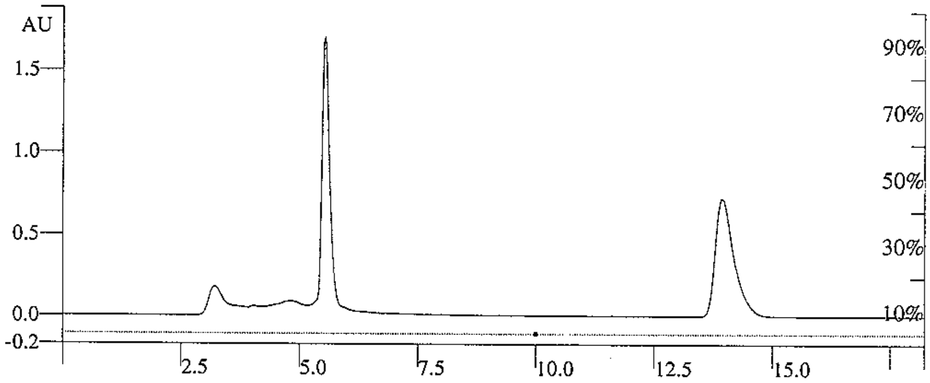

142.294
135.905
128.441
128.161
127.552
127.494
126.473
125.131
77.253
76.999
76.745
67.089
63.051
16.686
BIP23-4-A-BCM
Me
O
Ph
Ph
2n (CDCl3, rt)
ppm

Chiral HPLC analysis of compound **2n**

rac-**2n**

Data File:	c:\star\4-8-11 6;47;14 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP23-rac 225nm	Peak Measurement:	Peak Area
Operator (Inj):	OD-H PrOH/Hex 3/97 FR1.0	Calibration Level:	N/A
Injection Date:	04/08/11 06:47:14 PM	Run Time (min):	18.240

Injection Method: c:\star\yasuhiro\temporaly.mth

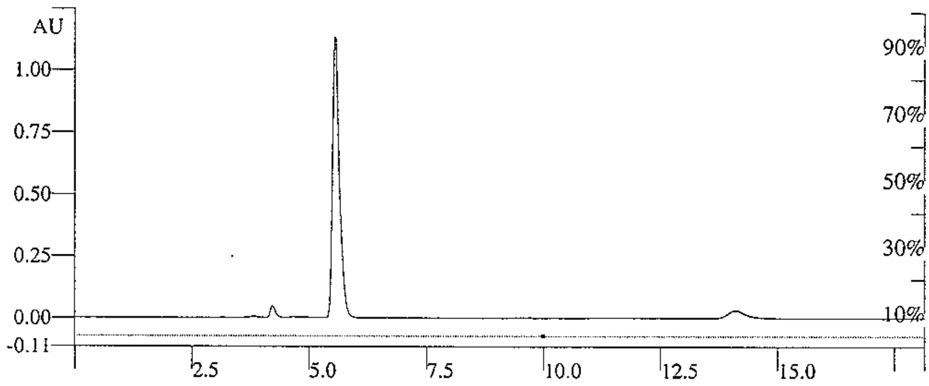


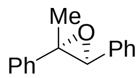
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	49.3615	5.533	0.000	95956504	0.00	BB	10.0
2	50.6385	13.933	0.000	98439104	0.00	BB	24.7
	100.0000		**0.000**	**194395616**			

chiral-**2n**

Data File:	c:\star\4-8-11 6;26;14 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP23-4-A 225nm	Peak Measurement:	Peak Area
Operator (Inj):	OD-H PrOH/Hex 3/97 FR1.0	Calibration Level:	N/A
Injection Date:	04/08/11 06:26:14 PM	Run Time (min):	18.160

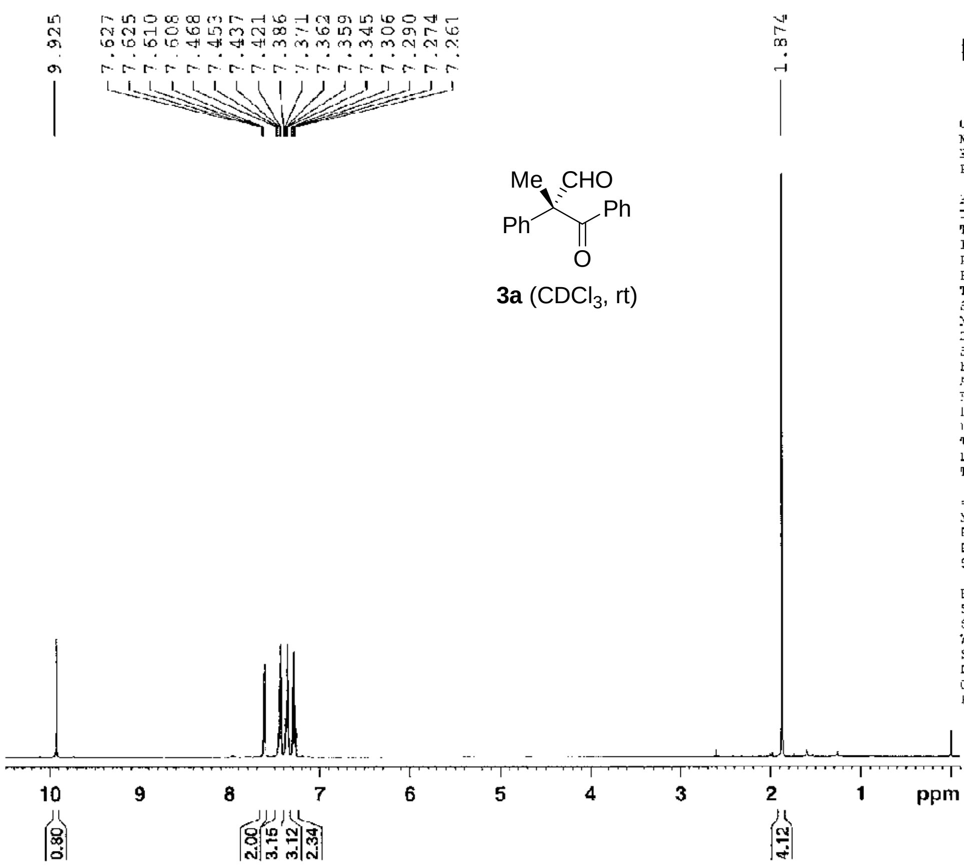
Injection Method: c:\star\yasuhiro\temporaly.mth





2n

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	93.4815	5.533	0.000	59468072	0.00	BB	9.1
2	6.5185	14.093	0.000	4146707	0.00	BB	23.0
	100.0000		**0.000**	**63614780**			



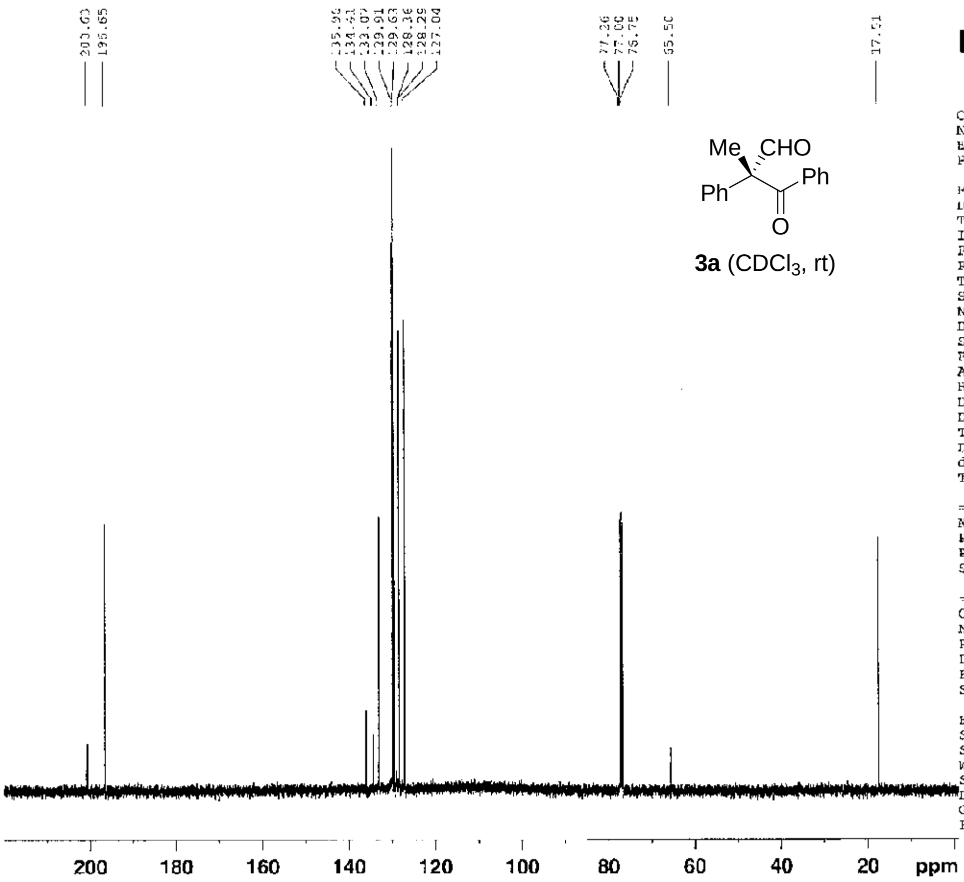
BRUKER

Current Data Parameters
NAME BIP84-1-A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110117
Time 16.11
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 57
DW 50.000 usec
DE 7.50 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz

F2 - Processing parameters
SI 32768
SF 499.8700172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

Current Data Parameters
NAME BIP84-1-A-BCM1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110117
Time 22.50
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT CDCl3
NS 400
DS 0
SWH 37593.984 Hz
FIDRES 0.275697 Hz
AQ 1.8136350 sec
RG 18390.4
DW 13.300 usec
DE 7.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 3.00 dB
SFO1 125.7024664 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz

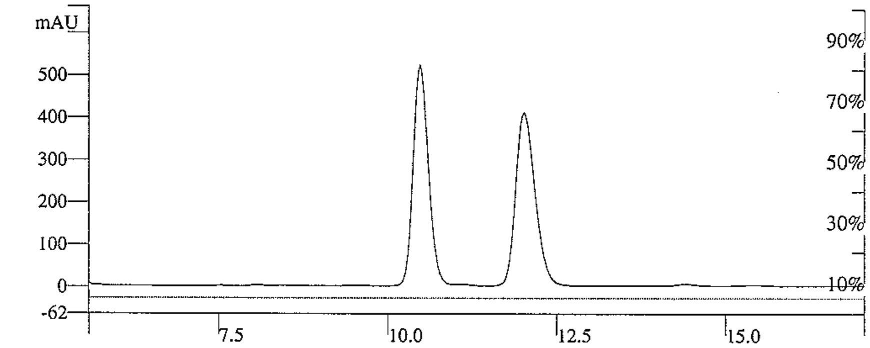
F2 - Processing parameters
SI 32768
SF 125.5924181 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Chiral HPLC analysis of compound **3a**

rac-**3a**

Data File:	c:\star\1-25-11 11;29;22 am -1.run	Run Mode:	Analysis
Sample ID:	BIP84-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 5/95 FR1.0	Calibration Level:	N/A
Injection Date:	01/25/11 11:29:22 AM	Run Time (min):	21.547

Injection Method: c:\star\yasuhiro\temporaly.mth

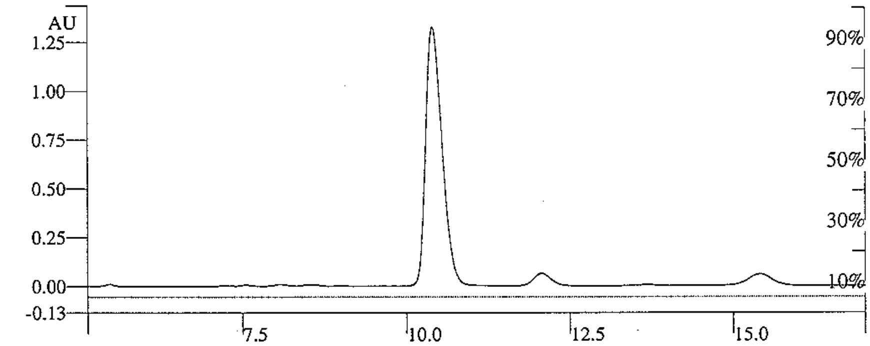


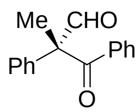
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	49.7899	10.467	0.000	41413496	0.00	BB	14.5
2	50.2101	12.013	0.000	41763036	0.00	BB	18.5
	100.0000		**0.000**	**83176528**			

chiral-**3a**

Data File:	c:\star\1-25-11 2;48;56 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP84-1-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	01/25/11 02:48:56 PM	Run Time (min):	18.907

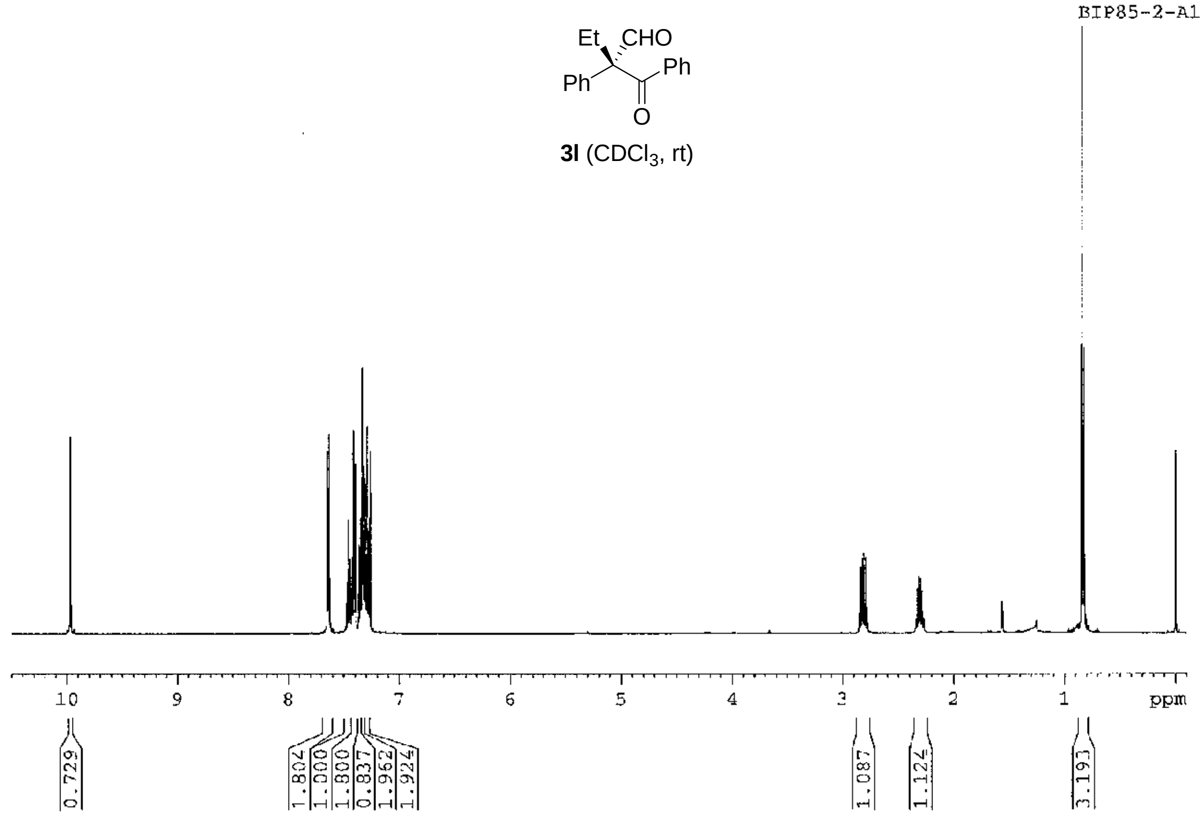
Injection Method: c:\star\yasuhiro\temporaly.mth

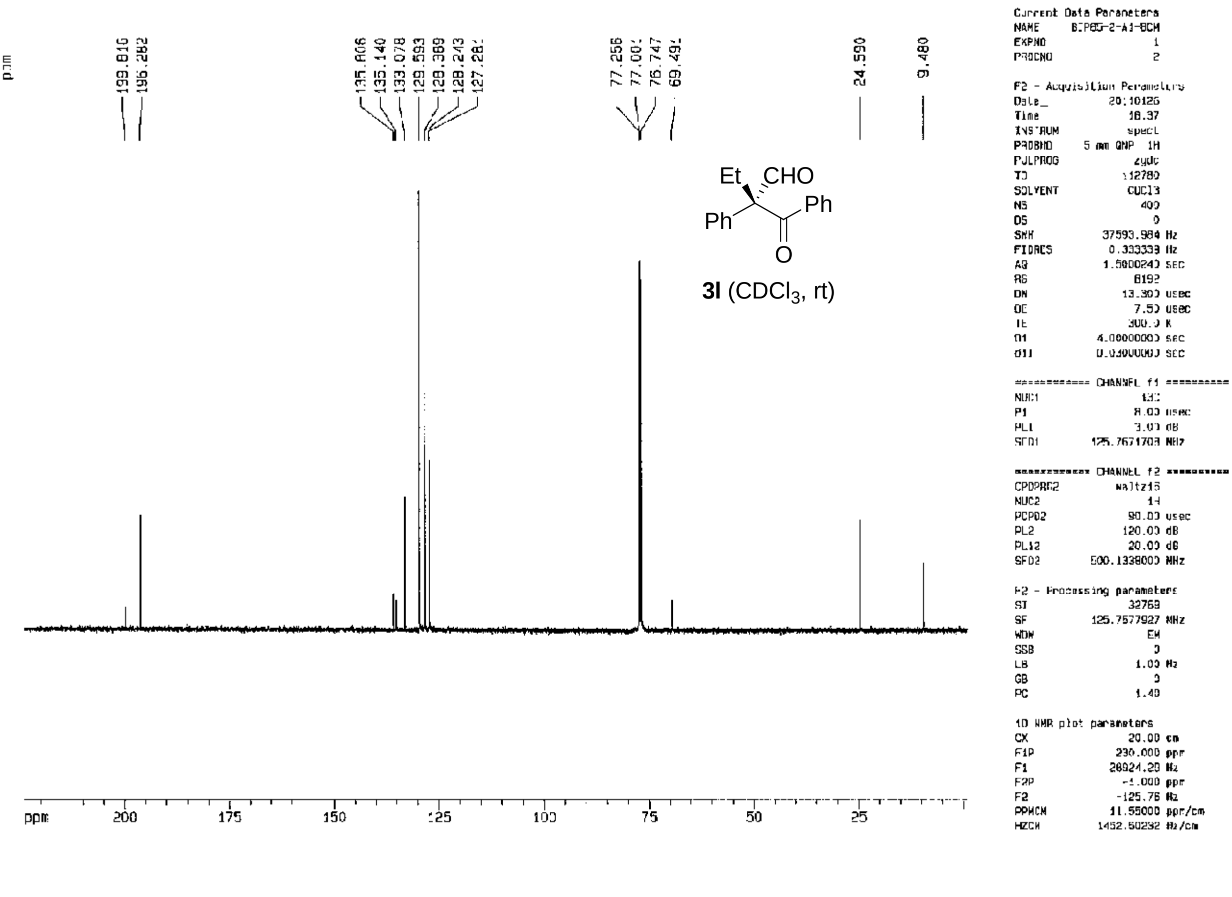




3a

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	95.2032	10.387	0.000	117952592	0.00	BB	16.3
2	4.7968	12.067	0.000	5943005	0.00	BB	16.9
	100.0000		**0.000**	**123895600**			


BIP85-2-A1
Et
CHO
Ph
Ph
O
3l (CDCl3, rt)
10
9
8
7
6
5
4
3
2
1
ppm
0.729
1.804
1.000
1.800
0.837
1.962
1.924
1.087
1.124
3.193

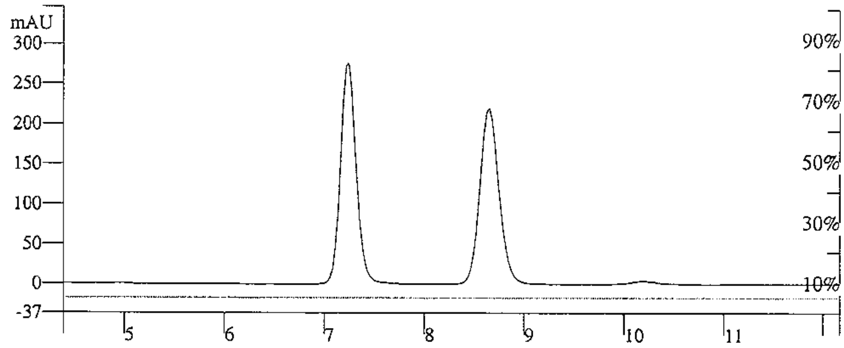

ppm
199.816
196.282
135.806
135.140
133.078
129.593
128.389
128.243
127.28
77.256
77.00
76.747
69.49
24.590
9.480
Et
CHO
Ph
Ph
O
3l (CDCl3, rt)
ppm
200
175
150
125
100
75
50
25
Current Data Parameters
NAME BIP85-2-A1-BCM
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
SOLVENT CDCl3
NS 400
SWH 37593.984 Hz
TE 300.0 K
NUC1 13C
SFO1 125.7671708 MHz
CPDPRG2 waltz16
NUC2 1H
SFO2 500.1338000 MHz
F2 - Processing parameters
SI 32768
SF 125.7577927 MHz
WDW EM
LB 1.00 Hz
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 230.000 ppm
F2 -125.76 Hz
PPMCM 11.55000 ppm/cm
HZCM 1452.50292 Hz/cm

Chiral HPLC analysis of compound 3l

rac-3l

Data File:	c:\star\1-25-11 3;12;47 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP85-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 5/95 FR1.0	Calibration Level:	N/A
Injection Date:	01/25/11 03:12:47 PM	Run Time (min):	15.627

Injection Method: c:\star\yasuhiro\temporaly.mth

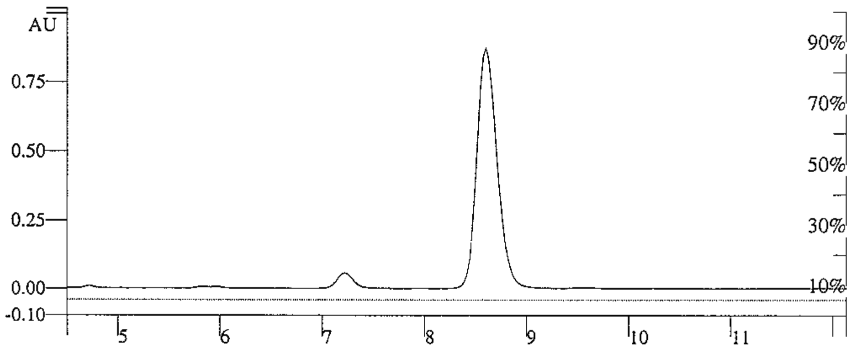


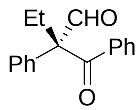
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	50.5380	7.240	0.000	15176258	0.00	BB	9.8
2	49.4620	8.653	0.000	14853138	0.00	BB	12.1
	100.0000		**0.000**	**30029396**			

chiral-3l

Data File:	c:\star\1-25-11 3;36;31 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP85-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	01/25/11 03:36:31 PM	Run Time (min):	13.067

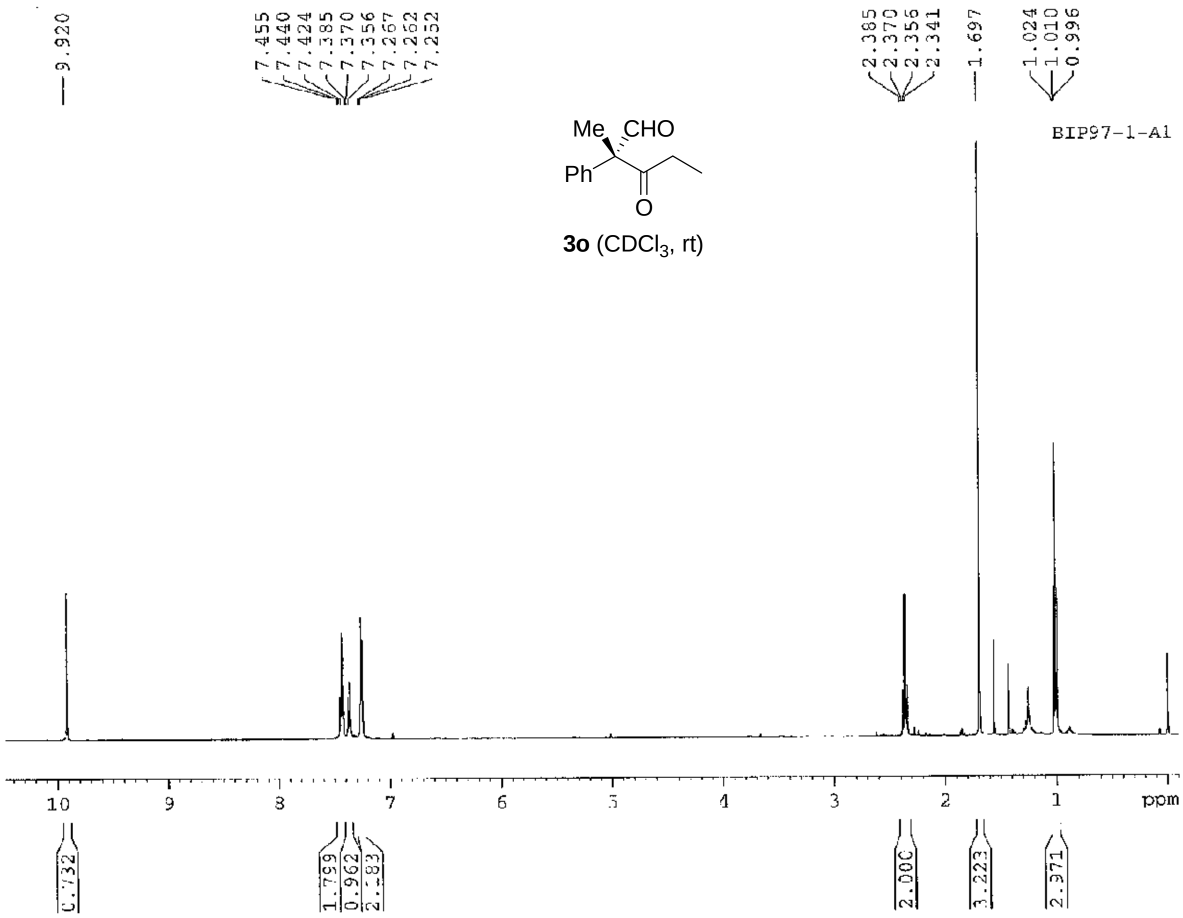
Injection Method: c:\star\yasuhiro\temporaly.mth

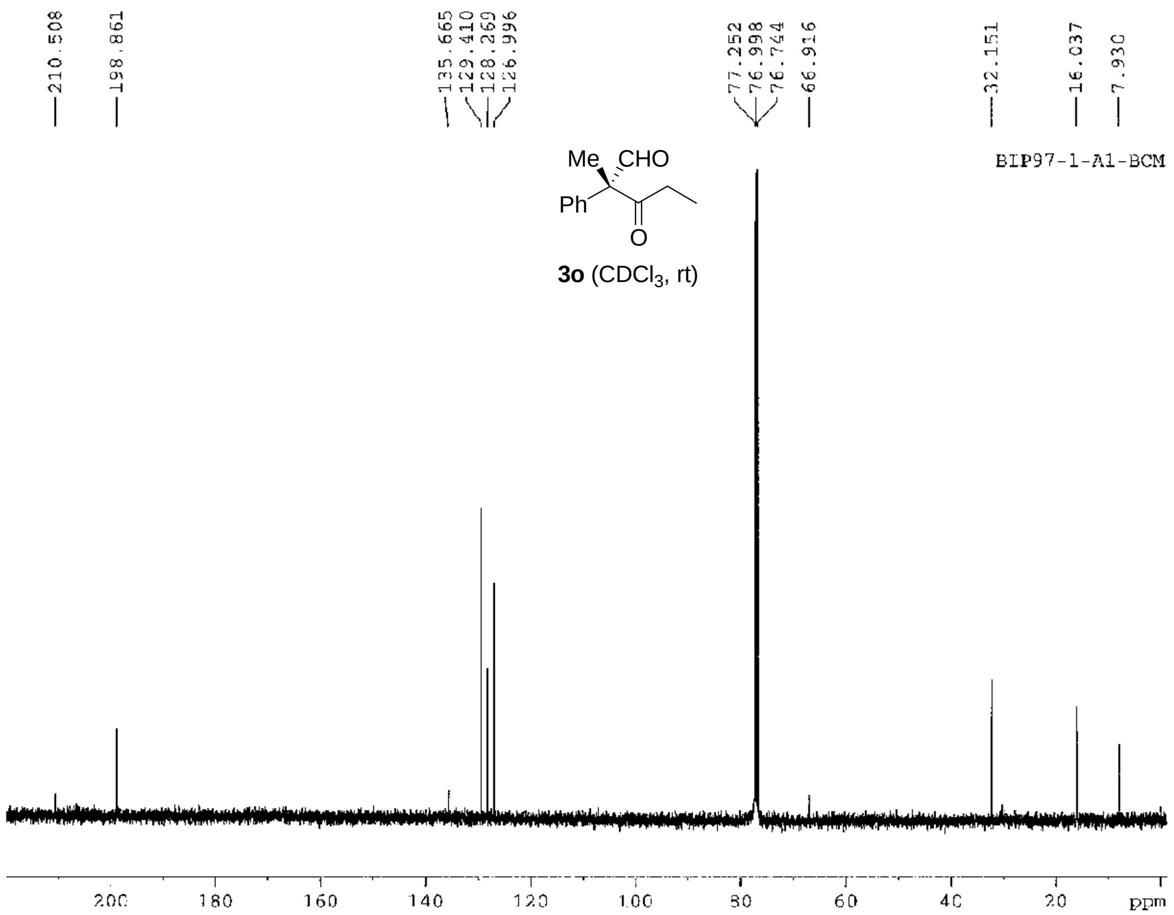




3l

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	4.8491	7.213	0.000	3153613	0.00	BB	10.0
2	95.1509	8.600	0.000	61880960	0.00	BB	12.8
	100.0000		**0.000**	**65034572**			


9.920
7.455
7.440
7.424
7.385
7.370
7.356
7.267
7.262
7.252
2.385
2.370
2.356
2.341
1.697
1.024
1.010
0.996
Me
CHO
Ph
O
3o (CDCl3, rt)
BIP97-1-A1
ppm

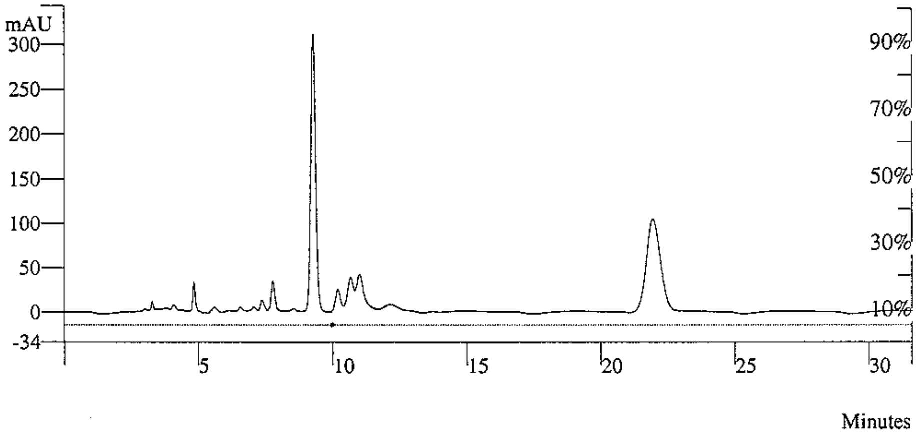

210.508
198.861
135.665
129.410
128.269
126.996
77.252
76.998
76.744
66.916
32.151
16.037
7.930
Me
CHO
Ph
O
3o (CDCl3, rt)
BIP97-1-A1-BCM
ppm

Chiral HPLC analysis of compound 3o

rac-3o

Data File:	c:\star\4-15-11 10;52;21 am -1.run	Run Mode:	Analysis
Sample ID:	BIP-S108-rac 210nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 5/95 FR1.0	Calibration Level:	N/A
Injection Date:	04/15/11 10:52:21 AM	Run Time (min):	31.627

Injection Method: c:\star\yasuhiro\temporaly.mth

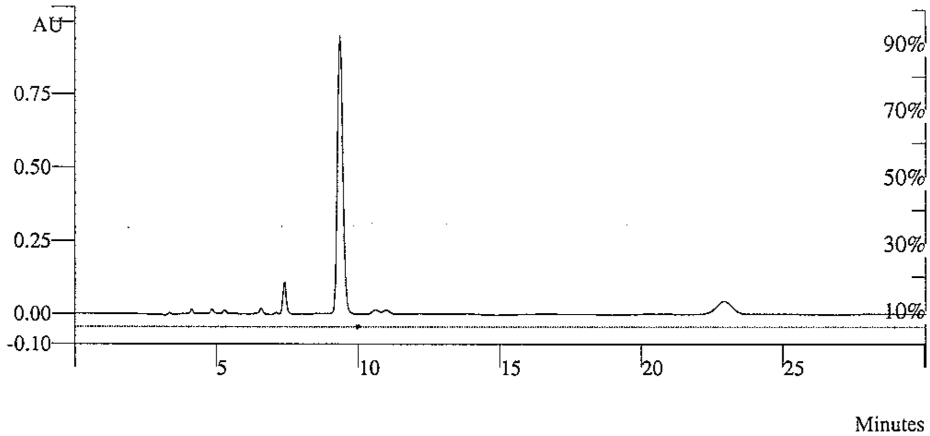


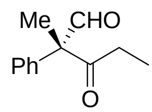
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	50.4974	9.267	0.000	19984194	0.00	BB	11.5
2	49.5026	21.933	0.000	19590520	0.00	BB	34.1
	100.0000		**0.000**	**39574712**			

chiral-3o

Data File:	c:\star\4-19-11 9;54;49 am -1.run	Run Mode:	Analysis
Sample ID:	BIP97-3-A 210nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	04/19/11 09:54:49 AM	Run Time (min):	30.080

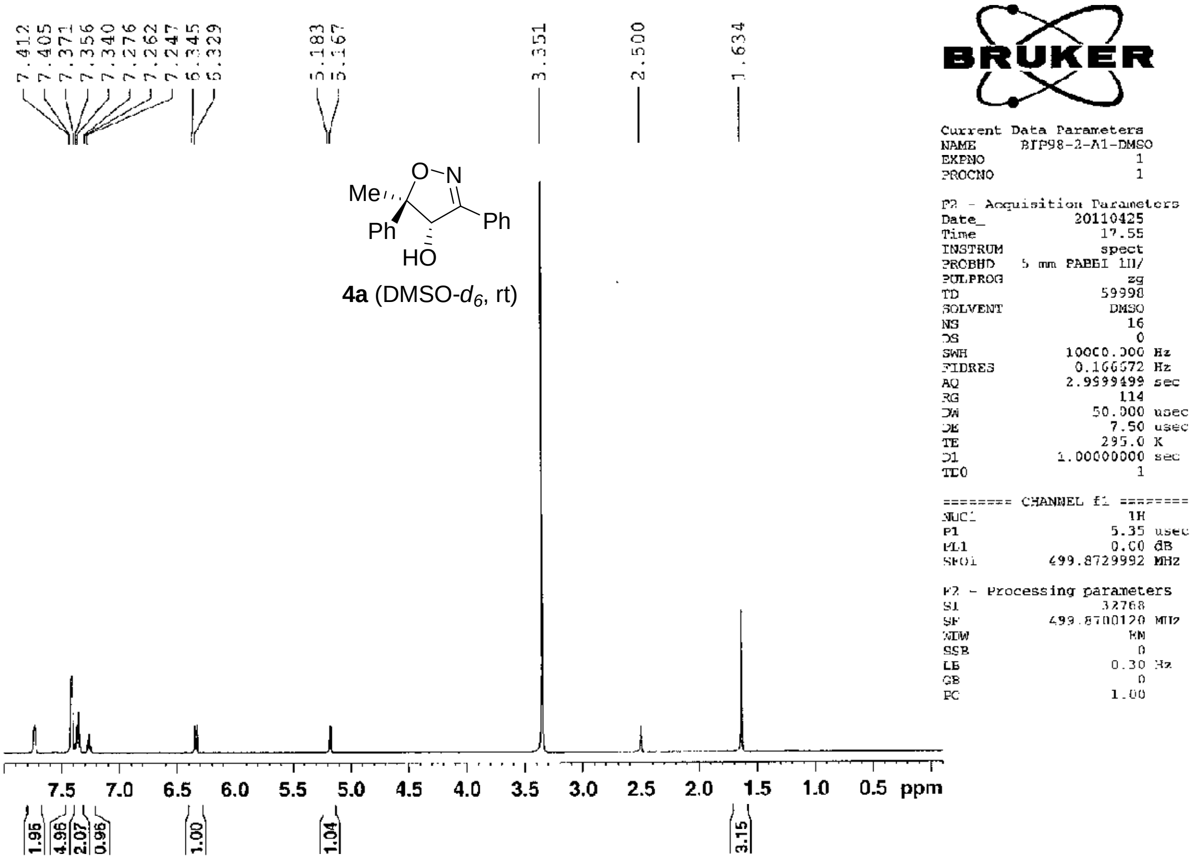
Injection Method: c:\star\yasuhiro\temporaly.mth

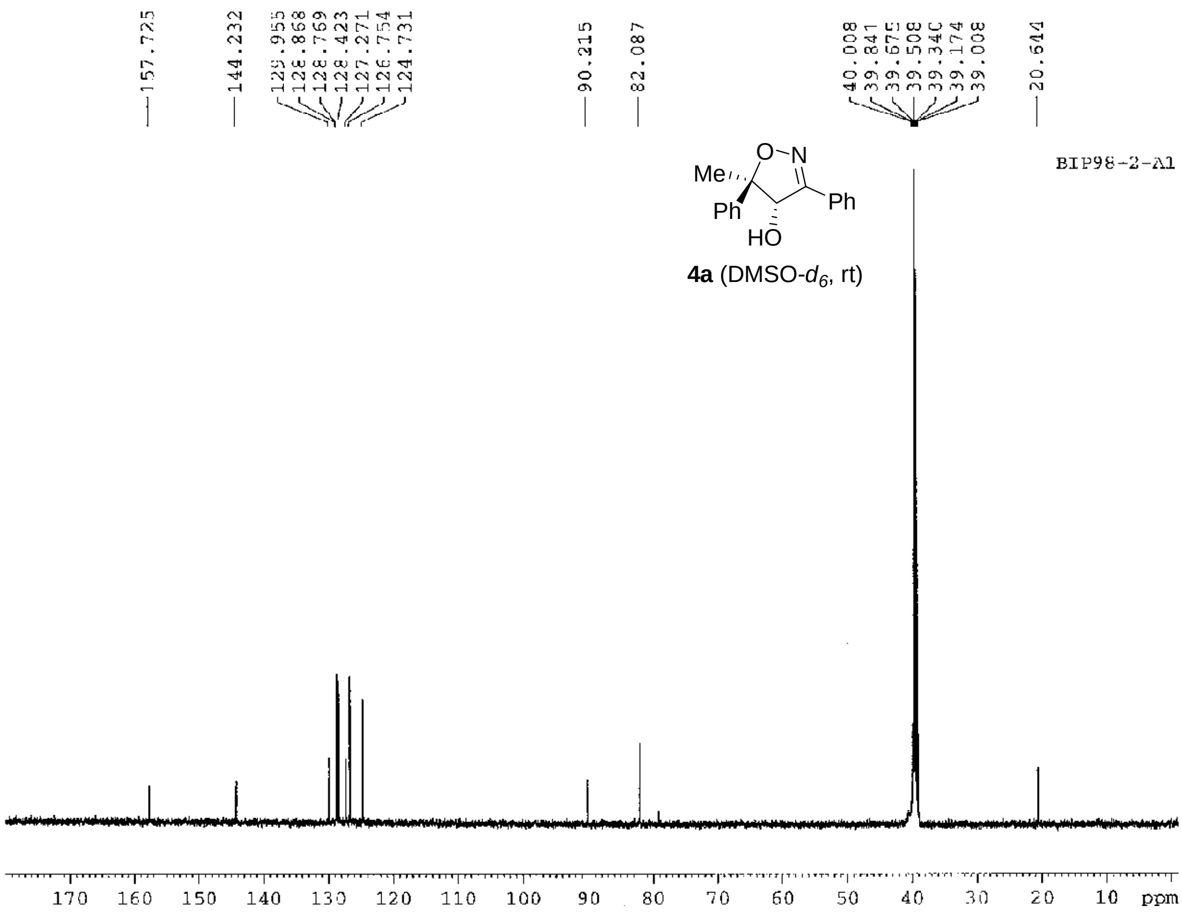




3o

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	88.7151	9.347	0.000	65603192	0.00	BB	12.5
2	11.2849	22.920	0.000	8344957	0.00	BB	35.3
	100.0000		**0.000**	**73948152**			


7.412
7.405
7.371
7.356
7.340
7.276
7.262
7.247
6.345
6.329
5.183
5.167
3.351
2.500
1.634
BRUKER
Current Data Parameters
NAME BIP98-2-A1-DMSO
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110425
Time 17.55
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT DMSO
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 114
DW 50.000 usec
DE 7.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.35 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O–N
Ph
Ph
HO
4a (DMSO-d_6, rt)
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.95
4.96
2.07
0.96
1.00
1.04
3.15

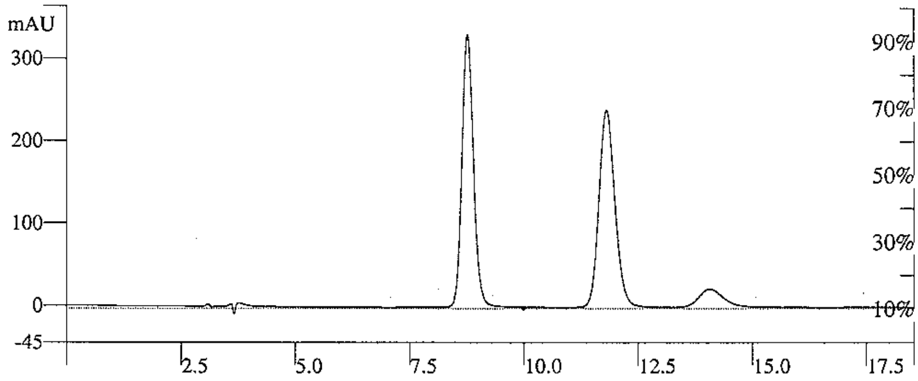

157.725
144.232
129.955
128.868
128.769
128.423
127.271
126.754
124.731
90.215
82.087
40.008
39.841
39.675
39.508
39.340
39.174
39.008
20.644
BIP98-2-A1
Me
O–N
Ph
Ph
HO
4a (DMSO-d_6, rt)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Chiral HPLC analysis of compound **4a**

rac-**4a**

Data File:	c:\star\4-19-11 6;38;34 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP98-rac 254nm	Peak Measurement:	Peak Area
Operator (Inj):	AS-H PrOH/Hex 10/90 FR1.0	Calibration Level:	N/A
Injection Date:	04/19/11 06:38:34 PM	Run Time (min):	18.560

Injection Method: c:\star\yasuhiro\temporaly.mth

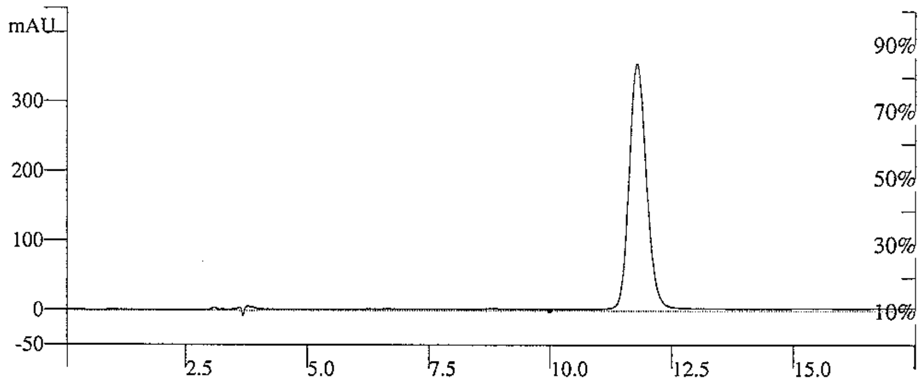


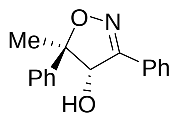
Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	49.9045	8.760	0.000	29551020	0.00	BB	16.0
2	50.0955	11.800	0.000	29664082	0.00	BB	22.5
	100.0000		**0.000**	**59215104**			

chiral-**4a**

Data File:	c:\star\4-19-11 7;23;25 pm -1.run	Run Mode:	Analysis
Sample ID:	BIP98-2-A 254nm	Peak Measurement:	Peak Area
Operator (Inj):		Calibration Level:	N/A
Injection Date:	04/19/11 07:23:25 PM	Run Time (min):	20.640

Injection Method: c:\star\yasuhiro\temporaly.mth





4a

Peak No	Result ()	Ret Time (min)	Time Offset (min)	Peak Area (counts)	Rel Ret Time	Sep. Code	Width 1/2 (sec)
1	0.1278	8.787	0.000	56742	0.00	BB	17.4
2	99.8722	11.773	0.000	44351980	0.00	BB	22.7
	100.0000		**0.000**	**44408720**			